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February 14, 2022

Via CM/ECF

Special Master Thomas P. Scrivo, Esq.
O'Toole Scrivo, LLC
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**Re: *Occidental Chemical Corp. v. 21st Century Fox Am., Inc., et al.*
Civil Action No. 2:18-cv-11273-MCA-LDW**

Dear Special Master Scrivo:

Late Friday, having exhausted every other strategy to avoid their obligation to give sworn testimony, Defendants lobbed in a midnight letter asserting that because “some” Defendants believe they have reached an “agreement in principle” to settle their liability *to the United States*, this entire case should “effectively end.”¹

Not so. This case seeks relief for *OxyChem's* claims for contribution and cost recovery against the Defendants for the pollution *they* caused in the Passaic River. The United States is not a party to this case. It does not own *OxyChem's* claims. And it has no statutory authority to settle or compromise *OxyChem's* claims. In any event, even if a settlement is eventually reached with an *unknown* number of Defendants, on *unknown* terms, the scope, validity, and impact of the “matters addressed” in any lodged settlement under CERCLA is the province of *this Court*, not the United States or the Defendants. *N.J. Dep't of Env'tl. Prot. v. Am. Thermoplastics Corp.*, 974 F.3d 486, 494-95 (3d Cir. 2020).

The rest of the SPG Defendants' letter is equally threadbare. In a transparent attempt to distract attention from their own wrongdoing, they argue again for a stay of their depositions, plainly fearful that their sworn evidence will compound the mountain of proof against them. Their fears are well-founded. Below are just two of *many* possible examples we could cite, showing the abundant evidence *OxyChem* has already uncovered in this case—evidence that *requires* Defendants to explain themselves under oath.

¹ Dkt. 1982 at 1 (emphasis added). Unless otherwise noted, all emphasis is added throughout.

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1. The Sherwin-Williams Company (“Sherwin-Williams”)

As set out in Dkt. 1894-4, Sherwin-Williams destroyed evidence of its polluting operations and waste disposal practices *after* EPA notified it of its potential responsibility for polluting the Passaic River and *after* the company was legally obligated to retain all relevant documents. These are the facts about Sherwin-Williams:

- **January 3, 1995.** EPA sent Sherwin-Williams a CERCLA Section 104(e) request.² That request required Sherwin-Williams to gather, produce, and preserve evidence pertaining to its responsibility for hazardous substances in sediments of the Passaic River, including DDT. To prepare the response, EPA directed Sherwin-Williams to “consult with all current and former employees and agents of your company,” and to produce all relevant documents to EPA. EPA warned Sherwin-Williams of severe consequences if the company’s response was not complete when made and updated regularly with any new information. EPA wrote:

“Be advised that *you are under a continuing obligation to supplement your response* if information not known or available to you as of the date of your submission of your response should later become known or available. *If at any time in the future you obtain or become aware of additional information and/or find that any portion of the submitted information is false, misleading or misrepresents the truth, you must promptly notify EPA. If any part of your response is found to be untrue, you may be subject to criminal prosecution.*”

- **March 2, 1995.** Sherwin-Williams, after consulting its *current* employees and no *former* employees, sent EPA a letter asserting, “No information has been obtained that would indicate the [Sherwin-Williams] Lister Avenue facility ever received, utilized, manufactured, discharged, released or disposed of ... DDT.”³ This “don’t ask, don’t tell” response was not, as explained below, remotely accurate when made. In the same response, Sherwin-Williams admitted it *had* documents regarding its handling of hazardous substances, “including manifest inventory forms and billing records,” all of which were stored at the Lister Avenue facility and all of which would be made available upon request.
- **October 4, 1995.** EPA sent a letter to the Chairman of the Board of Sherwin-Williams notifying the company of its potential liability under CERCLA Section 107 for polluting the Passaic River.⁴ By this point, Sherwin-Williams had not only *promised* to make the documents showing its handling of hazardous substances available to EPA, it had a legal duty to *preserve* them for inspection.⁵

² **Ex. A** (1/3/1995 104(e) request from EPA to J. Breen, Chairman, Sherwin-Williams).

³ **Ex. B** (3/2/1995 letter from D. McConnell, Sherwin-Williams to EPA).

⁴ **Ex. C** (10/4/1995 letter from EPA to J. Breen, Chairman, Sherwin-Williams).

⁵ *See, e.g., Mosaid Techs., Inc. v. Samsung Elec. Co., Ltd.*, 348 F.Supp.2d 332, 336 (D.N.J. 2004) (litigant has “duty to preserve what it knows, or reasonably should know, will likely be requested in a reasonably foreseeable litigation.”)

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- **January 11, 1999.** A Sherwin-Williams consultant interviewed three former and four current employees about the company’s pesticides operations—operations Sherwin-Williams had *denied existed* in its March 1995 104(e) response to EPA. Those interviews confirmed that “*pesticide formulation was conducted at the Newark facility*,” and that “[t]hose pesticides were warehoused on site and shipped from the Newark plant.”⁶ Despite its obligation to do so, Sherwin-Williams never supplemented its 104(e) response to inform EPA of this fact or other evidence it knew about its own pesticide operations.
- **1999.** Also in 1999, after its consultant reported this information, Sherwin-Williams ceased business activities at its Lister Avenue facility and *destroyed its building*. In interrogatory answers in this case, Sherwin-Williams admitted that there was *no document retention policy in place to preserve environmentally related documents in 1999 when “the former facility was decommissioned ... and the facility was demolished.”*⁷

Sherwin-Williams’ failure to disclose and preserve records also is not limited to pesticides or DDT. There is additional evidence that Sherwin-Williams’ *century* of paint, lacquer, and other operations used or formed PCBs, lead, and copper—all contaminants EPA identified as ubiquitous in Passaic River sediments.⁸

Given this record, any attempt to claim there is *no* evidence of Sherwin-Williams’ responsibility for polluting the Passaic River is misleading, at best. The facts above are damning: After it was under a legal duty to preserve documents and disclose all information known to its employees about its pesticide operations, Sherwin-Williams *denied* it handled DDT, *concealed* interviews of former employees about pesticide operations, *destroyed* its building, and *failed to preserve* critical documents, including inventory manifests and billing records. It would be surprising, therefore, to learn Sherwin-Williams was among the unnamed parties with whom EPA has allegedly “agreed to settle in principle.” But even if EPA is unwilling to pursue recoveries from Sherwin-Williams for the United States, Congress gave OxyChem the right to pursue contribution from CERCLA. And OxyChem intends to do so.

2. Givaudan Fragrances Corporation (“Givaudan”)

EPA has assured the New Jersey Attorney General’s office of its unchanged expectation that parties responsible for the presence of dioxins, furans, and/or PCBs in the Passaic River will not be permitted to settle because they are expected to implement the remedy in Operable Unit 2 of the Diamond Alkali Superfund Site.⁹ It would be surprising, therefore, if Givaudan is among the unnamed parties with whom the United States has allegedly settled. As demonstrated below,

⁶ **Ex. D** (1/11/1998 Report from ENSR Consulting to D. Gustafson, Sherwin-Williams) at p. 4.

⁷ **Ex. E** (9/30/2019 Sherwin-Williams Supplemental Responses to Standard Interrogatories) at p. 10.

⁸ *See, e.g., Ex. F* (Hu, et al., *Inadvertent Polychlorinated Biphenyls in Commercial Paint Pigments*, Environ. Sci. Technol., 44 (2010) at 2823; **Ex. G** (5/3/1946 “technical services” document describing use of lead at Newark plant) at p. 3; **Ex. H** (2003 document acknowledging handling of DDT and copper at Newark plant) at p. 35.

⁹ **Ex. I** (9/18/2017 Letter from EPA to Addressees at pg. 1, copied to John Dickinson at N.J. Attorney General’s Office).

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there is abundant evidence of Givaudan's responsibility for dioxin in the Passaic River.

In 2011, an EPA-sponsored study determined there was dioxin in the Passaic River that was not associated with the type of operations conducted at the former Diamond Alkali plant.¹⁰ The highest concentrations of that dioxin type were near Defendant Givaudan's former facility.

Givaudan manufactured two compounds at its plant in Clifton, New Jersey: a disinfectant called G-11 and 2,4,5 Trichlorophenol (also known as TCP). When manufactured in high temperature, alkaline environments, the manufacture of both chemicals can produce dioxin. But Givaudan has asserted for years that it was "highly unlikely" its G-11 and TCP processes produced dioxin, because it claimed it used only "*acidic conditions and low temperatures* in its process."¹¹ Givaudan made the same claim in this case, arguing in its interrogatory answers that "[t]he production of HCP (i.e., G-11) did not create or produce dioxin."¹²

But the evidence here paints a different picture. In 1945 Givaudan developed, and in 1948, Givaudan *patented* a *high temperature, alkaline process to manufacture G-11*.¹³ And while Givaudan told NJDEP that it did not even begin to manufacture G-11 at all until 1947, it produced in this case a 1941 batch process recipe for a "sodium salt method" to manufacture G-11 that requires high, not low, temperatures.¹⁴ The same document reflects that this "sodium salt method" could produce *half a ton per day* of G-11. As late as 1979, other documents show Givaudan's acid-based process for G-11 was *also* conducted at temperatures of 130° Celsius, not at low temperature as Givaudan claimed.¹⁵

Givaudan also manufactured 300,000 pounds of TCP.¹⁶ Its 1948 batch process recipe for manufacturing TCP calls for the addition of 312 *pounds* of caustic soda flakes to the kettle, after which the temperature is raised to 175° Celsius and held there for *four hours*.¹⁷ This recipe's *high temperatures* all occur when the solution is in an *alkaline* condition. Based on the methodology used in a 2017 paper examining how much dioxin could be generated by this type of high temperature, alkaline process, Givaudan's manufacture of TCP could have generated between 11 and 25 *kilograms* of dioxin.¹⁸

And where did it go? In a supplemental Section 104(e) response, Givaudan claimed:

The alleged existence of a possible surface water pathway that could have conveyed storm water flow . . . directly to the Passaic River is *not* supported by the historical

¹⁰ See **Ex. J** (Garvey et al., "Dioxin in the Passaic River (NJ), The Case for Two Dioxin Sources" (Feb. 10, 2011)).

¹¹ See **Ex. K** (Excerpt from Supplemental 104(e) response (2016)) at 7; **Ex. L** (Section 104(e) Response (1983)) at 2.

¹² See **Ex. M** (Givaudan Response to Standard Interrogatory No. 11) at 23.

¹³ See **Ex. N** (1945 "Improved Process for the Manufacture of Compound G-11") & **Ex. O** (1948 Patent).

¹⁴ See **Ex. P** (1941 "G-11 Process (Sodium Salt Method)" (GIV_NBC_0664675)).

¹⁵ See **Ex. Q** (1979 "G-11 N.P." Process) at "Step No. 17."

¹⁶ See **Ex. L** (104(e) Resp. (1983)) at 2 ("305,000 pounds of 'pre-purified' 2,4,5-TCP" produced in 1948 and 1949).

¹⁷ See **Ex. R** (1948 Process (GIVA-FED-0000342825)) at GIVA-FED-0000342847.

¹⁸ See **Ex. S** (Parette et al., "Modeling the formation of 2378-tetrachlorodibenzo-p-dioxin in the historical manufacture of 2,4,5-trichlorophenol," *Environmental Forensics*, 18:4, 307-317 (2017)).

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aerial photo review, or the digital topography evidence completed on the historical photos. There is no evidence of a defined drainage swale either on or off the property to the Passaic River in any of the historical aerial photos.¹⁹

Givaudan focused attention on “aerial photos” to support its claim that there was “no evidence” of drainage swales that drained into the Passaic River. But there is other evidence—including Givaudan’s own maps and consultant reports—showing there *were* surface swales on an area of Givaudan’s property heavily contaminated with a form of dioxin called TCDD, swales that drained stormwater into an outfall on the Passaic River. Those swales are reflected on a “Givaudan Corporation” drainage map dated June 30, 1970,²⁰ a map produced for the first time in this CERCLA case, contradicting Givaudan’s claim that there is “no evidence of a defined drainage swale either on or off the property to the Passaic River.” Another report documented 12,700 gallons of stormwater per minute exiting and flowing into the Passaic River in a 25-year storm.²¹

3. Defendants Still Have No *Valid* Ground for any Stay.

The evidence cited above proves one critical fact: subpoena power and sworn testimony matter. We know these facts about Givaudan *now* because OxyChem’s *litigation* compelled Givaudan to produce 388,106 pages of records of its former operations. This is far more information than Givaudan gave to EPA, and it contradicts Givaudan’s claims in its 2016 supplemental Section 104(e) response to EPA. We know the facts about Sherwin-Williams *now* because it was compelled, through this litigation, to produce the documents and answer sworn interrogatories in which it was compelled to admit it failed to preserve critical environmental documents. Defendants would plainly *prefer* to settle before these, and other uncomfortable truths come out about their wrongdoing. But their preference is *not* a ground on which this Court should stay the case. To the contrary, the depositions scheduled to begin in March are essential. They will reveal even more of the truth about Defendants’ wrongful actions, providing evidence that the Court can evaluate in assessing each party’s fair and equitable share of responsibility.

The same evidence shows why Defendants’ attacks on OxyChem’s alleged “true share of responsibility” (Dkt. 1982) are rhetoric that doesn’t match reality. ***EPA has admitted OxyChem never polluted the Passaic River.*** OxyChem’s sole alleged basis of liability here is its merger with Diamond Shamrock Chemicals Company, long after the Lister Plant closed, in a transaction where the Seller retained liability for that Plant and gave OxyChem a robust indemnity agreement to “hold harmless” OxyChem against all liabilities related to the Diamond Alkali Superfund Site. The evidence also shows that when those indemnitors collapsed into bankruptcy years later, OxyChem immediately stepped up *alone* to design the remedy for OU 2 at an estimated cost of \$165 million. OxyChem has performed well and cooperatively with EPA ever since, most recently by offering to *expand* its leadership to include the design and performance of the \$441 million interim remedy at OU4 *so long as* the Defendants who polluted the river are not permitted to evade *their* responsibility for the pollution *they caused* and that OxyChem did not.

¹⁹ See **Ex. K** (Excerpt from Supplemental 104(e) response (2016)) at 3.

²⁰ See **Ex. T** (Drainage Map (KLL0430050001)).

²¹ See **Ex. U** (May 1993 “Givaudan-Roure; River Road - Water Problem Report”) at GIVA-FED-0000092764.

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This is how CERCLA is supposed to work. Congress enacted CERCLA, and gave parties like OxyChem contribution rights, to encourage them to do exactly what OxyChem has done here: perform and then recover from others their fair share of costs. Congress chose the Court—not EPA—to make a fair and equitable allocation of responsibility, based on the evidence, because an *evidence-based* assessment is the only way to ensure *every* party pays what they owe.

The United States is free to settle its own claims. When all the evidence is known, settlement may make sense for many reasons. But the United States is not free to insulate Defendants Sherwin-Williams, Givaudan, and others from their responsibility to OxyChem for costs *OxyChem* has incurred and will continue to incur to clean up *their* pollution, which OxyChem *did not* cause, and which has burdened the Passaic River and vulnerable communities in the Ironbound for over a century.

Settlement by some Defendants will not “end this litigation.” It cannot and it should not. Depositions should proceed as scheduled. It is time Defendants tell the truth, under oath.

Respectfully submitted,



Kathy D. Patrick

/s/ John J. McDermott

John J. McDermott

cc: All counsel of record

Exhibit A

03 JAN 1995

EXPRESS MAIL
RETURN RECEIPT REQUESTED

John G. Breen, Chairman of the Board
 Sherwin Williams
 101 Prospect Avenue, NW
 Cleveland, OH 44115

Re: Request for Information Under 42 U.S.C. §9601 et seq.
 Diamond Alkali Superfund Site, Passaic River Study Area

Dear Mr. Breen:

The United States Environmental Protection Agency ("EPA") is investigating the presence of hazardous substances in the sediments of the Passaic River. EPA is charged with responding to the release and/or threatened release of hazardous substances, pollutants and contaminants into the environment and with enforcement responsibilities under the Comprehensive Environmental Response, Compensation and Liability Act of 1980, as amended ("CERCLA"), 42 U.S.C. §9601 et seq.

In this "Request for Information", EPA requests information concerning the nature and quantity of certain materials (hazardous substances and hazardous waste, as those terms are defined at Section 101(14) of CERCLA, 42 U.S.C. §9601(14), and Section 1004 of the Resource Conservation and Recovery Act ("RCRA"), 42 U.S.C. §6903 respectively) which may have been generated, treated, stored, or disposed at your facility located at 60 Lister Avenue in Newark, New Jersey. EPA makes its request pursuant to Section 104 of CERCLA, 42 U.S.C. §9604.

Pursuant to these statutory provisions, EPA hereby requires that you provide the information requested in Attachment A of this letter, and include, as required, the "Certification of Answers to Request for Information," with your notarized signature.

In preparing your response to this "Request for Information," please follow the instructions provided in Attachment B.

AKF000001

SYMBOL ---->	NJSB2/N	ORC	NJSB2/N	NJSB2	NJP/DD	ERRD/DD	ERRD
SURNAME -->	RICHMAN	NICH	DIFORTE	BASSO	TRISCO	PAVLOU	CALLAHAN
DATE ----->	10/10	12/22	NMD 12/22	ASB 12/22	ASB 12/22	ASB 12/25	

Your response to this "Request for Information" should be postmarked or received by EPA within thirty (30) calendar days of your receipt of this letter. Your response should be mailed to:

Mr. Lance R. Richman, P.G.
Emergency and Remedial Response Division
U.S. Environmental Protection Agency
26 Federal Plaza, Room 13-100
New York, New York 10278

with a copy to Ms. Patricia C. Hick, Assistant Regional Counsel, Office of Regional Counsel, Room 310 at the same address.

Your failure to respond to this "Request for Information" within the time specified above may subject you to an enforcement action under Section 104(e)(5) of CERCLA, 42 U.S.C. §9604(e)(5). An enforcement action may include the assessment of penalties of up to \$25,000 for each day of continued noncompliance.

Be advised that you are under a continuing obligation to supplement your response if information not known or not available to you as of the date of submission of your response should later become known or available. If at any time in the future you obtain or become aware of additional information and/or find that any portion of the submitted information is false, misleading or misrepresents the truth, you must promptly notify EPA. If any part of your response is found to be untrue, you may be subject to criminal prosecution.

If desired, you may assert a business confidentiality claim covering all or part of the information requested by this letter. The claim must be supported by each of the four factors specified in Section 104(e)(7)(E) of CERCLA, 42 U.S.C. §9604(e)(7)(E), and must be asserted at the time of submission, by placing on (or attaching to) the information a cover sheet, stamped or typed legend, or other suitable form of notice employing language such as "trade secret", or "proprietary", or "company confidential". Information covered by such a claim will be disclosed by EPA only to the extent and by means of procedures set forth in Title 40 C.F.R. Part 2, Subpart B. If no such claim accompanies the information when it is received by EPA, it may be made available to the public by EPA without further notice to you.

If you have any questions concerning this "Request for Information," please contact Mr. Richman, of my staff, at (212) 264-6695 or Ms. Hick at (212) 264-2642. Inquiries from attorneys should be directed to Ms. Hick. Your cooperation is appreciated.

Sincerely yours,

Kathleen Callahan, Director
Emergency and Remedial Response Division

Enclosures

CERTIFICATION OF ANSWERS TO REQUEST FOR INFORMATION

State of _____:

County of _____:

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this document (response to EPA Request for Information) and all documents submitted herewith, and that based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate, and complete, and that all documents submitted herewith are complete and authentic unless otherwise indicated. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment. I am also aware that my company is under a continuing obligation to supplement its response to EPA's Request for Information if any additional information relevant to the matters addressed in EPA's Request for Information or the company's response thereto should become known or available to the company.

NAME (print or type)

TITLE (print or type)

SIGNATURE

Sworn to before me this
day of _____, 19____

Notary Public

AKF000004

sherwin-williams company

ATTACHMENT A

REQUEST FOR INFORMATION

Background

The United States Environmental Protection Agency ("EPA") is investigating the release of hazardous substances into the Passaic River. EPA has information indicating that hazardous substances from your facility located at 60 Lister Avenue in Newark, New Jersey may have been discharged into the Passaic River.

Provide the information requested below, including copies of all available documentation that supports your answers.

1) How long has your company operated at the facility designated above? If your company no longer operates at this facility, during what years did your company operate at the facility?

2) a) Does your company have or has it in the past had a permit or permits issued pursuant to the Resource Conservation and Recovery Act, 42 U.S.C. §6901 et seq.? If "yes", please provide the years that your company held such a permit and its EPA Identification Number.

b) Does your company have or has it in the past had a permit or permits issued pursuant to the Federal Water Pollution Control Act, 33 U.S.C. § 1251, et seq.? If "yes", please provide the years that your company held such a permit.

3) Did your company receive, utilize, manufacture, discharge, release or dispose of any materials containing the following substances:

	Yes	No
2,3,7,8 tetrachlorodibenzo-p-dioxin or other dioxin compounds	—	—
Pentachlorophenol	—	—
dichlorodiphenyl-trichloroethane (DDT)	—	—
Cadmium	—	—
Copper	—	—
Mercury	—	—
Lead	—	—
Zinc	—	—
Methyl ethyl ketone	—	—
ethyl benzene	—	—
Toluene	—	—
Xylene	—	—

4) a) Provide a description of the manufacturing processes for which all hazardous substances, including, but not limited to, the substances listed in response to item (3), were a product or by-product.

b) During what parts of the manufacturing processes identified in the response to items (4)(a), above, were hazardous substances, including, but not limited to, the substances listed in response to item (3), generated?

i) Describe the chemical composition of these hazardous substances.

ii) For each process, what amount of hazardous substances was generated per volume of finished product?

iii) Were these hazardous substances combined with wastes from other processes? If so, wastes from what processes?

5) Describe the methods of collection, storage, treatment, and disposal of all hazardous substances, including, but not limited to, the substances listed in response to item (3) and (4). Include information on the following:

a) Identify all persons who arraigned for and managed the processing, treatment and disposal of hazardous substances.

b) If hazardous substances were taken off-site by a hauler or transporter, provide the names and addresses of the waste haulers and the disposal site locations.

c) Describe all storage practices employed by your company with respect to all hazardous substances from the time operations commenced until the present. Include all on-site and off-site storage activities.

6) a) For process waste waters generated at the facility which contained any hazardous substances, including, but not limited to, the substances listed in response to item (3) and (4):

i) Did the waste stream connect to a sanitary sewer and if so, during what years?

ii) Were they treated before being discharged to the sanitary sewer and if so, how?

iii) If the waste waters were not discharged to the sanitary sewer, where were they disposed and during what years?

b) For floor drains or other disposal drains at the facility:

i) Did the waste stream connect to a sanitary sewer and if so, during what years?

ii) Was the waste stream treated before being discharged to the sanitary sewer and if so, how?

iii) If the floor drains or other disposal drains at the facility were not discharged to the sanitary sewer, where did they discharge and during what years?

c) i) Did any storm sewers, catch basins or lagoons exist at any time at the facility and if so, during what years?

ii) If catch basins or lagoons existed, were they lined or un-lined?

iii) Where was the discharge from any of these structures released and during what years? Was this discharge treated before its release and if so, how and during what years?

d) Please supply diagrams of any waste water collection or disposal systems on the property.

7) a) For each hazardous substances, including, but not limited to, the substances listed in response to item (3) or identified in the responses to item (4), above, provide the total amount generated during the operation of the facility on an annual basis.

b) Were any hazardous substances, including, but not limited to, the substances listed in response to item (3) or identified in the responses to item (4), above, disposed of in the Passaic River or discharged to the Passaic River? If yes, estimate the amount of material discharged to or disposed of in the Passaic River and the frequency with which this discharge or disposal occurred.

8) Please identify any leaks, spills, explosions, fires, or other incidents of accidental material discharge that occurred at the facility during which or as a result of which any hazardous substances, including, but not limited to, the substances listed in response to item (3) or (4), were released on the property of the facility or discharged to the Passaic River. Provide any documents or information relating to these incidents.

9) Provide the date of any leaks, spills, explosions, fires or other incidents of accidental material discharge of any hazardous substances, including, but not limited to, the substances listed in response to item (3) and (4), on the property or into the waste water discharge system at the facility. Provide details of the ultimate disposal of any contaminated materials.

10) a) Was your facility ever subject to flooding. If so, was the flooding due to:

i) overflow from sanitary or storm sewer back-up, and/or

ii) flood overflow from the Passaic River?

b) Please provide the date and duration of each flood event.

11) Please provide a detailed description of any civil, criminal or administrative proceedings against your company for violations of any local, State or federal laws or regulations relating to water pollution or hazardous waste generation, storage, transport or disposal. Provide copies of all pleadings and depositions or other testimony given in these proceedings.

12) Provide a copy of each document which relates to the generation, purchase, use, handling, hauling, and/or disposal of all hazardous substances, including, but not limited to, the substances listed in response to item (3) and (4). If you are unable to provide a copy of any document, then identify the document by describing the nature of the document (e.g. letter, file memo, invoice, inventory form, billing record, hazardous waste manifest, etc.) Describe the relevant information contained therein. Identify by name and job title the person who prepared the document. If the document is not readily available, state where it is stored, maintained, or why it is unavailable.

13) Provide all other documents pertaining to the results of any analyses of ground water, surface water, ambient air, and any other environmental media performed at the facility.

14) a) Has your company owned the facility at the location designated above? If so, from whom did your company purchase the property and in what year? If your company subsequently sold the property, to whom did your company sell it and in what year? Please provide copies of any deeds and documents of sale.

b) If your company did not own the facility, from whom did your company rent the facility and for what years? Please provide copies of any rental agreements.

c) To the extent that you know, please provide the names of all parties who owned or operated the facility during the period from 1940 through the present. Describe the relationship, if any, of each of those parties with your company.

15) Answer the following questions regarding your business or company. In identifying a company that no longer exists, provide all the information requested, except for the agent for service of process. If your company did business under more than one name, list each name.

- a) State the legal name of your company.
- b) State the name and address of the president or the chairman of the board, or other presiding officers of your company.
- c) Identify the state of incorporation of your company and your company's agent for service of process in the state of incorporation and in New Jersey.
- d) Provide a copy of your company's "Certificate of Incorporation" and any amendments thereto.
- e) If your company is a subsidiary or affiliate of another company, or has subsidiaries, or is a successor to another company, identify these related companies. For each related company, describe the relationship to your company; indicate the date and manner in which each relationship was established.
- f) Identify any predecessor organization and the dates that such company became part of your company.
- g) Identify any other companies which were acquired by your company or merged with your company.
- h) Identify the date of incorporation, state of incorporation, agents for service of process in the state of incorporation and New Jersey, and nature of business activity, for each company identified the responses to items (11)(e), (f), and (g), above.
- i) Identify all previous owners or parent companies, address, and the date change in ownership occurred.

16) Provide the name, address, telephone number, title and occupation of the person(s) answering this "Request for Information" and state whether such person(s) has personal knowledge of the responses. In addition, identify each person who assisted in any way in responding to the "Request for Information" and specify the question to which each person assisted in responding.

ATTACHMENT B

INSTRUCTIONS FOR RESPONDING TO REQUEST FOR INFORMATION

1. A complete separate response must be made to each individual question in this "Request for Information".
2. Precede each answer with the number of the question to which it is addressed.
3. In preparing your response to each question, consult with all current or former employees and agents of your company who may be familiar with the matter to which the question pertains.
4. Interpret "and" as well as "or" to include within the scope of the question as much information as possible. If two interpretations of a question are possible, use the one that provides more information.
5. If you are unable to give a detailed and complete answer or to provide any of the information or documents requested, indicate the reasons for your inability to do so.
6. If you have reason to believe that an individual other than one employed by your company may be able to provide additional details or documentation in response to any question, state that person's name, last known address, phone number and the reasons for your belief.
7. For each document produced in response to this "Request for Information", indicate on the document, or in some other reasonable manner, the number of the question to which it applies.
8. If anything is deleted from a document produced in response to this "Request for Information", state the reason for, and the subject matter of, the deletion.
9. Provide all documents that relate to each question. If a document is requested but is not available, state the reason for its unavailability. In addition, to the best of your ability, identify any such document by author, date, subject matter, number of pages, and all recipients and their addresses.
10. As used herein "relate to" or "relating to" means constituting, defining, containing, embodying, reflecting, identifying, stating, referring to, dealing with, or in any way pertaining to. "Document" as used herein means any recording of information in tangible form, including memoranda, handwritten notes, invoices, checks, manifests, tape recordings, computer databases, or any tangible or physical objects however produced or reproduced upon which words or other information are affixed or recorded or from

which by appropriate transcription written matter or a tangible thing may be produced.

11. Whenever in this "Request for Information" there is a request to identify a person or an entity other than a person, state the person or entity's full name, last known employment, present or last known home address, and telephone number.

12. As used herein, the term "facility," "hazardous substance," "person," and "release" shall have the meaning set forth in Section 101(9), (14), (21) and (22) of CERCLA, 42 U.S.C. §9601(9), (14), (21), and (22), respectively.

13. In answering these questions, every source of information to which you have access should be consulted, regardless of whether the source is in your immediate possession or control. All documents or other information, including records of all types of manufacturing, treatment, transportation or disposal operations, in your possession or in the possession of the Corporation should be consulted. If you do not have access to certain information and/or documents, state the nature of this information and/or documents, and indicate in whose possession they can be found.

Exhibit B



The Sherwin-Williams Company
101 Prospect Avenue, N.W.
Cleveland, Ohio 44115-1075

March 2, 1995

VIA FEDERAL EXPRESS

Patricia Hick, Esq.
United States Environmental Protection Agency
Office of Regional Counsel
New Jersey Superfund Branch
290 Broadway, 17th Floor
New York, NY 10007-1866

MAR 03 1995

RECEIVED

Re: Request for Information Regarding the Diamond Alkali Superfund Site
Passaic River Study Area, New Jersey

Dear Ms. Hick:

This letter is in response to the Request for Information regarding the above-captioned site sent to The Sherwin-Williams Company for its facility which was located at 60 Lister Avenue in Newark, New Jersey. Sherwin-Williams understands this response to be timely submitted and reserves its rights to submit additional or new information if obtained. Without waiving any rights or privileges and not admitting to any facts or liabilities, Sherwin-Williams responds as follows:

1. Sherwin-Williams has operated at the 60 Lister Avenue facility since approximately 1902.
2. The Lister Avenue facility maintains a USEPA generator ID # NJD002451870 that is believed to have been issued to this facility in 1980.
 - 2 (b) This facility has been issued a storm water permit from the New Jersey Department of Environmental Protection in 1993 permit #NJ0088315 and the facility has a permit with the Passaic Valley Sewer Commission permit #20401500 that was issued in 1985.
3. No information has been obtained that would indicate that the Lister Avenue facility ever received, utilized, manufactured, discharged, released or disposed of 2, 3, 7, 8 tetrachlorodibenzo-p-dioxin or other dioxin compounds including dichlorodophenyl-trichloroethane (DDT). The remaining substances, pentachlorophenol, cadmium, copper, mercury, lead, zinc, methyl ethyl ketone, ethyl benzene, toluene and xylene may have been present in raw products used at the facility prior to 1984. Since 1984, this facility has produced a non-hazardous latex paint which may contain zinc.

LS

AKF000013



Patricia Hick, Esq.
March 2, 1995
Page - 2 -

4. (a) The Lister Avenue facility has not manufactured any products that contain any of the hazardous substance listed in question 3. This facility produced a water-based latex paint since 1984. Prior to 1984, this facility manufactured resins, varnish, oil and water-based latex paint.

(b) (i)(ii)(iii) Sherwin-Williams has not identified any information as to whether hazardous substances were generated during the manufacturing process or that any hazardous substances were ever mixed with any other waste produced from the facility.

5. Sherwin-Williams has not collected stored, treated or disposed of any hazardous substances since 1984 at this facility.

(a) Prior to 1984, Sue Free was responsible for disposal of hazardous substances from this facility.

(b) Sherwin-Williams used various waste haulers and trash brokers to dispose of hazardous waste at various waste disposal facilities.

(c) Sherwin-Williams stored its raw product in tanks, drums, containers and in warehouses for the production of paints and paint products throughout various areas of the facility.

6. (a) In response to 6(a)(i)(ii)(iii), Sherwin-Williams states that at least since 1984, the waste water streams from this facility are processed through a settling and filtration system to remove solids and sludges and the remaining waste water is discharged into the sanitary sewer system.

(b) In response to 6(b) and its subparts, Sherwin-Williams states that there are floor drains existing at the facility and are connected to a process system running to a settling tank to collect solids and then pass the waste water into the sanitary sewer system. There is no information that would indicate that these drains have not been connected to the sanitary sewer system since the plants began operation.

(c) (i) Sherwin-Williams has never had a catch basin or lagoons at this facility but storm sewers do exist on the property.

(ii) See response to 6(c)(i).

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Patricia Hick, Esq.
March 2, 1995
Page - 3 -

(iii) Sherwin-Williams has not obtained any information as to where the discharge from the storm sewer or sanitary sewer occurred.

(d) Sherwin-Williams has attached the only drawing found in response to this question.

7. (a) Sherwin-Williams has not generated any hazardous substance from this facility from 1984.

(b) Sherwin-Williams has not identified any information that would indicate any material was ever discharged into the Passaic River.

8. A small release of water-based latex paint (non-hazardous) was accidentally released into the river March 30, 1992. The U.S. Coast Guard and other authorities were notified of the release and inspected the river. No action was required of Sherwin-Williams.

9. Sherwin-Williams has not obtained any information other than that described in response to question number 8.

10. In response to this question and its subparts, the facility experienced a flood in December of 1992 when the sewers backed up as a result of the Passaic River cresting over the established bulkheads.

11. In response to this question, Sherwin-Williams has attached the requested documentation.

12. Sherwin-Williams states that the documentation as requested in question No. 12 including manifest inventory forms and billing records are located at the Lister Avenue facility and can be made available for inspection and copying at a mutually convenient time.

13. The facility has numerous records pertaining to sanitary sewer monitoring reports, BOD monitoring reports, air monitoring reports and groundwater reports. Due to the high volume of the documents, they can be produced for inspection at mutually convenient times.

AKF000015



Patricia Hick, Esq.
March 2, 1995
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14. (a) This facility has been owned and operated by The Sherwin-Williams Company since 1902.

- (b) See response to Question No. 14.
- (c) See response to Question No. 14.

15. In response to Question No. 15, and all of its subparts, Sherwin-Williams submits its Annual Report, Articles of Incorporation and Bylaws to which the answer to the Question is and its subparts can be ascertained.

16. The person answering this request is Donald J. McConnell, Counsel for The Sherwin-Williams Company, and obtained the information to respond to these questions from the following individuals:

- (a) Dr. Gordon Kuntz
Regulatory Compliance
The Sherwin-Williams Company
101 Prospect Avenue, N.W.
Cleveland, OH 44115
- (b) Sue Free
Environmental Specialist
The Sherwin-Williams Company
1450 Sherwin Avenue
Oakland, CA 94608
- (c) Wayne Murphy
Facility Manager
The Sherwin-Williams Company
60 Lister Avenue
Newark, NJ 07105
- (d) Marnie Sabatine
Environmental/Chemist
The Sherwin-Williams Company
60 Lister Avenue
Newark, NJ 07105

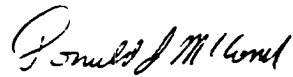
AKF000016



Patricia Hick, Esq.
March 2, 1995
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Pursuant to the Freedom of Information Act, The Sherwin-Williams Company is requesting a copy of any and all information your agency may have that would indicate the Lister Avenue facility is linked to the contamination of the Passaic River. Please forward all future correspondence directly to my attention. Should you have any questions or comments, please feel free to contact me.

Very truly yours,


Donald J. McConnell
Environmental Counsel
(216) 566-3741

DJM:ms

AKFG00017

Exhibit C



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY - REGION II

290 BROADWAY

NEW YORK, NEW YORK 10007-1866

OCT - 4 1985

GENERAL NOTICE LETTER
URGENT LEGAL MATTER
EXPRESS MAIL - RETURN RECEIPT REQUESTED

Mr. John G. Breen, Chairman of the Board
The Sherwin-Williams Company
101 Prospect Avenue, N.W.
Cleveland, Ohio 44115-1075

Re: Diamond Alkali Superfund Site
Notice of Potential Liability for
Response actions in the Passaic River Study Area

Dear Mr. Breen:

The United States Environmental Protection Agency ("EPA") is charged with responding to the release and/or threatened release of hazardous substances, pollutants, and contaminants into the environment and with enforcement responsibilities under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 ("CERCLA"), as amended, 42 U.S.C. §9601 *et seq.*

EPA has documented the release or threatened release of hazardous substances, pollutants and contaminants to the Passaic River Study Area which is a part of the Diamond Alkali Superfund Site ("Site"). By this letter EPA is notifying The Sherwin-Williams Company ("Sherwin-Williams") of its potential liability relating to the Site pursuant to Section 107 of CERCLA.

Sediment in the Passaic River contains numerous hazardous substances, pollutants and contaminants. Investigations undertaken by EPA indicate that hazardous materials were being released from the Sherwin-Williams facility at 60 Lister Avenue in Newark, New Jersey, into the Passaic River Study Area. Hazardous substances, pollutants and contaminants released from the Sherwin-Williams facility into the Passaic River Study Area present a risk to the environment and the humans who may ingest contaminated fish and shellfish. Therefore, Sherwin-Williams may be potentially liable for all response costs which the government may incur relating to the Passaic River Study Area.

Under Sections 106(a) and 107(a) of CERCLA, 42 U.S.C. §9606(a) and §9607(a) and other laws, potentially responsible parties ("PRPs") may be obligated to implement response actions deemed

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necessary by EPA to protect human health, welfare or the environment, and may be liable for all costs incurred by the government in responding to any release or threatened release at the Site. If response actions are performed by EPA rather than by the PRPs, those PRPs may be subject to legal action pursuant to Section 107(a) of CERCLA, 42 U.S.C. §9607(a), to recover public funds expended by EPA in response to the release and threatened release of hazardous materials at the Site. Such actions and costs may include, but need not be limited to, expenditures for conducting a Remedial Investigation/Feasibility Study ("RI/FS"), a Remedial Design/Remedial Action, and other investigation, planning, response, oversight, and enforcement activities. In addition, responsible parties may be required to pay damages for injury to, destruction of, or loss of natural resources, including the cost of assessing such damages.

While EPA has the discretionary authority to invoke special notice procedures, EPA hereby notifies you that it will not utilize the special notice procedures contained in Section 122(e) of CERCLA, 42 U.S.C. §9622(e). EPA has concluded that use of the special notice procedures in Section 122(e) of CERCLA would delay the implementation of the RI/FS which is currently being performed at the Site to determine the extent of contamination and to evaluate possible actions to mitigate any adverse effects. EPA will determine at a subsequent time whether additional measures are required to mitigate releases from the Site in order to protect the public health, welfare, and the environment. The decision not to use the special notice procedures does not preclude you from entering into discussions with EPA regarding your participation in activities at the Site.

By this letter, EPA encourages you, as a PRP, to voluntarily participate in the EPA-approved activities underway at the Site in conjunction with other PRPs. At the present time, the Occidental Chemical Corporation ("OCC") is performing an RI/FS at the Site under an Administrative Consent Order. OCC, through its successor, Maxus Energy Corporation, can be contacted at the addresses listed in the Attachment to this letter. Be advised that notice of your potential liability at the Site is being forwarded to OCC by EPA.

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EPA requests your cooperation in this matter. If you are interested in participating in the ongoing response action you should notify EPA of your intentions to join with OCC. Notification should be in writing and should be delivered to EPA no later than fourteen (14) days after the date that you receive this letter. Your letter should be sent to:

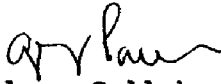
Lance R. Richman, P.G.
U.S. Environmental Protection Agency
Emergency and Remedial Response Division
290 Broadway, Floor 19
New York, NY 10007-1866,

with a copy to Ms. Amelia Wagner, Esq., of the Office of Regional Counsel at the same address.

If EPA does not receive a written response from you in the time specified above, EPA will assume that you voluntarily decline to participate in any of the response actions taking place at the Site. EPA reserves the right to pursue its available enforcement options with regard to the Site.

If you wish to discuss this matter further, please contact Mr. Lance R. Richman, P.G., of my staff at (212) 637-4409 or Ms. Wagner at (212) 637-3141. Please note that all communications from attorneys should be directed to Ms. Wagner.

Sincerely yours,


Kathleen Callahan, Director
Emergency and Remedial Response Division

Attachments

CC: Mr. Donald J. McConnell, Esq.
Counsel for The Sherwin-Williams Company

Ms. Carol Dinkins, Esq.
Vinson & Elkins, L.L.P.

Mr. Richard P. McNutt
Maxus Energy Corporation

844160003

ATTACHMENT

Contact for Maxus Energy Corporation:

Mr. Richard P. McNutt
Maxus Energy Corporation
1015 Belleville Turnpike
Kearny, New Jersey 07032

Counsel: Ms. Carol Dinkins, Esq.
Vinson & Elkins, L.L.P.
3700 Trammell Crow Center
2001 Ross Avenue
Dallas, Texas 75201-2916

PRPs in receipt of Notice Letters:

Mr. J. Roger Hirl
President and Chairman of the Board
Occidental Chemical Company
Occidental Tower
5005 LBJ Freeway
Dallas, Texas 75244

Brian C. Kelly, Esq.
Chris-Craft Industries, Inc.
600 Madison Avenue
New York, New York 10022

Counsel: Peter Simshauser, Esq.
Skadden, Arps, Slate, Meagher & Flom
300 South Grand Avenue
Los Angeles, California 90071-3144

Mr. Edgar S. Woolard, Jr., Chairman
E.I. du Pont de Nemours and Company
1007 Market Street
Wilmington, Delaware 19898

Mr. Robert D. McNeeley, President
Reilly Industries, Inc.
1510 Market Square Center
151 North Delaware Street
Indianapolis, IN 46204

844160004

Exhibit D



Consulting • Engineering • Remediation

281 Centennial Avenue
Piscataway, NJ 08854

(732) 457-0500

FAX (732) 457-0550

<http://www.ensr.com>

January 11, 1998

Mr. David Gustafson
Director of Engineering and Environment
The Sherwin-Williams Company
Coatings Division
101 Prospect Avenue, N. W.
Cleveland, Ohio 44115

PRIVILEGED AND CONFIDENTIAL

RE: Environmental Evaluation Summary for the Sherwin-Williams Facility located at 60 Lister Avenue Newark, New Jersey.

Dear Mr. Gustafson:

ENSR Consulting and Engineering (ENSR) was by retained the Sherwin-Williams Company (Sherwin-Williams) to conduct an Environmental Site Evaluation at their paint manufacturing facility located at 60 Lister Avenue, Newark, Essex County, New Jersey.

ENSR conducted a site visit at the site on July 22, 1998 for the purpose of initiating the site evaluation activities. The purpose of the site visit was to observe operations and identify potential areas of concern in accordance with the New Jersey Department of Environmental Protection's (NJDEP) requirements for preliminary assessments.

Site Location and Description

The Sherwin-Williams site is approximately a 13-acre parcel located in the Ironbound section of Newark. The area surrounding the facility consists of industrial properties with some nearby residential and commercial properties. This section of Newark has many known contaminated sites including the Diamond Shamrock site, a NPL site, which borders the facility to the east. The site is bordered to the north by the Passaic River, to the west by a former Conrail rail yard and Copco a former steel engineering company, and to the south by a railroad and Lister Avenue. Reichhold Chemicals, Inc. is located to the south across Lister Avenue. Two rail spurs are located on the Sherwin-Williams property. These rail spurs enter the property from the southwest and extend to the north and east. At least one of these spurs was used by Sherwin-Williams for the transportation of raw materials to the site. Figure 1 is a Site Location Map showing the site location, local topography, and surrounding areas.

Physical Setting

The Sherwin-Williams property is a relatively level property consisting of 20 existing buildings, including four manufacturing/production buildings. Approximately 50 percent of the site is currently covered by buildings and pavement. The southeast corner of the property consists of a parking lot. The area north of the parking lot consists of an unpaved area covered with vegetation. The southwest section of the property is almost entirely unpaved. Figure 2 provides a Site Plan showing the current site layout and the identified potential areas of environmental concern.



January 11, 1999
 Mr. David Gustafson
 The Sherwin-Williams Company
 Page 2 of 13

PRIVILEGED AND CONFIDENTIAL

Site History

To establish a history of the subject site, ENSR reviewed a series of aerial photographs, fire and factory insurance maps and historical site maps. Aerial photos from the mid-1930's, 1947, 1957, 1968, 1982, 1989 and 1997; and fire and factory insurance maps from 1892, 1908, 1931, 1941, 1950, 1952, 1963, 1970, 1973, 1988 and 1994 were used for the historical review of the site. Historical site plans and factory and fire insurance maps are included as Attachment 1. In addition, three former and four current Sherwin-Williams's employees were interviewed. Telephone summary sheets are included as Attachment 2.

Sherwin-Williams has operated a paint manufacturing facility at the Newark, New Jersey location since the early 1900s. The plant stored and produced oil- and water-based paints, lacquers, thinners, oils, solvents and alkyl resins. In 1984 the plant switched to the production of water-based paints only. The solvent-based paint operations were moved to other locations and the resin and lacquer production operations were shut down and demolished, along with the solvent and oil tank farm, and the Quonset Hut.

A review of the aerial photographs and the factory and fire insurance maps indicate that Sherwin-Williams has been occupying the site since the early 1900s. Prior to Sherwin-Williams the property was occupied by the Union Chemical Works and the E.C Shipyard. In the early 1900s the first buildings constructed by Sherwin-Williams were built on the west side of Brown Avenue in the area formerly occupied by the E.C. Shipyard. By 1908 the majority of the buildings were present on the western parcel of the property. The parcel east of Brown Avenue was occupied by the Consolidated Color and Chemical Company, which also occupied the parcel on the corner of Brown and Lister Avenues. By 1916 Sherwin-Williams had taken occupancy of the parcel of land east of Brown Avenue and began constructing buildings on this parcel.

Between 1916 and 1931, the site does not appear to have changed significantly. In the 1931 photograph aboveground storage tanks are present in the southwest portion of the Sherwin-Williams property and Consolidated Color and Chemical Company continues to occupy the parcel of land on the corner of Brown and Lister Avenue.

In the aerial photograph from the 1950s, the Quonset Hut is present along the western property boundary in the location of the C.R.R of NJ (manufacturers branch) railroad tracks. In the 1950s Roanoke Inc. took over the parcel of land on the corner of Brown and Lister Avenue which, according to a 1931 Sanborn fire insurance map, was previously owned by Consolidated Color and Chemical Co. Both the Consolidated Color and Chemical and Roanoke facilities consisted of a factory building, a smokestack, and at least three other buildings. Roanoke remained there until at least 1952. The use of this property and the operations conducted by Roanoke are not known at this time.

In an aerial photograph dated late 1960s, early 1970s, one Roanoke building, current building 29, is still present and the current New Emulsion Plant (NEP) building is present in the northeast corner of the property. The 1963 and 1970 fire insurance maps and the 1968 aerial photo



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The Sherwin-Williams Company
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PRIVILEGED AND CONFIDENTIAL

indicate the presence of ~~12~~ aboveground storage tanks along the northern corner of the property boundary. It is believed these tanks are associated with the Diamond Alkali property.

Follow
Brush?

The 1994 Sanborn fire insurance map identifies Roanoke as the owners of the parcel of land on the corner of Brown and Lister Avenue, which is identified as a parking lot. However, according to property deeds this portion of the property was acquired by Sherwin-Williams in 1964.

?

A review of the 1989 aerial photograph indicates the aboveground storage tank farm and the varnish manufacturing and storage buildings that were present since at least 1931 were removed by 1989. In addition, the Quonset Hut is no longer present; the location now appears as a concrete pad used as a drum storage area. Building 1 (formerly occupied by Consolidated Color & Chemical as an office and laboratory) at the northwest corner of Lister and Brown Avenue, at the Sherwin-Williams entrance, is no longer present in the 1989 photograph.

Previous Environmental Studies

In August 1986, a preliminary soil investigation was completed within the bermed tank farm located in the southwestern portion of the property. Soil samples were collected from the 6-inch interval above the water table and were analyzed for VOCs. Soil sample results indicated the presence of VOCs including benzene, tetrachloroethylene, xylenes, toluene, dichlorobenzenes, and ethylbenzene. A second phase of soil sampling was conducted in October 1986 to further delineate the potential extent of the contamination. A total of 16 soil borings were completed throughout the plant. Following the soil investigation, a groundwater investigation was initiated which included the installation of nine monitoring wells in March 1987 and January 1988. Water levels in March 1988 were reported to range from 1.88 to 9.27 feet below ground surface. Groundwater analytical results indicate the presence of benzene, chlorobenzenes, xylenes, trichloroethylene, vinyl chloride, and ethylbenzene at concentrations ranging from 1.2 to 8,100 parts per billion (ppb). Previous reports indicate contamination in some of the site wells (MW-2, MW-3, MW-6, MW-12) may be from sources other than the tank farm. As previously reported, the location of these wells and the groundwater flow direction at the site suggest a possible off-site source, located upgradient of the Sherwin-Williams site, may be contributing to groundwater contamination at the site. Contaminants detected in groundwater at the site exceed current NJDEP groundwater quality criteria.

In 1986 Sherwin-Williams installed a trench within their bermed tank farm to recover solvents found floating on the water table. The trench operated from 1986 to 1988 and reportedly recovered hundreds of gallons of solvent. The recovered solvent was reportedly pumped into an oil/water separator where it remained for a couple of months. The solvent was then pumped into a tanker for off-site disposal and the water portion was released into the sewer system. The trench operations ceased in 1988, and the trench was filled. The presence of solvent on the water table was reported by Sherwin-Williams to the US EPA in 1986.

In January 1988, additional soil borings were completed at previous soil boring locations to determine if contaminant concentrations had changed since the 1986 sampling event. Volatile compounds at concentrations ranging from 0.016 to 4,100 parts per million (ppm) were detected in the soil samples collected in 1988. Soil boring B9, located in Brown Street at the south end of



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building 14, contained the highest concentrations of VOCs in soil. Soil contaminant concentrations detected exceed current NJDEP soil clean-up criteria.

The tank farm and several buildings associated with the closed operations at the plant were demolished in 1987. During two recent excavations conducted at the facility, one south of building 28 and the other in the area of former buildings 48, 50, and 68, strong organic chemical odors were noted. The sources of the odors are unknown but are believed to be associated with past operations at the facility.

Sherwin-Williams Operations

Since the early 1900s, Sherwin-Williams has operated the site as a paint manufacturing facility. These operations included storing and producing oil- and water-based paint, resins, thinners, lacquers, oils, solvents and alkyd resins. The manufacturing/production processes appear to have occurred in several buildings on site. Currently, only water-based paints are manufactured at the Newark facility. According to facility personnel and former employees, all process lines are/were located aboveground and all discharges were to the Passaic Valley Sewer Commission (PVSC). In addition, interviews indicate that pesticide formulation was conducted at the Newark facility. The pesticides were warehoused on site and shipped from the Newark plant.

Raw materials are transported to the site by truck and railcar in bulk, drums, super sacks, bags, and totes. These materials, as well as, finished goods, off-spec goods, and empty paint cans are stored in various site buildings.

In 1960, several old buildings were razed. Modernization of the plant in the 1960s included the construction of buildings 27 (NEP) and 28 as finished goods warehouses, as this was Sherwin-Williams' major distribution center for the East Coast. The modernization also included the construction of building 18 (tank storage of non-flammable materials). In 1996, water-based paint manufacturing processes were installed in the NEP building. In the fourth quarter of 1998, NEP manufacturing facilities were removed and all floor penetrations were filled and the building was vacated. This building is located in the northeast corner of the property. Ten fiberglass storage tanks ranging in size from 3,000 to 7,500 gallons were used for raw material storage in building 27. Eight high-speed dispersion (HSD) tanks were also located in building 27. The paint was transferred from the HSD tanks to holding tanks where it was then transferred to filling tanks and into cans. Conveyor belts carried empty cans for labeling and for the addition of handles to the cans. Seven sumps were located beneath the filling tanks. In addition, three sumps were located beneath the holding tanks and four sumps were located beneath the wash tanks in this building.

Building 22 is a 5-story building located west of building 27. Historical maps indicate this building was used for paint operations. Currently, building 22 contains an air compressor for air actuated in the manufacturing process and one mechanical elevator on the 1st floor. Offices are located on the 2nd floor of building 22. The 5th floor is used as a rework area for returned paints. These paints are reworked and bulked into small drums. This area was previously used for the



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Mr. David Gustafson
The Sherwin-Williams Company
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storage of returned paints. The 4th floor is currently empty, but was previously also used for the storage of returned paint. The 3rd floor consists of a laboratory and storage areas for paint samples. All quality assurance lab work is done in the 3rd floor laboratory. A sink located in the laboratory currently goes to washwater tanks. Some sinks reportedly may have previously discharged to other areas. However, former employees indicate all known discharges were reportedly to PVSC.

Building 14 is a 5-story building, which formerly housed a laboratory on the 5th floor, which is currently empty. One elevator is located in this building. The 3rd floor of building 14 is used for the storage of raw material and colorants. The 1st floor contains packaging equipment and conveyor belts and is used for labeling and packaging. No floor drains, sumps, or floor cracks were observed during ENSR's site walk. Some paint staining was present on the floor.

Building 11 is a 4-story building, which houses the older manufacturing/process area. On the 4th floor, nine HSD tanks ranging in size from 500 to 1,500 gallons are located. One sink is located on the 4th floor and is connected to a washwater line. The 3rd floor of building 11 contains the holding tanks where all adjustments to the paint, including viscosity and pigment, are completed. One sink is located on the 3rd floor of building 11. Paint is transferred by pump to the holding tanks situated on the 2nd floor. The 1st floor of the building houses the filling area. Five active sumps are located on the 1st floor and at least three former sumps have been filled with concrete on this floor. These sumps currently are pumped to the wastewater tank. Additional sumps and sinks were located here prior to December 1997, which went to the Passaic Valley sewer system. During our site walk, no cracks were observed in the concrete floor.

Building 28, constructed in the 1960s, is used for the storage of finished goods only. No floor drains were observed during ENSR's site walk. Building 29 is also used for the storage of finished goods, as well as raw materials and empty cans.

Building 13 is a 4-story building. The 3rd floor is used for the storage of raw materials. Small mixers are also located on this floor. A locker room and cafeteria are located on the 2nd floor. One of the floors in building 13 is currently not in use, but was previously used for the storage of raw materials. Two loading docks are located at building 13.

Building 18, constructed about 1960, is a 1-story building containing AGSTs used to store water-based slurry, propylene glycol, ammonia, and various latices. The tanks are located on a concrete floor and the entire tank area is contained by building walls, ramps, and berms. No cracks were observed on the floor during ENSR's site walk.

Building 24, reconstructed in 1996, is used for the storage of raw materials in AGSTs, dry raw materials, and packing materials. Nine latex storage tanks and one slurry storage tank were observed in this building. The tanks are located on a concrete floor, which is reported to be new since 1997.



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PRIVILEGED AND CONFIDENTIAL

The maintenance building is located near the center of the site. One sink and one floor drain were observed in this building. In addition, four drums stored on pallets were observed in this building during ENSR's site walk. Some oil staining was observed on the concrete floor; however, no cracks were observed on the floor.

Identified Potential Areas of Environmental Concern

Several potential areas of environmental concern were identified during the environmental site evaluation. Historic site plans, aerial photographs, fire and factory insurance maps, and interviews with facility personnel were used to identify the following potential areas of environmental concern in accordance with the NJDEP's requirements for preliminary assessments.

Former aboveground storage tanks (AGSTs) – Several AGSTs existed in the southwestern portion of the property since at least 1931. These AGSTs were used for the storage of solvents and other raw materials used in the manufacturing processes. According to historical maps, two of the AGSTs were constructed with wood floors and contained mineral oil and mineral spirits. Soil and groundwater samples collected from this area in 1986 and 1988 indicate the presence of volatile organic compounds in the soil and groundwater. In addition, a solvent recovery trench was installed to recover solvents floating on the water table in the area of the former tank farm. A former 18,000-gallon fuel oil tank was identified in the area of the former varnishing manufacturing buildings on the 1934 fire insurance map.

Current aboveground storage tanks – A total of five AGSTs are currently located outside the Sherwin-Williams buildings near the northwestern corner of the property. Two of the five AGSTs contain wastewater and are located within a concrete bermed area. The three other AGSTs contain water-based slurry and are also located within a concrete bermed area, which adjoins the other bermed area. In March 1998, a leak in a fourth AGST containing slurry resulted in a release to the Passaic River due to cracks in the tank pad. This tank was removed and the tank pad and berm were repaired. No known samples were collected from this area following the cleanup. No cracks in the concrete pad were observed by ENSR during our site walk. Several AGSTs are located inside some of the facility buildings. These AGSTs are used for the storage of various water-based liquids (i.e. latex, slurry, washwater) and are located on concrete. No cracks in the concrete floors were observed during ENSR's site walk. One additional AGST was identified at the Sherwin-Williams site. This tank was identified as a former washwater tank. Reportedly, this tank has had two spills. The dates of these spills have not been determined.

Drum storage areas – One drum storage area is currently located outside the facility buildings. This area consists of an uncovered concrete pad used for the storage of returned goods and spoiled batches of paint. Aerial photographs and historical maps indicate that in the past several additional exterior areas were used throughout the property for drum staging areas. These storage areas include the following:

- storage of oils and varnishes along the railroad siding;



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- a drum storage area on a concrete pad in the former location of the Quonset Hut;
- a drum storage area south of former building 20 (now occupied by building 27);
- empty drum staging area along warehouse 24;
- drum storage area for oils and varnishes north of the former AGST farm; and
- a drum storage area north of the existing parking lot.

Asbestos - Based on our site walk, interviews with facility personnel, and an asbestos assessment conducted by Sherwin-Williams personnel in 1996, an estimated 5,600 linear feet of asbestos containing material (ACM) insulated pipe was observed. Observations during the asbestos assessment conducted in 1996 and ENSR's site walk indicate some of the ACM was in poor condition.

Boiler room - The boiler room is located in the maintenance shops area of the facility. Some oil staining was observed on the boiler room floor during ENSR's site walk and some minor cracks were observed in the floor.

Sumps - Several sumps were identified in three of the existing facility buildings. These sumps are reported to be concrete. Most sumps could not be inspected due to the presence of liquid; therefore, their integrity could not be confirmed. According to facility personnel, several sumps have been filled and are no longer in use.

Underground storage tanks - Two USTs were identified on the 1941 Factory Mutual Fire Insurance Company map. One buried 13,000-gallon naphtha tank was identified just north of the former AGST farm. One 500-gallon gasoline tank was identified in the southeastern corner of the property near Dock Street. It is unknown if these USTs are still present. Refer to Figure 2 for the location of these USTs. ?

Groundwater - Nine monitoring wells were installed in 1987 at the site as part of the soil and groundwater investigation associated with the AGST farm. Two additional wells were reported to exist on the Sherwin-Williams property as part of the ongoing investigative activities at the adjacent Diamond Shamrock site. Historical groundwater sampling results indicate the presence of volatile organic compounds (benzene, chlorobenzenes, xylenes, trichloroethylene, vinyl chloride, and ethylbenzene) in the groundwater at concentrations ranging from 1.2 to 8,100 ppb. Individual contaminant compounds detected in the groundwater at the site exceed current NJDEP groundwater quality criteria. The location of four (MW-4, MW-8, MW-12, MW-11) of the nine wells reported to be on site were identified during ENSR's site walk. The monitoring wells that were identified appeared to be in poor condition.

Loading/unloading areas - Raw materials currently and historically are transported to the site via rail and truck. The existing rail loading and unloading area is not paved. Staining was observed on the ground surface in this area during ENSR's site walk. No surface staining was



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observed in the area of the truck loading/unloading areas during our site walk. Three former rail spurs were identified at the Sherwin-Williams property on factory insurance maps. It is not known at this time which rail spurs were used for the loading and unloading of materials at the facility. In addition, employees indicate that a barge or ship unloading station for latex was previously located at the river. No surface staining was observed in this area during ENSR's site walk.

?
Liuscah
oil

Transformers – Two areas of transformers were observed at the facility during ENSR's site walk. These transformers were located on concrete pads and no staining was observed on the pads. These transformers were reported to contain low concentrations of polychlorinated biphenyls (PCBs). One additional transformer area was also identified on various historical maps and fire insurance maps.

Spills – In March 1998 a leak in an AGST containing a water-based slurry and cracks in the tank pad resulted in a release to the Passaic River. No known samples were collected from this area following the cleanup.

Oil staining was observed on the surface soil in the area between the railroad spurs, which service the facility. In addition, some paint staining was also observed on gravel present in the area.

Floor drains and process lines - Currently, all floor drains and sink areas discharge to on-site washwater tanks prior to discharging to the PVSC. Prior to 1997, some sumps and sinks discharged to the Passaic Valley sewer system. According to current and former employees, all existing and former production lines are reportedly located aboveground. Interviews with a current employee indicate floor drains were present in the resin plant; however the drains were filled with concrete in approximately 1973 to 1974.

Roof drains – Several roof drains are present on the Sherwin-Williams buildings, which are reported to discharge to the Passaic River. At least one dust collector was identified on the roof of Building 14; however, currently there are no processing areas in this building.

Other storage areas – Historical maps indicate several buildings and exterior areas were used for storage of rubbish, chemicals and raw materials. These storage areas include the following:

- fire insurance maps identified that rubbish was staged in the northwest corner of the property near Building 24;
- rubbish was stored in Building 12;
- former Buildings 20 and 113 were used to store insecticides as identified on the Associated Factory Mutual Fire Insurance map, revised 1941;
- coal storage areas near Building 113 and west of Buildings 92 and 93; and



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- former Buildings 62, 63, and 64 were used to store varnishes. Building 65 was used for manufacturing of lacquers and thinners. These buildings were demolished in 1985.

Former operations – Historical maps indicate that several former buildings were used for operations that included drum washing and manufacturing of oil-based products. These operations were conducted in the following buildings:

- former Building 55, located west of Brown Street, was a varnish manufacturing building
- former buildings 50, 51, 52 and 53, located east of the railroad siding and north of the former tank farm, were used for drum washing operations
- Sanborn Fire Insurance Maps indicate that Roanoke Inc. owned the portion of the Sherwin-Williams property along Lister Avenue. This area is now a parking lot. The buildings identified as part of the Roanoke facility include factory buildings, solvent warehouse, and paint and chemical warehouse. The nature of the operations and processes conducted by Roanoke at this location are not known at this time.

Dumpsters – Seven dumpsters were observed on the Sherwin-Williams property. No staining was observed in the area of the dumpsters during ENSR's site walk. Two compactors were also identified on site. Staining was observed on the ground surface in the area of the compactor located near the maintenance shop.

Passaic River – Storm sewers and non-contact cooling water from the former resin plant reportedly discharged to the Passaic River. No further information regarding these discharges was available.

Adjacent and Surrounding Properties

As part of ENSR's investigation of the subject site, ENSR reviewed the EDR data base report for the Sherwin-Williams property, which is a review of various governmental data by EDR of Southport, Connecticut. A copy of the EDR database report for the Sherwin-Williams property is provided in Attachment 3.

The following federal and state databases were searched for the area surrounding the subject property; the various search distances used are noted in parenthesis:

- NPL: for existing and proposed Superfund sites on the National Priorities List (1.0 mile of the subject property).



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- RCRIS/TSD: for reported sites that treat, store and/or dispose of hazardous waste and subject to the federal RCRA regulations (0.5 mile).
- SHWS: for identified hazardous waste sites designated under various state regulations (1.0 mile).
- CERCLIS: for abandoned, uncontrolled or inactive hazardous waste sites reported to the U.S. EPA. (0.5 mile).
- CORRACTS: for hazardous waste handlers with RCRA corrective action activity (1.0 mile).
- SWF/LF: for identified solid waste facilities and landfill sites designated under various state regulations (0.5 mile).
- LUST: for leaking underground storage tanks reported to the state under various state regulations (0.5 mile).
- UST: for underground storage tanks registered under various state regulations (0.25 mile).
- RCRIS/LQG: for reported large-quantity generators of hazardous waste (0.25 mile).
- RCRIS/SQG: for reported small-quantity generators of hazardous waste (0.25 mile).
- ERNS: for sites reporting spills to the U.S. EPA and/or the U.S. Coast Guard under various federal regulations (target property).
- FINDS: for sites that are regulated or tracked by the EPA for a variety of programs (target property).



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The Sherwin-Williams facility was identified in the CERCLIS, CORRACTS, RCRIS, and FINDS databases. The following properties were identified as potential environmental concerns located in the immediate vicinity of the Sherwin-Williams property.

- Diamond Alkali
80 Lister Avenue
Newark, NJ 07105

Diamond Alkali is currently on the final National Priorities List (NPL). This site was the location of a former chemical manufacturer/chemical process plant, which included the manufacturing of dioxin. Soil and groundwater contamination is known to exist at the site.

- Duralac Chemical Corporation
84 Lister Avenue
Newark, NJ 07105

This property is identified on the CERCLIS, FINDS, RCRIS-LQG and UST databases. Duralac Chemical Corp. is reported to manufacture paint and industrial coatings. The site is currently inactive.

- Reichhold Chemical Company
46-58 Albert Avenue
Newark, NJ 07105

This property is identified on the CERCLIS, FINDS, RCRIS-LQG, TRIS, RAATS, and UST databases. Reichhold Chemical is reported to be involved in the preparation of resins and polymers for use in the paint, coating, and graphic arts industries.

- Reichhold Chemicals Incorporated
46 Albert Avenue
Newark, NJ 07105

This facility is identified on the LUST database. The Reichhold Chemicals plant manufactured alkyd resins and is located hydraulically upgradient of the Sherwin-Williams property. In addition to the EDR Report, ENSR reviewed the Reichhold Chemicals Site Investigation (SI) Report prepared by O'Brien & Gere Engineers, Inc., dated November 1997. Due to the closure of their manufacturing operations the site is currently under ISRA review. Site Investigation activities at the facility indicate the presence of volatile organic compounds in the site soil and groundwater. A total of six USTs were closed at the facility in 1992. Three of the USTs were removed and three were closed in place. Results of the post-excavation samples collected during the UST removal were not included as part of the SI report; however, the report indicated that site soils were impacted by petroleum related compounds. As part of the ISRA Site Investigation, three groundwater monitoring wells were installed at the site. Groundwater samples collected from these wells indicate the presence of benzene, ethylbenzene, and xylenes at concentrations that exceed their



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respective NJDEP groundwater quality criteria. Light, non-aqueous phase liquid (LNAPL) has also been detected on the site groundwater in the area of the former USTs.

- Cellofilm Corp.
45-5 Cornelia Street
Newark, NJ 07105

This property is identified on the CERCLIS, FINDS, RCRIS-LQG and RAATS databases. The owner of Cellofilm is listed as Reichhold Chemicals, Inc.

Requests were made to New Jersey Department of Environmental Protection (NJDEP) through the Freedom of Information Act (FOIA) for any regulatory information on the subject site and several surrounding properties. Responses from NJDEP indicate files are available for review; however, at this time these files have not been reviewed.

Conclusions and Recommendations

Sherwin-Williams is evaluating the future of the Newark facility, and putting together a 5-year business plan. Options being evaluated include tearing down the current operations and relocating them to another area of the site, or closing down the site and moving the operations to another site altogether. ENSR conducted an environmental evaluation of the site to assist Sherwin-Williams in its evaluation of the potential options for the site. The environmental evaluation included a review of existing reports, site visits, identification of potential areas of environmental concern, review of historic background information on the site and interviews with past employees of the facility.

Potential areas of concern were identified in accordance with the NJDEP's requirements for preliminary assessments. A total of 16 categories of potential areas of environmental concern, including USTs, AGSTs, transformers, drum storage areas, asbestos, loading/unloading areas, sumps, floor drains, former operations, roof drains, boiler room, chemical storage areas, spills, dumpsters, other storage areas, and contaminated groundwater, have been identified at the Sherwin-Williams site. ENSR's environmental evaluation of the Sherwin-Williams site did not include the collection of environmental samples to determine the presence or absence of contamination at each of the identified potential areas of concern. However, based on our evaluation soil and/or groundwater contamination is suspected to be present at many of the identified areas of concern. ENSR recommends the collection of a limited number of soil and groundwater samples at select locations to provide a better understanding of the potential environmental impacts at the site and assist in developing a site-wide remedial strategy.

The presence of groundwater contamination at the Sherwin-Williams site was determined in previous investigations and will require vertical and horizontal delineation. The former monitoring wells used during these previous studies were observed to be in poor condition and their integrity is suspect. It is recommended that they be fully evaluated, and repaired or properly abandoned.



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Due to the presence of known contaminated sites surrounding the Sherwin-Williams site, a review of regulatory files regarding the adjacent and nearby properties is recommended to determine if off-site groundwater contamination is migrating beneath, and contributing to groundwater contamination at the subject property.

Should Sherwin-Williams decide to shut down operations at the Newark facility the site would be subject to the requirements of New Jersey's Industrial Site Recovery Act (ISRA). ISRA requires that prior to transfer of ownership, termination or closing of activities at an "industrial establishment"; owners/operators of these activities notify the New Jersey Department of Environmental Protection (NJDEP) and evaluate the potential for a release of hazardous substances at the site. If a release is confirmed the owner/operator is responsible for its clean-up. All remedial actions (including investigations and clean up) in New Jersey must be conducted in accordance with the Technical Requirements for Site Remediation (TRSR), N.J.A.C. 7:26E. The TRSR are actual regulations, not guidelines, on conducting investigation and remedial actions. The TRSR are rather prescriptive requirements that specify the minimal actions that the NJDEP will accept when conducting preliminary assessment, site and remedial investigations, and remedial action activities under all regulatory programs in New Jersey including ISRA.

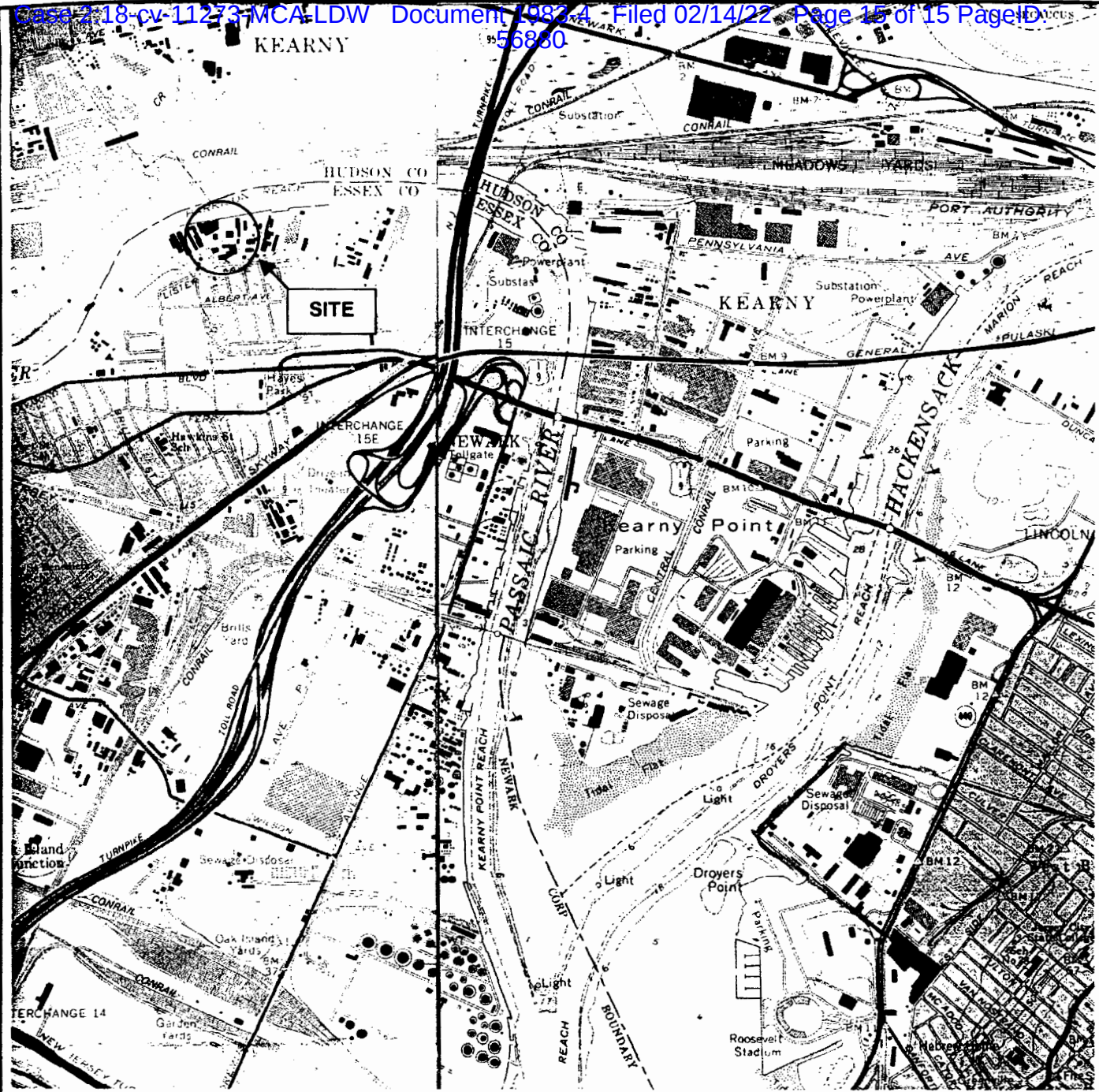
ENSR appreciates the opportunity to provide Sherwin-Williams with this environmental evaluation and look forward to assisting you with the next phase of this project. If you have any questions, please contact our office at (732) 457-0500.

Sincerely,

Tammy Grillon
Project Manager

r:\common\sherwill\pa

David J. Grupp
Manager, Mid-Atlantic Operations



SOURCE: U.S.G.S Topographic Map
Elizabeth, N.J. - N.Y. 1967 (photorevised 1981)

Scale:
 0 2000 4000 ft
 0 1/4 1/2 1 MILE

NORTH ↑

ENSR ENSR Consulting and Engineering			
FIGURE 1 SITE LOCATION MAP Sherwin Williams Company 60 Lister Avenue Newark, NJ			
DRAWN: CRS	DATE: October 30, 1998	PROJECT NO.: 8508-070	REV:
FILE NO.: SWSITELOC.DOC	CHECKED:		

Exhibit E

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**UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY
NEWARK VICINAGE**

OCCIDENTAL CHEMICAL)	
CORPORATION,)	Hon. Judge Madeleine Cox Arleo
)	Hon. Magistrate Judge Joseph A.
Plaintiff,)	Dickson.
)	Civil Action No. 2:18-CV-11273 (MCA-
v.)	JAD)
)	
21ST CENTURY FOX AMERICA, INC., <i>et al.</i> ,)	
)	
Defendants.)	
)	
)	
)	
)	

**The Sherwin-Williams Company responses to alleged
Interrogatory deficiencies**

The Sherwin-Williams Company (hereinafter referred to as “the Defendant”) hereby responds to the deficiency letter dated August 7, 2019 from plaintiff and addresses the deficiencies alleged by plaintiff in the supplemental interrogatory responses set forth below.

RESERVATION OF RIGHTS

The Defendant’s investigation and development of all facts and circumstances related to this litigation is ongoing. The responses to interrogatories are made without prejudice to, and are not a waiver of, Defendant’s right to rely on other facts or documents at trial. In addition, the responses do not waive, and Defendant hereby expressly reserves, its right to assert any and all objections as to the admissibility of such responses into evidence in this action, or in any

other proceedings. The Defendant expressly reserves the right to supplement, clarify, revise, or correct any or all of the responses and objections herein.

5. For each Operation identified in response to Interrogatory No. 2, Identify: (a) the raw materials used; (b) products and intermediates resulting from the Operations; and (c) any Waste Materials.

Interrogatory #5 response:

Known raw materials used by SW are listed on the document previously produced to plaintiff and identified as TSWC-FED-0047641 and 0047642 as set forth on the listing of hazardous substances used at the former SW facility.

The known waste materials produced by SW are identified in the documents previously produced to plaintiff and identified as TSWC-FED-0044531, 1981 TSD Facility Annual Report and the 1983 Generator's Report, TSWC-FED-0044475.

The site maps previously identified in Defendant's responses to Defendant's interrogatories are all of the site maps in the possession of Defendant. Each is identified by date, and the date means that the improvements shown on the site maps, and the products and materials listed thereon, were at and/or used at the SW property at that time. Inasmuch as the maps cannot apparently be read by plaintiff, revised copies of said maps have been reproduced such that the relevant information thereon can be read. The storage containers are identified as such on the maps. The products and/or materials used on Defendant's property as of the date of the site map are identified thereon. A written explanation of the location of storage areas and facilities is not believed to be as responsive as a review of the site maps, inasmuch as a written description of location(s) will be confusing and provide little assistance to plaintiff in its review of the former SW property and the activities that were undertaken thereon. A simple "zoom in" on a computer screen makes each of the site maps easy to read.

A listing of materials in storage, and their tank location, as set forth on the re-produced site maps is contained on the excel sheet included with Defendant's recent document production in response to the deficiency letter received by Defendant, TSWC-FED-0047913. The information therein is a compilation of information from the site maps produced to plaintiff and referenced above. In addition, an identification of storage locations is also included with these interrogatory answers, which includes information also set forth on the site maps produced to plaintiff and referenced above TSWC-FED-0047911.

Revised document submissions have been made by Defendant and those submissions were included in the document production filed on August 26, 2019 and included new documents

identified with bates stamp numbers for the initial pages of said documents: TSWC-FED-0001254, 0001121, 0001115, 0001252, 0001253, 0001255 and 0047862.

6. For each Operation identified in response to Interrogatory No. 2, Identify: (a) each specific location of any storage area(s) (such as tanks, pits, and barrels) used for raw materials and/or Waste Materials; (b) the time period during which the storage area was used; and (c) describe what was stored in each storage area.

Interrogatory #6 response:

The site maps provided/referenced in Defendant's initial interrogatory responses identify specific areas at the former SW facility where materials were stored for raw materials and waste materials. The maps provide a comprehensive account of storage area location and types of materials stored. Each site map is identified by date and that date provides the time period within which the former SW property was utilized for the activities identified, the location(s) where those activities were undertaken, and the areas utilized for storage and the contents of the tankage identified on the site map. The interpretive section of the 2001 ISRA Investigation Report discusses buildings and storage locations. (See TSWC-FED-0000022-27, inclusive). The aerial photographs referenced therein will be produced shortly. The various buildings referenced in the ISRA IR can also be determined by review of the site maps previously produced to plaintiff. (See TSWC-FED-0001254, 0001121, 0001115, 0001252, 0001253, 0001255, 0047862 and 0047912.

As noted in response to interrogatory #5 above, information regarding the materials stored in locations shown on the site maps produced to plaintiff and referenced above are included with the re-produced site maps, as well as information identifying the buildings within which storage of materials was undertaken, at the time frames referenced accordingly. Said documents were re-produced to counsel on August 26, 2019.

See also TSWC-FED-0047862 and the site maps re-produced to counsel on August 26, 2019, all of which have been approved by counsel for plaintiff for their clarity. Said documents were included in the Defendant's document production to counsel on August 26, 2019

7. For each Property at Issue identified in response to Interrogatory No. 1, Identify all catch basins, floor drains, tanks, sinks, sumps, trenches on the property, outfalls, air emissions, casualty fires, explosions, intentional dumping, and stormwater and storm sewers.

Interrogatory #7 response:

Known raw materials used by SW are listed on the document previously produced to plaintiff and identified as TSWC-FED-0047641 and 0047642 as set forth on the listing of hazardous substances used at the former SW facility.

The known waste materials produced by SW are identified in the documents previously produced to plaintiff and identified as TSWC-FED-0044531, 1981 TSD Facility Annual Report and the 1983 Generator's Report, TSWC-FED-0044475.

The site maps previously identified in Defendant's responses to plaintiff's interrogatories are all of the site maps in the possession of Defendant. Each is identified by date, and the date means that the improvements shown on the site maps, and the products and materials listed thereon, were at and/or used at the SW property at that time. Inasmuch as the maps cannot apparently be read by plaintiff, revised copies of said maps have been reproduced such that the relevant information thereon can be read. The storage containers are identified as such on the maps. The products and/or materials used on Defendant's property as of the date of the site map are identified thereon. A written explanation of the location of storage areas and facilities is not believed to be as responsive as a review of the site maps and the 2001 Investigation Report at TSWC-FED-00000022-27, inasmuch as a written description of location(s) will be confusing and provide little assistance to plaintiff in its review of the former SW property and the activities that were undertaken thereon. A simple "zoom in" on a computer screen makes each of the site maps easy to read.

A listing of materials in storage, and their tank location, is contained on the excel sheet included with Defendant's recent document production in response to the deficiency letter, TSWC-FED-0047913. The information therein is a compilation of information from the site maps produced to plaintiff and referenced above. In addition, an identification of storage locations is also included with these interrogatory answers, which includes information also set forth on the site maps produced to plaintiff and referenced above, TSWC-FED-0047911.

Revised document submissions have been made by Defendant and those submissions were included in the document production filed on August 26, 2019 and included new documents identified with bates stamp numbers for the initial pages of said documents: TSWC-FED-0001254, 0001121, 0001115, 0001252, 0001253, 0001255 and 0047862.

No explosions or major accidental releases of any chemicals are known to have occurred at Defendant's property. No accidental releases are known to have occurred other than those previously disclosed. However one or more explosions did occur at the former, adjacent Diamond Shamrock property. Said explosion(s) and day-to-day operations at the former, adjacent Diamond Shamrock property impacted and contaminated Defendant's property. See TSWC-FED-documents beginning at the following bates stamped pages: 0044962, 0045020,

0045032, 0045058, 0045084, 0045192, 0046652, 0046891, 40047058, 0047151, 0047937.

8. Describe any treatment performed on Waste Material identified in response to Interrogatory No. 5 before it was disposed of.

Interrogatory #8 response:

Other than the settling basin to collect solids identified in Defendant's initial responses to plaintiff's interrogatories, there was no treatment of process wastewater that was discharged into the PVSC treatment works system from 1924 to 1999. Defendant strikes its objection to the definition of the term "treatment" for this interrogatory response.

9. For each Operation identified in response to Interrogatory No. 2, Identify any environmental permits, including without limitation air quality, water quality, waste disposal, stormwater, waste discharge, and/or operating permits.

Interrogatory #9 response:

See TSWC-FED-0044116-158, inclusive.

10. For each Operation identified in response to Interrogatory No. 2, Identify each Disposal Company that handled any Waste Material that contained COCs and for each such company provide: (a) the dates during which that company handled Waste Material; (b) the chemical composition of the Waste Material handled by that company; (c) the methods of storage, handling, treatment, and disposal used by that company; and (d) the location(s) where that company disposed of the Waste Material.

Interrogatory #10 response:

Defendant identified all known disposal companies that handled any waste materials in the lower Passaic River catchment for the Defendant. The known disposal companies are set forth below in this interrogatory response. Any waste materials removed from the former SW property and transported and disposed outside of the lower Passaic River catchment is not the subject of the present lawsuit. The chemical composition of waste materials is set forth in the 1981 TSD Annual Report found at TSWC-FED-0044531 and the 1983 Generator's Report found at TSWC-FED-00444475. The alleged location(s) of the companies so identified is alleged in

plaintiff's Complaint. The list of known disposal companies is comprehensive. The dates said disposal companies were utilized by Defendant are set forth below:

D&J Trucking and Waste Company, Avenue P Landfill—was a facility licensed to operate in 1970 and that ceased accepting materials in 1974. If waste materials from the former SW property were taken to the Avenue P Landfill, they would have consisted of waste materials consistent with the 1981 TSD Annual Report and 1983 Generator's Report referenced above. It was alleged that Defendant disposed of approximately 1300 drums per year at the Avenue P Landfill during its operations.

Bayonne Barrel & Drum—this facility allegedly operated between the early 1940s and the early 1980s. It is believed that this facility received empty drums from Defendant for reconditioning.

Central Steel Drum—this facility allegedly operated between 1951 and the early 1990s.

With regard to the site referenced as Avenue P, see file materials found at TSWC-FED-0047863 etc. This site allegedly operated between 1960 and 1973,

Defendant has no knowledge or documentation of any other disposal facility or waste disposal activities that were undertaken in the lower Passaic River catchment.

11. For each Operation identified in response to Interrogatory No. 2, state whether any raw material, products or intermediates, or Waste Material contained any of the COCs and, if so, Identify: (a) which COC(s) it contained; (b) the raw material or Waste Material that contained the COC; and (c) the approximate quantity (by percentage and concentration) of each COC present in the raw material or Waste Material.

Interrogatory #11 response:

With regard to the percentage and concentration of any COCs in Defendant's waste is found in the PVSC wastewater sampling results, a copy of which in the possession of plaintiff is found at TSWC-FED-0047860-61. Said production is without prejudice to Defendant's position that upon connecting to the PVSC treatment works system in 1924, Defendant's process wastewater was not discharged into the Passaic River.

Information with regard to lead acetate is as follows:

The Defendant has not located any documentation or information that lead acetate was used at the former SW property. The Defendant previously acknowledged, however, that it is possible that lead acetate was used at the former SW property, and that was the sole basis for the inclusion of that information in Defendant's initial interrogatory responses. In any event, even

if used, the Defendant does not believe that lead acetate was produced at the former SW property. It would have been delivered as a raw material if so used. It is also more likely than not that if lead acetate was used it would have been stored in an above-ground tank at the former SW facility, and there is no document or any information located by Defendant that suggests any such storage ever took place. The investigation regarding documents or information on this subject is continuing and defendant reserves the right to supplement these interrogatory responses accordingly.

12. Identify any contracts You had with the Disposal Sites, any operator(s) of the Disposal Sites, or any party to haul Containers containing Waste Materials to the Disposal Sites and describe: (a) the chemical composition of the materials You disposed of at the Disposal Sites; (b) the time period of this disposal; and (c) the amounts of Waste Materials disposed at the Disposal Sites.

Interrogatory #12 response:

The list of known hazardous substances used at the former SW property is found at TSWC-FED-0047641 and 0047642. Neither dioxin nor PCBs were utilized by Defendant in its production of products produced at the former SW property, nor were they produced as by-products in any of the Defendant's production activities. The known chemicals in the waste materials are noted in the 1981 TSD Annual Report found at TSWC-FED-44531 and the 1983 Generator's Report found at TSWC-FED-0044475. The complete universe of contracts with disposal companies that had facilities in the Passaic River catchment in the possession of Defendant consists of those contracts identified in Defendant's initial responses to plaintiff's interrogatories. The representative amount of waste material disposed on an annual basis is set forth in the documents identified and referenced in the interrogatory #5 response as set forth in the documents beginning with the pages noted hereafter: TSWC-FED-0044531 and 00-44475.

15. Are You a party to any joint defense agreement(s) (whether written or unwritten) between or among You and any other Person relating to this litigation and/or any of the Properties at Issue? If so, Identify the parties to the agreement and state the date it was signed (or verbally agreed to) and became effective.

Interrogatory #15 response:

The joint defense agreements entered into by Defendant were noted in the initial interrogatory responses. The identity of "others" is requested, notwithstanding said information was previously provided to Defendant. The parties that entered into the October 24, 2017 joint defense agreement were all identified in Defendant's previous interrogatory responses. In the subsection referenced in (f), delete the word "includes" and replace with the word "are".

17. Identify the dates and describe the nature and results of any soil, groundwater, surface water, stormwater, sediment, wastewater, or other site media sampling that relates to COCs on any Property at Issue identified in response to Interrogatory No. 1 or in the Passaic River.

Interrogatory #17 response:

Plaintiff references the number of pages produced by Defendant on the issue of remedial investigation activities. It is noted that plaintiff requested that it receive all such materials, reports, including quality assurance/quality control documentation. Defendant has complied with that request, which has resulted in the substantial document production noted. The more specific information requested is set forth below, identifying the report in question, its date, and the pages therein that contain sampling data.

Each report contains a table of contents that identify the contents of the report and the reports contain headings that identify the sampling activities undertaken and the sampling results obtained. Each report is also dated and indicates the consulting firm author of the document. By identification of the pages below, Defendant notes that the reports include text explaining or discussing sampling results in pages and sections not identified as "sampling results" and plaintiff should so recognize in its review of the documents produced by Defendant.

1. 2003 Copco PAR, beginning at page TSWC-FED-0044548, pages 0044577 to 0044596
2. 2003 Copco SI/RIR, beginning at page TSWC-FED-0045222, pages 0045252 to 0045256
3. Final UST report, beginning at TSWC-FED-45392, page 0045406
4. Full RI report, beginning at TSWC-FED-45430, pages 0045490 to 0045967
5. MW 32 Groundwater 2007 report, beginning at TSWC-FED-0056544, pages 0056559 and 0056560
6. AOCARAR 2008, beginning at TSWC-FED-0043919, page 0043937 to 0043940
7. The complete contents of the two appendices found at TSWC-FED-0001336 and TSWC-FED-7864 respectively

8. ISRA Investigation Report 2002, beginning at TSWC-FED-1122, pages 1175 through 1240
9. Final Copco report 2017, beginning at TSWC-FED-0034308 through and the 27 pages thereafter
10. Final Soil RIR 2017, beginning at TSWC-FED-0047632, pages 0047669-676.
11. Phase V report, beginning at TSWC-FED-0036599, pages 0036617 to 0036621
12. Phase IV report, beginning at TSWC-FED-0034308, pages 0034326-0034330
13. Groundwater Monitoring Report 2010-2012, at TSWC-FED-0011301, pages 0011323 to 0011336
14. Remedial Action Report for AO 26, 2008, at TSWC-FED-0047937, pages 0047954 to 47959.

Defendant reserves the right to amend its answers to plaintiff's interrogatories as allowed pursuant to Court orders.

It is also noted that the list above does not include all of the document production specified pages pertaining to investigatory or remedial activities. The pages noted are specific portions of the various reports that contain "Analytical Results", as requested and in accordance with plaintiff's request. Pages that include sampling data quality control/quality control materials are not specified herein but are found at the document locations so noted in Defendant's previous productions. For example, Defendant's second production of documents includes such documents and materials (See TSWC-FED-0001336-0034307.) In addition, certain of the reports identified above include such materials, but have not been specifically identified herein. For example, preliminary sections of the identified reports contain descriptions of site history and activities, prior investigation results, etc. that are easily gleaned from the table of contents contained in the various reports identified.

18. For any Property at Issue identified in response to Interrogatory No. 1, Identify and describe any Response Action that has occurred on that location.

Interrogatory #18 response:

See documents produced by Defendant with the pages beginning at TSWC-FED-0043919, TSWC-FED-0045032, TSWC-FED-0045192, TSWC-FED-0045392.

19. For any Property at Issue identified in response to Interrogatory No. 1, Identify and describe any major capital improvements and/or major changes to the footprint of the property.

Interrogatory #19 response:

The site maps previously provided identify the improvements located at the former SW property as of the date noted. (See TSWC-FED-0001254, 0001121, 0001115, 0001252, 0001253, 0001255 and 0047862). It is not considered that any major capital improvements and/or footprint changes to the former SW property occurred that is/are not identified on the referenced site maps re-produced by Defendant to counsel on August 26, 2019. Defendant withdraws its objection to the term "major" for this interrogatory response.

21. Identify or describe Your document retention and destruction polic(ies) relating to the retention or destruction of business records relating to each Property at Issue or Operations identified in response to Interrogatory Nos. 1 or 2.

Interrogatory #21 response:

The Defendant has only recently implemented a company-wide document retention policy. That policy was placed in effect within the last three (3) years. Prior to that policy, the company had an environmentally related document retention policy that related to certain environmental documents. That document retention policy was not in effect at the time the former facility was decommissioned. Business activities at Defendant's facility ceased in 1999 and the facility was demolished at that time. All known documents relevant to the allegations set forth in plaintiff's Complaint have been reviewed as part of Defendant's document production. To the extent additional responsive documents are identified, they will be reviewed and produced if appropriate.

VERIFICATION

I, Stephen J. Perisutti, Vice President, Deputy General Counsel and Assistant Secretary of The Sherwin-Williams Company ("Sherwin-Williams"), state that I am authorized on behalf of Sherwin-Williams to verify the Initial Answers and Objections, and Supplemental Responses to Plaintiff's Interrogatories, and that while I do not have personal knowledge of all facts cited therein, the information has been collected and the Answers and Responses made after a reasonable search of available records and that the information contained therein is true and accurate based on my best knowledge, information and belief. The records relied upon are identified in the Initial Answers and Supplemental Responses and, unless otherwise noted therein, have all been produced to Plaintiff and counsel of record. Therefore, the foregoing Initial Answers and Supplemental Responses are verified on behalf of Defendant The Sherwin-Williams Company.

Dated: September 30, 2019



Exhibit F

Inadvertent Polychlorinated Biphenyls in Commercial Paint Pigments[†]

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A polychlorinated biphenyl (PCB) that was not produced as part of the Aroclor mixtures banned in the 1980s was recently reported in air samples collected in Chicago, Philadelphia, the Arctic, and several sites around the Great Lakes. In Chicago, the congener 3,3'-dichlorobiphenyl or PCB11 was found to be the fifth most concentrated congener and ubiquitous throughout the city. The congener exhibited strong seasonal concentration trends that suggest volatilization of this compound from common outdoor surfaces. Due to these findings and also the compound's presence in waters that received waste from paint manufacturing facilities, we hypothesized that PCB11 may be present in current commercial paint. In this study we measured PCBs in paint sold on the current retail market. We tested 33 commercial paint pigments purchased from three local paint stores. The pigment samples were analyzed for all 209 PCB congeners using gas chromatography with tandem mass spectrometry (GC-MS/MS). More than 50 PCB congeners including several dioxin-like PCBs were detected, and the PCB profiles varied due to different types of pigments and different manufacturing processes. PCB congeners were detected in azo and phthalocyanine pigments which are commonly used in paint but also in inks, textiles, paper, cosmetics, leather, plastics, food and other materials. Our findings suggest several possible mechanisms for the inadvertent production of specific PCB congeners during the manufacturing of paint pigments.

Introduction

Polychlorinated biphenyls (PCBs) are a family of 209 compounds, called congeners, produced commercially as Aroclors by chlorination of biphenyl. The Aroclor mixtures were marketed for use in electrical transformers, capacitors, heat transfer systems, and hydraulic systems (1, 2). Lower quantities were used in voltage regulators, adhesives, caulking compounds, inks, lubricants, paints, sealants, carbonless copy paper, coatings, electrical switches, plasticizers, circuit breakers, dust control agents, and older fluorescent lighting fixtures (2). Aroclors were used in paint formulations as drying oils (resins) and plasticizer or softening agents (liquids). Data provided to EPA indicate that PCBs have been found in dried paint at concentrations that range from less than 1 ppm to 97,000 ppm (3).

[†] Part of the special section "Sources, Exposures, and Toxicities of PCBs in Humans and the Environment".

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Some PCB congeners, usually called non-Aroclor PCBs, are not present or are very low in concentration due to unfavored or improbable formation during the Aroclor manufacturing process (2). PCB11 is one of such non-Aroclor PCB congeners. In air samples from Chicago collected in 2007, we found PCB11 widely distributed throughout the city (4). The compound was almost simultaneously reported in air of polar regions (5). Since then, its presence was also reported in air of Philadelphia (6) and five sites around the Great Lakes (7). It appears that PCB11 is a global pollutant. In addition, PCB11 was measured in the wastewater effluent from paint production. Possible production of PCB11 from dechlorination is not likely because its possible precursors are in very low concentration in Aroclors (8, 9). The widespread distribution of PCB11 throughout Chicago and elsewhere suggests volatilization of this compound from surfaces. Litten et al. reported that PCB11 was in surface waters and effluent waste streams from a pigment manufacturing plant around New York Harbor (10). Recently, Rodenburg et al. detected it in consumer goods including newspapers, magazines, and cardboard boxes, which usually contain color pigments (11). Therefore, we hypothesize that PCB11 and other PCB congeners are present as byproduct in current commercial pigments.

Paint is composed of pigments, solvents, resins, and various additives (12). Two major groups of paints are latex (water-based) and alkyd (oil-based) paints (13). The major difference between latex and alkyd paints is that the major liquid portion of latex paints is water while the liquid in oil-based paints consists of petroleum distillates and other organic solvents such as toluene and xylene. Latex paints are the most common type for house use from exterior paint and trim, to interior walls and woodwork. Generally, a paint store has about 10 different colors of base pigments, and paints are sold by mixing pigments with other components. To test our hypothesis, we purchased and analyzed paint pigments from three paint stores. According to IBISWorld Inc., in 2007 these companies account for about 70% of the market share in the United States.

Materials and Methods

Reagents. Paint pigments were purchased from three different paint retailers: Sherwin Williams, PPG Pittsburgh, and Vogel, in Iowa City, Iowa in 2009. A calibration standard solution with a full suite of 209 PCB congeners was prepared from five PCB congener solutions purchased from AccuStandard (New Haven, CT). Acetone and hexane (pesticide grade) were purchased from Fisher Scientific (Fair Lawn, NJ).

Sample Analysis. The extraction method was modified from U.S. EPA method 3545 (14). In brief, approximately 5.0 g of the fresh pigment sample was accurately weighed and mixed with combusted diatomaceous earth, then spiked with 50 μ L of 500 ng/mL surrogate standards containing PCB14 (3,5-dichlorobiphenyl), PCB65 (2,3,5,6-tetrachlorobiphenyl) and PCB166 (2,3,4,4',5,6-hexachlorobiphenyl) (Cambridge Isotope Laboratories, Inc.). The samples were extracted utilizing a pressurized fluid extraction (Accelerated Solvent Extractor, Dionex ASE-300) with a mixture of acetone and hexane (1:1, v/v). The extract was concentrated to \sim 2 mL from \sim 200 mL, and the concentrated extract was transferred to a glass test tube; \sim 2 mL of concentrated sulfuric acid was slowly added and mixed. Hexane (8 mL) was used to extract the acidified mixture 3 times after a 10-min mechanical shaking and centrifugation at 3000 rpm/min for 5 min. The pooled extract was concentrated down to \sim 2 mL and passed

through a silica gel column prepared with 0.1 g of combusted silica gel at the bottom and 1 g of acidified silica gel (2:1 silica gel:concentrated sulfuric acid by weight). Hexane (10 mL) was used to elute PCBs from the column and the eluate was concentrated down to ~0.5 mL for PCB analysis. All samples were analyzed in duplicate, and the average is reported.

The final extract was spiked with 20 ng of PCB204 (2,2',3,4,4',5,6,6'-octachlorobiphenyl) as internal standard (Cambridge Isotope Laboratories, Inc.). All 209 PCB congeners, in about 170 chromatographic peaks, were analyzed using a gas chromatograph with mass selective detection (GC-MS/MS) modified from the EPA method 1668A (15). The quantification of PCB congeners was performed by an Agilent 6890N gas chromatograph with an Agilent 7683 series autosampler coupled to a Waters Micromass Quattro micro GC mass spectrometer (Milford, MA) operating under electron impact (EI) positive mode at 70 eV and multiple reaction monitoring (MRM), and the trap current was 200 μ A. The retention windows were defined by PCB parent/daughter ion pairs from mono- to deca- homologues which were 188/152, 222/152.10, 255.96/186, 291.92/222, 325.88/255.90, 359.84/289.90, 393.80/323.90, 427.76/357.80, 461.72/391.83, 497.68/427.70, respectively.

Quality Assurance/Quality Control. During extraction of paint pigments using ASE, samples and blanks (combusted diatomaceous earth) were alternated on the instrument to avoid, and detect, any cross contamination between pigment samples. The average recoveries of PCB 14, PCB 65, and PCB 166 surrogate standards injected in every sample were $56 \pm 15\%$, $85 \pm 25\%$, and $86 \pm 20\%$, respectively. Di- to tri- PCB concentrations in samples were corrected for PCB14 recovery and tetra- to deca-PCBs for PCB166 based on our analytical method validation using Standard Reference Material 1944.

Results and Discussion

Inorganic and Organic Pigments. Inorganic pigments are produced from either naturally mined pigments (sienna, umber, ochre) or synthetically manufactured pigments (iron oxide, carbon black, etc). Titanium dioxide is the most important white pigment in the industry because of its high refractive index, reflectance, ease of dispersion, brightness, and opacity (16, 17). Titanium dioxides and iron oxides account for approximately 70% and 15% of world consumption of inorganic pigments (18, 19). No PCBs were found in inorganic pigments which primarily contain titanium dioxide, iron oxide, raw umber, or carbon black (Figure 1 and Table 1). PCB formation is expected to be associated with chlorinated solvent or intermediates used in the manufacturing process of pigments.

PCBs were primarily found in organic paint pigments with a concentration range from 2 to 200 ng/g fresh weight (f.w.) in 15 of 33 analyzed paint pigments (Figure 1 and Table 1) in this study. Pigment chemical structures were provided by Sherwin Williams. PPG Pittsburgh and Vogel did not provide this information, although we were able to determine several pigment types based on their material safety data sheets. Most orange, red, and yellow pigments are made from azo pigments, and PCBs are only found in two groups of organic pigments: azo pigments and phthalocyanine pigments. For pigment samples from Sherwin Williams, we clearly see PCBs are only present in these two types of pigment. Chlorinated solvents or intermediates are usually involved to produce these two types of organic pigments, and side-reactions of these chlorinated compounds result in formation of PCBs during the manufacturing process. The EPA is aware of the presence of PCBs in diarylide pigments and phthalocyanine pigments. Diarylide pigments belong to the azo category of pigments (20). However, we observed the presence of PCBs not only in diarylide pigments but also in other azo pigments

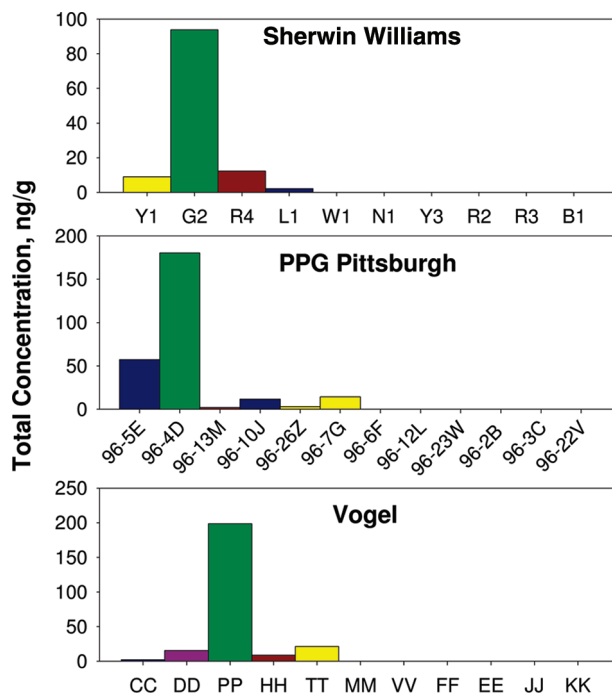


FIGURE 1. Σ PCB concentrations in 33 commercial paint pigments purchased from Sherwin Williams, PPG Pittsburgh, and Vogel paint stores.

such as Hansa yellow, quinacridone, isoindolinone, and maybe more, since some pigment types are unknown.

Azo and phthalocyanine pigments and chemically identical dyes are the most important groups of synthetic colorants with a great variety of industrial applications. They are used for coloring paints, inks, textiles, paper, cosmetics, leather, plastics, food and other materials (21, 29). The widespread use of these pigments explains the presence of PCB11 in commercial goods common throughout modern society, such as newspapers, magazines, and cardboard boxes (11). Although we do not know if inadvertent PCBs have adverse effects on human health, there are many potential routes for human exposure to these PCBs through inhalation, dermal exposure, and ingestion due to their physicochemical characteristics of semivolatility, hydrophobicity, and persistence.

Congener Profiles. The detailed PCB distribution profile in each pigment is provided in Tables S1–S3, and two examples are presented in Figure 2. The pigments, Y1 of Sherwin Williams and 96-26Z of PPG Pittsburgh, are both yellow and made from monoazo yellow pigments. The synthesis of monoazo yellow pigments involves the coupling of a diazotized substituted aniline with a coupling component containing an active methylene moiety in a linear structure (18). There are different PCB distribution profiles in different pigments due to various manufacturing processes for different pigments or even the same pigments. The same type pigment might have different starting materials, intermediates, or manufacturing conditions. For a particular manufacturing process, only very limited numbers of chlorinated compounds are involved; however, up to 22 congeners were detected in one pigment. Among these detected congeners, PCBs 77, 114, and 123 are dioxin-like congeners which have distinct toxic properties. A variety of PCB profiles in paint pigments were observed in this study (Tables S1–S3), although the reason for their presence is not completely understood. PCB11 was most often detected: it was found in 13 of 15 pigment samples for which any PCBs were detected, followed by PCBs 8, 6, 4, 1, 12/13, 2, 3, and 209—each with more than 40% detection frequency (Figure 2). PCB congeners of all chlorination levels were found in the pigments.

TABLE 1. Colors and Types of Commercial Paint Pigments Purchased from Three Paint Stores^a

paint store	code	color	pigment type
Sherwin Williams	Y1	yellow	hansa yellow
	G2	green	phthalocyanine green
	R4	red	isoindolinone
	L1	blue	phthalocyanine blue
	W1	white	titanium dioxide
	N1	raw umber	raw umber titanium dioxide
	Y3	deep gold	iron oxide
	R2	maroon	iron oxide
	R3	magenta	quinacridone
	B1	black	carbon black
	PPG	96-5E	blue
96-4D		green	phthalocyanine green
96-13M		durable red	/
96-10J		carbazole violet	/
96-26Z		medium yellow	monoazo yellow
96-7G		durable yellow	/
96-6F		red	iron oxide
96-12 L		raw umber	/
96-23W		white	titanium dioxide
96-2B		lamp black	/
96-3C		yellow oxide	iron oxide
96-22 V		violet	quinacridone
Vogel		CC	blue
	DD	magenta	/
	PP	green	phthalocyanine green
	HH	exterior red	/
	TT	medium yellow	/
	MM	red oxide	/
	VV	white	/
	FF	raw umber	/
	EE	black	/
	JJ	yellow oxide	/
	KK	brown oxide	/

^a "/": proprietary.

However, lower chlorinated PCB congeners dominated in most pigment samples except in phthalocyanine green pigments which contain very high levels of PCB209 relative to other detected congeners. PCB209 accounts for approximately 66%, 33%, and 50% of total PCBs in phthalocyanine green pigments of G2, 86-4D, and PP, respectively, from three different paint stores. It is interesting that although dissimilar in structure, both PCB 11 and PCB 209 are non-Aroclors that could be used as signatures of pigment use or discharge.

Formation Mechanism. Phthalocyanine Pigments. Copper phthalocyanine pigments are the most widely used blue and green pigments for various applications (16, 21). Two different general processes are used for commercial production of phthalocyanine pigments: one is from phthalic anhydride, urea, and copper or a copper salt, and the second is from phthalonitrile and copper or a copper salt (22). The first route is less expensive and is usually used to produce phthalocyanine pigments for high-volume and low-cost applications such as paint pigments and dyes for textiles and paper; the second route is more expensive but produces high-quality and high-purity phthalocyanine pigments such as charge generation materials for electrophotography (21). The first urea process usually involves organochlorine solvent such as di- or trichlorobenzene as the reaction medium. Uyeta et al. showed that starting materials (urea, phthalic anhydride, copper chloride, ammonium molybdate) and the initial reaction medium (di- or trichlorobenzene) did not contain PCB congeners (22), so they are not a direct source of PCBs in pigments.

PCB formation mechanisms (Figure 3) are proposed for the urea manufacturing process. Phthalocyanine blue is

produced from starting materials without chlorines, and phthalocyanine green is derived from phthalocyanine blue by chlorination (21). Lower chlorinated PCB congeners are produced as by-products during the manufacturing process of phthalocyanine blue pigments from the reaction medium chlorobenzene (23). The reaction medium, dichlorobenzene or trichlorobenzene, can form tetra-, penta-, and/or hexa-PCB congeners by a reaction with each other under heat through a free radical mechanism (the dashed arrow pathway in Figure 3) (24, 25). The resulting PCB congeners may thermally degrade further into lower chlorinated congeners by the same mechanism (26). Mono- through tetra-chlorobiphenyls have been created through a free radical mechanism from pyrolysis and combustion of other chlorinated organics at temperatures ranging from 300 to 700 °C (27). The free radical mechanism to form PCBs from chlorobenzenes has been experimentally demonstrated (25). The temperature is usually below 300 °C during the pigment manufacturing process; however, the presence of copper chloride and ammonium molybdate as a catalyst might promote this mechanism at a lower temperature range (25, 28). To gain proper brightness, shade, strength, and flow properties of synthetic pigments, various factors including the reaction temperature and the drying temperature might be altered to meet these purposes (20). With increase of the reaction temperature, the total PCB formation increases independent of reaction time after the initial 2 h (25). Lower chlorinated benzenes might produce more PCB congeners than higher ones.

During the process of perchlorination from phthalocyanine blue to phthalocyanine green, decachlorobiphenyl (PCB 209) is formed along with some other highly chlorinated

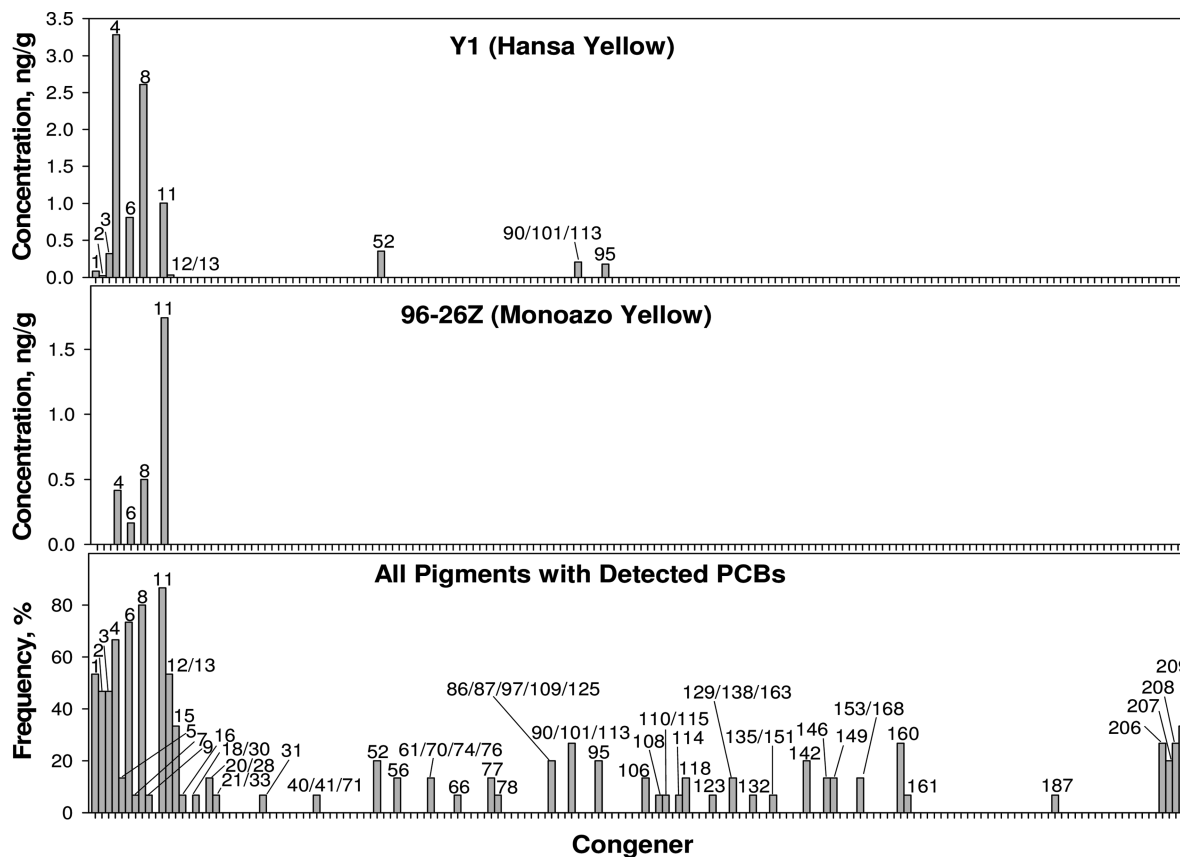


FIGURE 2. Examples of PCB profiles in paint pigments (top two plots) and the frequency of congener detection in the 15 pigments with detected PCBs (bottom plot).

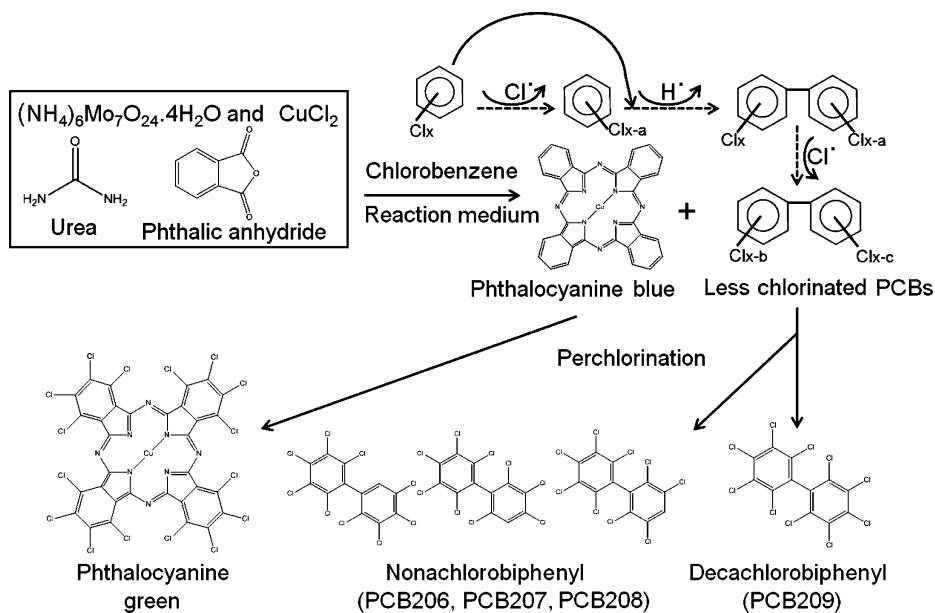


FIGURE 3. PCB formation mechanisms in the manufacture process of phthalocyanine blue and phthalocyanine green. The subscripts x, a, b, and c refer to the number of chlorine atoms.

congeners such as nonachlorobiphenyls (PCBs 206, 207, and 208) from less chlorinated congeners (Figure 3). This explains the presence of much more nona- and deca-PCB congeners in phthalocyanine green than in phthalocyanine blue, which can be observed by comparison of PCB distribution profiles in L1 and G2 from Sherwin Williams, 96-5E and 96-4D from PPG Pittsburgh, and CC and PP from Vogel.

Azo Pigments. Azo pigments are the most important group of synthetic colorants with the largest fraction (more than

50%) of organic pigments on the market (29). Azo pigments have a wide range of colors covering almost the entire visible spectrum although blue and green colors are mostly provided by phthalocyanine and two other pigments (18). Some vivid colors of azo pigments are commercially dominant, especially reds, oranges, and yellows.

Azo pigments are almost exclusively produced through a reaction sequence of diazotization and coupling to afford the azo group ($-\text{N}=\text{N}-$) which is the chromophore respon-

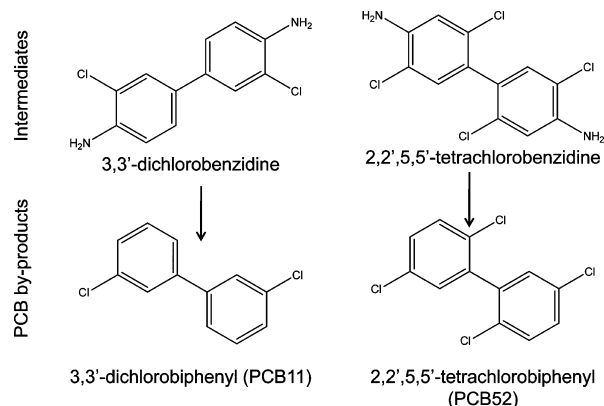


FIGURE 4. Possible formation pathways of PCB11 and PCB52 in pigments.

sible for its vivid colors (18). The reaction involves a primary aromatic amine as a diazo component and a nucleophilic aromatic or aliphatic compound with active methylene groups as a coupling component. An aromatic amine such as a mono-, di, or trichlorinated aniline is typically involved in the diazotization reaction as a diazo component, and frequently they are also used as coupling components. Another important group of diazo components for azo pigment formation include 3,3'-dichlorobenzidine, and to a lesser extent 2,2',5,5'-tetrachlorobenzidine, 3,3'-dimethoxybenzidine, and 3,3'-dimethylbenzidine (18). The last two compounds do not contain chlorines which are required elements for formation of PCB congeners.

For synthesis of azo pigments, there are more than 10 common intermediates and starting materials such as chlorinated aniline and chlorinated benzidines that can potentially have side-reactions to produce PCBs. PCBs are probably formed by coupling of bis-diazotized dichlorobenzidines or tetrachlorobenzidines under basic conditions as a result of the decomposition of the diazo moiety. Polychlorinated anilines can be also used to form PCBs through the free radical mechanism, and the free radical rearrangement of chlorine positions might play a significant role in varieties of PCB congeners with limited chlorinated intermediates. Lower PCB congeners may be formed by carrying out the coupling process at lower pH or in the presence of unsaturated aliphatic compounds such as acylamides (20). For example, 3,3'-dichlorobenzidine and 2,2',5,5'-tetrachlorobenzidine are probably the diazo components for TT and HH pigments that contain high PCB11 and PCB52 (Table S3), respectively. Unfortunately, we cannot verify the pathways for pigments considered proprietary by manufacturers; however, the links of intermediates and PCB by-products can be illustrated structurally (Figure 4). The azo pigments based on 3,3'-dichlorobenzidine appear preponderant (30), which might explain partially the consistency of PCB11 detection.

Environmental Emission. A wide variety of organic pigments are commercially available; however, in terms of chemical structure, almost all currently produced organic pigments belong to four different groups: azo pigments and lakes (salt type), phthalocyanine pigments, polycyclic pigments, and heterocyclic pigments (20). In spite of accelerated progress in the synthesis of organic pigments, commercially available pigments at present are chemically identical to those produced historically since the use of synthetic pigments. PCB congeners are primarily detected in azo pigments and phthalocyanine pigments. PCB11 is consistently detected in almost all azo and phthalocyanine pigments, and it is absent or in very low relative concentrations in commercial Aroclor mixtures. Therefore, PCB11 can be regarded as a key indicator of PCB emission from de novo synthesis as by-products of

industrial synthetic process of paint pigments. PCB11 is the fifth highest congener and ubiquitous in Chicago air (4). Although we do not know the contribution of PCB congeners from paint pigments to the airborne PCBs in the environment, these congeners, especially low chlorinated congeners, might contribute a significant portion as PCB11 because of their high volatility.

Based on 40 CFR 761.80, PCBs are allowed at less than 25 mg/kg with a 50 mg/kg maximum in commerce of diarylide pigments or phthalocyanine pigments when leaving a manufacturing site or imported to the United States. PCB levels in the examined paint pigments are all below the regulatory standard; however, paints are being extensively and constantly used especially in urban areas. PCBs might accumulate due to their resistance to degradation in the environment. It has been reported that PCB11 and total PCB levels in air are directly proportional to human population density (7, 31, 32). To our knowledge, pigments or dyes are the only significant source of PCB11. The elevation of PCB11 in air must be associated with human activity utilizing pigments or dyes. The presence of PCB11 indicates paint should be an important source of airborne PCBs although the link of PCBs in paint pigments and PCBs in air is still not clear.

Acknowledgments

We thank our analytical core coleader, Craig Just, and our laboratory director, Collin Just. We also thank Kristin Isley and Timothy Schulz for assisting with sample preparation in the laboratory. Funding for this work was provided by the National Institute for Environmental Health Sciences (NIEHS/NIH) Superfund Basic Research Program (ES013661).

Supporting Information Available

Three tables. This information is available free of charge via the Internet at <http://pubs.acs.org>.

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ES902413K

Exhibit G

Technical Service

*new work / Sat
Factory / papers
Rec'd from
C.B. Klay
W.R.S.*

MAY 3 1946

In the Technical Service Department all raw materials are tested for strength and color before they can be used. All experimental and research work is taken care of in this Department.

When the factory makes a batch of paint, sample cans are taken from the tank and sent to the Technical Service Dept. where they are checked against the standard sample for color, gloss, consistency and drying, brushed out, sprayed or baked, whatever the case may be. The weight of the paint per gallon must also be up to standard before the Factory can be notified to go ahead and fill.

A weather-meter is installed in this Department for testing out our products. Panels are put in this machine and given practically the same test as they would get if they were exposed to the elements.

To match a wet sample, it is centrifuged which separates the pigment and vehicle and then analyzed. Samples of oil are tested for characteristics such as acid value, iodine number, percentage of foets and specific gravity, and must meet our specifications. Thinners are tested for specific gravity, odor, flash point and color.

"B" Grinding Department

In this Department all mixing and grinding is done. They have 16-36" mills, and two pebble mills in this Dept.

The first process in making paint--The pigment is put into mixing tanks with a certain percentage of oil to bring the paint to a semi paste consistency; after being in the tank for approximately three hours (3), the paint is then turned into a grinding mill; from the mill it goes into a filling tank where the oils, thinners, or driers are added and the proper shading done, before samples can be taken from the filling tank and sent to the Technical Service Dept. for final approval.

Pebble Mills:—One is of 150 gallons capacity and the other 500 gallons capacity. The small mill takes about 3½ hrs. to make a batch of paint and the large mill takes about 17 hrs. Pebble mills are so designed that it takes only one operation to make a batch of paint, all the pigments and vehicles are put in the tank at the same time.

In this Department some of the following lines of paint are made: S.W.P.; Flattons; Roof & Bridge; Floor Paint; Family Paint; Trimrite; Stucco & Concrete; Shingle Stain; Copper Paint, etc.

Under normal conditions this Department will make approximately 5000 gallons of paint a day. Last year this Dept. made on an average of 106,000 gallons of paint a month, and during the year made about 1,624,500 gallons.

"A" Department

In this Department the same process of making paint is carried out as in "B" Grinding Department, the only difference is the paint is filled directly into the package in semi paste form--the thinners and driers are added by the customer. Semi Paste; Flock Colors; Freight Car Paints; A.I.P.; Oil Colors; Machine Fillers; Zines; etc. are some of the paints manufactured in this Department.

THIS DEPARTMENT WAS DISCONTINUED C.B.

The large chasers are used to grind certain pigments finer.

Last year, this Department averaged about 17,600 gallons of paint a month and during the year about 181,000 gallons of paint. They also filled on an average of 41,900 pounds of paste goods a month and during the year, made about 526,000 pounds.

"D" Department

In this Department we have 8 tandem mills, 6-21" mills, one pebble mill and three roller mills.

The tandem mills are used exclusively for the best grades of Enamel paint, they are water cooled. The roller mills are more economical and still put out a fine piece of goods in less time than it took under the old system of making paint on the 21" mill, pigment on these mills can be ground finer, and the output greatly increased and better results obtained.

In this Department, they make Enamels; Brighten-Up Stains; Oil Stains; Enameloid; Eloc-lac; Kem Colors; Lustral Enamels; Wagon and Implement Paints; etc.

Last year this Department averaged about 28,400 gallons of paint a month and during the year made about 394,000 gallons.

"B" Filling Department

After the Factory receives the final approval from the Technical Service Dept., the paint is ready to be filled. The cans are properly stamped showing date of manufacture, the scales set to the correct weight, before filling.

One thing of importance is the special care taken to see that no skin paint, dirt, or foreign matter goes in the can. This is prevented by screens and separators. The new Kem Enamel Department is also on this floor.

Labeling Department

All labels except bottles and five gallon pails are put on the cans by machine which makes a neat looking package. After the cans are labeled, they are put into cartons; the cartons are then run thru a closing machine and transferred to the new warehouse building by conveyors.

Varnish Dept.

Varnish is a combination of oil and resin under heat. The gum and resin varies not only as to the quality of the Varnish but also as to the purpose for which it is to be used.

China Wood Oil is the oil generally used in varnishes, which are to be exposed to the elements because of its water resisting qualities.

- 1 -

Varnish Dept. (Cont'd.)

Varnish intended for interior enamels usually are made with some proportion of linseed oil.

China Wood Oil as the name indicates comes from China and is the oil expelled from the tung nut.

Gums are found in most tropical climates. Some deposits are buried in the ground; others are close to the surface. They are the hardened sap of trees and other vegetable matter that have been buried in the ground for centuries. Gums vary as to hardness and color. Those generally processed are: Congo, East India, Manila and Damar. It was the near exhaustion of some gum fields that brought synthetic resins into varnish production.

Shellac gum comes from India and it is the product of the lac bug. Zinsser has a booklet entitled "The Story of Shellac" which may be had for the asking. It is very interesting and instructive. Shellac is a soft resin and dissolves readily in alcohol in three to four hours.

Output last year:-

~~Varnish - Average per month - 80,000 gals.
During the year - 1,300,000 gals.~~

~~Shellac - Average per month - 16,000 gals.
During the year - 218,000 gals.~~

~~Litho-Varnish - Average per mo. 5100 gals.
During the year - 62,000 gals.~~

In order to give you some idea what goes into the manufacture of paint, please note the following:

1. Minerals used in paints:- Lead, Zinc, Titanium, Iron Oxides.
2. Ochre, Umber and Sienna are earth materials.
3. Silica, China Clay, Calcium Carbonate, Asbestine, Barium Sulphate, Magnesium Carbonate. These are inert materials.
4. Oslo, our leaded zinc, is processed so that each particle would analyze the same as 100 lbs. White lead is not mixed with zinc but lead ore is sublimed or burned in the mineral form with zinc ore thus forming the white pigment.

Asbestine is a natural product but is cleansed of foreign matter so that the analysis shows nearly all magnesium silicate.

Lithopone is made by taking the ores of zinc and barytes and in refining them together. Lithopone is made in various proportions of zinc sulphate and barium sulphate.

Barytes is a natural product which is mined and cleansed of foreign matter.

5. Green, blue and yellow are made from minerals but by a chemical process which produces the colors. Green is made of lead chromate pigment and iron blue, the yellows from lead chromates and the blues from iron or dyes.

Exhibit H

Gordon S. Kuntz, Ph.D.
Sherwin-Williams Company
101 Prospect Avenue, N.W.
Cleveland, Ohio 44115-1075

RE: Sherwin-Williams Company (Sherwin-Williams)
60 Lister Avenue
City of Newark, Essex County
ISRA Case No. E99382

- ISRA Investigation Report (PA/SI/RI) Dated: October 26, 2001
- July 3, 2002 Meeting
- Remedial Investigation Workplan for Groundwater Dated: August 5, 2002
- ISRA Investigation Report Addendum Dated: August 2002
- Additional Groundwater RIR Dated: November 2002
- Bedrock and Surficial Groundwater Aquifers Investigation Dated: November 2002
- Dioxin Groundwater Investigation (May 2002) Dated: December 2002
- Site Investigation Report – 13,000 Gallon Naphtha UST Dated: December 2002
- May 21, 2003 Meeting
- Railroad Reaches Remediation Report Dated: October 1986, Submitted May 2003
- August 13, 2003 Permit Pre-Application Meeting

Dear Dr. Kuntz:

Please be advised that the New Jersey Department of Environmental Protection (NJDEP) has completed its review of the above referenced documents. All of the reports were prepared by Weston Solutions, Inc. on behalf of Sherwin-Williams, with the exception of the October 1986 Railroad Reaches Remediation Report. The Railroad Reaches Report was prepared by International Technology Corporation on behalf of Diamond Shamrock Corporate Company, and Weston Solutions, Inc. submitted a copy of the report in May 2003. The NJDEP is willing to meet with Sherwin-Williams at the NJDEP and/or in a conference call to discuss the comments below regarding the referenced reports.

A. Site History

Sherwin-Williams Summary: The site consists of an approximately thirteen-acre parcel. The site is underlain by historic fill material imported prior to the year 1892. The surrounding land use is heavy industrial with some residential and commercial properties also present within the area. Regional ground water contamination has been documented. Numerous industrial facilities in the surrounding area have had documented impacts on the environment.

Prior to 1910	Union Chemical Works occupied the northeast portion of the site and EC Brown Shipyard occupied the site. A lead Foundry was also noted to be present in the eastern most portion of the site.
1908 to 1937	Consolidated Color and Chemical Company (CCC) occupied parcels of land on the southeastern portion of the site.
1937 to 1960s	Parcels occupied by Roanoke, Inc. – A successor company to Consolidated Color and Chemical Company.
1960s to 1999	Sherwin-Williams took over the parcels occupied by Roanoke, Inc. and converted the area for use as employee parking.

NJDEP Comments: The VOCs detected within this area are not delineated. Delineation of VOCs to the north of B-2 and B-4 is required. The presence of higher concentrations of CaPAHs at location B-1 also must be addressed. Delineation should continue to the property boundary. Delineation of zinc north of B-4 (0-6" bgs) should also be conducted. Sherwin Williams shall clarify whether the black sand layer that had an organic odor was sampled. If this layer was not sampled, sampling is required.

Sherwin-Williams should supply a list of the insecticides that were stored at this AOC.

Sherwin-Williams Response

VOCs were not detected above the MSSCC at either sample interval from boring B-4. Sherwin-Williams does not propose any additional sampling around B-4. Samples were collected to the north of Boring B-2 to complete horizontal delineation. Vertical delineation was completed with samples collected from previous investigations. No further delineation is proposed for this AOC. A summary of the results is provided in the attached report under AOC 23 of the attached addendum report.

The comments regarding CaPAHs and zinc are discussed in detail below in the response to Comment 29.

A sample was not collected for VOC analysis from the black sand unit that had the burnt organic odor. The black sand unit was a natural sand layer present immediately above the meadow mat unit. The organic smell was from the naturally decaying organic material in the meadow mat unit. Sherwin-Williams proposes to collect no samples from this unit based on the following:

- since contamination above this interval is very limited (see figure 8A VOC exceedances – AOC 23 sampling results) in the intervals above it; and
- groundwater contamination in both the permanent and temporary wells surrounding the area where the black sand unit was present is very limited.

The insecticides stored at this AOC were DDT and copper arsenate.

24. AOC 24 – Site Groundwater

Groundwater sampling has been performed in an effort to determine the existence, nature, and extent of the groundwater contamination. Sherwin-Williams states the objective of the groundwater investigation is to collect sufficient data to fully delineate both the horizontal and the vertical extent of groundwater contamination from potential on-site and off-site sources.

a. Groundwater Screening Investigation

The aquifer screening investigation was divided into surficial and deep investigations. Groundwater was encountered at depths ranging from approximately 2 feet bgs near the Passaic River to approximately 6 feet bgs near Lister Avenue. The surficial locations are defined as samples from the water-bearing unit above the meadow mat, which was encountered at approximately 7 to 11 feet bgs, and the deep locations are from below the meadow mat. Surficial groundwater samples were collected at 25 locations from the surficial overburden aquifer.

The 25 surficial temporary screening points were installed using a Geoprobe rig. The borehole was advanced to the top of the meadow mat lithologic unit. A one-inch PVC well screen (5-foot section) and casing was installed in the borehole. Prior to collecting the analytical samples, approximately 2 gallons of water was purged from the well using a peristaltic pump. The analytical sample was then collected using a disposable bailer. During September

Sherwin-Williams Response

Sherwin-Williams will comply with the above requirements. No response required.

If you have any questions, please contact the Case Manager, Robert P. Posey, at (609) 984-2159.

Prepared By:

Approved By:

Robert P. Posey, Case Manager
Bureau of Northern Case Management

Ann H. Wolf, Supervisor
Bureau of Northern Case Management

c: Chris Lacy, BEERA
David Morrow, BGWPA
Isabel Rodrigues, US EPA Region 2
Newark Health Department
Rob Gascoyne, Weston Solutions, Inc.

Exhibit I



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION 2
290 BROADWAY
NEW YORK, NY 10007-1866

SEP 18 2017

**BY EMAIL AND CERTIFIED MAIL
RETURN RECEIPT REQUESTED**

To: See List of Addressees - Attachment A

Re: Allocation for Operable Unit 2 Remedial Action
Diamond Alkali Superfund Site, Essex and Hudson Counties, New Jersey

Dear Sir/Madam:

On August 28, 2017, the U.S. Environmental Protection Agency ("EPA") hosted a meeting at its New York City offices to provide you with an opportunity to share your views on the Agency's proposed settlement framework for implementation of the remedy selected for the lower 8.3 miles of the Passaic River, which is Operable Unit 2 ("OU2") of the Diamond Alkali Superfund Site (the "Site"). I want to once again thank all of the parties that participated in the meeting. Your participation has helped both EPA and the other parties better understand the issues and concerns regarding the framework.

After careful consideration, the Agency has concluded that the allocation process should include all of the potentially responsible parties ("PRPs") for OU2 (apart from the Passaic Valley Sewerage Commission ("PVSC"), the four municipal PRPs referred to below, and the PRPs that settle pursuant to the "early" cash-out settlement that EPA offered in March 2017), and should not be limited to the "middle tier" parties. Transparency and fairness are concepts that EPA has consistently stated are of importance to the Agency in this matter and, after considering your comments and concerns, we think those concepts are best served by having one allocation for all of these parties.

Numerous parties at the August 28, 2017 meeting expressed concern regarding the financial burden that would be placed on PRPs that are not responsible for the release of dioxins, furans and/or polychlorinated biphenyls ("PCBs") into the Lower Passaic River if those parties are not given the opportunity to settle with the United States for their OU2 liability, as opposed to having to implement the remedial action for OU2. EPA appreciates those concerns. As we have stated, we anticipate that with the help of the allocation process, EPA will be able to offer cash-out settlements to a number of the parties.

Similarly, EPA's expectation that the private PRPs responsible for the release of dioxins, furans and/or PCBs will perform the OU2 remedial action has not changed. It is therefore our goal that, in addition to supporting potential additional cash-out settlements, the allocation will lead to a consent decree in which those parties agree to perform the OU2 remedial action under EPA oversight.

To perform the allocation, EPA has retained AlterEcho and its senior allocation specialist, Mr. David Batson, Esq., through the Agency's prime contract with CSRA. EPA and AlterEcho invite you to attend a meeting to introduce the allocation process. Among other things, the allocation will provide opportunities for participating parties to comment on factors that should be part of the allocation and to contribute relevant information about themselves and other parties for use in the allocation. This meeting will be held on October 13, 2017 at 9:00 A.M. on the 27th floor of EPA's offices, which are located at 290 Broadway, New York, NY 10007.

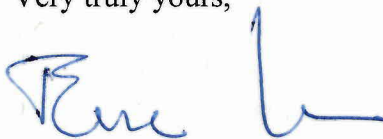
Mr. Batson has requested that each party designate a primary contact for future communications on the allocation and that the primary contact attend the October 13th meeting in person. EPA has established a conference line for others wishing to participate. The call-in number is 866-299-3188, and the conference code is 212-637-3136. Please respond to EPA by October 5, 2017 with the following information: 1) name of and party represented by the primary contact attending the meeting; 2) names of other representatives planning to call in for each such party. Your response should be directed to Alice Yeh, Remedial Project Manager, Emergency and Remedial Response Division at yeh.alice@EPA.gov or U.S. EPA Region 2, 290 Broadway - 19th Floor, New York, NY 10007.

After the allocator assigns shares to the parties, EPA will make a decision as to which parties should receive cash-out settlement offers, the dollar amount of each offer, and how the money raised by the cash-out settlements will be applied towards OU2 costs.

During the August 28, 2017 meeting, several parties raised questions concerning EPA's enforcement approach for PVSC and the municipalities to which EPA issued notices of potential liability (the City of Newark, Borough of East Newark, Town of Harrison and Town of Kearny). EPA has initiated discussions with PVSC and the municipalities about substantial contributions that, collectively, they might make to the OU2 remedy. At this time, we do not believe it would be helpful to include them in the allocation.

If you have any questions regarding this matter, please contact Assistant Regional Counsel Juan Fajardo at 212-637-3132 or fajardo.juan@epa.gov.

Very truly yours,



Eric J. Wilson
Deputy Director for Enforcement and Homeland Security
Emergency and Remedial Response Division

cc: Brian Donohue, Esq., USDOJ
Mark Barash, Esq., USDOJ
Kate Barfield, Esq., NOAA
John Dickinson, Esq., New Jersey Attorney General's Office

Attachment A – List of Addressees

Diamond Alkali Superfund Site

Lower 8.3 Miles - Passaic River

Company	Contact Information	Facility
<p>A.E. Staley Manufacturing Co., Inc. 2200 E. Eldorado Street Decatur, IL 62521-1578</p> <p>Now Tate & Lyle Ingredients Americas LLC</p>	<p>John R. Holsinger, Esq. Two University Plaza, Suite 300 Hackensack, NJ 07601 201-487-9000 (T) johnh@jrholdersinger.com</p> <p>Heidi R. Balsley, Esquire Corporate Counsel A.E. Staley Manufacturing Co., Inc. 2200 E. Eldorado Street Decatur, IL 62521 Heidi.Balsley@tateandlyle.com</p>	<p>320 Schuyler Avenue and 100 Third Avenue Kearny, NJ</p>
<p>Alden Leeds Inc. 55 Jacobus Ave. Kearny, NJ 07032</p>	<p>Mark Epstein, President Alden Leeds Inc. 55 Jacobus Ave. Kearny, NJ 07032</p> <p>Joseph Fiorenzo, Esq. Sills Cummis & Gross The Legal Center One Riverfront Plaza Newark, NJ 07102 973-643-7000 (T) jfiorenzo@sillscummis.com</p>	<p>2145 McCarter Highway Newark, NJ</p> <p>55 Jacobus Avenue Kearny, NJ</p>
<p>Alliance Chemical, Inc. Linden Avenue Ridgefield, NJ 07657</p>	<p>Fredi Pearlmutter, Esq. Lindabury, McCormick, Estabrook & Cooper, P.C. 53 Cardinal Drive Box 2369 Westfield, NJ 07091 908-233-6800 (T) fpearlmutter@lindabury.com</p>	<p>33 Avenue P Newark, NJ</p>

<p>American Ref-Fuel Co. 155 Chestnut Ridge Road Montvale, NJ 07645</p> <p>Now Covanta Essex Company</p>	<p>Nancy Tammi, Esq. VP, Associate General Counsel Covanta 445 South Street Morristown, NJ 07960 862-345-5133</p> <p>Barbara Hopkinson Kelly, Esq. Wilson Elser Moskowitz Edelman & Dicker LLP 200 Campus Drive Florham Park, NJ 07932-0668 973.735.5765 (Direct) 609.213.8589 (Cell) 973.624.0808 (Fax) barbara.kelly@wilsonelser.com</p>	<p>183 Raymond Blvd & 66 Blanchard St Newark, NJ</p>
<p>Arkema Incorporated 2000 Market Street Philadelphia, PA 19103-3222</p>	<p>Paula Martin, Esq. Doug Loutzenhiser Legacy Site Services, LLC 468 Thomas Jones Way, Suite 150 Exton, PA 19341-2528 Paula.martin@total.com</p>	<p>Wallace & Tiernan 25 Main Street Belleville, NJ</p>
<p>Ashland, Inc. 5200 Blazer Parkway Dublin, OH 43017</p>	<p>Robin E. Lampkin Ashland Inc. 5200 Blazer Parkway Dublin, OH 43017 Telephone: 614-790-3019 realmpkin@ashland.com</p> <p>William S. Hatfield, Esq. Gibbons P.C. One Gateway Center Newark, NJ 07102 whatfield@gibbonslaw.com</p>	<p>221 Foundry St. Newark, NJ</p>
<p>Atlas Refining, Inc. 142 Lockwood Street Newark, NJ 07105</p> <p>Now Atlas Refinery, Inc.</p>	<p>Steven Schroeder, Jr., President & CEO Atlas Refinery, Inc. 142 Lockwood Street Newark, NJ 07105</p> <p>Thomas H. Prol, Esq. Laddey, Clark & Ryan, LLP 60 Blue Heron Road, Suite 300 Sparta, NJ 07871 tryan@lclrlaw.com tprol@lclrlaw.com</p>	<p>142 Lockwood St. Newark, NJ</p>

<p>Automatic Electro Plating Corp. 185 Foundry Street, Suite 3 Newark, NJ 07105</p>	<p>Michael O'Rourke, President Automatic Electro Plating Corp. 1017 Applegate Parkway Waxhaw, NC 28173-6738 Michael.orourke@aol.com</p>	<p>185 Foundry Street Complex Newark, NJ (Bldgs 19, 21, 22)</p>
<p>BASF Catalysts LLC 100 Campus Drive Florham Park, NJ</p>	<p>Karyllan D. Mack, Esq. (see below)</p>	<p>Engelhard Corporation One West Central Avenue East Newark, NJ</p>
<p>BASF Corp. 3000 Continental Drive Mount Olive, NJ 07828</p>	<p>Karyllan D. Mack, Esq. Environmental Counsel BASF Corporation 100 Park Avenue Florham Park, NJ 07932 Karyllan.mack@basf.com</p> <p>David Schneider, Esquire Bressler, Amery & Ross Post Office Box 1980 Morristown, NJ 07962 dschneider@bressler.com</p>	<p>50 Central Ave. Kearny, NJ & 150 Wagaraw Rd Hawthorne, NJ</p>
<p>Benjamin Moore & Co. 51 Chestnut Ridge Rd. Montvale, NJ 07645</p>	<p>Paul Sangillo, Esq. Benjamin Moore & Co. 101 Paragon Drive Montvale, NJ 07645 201.949.6318 (T) Paul.sangillo@benjaminmoore.com</p> <p>Eric S. Aronson, Esq. David G. Mandelbaum, Esq. GreenbergTraurig 500 Campus Drive Suite 400 Florham Park, NJ 07932 aronsone@gtlaw.com</p>	<p>134 Lister Ave. Newark, NJ</p>
<p>Berol Corporation c/o Newell Rubbermaid Inc. 2707 Butterfield Road, Suite 100 Oak Brook, IL 60523</p>	<p>Andrew Sawula, Esq. Schiff Hardin LLP One Westminster Place, Suite 200 Lake Forest, IL 60045 847-295-4336 (T) asawula@schiffhardin.com</p>	<p>Faber-Castell Corporation 41 Dickerson Street Newark, NJ</p>

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<p>Viacom Inc. 11 Stanwix St. Pittsburgh, PA 15222</p> <p>Now CBS Corporation</p>	<p>Jeffrey B. Groy, Esq. VP, Sr. Counsel/ Environmental CBS Law Department CBS Corporation 2 East Mifflin Street, Suite 200 Madison, WI 53703 262-705-0579(T) jeff.groy@cbs.com</p>	<p>Westinghouse Electric 95 Orange St. Newark, NJ</p>
<p>Vulcan Materials Co. 1200 Urban Center Drive Birmingham, AL 35242</p> <p>Now Legacy Vulcan Corp.</p>	<p>Eva Fromm O'Brien, Esq. Fulbright & Jaworski Fulbright Tower 1301 McKinney Suite 5100 Houston, TX 77010-3095 713-651-5321 (T) 713-651-5246 (F) eobrien@fulbright.com</p> <p>John M. Floyd, Esq. Senior Attorney Vulcan Materials Company 1200 Urban Center Drive Birmingham, AL 35242 205-298-3745 (Direct) 205-492-4219 (Cell) 205-298-2960 (F) floydj@vmcmail.com</p>	<p>600 Doremus Ave. Newark, NJ</p>

<p>Roman Asphalt Corporation 14 Ogden Street Newark, NJ 07104</p>	<p>Michael V. Calabro, Esq. Law Offices of Michael V. Calabro 475 Bloomfield Avenue Newark, NJ 07107 973 482-1085 Mcalabro475@gmail.com</p>	<p>14 Ogden Street Newark, NJ</p>
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Exhibit J

Dioxin in the Passaic River (NJ): The Case for 2 Dioxin Sources

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Juliana Atmadja
Solomon S. Gbondo-Tugbawa,
Shane McDonald

The Louis Berger Group:
Morristown, NJ, Elmsford, NY & Exton, PA

*Battelle Sixth International Conference on the Remediation of
Contaminated Sediments*

New Orleans, LA

February 10, 2011



The Louis Berger Group, Inc.



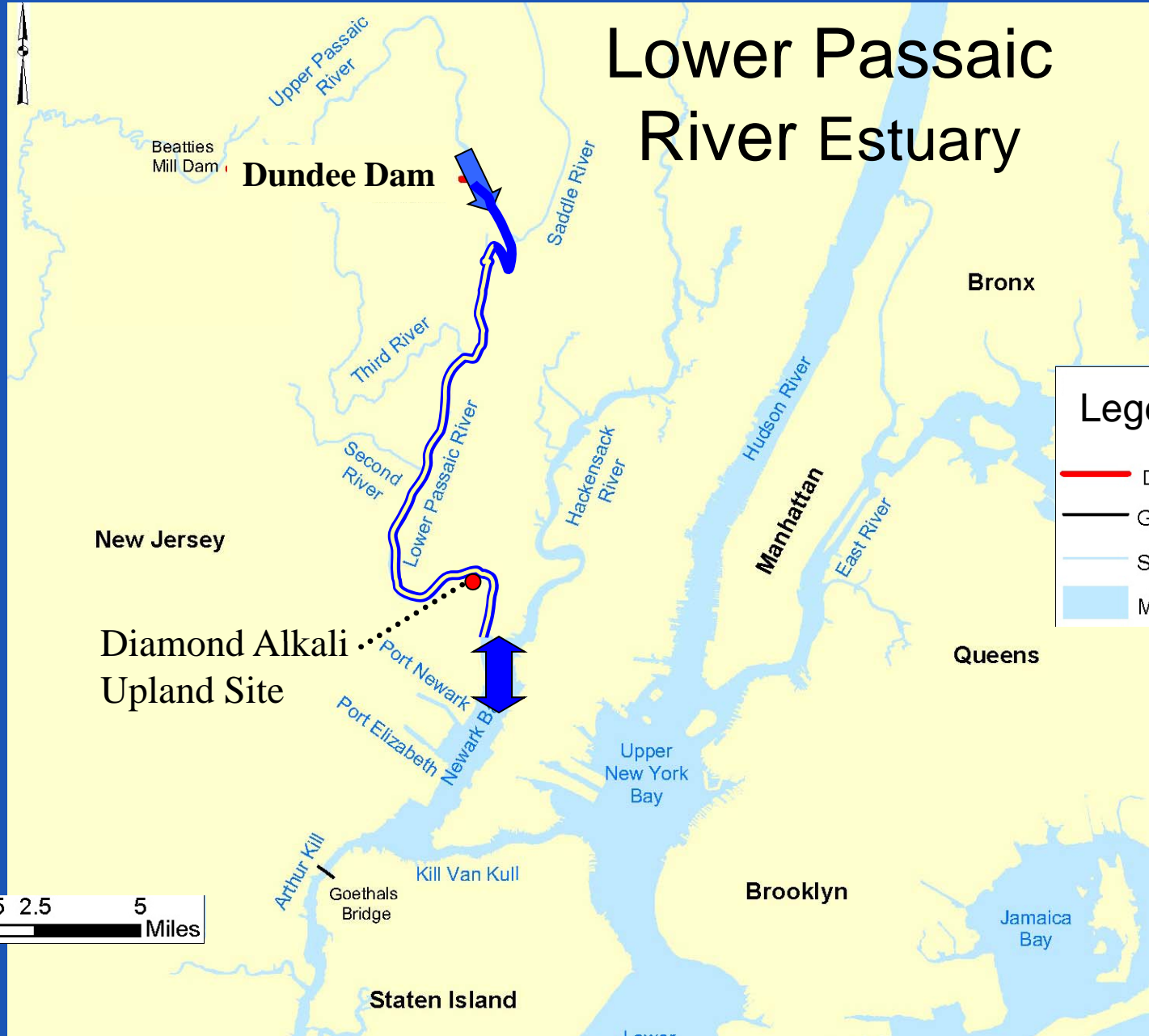
Outline

- Site Background
- Dioxin Ratios in New York Harbor.
- Dated Sediment Results for Dioxin
- Principal Components Analysis and Dioxin Ratios
- Conclusions

Although the information in this presentation has been funded by the USEPA, it does not necessarily reflect the views of the agency and no official endorsement should be inferred.

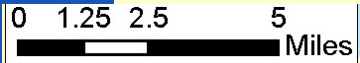


Lower Passaic River Estuary



Legend

- Dams
- Goethals Bridge
- Streams/Rivers
- Major Waterbodies



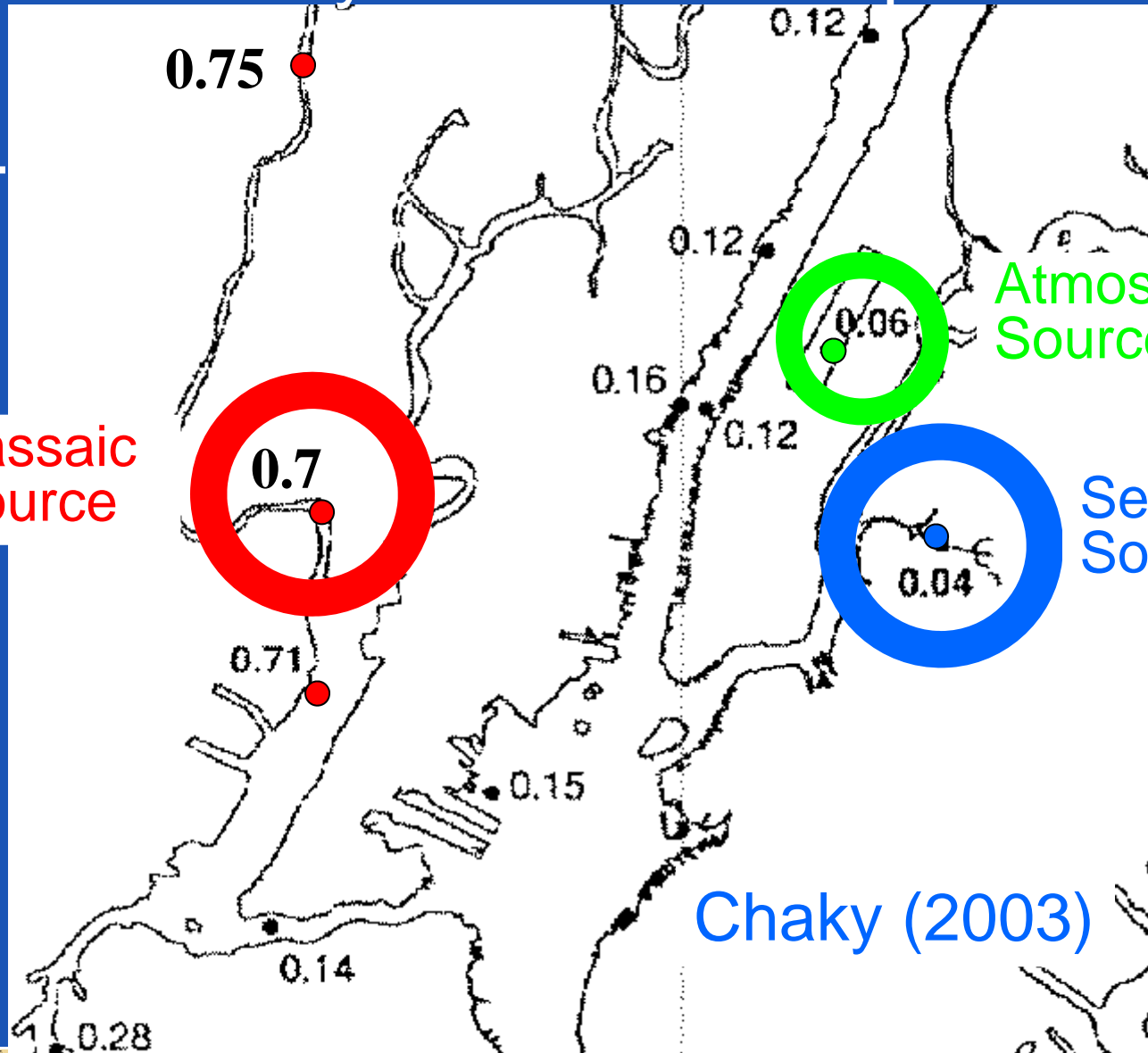
Lower Passaic River sediments have a unique signature that readily identifies their presence

$\frac{2,3,7,8\text{-TCDD}}{\text{Total TCDD}}$

Passaic Source

Atmospheric Source

Sewage Source

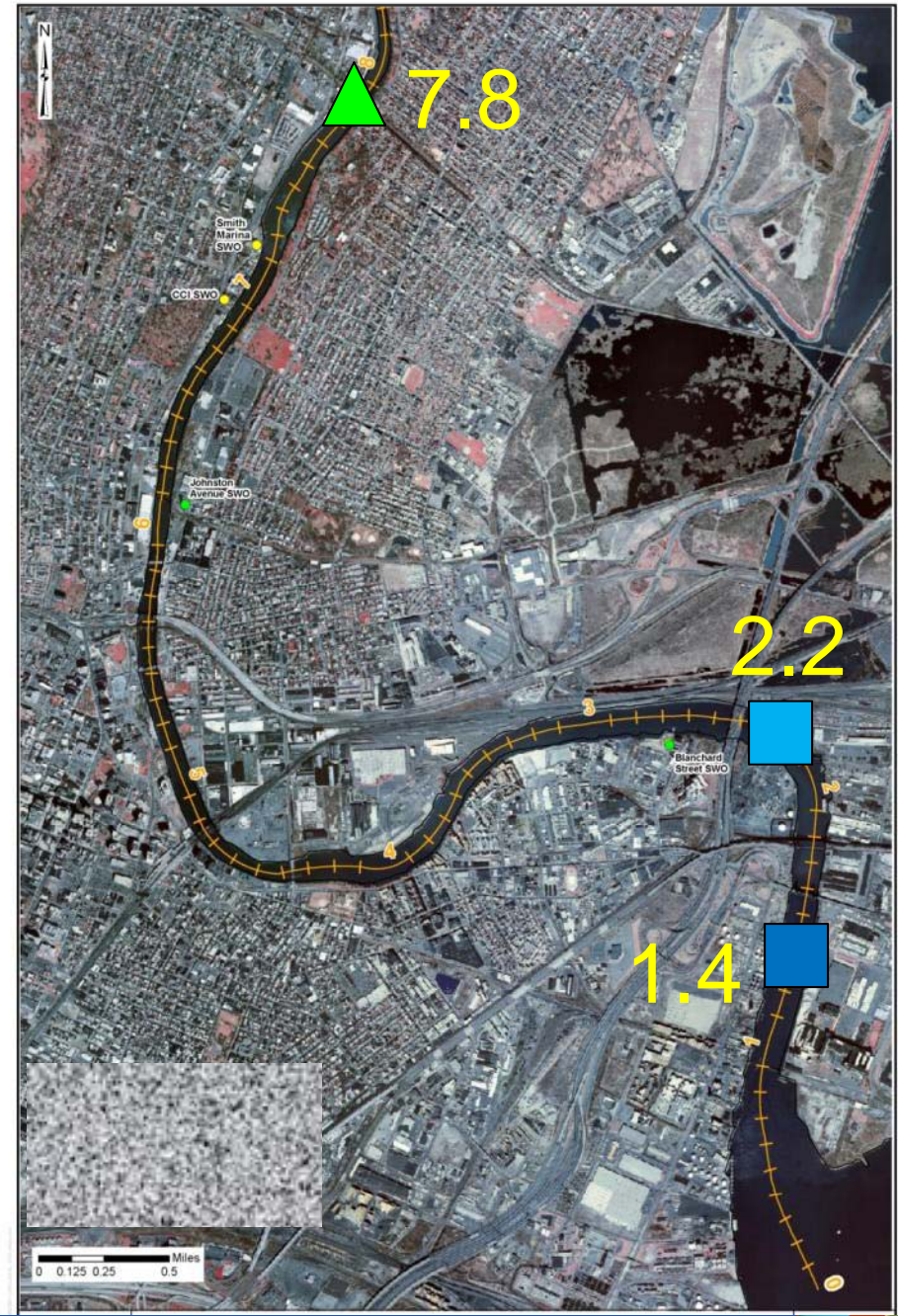
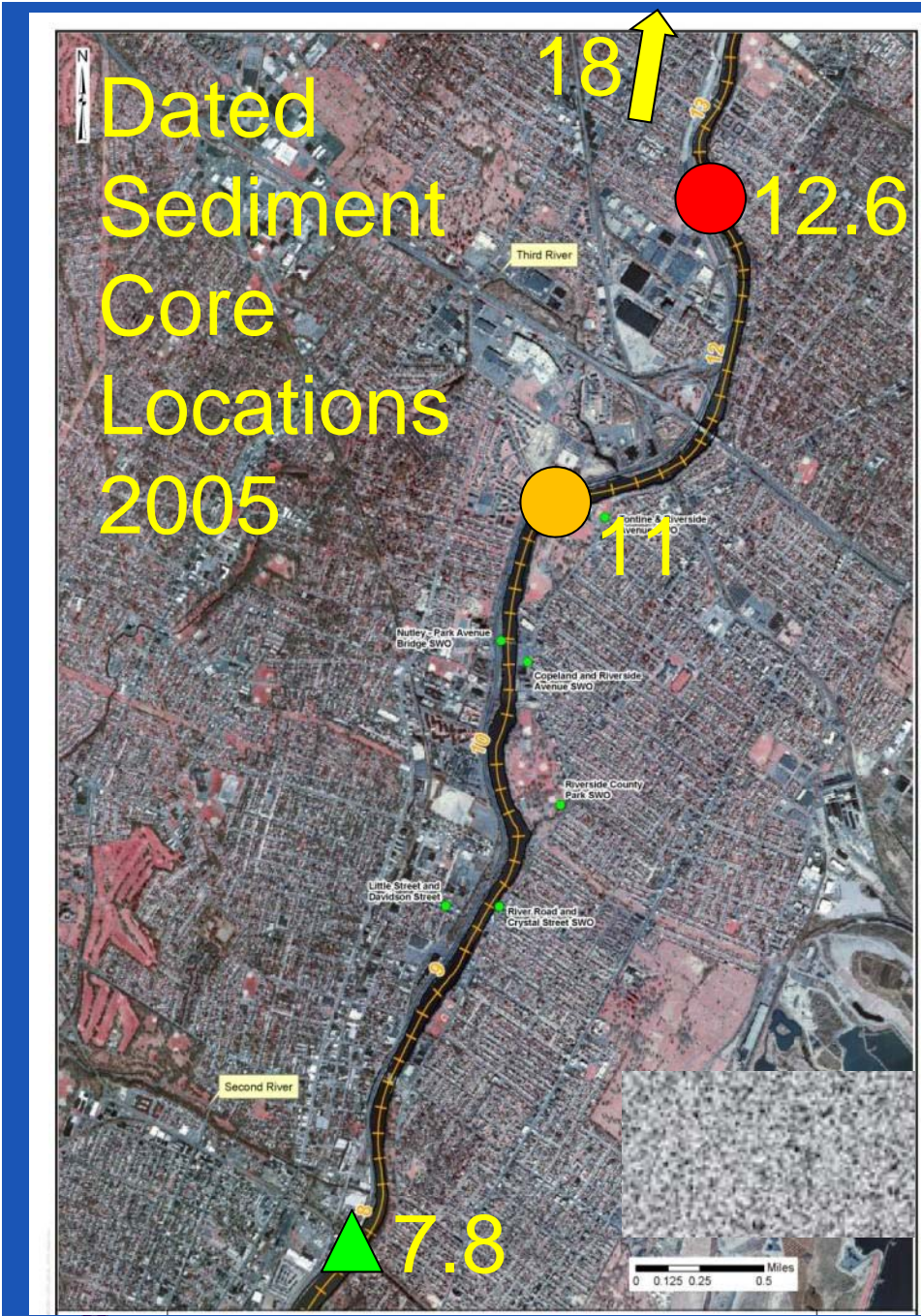


Chaky (2003)

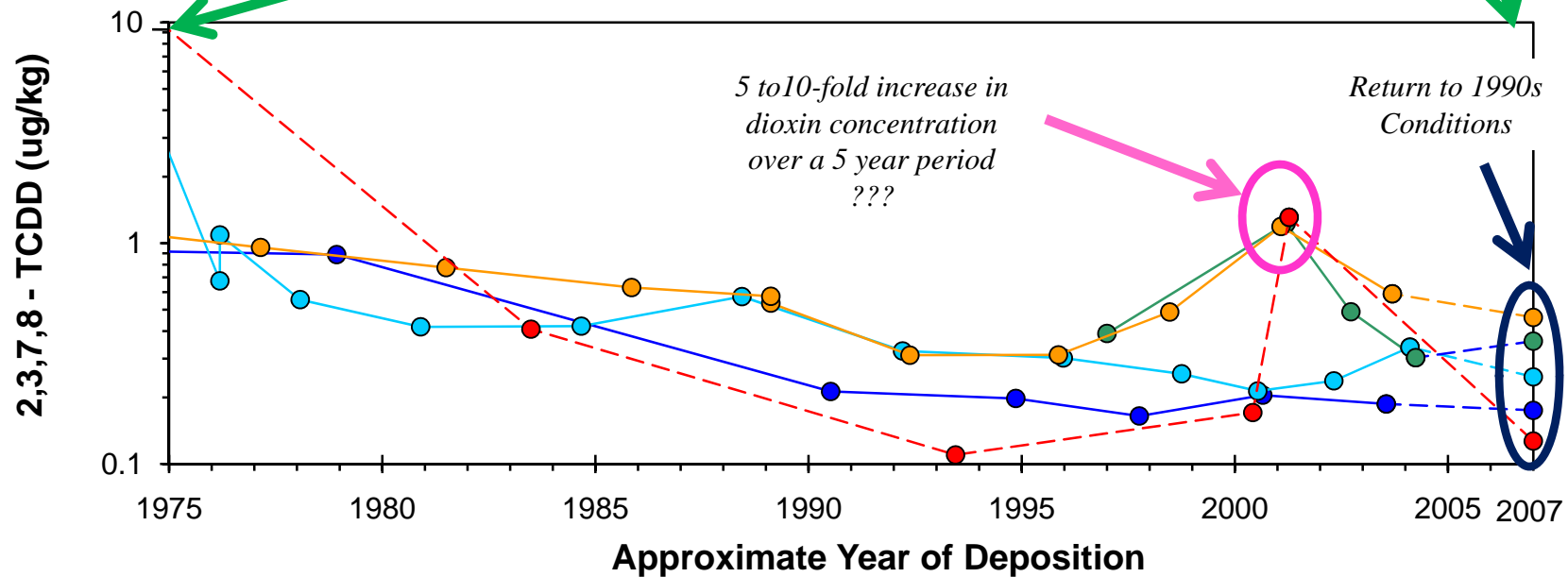
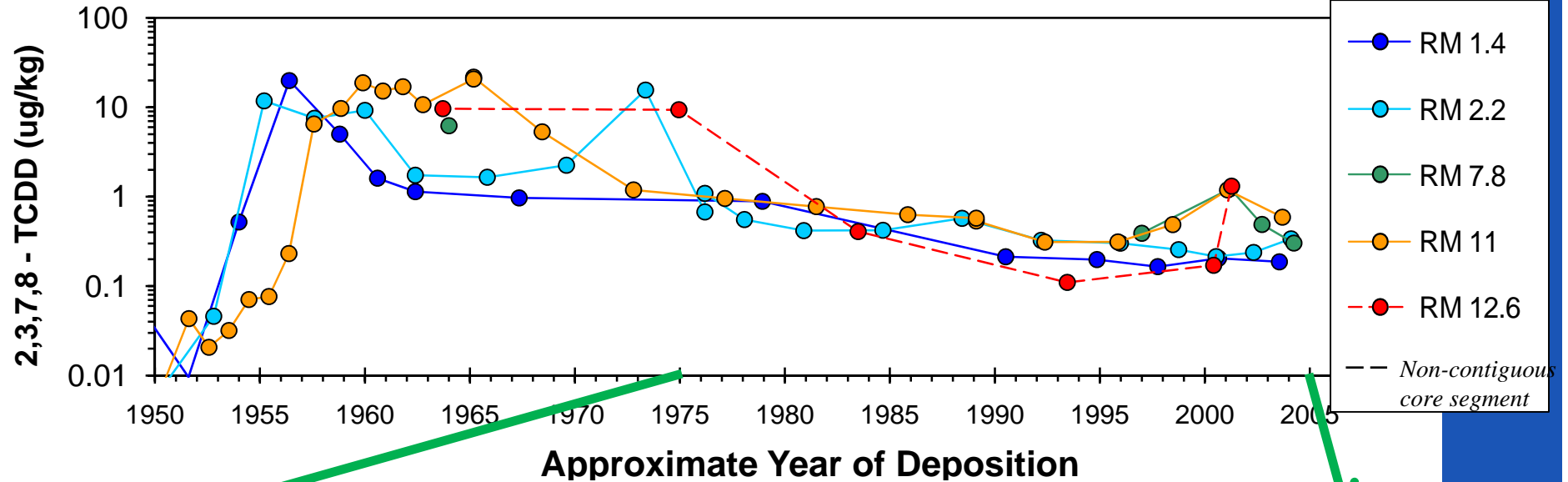


The Louis Berger Group, Inc.





The History of 2,3,7,8-TCDD as Recorded in the Sediments

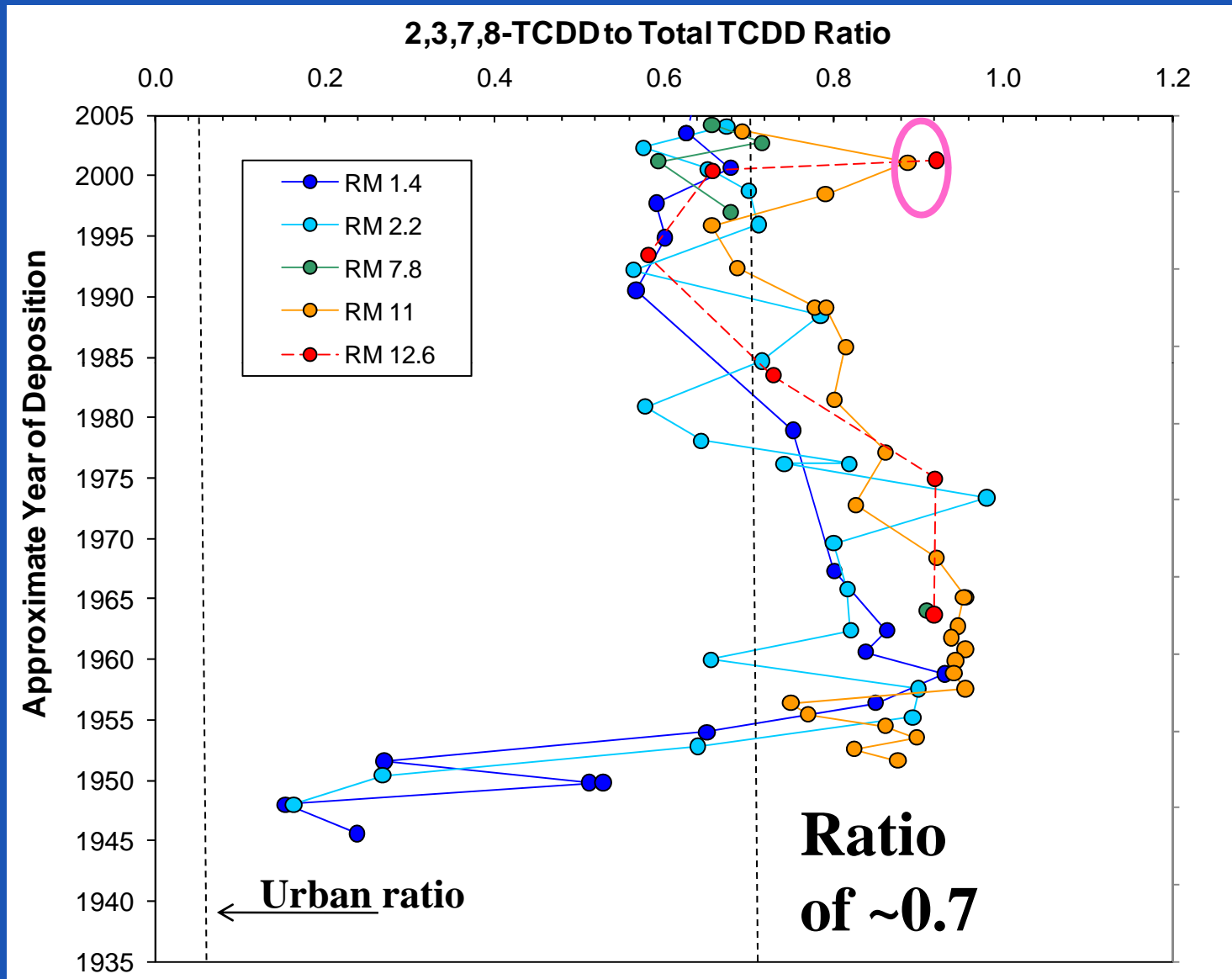


Possible Causes

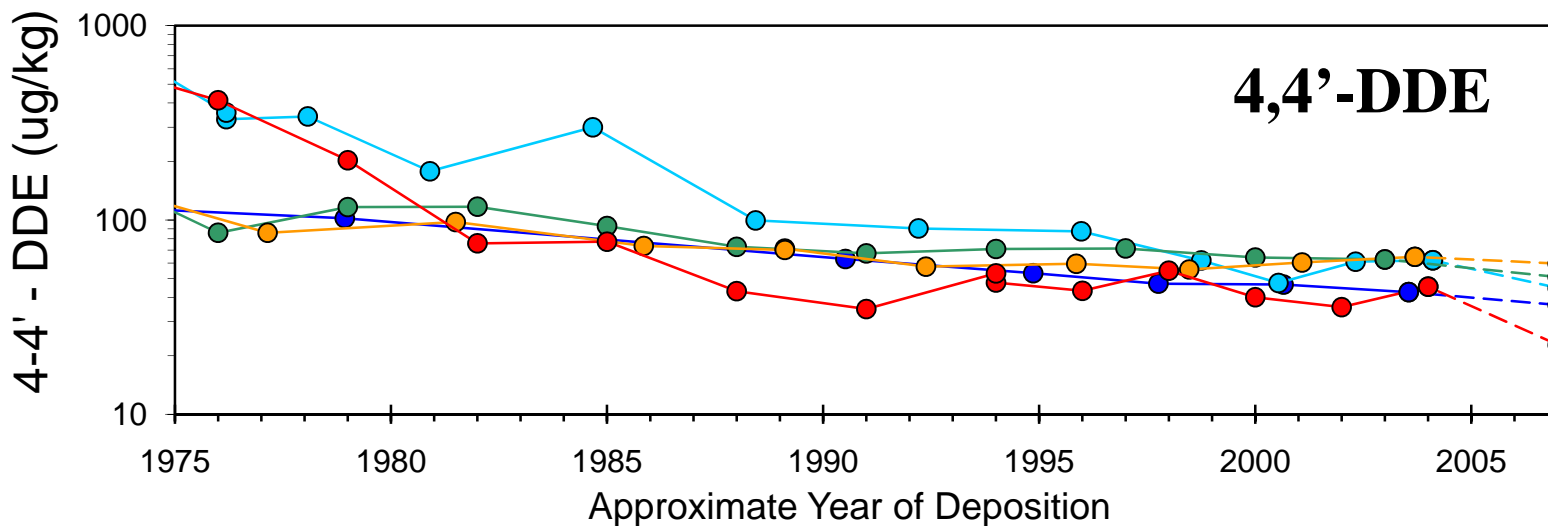
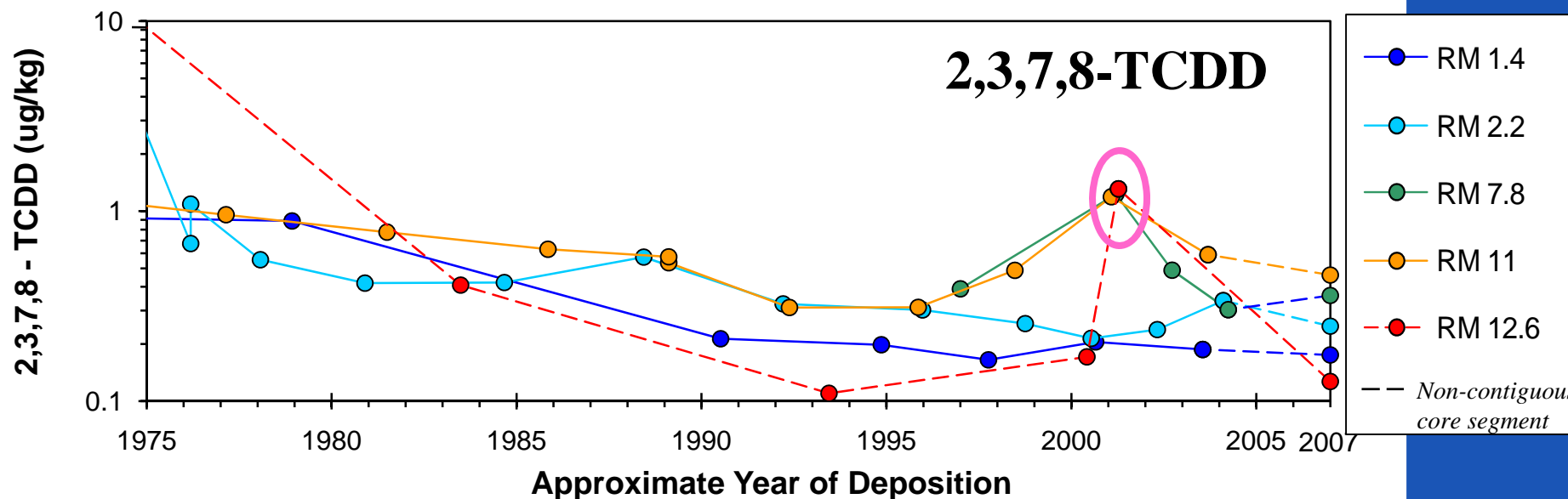
- Sampling Artifact
 - (but result observed in three separate cores at approximately the same time horizon)
- Resuspension
 - (but impact limited to upper 3 cores)
 - (and other contaminants do not show impact)
- External Source
 - (but no known dischargers)



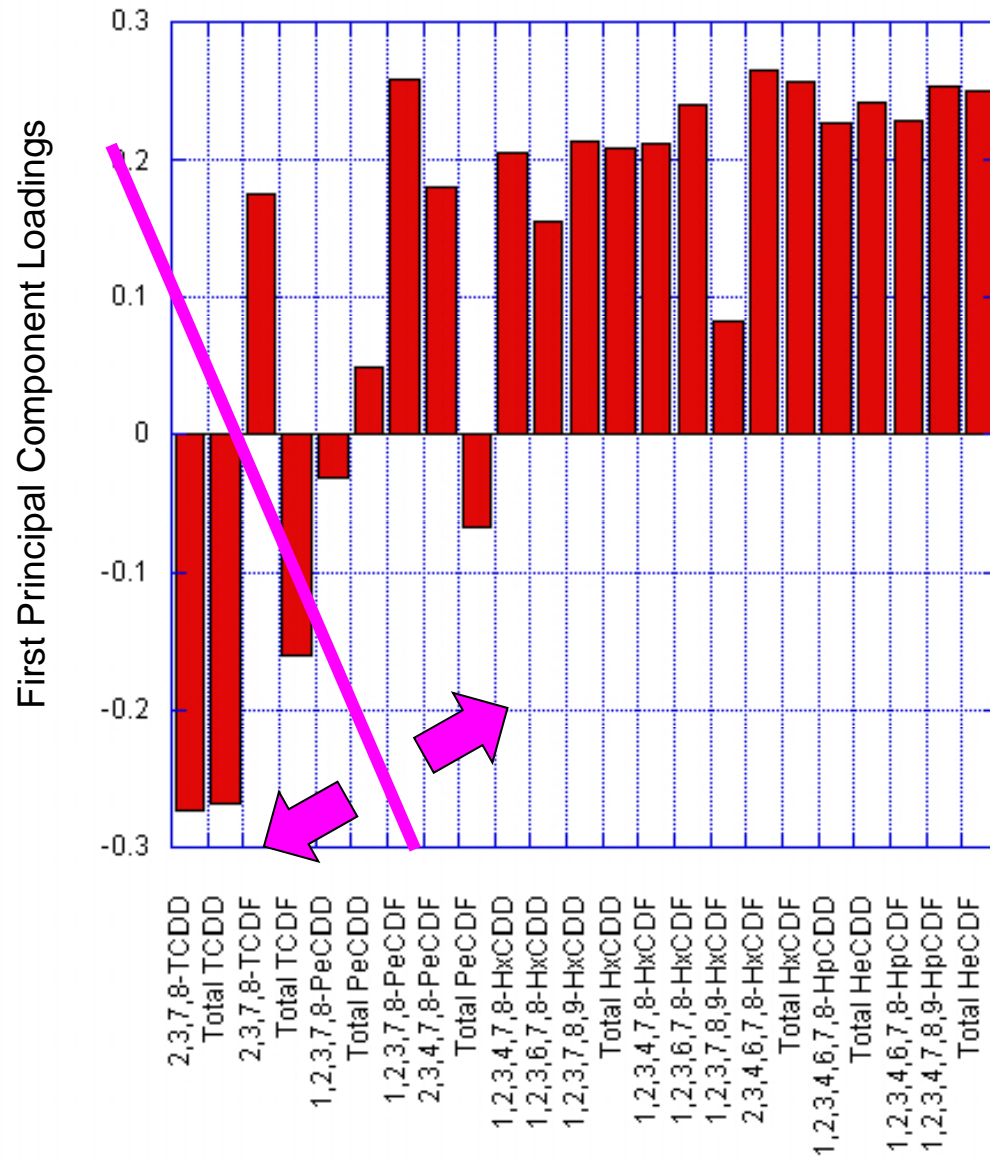
Dioxin ratios suggest change at time of event

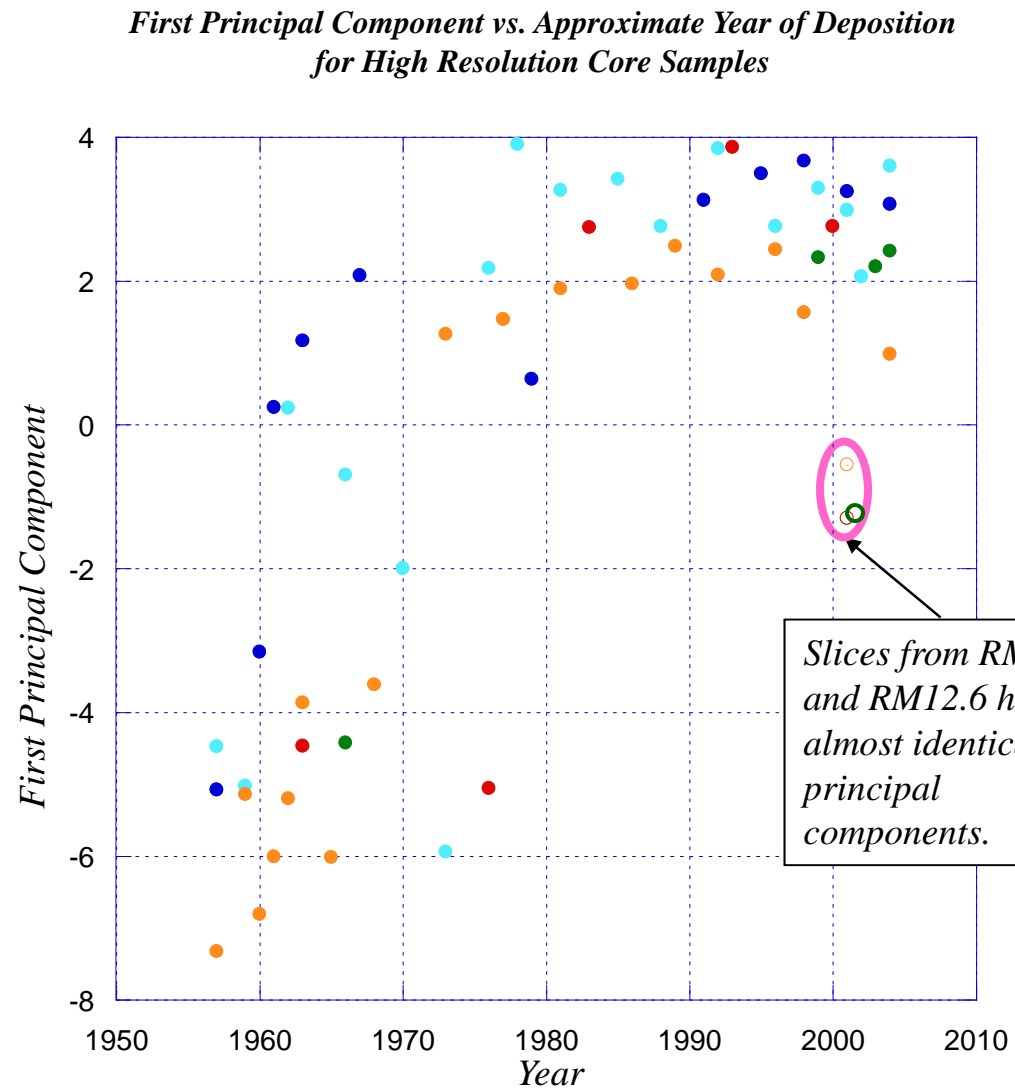


No Similar Event is seen in other Contaminants



First Principal Component Loadings for Dioxins and Furans in Dated Sediment Core Samples





Legend

High Resolution Core Slices

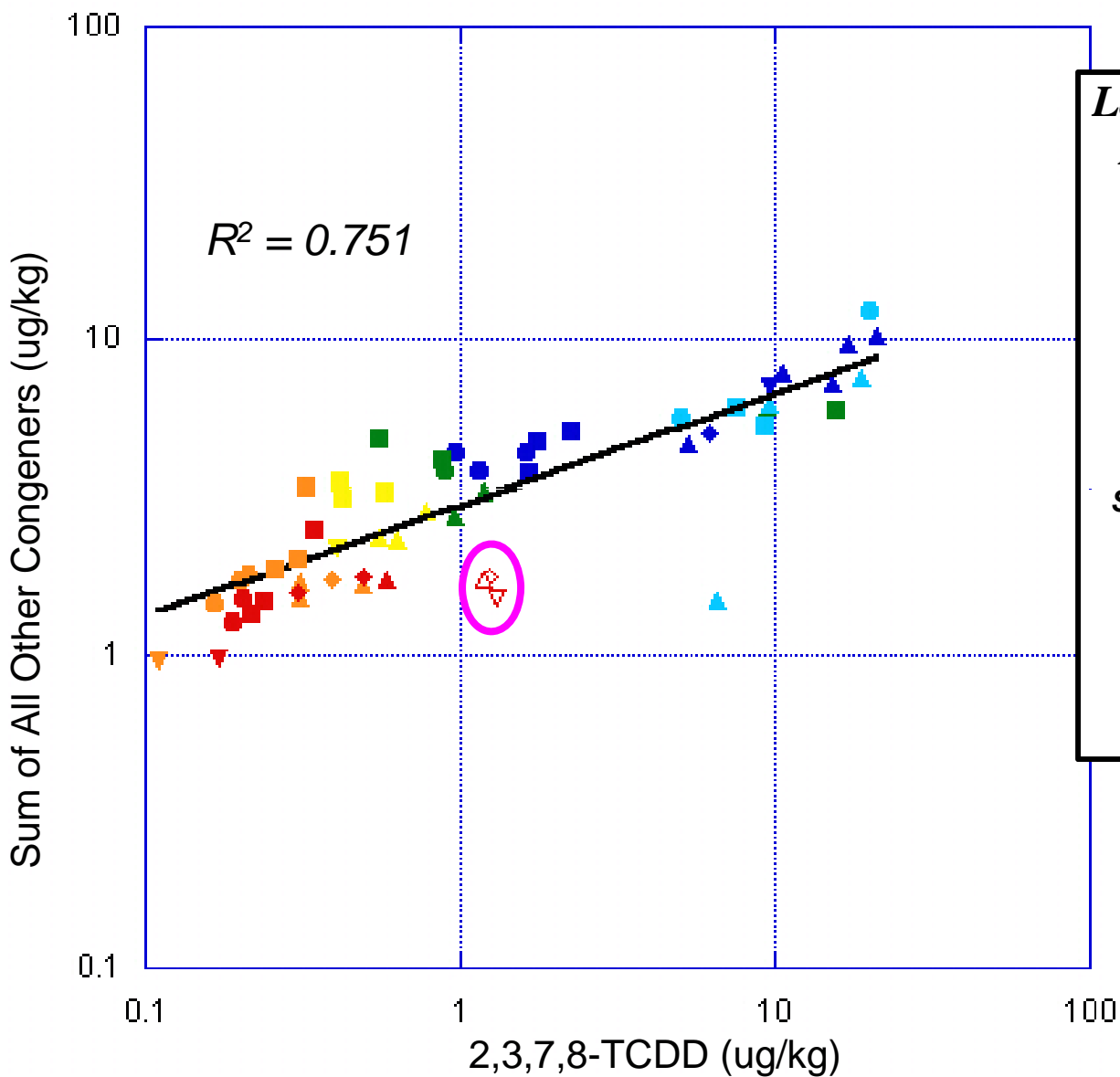
- River Mile 1.4
- River Mile 2.2
- River Mile 7.8
- River Mile 11
- River Mile 12.6

Slices with High Dioxin Concentration

- RM 7.8, 2001 slice
- RM 11, 2001 slice
- RM 12.6, 2001 slice

First Principal Component Shows Circa 2000 Event Pattern to be Distinct from Post-1970 Sediment Patterns





Legend

Dated Sediment Core Slices

- River Mile 1.4
- River Mile 2.2
- ◆ River Mile 7.8
- ▲ River Mile 11
- ▼ River Mile 12.6
- 1950s
- 1960s
- 1970s
- 1980s
- 1990s
- 2000s

Slices with High Dioxin Concentration

- ◇ RM 7.8, 2001 slice
- △ RM 11, 2001 slice
- ▽ RM 12.6, 2001 slice

— Regression Line

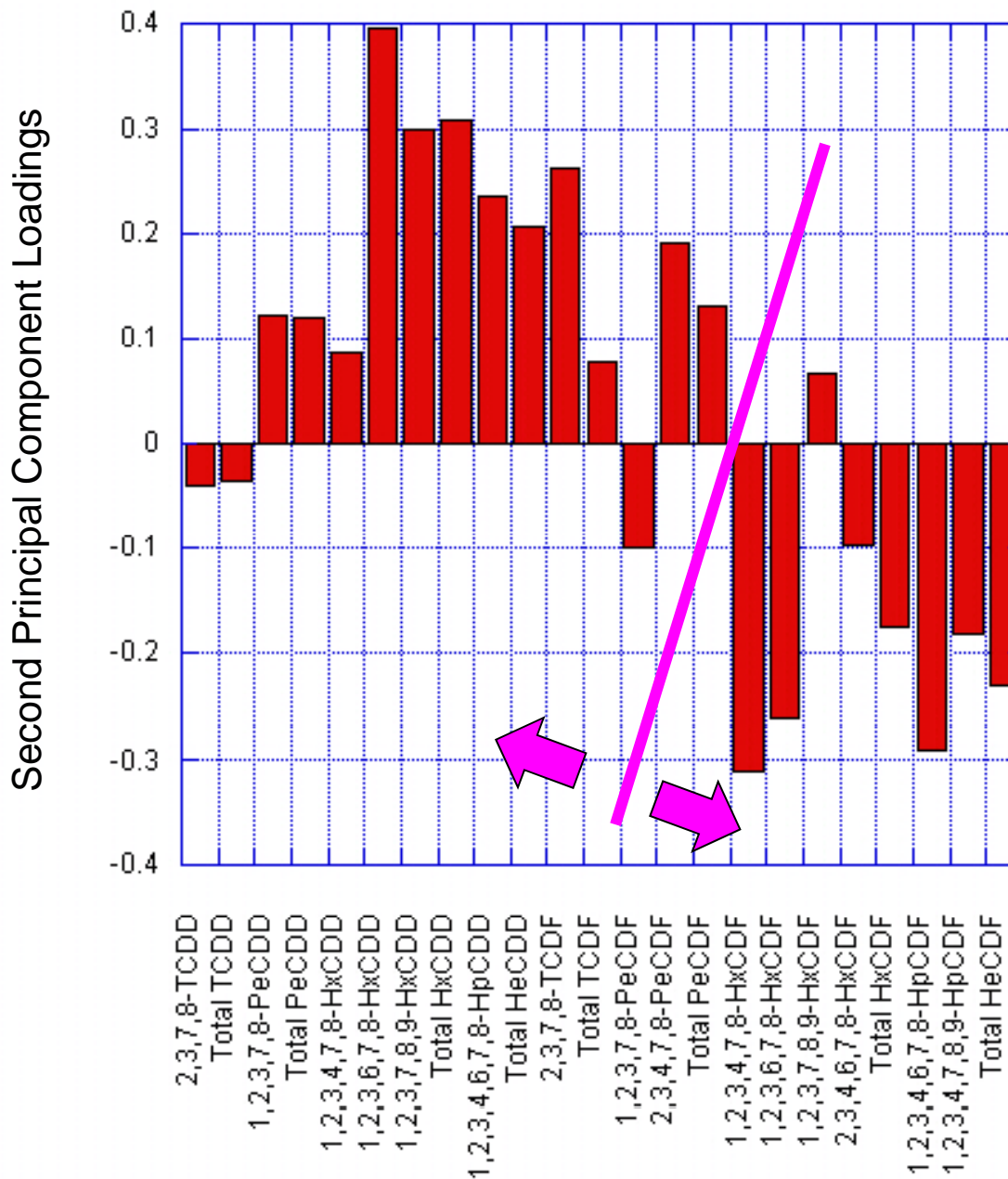
Dioxin Ratio Shows Uniqueness of Circa 2000 Event

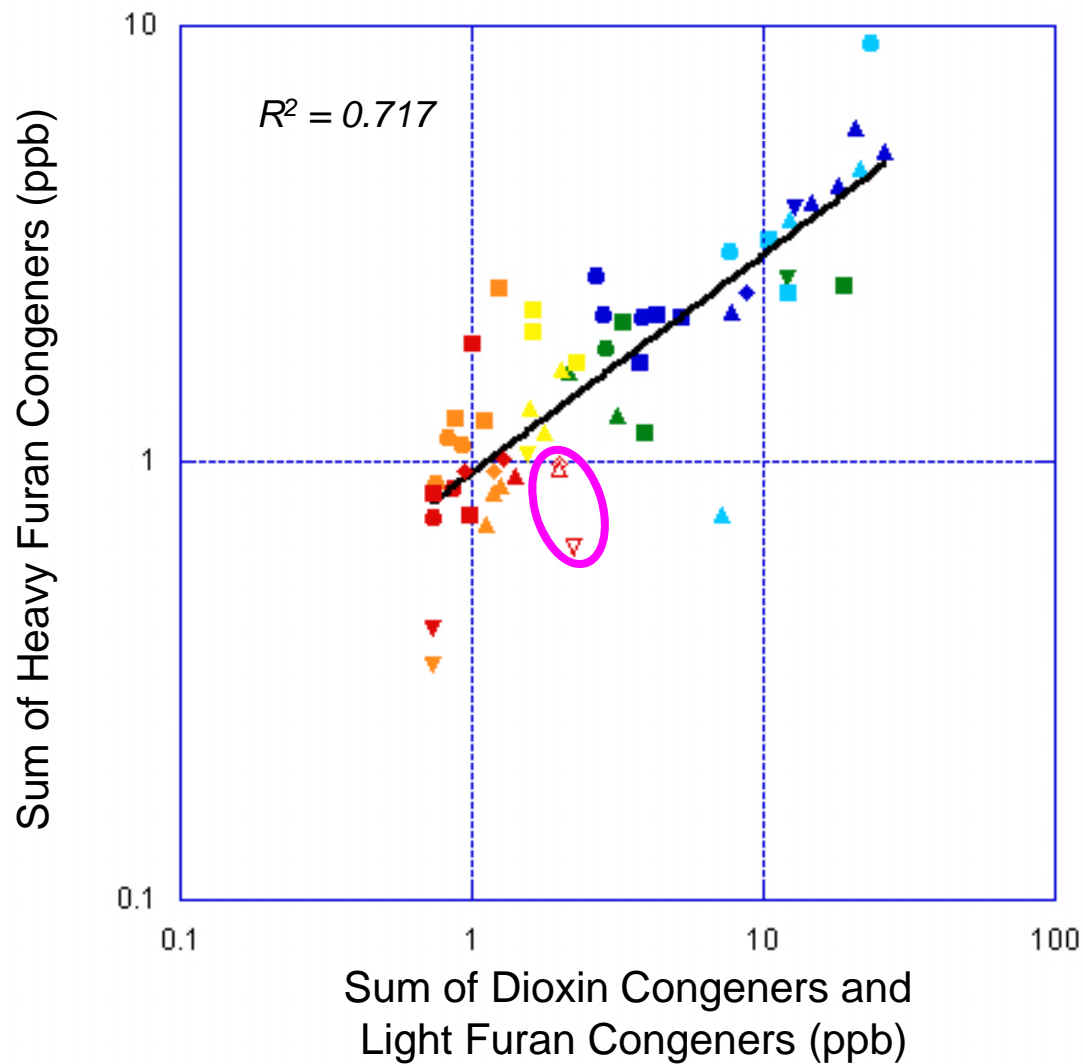


The Louis Berger Group, Inc.



Second Principal Component Loadings for Dioxins and Furans in Dated Sediment Core Samples





Legend

Dated Sediment Core Slices

- River Mile 1.4
- River Mile 2.2
- ◆ River Mile 7.8
- ▲ River Mile 11
- ▼ River Mile 12.6
- 1950s
- 1960s
- 1970s
- 1980s
- 1990s
- 2000s

Slices with High Dioxin Concentration

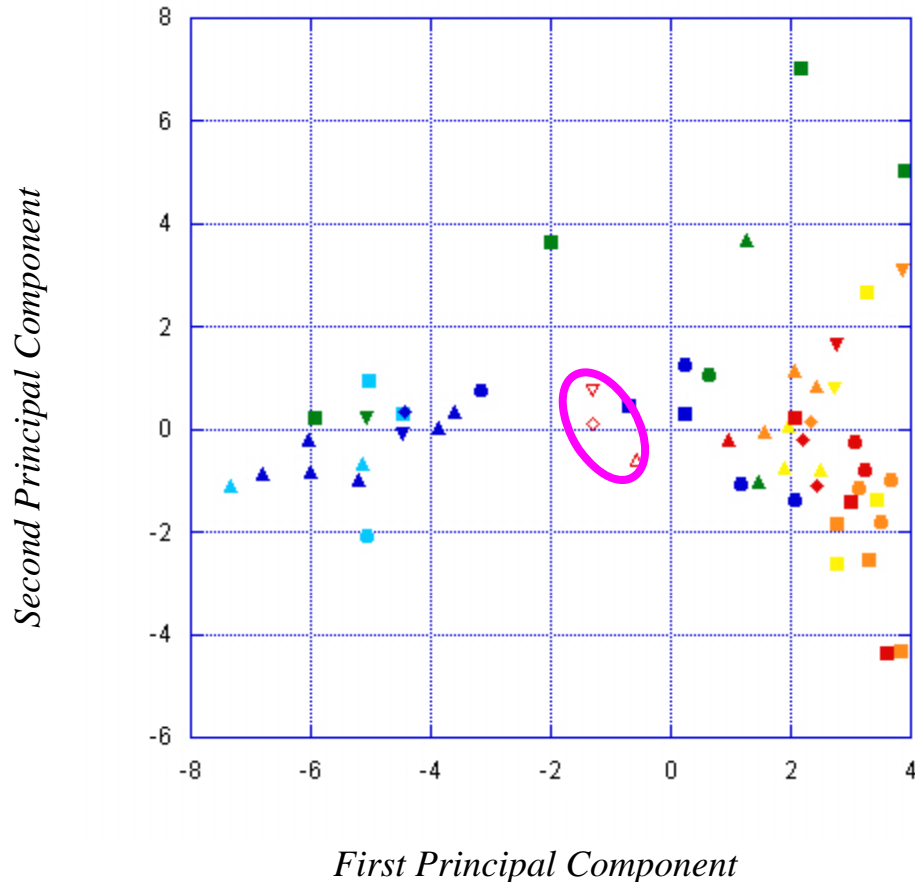
- ◇ RM 7.8, 2001 slice
- △ RM 11, 2001 slice
- ▽ RM 12.6, 2001 slice

— Regression Line

Factors Suggested by Second Principal Component Do Not Uniquely Identify the Circa 2000 Event



First and Second Principal Components for High Resolution Core Samples



Legend

High Resolution Core Slices

- River Mile 1.4
- River Mile 2.2
- ◆ River Mile 7.8
- ▲ River Mile 11
- ▼ River Mile 12.6
- 1950s
- 1960s
- 1970s
- 1980s
- 1990s
- 2000s

Slices with High Dioxin Concentration

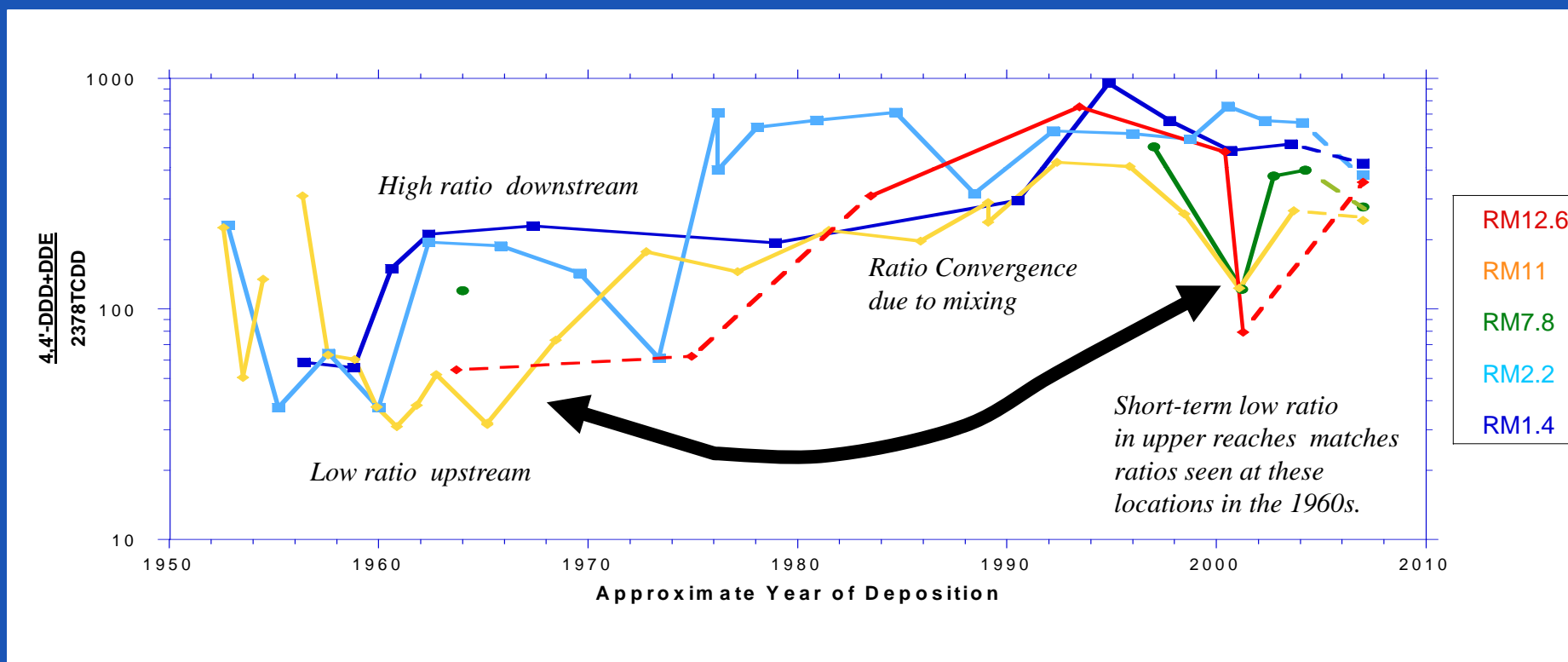
- ◇ RM 7.8, 2001 slice
- △ RM 11, 2001 slice
- ▽ RM 12.6, 2001 slice

Combined Principal Components Show Circa 2000 Event to Lie Between Patterns Observed Upriver and Downriver in the 1960s



Dioxin to DDT Ratio Shows Variation over Time Between Upper and Lower Reaches.

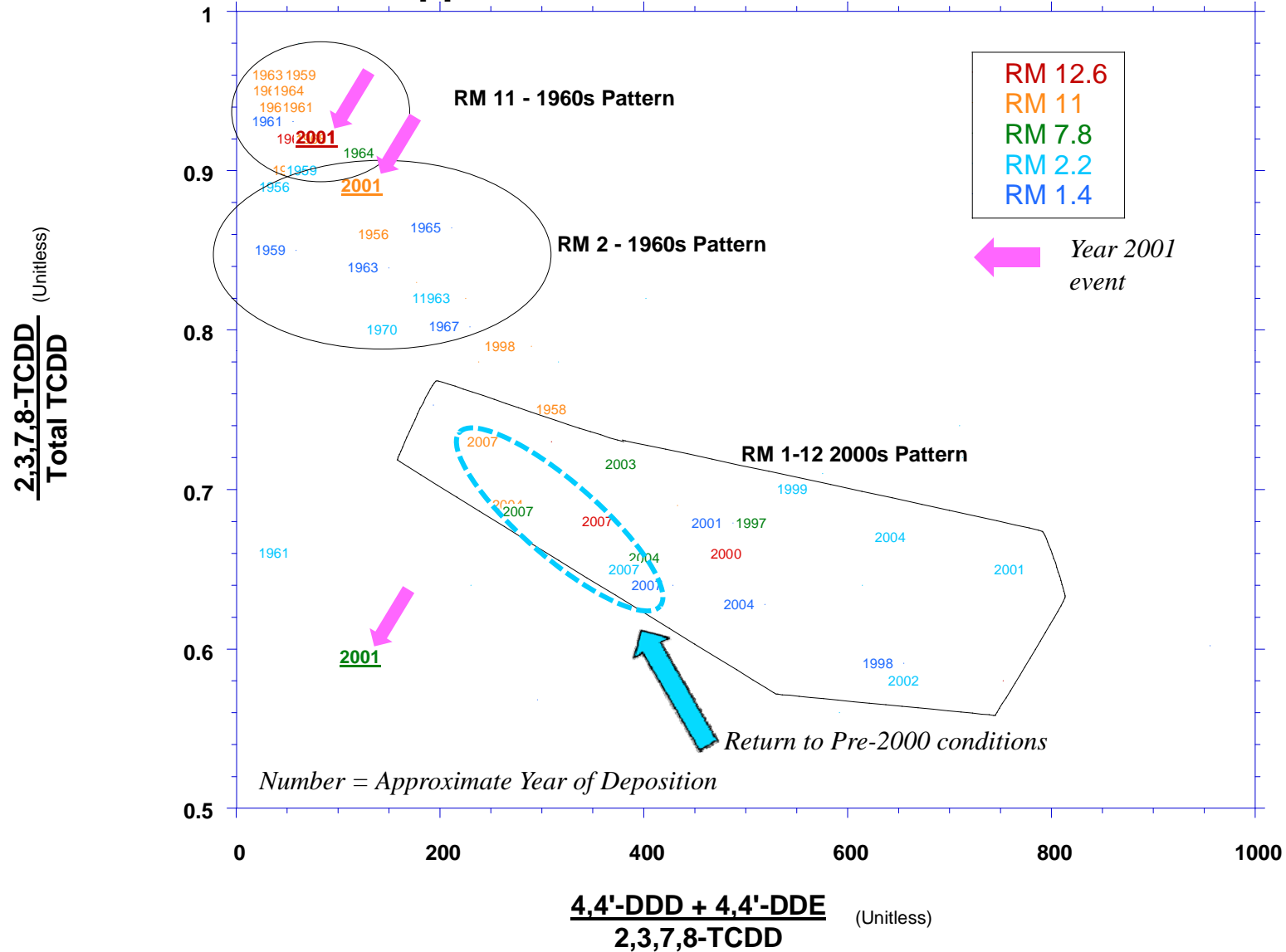
Recent event is similar to 1960s conditions seen in the same area



(4,4'-DDD+4,4'-DDE)/2,3,7,8-TCDD Ratio
Dated Sediment Cores



Combining Ratios Identifies Unique Patterns of Current and Historical Contamination in the Upper and Lower Reaches of the Lower Passaic River



Conclusions

- Dated sediment cores provide a detailed temporal record of relative contaminant loads to the estuary.
- Event circa 2000 is documented across three cores and can be identified as an external dioxin load, with a unique dioxin pattern.
 - Lack of response in other contaminants rules out sediment resuspension.
- Pattern observed in 2000 similar to one of 2 patterns observed in 1960s.
- Combined use of Dioxin and DDT ratios uniquely identifies upper reach contamination relative to lower reach in 1960s.
- Ratios in upper and lower reaches converge over time as sediments are mixed by tidal circulation.
- Temporary release circa 2000 has ended and estuary conditions have returned to 1990s levels.



Questions???

- www.ourpassaic.org
- www.ournewarkbay.org



USEPA



USFWS



USACE



NJDEP



NJDOT



NOAA

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Exhibit K

GIVAUDAN FRAGRANCES CORPORATION
SUPPLEMENTAL RESPONSE TO 104(e) REQUEST FOR INFORMATION

United States Environmental Protection Agency (“USEPA”) has issued several requests for information about the former Givaudan facility in Clifton, New Jersey (the “Givaudan Site” or “Clifton Site”) with regard to the Lower Passaic River Study Area. USEPA’s requests have sought information regarding the following general categories: the history of the former Givaudan Site, plant production information, liquid and solid waste management, permits, material and waste testing data, plant demolition, and remedial actions, with a specific focus on the compound identified as 2,4,5 Trichlorophenol (“TCP”) and a product manufactured using TCP known as Hexachlorophene (a/k/a “G-11”). Givaudan’s prior responses to USEPA were based upon the information and data then available. Givaudan hereby supplements its prior 104(e) responses dated October 26, 1983, July 12, 2004, June 27, 2006, October 14, 2009 and December 3, 2009 for the Clifton Site.

Since 2009, Givaudan has met with USEPA on several occasions and has exchanged correspondence concerning the former Clifton facility and its alleged nexus to contamination in the Lower Passaic River. USEPA has expressed interest in obtaining additional information related to the Clifton Site conditions over time, including the former plant process sewer system. Specifically, USEPA has sought confirmation of when the former Givaudan Site connected to the City of Clifton sewer system, which discharges to the Passaic Valley Sewer Commission (“PVSC”) main trunk line that runs parallel to the Lower Passaic River along Route 21.

In an effort to respond to USEPA’s requests, Givaudan has expended considerable efforts and resources to research and compile additional information concerning the former Givaudan Site. Those efforts included obtaining and evaluating additional historical aerial photographs, available historical sewer records, and other materials from public and other sources. The additional information gathered through Givaudan’s efforts are provided in this supplemental 104(e) response and include:

- (1) a collection of historical aerial photos and topographic information (with interpretative notes);
- (2) additional sewer maps obtained from the City of Clifton and the New Jersey Department of Transportation (“NJDOT”), detailing when the sewer lines were installed in the area surrounding the former Givaudan site; and
- (3) additional documents found in public and archived files related to other topics included in USEPA’s requests for information.

Givaudan supplements its prior 104(e) responses with the enclosed binders, Volume 1 (Tab No.’s 1-55) and Volume 2 (Sewer Chronology, Exhibits A-T) (also provided via disk). The following

is a summary of the former Clifton facility information and documentation provided in this supplemental 104(e) response.

HISTORICAL AERIAL PHOTOGRAPHY AND TOPOGRAPHIC REVIEW CONFIRMS NO SURFACE WATER DRAINAGE PATHWAY TO THE PASSAIC RIVER FROM THE FORMER GIVAUDAN SITE

Through private vendors and archived public sources, Givaudan obtained dozens of additional aerial photographs of the Clifton Site spanning the years 1931 to 2012 (*see* Tab 1 for all Aerial Photograph and Topographic Exhibits). An emphasis was placed on securing aerials that covered the early years of plant operations up through the 1970s. This information supplements the aerial photography submitted as part of the 2009 104(e) supplemental response. The source and scale of these photographs are noted within the Aerial Photograph Exhibits in the binder included with this supplemental response.

Several of these photographs include aerial stereo pairs, which allow a trained aerial photo interpreter to view greater detail to support the interpretations presented with the aerial photographs. There are also oblique aerial photographs that provide an additional viewing angle of the former Clifton site and the surrounding area. Based on the search conducted, the 1931 photograph is the earliest aerial available for the former Clifton Site.

In addition, the attachments include an 1870-1887 historical topography map (Aerial Photograph Exhibit 3), two aerial photographs that were photo digitized to obtain topographic information on a portion of the former Clifton site (Aerial Photograph Exhibits 19 and 24), and a 1979 aerial photograph that has been overlaid with the 1982 plant topography (Aerial Photograph Exhibit 32). Note that copies of Sanborn Maps for the years 1935, 1951, 1952, 1965, 1970 and 1984 are also provided for reference, which show the plant expansion over time (*see* Tab No. 36).

Aerial Photograph Exhibit 1 shows the plant at full build-out circa 1979, as well as the surrounding area. Plant building numbers and key roads around the plant are labeled for reference on this exhibit.

Aerial Photograph Exhibit 2 shows the dates on which Givaudan purchased the various lots that make up the entirety of the former Clifton Site, which includes buildings on both the north and south sides of Delawanna Avenue (the main east/west trending road that divides the plant property). However, the remainder of the aerial photography review focuses on the south side of Delawanna Avenue where production and waste management activities took place. As part of the aerial photo interpretation, the then current property line of the former Clifton Site at the time of the photograph is shown, illustrating actual ownership and conditions onsite and in the area surrounding the property at the time of the photograph. The property line transferred onto each

photograph is based on the property title history and the City of Clifton title maps (*see* Tab No. 37). The property title history identifies the parcels that Givaudan purchased over time that encompass the former Clifton Site, beginning in 1924 and ending with the last purchase in 1978 for the south side and 1982 for the north side (*id.*).

Aerial Photograph Exhibit 3 is the 1870-1887 historical topography map. The red outline shows the boundary of the former Clifton Site and the yellow line shows the area where topography was developed using digital plotting on the 1954 photograph (Aerial Photograph Exhibit 18) and the 1961 photograph (Aerial Photograph Exhibit 23). Aerial Photograph Exhibit 32 shows the 1982 plant topography overlain on a 1979 aerial photo. The 1870-1887 topography shows that prior to any development, Delawanna Avenue and the rail line along the western boundary of the former Clifton Site were present and their location and orientation have not changed since that time. Both Delawanna Avenue and the rail line are at a higher elevation compared to the former Clifton Site. The configuration of River Road was the same from that time until it changed on the eastern side of the property when Route 3 and Route 21 were constructed between 1959 and 1961. Within the former Clifton Site boundary (inside the red line), the lowest elevation is to the south along the rail line at River Road, which low point remained the same throughout the development of the property. The area to the east/northeast is a natural topographic high that was partially excavated over time to allow for plant expansion, and the eastern bluff that remains there is the current location of a residential community, which is elevated approximately 20 feet above the former Clifton Site.

The area east of the former Clifton Site (within the yellow line) is topographically higher than the Givaudan Site and that elevation has remained the same over time. Using digitized topography, Aerial Photograph Exhibit 19 (1954 photo) shows that the River Road/rail line location is the low spot, with higher elevations to the east along River Road.

One key observation on all of the historical aerial photographs is that there is no visible channelized flow or surface drainage feature visible on or off of the plant property. The alleged existence of a possible surface water pathway that could have conveyed storm water flow from the former Clifton property directly to the Passaic River is *not* supported by the historical aerial photo review, or the digital topography analysis completed on the historical photos. There is no evidence of a defined drainage swale either on or off the property to the Passaic River in any of the historical aerial photos, and topographic relief is higher around the property and significantly elevated to the east, with the lowest elevation consistently identified at River Road and the rail line to the south.

There was no overland path for runoff to the east from the former Givaudan Site to the Passaic River. Aerial Photograph Exhibit 24 (1961 photo) provides digitized topography after the construction of Route 3/Route 21, which shows that fill material was used for construction of

that roadways' current on/off ramps. The River Road/railroad area on the southern end of the plant remains the low point at the Clifton Site. Aerial Photograph Exhibit 32 shows the 1982 topography overlaid on the 1979 aerial photo, which confirms that the River Road/railroad area remained the low point at the Clifton Site. With higher elevations to the east, and the unchanged presence of Delawanna Ave. along with the railroad on the north and west sides of the plant, the only location for surface water runoff from the former Clifton Site would have been the juncture of River Road and the rail-road at the southern end of the plant. Surface water runoff (if any) only occurred during extreme precipitation events as storm water was collected in the onsite pond or percolated into the unpaved areas at the property. Any surface runoff would have been collected in the City of Clifton storm sewer system on River Road near the railroad underpass (*see* Tab No. 50).

FROM THE LATE 1940s THROUGH EARLY 1950s, THE FORMER CLIFTON SITE HANDLED PROCESS WASTEWATER ONSITE

The remaining aerial photographs document site development over time. Key observations include the presence of three enclosed water features on the Clifton Site. Two of these features are first visible on the 1947 aerial photograph (Aerial Photograph Exhibit 8): a thin elongated feature believed to be the Spent Acid Pit ("SAP") referred to in earlier submittals, and the area known as the storm water pond, which remains visible until the plant is closed. The third enclosed surface water feature is clearly visible on the 1949 Oblique (Aerial Photograph Exhibit 12), adjacent to the SAP and north of the storm water pond. That feature appears to be an impoundment that handled process waste water. By 1953 (Aerial Photograph Exhibit 16), the only surface water feature visible is the storm water pond. The SAP no longer contains standing liquid, and there is no remaining evidence of the third water feature. The occurrence and eventual disappearance of the SAP and third surface water feature support prior information submitted to USEPA that the plant used the SAP and third surface water feature to handle plant process waste water and possibly non-contact cooling water, while the storm water pond was consistently used for collecting rainwater until plant closure (*see* Tab No. 27, well driller logs confirming the use of onsite pits for liquid waste disposal in 1949). The documentation submitted to USEPA indicated that, prior to 1947, waste was discharged onsite into cesspools and pits. Some liquid waste (solvents) was also used as supplemental fuel in the plant boiler. The boiler house is visible on the 1947 photograph, and also may be present in the 1940 photograph.

In addition to the interpretation of aerial photography and the sewer chronology, Givaudan located a separate document from a well driller who did work at the plant, which confirms that plant waste was disposed into pits (e.g. the SAP and third water feature) (*see* Tab No. 27). Further, a 1951 Givaudan memo discusses the recovery of G-11 from the former waste pits (*see* Tab No. 48), which also supports the facility's practice of discharging and handling its process

waste water from G-11 on site before the plant connected to the city sewer line on River Road by 1951-52.

THE GIVAUDAN SITE WAS CONNECTED TO THE CLIFTON CITY SEWER SYSTEM AS EARLY AS 1926 AND NO LATER THAN 1951/1952

As documented in prior submittals, the earliest engineering drawing referencing a plant sewer system for the Clifton Site is dated 1946. Until that time, the documentation indicates that process waste generated by the plant was disposed of in cesspools and pits, with some spent solvents used as supplemental fuel in the plant boiler. The installation of a dedicated plant sewer system in 1946 and the appearance of the SAP in 1947, followed by the development of a third surface water impoundment by 1949, supports the prior information that plant process waste water was initially all handled onsite. Also, an onsite storm water management system was evident by 1947 and is supported by the appearance of the storm water pond and a storm water conveyance system (1946 engineering drawing), which remained in use until the plant closed.

Regarding the Clifton Site's connection to the City of Clifton sewer system, Givaudan provides the following information along with some background documentation (*see also* attached Sewer Chronology with Exhibits A-T). The City of Clifton (formerly Acquackanonk Township) began the process of planning for a dedicated sewer system in the early 1900s. By 1911, a contract was in place with the Passaic Valley Sewerage Commission ("PVSC") to construct a sewer system that would connect to the main trunk line that PVSC planned to install parallel to the Passaic River. There is documentation in historical City of Clifton records that parts of the City had sewer access before the 1920s (*see* Tab No. 25). By the early 1920s, the City had passed several resolutions for the design and installation of a dedicated sewer system for the majority of Clifton. With the completion of the PVSC trunk sewer by 1921, the City began adding connections for the discharge of its sewage to the PVSC.

According to City of Clifton meeting notes, the majority of the City sewer system was in place and operating by 1927 (*see* Exhibit C to Sewer Chronology). Copies of sewer maps for the Delawanna area of Clifton, which include Delawanna Avenue, River Road and Oak Street in the vicinity of the former Givaudan Site, indicate that these sewers were installed by 1927. In 1930, the City passed Ordinance #989, which prohibited the discharge of sewage into the Passaic River or its Tributaries (*see* Exhibit H to Sewer Chronology). Subsequently, a series of ordinances were passed requiring hook ups by all businesses in Clifton to the City sewer when it was installed and establishing rates for sewer usage. A 1945 City Planning map shows that essentially the entirety of the City had both sanitary and storm sewers by that date (*see* Tab No. 51). There is no evidence to support any allegation that the City of Clifton sewer line could have discharged to the Yantacaw Pond, the Third River, or the Passaic River. The Clifton sewer

system along Delawanna Avenue, River Road, and Oak Road was connected to the PVSC system by 1926-27.

In 1951-1952, an additional sewer line was installed on Delawanna Avenue to collect domestic sewage from the residential area constructed on the bluff to the east of the former Clifton Site. One of the drawings generated as part of this construction indicates that the Givaudan Site was already serviced by a City sewer (*see* Exhibit P to Sewer Chronology), which sewer line is believed to have been in place since 1926/1927.

The connection of the plant process waste stream (Outfall 001) to the River Road sewer system appears to have been made no later than 1951-52, and perhaps several years earlier. The City of Clifton sewer maps indicate that this stretch of River Road had sewer lines installed in 1927 (*see* Attachment E to Sewer Chronology). However, Givaudan did not have access to this area until it purchased Parcel 3 in 1939 (*see* Aerial Photograph Exhibit 2). The latest a sewer connection would have been made is 1951-52, as it coincides with the absence of the large rectangular surface water impoundment and the appearance of Building 74, which is identified as part of the Clifton Site's early waste water pre-treatment system (*see* Aerial Photograph Exhibit 14). Also, the SAP is no longer visible by the 1953 aerial photo and Building 83 is present, which is designated as a Waste Neutralization System (*see* Aerial Photograph Exhibit 16). A review of New Jersey Department of Transportation ("NJDOT") engineering drawings for the Route 3/21 project show that the former plant had connected to the River Road sewer before 1955 (*see* Exhibit T to Sewer Chronology). Based on these documents, Givaudan believes that the Clifton Site may have connected to the River Road sewer line as early as 1946, but no later than 1951-52. This conclusion is supported by the aerial photo documentation, the local ordinances in place, and the NJDOT engineering drawings, as well as facility documents.

As discussed above, based on the aerial photo interpretation, there was no defined surface water runoff or pathway from the Clifton Site to the Passaic River, and that plant process waste effluent was contained on the property prior to connecting to the City sewer system. Further, based on the historical sewer documentation, it is evident that the former Givaudan Site was connected to the Delawanna Avenue sewer line as early as 1927 and to the River Road sewer line as early as 1946, but no later than 1951-52. Additional support for this conclusion is contained within Tab No. 35, which includes 1953 correspondence related to Givaudan's agreement to repair sewer lines on River Road, and a reference to 1946 correspondence related to maintenance responsibility.

ADDITIONAL DOCUMENTS RELATED TO THE GIVAUDAN SITE OPERATIONS

In the course of reviewing archived information and other public sources, Givaudan located additional documents that are responsive to USEPA's prior 104(e) requests. The enclosed Binder

(Volume 1) includes those documents and provides an index and summary of each document. The following is a brief overview discussion of these additional reference materials.

GIVAUDAN'S MANUFACTURING PROCESS AND STRICT QUALITY CONTROLS ON ITS TCP FEEDSTOCK RESULTED IN MINIMAL (IF ANY) TCDD CONTENT

Additional information from published sources indicates that it was highly unlikely that Givaudan's G-11 manufacturing process generated TCDD because it used acidic conditions and low temperatures in its process (*see* Tab No.'s 23 and 26). Documents prepared by Dow state that TCDD may be produced during use of TCP under alkaline conditions and in temperatures greater than 100 degrees centigrade, which is distinct from the acid process employed at Givaudan (*see* Tab No. 32). These documents provide an independent technical basis for the conclusion that the Givaudan G-11 manufacturing process would not have generated TCDD.

An independent investigation of TCP and TCDD was conducted by the National Institute for Occupational Safety and Health ("NIOSH"), led by Dr. Fingerhut. In 1983, Dr. Fingerhut studied and researched the potential threat to workers at plants that produced and/or handled TCP and related chemicals known to be associated with TCDD. The results of her work were published for each facility studied, including an evaluation of the former Givaudan Site. One of Dr. Fingerhut's key findings was that the Givaudan Site had the lowest range of TCDD in its TCP and G-11, whereas the highest concentration of TCDD was found in plants where Agent Orange was produced, such as the Diamond Alkali site in Newark, New Jersey (*see* Tab No.'s 23, 46, and 47).

Documents provided to USEPA in 1983 summarize available information on Givaudan's 1948/1949-era pilot production of TCP and the management of waste generated from that process. Those documents indicate that the TCP produced by Givaudan, and used to manufacture G-11, was purified and therefore had lower TCDD content. Based on information gathered by Givaudan in 1983, the TCP waste products were drummed for disposal and picked up by waste haulers (*see* Tab No.'s 2 and 3). A 1967 memo states that Givaudan provided its TCP production and purification process to Hooker Chemical in return for Hooker agreeing to be the primary supplier of TCP to Givaudan (*see* Tab No. 52). This agreement gave Givaudan confidence that the Clifton Site would be supplied with high quality TCP, with low TCDD content, for use in producing G-11. Historical testing of Hooker Chemical TCP process waste streams demonstrated that the TCDD generated from the production of TCP was captured in the still residue and crude charged to the still material, which purified the TCP to reduce impurities, including TCDD content (*see* Tab No.'s 10 and 11).

In August 1976, USEPA visited the former Givaudan Site and obtained three samples from the TCP material on hand for testing (*see* Tab No. 28). USEPA's testing confirmed the TCP results

that Givaudan shared for the same material, which was in the low part per billion range for TCDD (*see* Tab No. 29).

In 1977, Givaudan met and contracted with Dow as a TCP supplier, requiring Dow to meet Givaudan's specifications for TCP to manufacture its G-11 (*see* Tab No.'s 30, 31, and 32). These documents support Givaudan's consistent approach to quality control for the TCP raw material used to make G-11, which required a purified form of TCP with low impurities. In 1978, Dow TCP drums were tested and confirmed TCDD concentrations at less than 0.01ppm (*see* Tab No. 33).

A 1983 Givaudan memo states that the production of up to 2,200 pounds of G-11 was lost during the State-imposed temporary shutdown of the G-11 operation pending the results of the TCDD investigation (*see* Tab No. 40). At that time, Givaudan performed additional testing of TCP to confirm that each lot of TCP had low levels of TCDD so that it could be used when G-11 production was allowed to resume. Testing confirmed that the TCP lots contained less than 1 part per billion of TCDD. Documentation also confirms that buildings 58, 59, and 60 were cleaned and that all waste material from this work was stored in building 54 (*see* Tab No. 42). Also provided is additional documentation related to the duties of the G-11 operators, which details each step of the manufacturing process and documents how waste residues were reclaimed for reuse in the process, collected in containers for disposal, and acid waste and process water was sent to the sewer (*see* Tab No.'s 44 and 45). In addition, the G-11 process had a catch-all tank that caught solids and residues before discharging waste water to the sewer.

In its prior 104(e) responses, Givaudan provided results of TCP and G-11 testing available at that time. Routine testing of G-11 for TCDD was in place by at least 1978 (*see* Tab No. 39). Included with this submittal is additional testing data to show that Givaudan maintained tight quality control on specifications for both the TCP raw materials and G-11 product in their manufacturing process (*see* Tab No.'s 6, 7, 8, 9 and 10). Givaudan imposed similar quality control on its raw material suppliers when evaluating options for purchasing TCP (*see* Tab No. 17). Testing of G-11 was also completed in 1983 by the Food and Drug Administration (FDA) to document the quality of the material. After these tests, the FDA took no further action with respect to Givaudan's G-11 product (*see* Tab No. 43).

GIVAUDAN'S WASTE MANAGEMENT PRACTICES PREVENTED OFFSITE MIGRATION OF CONTAMINATION

In prior 104(e) responses, Givaudan provided summary tables of the quantity and disposal locations for waste generated during the remediation. Attached documentation demonstrates that any TCDD-impacted soil identified at the Clifton Site was excavated, placed in drums and stored inside secured areas protected from the weather pending decisions on final handling of this

material (*see* Tab No. 4). In addition, Givaudan has located copies of receipts and waste manifest forms obtained from archived files that provide supporting documentation concerning TCP and G-11 waste drums disposed between 1978 and 1983 (*see* Tab No.'s 13, 14 and 16). Separate documentation was found for the removal and disposal of PCB oils and G-11 filter cake in 1982 (*see* Tab No. 15). In prior 104(e) responses, Givaudan provided information that waste solvents were used as a fuel supplement for the plant boilers. Additional supporting documentation is provided herein (*see* Tab No.'s 20 and 21).

TCDD-IMPACTED SOIL REMEDIATION

In its prior submissions, Givaudan provided USEPA with the results of the 1980s TCDD soil investigation, including the reports and maps prepared as part of the remedial work completed under the Administrative Consent Order (ACO) that Givaudan entered into with the State of New Jersey. Included with this submittal are additional communications with the NJDEP related to the investigation and remediation work, which document various approvals and agreements between the parties (*see* Tab No.'s 18, 19 and 22). As noted in the March 5, 1987 Administrative Consent Order for TCDD, results of the investigation conducted by Givaudan under the supervision of NJDEP, in conjunction with investigations by USEPA and the Department of Health, confirmed that there was “no evidence that TCDD contamination has migrated off the Site.” (*See* Tab No. 24, at ¶32).

USEPA PASSAIC RIVER SOURCE INVESTIGATION

In August 2015, USEPA conducted soil sampling within the containment cell at the Clifton Site. Lockheed Martin (LM) and Scientific Engineering, Response and Analytical Services (SERAS) conducted this work and AMO Environmental Decisions was retained as the Licensed Site Remediation Professional (“LSRP”). The “Waste Cell Repair Report” provided by USEPA’s LSRP noted that the asphalt cap was thicker than expected. In addition, field change forms prepared by SERAS noted that the material to be sampled was at a much greater depth than expected. Ultimately, USEPA’s sampling confirmed that concentrations of TCDD within the cell at the Clifton Site were all below 10 ppb (*see* Tab No. 53). The materials that USEPA sampled in the cell are the soils around the plant that were sampled and remediated under NJDEP oversight, which resulted in the issuance of an approved No Further Action letter in 2002. USEPA’s investigation confirmed that only low levels of TCDD were identified and properly remediated at the Clifton Site.

CONCLUSION

Givaudan has provided documentation that the G-11 process wastewater was handled onsite at the plant until 1951-52 (at the latest), before it was discharged to PVSC via the Clifton City

sewer system. After Givaudan acquired land that abutted River Road as part of the plant expansion in 1939, the facility connected to the River Road sewer, which was required by local ordinance. The facility's sewer connection at Delawanna Avenue could have been made as early as 1927, as the City sewer maps indicate that the Delawanna Avenue line was installed in 1927, and later sewer maps show that a connection at the location of the former Givaudan plant was in place.

A review of City of Clifton ordinances, meeting notes, aerial photography, topography, and sewer maps confirms that Givaudan did not discharge its wastewater to Yantacaw Pond, the Third River, or the Passaic River. Further, there was no overland drainage ditch or pathway from the Clifton Site to the Passaic River. Multiple lines of evidence support the fact that the lowest point of elevation on the Givaudan Site was to the south at River Road adjacent to the rail line, and that elevations were higher to the north along Delawanna Avenue and to the east where the residential neighborhood is located, such that no surface water runoff could flow off the plant property other than the low point at River Road and the rail line.

Finally, historical documents and sampling data confirms that the TCDD level in the TCP used to make G-11, and the G-11 product itself, was carefully monitored to follow strict quality control guidelines. This conclusion is also supported by third parties that independently reviewed Givaudan's G-11 manufacturing process. These conclusions are confirmed by the historical documents and recent USEPA sampling data from its investigation of the Clifton cell in 2015.

CERTIFICATION OF SUPPLEMENTAL RESPONSE
TO REQUEST FOR INFORMATION

State of New Jersey

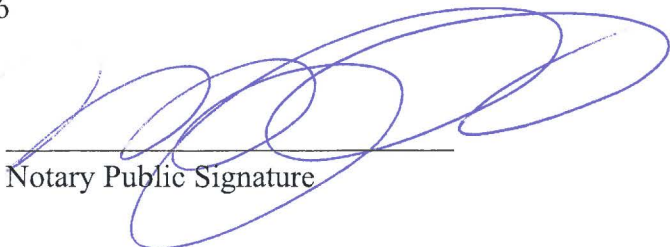
County of Morris

I certify under penalty of law that I have personally examined and am familiar with the information submitted in this document (Supplemental Response to EPA Request for Information), and that based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate, and complete, and that all documents submitted herewith are complete and authentic unless otherwise indicated. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment. I am also aware that my company is under a continuing obligation to supplement its response to EPA's Request for Information if any additional information relevant to the matters addressed in EPA's Request for Information or the company's response thereto should become known or available to the company.

John Trombley
Givaudan Fragrances Corp.
Head of Consumer Products


SIGNATURE

Sworn to before me this 2nd day of November, 2016


Notary Public Signature

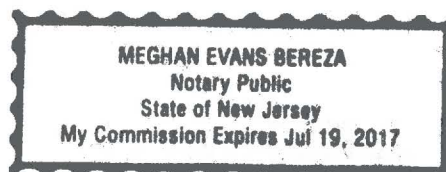


Exhibit L

Copy: HKW 11/29/83

G I V A U D A N

GIVAUDAN CORPORATION

125 Delawanna Avenue
Clifton, New Jersey 07014
Phone: (201) 546-8000
Cable: Givaudanco, Clifton
Telex: 138901

October 26, 1983

Mr. Raymond Basso
Hazardous Waste Site Branch
U.S. Environmental Protection Agency
Region II
26 Federal Plaza - Room 402
New York, New York 10278

RE: CERCLA 104/RCRA 3007 REQUEST FOR INFORMATION
Givaudan Corporation
Clifton, New Jersey

Dear Mr. Basso:

The purpose of this letter is to respond to the questions contained on Attachment I to the September 15, 1983 letter from Mr. William J. Librizzi, Director of your Office of Emergency and Remedial Response, to Mr. George Talarico of Givaudan Corporation ("Givaudan") received by Givaudan on September 19, 1983. On October 19, 1983, you granted Givaudan a one-week extension of time to respond.

The questions contained on Attachment I solicit information regarding the manufacturing or processing of "technical grade" 2,4,5-TCP and products made therefrom, as opposed to the manufacturing and processing of, or products made from, "pre-purified" 2,4,5-TCP, and Givaudan has responded to the questions on that basis. Information which would be responsive to the questions had they been intended to apply to "pre-purified" 2,4,5-TCP, however, has been provided by Givaudan to the New Jersey Department of Environmental Protection ("DEP") and is available to you should you wish to have it.

For example, Question 1 solicits information regarding formulations or pesticide derivatives of "technical grade" 2,4,5-TCP. One example given is hexachlorophene made from "technical grade" 2,4,5-TCP. Although Givaudan has manufactured hexachlorophene for many years, all the hexachlorophene manufactured and marketed by Givaudan has been produced from "pre-purified" 2,4,5-TCP; none of it has been produced using "technical grade" 2,4,5-

GIVAUDAN CORPORATION

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TCP. Accordingly, Givaudan has answered the questions only with respect to its limited production, in 1948 and 1949, of "technical grade" 2,4,5-TCP and not with respect to its production of hexachlorophene from "pre-purified" 2,4,5-TCP. EPA's proposed dioxin regulations, published on April 4, 1983, correctly recognized the distinction made by the questions on Attachment I between hexachlorophene manufactured using "technical grade" 2,4,5-TCP, in which 2,3,7,8-TCDD contamination might have occurred, on the one hand, and hexachlorophene made with "pre-purified" 2,4,5-TCP, using a reaction which occurs at rather low temperatures and at acid pH, in which 2,3,7,8-TCDD contamination is not expected to occur, on the other. 40 C.F.R. parts 261, 264, 265 and 775, 48 Fed. Reg. 14514 (April 4, 1983), note 7. All hexachlorophene manufactured and marketed by Givaudan has been produced using only "pre-purified" 2,4,5-TCP utilizing a process such as is described in note 7 of the proposed regulations, so that no detectable levels of 2,3,7,8-TCDD contamination are expected to have occurred. Analyses of Givaudan's finished hexachlorophene have verified the accuracy of that expectation.

The responses are numbered to correspond with the numbered questions contained on that Attachment I. Where indicated, some additional information is submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.:

1. Givaudan does not currently manufacture "technical grade" 2,4,5-TCP and has not done so for nearly 35 years, since 1949. In 1948 and 1949, Givaudan manufactured "technical grade" 2,4,5-TCP, which was distilled into "pre-purified" 2,4,5-TCP and used in hexachlorophene manufacture. 305,000 pounds of "pre-purified" 2,4,5-TCP was produced during that period from "technical grade" 2,4,5-TCP.

2. a) All "technical grade" 2,4,5-TCP Givaudan produced is believed to have been used to produce "pre-purified" 2,4,5-TCP.

b) Givaudan records show the purchase of a small amount of "technical grade" 2,4,5-TCP from Dow Chemical for experimental purposes only. Copies of all available 2,3,7,8-TCDD analyses have already been provided to and are on file with DEP in connection with their investigation of possible 2,3,7,8-TCDD contamination at Givaudan's facility.

3. Information responsive to Question 3 has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

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4. a) Givaudan believes that "technical grade" 2,4,5-TCP was manufactured by the alkaline hydrolysis of 1,2,4,5 tetrachlorobenzene with caustic soda dissolved in ethylene glycol. After reaction, a batch was neutralized with muriatic acid and the sodium chloride precipitate was removed by filtration. The filtrate was diluted with water and the TCP was extracted with benzene. The benzene extract was washed with water and the benzene was removed by distillation. Ethylene glycol was recovered by fractionation and was reused in the process.

Further information responsive to Question 4 a) has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

4. b) No "technical grade" 2,4,5-TCP has been manufactured by Givaudan for nearly 35 years. In the relevant 1948 and 1949 time period, a vacuum still was used to purify 2,4,5-TCP. From our knowledge of TCP manufacturing operations, Givaudan believes that the following wastes in approximately the following quantity ranges were generated during the 2,4,5-TCP purification step:

- | | |
|--------------------|-----------------|
| 1. Light Fractions | 2-3 lb./lb. |
| 2. Still Bottoms | 0.2-0.3 lb./lb. |

It is believed that none of these wastes were combined with wastes from other processes.

Further information responsive to Question 4 b) has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

5. Givaudan has no records from which specific decontamination procedures used with respect to "technical grade" 2,4,5-TCP manufacturing equipment can be determined; however, Givaudan's standard equipment cleaning and decontamination procedures, which are believed to have been followed with respect to equipment at one time used in the manufacture of "technical grade" 2,4,5-TCP, include thorough steam cleaning and solvent washing which Givaudan believes eliminated any possibility of 2,3,7,8-TCDD contamination. Because of the strong odor of "technical grade" 2,4,5-TCP, especially thorough cleaning and decontamination procedures are believed to have been used before reuse of equipment for manufacturing or processing of other chemical substances.

6. It is believed that the light fractions and still bottoms described in 4 b) above were drummed. Givaudan has no records describing the methods of collection, storage or disposal of such wastes, the names and addresses of haulers who might have hauled such wastes, or disposal site locations.

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Page 4

Further information responsive to Question 6 has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

7. a) There are no drums in storage containing light fractions or still bottoms generated in the manufacturing of "technical grade" 2,4,5-TCP. Givaudan has no records from which the total amount of such wastes generated during the history of its facility can be determined.

Further information responsive to Question 7(a) has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

7. b) Givaudan has no records from which the total amount of wastes generated in the manufacture of "technical grade" 2,4,5-TCP can be determined, nor can Givaudan determine the dates of disposal, the amount disposed of on each occasion, the waste hauler or the disposal location. Whatever disposal activity that may have occurred, of course, took place nearly 35 years ago.

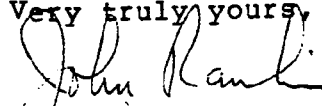
Further information responsive to Question 7(b) has been submitted on a separate sheet under a confidentiality claim pursuant to 40 C.F.R. 2.200 et seq.

8. Copies of all records of 2,3,7,8-TCDD testing with respect to the wastes identified in 4 and 5 have been provided by Givaudan to and are on file with DEP.

9. DEP is coordinating the investigation of possible 2,3,7,8-TCDD contamination at the Givaudan site. This investigation has involved the production by Givaudan to DEP of hundreds of analyses and large volumes of documents. DEP has all of this information, and Givaudan requests that, to the extent necessary, these documents be consulted at DEP, so that needless duplication of this burdensome document production can be avoided.

If there are any questions regarding this matter, please contact me at (201) 365-8521.

Very truly yours,



John Rankin, Vice President
Operations

Att.



DOW CHEMICAL U.S.A.

MICHIGAN DIVISION
MIDLAND, MICHIGAN 48640

February 6, 1980

FEB. 11, 1980

COPIES: J. BRADERICK

L. LEVY ~~LEV~~ WILL THIS MAKE HEX. NON-US?

M. MANOWITZ

P. GROSS

L. BLECKER

Ref

Mr Robert Aron
Givaudan Corp.
100 Delawana Ave.
Clifton, New Jersey 07014

RE: 2, 4, 5 TRICHLOROPHENOL TECHNICAL GRADE

Dear Sir;

Inspection of the remaining drums of 2, 4, 5 Trichlorophenol technical grade (TCP), indicates that lot identity can be assigned to only 8 drums. These drums have received minor damage and some superficial rusting has occurred. Nevertheless, the integrity of the drums appears to have been maintained. The TCP is part of lot number MM06139, and has the following analysis:

<u>COMPONENT</u>	<u>% BY WEIGHT</u>
3, 4 Dichlorophenol	0.04
2, 4; 2, 5; 2, 6 Dichlorophenols	0.17
2, 3, 6 Trichlorophenol	0.28
2, 4, 5 Trichlorophenol	95.12
Monochloromethoxyphenol	N.D.
Dichloromethoxyphenol	4.17
Trichloroanisole	0.01
Tetrachlorophenol	N.D.
2, 3, 7, 8 Tetrachlorodibenzo-p-dioxin	<0.01 ppm

If I can be of further assistance please do not hesitate to call.

Regards,

J.R. Ulrich
John R. Ulrich, Supt.
Trichlorophenol Plant
Dow Chemical Company
Midland, Michigan 48640

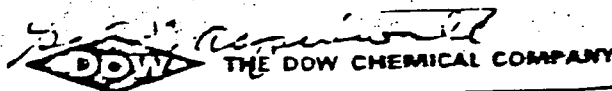
(517) 636-3219

cc: Dave Cheek - Dow Chemical Co.,

J.T. STICKLER, B.T.
A SAMPLE SENT TO 152
2/11/80



CERTIFICATE OF ANALYSIS



DESCRIPTION OF MATERIAL 2,4,5-Trichlorophenol, Technical		DATE 12-27-78
DOW ORDER NO. 4645599	AMOUNT 6 drums	CAR OR TRUCK NO.
LOT NUMBERS		CUSTOMER ORDER NO. R21661
CUSTOMER NAME AND ADDRESS Givaudan Corporation Attention: Mr. G.T. VonEssen, Purchasing Dept. 100 Delawanna Avenue Clifton, NJ 07014		DATE SHIPPED 12-27-78
		DRY LAB FILE NO.

I CERTIFY: That the material shipped has the following analysis:

	<u>Lot Number</u> MM07248
2,4,5-Trichlorophenol	94.8%
2,3,6-Trichlorophenol	0.3%
Dichlorophenols	0.1%
Dichloromethoxyphenol	4.7%
2,3,7,8-Tetrachlorodibenzo-p-Dioxin	<u><0.01 ppm</u>

GIVAUDAN

GIVAUDAN CORPORATION

125 Delawanna Avenue
Clifton, New Jersey 07014
Phone: (201) 546-8000
Cable: Givaudanco, Clifton
Telex: 138901

CONFIDENTIAL

CONFIDENTIAL

PROPRIETARY AND TRADE SECRET INFORMATION

GIVAUDAN CORPORATION

CONFIDENTIAL

RIDER TO LETTER, DATED OCTOBER 26, 1983
from Givaudan Corp. to Mr. Raymond Basso

CONFIDENTIAL

PROPRIETARY AND TRADE SECRET INFORMATION

3. Because nearly 35 years have elapsed since Givaudan last produced "technical grade" 2,4,5-TCP, records from which information responsive to Question 3 can be determined are extremely limited. Chemical substances currently manufactured or processed using two pieces of equipment that are believed at one time to have been used in the manufacture of "technical grade" 2,4,5-TCP are Musk Tibetene, (2,6 dinitro-2,4,5-trimethyl tertiary-butyl benzene), Musk Xylol (2,4,6-trinitro-tertiary butyl meta Xylene), Cetonal crude (mixture of 2-methyl-4-(2,6,6,-trimethyl-2-cyclohexenyl) butanal, and 2-methyl-4-(2,6,6,-trimethyl-1-cyclohexenyl)-butanal), and Melonal crude (2,6-dimethyl-5-hepten-1-al). The two pieces of equipment in question have been repeatedly cleaned and painted during the 35-year period since they were last used to produce "technical grade" 2,4,5-TCP and analyses of recent wipe samples have disclosed no detectable levels of 2,3,7,8-TCDD contamination.

Givaudan has no records from which a determination can be made as to past use of specific pieces of equipment in the manufacture or processing of specific chemical substances, nor can past dates of manufacture, quantities produced or product names of specific chemical substances manufactured or processed in specific pieces of equipment be determined.

4. a) (Second Paragraph)

Musk Tibetene is produced by dissolving 5-tertiary butyl-2,3-tri-methyl-4,6-dinitrobenzene crude in heptane. Reactants are washed with sodium hydroxide liquid and the alkaline extract is drained to sewer. The heptane solution is cooled, the crystals are filtered to yield Musk Tibetene and the mother liquor is distilled. The residue from this distillation is recycled until depleted of product and then sent for disposal. The recovered heptane is reused.

Musk Xylol is produced by dissolving crude 5-tertiary butyl-1,3-dimethyl-2-4-6-trinitrobenzene in heptane. The hot heptane solution is washed with sodium hydroxide which is separated and drained to sewer. The hot heptane solution is cooled and filtered to produce crystalline Musk Xylol and mother liquor. Distillation of the mother liquor produces a residue. The residue is reworked until depleted and then reused at the Givaudan site. The recovered heptane is reused.

GIVAUDAN CORPORATION

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Melonal Crude is produced by reacting methyl heptenone with ethyl chloroacetate in the presence of sodium methylate to form "Melonal Glycidic Ester" crude. The ester is saponified by the addition of alkali, acidified and the resulting acid separated by extraction. Melonal crude is produced by decarboxylation of the glycidic acid.

Cetonal Crude is produced by reacting a mixture of dihydro alpha and B-ionone with ethyl chloroacetate in the presence of sodium methylate utilizing toluene as a solvent. The crude product is quenched on water and separated. The resulting glycidic ester is saponified with caustic and recovered by acidification and extracted with toluene. The toluene solution is decarboxylated to yield Cetonal Crude.

4. b) (Second Paragraph)

The Musk Xylol, Melonal Crude, and Cetonal Crude processes produce no wastes. The Musk Tibetene process produces a still bottom residue. This residue is generated at the rate of 0.25 lb./lb. of product. The residues have never been analyzed; therefore, chemical composition is unknown. The Musk Tibetene residues are not combined with other wastes.

6. Givaudan has no records from which past collection, storage or disposal practices with respect to wastes generated in the manufacture of Musk Tibetene can be determined. Currently, such wastes are collected in drums and stored in Givaudan's RCRA permitted hazardous waste disposal facility for future disposal.
7. No wastes described in 4 or 5, except those produced by the Musk Tibetene process, are currently in storage. The total volume generated of these wastes is unknown. The Musk Tibetene wastes are currently stored in drums on site. The total volume of such wastes generated or stored during the life of the Givaudan facility is unknown.

Exhibit M

GIBBONS P.C.

One Gateway Center
Newark, NJ 07102-5310
Tel: (973) 596-4500
Fax: (973) 596-0545
Attorneys for Defendant
Givaudan Fragrances Corporation

**IN THE UNITED STATES DISTRICT COURT
FOR THE DISTRICT OF NEW JERSEY
NEWARK VICINAGE**

OCCIDENTAL CHEMICAL
CORPORATION,

Plaintiff,

v.

21ST CENTURY FOX AMERICA, INC.; *et*
al.,

Defendants.

Hon. Madeline Cox Arleo
Hon. Joseph A. Dickson

Civil Action No. 2:18-cv-11273

**OBJECTIONS AND ANSWERS OF
DEFENDANT GIVAUDAN
FRAGRANCES CORPORATION TO
STANDARD SET OF
INTERROGATORIES TO BE
ANSWERED BY PLAINTIFF AND
DEFENDANTS**

Defendant Givaudan Fragrances Corporation (“Givaudan” or “Defendant”), by its attorneys, hereby responds to the Standard Set of Interrogatories to Be Answered by Plaintiffs and Defendants (the “Interrogatories”) propounded on April 15, 2019 based on the information presently known and available to Defendant and subject to the general and specific objections set forth below.

GENERAL OBJECTIONS

The following General Objections apply to each of the Interrogatories, and Defendant expressly incorporates the following General Objections in each of its responses. Each response is provided subject to, and without waiver of, these General Objections and any further objections stated in the individual responses.

11. For each Operation identified in response to Interrogatory No. 2, state whether any raw material, products or intermediates, or Waste Material contained any of the COCs and, if so, Identify: (a) which COC(s) it contained; (b) the raw material or Waste Material that contained the COC; and (c) the approximate quantity (by percentage and concentration) of each COC present in the raw material or Waste Material.

ANSWER: Defendant incorporates its objections to Interrogatory No. 2 and makes those objections in response to this Interrogatory. Defendant objects to this Interrogatory as it duplicative, and has been asked and answered in one or more prior Interrogatories. Defendant objects to this Interrogatory as overbroad, as it unconstrained in time and as it seeks to encompass information that has no bearing on the issues in this litigation. Defendant objects to this Interrogatory as vague, as the terms, “raw material,” “products,” and “intermediates,” are undefined. Further, the term “Identify” is defined only as relating to persons or business entities and documents. Defendants objects to this Interrogatory to the extent it seeks information related to trade secret or other confidentiality, or to the extent it seeks information protected by privilege such as the attorney-client privilege or the work product doctrine.

Subject to and without waiving any objections, Defendant answers as follows.

Dioxin (2,3,7,8 TCDD) was present at very low concentrations in a raw material, a purified form of Trichlorophenol (TCP), which Givaudan used to manufacture Hexachlorophene (HCP a/k/a G11), (see GIVA-FED-0000007875), a pharmaceutical grade bactericide. Givaudan used a highly purified form of TCP to meet the strict standards required in the finished G11 product. From approximately 1947 to 1949, Givaudan initiated a pilot production of purified TCP at the plant. In 1948, Givaudan entered into a contract with Hooker Chemical wherein Givaudan provided Hooker its patent for manufacturing purified TCP in exchange for Hooker exclusively supplying Givaudan with TCP by this method (See Attachment 52 in the 2016 104(e), GIVA-FED-0000040111 – GIVA-FED-

0000040526). The supply agreement with Hooker ended in the early 1970's. Between 1971 and 1975, Givaudan used available inventory of purified TCP and did not purchase any additional TCP during this time period. (See GIV_NBC_0473569 – GIV_NBC_0474645). Thereafter, Givaudan began purchasing purified TCP from other suppliers, but only if it met Givaudan's strict quality control standards. (Id.). Any shipments of purified TCP that did not meet Givaudan's strict quality control standards were returned to the manufacturer. Records show that only some off-specification TCP material was disposed of by Givaudan. (See GIVA-FED-0000009951 – GIVA-FED-0000010538, GIVA-FED-0000012452 – GIVA-FED-0000012455, GIVA-FED-0000017952 – GIVA-FED-0000018001, GIV_NBC_0498074 – GIV_NBC_0498118, and GIVA-FED-0000040111 – GIVA-FED-0000040526).

Dioxin is reported to be generated in the production of TCP. The purified TCP arrived as solid flakes/powder in drums that were stored in a designated drum storage area for this product. Givaudan began limited production of purified TCP in small scale distillation batches in 1947, and increased production during 1948. In 1949, it supplemented its limited TCP feedstock with purchases of Dowicide. By 1950, onsite production of purified TCP ceased and all purified TCP was purchased from Hooker Chemical. In the G11 manufacturing process, virtually all residues of dioxin from the TCP were contained in filter bottoms (which were drummed for offsite disposal), with less than 1% potentially transferred in waste water from the HCP manufacturing operation. (GIV-NBC_0681645 – GIV_NBC_0681668). Some trace levels of dioxin may also have been retained in the finished G11 product.

The production of HCP (*i.e.* G11) did not create or produce dioxin. Low levels of TCDD were only present as a result of impurities in the TCP feed stock. (See March 1, 1984 Waste Streams from HCP Manufacturing, Radian (GIVA-FED-0000012573 – GIVA-FED-0000012598); Dioxins – EPA-600, November 1980, pages 106 to 108 (GIVA-FED-0000011008 – GIVA-FED-0000011414); A Retrospective Job Exposure Matrix for Estimating Exposure to 2,3,7,8 TCDD – NIOSH March 1999 (GIVA-FED-0000008978 – GIVA-FED-0000009000); 1991 NIOSH Fingerhut Report; EPA Sponsored Study (GIVA-FED-0000015591 – GIVA-FED-0000015622)).

Givaudan is not aware of any other designated COC used as a raw material or generated by the manufacturing operations.

12. Identify any contracts You had with the Disposal Sites, any operator(s) of the Disposal Sites, or any party to haul Containers containing Waste Materials to the Disposal Sites and describe: (a) the chemical composition of the materials You disposed of at the Disposal Sites; (b) the time period of this disposal; and (c) the amounts of Waste Materials disposed at the Disposal Sites.

ANSWER: Defendant objects to this Interrogatory to the extent it is overbroad, as it is unconstrained in time and as it seeks to encompass information that has no bearing on the issues in this litigation. Defendant objects to this Interrogatory as vague, as the terms “chemical composition” and “materials” are undefined. Defendant objects to this Interrogatory to the extent it assumes any action or inaction by the Defendant. Defendant objects to this interrogatory to the extent it is incomprehensible as it refers to “the” unspecified “Disposal Sites.” Defendant objects to this Interrogatory to the extent it seeks information related to trade secret or other confidentiality, or to the extent it seeks information protected by privilege such as the attorney-client privilege or the work product doctrine.

Subject to and without waiving any objections, Defendant answers as follows.

CORPORATE CERTIFICATION

I, Richard Wroblewski, am the authorized agent for Givaudan Fragrances Corporation, and I sign the foregoing Answers to Interrogatories for and on behalf of Givaudan Fragrances Corporation. The answers to Interrogatories are not within the personal knowledge of the undersigned, but the facts stated in the foregoing answers to Interrogatories have been assembled by counsel and employees of Givaudan Fragrances Corporation, including the undersigned, and the matters set forth in the aforesaid answers to Interrogatories are in accordance with the information available and are true, insofar as it is possible to verify them. Those with personal knowledge of the facts set forth in the Answers are identified therein or in the documents previously provided in the Response to the Request for Production of Documents. I am aware that if any of the foregoing statements are willfully false, I am subject to punishment.

Dated: July 11, 2019




Exhibit N

MICROFILMED

Dr. W. S. Cump
January 8, 1945

MAR 29 1951

1. ~~Dr. Cump~~
2. ~~Dr. Cump~~
3. Dr. Cump 6/28/62
4. destroyed 9/28/70
Dr. Russell (Geneva)
12/5/62

IMPROVED PROCESS FOR THE MANUFACTURE
OF COMPOUND G-11

The large amounts of sulfuric acid needed in the present process for the manufacture of G-11 and the difficulty of the disposal of the waste sulfuric acid made it desirable to find a method where considerably smaller amounts of sulfuric acid would be applied or where the acid could be reused.

In the first experiments, the 93% sulfuric acid was replaced by 62% and 80% acid which could be filtered off from the reaction product and re-used. However, the results in regard to the quality of the G-11 were inferior.

At present, 715 g. of sulfuric acid 93% are employed for 100 g. of trichlorophenol; it was found that this amount of acid could be cut down to one-third without diminishing the yield or changing the quality of the product.

A much bigger reduction of the amount of sulfuric acid used could only be achieved by carrying out the reaction at a temperature high enough to keep the mixture in a liquid state. Aqueous formaldehyde had to be replaced by trioxane or paraformaldehyde. A large number of experiments showed that the optimum temperature lies between 130° and 140°, that the time of reaction should be very short (a few minutes), that oleum 20% which gives slightly better results than 93% sulfuric acid should be used to the amount of 65 g. per one mol (198 g.) of trichlorophenol. An excess of paraformaldehyde (25% above the theoretical amount) must be used in order to re-

duce unchanged trichlorophenol to a minimum. Iron apparatus is entirely satisfactory.

The modified process is illustrated by the following example:

A mixture of 198 g. of Dowicide #2, purified by vacuum distillation, and of 18.8 g. of paraformaldehyde are heated to 75° and well stirred. 65 g. of oleum 20% is added dropwise and the addition is so regulated that the temperature rises slowly without outside heat, reaching about 135° at the end. The addition of the oleum takes 10 to 15 min. The mixture is stirred for two minutes more and then allowed to run into a solution of 100 g. of caustic soda flakes in 1000 cc of water. The reaction flask is washed with a solution of 25 g. of caustic soda flakes in 250 cc of water. The combined alkaline solutions are heated to boiling until practically all of the melt has gone into solution, and are filtered over filter-cel. 6 g. of alkali-insoluble material remain on the filter pad.

Under good stirring, 62% sulfuric acid (about 120 g.) are added dropwise to the alkaline solution until a pH of 10.3 was reached. The G-11 sodium salt formed is filtered off, and washed with 200 cc of water. It is then suspended in 2 l. of water and acidified with sulfuric acid under stirring until congo red paper turns blue; about 30 g. of 62% sulfuric acid are needed. The G-11 Tech. is filtered, washed with water until acid-free and dried. 170 g. (84% of the theory) of the m.p. 154°-158° are obtained.

The alkaline mother liquor of the G-11 sodium salt was acidified and steam distilled. No unreacted Dowicide #2 was recovered; however, 24 g. of resinous material remained.

Yield:	G-11 Tech.	170 g.
	Resin alkali-soluble	24 g.
	<u>Alkali-insoluble</u>	<u>6 g.</u>
	Total	200 g.

W. Gump.

Dr. W. S. Gump
Delawanna, New Jersey
January 8, 1945.

Exhibit O

Patented Feb. 10, 1948

2,435,593

UNITED STATES PATENT OFFICE

2,435,593

PROCESS FOR MAKING BIS-(3,5,6-TRICHLORO-2-HYDROXYPHENYL) METHANE

Max Luthy, Ridgewood, and William S. Gump, Montclair, N. J., assignors to Burton T. Bush, Inc., New York, N. Y., a corporation of New Jersey

No Drawing. Application June 14, 1945,
Serial No. 599,507

17 Claims. (Cl. 260-619)

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This invention relates to an improved process for making bis-(3,5,6-trichloro-2-hydroxyphenyl) methane (sometimes referred to also as 2,2'-dihydroxy-3,5,6-3',5',6'-hexachloro diphenyl methane).

Bis-(3,5,6-trichloro-2-hydroxyphenyl) methane is a substance having desirable bactericidal and fungicidal properties. It may be employed to advantage in tooth pastes, ointments, creams, lotions and rubber goods, inter alia. In addition, when incorporated in small amounts in soaps, it exhibits the surprising quality—for a phenolic substance—of rendering such soaps germicidal.

We are aware of the prior art method for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane. This known method, as well as the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane itself, has been patented by one of us. The known method involves the condensation of 2,4,5-trichlorophenol with formaldehyde in the presence of sulfuric acid. Large amounts of sulfuric acid are required in the patented process. Also, used therein during the condensation reaction are solvents like methyl alcohol. Moreover, the prior art condensation process calls for low temperatures, e. g., 0° to 5° C., and requires about 24 hours of reaction time.

In accordance with our present invention we avoid the use of large amounts of sulfuric acid, dispense entirely with the use of a solvent during the condensation reaction, conduct the reaction at elevated temperatures (thereby avoiding the need for expensive cooling equipment and material), and obtain substantially complete reaction within a period of minutes instead of hours. In economic terms, our present process results in savings in time and materials, reduction in costs, and increase in productivity of a given unit.

In accordance with our present invention, we react 2,4,5-trichlorophenol and a suitable formaldehyde-yielding material at an elevated temperature in the presence of a minor amount of strong sulfuric acid or oleum and over a period which need not exceed thirty minutes. The reaction products may be worked up in known manner (see U. S. Patent No. 2,250,480) to obtain bis-(3,5,6-trichloro-2-hydroxyphenyl) methane. Alternatively, the hereinafter-described novel purification method, forming a particular aspect of this invention, may be employed to yield substantially pure bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in a simple and commercially desirable manner.

Suitable formaldehyde-yielding materials which can be employed herein include paraform-

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aldehyde and trioxane. Any other substance which will yield formaldehyde under the reaction conditions may also be employed.

As the condensation agent, sulfuric acid may be employed in various acid concentrations. Excellent results have been obtained with sulfuric acid of 100% H₂SO₄ concentration up to and including oleum 20%. However, aqueous sulfuric acid solution of lower strength (e. g., 93% acid strength) and oleum of higher strength (e. g., oleum 50%) can also be employed. As will be understood by those skilled in the art, it is desired to bind the water formed during the reaction. Consequently, it is preferable to utilize oleum, e. g., oleum 20%.

The amounts of 2,4,5-trichlorophenol, formaldehyde-yielding substance and sulfuric acid employed can be varied over wide limits. It has been found advantageous to use the 2,4,5-trichlorophenol and formaldehyde-yielding substance in amounts such that there are present about 2 mols of the trichlorophenol to each mol of CH₂O. Especially desirable results are obtained when formaldehyde-yielding material is used in about 25% excess over that called for by the ratio of 2 mols of 2,4,5-trichlorophenol to 1 mol of CH₂O. When such an excess of formaldehyde-yielding material is employed it is found that there is a practically complete conversion of the trichlorophenol, thereby eliminating the necessity of having to separate or recover unreacted trichlorophenol.

One of the features of our process is the reduction in the amount of sulfuric acid used. Accordingly, it will be understood that it is desirable to use the smallest amount of sulfuric acid or oleum which is needed to accomplish the desired condensation of the trichlorophenol and CH₂O. We have found that especially desirable results are obtained when the quantity of acid or oleum used is of the order of about one third of the weight of the trichlorophenol employed.

The condensation may be conducted at elevated temperatures within a rather wide range. It is desirable to start the reaction at a temperature at or above the melting point of 2,4,5-trichlorophenol, and to maintain throughout the reaction period such temperature conditions as will permit of the stirring of the contents with equipment normally used for agitation. We have found that the initial temperature may be as low as 65° C. and that the temperature may be permitted to rise to 130° C. or even 150° C. during the reaction. If desired, the entire reaction may be conducted at the higher temperatures. In all

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cases, as will be understood by those skilled in the art, means for controlling the temperature of the contents of the reaction chamber should be at hand.

Though we do not wish to be limited to any particular method of and order in bringing the 2,4,5-trichlorophenol, formaldehyde-yielding material and sulfuric acid or oleum together, we have found it desirable to introduce the 2,4,5-trichlorophenol and formaldehyde-yielding material first into the reaction vessel and then to raise the temperature to at least about 65° C. before introducing the sulfuric acid or oleum. Also, we prefer to add the sulfuric acid or oleum slowly, over a 10 to 15 minute period, though if suitable precautions are taken, e. g., to control the temperature of the contents of the reaction vessel within the aforementioned range, and if rapid dispersion of the acid is effected, the acid may be charged into the reaction vessel much more rapidly.

As will be understood by those skilled in the art, the process is not restricted to any particular length of time of reaction. Suffice it to say that the reaction should be conducted as long as it takes to convert substantially all of 2,4,5-trichlorophenol into bis-(3,5,6-trichloro-2-hydroxyphenyl) methane. The time that this will require will depend on various factors, such as the ratio and amounts of the reactants and condensation agent, the facilities available to remove the heat liberated by the reaction, and the temperature of reaction. We have found that the reaction is substantially complete in thirty minutes and, in some cases, five minutes are long enough.

As noted above, a particular aspect of the present invention involves a novel method of isolating in substantially pure form the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane formed during the reaction. This novel method is much simpler and more commercially feasible than the known method of isolating and purifying the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane formed by condensing 2,4,5-trichlorophenol and a formaldehyde-yielding material.

In general, the novel method involves the formation and isolation of a mono-alkali metal salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane and the regeneration of the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by treatment of the mono-alkali metal salt with acid. Substantially pure bis-(3,5,6-trichloro-2-hydroxyphenyl) methane may be obtained from the regenerated bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by crystallization from a suitable solvent, e. g., toluene or benzene.

In carrying out this novel method of isolating and purifying bis-(3,5,6-trichloro-2-hydroxyphenyl) methane the condensation of 2,4,5-trichlorophenol and formaldehyde-yielding material is effected in accordance with this invention and the contents of the reaction vessel are made alkaline by running it into an excess of an aqueous solution of a base, such as sodium or potassium hydroxide. Alkali-insoluble material is then filtered off through a suitable filter medium. Prior to filtration, the contents may be boiled for several minutes to facilitate solution of alkali-soluble material.

The pH of the alkaline solution is then brought down, preferably to about 10.3 to about 11. A suitable agent for this purpose is strong sulfuric acid, e. g., of 62% acid strength. The mono-alkali metal salt of bis-(3,5,6-trichloro-2-hydroxy-

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phenol) methane precipitates out of solution at a pH within the range of about 10.3 to about 11. It is filtered and then washed with water. Unreacted 2,4,5-trichlorophenol and other impurities remain in solution. The mono-alkali-metal salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane is then suspended in a large quantity of water and acidified, e. g. with 62% strength sulfuric acid. Bis-(3,5,6-trichloro-2-hydroxyphenyl) methane is thereby regenerated. Crystallization from a suitable solvent, e. g., toluene yields substantially pure bis-(3,5,6-trichloro-2-hydroxyphenyl) methane.

In order to explain the invention more specifically the following examples are given, but it is understood that they are for purposes of illustration and are not to be construed as limiting the scope of the invention.

Example I

A mixture of 198 grams of 2,4,5-trichlorophenol and 18.8 grams of paraformaldehyde were heated to 65° C. and well stirred. 65 grams of oleum 20% was added dropwise and the addition was so regulated that the temperature increased, without the application of external heat, until it reached 135° C. at the end of the acid addition, which took 10 to 15 minutes. The contents of the reaction vessel were stirred for two minutes more and then allowed to run into a solution of 100 grams of sodium hydroxide in 1000 cc. of water.

The reaction flask was washed with a solution of 25 grams of sodium hydroxide in 250 cc. of water. The combined alkaline solutions were heated to boiling for five minutes. A small amount (6 grams) of alkali-insoluble material remained and was filtered off. Sulfuric acid (62% H₂SO₄ content) was then added at room temperature dropwise under stirring to the filtrate until a pH of 10.3 was reached. This required about 80 grams of the acid. The mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane precipitated out of solution and was filtered and then washed with 200 cc. of water. The salt was then suspended in 2000 cc. of water and sulfuric acid (62% H₂SO₄ content) was added under stirring until the contents were acid to Congo red paper. This required about 30 grams of the acid.

The resulting bis-(3,5,6-trichloro-2-hydroxyphenyl) methane was filtered, washed with water until acid-free and dried to constant weight at 100° C. (170 grams, melting point 154°-158° C.) Crystallization of the 170 grams of dried bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from 300 grams toluene yielded a first crop amounting to 105 grams of substantially pure bis-(3,5,6-trichloro-2-hydroxyphenyl) methane, having a melting point of 161°-163° C.

Example II

A mixture of 198 grams of 2,4,5-trichlorophenol and 18.8 grams of paraformaldehyde were heated to 65° C. and well stirred. 65 grams of oleum 20% was added dropwise and the addition was so regulated that the temperature increased, without the application of external heat, until it reached 135° C. at the end of the acid addition, which took 10 to 15 minutes. The contents of the reaction vessel were stirred for two minutes more and then allowed to run into a solution of 100 grams of sodium hydroxide in 1000 cc. of water.

The contents of the reaction vessel were stirred for 2 minutes more and then allowed to run into a solution of 150 grams of caustic potash flakes

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(94% KOH) in 1000 cc. of water. The reaction flask was washed with a solution of 25 grams of caustic potash flakes in 250 cc. of water. The combined alkaline solutions were heated to boiling for 10 minutes and filtered while hot. A small amount of alkali-insoluble material remained on the filter. Sulfuric acid (62% H₂SO₄ content) was then added at room temperature dropwise under stirring to the filtrate until a pH of 10.3 was reached. This required about 80 grams of the acid.

The mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane precipitated out of solution, was filtered off, and then washed with 500 cc. of water. The salt was then suspended in 2000 cc. of water and sulfuric acid (62% H₂SO₄ content) was added under stirring until the contents were acid to Congo red paper. This required about 30 grams of acid. The resulting bis-(3,5,6-trichloro-2-hydroxyphenyl) methane was filtered, washed with hot water until free of sulfuric acid and potassium sulfate and dried to constant weight at 100° C. (185 grams, melting point 154°-157° C.). Crystallisation of the 185 grams of dried bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from 320 grams toluene yielded a first crop amounting to 115 grams of substantially pure bis-(3,5,6-trichloro-2-hydroxyphenyl) methane, having a melting point of 161°-163° C.

Similar results as to yield and product, i. e., the mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane, are obtained, if in this example the 150 grams of caustic potash flakes are replaced by a mixture of caustic alkalis consisting of 56 grams of caustic soda and 72 grams of caustic potash flakes.

The foregoing illustrates the practice of this invention which however is not to be limited thereby but is to be construed as broadly as permissible in view of the prior art and limited solely by the appended claims.

We claim:

1. In the process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by condensing 2,4,5-trichlorophenol with a formaldehyde-yielding substance, the improvement which comprises conducting the condensation at elevated temperatures and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an amount not substantially more than one-third the weight of the 2,4,5-trichlorophenol employed.

2. In the process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by condensing 2,4,5-trichlorophenol with a formaldehyde-yielding substance, the improvement which comprises conducting the condensation at elevated temperatures within the range of about 65° C. to about 150° C. and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an amount not substantially more than one-third the weight of the 2,4,5-trichlorophenol employed.

3. In the process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by condensing 2,4,5-trichlorophenol with a formaldehyde-yielding substance, the improvement which comprises conducting the condensation at elevated temperatures within the range of about 65° C. to about 150° C. and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an

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amount equal to not more than one-third the weight of the 2,4,5-trichlorophenol employed.

4. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting 2,4,5-trichlorophenol with paraformaldehyde at elevated temperatures within the range of about 65° C. to about 150° C. and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an amount not substantially more than one-third the weight of the 2,4,5-trichlorophenol employed.

5. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C.

6. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to about 30 minutes.

7. In the process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by condensing 2,4,5-trichlorophenol with a formaldehyde-yielding substance, the improvement which comprises conducting the condensation at elevated temperatures and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an amount not substantially more than one-third the weight of the 2,4,5-trichlorophenol employed, and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono alkali metal salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

8. In the process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane by condensing 2,4,5-trichlorophenol with a formaldehyde-yielding substance, the improvement which comprises conducting the condensation at elevated temperatures and in the presence of a substance selected from the group consisting of sulfuric acid having at least about 93% H₂SO₄ content by weight and oleum, said substance being employed in an amount not substantially more than one-third the weight of the 2,4,5-trichlorophenol employed, and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

9. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 150° C., and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substan-

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tially pure form by forming and isolating the mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

10. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 150° C., and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

11. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to about 30 minutes, and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono alkali metal salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

12. The process for preparing bis-(3, 5, 6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to 30 minutes, and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

13. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to 30 minutes, and isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt.

14. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 150° C., isolating the bis-(3,5,6-tri-

8

chloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt by acidifying with sulfuric acid, and recrystallizing the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from a suitable solvent.

15. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 150° C., isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt by acidifying with sulfuric acid, and recrystallizing the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from a suitable solvent.

16. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to 30 minutes, isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono sodium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt by acidifying with sulfuric acid, and recrystallizing the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from a suitable solvent.

17. The process for preparing bis-(3,5,6-trichloro-2-hydroxyphenyl) methane which comprises reacting about 198 parts by weight of 2,4,5-trichlorophenol with about 18 parts by weight of paraformaldehyde in the presence of about 65 parts by weight of oleum 20%, at elevated temperatures within the range of about 65° C. to about 135° C. for a period of about 5 to 30 minutes, isolating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane in substantially pure form by forming and isolating the mono potassium salt of bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from the reaction products and regenerating the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from said salt by acidifying with sulfuric acid, and recrystallizing the bis-(3,5,6-trichloro-2-hydroxyphenyl) methane from a suitable solvent.

MAX LUTHY.
WILLIAM S. GUMP.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

70	Number	Name	Date
	2,250,480	Gump	July 29, 1941

Exhibit P

H.C. Grebe

November 18, 1941

G-11 Process

(Sodium Salt Method)

Building 36-D.

After the condensation of 500 lbs. of Dowicide #2 with for-
maldehyde, 1,000 lbs. of ice are added to the 800^{gallon} steel reactor
(A) as is the present procedure. The strong acid slurry is then
pumped into the existing 600 and 800 gallon lead-lined tanks (B)
and (C) which have been previously charged with 400 and 530 gallons
of water respectively. The diluted slurry is pumped by means of
the same Duriron pump to a 60" acid resistant centrifuge (1). The
acid filtrate containing 26% sulfuric acid and wash are sent to
the sewer. The wet crude G-11 cake is discharged through the bot-
tom of the centrifuge into a cart and is transferred to building
47. On the basis of experiments carried out in a 12" centrifuge
(RPM 1,500), the wet cake which contains 45% water at this stage
has a volume of 19 cubic feet and weighs 910 lbs. Thus, on the
basis of a 2" cake in the centrifuge, 4 loads will be necessary.

Building 47.

The wet G-11 cake is hoisted to a platform and dumped into
the 1,000 gallon steel tank (2) in which 750 gallons of water and
110 lbs. of caustic soda flakes have been previously heated to
about 80° C by means of live steam. The temperature is then
raised to the boiling point by an additional amount of live steam
in order to obtain complete solution of the G-11. The steam in-
let should be equipped with an appropriate silencer.

- 1.
- 2.
- 3.

X. Dr. N. K. ... 10/21/41
X. ... 12/1/41
X. ... Ansell (Genova) 12/1/41
H. Brandman - 12/1/41
J. Virgilio - 3/21/78

2.

Several pounds of Super-Cel are thrown into the hot alkaline solution. The solution is then pumped through a small washing type filter press, (4) (which has been previously heated to about 95° C), in order to remove dirt and a small amount of caustic insoluble resin. The filter should have a capacity of approximately 1 cubic foot and ample filtering area so as to permit filtration to proceed at the rate of 50 gallons per minute. (On the basis of some laboratory experiments the actual volume of cake should be only $\frac{1}{2}$ cubic foot but for possible variations in the plant a volume of 1 cubic foot is suggested). The pipe lines to and from the filter press are wrapped with a copper steam coil. In addition, the live steam line connected to the bottom outlet of tank (2) is used to blow out the pipe line in either direction. The solution must be maintained above 90° C in order to prevent the precipitation of the G-11 sodium salt. If necessary, a small amount of live steam can be bled into the pump line by means of the above-mentioned steam line. (The solubility of the sodium salt can be increased by the use of an additional amount of caustic soda). The filtrate is returned to tank (2) until it becomes clear and it is then run into the 1,200 gallon steel kettle (5) which has been previously charged with 100 gallons of water.

The filter press is given a hot water wash and is blown with air. This wash water is also sent to tank (5). The press is dumped when time permits.

The pH of the hot alkaline solution is reduced to about 10.5 by means of the slow addition of approximately 95 pounds of 62% sulfuric acid from the lead-lined acid egg (8). The small lead-lined acid tank in building 45 which is no longer required for the Cuminaldehyde

3.

process can be used for this purpose. The sodium salt begins to precipitate when approximately one-half of the required amount of sulfuric acid has been added. The sodium salt precipitates in the form of long fibrous crystals which mat readily and make the slurry very thick. Therefore, very efficient and thorough agitation must be employed during the addition of the acid in order to prevent the precipitation of impurities at a lower pH which would exist in "patches" throughout the slurry if agitation is not thorough. Such impurities would be slow to redissolve on continued stirring. The acid inlet should be installed so that the acid does not run down the wall of the tank but falls clear of the sides.

The slurry is cooled to room temperature by circulating cold water through the jacket. (Steam should also be connected to this jacket in case it ever becomes necessary to heat the solution before precipitation as may be necessary after a shut down). A close fitting agitator is required for this cooling process as the sodium salt will deposit on the cooling surface and considerably retard the rate of cooling. This deposit is very soft and therefore a scraping type agitator is not necessary. (Sometimes this sodium salt deposit builds up to a thickness of more than 2" on the walls of the crystallizer located in 47 building. This crystallizer is equipped with a turbine type agitator).

After cooling to room temperature, the slurry is pumped to a 48" centrifuge (7). The pump must not be a close clearance pump as the G-11 sodium salt will not pass through the screen of a "Westco" type pump. Based on experiments in the 12" centrifuge, the G-11

4.

790 / 60 = 13.2

sodium salt cake weighs approximately 790 pounds, contains 50% moisture and has a density of 60 pounds per cubic ft. Thus, for a 2" cake, 4 loads will be necessary in a 48" centrifuge. The pH of the filtrate is 10.5 and contains sodium sulfate and the sodium salts of Dowicide and resinous by-products. A bronze centrifuge basket will probably be suitable, however, the possibility of contaminating the G-11 sodium salt with traces of copper should be considered, as traces of copper are known to cause discoloration of soap.

In this process, no attempt is made to recover the Dowicide in the filtrate and therefore the filtrate is discharged to the sewer. More experiments will be made in the laboratory in order to determine if the Dowicide content can be definitely reduced to such a value that it will not be economical to recover it from the filtrate and at the same time obtain a satisfactory yield. If the Dowicide must be recovered, additional equipment, i.e., a 1,200 gal. lead-lined tank, pump and vacuum still will be required.

The G-11 sodium cake after washing with water in the centrifuge is discharged through the bottom of the centrifuge. The cake is hoisted to a platform and dumped into the 1,000 gallon lead-lined(9) steel tank which has been previously charged with 800 gallons of water. The slurry is made acid to Congo Red by means of the addition of approximately 80 lbs. of 62% sulfuric acid in order to decompose the sodium salt and obtain the free acid. Agitation in this tank must also be thorough as the slurry is quite thick. In addition, the agitator must be capable of breaking up the G-11 sodium salt cake so that the sulfuric acid can react with the individual particles and thus insure the complete conversion of the sodium salt to the free acid.

5.

Sodium bicarbonate is then added in order to raise the pH to approximately 7.5 and the G-11 slurry is then pumped to a 48" centrifuge equipped with a bronze basket. The pump (10) should be similar to pump (6) as the G-11 clogs the strainers of Westco pumps. Based on a 2" cake approximately $5\frac{1}{2}$ loads will be required to filter the batch. (Wt. wet cake - 680 lbs.; moisture 45%; density 42 lbs./1 cu. ft.). In view of the fact that more than 4 loads will be required to filter the batch, it may be advisable to use a 60" centrifuge. Experiments will be made in the laboratory to determine if the G-11 can be precipitated in a denser form and thus be able to filter the batch in less than 5 loads in a 48" centrifuge (cake thickness 2").

The filtrates from the centrifuges should run first to 200 gallon "catch all" tanks (separate one for each centrifuge) before flowing to the sewer so that in case the filter medium breaks or the filtrate does not run clear, no G-11 will be lost. The centrifuge pumps should be connected so that the cloudy filtrate in the "catch all" tank can be recirculated through the centrifuge.

After washing with water, the G-11 cake is dried in the existing vacuum shelf drier.

Capacity of Equipment.

With the exception of reactor (A), the above equipment should be capable of processing 3 batches (375 lbs. G-11 Refined per batch) or approximately 1,000 pounds per twenty-four hour day. In order to attain this production rate, at least 2 more or one larger reactor must be installed.

6.

If centrifuges cannot be obtained, consideration should be given to other types of filtration equipment such as a Vallez Filter which could replace either of the last two centrifuges (7) or (11), or a Sweetland Filter which could replace the centrifuge (7) in which the sodium salt is filtered.

H. G. Krebs

Delawanna, N. J.

November 18, 1941

Exhibit Q

V. Weglowski
5/18/79

PRODUCT (FROM 2,4,5-TRICHLOROPHENOL PURE USING TOLUENE AS SOLVENT)

- 1) J. Pons
 - 2) P. Doucette
 - 3) R. Genet
 - 4) A. Gessner
 - 5) S. Gold
 - 6) P. Gross
 - 7) N. Panagiotakis
 - 8) C. Snyder
 - 9) V. Weglowski
 - 10) Production Directors
 - 11) Geneva (2)
- H. Kuhnau 2-20-81*
V. Weglowski 11-81
J. Rankin-12/17/82

OPERATION Reaction

Bldg. No.	Unit No.	Type of Unit	PART I
58	TZ-170	Glass Lined Reactor, 750-Gallon	
82	TZ-114	Glass Lined Reactor, 300-Gallon	
82	TU-147	Glass Lined Feed Tank, 150-Gallon	

EQUIP

Wt. Lbs.	Material	Analysis	Remarks
600	Trichlorophenol Pure	Specific Gravity: 1.30	
1,650	Sulfuric Acid 93%	Specific Gravity: 1.83	
198	Methyl Alcohol	Specific Gravity: 0.792	
132	Aqueous Formaldehyde, 37%		
600	Water		

CHARGE

TRICHLOROPHENOL: WARNING! Dust and fumes irritating to eyes, nose and throat. Do not breathe dusts or fumes. Avoid contact with skin. Wear chemical goggles or face shield, apron, rubber gloves and OV respirator with dust filter when handling.

SAFETY

SULFURIC ACID 93%: DANGER! Causes severe burns. Avoid all contact. Wear chemical goggles and/or face shield, apron and rubber gloves. Avoid breathing mist, wear acid gas respirator if irritating to respiratory system.

METHYL ALCOHOL: DANGER! Flammable. Prolonged or repeated breathing of vapor harmful. Ground all equipment and containers. Wear gloves, full goggles and/or face shield when handling.

FORMALDEHYDE 37%: DANGER! Vapor or liquid causes skin, eye, nose and throat irritation. Wear OV respirator, rubber gloves and full goggles and/or face shield when handling. Avoid all contact with skin.

OPERATING PROCEDURE

Step No.	Procedure	[With Brief Special Remarks]
1.	Check that reactor TZ-114 in Building 82 is clean.	
2.	Charge by vacuum 1,650 lbs. Sulfuric Acid 93% to TZ-114.	
3.	Charge by vacuum 198 lbs. Methyl Alcohol to feed Tank TU-147.	
4.	Turn on agitation and cool batch in TZ-114 to 20°C with cooling water.	
5.	With cooling water on, slowly feed 198 lbs. Methyl Alcohol over 1/2 hour to Sulfuric Acid. Keep batch temperature below 40°C with cooling water and controlling feed of Methyl Alcohol.	
6.	Drain to polyethylene lined drums at 20-25°C and move to Building 58. <u>Wear full face shield, apron and rubber gloves while draining.</u>	
7.	Check that reactor TZ-170 in Building 58 is clean.	
8.	Charge by vacuum Sulfuric Acid/Methyl Alcohol mixture to reactor TZ-170. Start agitation.	
9.	Heat charge in reactor to 70-75°C.	

OPERATING PROCEDURE CONTINUED

10. Charge by vacuum 600 lbs. (1 drum) TCP Pure from hot box.
11. With reactor at 75°C, slowly, over approximately 30 minutes, add from a suspended drum with scale 132 lbs. Aqueous Formaldehyde. Maintain batch at 75-80°C with cooling water, if necessary.
12. Heat charge in reactor to 80°C and agitate 1 hour.
13. After 1 hour agitation, slowly, over 30 minutes, add 600 lbs. (72-gal.) of water through the meter, maintaining the temperature below 90°C. Agitate 5 minutes.
14. After water is added, take a gallon sample. Sample taken from manhole without agitation. Wear full protective gear, face shield, respirator with OV cartridge, rubber gloves and apron. (See Note 1).

PRODUCTS

Wt.	Material	Analysis	Containers, Disposition, etc.
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MISC. NOTES

Note 1: Sample is taken to check consistency of batch. Should be granular. See supervisor before continuing.

	Prepared By	Date	Rev. By Safety Dept.	Date
ORIGINAL	<i>V. Weglowski</i>	5/18/79	<i>A. Druetta</i>	5/31/79
REVISED				

PRODUCT G-11® N.P. PART II

OPERATION Steaming and Separation

Bldg. No.	Unit No.	Type of Unit	Remarks
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58	TZ-170	Glass Lined Reactor, 750-Gallon	
59	TY-134	#1 Settler, 1,000-Gallon Steel	

Wt.	Material	Analysis	Remarks
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1,800 lbs.	Toluene		
400 lbs.	Toluene for wash		

TOLUENE: WARNING! Flammable. Breathing vapor may be harmful. Causes eye injury and skin irritation. Ground all containers. Wear gloves and goggles or face shield when handling.

Step No.	Procedure	[With Brief Special Remarks]
----------	-----------	------------------------------

- | | | |
|-----|---|--|
| 15. | Open drain valves from reactor condenser to run distillate to sewer. Turn on warm water (~70°) to condenser (to prevent TCP from congealing). (Note 2). | |
| 16. | Heat reactor TZ-170 to 100°C. | |
| 17. | With steam on the reactor jacket, inject live steam through bottom drain. Pot temperature rises rapidly to 125-130°C, then slowly drops to 115-120°C during steaming. | |
| 18. | Steam for 2 hours and check distillate with 10% Ferric Chloride Solution (See Note 3). Continue steaming if TCP test positive. | |
| 19. | When steaming is complete, cool batch to 100°C and take a level reading from 2" nozzle. If reading 50" (450-gal.) or greater proceed. If less than 50", stop agitator and allow the batch to settle for 1/2 hour. Then siphon off top water layer to 50" level. | |
| 20. | Set valves to run distillate from reactor condenser back to reactor. Change condenser water from warm to cold. | |

EQUIP

CHARGE

SAFETY

OPERATING PROCEDURE

OPERATING PROCEDURE CONTINUED

21. Charge by vacuum 1,800 lbs. Toluene to TZ-170.
22. Heat to reflux (92-95°C). Reflux 1/2 hour, then shut agitation and jacket steam and settle 1 hour. Keep batch temperature at 95°C.
23. Drain bottom acid layer from reactor to polyethylene lined drums. Wear full protective gear, face shield, rubber gloves and apron. Take 1 gallon sample. Have 8-10 polyethylene lined drums on hand per batch.
24. Preheat settler TY-134 in Building 59. Transfer batch charge from reactor to settler TY-134, then immediately transfer to treatment tank TZ-168 in Building 59.
25. Charge by vacuum 400 lbs. Toluene to reactor TZ-170, pump to settler TY-134, then to treatment tank TZ-168 to clean reactor, settler, and transfer lines.

PRODUCTS

Wt.	Material	Analysis	Containers, Disposition, etc.
3,700-4,000 lbs.	Spent Sulfuric Acid		55-gal. polyethylene lined drums. If material balance of batch OK, drain to sewer.

MISC. NOTES

- Note 2: Discharge connection pipe from condenser during steaming should be extended to lead directly to sewer.
- Note 3: For TCP test - 25 mls. distillate at 25°C, add 5 drops 10% FeCl₃ solution. If purple, continue steaming. If yellow or yellowish orange, steaming complete.

	Prepared By	Date	Rev. By Safety Dept.	Date
ORIGINAL	<i>J. Wylowski</i>	5/18/79	<i>P. Donetto</i>	5/31/79
REVISED				

PRODUCT G-11® N.P. PART III

OPERATION Decolorization and Filtration

Bldg. No.	Unit No.	Type of Unit	Remarks
59	TZ-168	Treatment Tank, 1,000-Gallon Steel	
59	FL-9633	Sparkler Filter	
59	TZ-31	Storage Tank, 1,000-Gallon Glass-Lined	

Wt.	Material	Analysis	Remarks
70 lbs.	Filtrol Grade #4		
3 lbs.	Hyflo Super-Cel		
100 gals.	Toluene		

FILTROL: CAUTION! Not known to be hazardous. Wear dust mask if heavy dusting occurs while handling.

SUPER-CEL: WARNING! Diatomaceous Silica. Inhaling dust harmful. Dust mask or cartridge respirator with dust filter MUST be worn when handling.

TOLUENE: WARNING! Flammable. Breathing vapor may be harmful. Causes eye injury and skin irritation. Ground all containers. Wear gloves and goggles or face shield when handling.

STIK TIGHT ROUGH CEMENT: CAUTION! May be irritating. Wear dust mask if heavy dusting occurs while handling. Material is asbestos free.

Step No.	Procedure	[With Brief Special Remarks]
1.	To G-11/Toluene solution in treatment tank TZ-168, charge 70 lbs. Filtrol #4 and 3 lbs. Hyflo Super-Cel. <u>Wear OV respirator with dust filter when charging.</u>	
2.	Reflux with agitation for 1-1/2 hours with temperature at 110-113°C. A small amount of water will be azeotropically distilled to the separator (10 lbs.).	
3.	Heat jacket of sparkler filter FL-9633 for 15 minutes with full steam.	
4.	Cool batch in treatment tank TZ-168 to 100°C.	
5.	Partially open vent on top of sparkler, close sparkler drain valve and pump batch from treatment tank to sparkler expelling air through the vent valve.	
6.	As soon as sparkler is full, shut vent valve and open sparkler discharge valve sending the filtrate back to the treatment tank on recycle. Keep sight glass full at all times by proper valve adjustment.	

EQUIP

CHARGE

SAFETY

OPERATING

PROCEDURE

OPERATING PROCEDURE CONTINUED

7. Recycle at 95-100°C for at least 15 minutes. When batch is sparkling clear, take 1-gallon sample and label with time, date, batch and operator. Check with supervisor before proceeding.
8. If batch is not sparkling clear, add 4 lbs. of Super-Cel and 4 lbs. Stik Tight Rough Cement to the pump filter basket and recycle until batch is clear. If batch does not become sparkling clear, check with supervisor.
9. When batch is sparkling clear, switch from recycle and filter batch into storage tank TZ-31, adjusting valves as required to keep sight glass full, sparkling clear and free of air bubbles. At this point, do not leave filtration unattended. Go back to recycle tank TZ-168 if filtrate not sparkling clear. Do not pump filtrate which is not clear into storage tank TZ-31.
10. Stop agitation in treatment tank TZ-168 when level is low. When treatment tank is empty, stop pump and reset valves for recycle of filtrate to treatment tank for the next batch.
11. Charge 100 gallons Toluene to treatment tank TZ-168 and heat to reflux. At this point, open vent valve on sparkler and close sparkler discharge valve. Pump agitated wash to sparkler, expelling air from sparkler vent valve. When sparkler is full, shut vent valve, open discharge valve to treatment tank and recycle wash back to treatment tank TZ-168 until sparkling clear. When sparkling clear, pump wash into storage tank TZ-31. If wash not clear, check with supervisor.
12. When treatment tank is empty, repeat Step #11 but return this wash to treatment tank and allow sparkler to drain to treatment tank.
13. Filtrate which is not sparkling clear must never be pumped to storage tank TZ-31. Take a gallon sample from TZ-31 after filtration. Also take a 5-lb. sample of the filter cake from sparkler FL-9633. Wear OV cartridge respirator with dust mask, rubber gloves and apron when handling samples.

Wt.	Material	Analysis	Containers, Disposition, etc.
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PRODUCTS

MISC. NOTES

	Prepared By	Date	Rev. By Safety Dept.	Date
ORIGINAL	<i>J. Weglowski</i>	<i>5/18/79</i>	<i>P. Duetto</i>	<i>5/31/79</i>
REVISED				

PROCESS INSTRUCTION FORM

GIVAUDAN CORPORATION

PRODUCT G-11@ N.P.

PART IV

OPERATION Steam Distillation (Solvent Stripping)

E
Q
U
I
P

Bldg. No.	Unir No.	Type of Unit	Remarks
59	TZ-31	Storage Tank, 1,000-Gallon, Glass	
59	TZ-2 or	Steam Still, 1,000-Gallon, Glass	
59	TZ-48		

C
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Wt.	Material	Analysis	Remarks
2,800-2,900	600 lbs. G-11 2,900 lbs. Toluene 1-2 lbs. Oxalic Acid 400 lbs. Toluene for wash	} Solution	

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F
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Y

G-11: May be irritating - full goggles and dust mask must be worn when exposed to powder.

TOLUENE: WARNING! Flammable. Breathing vapor may be harmful. Causes eye injury and skin irritation. Ground all containers. Wear gloves and goggles or face shield when handling.

OXALIC ACID: DANGER! POISONOUS - Dust or solution can cause severe burns of the skin, eyes and respiratory tract. Dust mask, full goggles, and rubber gloves must be worn when handling.

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Step No.	Procedure	[With Brief Special Remarks]
1.	Check that the Tiger filters and the GAF polishing filters are properly cleaned, installed, heated and ready for service.	
2.	Check that distillate lines are open to the outside portable tankcar and blocked off to the underground ethylene dichloride storage tank. Portable tankcar will serve as a separator with the bottom aqueous layer to sewer and top Toluene layer to drums.	
3.	Charge steam still TZ-2 or TZ-48 with 250 gallons of water and 1-2 lbs. Oxalic Acid. Start agitator.	
4.	Heat steam still to reflux (98-100°C) with live steam and jacket steam. Set valves so that distillate will pass through liquid cooler and separator.	
5.	Keep G-11/Toluene solution in storage tank TZ-31 at 90°C with steam on jacket.	

6. When steam still temperature reaches 98-100°C, feed G-11/Toluene solution to steam still by way of polishing filters. Start with the following: Live steam - 6 lbs. then increase to 10 lbs.
 Roto setting for feed - 2 then increase to 3
 Keep jacket steam on during the steam distillation. Steam still should never be run at positive pressure, which would indicate steam pressure is too high, feed rate too high or that the system has a partial plug.
7. Keep batch in steam still at 95-98°C. If batch temperature drops below 95°C, increase jacket steam and slow feed rate. Record steam pressure in jacket and live steam readings, rotameter settings and condenser water pressure. Take 1 gallon sample of water and Toluene distillates.
8. When the holding tank TZ-31 is empty, rinse the lines between holding tank and steam still with 50 gallons of Toluene and feed to steam still.
9. Distill for 1/2 hour after the feeding is completed. Then check distillate for Toluene. When no Toluene is present, check the distillate for TCP with 10% Ferric Chloride.
10. When batch is free of TCP, cool batch to 90°C, then pump batch to cooling tank in Building 60 (TZ-30). Wash out still with water.
11. Batch is then filtered (Building 60), dried (Building 60) and ground (Building 47).

Wt.	Material	Analysis	Containers, Disposition, etc.
550-585 lbs.	G-11 N.P.	Purity: 98% Melting Point: 161-167 Color: White to Beige	Plastic Lined Lever Paks

- 1) TCP Test: Add 5 drops 10% Ferric Chloride to 25 mls. of (1 oz.) distillate. If the color is purple, TCP is present, continue steaming. If the color is yellow or yellowish-orange, steaming is complete.

	Prepared By	Date	Rev. By Safety Dept.	Date
ORIGINAL	<i>Z. Weglowski</i>	<i>5/18/79</i>	<i>P. Mucillo</i>	<i>5/31/79</i>
REVISED				

Exhibit R

MICROFILMED

MAR 30 1950

2,4,5 TRICHLORPHENOL

*Krebs
8-24-48*

Manufactured at
GIVAUDAN-DELAWANNA INC.
Delawanna, N. J.

This copy issued to

Date issued

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- 4. ~~Mr. Krebs - 9/22-48~~
- 5. Geneva - 7/12-50 - #884

Process for 2,4,5 Trichlorophenol

Sec. No. Authors Page 1.

Date 8-24-48 Original Process H. G. Krebs



CURRENT PROCESS

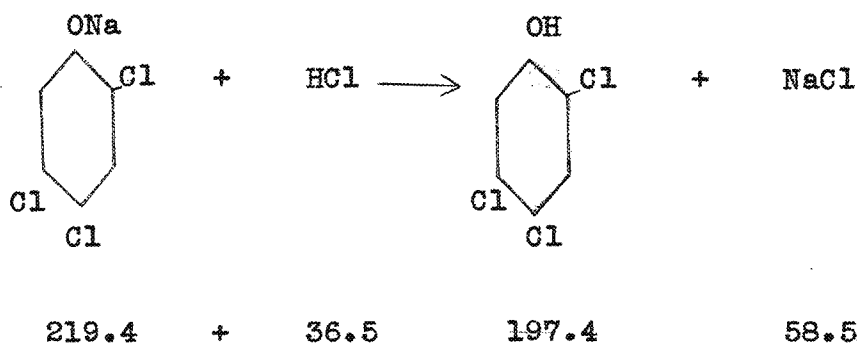
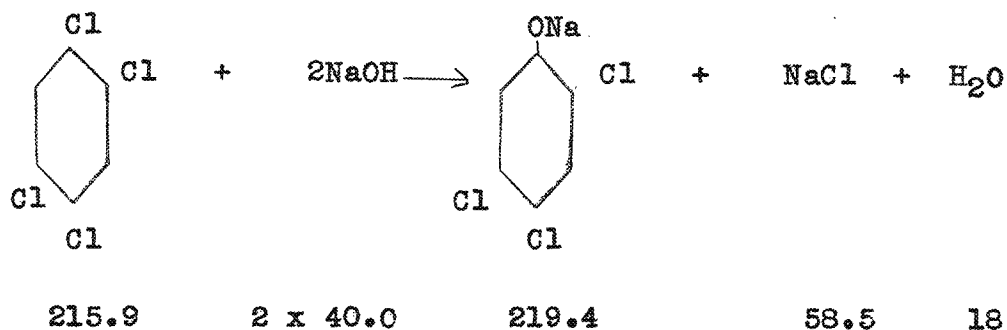
Section I	Chemistry of Process	2 pages
Section II	Raw Materials	3 "
Section III	Equipment	9 "
Section IV	Material and Labor Requirements	2 "
Section V	Operating Procedure	19 "
Section VI	Hazards of Process	2 "
Section VII	Products and By-Products	1 page
Section VIII	Cost Data	1 "
Section IX	Literature and Research Sources	1 "
Section X	Drawings	2 pages

OBSOLETE SECTIONS

SECTION I

CHEMISTRY OF PROCESS

2,4,5 Trichlorophenol is made by hydrolyzing 1,2,4,5 tetrachlorobenzene with an excess of caustic soda dissolved in ethylene glycol at a temperature of 170 to 175°C. After the reaction the batch is neutralized with muriatic acid and the sodium chloride which is precipitated is removed by filtration. The filtrate is diluted with water and the trichlorophenol is extracted with benzol. The benzol extract is washed with water and the benzol is removed by distillation. The crude trichlorophenol is vacuum distilled, and a product with a congealing point of 63°C is obtained. The ethylene glycol which is recovered by fractionation is reused in the process.



The theoretical weight yield based on tetrachlorbenzene is $91\frac{1}{2}\%$. With the fair grade of tetrachlorbenzene employed an actual weight yield of 70 to 76% has been obtained in the plant.

SECTION II

RAW MATERIALS

1,2,4,5 Tetrachlorbenzene:

Melting Point: 136°C minimum

Purchased from Hooker Electrochemical Co. Received in M3208 leverpak containers holding 200 pounds.

Caustic Soda Flakes:

Purity: Minimum 96% as NaOH

Purchased from Sergeant Pulp & Chemical Co., Inc. Received in department in steel drums containing 400 pounds of flakes.

Ethylene Glycol:

Specific Gravity 25/25°C: 1.1115 to 1.1130

Boiling Range: 195 to 205°C

Purchased from Dow Chemical Co. and Carbide & Carbon Chemicals Corp. Received in department in steel drums containing 500 pounds.

Recovered Ethylene Glycol:

As recovered in this process.

Specific Gravity 25/25°C: 1.110 minimum

Muriatic Acid 20° Baumé:

Purity: 31.5 to 35.2% as HCl

Specific Gravity 25/25°C: 1.1528 to 1.1709

Purchased from A. H. Mathieu & Co. and Riches Nelson Co. Received in department in carboys containing 115, 118 or 131 pounds of acid.

Isopropyl Alcohol 99%:

Specific Gravity 25/25°C: 0.783 to 0.787

Distilling Range: Total range not more than 1.5°C including the temperature 82.5°C.

Non-Volatile Matter: Maximum 5 mg. per 100 ml.

Purchased from Standard Alcohol Co., Standard Oil Co., Shell Chemical Co. and Enjay Co., Received in tank cars. Delivered to department in 55 gallon drums containing 300 pounds.

Recovered Isopropyl Alcohol:

As obtained by fractionating weak isopropyl alcohol recovered in this process.

Purity: 85% by weight minimum

Specific Gravity 25/25°C: 0.820 maximum

Benzol:

Specific Gravity 25/25°C: 0.875 to 0.877

Congealing Point: 5°C minimum

Distilling Range: Not more than 1°C including the temperature 80.1°C

Purchased from Stoney Mueller Inc., Calco Chemical Div., and Jones & Laughlin Steel Co. Received in tank cars. Delivered to department in 55 gallon drums.

Benzol Recovered:

As recovered in this process.

Specific Gravity 25/25°C: 0.870 to 0.877

Soda Ash:

Purity: Minimum 98% as Na_2CO_3

Purchased from A. H. Mathieu, Solvay Sales and Solvay
Process. Received in department in paper bags contain-
ing 100 pounds.

#4619 150 gal. half jacketed steel tank with bolted cover, equipped with thermometer well and jacketed vent pipe leading through the roof. This tank is used to vent residual 20 lb. pressure from hydrolysis kettle at the end of the reaction.

SECTION III

EQUIPMENT

Building 54

#8454 Stainless Steel Reaction Kettle; capacity 250 gallons; gas fired, anchor type agitator driven at 52 RPM through #8412 Nettco speed reducer by 2 horsepower motor #8673; stainless steel cooling coil with 14 square feet of cooling surface; top of kettle equipped with 6" charging hole, 1 $\frac{1}{4}$ " drum charging leg with flexible hose, thermometer well for 0 to 300°C Motoco thermometer and Gotham recorder, steam jacketed vent line, combination vacuum, vent and air lines, cooling coil inlet and outlet; 2" relief valve set at 25 pounds, 0 to 60 pound pressure gage; 2" outlet on bottom of kettle closed by 2" nickel plug cock connected to 1 $\frac{1}{2}$ " stainless steel blow line to building 60 and to drain cock. Used for hydrolysis reaction.

#4619 —

#8543 Gotham single pen recorder 0 to 300°C. Used to record temperature in hydrolysis kettle #8454.

#8484 Air Hoist; Ingersoll-Rand; capacity 500 pounds. Used to charge hydrolysis kettle #8454.

#8868⁷ Monel High Vacuum Still; capacity 136 gallons, gas fired, oil jacket, equipped with 1 $\frac{1}{4}$ " drum charging leg with flexible metallic hose; thermometer well in oil jacket for 0 to 300°C Motoco thermometer; thermometer well in kettle for bulb of Gotham recorder; 15 pound steam traced relief valve and vent, 18" diameter packed monel column filled to a height

of 10 feet with 1" porcelain Raschig rings; monel coil dèphlegmator with 5.5 sq. ft. of cooling surface at top of column. Used to distill the crude trichlorophenol.

#8824 Monel Coil Condenser - 36 square feet of cooling surface; cooled by recirculated warm water at 70°C; inlet connected by gooseneck to top of 18" diameter packed column, outlet to either monel receiver #8884 or #8885. Used to condense trichlorophenol vapors from monel still #8868.

#8884 & #8885 Monel Water Jacketed Receivers; capacity 25 gallons each; jackets supplied with water at 70°C from water recirculation system; steam heated level gage glass on side; top connections to condenser #8824, to 0 to 30" vacuum dial type gage, to Zimmerli gage, to ^{jacketed} vacuum trap which in turn is connected to coarse, medium and high vacuum supply; $\frac{3}{4}$ " drain cock on bottom. Used to receive trichlorophenol condensate.

#8691 Worthington Iron Centrifugal Pump; capacity 25 GPM; $1\frac{1}{2}$ " inlet connected to hot water tank automatically controlled at 70°C by TR-21 Sarco regulator; outlet connected to jackets of monel receivers, monel condenser and to monel dèphlegmator coil through 0 to 5 GPM rotameter. 0 - 100°C Motoco thermometers at inlet and outlet of dèphlegmator coil, Used to circulate warm water through monel condenser #8824, dèphlegmator coil, and jackets of monel receivers #8884 & #8885.

#8772 Gotham Recorder - three pen 30 to 240°C. Used to record still, vapor and distillate temperatures of monel still.

#8728 Glycol Recovery Still; steel; capacity 600 gallons; 100 pound code jacket connected to steam supply and cooling water; top connected to #8787 30" diameter packed column, to drum charging line, to 275 gallon storage tanks #5640, 5641 5642 & 9029; thermometer well for one bulb of three pen Gotham recorder and one to 200°C Motoco thermometer; drain on bottom to sewer and to residue drum. Used to distill the weak ethylene glycol.

#8787 Packed Column; steel; diameter 30 inches; height 11 feet; filled with 1" porcelain Raschig rings to a height of 9½ feet; reflux distributor at top; two wall collectors and distributors; bottom connected to top of still #8728; top connected by gooseneck to condenser #8767; side connection at top for liquid reflux connected to reflux proportioner. Used to fractionate the ethylene glycol.

#8767 Steel Water Tube Condenser; 15" diameter x 56" length; 60.6 sq. ft. cooling surface; 1½" water inlet and outlet and condensate connections; vapor inlet connected to top of column #8787; distillate outlet connected ^{to} reflux proportioner to liquid cooler. Used to condense vapors from column #8787.

#8670 & 8671 Steel Vacuum Receivers; capacity each 150 gallons; 31½" inside diameter; distillate inlet at top from liquid cooler; top connected to coarse, medium and fine vacuum supply; level gage glass on side; bottom outlet to drum line running to outside of building, to drum line inside of building, and to 750 gallon steel tank. Used to receive benzol, isopropyl,

alcohol, water and ethylene glycol fractions from liquid cooler.

Panel Board for glycol still; equipped with four 0 to 150°C, Motoco thermometers for indicating temperature of reflux flowing to column, of condensate from tubular condenser #8767, of water to and from tubular condenser; also Zimmerli gage for indicating absolute pressure at top of column; mercury U tube gage for indicating pressure drop in column; Gotham temperature recorder #8636 mounted in center of board.

#8599 Horizontal Steel Tank; 750 gallon capacity; inlet at top from receivers #8670 and 8671; level gage glass on side; bottom connected to Westco pump #8561. Used to receive water fraction from glycol recovery still. 3'11 $\frac{1}{2}$ " I.D.

#8561 Westco Turbine Pump; all bronze; capacity 25 GPM. Used to pump recovered water from 750 gallon storage tank to building 60.

#8636 Gotham Temperature Recorder; three pen; range 0 to 200°C. Used to record temperature of still, vapor and distillate of ethylene glycol recovery still #8728.

#8656 Nash Hytor Vacuum Pump; Size A1-673; $\frac{1}{2}$ " water inlet; 1 $\frac{1}{2}$ " vacuum inlet; 1 $\frac{1}{2}$ " discharge; driven at 1750 RPM by 3 horsepower motor #8891. Used to supply coarse vacuum for glycol recovery still #8728 for charging and distilling light fractions.

* This kettle has an 8" galvanized vent pipe leading outside the building to remove fumes while the hot batch is being blown over.

Building 60

#6220 Kettle; stainless steel; rated capacity 250 gallons; jacket connected to steam and cooling water; turbine type agitator driven at 190 RPM through #8412 speed reducer by 2 horsepower motor #8673; top of kettle fitted with 6" hand hole, thermometer well for 0 to 200°C Motoco thermometer, vent and vacuum connection, 1½" inlet from hydrolysis kettle #8454 in building 54, ½" Saran muriatic acid connection; bottom connection to drain cock and to Westco pump #8449. Used to cool and neutralize glycol slurry from hydrolysis kettle. This kettle is actually too small for this process. *

#8858 Sigma Pump, Model T-6, pulsating rubber tube type pump; capacity ½ gallon per minute; driven at 280 RPM through pulleys by ¼ horsepower motor #8254; inlet from acid carboys outside of building; outlet connected to Saran tubing to neutralizer #6220. Bypass on pump so that acid can be fed to neutralizer by vacuum without the use of pump. Used to feed last portion of muriatic acid at a slow rate to neutralizer #6220.

#8449 Westco Turbine Pump; bronze; capacity 25 gallons per minute driven by 1½ horsepower motor #8250. Used to pump slurry from neutralizer #6220 to filter press #4653.

#4653 Shriver Filter Press - 24"; cast iron head; 17 - 1" thick bronze frames; bronze plates; through washing; feed inlet connected to Westco pump #8449, wash port connected to isopropyl alcohol tank #8565; filtrate ports connected to 1½" pipe line to extraction tank #6763 and to return line to

neutralizer #6220; air connection for blowing press. Number 8 Jabeeco cotton duck filter cloth is used as the filter medium. This press will be replaced with an all bronze press. Used to remove sodium chloride from neutralizer slurry.

#8565 Steel Tank; capacity 75 gallons, $23\frac{1}{2}$ " inside diameter x 38" straight height; top provided with $1\frac{1}{4}$ " charging line, 1" coarse vacuum and vent connection; level gage on side equipped with spring closing valve, 1" bottom outlet connected to wash port of filter press #4653 and also to suction line of filter press #4653. Used as a storage tank for isopropyl alcohol.

#6763 Lead Lined Extraction Tank; capacity 1130 gallons; 36" diameter 10 bladed hard lead turbine type agitator driven at 71 RPM through Nettco speed reducer #6770 by $7\frac{1}{2}$ HP motor #8538; thermometer well for 0 to 150°C Motoco thermometer; top provided with $1\frac{1}{4}$ " inlet from filter press, 1" water line connected to water meter for either fresh or recovered water, $1\frac{1}{2}$ " vent, manhole cover with 6" charging hole, $1\frac{1}{4}$ " feed connection from steel tank #6235; monel decanter arm mounted in side of tank, 2" bottom outlet connected to $1\frac{1}{4}$ " drain cock, $1\frac{1}{4}$ " drum line and $1\frac{1}{4}$ " brass line to steel neutralizer #6219. Used to dilute glycol filtrate from press #4653 and to extract the trichlorophenol from the dilute glycol by means of benzol.

#6235 Steel Tank; capacity 250 gallons, $35\frac{1}{2}$ " inside diameter x 56" straight side; $1\frac{1}{4}$ " top connection from Westco pump #8448, $1\frac{1}{2}$ " overflow line to underground tank #8600, level

In place of #8528

#9176 500 gal. Monel metal Still; turbine agitator,
Monel heating and cooling coil, the latter equipped with
safety valve set at 50 lb. pressure.

gage glass on side with spring closing valve, $1\frac{1}{2}$ " outlet on bottom connected to extraction tank #6763. Used to measure benzol which is added to extraction tank #6763.

#8528 Copper Agitated Still; capacity 350 gallons, copper heating and cooling coil with 13.4 sq. ft. of surface; 20" diameter 8 bladed bronze Nettco turbine agitator driven at 142 RPM through Nettco Speed reducer #5205 by 3 horsepower motor #8518; top equipped with gooseneck connection to condenser #5713, safety valve set for 10 pounds per sq. inch, 6" hand hole opening, thermometer well for 0 to 200°C Motoco temperature indicator, drum charging line, vacuum connection, suction line to decanter of lead lined extraction tank #6763; bottom drain to drum or sewer. Used to receive extracts from extractor #6763, to wash benzol extract and to remove benzol from the crude trichlorophenol.

#5713 Copper Coil Condenser; 38 sq. ft. of cooling surface; inlet from gooseneck of copper still #8528; discharge to vacuum receiver #8526 and to underground tank #8600. Used to condense vapors from copper still #8528.

#8526 Steel Receiver; capacity 25 gallons; $17\frac{1}{2}$ " inside diameter by 24" straight side; connections on top to condenser #5713 and to coarse vacuum and vent line; level gage glass on side; bottom connection to drain valve and to underground tank #8600. Used to receive condensate from condenser #5713.

#8600 Underground Tank; capacity 750 gallons; 48" inside diameter by 96" straight side; in horizontal position; top connections for vent, fill line to surface, fill line from

condenser #5713, overflow line from tank #6235, suction line to Westco pump #8448, gage connection to remote level indicator #8647; 18" manhole; located on south side of building 60. Used to receive dry benzol condensate from condenser #5713.

#8647 Uehling Tank-O-Meter; scales 2 to 48" and 10 to 750 gallons; equipped with hand air pump. Used to measure amount of benzol in underground tank #8600.

#8448 Westco Turbine Pump; all bronze; capacity 25 gallons per minute; driven at 1750 RPM by one horsepower motor #8540. Used to pump benzol from underground tank #8600 to measuring tank #6235.

#6219 Steel Neutralizer; capacity 500 gallons; 53-3/8" inside diameter, straight length - 48"; 18" diameter 6 bladed cast iron Nettco turbine agitator driven at 90 RPM through speed reducer #8883 by 2 horsepower motor #8981; top connections for fill line from bottom of extractor #6763, drum charging line, vacuum and vent lines; bottom drain to drum, sewer and outside storage tanks #5640, 5641, 5642 and 9029. Used to neutralize dilute ethylene glycol from extraction tank #6763.

#5640, 5641, 5642 and 9029 Storage Tanks; steel; fuel oil type; each 275 gallons; 60" length by 44" height by 27" width; located outside of building 60 on North side; top of all tanks connected to common header which in turn is connected to vent; bottom of each tank connected to fill line from neutralizer #6219 and to suction line of Westco transfer

pump #8500. Used to store dilute ethylene glycol from steel neutralizer #6219.

#8500 Westco Turbine Pump; all bronze; capacity 28 GPM; driven at 1750 RPM by one horsepower motor #8980; suction connected to bottom of storage tanks #5640, 5641, 5642 and 9029, and to bottom of neutralizer #6219; discharge connected to overhead pipe line to glycol recovery still #8728 in building 54. Used to pump weak ethylene glycol from storage tanks to ethylene glycol recovery still #8728.

SECTION IV

MATERIAL AND LABOR REQUIREMENTS

Raw Materials

Hydrolysis

Ethylene Glycol - fresh or recovered	1,380 pounds
Tetrachlorbenzene	600 "
Caustic Soda Flakes	312 "

Neutralization

31% Muriatic Acid	approx.	575	"
99% Isopropyl Alcohol (or recovered)	"	170	"

Extraction and Washing

Benzol - fresh or recovered	1,500 pounds		
Muriatic Acid	approx.	25	"

Neutralization of Extracted Dilute Ethylene Glycol

Soda Ash	approx.	30 pounds
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Labor Requirements

The department has been operated with one first class operator, two second class operators and ~~four~~³ chemical laborers for the three shifts. It is recommended that all the operators be at least second class operators.

On the day shift, there are ~~three~~² operators. One in building 54, another in building 60 and the third who is the first class operator works in both buildings. During the second and third shifts there is an operator in each building. Thus a total of ~~seven~~⁶ operators are required for twenty four hour

operation.

One hydrolysis batch is made on each shift so that 3 batches are made every day.

The recovered isopropyl alcohol is rectified in another department.

SECTION V

OPERATING PROCEDURE

Hydrolysis:

Hydrolysis kettle #8454 is charged with 1,380 lbs. of either fresh or recovered ethylene glycol with vacuum. The cock on the steam heated vent line is opened and the steam to the vent line and copper tubing around the pipe connections on top of the kettle is turned on. The agitator is started and 312 lbs. of caustic soda flakes which have been previously weighed into an old tetrachlorbenzene fiber drum are charged by means of the elevator and air hoist. Then 600 lbs. of tetrachlorbenzene, which has been loosened by beating the fiber containers with a short section of $1\frac{1}{2}$ " lead lined pipe, are charged. The hand hole cover is bolted down and the gas flame is turned on full. When the batch temperature reaches 140°C , the cock in the steam heated vent line is closed. At 165°C , the gas flame is turned off completely, and at 172°C the cooling water to the cooling coil is turned on full. As soon as the temperature levels off at 175 to 176°C , the cooling water is turned off. The temperature is maintained for four hours at 175°C . A quarter inch flame on all the burners is sufficient to maintain 175°C . A pressure of approximately 20 pounds per sq. inch is obtained at 175°C and in view of the fact that the safety valve on the kettle is set for 25 pounds, this temperature should not be allowed to rise more than a few degrees above 175°C at any time. At the end of the four hour period and with the gas still on, the pressure

within the hydrolyzer is slowly vented into condensate tank #4619. The latter is cooled by circulating water in the jacket during this operation. Venting must not be done too fast since some hot vapors may go past the cooling surface and be lost from the condensate tank through the roof vent. When the pressure within the hydrolyzer has dropped to 0, the temperature of the batch is quickly brought to 185°C and kept here ten minutes, then the batch is ready to be cooled to 105°C. If the safety valve does open at any time, the vent line from the safety valve should be inspected before the next batch is started in order to insure that no blockage has occurred. In addition, this vent line should be inspected at least once a month as there is always the possibility that the safety valve has leaked slightly and has clogged the vent line. After the cock in the vent line is closed, the temperature must be watched very closely as a heat of reaction develops in the vicinity of 150 to 160°C. At the end of the heating period the batch is cooled to approximately 105°C (not below 95°C as the batch thickens if the temperature is too low) by means of cooling water to the coil. About 10 minutes before the batch is to be blown to Building 60, the plug is removed from the cross on the bottom discharge connection of the hydrolysis kettle, and the nickel cock and pipe connections at the bottom are heated with the gas torch for about 10 minutes. The plug is replaced and the batch is blown to Building 60 by means of air.

The contents of the vent tank #4619 are removed when

the batch has been blown out of the hydrolyzer. This condensate is in two layers, a lower layer containing a small amount of glycol and some dissolved halogenated benzenes and an upper oily layer consisting of liquid by-products. These are drummed up for disposal.

Neutralization:

Before the batch is blown over from building 54, the three bottom valves on neutralizer #6220 are closed and the agitator and cooling water to the jacket are turned on. After the batch has been blown, the level in the neutralizer is measured and recorded. The distance between the bottom of manhole and the top surface of the batch should be 13 inches. When the temperature of the batch falls to 75°C, neutralization of the batch is started by adding approximately 550 pounds of muriatic acid from carboys by means of vacuum. The cooling water to the jacket is used during this neutralization. If the temperature of the batch is allowed to fall to about 55°C before the acid addition is started, caking will occur around the sides and the batch will thicken so that the acid will not mix and there will be danger of the free acid corroding the top of the stainless steel neutralizer. The hand hole cover is removed and the batch is checked with congo red paper. The batch should still be alkaline. Additional muriatic acid is added at a slow rate by means of the Sigma pump #8858 until the batch is acid to blue litmus paper but not to congo red paper. A total of approximately 575 pounds of acid is required. The cooling water is turned off when the batch has cooled to 25°C.

Filtration:

While the batch is being cooled in the neutralizer, the lead lined extraction tank #6763 is charged with 50 gallons of recovered water from building 54.

As soon as the temperature of the slurry in the neutralizer has fallen to 25°C, filtration is started. During the operation of the filter press, a plastic cover is placed over the press in order to guard against any spurts of liquor from the press. In addition, the operator must wear a face shield when working around the press. The slurry is filtered in filter press #4653 set up with 17 frames. The filtrate is returned to the neutralizer until it runs clear and then it is switched over to the lead lined extraction tank #6763 after the 36" diameter agitator has been started. Filtration of the batch requires approximately 30 minutes. When the neutralizer is empty, the pipe lines and pump are flushed with two pails of drip pan isopropyl alcohol drainings obtained from the previous filtration. (If none is available, a pail of isopropyl alcohol is used.)

The filter press is then blown with air for 2 minutes into the lead lined extractor. The press is washed by gravity with either fresh or recovered isopropyl alcohol from wall tank #8565. When the alcohol appears in the sight glass in the filtrate line, the wash valve is closed and the press is allowed to soak for 5 minutes. The press is then blown for 5 minutes into the extractor. Approximately 6 inches of isopropyl alcohol (75 pounds) measured in steel tank #8565 are required for the wash.

The damp salt cake which weighs approximately 300 pounds is removed from the press and is discarded. (The press is dumped while the batch is settling in the extractor. After cleaning, the filter cloths must be thoroughly scrubbed with a brush and warm water.)

Extraction:

Muriatic acid is added to the batch in lead lined extractor #6763 until it turns Congo Red paper blue or very black. Usually 15 to 20 pounds of 31% muriatic acid are required. The agitator in the extractor is turned off and 25 inches of benzol (770 pounds) measured in wall tank #6235 are added. The batch is agitated for 2 minutes and allowed to settle for twenty five minutes.

The benzol layer at the top is decanted by applying a vacuum no greater than 10 inches to the empty Monel still #9176 and opening the valves in the decanter line. The decanter arm is lowered as far as possible in the extractor so that no glycol appears in the sight glass. The valve under the sight glass is opened full and the flow in the decantation line is controlled by means of the valve above the sight glass. After the first benzol extract has been removed, 12 inches of benzol (365 pounds) are again dropped into the extractor. After stirring for 2 minutes, the batch is allowed to settle for twenty five minutes. The second extraction is removed as before and then the last 12 inches of benzol are added. After stirring for 2 minutes, the batch is allowed to settle for twenty five minutes and the last extraction is decanted.

Washing of Benzol Extract:

The vacuum on Monel still #9176 is released and any glycol on the bottom is drained and returned to lead extractor #6763. The level (i.e. the distance between the bottom of the hand hole and the top surface of the benzol extract) is approximately 10 inches. The agitator is turned on for one minute in order to mix the extracts. After the agitator is stopped, thirty gallons of fresh water measured by means of the water meter and one gallon of muriatic acid mixed with 2 gallons of water in a copper pail are added to the copper still and the batch is agitated for three minutes. After settling for twenty five minutes, the water wash on the bottom is drained to the sewer. Any muck is saved for separation in a bottle. The batch is washed a second and third time in the same manner using 30 gallons of water each time without any acid. All the clear water washes are also sent to the sewer.

Small amounts of glycol are removed by these washes and it is very important that the washing be efficient, otherwise the congealing point of the distilled trichlorophenol will be low. If the still were larger, the amount of wash water could be increased, and two washes would suffice.

Removal of Benzol:

The valves on the copper still are set for atmospheric distillation into receiver #8526. The agitator is started and the steam is turned on slowly until the benzol begins to distill (approximately 70°C still temperature). The steam

is then increased in order to distill the benzol as quickly as possible without boiling over the batch. When receiver #8526 is almost full, the distillate is switched to the underground tank. (The amount of benzol already in the underground tank should be noted on the log sheet, so that the total amount of benzol recovered from each batch can be determined. This is approximately 195 gallons.) The water is drained from receiver #8526 and is discarded. The benzol in this receiver is run into the underground tank. When the steam pressure has increased to 50 pounds (still temperature approximately 125°C) and the rate of distillation has slowed down, the distillate is switched back to receiver #8526 and vacuum is applied slowly until full vacuum (20 to 25 inches) is attained. Ten minutes after the benzol has stopped distilling at full vacuum and at a still temperature of at least 125°C, the vacuum and steam are turned off and the batch is cooled to 90°C with cooling water. The steam to the copper coil around the bottom discharge pipe is turned on in order to prevent the phenol congealing in the pipe line when it is drained. The crude is drained into a tared galvanized or tin lined drum and weighed. (A 4 oz. sample of the crude is obtained for the laboratory.) The average weight of crude is approximately 575 pounds.

The drum of crude is placed in building 68 hot box in order to keep it in a molten state.

Distillation of the Crude Trichlorophenol:

Three batches of crude are charged to the monel still #8867 by means of vacuum. (The operator must wear a face shield

and gloves whenever molten trichlorophenol is being handled.) The steam to all the steam traced lines and to the hot water circulation tank is turned on. The hot water pump is started so that water at 70°C is circulated through the jackets of the Monel receivers, through the condenser and through the dephlegmator coil. The flow of water to the dephlegmator is adjusted to 5 gallons per minute as indicated by the rotameter. The three gas burners under the still are turned on full and cooling water to the vacuum traps is also turned on. The valves are set so that the first fraction distills into Monel receiver #8884 under coarse vacuum. Distillation usually starts when the still temperature is approximately 160°C and the vacuum is 20 mm. At this time the two inner burners are turned off and the outer burner is reduced to about a 5 inch flame. The steam supply to the hot water tank is reduced. After the first receiver is half filled, the distillate is switched to the other Monel receiver #8885 and the vacuum is changed from coarse to medium. All gas flames are turned off in the room and the benzol in the vacuum trap of the first Monel receiver #8884 is drained into a pail and is transferred to a drum outside the building. A pail is kept under the trap and the trap cooling water is turned off and the steam to the jacket of the trap is turned on to ten pounds. The bottom drain valve is rammed with a bent rod to insure all the benzol has been drained and then the gas flames in the room are ignited again. After the trap of the first receiver has been heated for ten minutes, the drain valve is closed, the steam turned off and the cooling water is started

through the jacket as before. The trichlorophenol in the first receiver which amounts to approximately 140 pounds is run directly into the light fraction drum. Distillation is continued at 5 to 8 mm. until the level of the molten trichlorophenol in the second Monel receiver #8885 can be seen in the level gage glass. The distillate is switched back to the first receiver and after one third of a pail of trichlorophenol is withdrawn from the second receiver, a 2 oz. sample is obtained. The congealing point and solubility of a few drops in 5% NaOH are determined on it. If the congealing point is 63.5°C or higher and it is clearly soluble in NaOH, the contents of the pail and receiver are run into the "B" drum. This drum should have a good galvanized or tin lining. If the congealing point is below 63.5°C, the sample generally is cloudy in the NaOH solution and the trichlorophenol is run into the light fraction drum again. The above procedure is repeated until the "B" cut is obtained. At this point the flow of hot water to the dephlegmator coil is reduced to one gallon per minute. During the "B" cut the receivers are filled to within one inch of the top of the level gage glasses, and the contents are run directly into the "B" drum. The oil bath temperature is between 250 and 270°C during this period.

After the second receiver has been drained, the cooling water to the corresponding trap is replaced with steam and the trap is allowed to drain into a pail. Usually it is necessary to preheat the drain valve with a steam hose before draining. After ten minutes, the steam flow is stopped and the cooling water is started again. Each time a receiver is drained,

the corresponding trap is heated. However, it is not necessary to drain the traps after the first two times as only a small amount of material collects in the bottom of the traps.

Near the end of the distillation, the distillation rate begins to slow down and the still temperature rises. From now on the receivers are filled only to the bottom of the level gage glass and the congealing point and solubility are determined each time the receiver is drained. As soon as the congealing point falls below 63.5°C , the trichlorophenol is drained into the "heavy fraction" drum. It may happen at this stage that the congealing point is low and the solubility is clear, but good trichlorophenol meets both criteria. Approximately 100 pounds of this fraction are usually obtained. The still temperature is not allowed to rise above 210°C , otherwise decomposition occurs in the still and the residue becomes hard and can be removed only by chopping and scraping. When the distillation is completed, the flames, hot water and steam supplies are turned off and the vacuum is released slowly. When the temperature in the still falls to 135°C , the residue is withdrawn by means of a suction drum. The residue which contains the monochlorophenyl ether of diethylene glycol is delivered into stock at no value. It must be remembered that after repeated heating the oil in the still jacket will polymerize and carbonize. This occurrence is usually indicated by an increasing temperature lag between both temperature and still temperature. Good bath oil generally shows a temperature lag of $60-65^{\circ}$; when the lag exceeds 110° , the condition of the bath oil should be checked and the oil renewed.

The vacuum traps are heated by means of steam and are completely drained.

The following data are obtained from a typical distillation:

Charge: 3 Batches of Crude - 1,754 lbs. total.

TIME	TEMPERATURE			Circulating Water	VACUUM MM HG	DEPHLEGMATOR		PRODUCT		RE-CEIVER #	REMARKS
	Still OC	Col- umn OC	Dis- til- late OC			Oil Jack- et OC	Rota- meter gal/ min.	in- let water OC	out- let water OC		
5:30											
6:30	120	90	82	250	20	4.5	62	62		8884	Still char- ged. Burners on full.
7:30	156	96	76	250	18	4.5	72	84	Light	8885	Distilling
8:30	158	130	82	265	12	4.5	66	95	Light 57.0	8885	
9:30	158	128	84	250	12	4.5	76	100	Light 61.0	8885	
10:30	150	130	88	275	12	1.0	80	100	B 62.0	8885	Switch Re- ceivers
11:30	150	130	84	275	12	1.0	80	100	B 62.6	8884	
12:30	150	132	84	275	12	1.0	78	100	B	8885	
1:30	150	128	82	275	5	1.0	78	100	B 64.2	8884	
2:30	156	132	88	275	5	1.0	70	100	B	8885	
3:30	158	132	84	280	6	1.0	70	100	B 62.3	8884	
4:30	184	130	98	285	9	1.0	80	100	Heavy 60.5	8885	Heat off.
5:30	198	130	98	275	12	1.0	82	98	Heavy	8885	

Light Fraction - 348 lbs. 19.8% of charge
 B - 1,118 lbs. 63.5% "
 Heavy Fraction - 110 lbs. 6.3%
 Residue - 115 lbs. 6.5%
 1,691 lbs. 96.1%

Process for 2,4,5 Trichlorophenol

When sufficient quantities of light and heavy fractions have been obtained, they are distilled separately. Upon repeated distillation of the light fractions, a very light fraction is obtained which does not congeal at room temperature. This fraction which contains a large amount of 1,2,4 trichlorobenzene is delivered into stock at no inventory value.

Neutralization of Weak Ethylene Glycol:

Twenty pounds of solid soda ash are added to the empty steel neutralizer #6219. The glycol remaining in the lead lined extraction tank #6763 is transferred to the steel neutralizer by applying vacuum to the neutralizer. The sight glass in the transfer line is observed and as soon as the benzol layer appears, the flow is stopped by turning off the two valves under the extraction tank and releasing the vacuum in the neutralizer. The benzol in the pipe line between the bottom valve on the extraction tank and the valve next to the sight glass is drained into a pail and is returned to the extraction tank. The small amount of benzol in the extraction tank is allowed to remain there for processing with the next batch.

The agitator in the neutralizer is started and the glycol is neutralized by adding solid soda ash to a pH between 6 and 7 as determined by means of Hydrion paper. Approximately 10 pounds of soda ash are usually required. The level in the neutralizer at this time is 33" below the bottom of the hand hole (equivalent to 260 gallons).

The neutralized glycol is allowed to flow by gravity from the neutralizer into the four outside storage tanks #5640, 5641, 5642 & 9029.

Distillation of Weak Ethylene Glycol:

The glycol recovery still #8728 in building 54 is charged with a 28" level (distance between bottom of hand hole and top surface of charge) of weak ethylene glycol (400 gallons) from the outside storage tanks 5640, 5641, 5642 & 9029 by means of Westco transfer pump #8500 located in building 60. The starting switch for this pump is located near the still. Before pumping, the hand hole cover on the still is removed and the still is connected to the coarse vacuum pump #8656 so that no benzol fumes which might be present in the weak glycol will enter the room. After obtaining the exact level, the still is closed, the steam is turned on full and the valves are adjusted so as to distill into receiver #8670 under coarse vacuum from Nash pump #8656. The air bleed on the pump is opened wide so that a vacuum of 22 inches of mercury is obtained at the still. The cooling water is turned on full and the reflux regulator is set for total reflux. Distillation begins when the still temperature is approximately 75°C. The first fraction consists of weak isopropyl alcohol. Although the regulator is set for total reflux, product is obtained as only partial condensation occurs in the water tube condenser and the liquid cooler serves as both a condenser and cooler during the alcohol cut. The steam pressure maintained on the jacket of the still is limited by the cooling water supply. With the cooling water on full enough steam is used so that the distillate temperature is about 35°C. After 50 gallons of distillate have been obtained, all the alcohol has distilled and the water cut is obtained. The weak isopropyl alcohol (approximately 25 to 30% alcohol) is drained into a grounded drum outside the

building. During this period the reflux regulator is set for 1 to 1 reflux and the air bleed on the Nash pump is closed. The water cut is distilled first into #8671 receiver up to the 24" mark on the level gage glass (105 gallons) and then into #8670. When the distillation rate slows down, the vacuum is switched from the Nash pump to the tower coarse vacuum. The water in receiver #8671 is run to the sewer and the water obtained in the second receiver is saved in the recovered water tank #8599. At the end of the water cut the column temperature rises sharply. When it reaches 100°C and the vacuum is approximately 20 mm of mercury, the distillate which is now glycol is switched to empty receiver #8671 and the vacuum is changed from coarse to medium. The reflux regulator is set on zero and the valve in the reflux line is closed. At this time the cooling water is also reduced so as to maintain a 35°C distillate. Distillation is continued until the distillate slows down to a slow drip and the still temperature is above 150°C. The vacuum is released and any fluid residue is drained into a drum. (This residue contains about 35% glycol and is distilled in the same still when a sufficient quantity is available.) In general no fluid residue is obtained. The hot still is immediately washed with cold water from a 1½" hose using a full stream to dislodge the salts in the bottom of the still. This cleaning which usually takes forty five minutes must be very thorough or the following distillation will require more than ten hours.

Fifteen hundred to 1,700 pounds of glycol are obtained from each batch. Two batches can be run every 24 hours and in view of the fact that one still batch is equivalent to slightly more than 1½ batches of weak glycol, there is

sufficient capacity if no interruptions are caused by poor vacuum or cooling water supply.

The following data were obtained from a typical distillation:

Charge - 28" level (400 gals.)

TIME	TEMPERATURE		VACUUM		REFLUX	STEAM Pressure lbs/sq. in.	PRODUCT	RECEIVER		REMARKS
	Still Col-umn	Distil-late	Top of Col-umn	At Receiver				#	Ht. Glass in.	
	°C	°C	MM Hg	Gage Hg.						
8:50										Still charged & started.
9:05	75			24		20	Weak IPA	8671	0	Distilling
9:35	98	40		24	total	20		8671	7	
9:45	104	40		26	1:1	40	Water	8671	8	
11:15	106	80		26	1:1	70	Water	8671	-	
12:05	130	70		29	1:1	80	Water	8671	24	
12:45	134	100	28	30	0	90	Glycol	8671	7 1/2	Glycol Dis-tilling.
1:40	134	112	28	30	0	90	Glycol	8670	8	
2:50	132	112	18	30	0	90	Glycol	8670	22	
3:30	130	110	12	30	0	90	Glycol	8670	30	
4:20	130	112	10	30	0	90	Glycol	8671	0	Switch Rec.
5:20	140	114	10	30	0	90	Glycol	8671	2	
6:20	150	122	10	30	0	90	Glycol	8671	3 1/2	
6:50	152	122	10	30	0	90	Glycol	8671	4 1/2	Heat off - Still Washed.

Weak IPA - 387 lbs. - (27% IPA)
 Ethylene Glycol - 1,525 lbs.
 No Fluid Residue

Process for 2,4,5 Trichlorophenol

The following schedule lists the operations in building 54.

Hydrolysis

Equipment #8454

<u>Time</u>	<u>Operation</u>
7:30 A.M. - Batch C	Charge ethylene glycol, caustic soda flakes and tetrachlorbenzene.
8:15 A.M.	Heat on.
9:05 A.M.	Vent closed.
9:35 A.M.	At temperature.
1:35 P.M.	Begin to cool.
2:05 P.M.	Begin to blow batch to Bldg. 60.
2:30 P.M.	Batch blown.

Glycol Distillation

Equipment #8728

<u>Time</u>	<u>Operation</u>
8:20 A.M.	Charge 600 gallon still.
8:50 A.M.	Steam on.
9:05 A.M.	Distillation begins - alcohol cut.
9:45 A.M.	Water cut begins.
12:45 P.M.	Glycol begins to distill.
6:50 P.M.	Heat off - Drain.
6:55 P.M.	Washing still.
7:55 P.M.	Still clean - ready for charging.

Distillation of Crude Trichlorphenol

Equipment #8868

<u>Time</u>	<u>Operation</u>
9:00 A.M.	Charging still.
9:30 A.M.	Heat on.
11:30 A.M.	Distillation begins.
9:30 P.M.	Heat off.
1:30 A.M.	Unloading residue

The following schedule lists the operations in building 60.

Neutralization

Equipment #6220

<u>Time</u>	<u>Operation</u>
8:00 A.M. - Batch B	Cooling batch to 25°C after neutralization.
8:45 A.M.	Start to filter.
9:15 A.M.	Neutralizer empty.
2:30 P.M. - Batch C	Next batch received from Bldg. 54.
3:00 P.M.	Start to neutralize after cooling.
4:00 P.M.	Neutralization completed - cooling to 25°C.

Filtration

Equipment #4653

<u>Time</u>	<u>Operation</u>
8:00 A.M. - Batch B	Add 50 gallons water to lead extractor.
8:45 A.M.	Start to filter.
9:15 A.M.	Filtration finished - blow press.
9:20 A.M.	First isopropyl alcohol wash on.
9:25 A.M.	Blow press.
9:50 A.M.	Clean press.

Extraction

Equipment #6763

<u>Time</u>	<u>Operation</u>
9:30 A.M. - Batch B	Making batch acid to Congo.
9:40 A.M.	25" Benzol added to extractor.
9:45 A.M.	Agitate and let settle for 25 minutes.
10:10 A.M.	Taking off extraction.
10:30 A.M.	First extraction off - second on.
10:35 A.M.	Settle for 25 minutes.
11:00 A.M.	Taking off second extraction.
11:05 A.M.	Second extraction off - third on.
11:10 A.M.	Settle for 25 minutes.
11:35 A.M.	Taking off last extraction.
11:40 A.M.	Last extraction off.
12:00 A.M.	Transfer weak glycol from lead extractor to steel neutralizer by vacuum.
12:15 P.M.	Extractor empty.

Neutralization of Weak
Ethylene Glycol

Equipment #6219

<u>Time</u>	<u>Operation</u>
11:55 A.M. - Batch B	Add soda ash to steel neutralizer.
12:00 A.M.	Start to receive weak glycol from lead extractor.
12:15 P.M.	Weak glycol in neutralizer.
12:30 P.M.	Neutralize glycol.
12:35 P.M.	Drain to outside storage tanks by gravity.
12:55 P.M.	Empty.

Benzol Extract Washing
and Distillation

Equipment #8528

<u>Time</u>	<u>Operation</u>
6:05 A.M. - Batch A	Benzol Starting to distill.
9:30 A.M.	All benzol off.
9:45 A.M.	Cooled to 90° and drained.
10:10 A.M. - Batch B	Begin to receive extractions.
11:45 A.M.	Drain glycol from bottom of copper still.
11:50 A.M.	Add first acid wash and agitate.
11:55 A.M.	Settle for 25 minutes.
12:20 P.M.	Draining first wash.
12:25 P.M.	Add second wash and agitate.
12:30 P.M.	Settle for 25 minutes.
12:55 P.M.	Draining second wash.
1:00 P.M.	Add third wash and agitate.
1:05 P.M.	Settle for 25 minutes.
1:30 P.M.	Drain last wash.
1:35 P.M.	Steam on still.
2:05 P.M.	Starting to distill.
5:30 P.M.	All benzol off.
5:45 P.M.	Cooled to 90°C - Draining.

The hydrolysis schedule in building 54 and the schedules in building 60 are repeated for each shift. During the week the schedule advances several hours so that the stills are finished and drained by 7:30 A.M. Saturday morning.

SECTION VI

HAZARDS OF PROCESS

Health

Caustic Soda Flakes:

Long rubber gloves and an eye shield must be worn by the operator when handling caustic soda flakes, as they produce painful burns upon coming in contact with the skin.

Muriatic Acid:

Long rubber gloves and an eye shield must be worn by the operator when handling muriatic acid.

Benzol:

When inhaled benzol acts as a nerve poison causing faintness and cyanosis. Benzol is also absorbed through the skin and produces the same symptoms as an inhalation. It can also produce dermatitis through the removal of the natural oils from the skin tissues. Benzol is also a cumulative poison and therefore, any small leaks from pipes or equipment must be taken care of immediately.

Trichlorophenol:

Molten trichlorophenol produces burns in contact with the skin, although the burns are slight compared with those produced by phenol and parachlorophenol. Nevertheless, long rubber gloves and a face shield must be worn whenever trichlorophenol is handled. Any spillages on the skin should be removed mechanically as much as possible and then the area should be bathe with 50% isopropyl alcohol and then thoroughly washed with soap and water.

Fire

Benzol has a low flash point of -11°C and precautions must be taken that no sources of ignition exist when benzol vapors are present. Whenever benzol is being transferred from or to a drum, the ground clamp and wire must be used.

The flash point of isopropyl alcohol is $+12^{\circ}\text{C}$ and therefore presents a fire hazard. The same precautions as indicated above for benzol should be taken.

Ethylene glycol has a flash point of 115°C and therefore presents a fire hazard at elevated temperatures.

SECTION VII

PRODUCTS AND BY-PRODUCTS

Trichlorophenol:

Congealing Point: 63.5°
 ~~-62.0°~~ C minimum

Light Transmission: 90% minimum
(50% solution by
volume in 2B alcohol)

Delivered into stock in 55 gallon galvanized or tin
lined drums containing approximately 575 pounds.

Residue:

Delivered into stock at no inventory value in 75 gallon
black iron drums containing approximately 1,000 pounds.
This residue contains about 50% of the monochlorophenyl
ether of diethylene glycol.

SECTION VIII

COST DATA

Materials consumed per 100 pounds of Trichlorphenol

Distilled produced:

Tetrachlorbenzene	135 pounds
Caustic Soda Flakes	71 "
Ethylene Glycol	90 "
Muriatic Acid 31%	137 "
Isopropyl Alcohol 99% (with recovery)	25 "
Benzol	20 "
Soda Ash	6.8 "

Labor required

To operate three shifts, $\frac{6}{7}$ men are required. One hydrolysis batch is made per shift and with a yield of 74%, 444 pounds are obtained from each batch,

therefore $\frac{7 \times 8}{3 \times 444/100} = 4.2$ man hours per 100 pounds of
Trichlorphenol Distilled
(no recovery of IPA)

Allowing 10% more for night supervision, repairs, cleaning, and rectification of recovered isopropyl alcohol, a labor requirement of 4.6 man hours per 100 pounds of trichlorphenol is obtained.

SECTION IX

LITERATURE AND RESEARCH SOURCES

Conversion of 1,2,4,5 Tetrachlorbenzene into 2,4,5 Trichlorophenol by W. S. Gump, Dec. 29, 1945.

2,4,5 Trichlorophenol Process by H. G. Krebs, April 17, 1943.

2,4,5 Trichlorophenol (Ethylene Glycol Process) by H. G. Krebs, Dec. 12, 1947

Trichlorophenol File in R.L. 3.

The Toxicity and Potential Dangers of Ethylene Glycol by Wiley, Hueper and vonOettingen, Journal of Industrial Hygiene and Toxicology, Feb. 1936.

Dow Glycols - Published in 1947 by the Dow Chemical Co.

Crosby-Fiske-Forster Handbook of Fire Protection.

Poisons, Their Properties, Chemical Identification, Symptoms and Emergency Treatments by J. Brookes and H. N. Alyea.

SECTION X

DRAWINGS

Tracings of the following drawings pertaining to the process are on file in the Engineering Department:

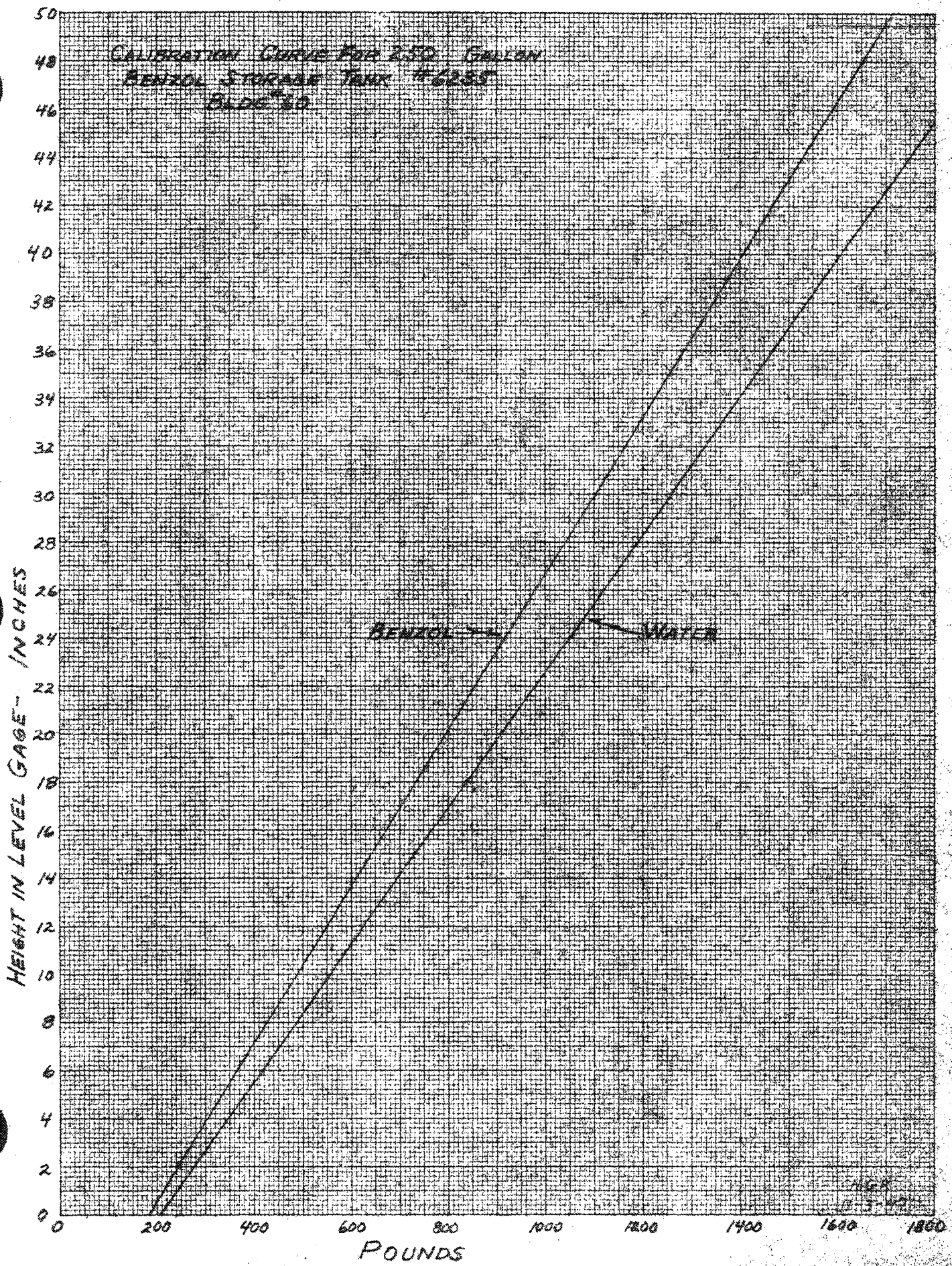
<u>Description</u>	Equipment No.	Drawing No.
500 Gallon Steel Neutralizer	6219	B-3072
750 Gallon Steel Recovered Water Storage Tank	8599	C-2861
750 Gallon Steel Recovered Benzol Storage Tank	8600	C-2861
150 Gallon Steel Receivers	8670 & 8671	C-2890
18" Monel Column for Monel Still	----	B-2912
185 Gallon Monel High Vacuum Still	8868	2899
25 Gallon Vacuum Receiver	8526	C-2846
Gooseneck for Monel High Vacuum Still	----	C-3008
600 Gallon Steel Recovery Still	8728	B-2862
Stainless Steel Pan for Filter Press #4653	5306	C-2948
Location of Outlets on Water Tube Steel Condenser	8767	C-2971
Reflux Preheater for 600 Gallon Still	8753	D-2895
1,130 Gallon Lead Lined Extraction Tank	6763	2256
1,130 Gallon Lead Lined Extraction Tank Coil	----	B-2850
Circulating Water Tank for Monel Still	----	C-2918
Liquid Cooler for Glycol Still	----	B-2897
Instrument Panel for 600 Gallon Still	----	2900
30" Column for Glycol Still	8787	B-2865
Detail of Flanges for 30" Column	----	C-2866
Monel Coil Condenser for Monel Still	8824	2061
25 Gallon Monel Receivers	8884 & 8885	C-2909
250 Gallon Stainless Steel Neutralizer	6220	2856
Gas Burner Support for Monel Still	----	C-2934
250 Gallon Hydrolysis Kettle	8454	B-2733-1

250 Gallon Hydrolysis Kettle Details	----	A-2738-2
350 Gallon Copper Still	8528	B-2852
Assembly of Sigmamotor Pump and Motor	8858	A-3017
75 Gallon Alcohol Tank	8565	2028-A
Installation of Neutralizer #6219, Pump #8500 and 4 - 275 Gallon Steel Storage Tanks	----	A-3078
250 Gallon Benzol Feed Tank	6235	2255
Decanter on Lead Lined Extractor	----	2414
T.C.P. Process Flow Sheet	----	C-2812
2,4,5 T.C.P. Process Bldg. #54 West Wall	----	2839
2,4,5 T.C.P. Process Bldg. #60 West Wall	----	2843

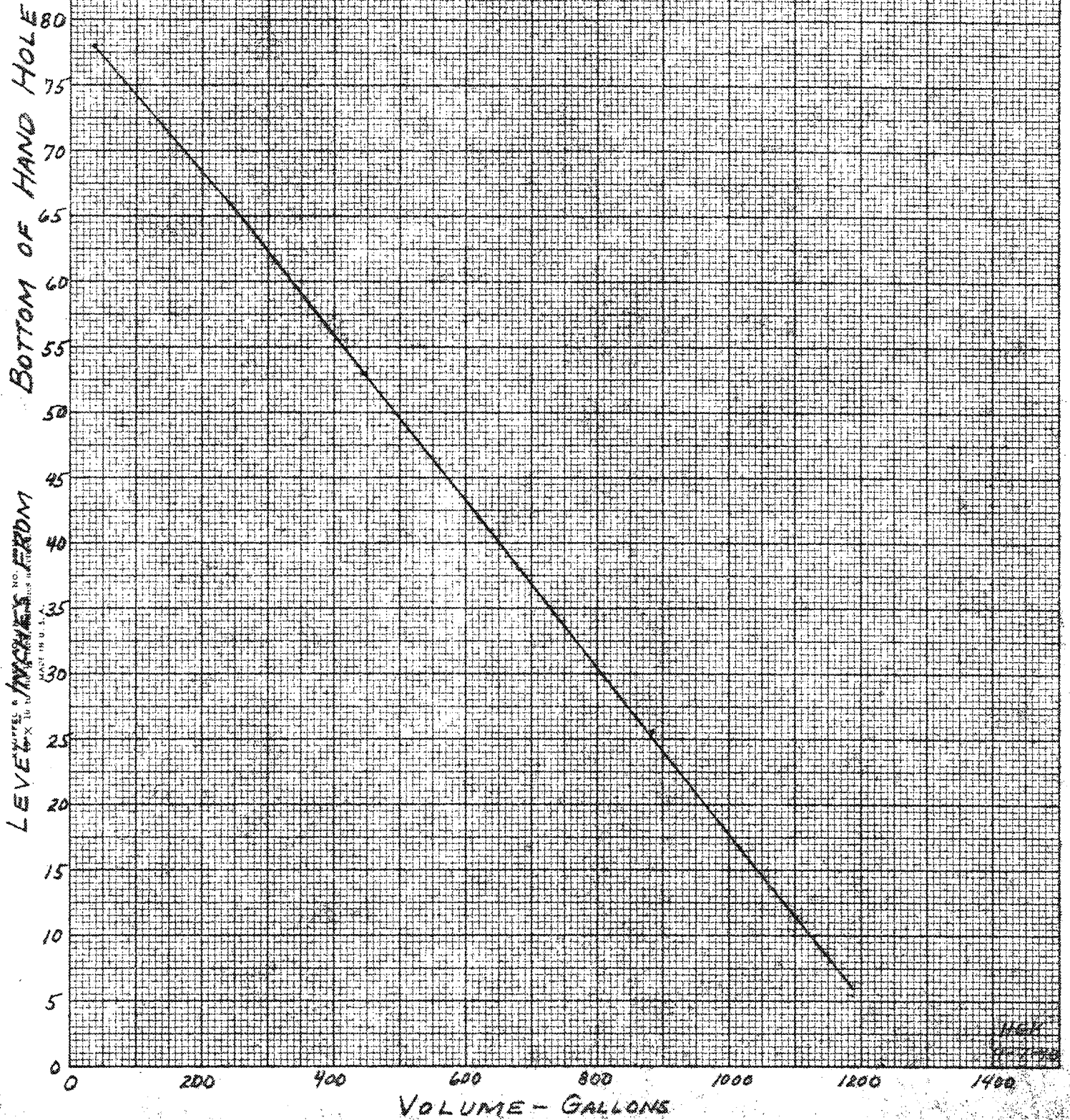
LIST OF GRAPHS AND DRAWING
CONTAINED IN THIS PROCESS PROCEDURE

Calibration Curve for 500 gallon Steel Neutralizer	#6219
Calibration Curve for 250 gallon Benzol Storage Tank	#6235
Calibration Curve for 1,100 gallon Lead Lined Extractor	#6763
Calibration Curve for 25 gallon Vacuum Receiver	#8526
Calibration Curve for 350 gallon Copper Still	#8528
Calibration Curve for 600 gallon Steel Still	#8728
T.C.P. Process Flow Sheet	C-2812

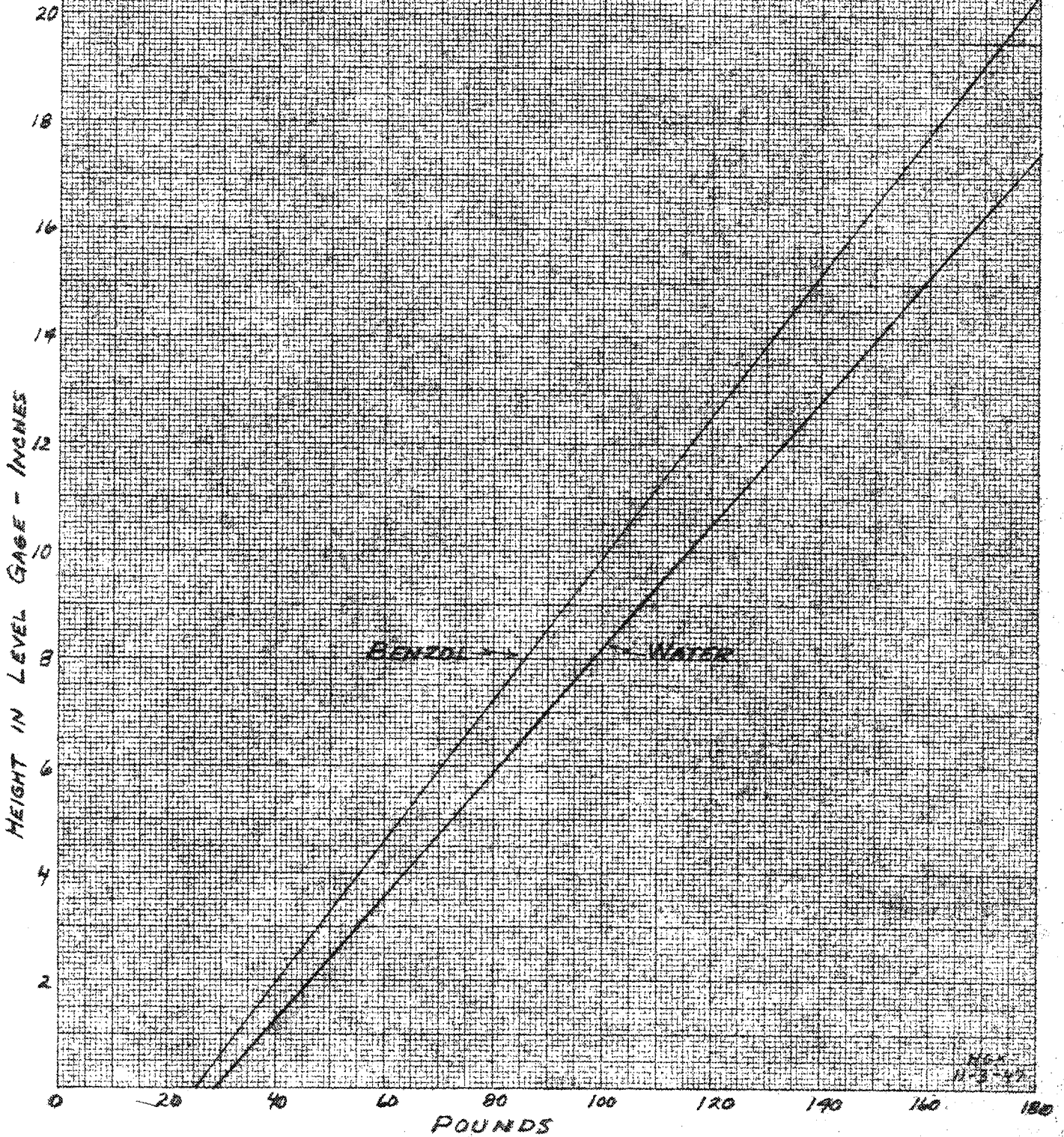
REUFFEL & SEER CO., N. Y. NO. 359H-14
MILWAUKEE, WIS. LINES APPROVED, CAN. TRADE MARK
MADE IN U. S. A.



CALIBRATION CURVE FOR 1100 GALLON
LEAD LINED EXTRACTOR #6763
BLDG. 60

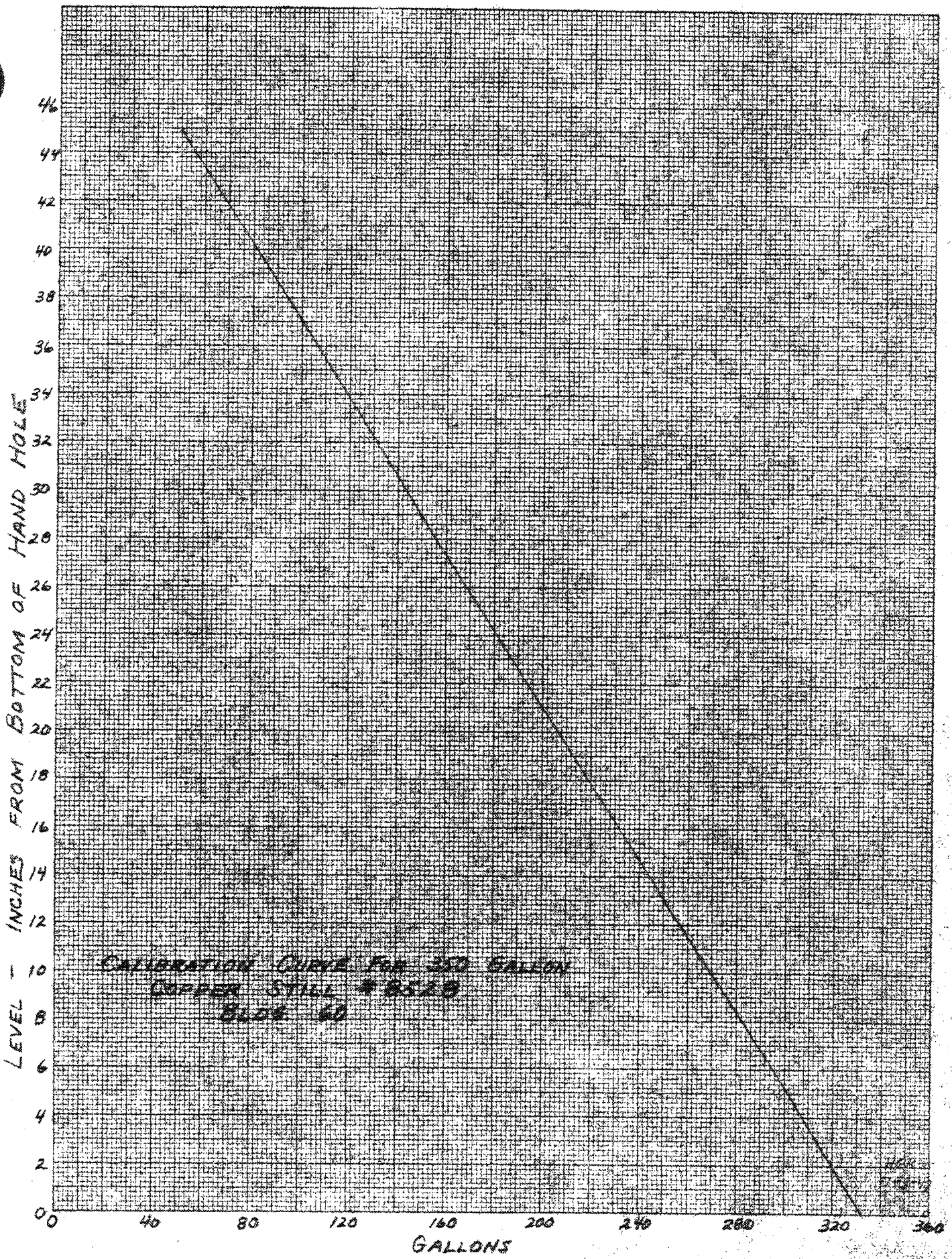


CALIBRATION CURVE FOR 2.5 GALLON
VACUUM RECEIVER # 2576
BLDG. 60



KEUFFEL & ESSER CO., R. Y. NO. 3991-11
MILWAUKEE, WIS. (LITHO. BOHEMIA) CHINA BRASS
MADE IN U. S. A.

11-2-47



KEUFFEL & ESSER CO., N. Y. NO. 8589-14
MULTIPLYERS, 3 mm. Bands provided, can. Paper heavy
MADE IN U. S. A.

KEUFFNER & ESSER CO., N. Y. NO. 89841, IF
1 1/2" X 10" TO THE 1/8" GRID, 50# THIS APPROX.
MADE IN U.S.A.

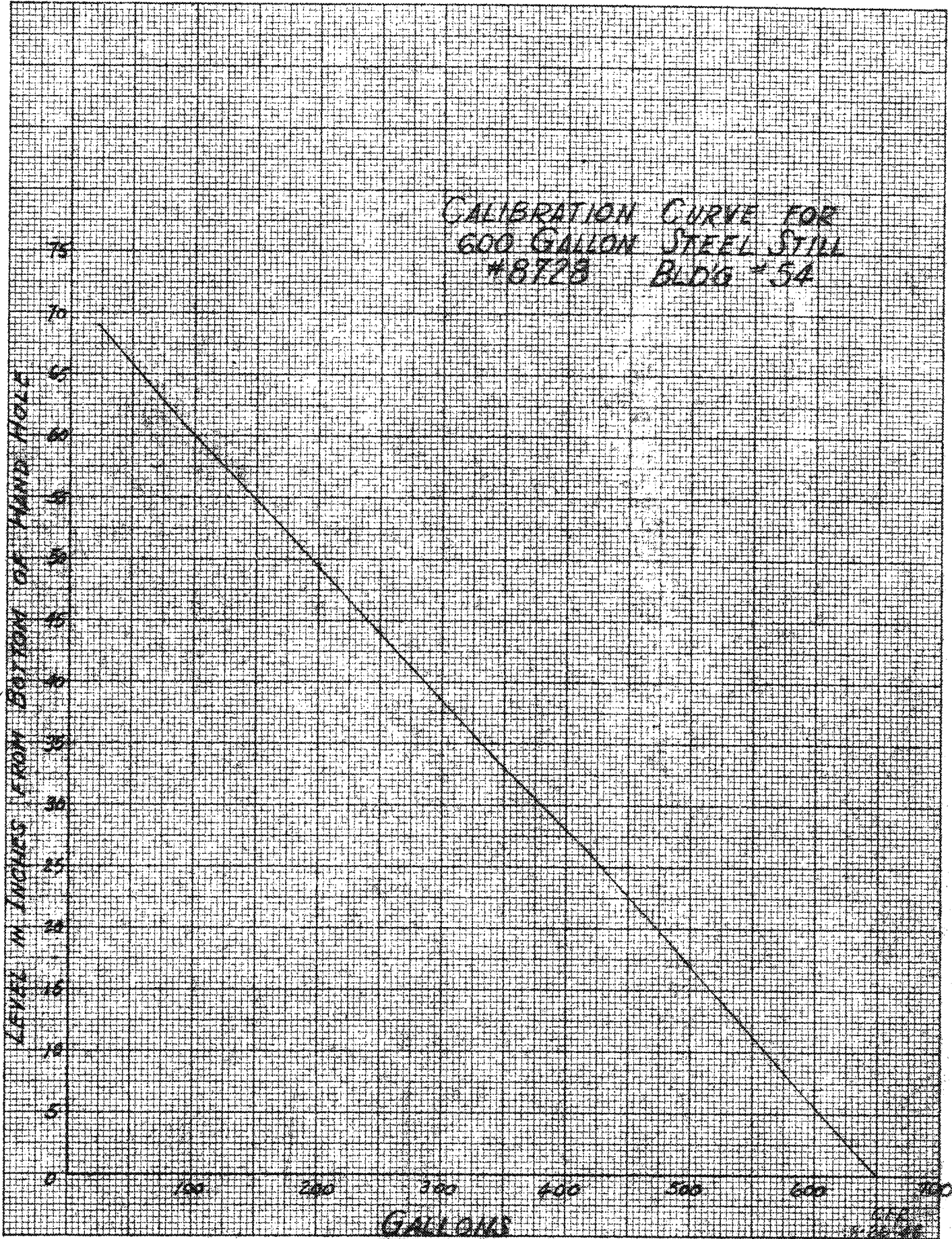


Exhibit S



Environmental Forensics

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Modeling the formation of 2,3,7,8-tetrachlorodibenzo-p-dioxin in the historical manufacture of 2,4,5-trichlorophenol

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Modeling the formation of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin in the historical manufacture of 2,4,5-trichlorophenol

Robert Parette^a, Robert McCrindle^{b,c}, Katherine S. McMahon^d, Valerie J. Watson^a, Michael J. Janik^e, Darrell Velegol^e, Frank L. Dorman^f, and Wendy N. Pearson^a

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ABSTRACT

A universal model for the simulation of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin concentrations in the historical manufacture of 2,4,5-trichlorophenol was developed. The model successfully simulates the conversion of 1,2,4,5-tetrachlorobenzene to 2,4,5-trichlorophenol, and modeled the concentrations of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin with an r^2 of .941 against measured data from 38 experimental runs. The high r^2 supports the proposed formation mechanism (second order with respect to 2,4,5-trichlorophenol). Simultaneous degradation of 2,3,7,8-tetrachlorodibenzo-*p*-dioxin by methoxylation/hydroxylation was hypothesized to play a role; however, modeling indicated this was insignificant in the industrial production of 2,4,5-trichlorophenol. This model can be a useful tool in environmental forensic investigations of sites where 2,4,5-trichlorophenol was produced.

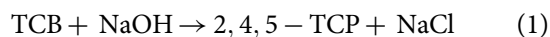
KEYWORDS

2,3,7,8-Tetrachlorodibenzo-*p*-dioxin (2,3,7,8-TCDD); 2,4,5-trichlorophenol (2,4,5-TCP); 2,4,5-trichlorophenoxyacetic acid (2,4,5-T); dioxin formation; modeling

Introduction

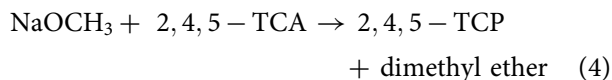
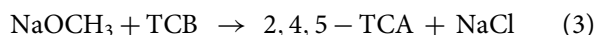
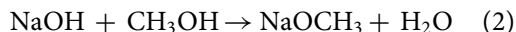
2,4,5-Trichlorophenoxyacetic acid, commonly referred to as 2,4,5-T, is an herbicide that was produced in the U.S. beginning in the mid-1940s. Production of 2,4,5-T, from the raw material 2,4,5-trichlorophenol (2,4,5-TCP), reached a peak during the second half of the 1960s as it comprised approximately 50% of the infamous “Agent Orange” used in the Vietnam War (U.S. Tariff Commission, 1941–1984; Hay, 1982; Young, 2009). The manufacture of 2,4,5-T was the largest single use for 2,4,5-TCP, but 2,4,5-TCP was also used as a germicide, in the manufacture of other herbicides, and in the manufacture of hexachlorophene (Doedens, 1964; Piacitelli et al., 1990).

To produce 2,4,5-TCP, 1,2,4,5-tetrachlorobenzene (TCB) was hydrolyzed in the presence of sodium hydroxide (NaOH). Methanol (CH₃OH) was typically used as the solvent, though one major U. S. manufacturer utilized ethylene glycol in place of methanol (Doedens, 1964; Marlow and Fingerhut, 1984, 1986; Marlow et al., 1989, 1991a). In its most simplistic form, the reaction to produce 2,4,5-TCP can be written (Hay, 1982):



Although 2,4,5-TCP and 2,4,5-T were produced commercially beginning in the mid-1940s (U.S. Tariff Commission, 1941–1984), it took nearly two decades before the chemistry of 2,4,5-TCP production was rationalized in the scientific literature. 2,4,5-Trichloroanisole (2,4,5-TCA), also formed in the manufacture of 2,4,5-TCP, was thought to be a by-product caused by a reaction between methanol and 2,4,5-TCP, and methanol was thought to behave only as a solvent in the primary reaction (Jenney and Nicolaisen, 1956; Doedens, 1964). However, in the early 1960s Shein and Ignatov (1962a, 1962b) found that the formation of 2,4,5-TCP was actually a two-step reaction (competitive consecutive second-order reactions), and that methanol played a larger role than simply as a solvent. Addition of sodium hydroxide to methanol led to the formation of sodium methoxide (NaOCH₃) (Equation (2)). Sodium methoxide in turn reacted with 1,2,4,5-tetrachlorobenzene to form 2,4,5-TCA (Equation (3)). The 2,4,5-TCA intermediate then reacted with another molecule of sodium methoxide to form 2,4,5-TCP (Equation (4)). In this environment, 2,4,5-TCP would be present as its sodium salt, but for simplicity is denoted as 2,4,5-TCP throughout

this article.



In the production of 2,4,5-TCP, the highly toxic (and severe chloracne) 2,3,7,8-tetrachlorodibenzo-*p*-dioxin (TCDD) was formed as an inadvertent by-product. 2,3,7,8-TCDD was first identified as a by-product in 2,4,5-TCP manufacture by Kimmig and Schulz in 1957, though industrial producers of 2,4,5-TCP were already aware that an unknown severe chloracne formed during its manufacture (Marlow and Fingerhut, 1986; Marlow et al., 1989). However, very little research has been directed at understanding the formation of 2,3,7,8-TCDD in the 2,4,5-TCP reaction processes.

The lack of attention in the scientific literature given to the formation of 2,3,7,8-TCDD in the manufacture of 2,4,5-TCP was perhaps due to the hazards of working with an extremely toxic material, particularly in an exothermic process associated with a number of previous industrial accidents, including the well-known incident in Seveso, Italy (Hay, 1982; Marlow and Fingerhut, 1986; Marlow et al., 1989, 1990; Hauptmanns, 2007; Young, 2009). As the process to manufacture 2,4,5-TCP became largely obsolete after restrictions and bans were placed on the use of 2,4,5-T due to the presence of 2,3,7,8-TCDD (Hutzinger et al., 1985), it is also likely that scientists shifted their attention to understanding the formation of polychlorinated dibenzo-dioxins and furans (PCDD/Fs) in ongoing processes so that future inadvertent production of PCDD/Fs could be reduced. Over the past several decades, a number of researchers have examined the formation of PCDD/Fs from chlorinated aromatic precursors in the gas phase or thermal processes such as incineration or pyrolysis (Langer et al., 1973; Lahaniatis et al., 1985; Ritter and Bozzelli, 1994). Some studies utilized quantum chemical modeling to avoid the dangers of working with an extremely toxic chemical, specifically to examine 2,3,7,8-TCDD formation in the gas phase (Okamoto and Tomonari, 1999; Qu et al., 2009).

The ability to understand how much of a particular by-product would have formed in a particular process based on the manufacturing conditions can be a key component of an environmental forensics investigation to reconstruct historical chemical releases. This is particularly true in the case of 2,3,7,8-TCDD, in which 2,4,5-TCP production predated the analytical measurement of 2,3,7,8-TCDD by approximately two decades, and where

manufacturing practices changed over this same time period. To the authors' knowledge, the only studies performed in an attempt to elicit the formation mechanisms or to model the formation of 2,3,7,8-TCDD in the manufacture of 2,4,5-TCP were an internal Monsanto study performed in the late 1960s (Sims and Udell, 1969) and one by Braun and Schönbacher (1997). However, these studies modeled specific 2,4,5-TCP processes, thereby limiting their applicability to 2,4,5-TCP processes across the industry.

The Sims and Udell model was a mathematical model designed to predict the time it would take to achieve a 2,4,5-TCP yield of 99% and the amount of 2,3,7,8-TCDD that would be present at this time for a given set of conditions. This model did not explicitly represent rates of elementary chemical reactions, but instead had empirically derived input parameters for the temperature, TCB addition time, and mole ratios of CH₃OH, NaOH, and H₂O to TCB. As such, it was applicable only to processes where all the input parameters fell within the limitations of the data used to fit the model. For example, the shortest TCB addition time within the model confines was 30 min. Therefore, this model was not applicable to processes in which all the TCB was in the reactor at time zero. In many historical 2,4,5-TCP processes, either all the raw materials were added simultaneously at the start, or all the TCB was in the reactor initially with sodium hydroxide metered in over time.

The model of Braun and Schönbacher (1997) did take chemical mechanisms into consideration and provided reaction rate constants for the conversion of TCB to 2,4,5-TCA, of 2,4,5-TCA to 2,4,5-TCP, and for the formation of 2,3,7,8-TCDD. The reaction rate constants in this model changed only as a function of temperature. However, it is known that reaction rate constants for Equations (3) and (4) vary quite significantly as a function of the ratio of the solvents (methanol and water) present (Shein and Ignatov, 1963), while the impact of solvent composition on 2,3,7,8-TCDD is unknown. Furthermore, in the Braun and Schönbacher model, the formation of 2,3,7,8-TCDD was controlled by two separate reactions; one was first order with respect to both 2,4,5-TCA and NaOH, and the other was first order with respect to both 2,4,5-TCP and NaOH (Braun and Schönbacher, 1997; Hauptmanns, 2007). It is unclear how the equations for the formation of 2,3,7,8-TCDD were developed or how to reconcile them with an elementary chemical mechanism. Therefore the Braun and Schönbacher model is limited to the process for which it was designed to mimic.

The goal of this research was to utilize historical experimental data to develop a universal model to

predict the concentration of 2,3,7,8-TCDD that would have formed during the manufacture of 2,4,5-TCP, taking into account such factors as the temperature, the ratios of the input chemicals (including solvents), and the rate at which the chemicals were added into the reactor. In developing such a model, an understanding of the mechanisms that control the formation of 2,3,7,8-TCDD in 2,4,5-TCP manufacturing processes could be gained. Such a model can be a useful tool in environmental forensics investigations to understand amounts of 2,3,7,8-TCDD produced at sites where 2,4,5-TCP was manufactured.

Methodology

Modeling of TCB, 2,4,5-TCA, and 2,4,5-TCP concentrations

The liquid volume within the reactor was estimated based on the amounts of methanol, water, and TCB and their densities at the temperature of the reactor (Jungfleisch, 1868; Felder and Rousseau, 1986; Goodwin, 1987). Water was also present in industrial TCP processes as these processes utilized recycled methanol, and in some cases used sodium hydroxide in the form of an aqueous solution. In one instance discussed below, 2,4,5-TCA was used in the model instead of TCB. The density of 2,4,5-TCA was estimated based on the density of 2,4,6-TCA at 25°C of 1.64 g/mL (Lide, 2006), assuming the same density vs. temperature relationship observed for TCB. 1,4-Dichloro-2,5-dimethoxybenzene was also present in this instance where 2,4,5-TCA was used in the model, and its density is very close to that of 2,4,6-TCA (U.S. Environmental Protection Agency, 1980). The ΔV_{mixing} between the components in the model reactor was assumed to be zero. It is known that the ΔV_{mixing} between methanol and benzene, chlorobenzene, or bromobenzene in the temperature range of 20 to 40°C is minimal (Williams et al., 1948; Gupta et al., 2009).

The liquid volume in the reactor may change somewhat due to the addition of NaOH (Freeguard et al., 1965; Krey, 1972). However, data for the apparent molar volume of NaOCH₃ in methanol were found only for 25°C and no data was found at any temperature for NaOH in water/methanol mixtures. Therefore, it was assumed the addition of NaOH did not influence the liquid volume in the reactor. It was also assumed that the liquid volume in the reactor did not change as a result of the progression of the reaction of TCB to 2,4,5-TCP. Concentrations of constituents in the liquid phase were not adjusted to

account for volatilization to the vapor phase within the modeled autoclave reactor as it would not significantly impact the concentrations in the liquid phase, particularly in an autoclave vessel where the volume was primarily liquid. Unless otherwise specified, the temperature data used throughout were assumed to be under isothermal conditions.

The model was run in Microsoft Excel by calculating the time-dependent concentrations from the set of differential equations using finite differences over small time steps (0.1 min). In the competitive consecutive second-order reactions to form 2,4,5-TCP (Equations (3) and (4)), the following differential equations were used in the model (Frost and Schwemer, 1952):

$$\begin{aligned} d[\text{NaOCH}_3]/dt = & -k_1[\text{NaOCH}_3][\text{TCB}] \\ & -k_2[\text{NaOCH}_3][2,4,5\text{-TCA}] \end{aligned} \quad (5)$$

$$d[\text{TCB}]/dt = -k_1[\text{NaOCH}_3][\text{TCB}] \quad (6)$$

$$\begin{aligned} d[2,4,5\text{-TCA}]/dt = & k_1[\text{NaOCH}_3][\text{TCB}] \\ & -k_2[\text{NaOCH}_3][2,4,5\text{-TCA}] \end{aligned} \quad (7)$$

$$d[2,4,5\text{-TCP}]/dt = k_2[\text{NaOCH}_3][2,4,5\text{-TCA}] \quad (8)$$

The composition of the solvent influences the species of the nucleophile (e.g., methoxide/hydroxide), the dissociation of ions pairs, and the solvation effects, all of which play a role in altering reaction rate constants (Anderson et al., 1969). Shein and Ignatov (1963) demonstrated that the reaction rate constants for Equations (3) and (4) were significantly reduced by the addition of water to methanol. Shein and Ignatov (1962b, 1963) did not consider the volume of TCB (or TCA) in their reactor when determining reaction rate constants. However, the concentrations of TCB (or TCA) in their laboratory tests were relatively dilute, and incorporating this additional volume would have changed the reaction rate constants only approximately the equivalent of a 1 to 2°C temperature change, likely within the experimental error. Therefore, reaction rate constants given by Shein and Ignatov were used “as is.”

Shein and Ignatov (1963) utilized these reaction rate constants (at different temperatures) to determine the activation energy (E_a) and the pre-exponential factor (A) for Equations (3) and (4) for different ratios of methanol to water through Equation (9) below. They also demonstrated that the activation energy (E_a) and pre-exponential factor ($\ln A$) for Equations (3) and (4) changed linearly with respect to the percentage of methanol (volume basis) in a methanol/water solvent mixture. E_a and $\ln A$ for Equations (3) and (4) as a function of the

percentage of methanol in the solvent were estimated based on a best-fit linear trend line from the E_a and $\ln A$ data given by Shein and Ignatov (1963). The reaction rate constants under these temperature/solvent conditions were then obtained using Equation (9) (the Arrhenius equation):

$$\ln k = \ln A - E_a/RT \quad (9)$$

This approach allows NaOCH_3 and NaOH to be considered as a single species, which avoids having to calculate equilibrium between NaOCH_3 and NaOH , each with its own reaction rate constant. The model was verified with data at various reactions conditions for which TCB, 2,4,5-TCA, and 2,4,5-TCP concentrations were reported as a function of time, before proceeding to incorporate the formation of 2,3,7,8-TCDD into the model.

Modeling of 2,3,7,8-TCDD concentration in the 2,4,5-TCP process

The reaction model described above was calibrated to include 2,3,7,8-TCDD using data from 38 experimental runs conducted in the late 1960s by Sims and Udell (1969). These 38 runs utilized different reaction conditions where the temperature, the ratio of chemicals (including solvents), and the time over which TCB was added to the reactors were varied. These 38 runs typically encompass conditions for the reaction industry-wide when 2,4,5-TCP was manufactured. For most of these runs, a 2,3,7,8-TCDD concentration for only a single time was given in the report (interpolated or extrapolated from analytical results of samples taken periodically throughout the run for the time at which a 99% 2,4,5-TCP yield was achieved). The experimental conditions for each of these runs, as well as the 2,3,7,8-TCDD concentration at a specified time (following the TCB addition), are listed in Table 1. The listed 2,3,7,8-TCDD concentrations are in parts per million (ppm) with respect to the 2,4,5-TCP product.

Given a 2,3,7,8-TCDD concentration reported at only a single point in time, a number of chemical mechanisms could be theoretically possible to account for 2,3,7,8-TCDD formation. In the development of this model, it was hypothesized that the dominant route to the formation of 2,3,7,8-TCDD was a result of the interaction of two molecules of 2,4,5-TCP (Equation (10)). This is consistent with the understanding of a number of former 2,4,5-TCP manufacturers (Brooker, 1981; Marlow and Fingerhut, 1984; Marlow et al., 1991a, 1991b). One industrial producer of 2,4,5-TCP specifically stated that

“the rate of formation of TCDD is greater towards the end of hydrolysis, being dependent upon sodium trichlorophenate concentration” (Brooker, 1981). Additionally, for two conditions within the 38 runs performed by Sims and Udell (1969), 2,3,7,8-TCDD concentrations were reported as a function of time. 2,3,7,8-TCDD concentrations did not rise appreciably until the conversion to 2,4,5-TCP was greater than 90% and appeared linear thereafter, consistent with a second-order dependence on the 2,4,5-TCP concentration. In accordance with Equation (10), Equation (8) was modified to Equation (11):

$$d[+2,3,7,8\text{-TCDD}]/dt = k_3[2,4,5\text{-TCP}] \times [2,4,5\text{-TCP}] \quad (10)$$

$$d[2,4,5\text{-TCP}]/dt = k_2[\text{NaOCH}_3][2,4,5\text{-TCA}] - 2 \times k_3[2,4,5\text{-TCP}][2,4,5\text{-TCP}] \quad (11)$$

Though the formation of 2,3,7,8-TCDD from 2,4,5-TCP is simulated as a one-step process in the model, it would require the formation of two ether linkages between the molecules of 2,4,5-TCP, which likely did not happen simultaneously. Formation of 2,3,7,8-TCDD from 2,4,5-TCP likely involved a polychlorinated phenoxyphenol intermediate, which has been found to be the major higher molecular weight impurity in a number of chlorophenols. The formation of PCDDs from polychlorinated phenoxyphenols containing an $-\text{OH}$ group in the 2-position and a chlorine atom in the 2'-position is a well-known reaction, and these particular chlorinated phenoxyphenol compounds have also been shown to undergo thermal ring closure to PCDDs in laboratory gas chromatographic analyses (Pohland and Yang, 1972; Rappe and Nilsson, 1972; Kende et al., 1974; Nilsson and Renberg, 1974; Nilsson and Andersson, 1977; Kunzevich et al., 1996). Polychlorinated 2-phenoxyphenols such as 4,5-dichloro-2-(2,4,5-trichlorophenoxy)phenol have been called “predioxin” and “pre-TCDD” (Nilsson and Renberg, 1974; Busch et al., 1980; Miller et al., 1983; Okamoto and Tomonari, 1999). However, to the authors' knowledge, formation of 2,3,7,8-TCDD directly from 4,5-dichloro-2-(2,4,5-trichlorophenoxy)phenol without the use of a catalyst or other external initiator (such as UV light) has not been studied. Formation of 2,3,7,8-TCDD via a mechanism involving radicals was considered but was not expected to be important. Though radicals can lead to the formation of 2,3,7,8-TCDD in high-temperature gas phase reactions (Okamoto and Tomonari, 1999; Evans and Dellinger, 2005), radicals

Table 1. Conditions and 2,3,7,8-TCDD results of 38 experimental runs by Sims and Udell (1969).

Run	Temp (°C)	TCB add. time (hr)	CH ₃ OH mol	NaOH mol	H ₂ O mol	TCB mol	Time after TCB add. (hr)	2,3,7,8-TCDD (ppm)
1	170	4.25	7.36	2.52	5.36	0.8	5.4	57
2	170	2.2	7.36	2.52	5.36	0.8	5	42
3	180	2.1	7.36	2.52	5.36	0.8	3.8	140
4	180	2.1	7.36	2.52	5.36	0.8	3.3	120
5	162	1.9	7.36	2.52	5.36	0.8	8.2	15
6	161	2.1	7.36	2.52	5.36	0.87	8.2	15
7	175	1.6	7.7	2.23	5.34	0.642	2.4	22
8	175	1.6	8.46	1.74	3.11	0.709	5.2	55
9	175	1.4	6.39	3.48	5.32	1.07	5	82
10	175	1.1	5.56	2.3	7.72	0.927	13	150
11	170	2	7.21	2.4	5.11	0.802	5.7	50
12	170	1.9	7.21	3.19	5.11	0.802	4	21
13	170	1.9	7.21	1.715	5.11	0.802	15	100
14	169	1.9	8.68	1.74	3.72	0.583	3.3	11
15	170	1.9	6.48	2.16	7.45	0.722	7.8	60
16	170	1.8	8.68	2.72	2.17	0.9	3.4	19
17	171	0.5	7.21	2.4	5.11	0.802	6.3	46
18	178	1.9	7.21	2.4	5.11	0.802	3.3	75
19	170	3.5	7.21	2.4	5.11	0.802	4.6	39
20	165	1.8	7.21	2.4	5.11	0.802	8.2	28
21	165	1.3	7.7	2.23	5.34	0.642	5.3	20
22	165	1.25	8.46	1.74	3.11	0.709	7.6	17
23	165	1.25	6.39	3.7	5.32	1.07	15	40
24	175	2.5	7.7	2.23	5.34	0.642	3.3	28
25	175	2.6	8.46	1.74	3.11	0.709	5.8	92
26	175	2.6	6.39	3.7	5.32	1.07	5.8	160
27	175	2.6	5.56	2.3	7.72	0.927	20	400
28	166	1.4	9.35	2.48	1.95	0.616	2.5	5
29	170	2	9.35	2.48	1.95	0.616	2.9	11
30	170	2	9.35	2.48	1.95	0.616	2.4	10
31	170	2	9.35	2.48	1.95	0.616	2	8
32	170	2	9.35	2.48	1.95	0.616	1.5	5
33	161	1.25	9.35	2.48	1.95	0.616	3.8	5
34	170	2	4.68	2.48	1.95	0.616	3.8	56
35	170	3.5	9.35	2.48	1.95	0.616	1.7	10
36	170	2	9.35	2.48	1.95	0.616	1.7	5
37	160	3.5	7.1	2.52	5.2	0.8	12	15
38	180	0.5	7.1	2.52	5.2	0.8	4	120

are not expected to play a significant role in the formation of 2,3,7,8-TCDD in the temperature range utilized in 2,4,5-TCP manufacture.

It was also hypothesized that simultaneous degradation of 2,3,7,8-TCDD (methoxylation or hydrolysis by NaOCH₃/NaOH) impacted the concentration of 2,3,7,8-TCDD during the manufacture of 2,4,5-TCP (Equation (12)). It is well known that PCDDs and PCDFs can be degraded by alkali during analytical sample processing. The degree of degradation is known to increase with the degree of chlorination, and is greater in PCDFs than PCDDs for PCDD/Fs containing the same number of chlorine atoms (Albro and Corbett, 1977; Firestone, 1977; Lamparski et al., 1978; Ryan et al., 1989; Gräbel and Hagenmaier, 1998; Nakao et al., 2002; Tsutsumi et al., 2003). Treatment of PCDD/Fs with ethanolic KOH resulted in the replacement of a chlorine atom with an ethoxy- group (Ryan et al., 1989). Therefore, presumably the treatment of PCDD/Fs with methanolic KOH would lead to the replacement of a chlorine atom with a methoxy- group. In aqueous KOH, hydrolysis

of PCDD/Fs occurs (Firestone, 1977).

$$d[-2, 3, 7, 8 - \text{TCDD}] / dt = -k_4[2, 3, 7, 8 - \text{TCDD}] \times [\text{NaOCH}_3] \quad (12)$$

Though 2,3,7,8-TCDD was reported to be stable in hot alkali in laboratory sample digestion (Firestone, 1991), the conditions utilized in the laboratory sample processing are mild in comparison to conditions in the industrial manufacture of 2,4,5-TCP. Research by Howard and Sidwell (1982) and Gräbel and Hagenmaier (1998) clearly demonstrated significant degradation of 2,3,7,8-TCDD can occur by NaOCH₃/NaOH in either a methanol or water solvent. In the work by Howard and Sidwell, 2,3,7,8-TCDD was reduced by 96.3% over 4.5 hours at 160°C when 200 g of chlorinated methoxybenzenes (approximately a 50/50 mixture of 2,4,5-TCA and dichloro-dimethoxybenzene based on the percent chlorine) containing an initial 2,3,7,8-TCDD concentration of 39.7 ppm was mixed with 350 g of methanol and 400 g NaOCH₃. Assuming 2,3,7,8-TCDD degradation is

first order with respect to both NaOH and 2,3,7,8-TCDD, this suggests a reaction rate constant on the order of $0.001 \text{ L} \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ under these conditions (NaOCH₃ in methanol at 160°C).

Gräbel and Hagenmaier (1998) studied the degradation of 2,3,7,8-TCDD at 280°C in a 1 M NaOH aqueous solution. Two g of soil from a former 2,4,5-TCP manufacturing site containing an initial 2,3,7,8-TCDD concentration of 99 ng/g soil were placed in contact with 60 mL of the 1 M NaOH solution in an autoclave heated by an aluminum heating block on a shaker. 2,3,7,8-TCDD degradation was measured at times of 10, 40, and 60 min. If it is assumed that 2,3,7,8-TCDD on the soil was extracted into the 1 M NaOH solution, these data suggest a reaction rate constant in the range of 0.1 to 0.4 $\text{L} \cdot \text{mol}^{-1} \cdot \text{min}^{-1}$ under these conditions (NaOH in water at 280°C). Soyfer et al. (1999) found that water at a temperature of 250°C was an effective solvent for the extraction of PCDDs from soil.

The previous studies of 2,3,7,8-TCDD formation in 2,4,5-TCP manufacture did not consider concurrent degradation of 2,3,7,8-TCDD (Sims and Udell, 1969; Braun and Schönbacher, 1997). However, the study conducted by Sims and Udell (1969) found that the ratio of NaOH to TCB played an important role in 2,3,7,8-TCDD formation, as higher ratios resulted in lower concentrations of 2,3,7,8-TCDD, providing support for the hypothesis that concurrent methoxylation/hydrolysis of 2,3,7,8-TCDD occurred during the manufacture of 2,4,5-TCP. Other degradation pathways for 2,3,7,8-TCDD, such as dechlorination to lesser chlorinated PCDDs, thermal decomposition, and photolysis were considered but were not expected to be significant in the industrial manufacture of 2,4,5-TCP (Stehl et al., 1973; Ryan et al., 1989; Samsonov et al., 1992; Gräbel and Hagenmaier, 1998; Zheng et al., 1999; Konstantinov et al., 2000; Tsutsumi et al., 2003).

The Solver function (using the GRG nonlinear method) within Microsoft Excel was utilized to solve for E_a and $\ln A$ for both 2,3,7,8-TCDD formation and degradation reactions that provided the maximum r^2 value for the model in comparison to the data for the 38 experimental runs of Sims and Udell (1969). As E_a and $\ln A$ for Equations (3) and (4) had been previously shown in the literature to change linearly with respect to the percentage of methanol in a water/methanol solvent, E_a and $\ln A$ for both 2,3,7,8-TCDD formation and degradation were allowed to change linearly with respect to the composition of the solvent within this model. As the Solver routine located a set of parameters giving a local maximum for r^2 that depended upon the initial guess solution and any constraints placed upon the fit constants, a number of different inputs and constraints were evaluated to find the global best fit solution (highest r^2 between the model

and the data of Sims and Udell). 2,3,7,8-TCDD degradation rates observed by Howard and Sidwell (1982) and Gräbel and Hagenmaier (1998) were used as constraints within the model.

Results and discussion

Verification of the model for TCB, 2,4,5-TCA, and 2,4,5-TCP concentrations

The reaction model concentrations compared favorably to three different 2,4,5-TCP processes for which data for TCB, 2,4,5-TCA, and 2,4,5-TCP concentrations were available as a function of time, as shown in Figure 1. The data in Figure 1a are from a batch reaction in “90%” methanol at 170°C, in which all the raw materials were added at time zero (Shein and Ignatov, 1962a). Though the Shein and Ignatov paper (1962a) referred to this methanol concentration as 90%, it is actually 92% (by mass) based on the information contained in the article. The data in Figure 1b originate from a process at 175°C in which all TCB was added at time zero and a methanol/water/sodium hydroxide mixture was metered into the reactor over 75 min (Perkins, 1961). The data in Figure 1c were obtained from a process where all the TCB and methanol, and some sodium hydroxide, were added at time zero, with a 50% sodium hydroxide solution (NaOH in water) metered in over 30 min at a

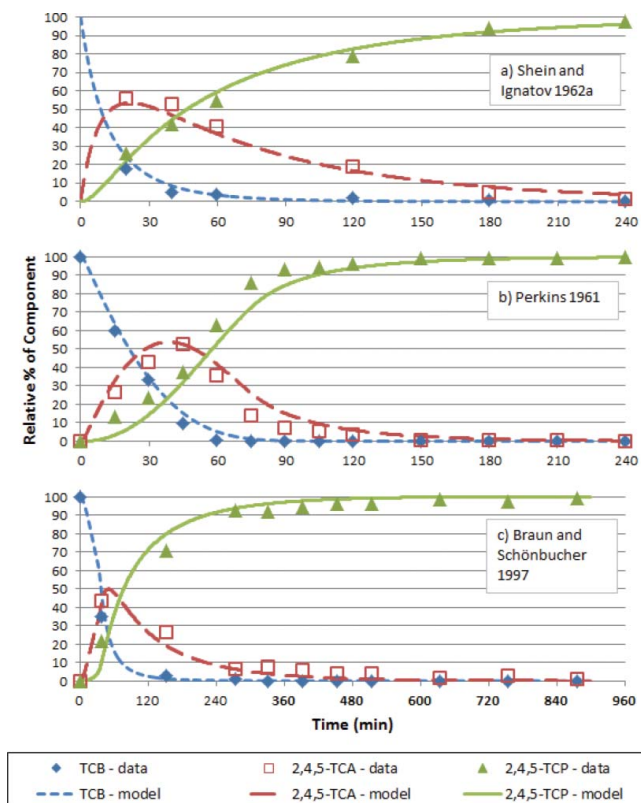


Figure 1. Reaction model vs. TCB, 2,4,5-TCA, and 2,4,5-TCP data.

temperature of 145°C. The temperature was then increased from 145 to 160°C over 5 min, and held at 160°C for the remainder of the reaction (Braun and Schönbacher, 1997). The purity of the methanol initially present in the reactor was not specified by Braun and Schönbacher (1997), therefore it was assumed to be 95%. Utilizing either 90% or 100% methanol in the model did not change the results significantly. The data in Figure 1c were normalized to remove the impurities/by-products, which were measured at 6% in the TCB raw material and generally measured around 10% throughout the test. The good agreement between model-predicted concentrations and measured values, observed over a variety of reactions conditions, demonstrates the robustness of the model for interconversion among TCB, 2,4,5-TCA, and 2,4,5-TCP.

Development of model for 2,3,7,8-TCDD

Once the model demonstrated successful simulation of the conversion of TCB to 2,4,5-TCA to 2,4,5-TCP, a variety of initial inputs and constraints were considered to identify local minima and the global best fit with maximum r^2 value between the modeled 2,3,7,8-TCDD concentrations and the 38 experimental runs of Sims and Udell (1969). Though multiple local minima were found with rather discrepant parameters, these results indicated that the addition of water to methanol caused the E_a for 2,3,7,8-TCDD degradation to increase significantly. These results further indicated that the reaction rate constant for 2,3,7,8-TCDD degradation would decrease rapidly with the addition of water to methanol, making the degradation reaction negligible in modeling the time-dependent 2,3,7,8-TCDD concentration. This was verified by removing the degradation reaction from the model and observing no change (within three significant digits) to the overall r^2 fit of the model.

Though 2,3,7,8-TCDD degradation is known to occur by reaction with $\text{NaOCH}_3/\text{NaOH}$ in either methanol or water under certain conditions, this reaction did not appear to be important in the conditions used to manufacture 2,4,5-TCP, contrary to the proposed hypothesis. Due to the limited data where 2,3,7,8-TCDD degradation was shown to occur and the large amount of data (38 experimental runs) where 2,3,7,8-TCDD degradation appeared to not play a significant role, little to no meaning was ascribed to the wide range of E_a and $\ln A$ values generated in the modeling for 2,3,7,8-TCDD degradation by $\text{NaOCH}_3/\text{NaOH}$. Therefore no E_a or $\ln A$ values are being reported for 2,3,7,8-TCDD degradation.

With 2,3,7,8-TCDD degradation eliminated from the model, the focus shifted to the formation of 2,3,7,8-TCDD. Though a good r^2 value for the model was

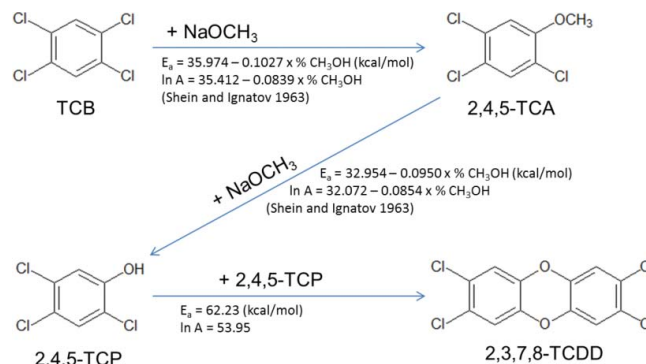


Figure 2. Complete reaction model with model parameters.

achieved when E_a and $\ln A$ values for 2,3,7,8-TCDD formation were allowed to change as a function of the solvent composition, the same r^2 was reached when E_a and $\ln A$ were fixed (not allowed to vary based on the amounts of methanol and water in the solvent), suggesting that solvent composition (water/methanol) was not an important factor in the formation of 2,3,7,8-TCDD during the manufacture of 2,4,5-TCP. To investigate this further, the reaction rate constant to best fit the data for each experimental run of Sims and Udell (1969) were determined in the model. Runs were separated into groups depending upon the percentage of methanol in the solvent, and the natural log of the reaction rate constants were then plotted as a function of $1/T$ (K). No evidence was observed to suggest that the composition of the solvent (methanol/water) made any difference with respect to the formation of 2,3,7,8-TCDD. Therefore, the best-fit E_a for the formation of 2,3,7,8-TCDD was 62.23 kcal/mol and the $\ln A$ was 53.95, independent of the composition of the solvent. The complete reaction model is shown in Figure 2.

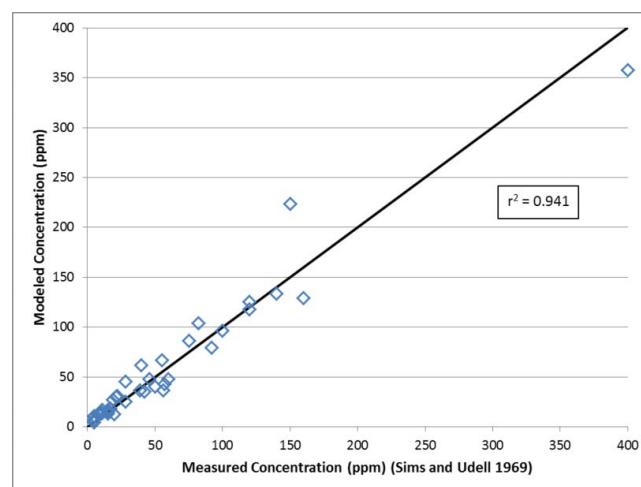


Figure 3. Simulated 2,3,7,8-TCDD vs. measured 2,3,7,8-TCDD for the 38 experimental runs of Sims and Udell (1969).

Using these inputs for E_a and $\ln A$ for 2,3,7,8-TCDD formation, the model successfully simulated the amount of 2,3,7,8-TCDD produced in the 38 experimental runs by Sims and Udell (1969), as shown in Figure 3 and Table 2. The r^2 value of .941, shown in Figure 3 (and Table 2), is with respect to the $y = x$ line, as all simulated data points (plotted as a function of the measured data points) would fall on this line in a perfect simulation. This r^2 value supports the hypothesis that the dominant mechanism for the formation of 2,3,7,8-TCDD in the manufacture of 2,4,5-TCP was the reaction of two molecules of 2,4,5-TCP. The mean absolute error for the model against this dataset was 10.6 ppm.

For comparative purposes, simulated results utilizing the Sims and Udell mathematical model are shown in Table 2. This empirical model, which was not based on elementary reaction kinetics, had an r^2 of .916 and a

Table 2. Modeled 2,3,7,8-TCDD concentrations vs. measured concentrations.

Sims and Udell Run No.	2,3,7,8-TCDD (ppm)		Sims and Udell Mathematical Model ($r^2 = .916$)	Model using k_s for TCDD formation from Braun and Schönbacher (1997) ($r^2 = .577$)
	2,3,7,8-TCDD data	Model ($r^2 = .941$)		
1	57	43.1	62.6	68.8
2	42	35.5	30.4	53.9
3	140	133.6	128.7	171.1
4	120	118.1	128.7	156.8
5	15	14.9	18.4	24.7
6	15	13.3	20.0	19.2
7	22	30.8	28.4	61.4
8	55	67.1	53.3	45.3
9	82	104.1	94.9	139.1
10	150	223.7	233.1	119.2
11	50	40.3	34.3	52.4
12	21	29.9	24.2	71.0
13	100	96.3	89.9	28.9
14	11	13.7	14.2	23.6
15	60	47.9	60.9	60.7
16	19	27.0	23.0	38.4
17	46	47.7	42.2	55.0
18	75	86.4	102.4	106.4
19	39	36.5	53.5	54.0
20	28	25.0	22.8	33.6
21	20	12.4	19.7	26.1
22	17	18.6	17.3	15.4
23	40	61.6	39.9	96.7
24	28	45.2	26.8	84.9
25	92	79.6	91.5	54.7
26	160	129.2	148.8	202.3
27	400	357.7	320.7	183.6
28	5	7.1	5.2	21.1
29	11	16.9	5.7	43.9
30	10	14.4	5.7	39.3
31	8	12.4	5.7	35.5
32	5	9.8	5.7	30.9
33	5	4.5	6.0	13.7
34	56	36.2	46.8	85.1
35	10	13.5	8.5	42.6
36	5	10.8	5.7	32.8
37	15	16.9	14.9	27.5
38	120	125.4	94.9	141.9

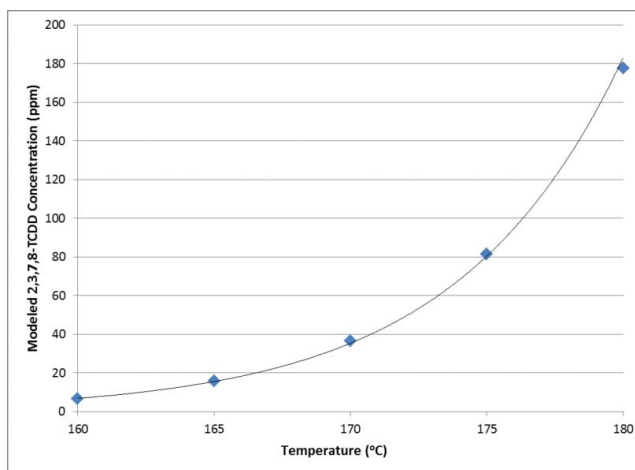


Figure 4. Simulated 2,3,7,8-TCDD concentrations for Sims and Udell’s experimental run 19 with different temperatures.

mean absolute error of 10.0 ppm. Also shown in Table 2 are our model results, modified to use the chemical mechanisms for the formation of 2,3,7,8-TCDD and rate constants as given by Braun and Schönbacher (1997) instead of Equation (10) and the best-fit E_a and $\ln A$ for k_3 above. Utilizing these alternative chemical mechanisms (and associated reaction rate constants), an r^2 of only .577 was obtained, further supporting a second-order dependence on 2,4,5-TCP as the dominant chemical mechanism in the formation of 2,3,7,8-TCDD.

It is evident from the model results that the formation of 2,3,7,8-TCDD (for a given time) in the manufacture of 2,4,5-TCP was sensitive to temperature. The modeled results shown in Table 2 could all be made to match the measured 2,3,7,8-TCDD concentration by changing the input temperature within $\pm 3^\circ\text{C}$, with the exception of run 32 (-4°C), and run 36 (-4.6°C). These temperature differences are likely within experimental error, demonstrating the predictive accuracy of the model. A further example of the model’s sensitivity to temperature is shown in Figure 4, where the temperature of the model for Sims and Udell’s run 19 was varied from 160 to 180°C . This experimental run was selected because Sims and Udell (1969) stated that this run was the most representative of the 2,4,5-TCP process utilized by Monsanto in the late 1960s. As shown in Figure 4, when holding the other inputs constant and changing only the temperature, the model predicted over a 2,400% increase in 2,3,7,8-TCDD concentration over only a 20°C temperature range. However, one cannot simply conclude that 2,3,7,8-TCDD concentrations would be increased or decreased to these levels by intentional operation at a temperature other than 170°C (the temperature of run 19). The duration of the industrial 2,4,5-TCP process was based upon the length of time required to achieve

the desired yield of 2,4,5-TCP. Lower temperatures would have required longer reaction times, and higher temperatures shorter reaction times. Due to the increased time required at lower temperatures, Sims and Udell (1969) concluded generally that the 2,3,7,8-TCDD concentration produced in 2,4,5-TCP manufacture was relatively insensitive to temperatures between 160 and 170°C.

Conclusions

A model for the simulation of 2,3,7,8-TCDD concentrations in the historical manufacture of 2,4,5-TCP was developed. The model successfully simulated the conversion of TCB to 2,4,5-TCA to 2,4,5-TCP, and modeled the concentration of 2,3,7,8-TCDD with an r^2 of .941 compared to measured data from 38 experimental runs. The high r^2 value obtained supported the proposed chemical formation mechanism for 2,3,7,8-TCDD (second order with respect to 2,4,5-trichlorophenol). Simultaneous degradation of 2,3,7,8-TCDD by methoxylation/hydroxylation was hypothesized to play a role in the concentration of 2,3,7,8-TCDD produced in 2,4,5-TCP manufacture, but the modeling indicated that TCDD degradation was insignificant in industrial 2,4,5-TCP processes. This model can be a useful tool in environmental forensic investigations of sites where 2,4,5-TCP was produced, as 2,4,5-TCP production predated the analytical measurement of 2,3,7,8-TCDD by approximately two decades.

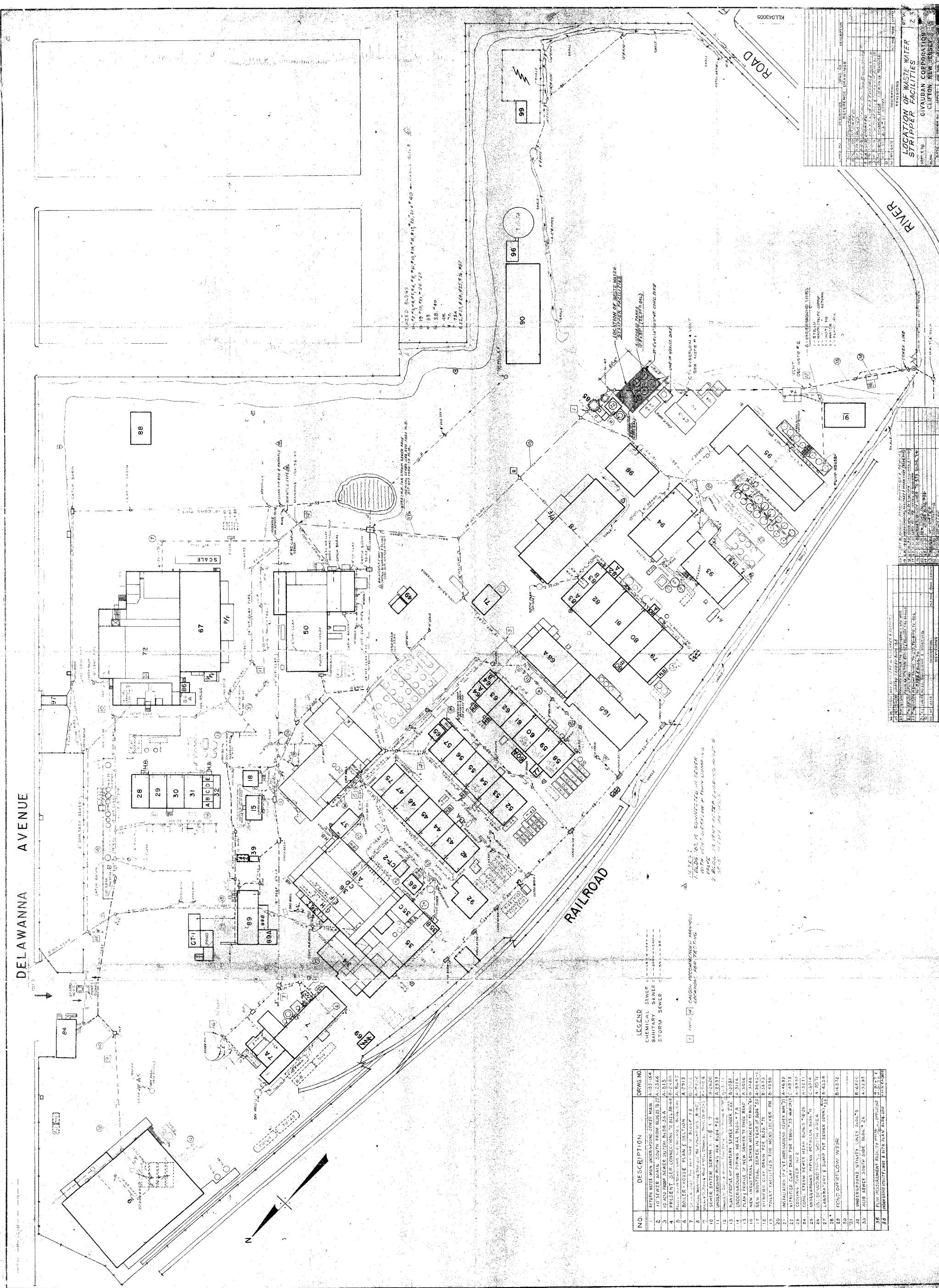
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Exhibit T



NO.	DESCRIPTION	DRWG NO.
1	RETURN WASTE MAIN UNDERGROUND SEWER MAIN	A-331-64
2	TO SEWER MAIN SOUTH FROM BLDGS 18, 22, A-2366	A-2366
3	TO SEWER MAIN SOUTH FROM BLDGS 18, 22, A-515	A-515
4	PRESENT SEWER CONNECTIONS TO BLDGS 28-32	A-28-32
5	PRESENT SEWER CONNECTIONS TO BLDGS 35-40	A-35-40
6	PRESENT SEWER CONNECTIONS TO BLDGS 43-48	A-43-48
7	PRESENT SEWER CONNECTIONS TO BLDGS 51-56	A-51-56
8	PRESENT SEWER CONNECTIONS TO BLDGS 59-64	A-59-64
9	PRESENT SEWER CONNECTIONS TO BLDGS 67-72	A-67-72
10	PRESENT SEWER CONNECTIONS TO BLDGS 75-80	A-75-80
11	PRESENT SEWER CONNECTIONS TO BLDGS 83-88	A-83-88
12	PRESENT SEWER CONNECTIONS TO BLDGS 91-96	A-91-96
13	PRESENT SEWER CONNECTIONS TO BLDGS 99	A-99
14	UNDERGROUND PIPING FOR SANITARY SEWER UNDER 7A	A-7A
15	NEW INDUSTRIAL SEWER MAIN SOUTH OF RIVER ROAD	A-3058
16	NEW INDUSTRIAL SEWER MAIN SOUTH OF RIVER ROAD	A-3188
17	NEW INDUSTRIAL SEWER MAIN SOUTH OF RIVER ROAD	A-3643-1
18	VITRIFIED CLAY DRAIN FOR BLDG #76	B-3333
19	TOILET FACILITIES FOR WAREHOUSE #90	B-4929
20	INSTALLATION OF 4\"/>	

LEGEND

--- CLAY SEWER
 --- SANITARY SEWER
 --- STORM SEWER
 [Symbol] CATCH BASIN
 [Symbol] MANHOLE
 [Symbol] UNDERGROUND MANHOLE
 [Symbol] UNDERGROUND PIPING
 [Symbol] UNDERGROUND UTILITY LINES
 [Symbol] VITRIFIED CLAY DRAIN

NO.	DESCRIPTION	DATE	BY
1	ISSUED FOR PERMIT	11/15/83	SK
2	ISSUED FOR CONSTRUCTION	11/15/83	SK
3	ISSUED FOR AS-BUILT	11/15/83	SK

NO.	DESCRIPTION	DATE	BY
1	ISSUED FOR PERMIT	11/15/83	SK
2	ISSUED FOR CONSTRUCTION	11/15/83	SK
3	ISSUED FOR AS-BUILT	11/15/83	SK

Exhibit U

**GIVAUDAN-ROURE
RIVER ROAD - WATER PROBLEM
REPORT**

MAY 1993

PREPARED BY
CORPORATE ENVIRONMENTAL ENGINEERING INC
FLEMINGTON, NEW JERSEY

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EXHIBITS AND SKETCHES

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Appendix A	List of Drawings Reviewed
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ABSTRACT

Problem Definition

As per Memorandum dated March 2, 1992 from Don Hogan, Givaudan-Roure to a distribution of other Givaudan-Roure personnel, Passaic County DPW received a complaint of icing on the roadway under the Railroad overpass on River Road. As reported by DPW personnel, the icing problem appeared to be caused by stormwater runoff entering River Road from the Givaudan-Roure site. (Runoff enters River Road through a curb cut along northern side of River Road at south western corner of the Property.) Mr. Hogan confirmed that runoff from Givaudan-Roure was entering River Road through the curb cut during inspection of the area on the morning following the complaint. As a result, CEE has been retained to investigate the extent of Givaudan-Roure's contribution to the problem and to identify possible solutions.

Problem Resolution

The investigation and identification consisted of:

1. *Evaluation of existing site drainage to determine what portion of the site contributes stormwater runoff to the area.*

Through review of existing drawing and maps along with field observation, the Givaudan-Roure Property south of Delawanna Avenue was divided in to areas draining to discharge points. In this manner, it was determined that 6.05 acres, (263,400 SF) drain to the River Road Curb cut. Of that, 3.1 acres drain directly from Givaudan-Roure to River Road through the curb cut. The remaining 2.95 acres drain to the ditch along the railroad track Right of Way and there to the curb cut.

2. *Evaluation of stormwater runoff flow pattern in River Road and impact of the physical condition of River Road on the Icing problem.*

Through field observation and review of existing drawings, it was determined that stormwater runoff on River Road flows westerly in the roadway gutters from a high point just east of the curb cut to inlets located at the intersection with Oak Street. This drainage system discharges into the Third River where River Road crosses the Third River.

Field observation of the physical condition of River Road in the vicinity of the icing problem revealed that a low spot exists in the gutter profile. In addition, the pavement is in poor conditions. Both conditions contribute to the icing problem.

3. *Quantification of Peak rate of runoff reaching River Road from the site.*

Application of the Rational Method to the total area from Givaudan-Roure that contributes runoff to the curb cut yields a 25 year peak rate of runoff or 23.8 cfs or 10,686 gpm.

4. *Identification of Solution Options and Feasibility.*

Several options were investigated, including methods to reduce both the volume and peak rate of runoff reaching the curb cut and methods to convey the runoff past the curb cut and area of icing to eliminate the problem. The feasibility of these methods was reviewed for economic constructibility and

practicality, both from a regulatory point of view and from a maintenance point of view.

The following options were considered:

- 1 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the existing retention lagoon
- 2 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to a new on-site detention impoundment
- 3 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the Delawanna Avenue stormsewer system,
- 4 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the PVSC sanitary sewer system
- 5 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the NJDOT/River Road stormsewer system,
- 6 Intercepting the runoff entering River Road from the SW curb cut and conveying it through a new piped system to its current destination, the Oak Street/River Road drainage system.
- 7 Intercepting all of the runoff entering River Road from the curb cut and conveying it through a new piped system to its current destination, the Oak Street/River Road drainage system.

Implementation of these options may require that one or more of the following actions be taken:

- Construction of a drainage system to move stormwater from one subdrainage basin to another.
- Construction of a pumping facility to overcome differences in elevations.
- Tying up substantial portions of currently undeveloped portions of the site with a surface impoundment or some sort of subsurface impoundment system with the associated maintenance problems.
- Construction of piping in portions of the site congested with existing buildings and underground utilities.

In addition, the possible options may involve one or more of the following regulatory concerns:

- Building permits, site plan approval, SCS certification, NJDEPE approvals.
- Prohibited discharge for which the receiving agency would probably not make an exception.
- Providing proof that the downstream receiving system has adequate excess capacity or providing detention to reduce the peak rate of runoff.

Conclusions and Option Recommendation

The icing problem appears to have two elements:

1. Ponding of stormwater runoff under the railroad overpass caused by deteriorating pavement and poor gutter line profile of River Road.
2. Greater rate of runoff than the road gutter can convey to the downstream receiving drainage system.

Regardless of the option chosen to reduce the peak rate of runoff to River Road, the deteriorating pavement and poor gutter line must be rectified in order to minimize the icing problem. The stormwater contributing to the peak rate of runoff drains from the railroad Right of Way, River Road and the Givaudan-Roure site. Based upon relative drainage area, Givaudan-Roure contributes the majority.

The recommended option consists of installing an inlet on-site near River Road to intercept the runoff from SW curb cut. This inlet will be connected to the existing system at Oak Street via a new dedicated stormsewer pipe in River Road, thereby reducing the rate of runoff reaching the gutter. The runoff from RR curb cut will continue to drain to River Road as it currently does. See Sketch 1.

INTRODUCTION

As per Memorandum dated March 2, 1992 from Don Hogan, Givaudan-Roure Corporation, 125 Delawanna Avenue, Clifton, Passaic County New Jersey, to a distribution of other Givaudan-Roure personnel, Passaic County Department of Public Works (DPW) received a complaint of icing on the roadway under the Railroad overpass on River Road adjacent to the southern property line.

As reported by DPW personnel, the icing problem appeared to be caused by stormwater runoff entering River Road from the Givaudan-Roure site. (Runoff enters River Road through a curb cut along northern side of River Road at south western corner of the Property.) Mr. Hogan confirmed that runoff from Givaudan-Roure was entering River Road through the curb cut during observation of the area on the morning following the complaint. As a result, Corporate Environmental Engineering Inc (CEE) has been retained to investigate the extent of Givaudan-Roure's contribution to the problem and to identify possible solutions.

For the purposes of this report, facility or site refers to the Specialty Division of Givaudan-Roure, located between Delawanna Avenue and River Road.

As an aside, please note that this stormwater problem does not violate the recently request NJPDES General Permit for Stormwater discharge to surface waters as long as the stormwater contains no pollutants picked up from the Givaudan-Roure site. Quantity of stormwater runoff is not an issue with the fore mentioned General Permit.

EVALUATION OF EXISTING SITE DRAINAGE

The existing site drainage was evaluated to determine what portion of the site contributes to the area of concern, River Road at the Railroad overpass. The evaluation consisted field observation supplemented by review of existing drawings obtained from Givaudan-Roure, Conrail, NJDOT, the City of Clifton, the County of Passaic (Ebasco, consultant to the county for the River Road Third River Bridge replacement). As a result of the evaluation, seventeen distinct areas have been identified. These areas either drain to the process wastewater sewer, to one of three on-site points with no off-site surface water discharge or to one of six points discharging off-site. The seventeen areas are delineated on Exhibit 1 and described in Table 1. The six discharge points locations are indicated on Exhibit 1 and described in Table 2.

DISCHARGE POINT DESTINATIONS AND RUNOFF FLOW PATTERNS IN RIVER RD

Discharge points 1, 2, and 3 all flow into a 15 inch stormsewer with an estimated capacity of 9 cfs or 4,100 gpm, part of the stormsewer system in Delawanna Avenue. This system conveys stormwater under the railroad overpass, south on Oak Street to the its intersection with William Street and to the outfall in northern shoreline of the Yantacaw Pond, a part of the Third River drainage basin. See Exhibit 2 for the schematic layout. Since Delawanna Avenue and Oak Street are Passaic County roads, this system is owned by the County.

Discharge point 4 flows through a 16 inch pipe into an NJDOT inlet in River Road. This inlet is connected via an 18 inch RCP with an approximate capacity of 10 cfs or 4,600 gpm to the NJDOT system for the Route 21/Route 3 roadway and ramps. This system eventually outlets into the Passaic

River somewhat east of the site and upstream of the Third River confluence with the Passaic River. See Exhibit 2 for the schematic layout. For additional information previously obtained for this connection, refer to the letter, section entitled "Status of the 16 Inch Connection", from CEE to Mr. Andrei Aroneanu of Givaudan-Roure, dated November 4, 1992. A copy of this letter is located in the Appendix.

Discharge points 5 and 6 both drain to the curb cut in the northern curb line of River Road at the southern corner of the site adjacent to the railroad ROW and overpass. See Sketches 1 and 2 for configuration of discharge points 5 and 6. From the curb cut, the runoff flows into the River Road curb line gutter with an approximated capacity of 3 cfs or 1,300 gpm and flows east toward the Oak Street intersection. At this point, the runoff is collected by existing inlets and conveyed by an existing 12 inch, 18 inch and 24 inch system that continues down River Road and outfalls into the Third River. It appears that this system was constructed as part of the Route 3 construction some time after 1927, the date of the plans. See Exhibit 2 for the schematic layout.

Through field observation, it was revealed that the free flow of stormwater in the River Road gutter is impeded by the deterioration of the pavement and a low spot in the gutter line profile. The pavement in the area under the overpass and adjacent to the curb cut appears to be asphalt on top of concrete pavement. The asphalt has deteriorated so that the concrete pavement is visible, creating depressions in the pavement. These conditions cause ponding of the stormwater.

Finally, a high point divides the River Road drainage between the NJDOT system and the Oak Street system. This high point is located approximately 175 feet east of the overpass.

QUANTIFICATION OF RUNOFF RATES FROM SITE

For each of six off-site discharge points, the peak rate of discharge has been calculated. The peak rate of runoff for the 25 year storm has been calculated using the Rational Method:

$$Q=CiA$$

where Q = peak rate of runoff in cubic feet per second
C = roughness coefficient
i = rainfall intensity in inches per hour
A = drainage area in acres

To prepare these calculations, work previously completed for other projects including the Closure Plan for the Lagoon prepared in September 1992 by CEE and drainage calculations for the Site Plan for the Proposed Neutralization Facility (building 210) prepared in January 1993 by CEE has been used. This work has been updated based upon information obtained for this project. The results of these calculations are summarized by Discharge Point in Table 3 and by Destination in Table 4.

Note that these calculations consider *only* runoff from Givaudan-Roure property. For example the peak rate of runoff calculated for A14 does not include runoff from the railroad ROW, itself, which is estimated to be 2.5 cfs or 1,100 gpm.

AVAILABLE EXISTING SOILS DATA

The available existing soils data consists of:

1. "Geotechnical Report, Waste Water Treatment Facility, Tank Foundation and Pipe Rack" , prepared by Woodward-Clyde Consultants, dated March 18, 1992 including an excerpt of the original geotechnical investigation report, dated July 2, 1981.

Briefly, fill material consists of silty sand over lying coarse to fine sand. The fill depth in the southern portion is 3 to 18 feet. Based upon recommendations by the consultant, the fill material was removed to construct the diked area for the Neutralization facility because too much clay was encountered. A copy of each of the reports is located in the Appendix D for informational purposes.

2. "Soil Survey of Passaic County, NJ", prepared by the USDA Soil Conservation Service in Cooperation with NJ Agricultural; Extension Station And Cook College, Rutgers University, dated October 1975.

This Survey identifies the soils in this area as being an "Urban Complex", or the area has been filled and no longer consists of undisturbed soils. Therefore, the engineering value of these soils is not addressed in the Survey. A copy of the pertinent section is included in the Appendix D, for informational purposes.

CONCEPTUAL SOLUTIONS EVALUATION

As stated previously, the icing problem appears to have been caused by ponding stormwater runoff flowing from uphill portions of River Road, off the Givaudan-Roure property and off the Railroad Right of Way through the curb cut in the northern curb of River Road just east of the Railroad Overpass of River Road. The ponding is caused by deterioration of the pavement and poor gutter line profile.

To minimize the icing, following must be implemented:

1. Repair deteriorated pavement and correct low spot in gutter line in the area of the Railroad crossing to eliminate ponding.
2. Reduce the peak rate of runoff that enters River Road.

The following options were considered:

- 1 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the existing retention lagoon
- 2 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to a new on-site detention impoundment
- 3 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the Delawanna Avenue stormsewer system,

- 4 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the PVSC sanitary sewer system
- 5 Rerouting a portion of the runoff entering River Road from the SW and RR curb cuts to the NJDOT/River Road stormsewer system,
- 6 Intercepting the runoff entering River Road from the SW curb cut and conveying it through a new piped system to its current destination, the Oak Street/River Road drainage system.
- 7 Intercepting the runoff flowing from the SW and RR curb cut and conveying it through a new piped system to its current destination, the Oak Street/River Road drainage system.

These seven options along with the feasibility factors are summarized in Table 5. The original proposal for this project called for equal investigation of all the options. However, as the investigation proceeded, some of the options appeared to be less practical than the others. Therefore, the effort was redirected at obtaining more information for those more practical solutions.

As a result, the sixth and seventh solutions were investigated to a further extent than the others. The following investigations. The following data was obtained:

1. Conversations with Ebasco revealed that the design for the replacement for the River Road bridge over the Third River is in preliminary design. It is a Passaic County project with NJ State and Federal funding. The area in question, the curb cut at the railroad overpass of River Road, is part of the drainage area for the existing drainage system associated with this bridge. However, the Ebasco's contract only extends to the drainage up to 100 feet away from the bridge and does not include any capacity analysis. Possible options may exist to coordinate drainage improvements, if needed.
2. Conversations with Steven Edmond, Passaic County Acting Engineer indicated that the County would agree to accept drainage improvements constructed within the River Road Right of Way and would assist Givaudan-Roure to obtain the necessary permits. The County would want to review the plans and execute an agreement with Givaudan-Roure prior to construction. Mr. Edmond indicated that he was pleased that Givaudan-Roure was considering steps to help correct the problem.

The question of Givaudan-Roure's liability after the County has accepted the system was not discussed. (See item 3, page 5 of Henry Kent-Smith's memorandum to Dr. Taveras.) This question should be addressed before a significant amount of additional work is done on this option.

3. Plans obtained from NJDOT for the construction of Route 3 dated 1948 only contained alignment and pipe size information for the drainage system beginning at Oak Street. One can infer that the River Road to the Third Street bridge drainage system was constructed before this date. These plans appear to reconstruction plans. NJDOT plans at the Trenton office could be further investigated. Previous efforts did not extend as far south and east. River Road is a County Road and the County only has the DOT plans. No other plans are available from this source. **Invert data may have to be obtained through field survey.**

CONCLUSIONS AND RECOMMENDATIONS

The icing problem appears to have two elements:

1. Ponding of stormwater runoff under the railroad overpass caused by deteriorating pavement and poor gutter line profile of River Road
2. Greater rate of runoff than the road gutter can convey to the downstream receiving drainage system.

Regardless of the option chosen to reduce the peak rate of runoff to River Road, the deteriorating pavement and poor gutter line must be rectified in order to minimize the icing problem. The stormwater contributing to the peak rate of runoff drains from the railroad Right of Way, River Road and the Givaudan-Roure site. Based upon relative drainage area, Givaudan-Roure contributes the majority.

The recommended option consists of installing an inlet on-site near River Road to intercept a runoff from A11 to reduce the total runoff reaching River Road. The inlet will be connected to the existing system at Oak Street via a new drainage pipe in River Road. See sketch of Proposed Alignment.

We recommend this course of action for the following reasons:

1. The stormwater *belongs* in this drainage basin.
2. This option collects only stormwater flowing from Givaudan-Roure, Option 7 collects all stormwater flowing to River Road, including that from the Railroad ROW (estimated to be 2.5 cfs) and River Road (estimated to be 3.6 cfs), itself.
3. It appears that the County will be cooperative.
4. It *may* be possible to coordinate the construction with that of Third River Bridge and minimize that cost.
5. No pumping stations are involved, therefore no associated maintenance.
6. Little construction on-site is required.
7. Little Givaudan-Roure property is impacted by the drainage improvements. (i.e., detention impoundments)

Possible complications consist of:

1. Existing stormsewer system at Oak Street to the Third River is undersized, requiring either a parallel system of replacement. A possible but undesirable alternative consists of providing some on-site detention. The volume would be less than for other solutions requiring detention.
2. Existing utilities in River Road and/or the existing slope may cause alignment difficulties and increase construction costs.

We recommend preparation of a concept design plan based on more complete data regarding the slope of River Road center line and gutter line along with inverts of the existing drainage system. (See note) Without these, required pipe sizes and existing pipe and gutter capacities calculated can be order of magnitude only. It may be possible to locate plans of the original River Road bridge over the Third River by reviewing the plans on microfilm in at the Trenton, NJDOT office. The original effort connected with the lagoon closure did not extend that far south on River Road. Otherwise, we recommend that the data should be obtained through field survey. In addition, utility mark outs in that area should be requested. In this manner, complications introduced by the utilities can be assessed.

Once a concept design plan has been prepared, the County should be officially approached. Repair of the roadway surface and coordination with the bridge design consultant should be part of the agenda. If the County prefers Option 7, then, for negotiation purposes, the County should be made aware of the contribution to runoff from the Railroad and River Road.

CEE is available to prepare both conceptual design plans and final design plans.

NOTE: Givaudan-Roure may want to consider opening a dialogue with the County and unofficially present the concept option plan (Sketch 1 for Option 6, without pipe sizes) to the County.

TABLES

TABLE 1			
EVALUATION OF EXISTING SITE DRAINAGE			
AREA NO.	AREA (AC/SF)	DESCRIPTION OF AREA	DESTINATION
A1	0.011/ 490	Diked area adjacent to and east of Building 7 that is connected to process wastewater sewer.	See note 1.
A2	0.144/ 6,290	Drainage area draining to abandoned rail unloading area behind rear loading dock of Building 9. There is no apparent outlet. Stormwater runoff percolates into ground and evaporates.	See note 2.
A3	0.577/ 25,130	Building 9 roof drains connect via piping to storm sewer in Delawanna Ave.	Discharge point 2.
A4	0.062/ 2,680	Grassed area located in the north western corner of the facility that sheet flows to Delawanna Ave. and into the storm sewer.	Discharge point 1.
A5	0.027/ 1,150	Paved drainage area, located along Delawanna Avenue, drains to grate at low spot over underground tank location. Stormwater runoff percolates into the ground and evaporates.	See note 2.
A6	0.213/ 9,300	Paved drainage area located adjacent to the Main Guard House that sheet flows into the Delawanna Avenue storm sewer.	Discharge point 3.
A7	0.134/ 5,850	Paved or diked drainage area located between the 30's buildings and the 40's Buildings, that drains to an inlet connected to the process wastewater sewer.	See note 1.
A8	0.046/ 2,000	Paved areas located west and south of the 80's Buildings. The paved areas drain into diked areas through gunnels. The diked areas are pumped out by portable pumps into the process wastewater sewer.	See note 1.
A9	0.017/ 730	Paved area that drains to a process wastewater sewer inlet.	See note 1.

Total Acreage as per Fletcher Survey - 30.5 Acres

- Notes:
- 1 Area excluded from calculations because it *is connected to process wastewater sewer.*
 - 2 Area excluded from calculations because it *does not contribute to off-site runoff.*

TABLE 1
EVALUATION OF EXISTING SITE DRAINAGE

AREA NO.	AREA (AC/SF)	DESCRIPTION OF AREA	DESTINATION
A10	0.121/ 5,270	Paved area that drains into diked area through gunnels; diked area is connected to the process waste water sewer via ejector pump.	See note 1.
A11	3.306/ 144,015	Paved, soil and stone covered drainage area located in the southern portion of facility adjacent to the proposed neutralization facility that drains to the curb cut in the River Road frontage near the Rail Road tracks.	Discharge point 5.
A12	6.249/ 272,220	Paved, roof and grass covered drainage area tributary to the swales and pipes in the south eastern portion of the facility that drains to the NJDOT inlet in River Road.	Discharge point 4.
A13	16.182/ 704,870	Paved, roof and grass covered drainage area located in the north eastern portion of the facility that is tributary to the retention pond through a system of pipes and over land flow.	See note 2.
A14	2.945/ 128,280	Paved and roofed areas located along the railroad tracks that drain via storm sewer system and gunnels in asphalt curb to drainage ditch along railroad tracks and to the curb cut in the River Road frontage next to the rail road tracks.	Discharge point 6.
A15	0.217/ 9,460	Paved areas that drain into the diked areas through gunnels located north and south of building 95 that are connected to the process wastewater sewer.	See note 1.
A16	0.069/ 3,020	Diked area located around the pretreatment facility that is connected to the process wastewater sewer.	See note 1.
A17	0.180/ 7,850	Paved area that drains via gunnels into a diked area know as tank farm 201. This area is pumped into the process wastewater sewer.	See note 1.

Total Acreage as per Fletcher Survey - 30.5 Acres

- Notes:
- 1 Area excluded from calculations because it *is connected to process wastewater sewer.*
 - 2 Area excluded from calculations because it *does not contribute to off-site runoff.*

May 20, 1993

TABLE 2

STORMWATER DISCHARGE POINTS
(Locations where stormwater runoff flows off-site.)

NO.	NAME	LOCATION	DESCRIPTION	AREAS ¹	DESTINATION
1	NW Corner	North west of site.	Sheet flow into Delawanna Avenue gutter.	A4	Delawanna Ave. Storm Sewer to Third River
2	Bldg 9	Roof drains, Building 9.	Building 9 roof drainage into storm sewer in Delawanna Avenue.	A3	Delawanna Ave. Storm Sewer to Third River
3	Guard House	Adjacent to Guard House.	Sheet flow into Delawanna Avenue gutter.	A6	Delawanna Ave. Storm Sewer to Third River
4	NJDOT Inlet	NJDOT inlet in River Road.	Stormwater runoff collected via swales and pipes from south eastern portion of facility into storm sewer in River Road.	A12	NJDOT RT. 21/3 Storm Sewer to Passaic River
5	SW Curb Cut	Curb cut in River Road frontage.	Sheet flow and shallow gutter flow from southern portion of site around neutralization facility and south of building 95 into River Road gutter.	A11	Passaic County Storm Sewer at Oak St and River Rd to Third River
6	RR Curb Cut	Storm sewer and gunnels along rail road property line.	Stormwater runoff collected via storm sewer pipes and through gunnels in asphalt curb to ditch on rail road ROW and from there to River Road gutter via curb cut.	A14	Passaic County Storm Sewer at Oak St and River Rd to Third River

Note:

1 Area tributary to the outfall.

TABLE 3
QUANTIFICATION OF RUNOFF RATES FROM SITE
SUMMARY OF PEAK RATE OF RUNOFF BY DISCHARGE POINTS

POINT NUMBER	AREA NUMBER	NAME	AREA (acres)	WT "C"	T _c (min.)	i ³ (25 yr storm-in/hr)	PEAK RATE OF RUNOFF, 25 YR STORM	
							CFS	GPM
1	A4	NW Corner	.062	.25	6 ⁴	7.0	.1	49
2	A3	Building 9	.577	.98	6 ⁴	7.0	4.0	1,776
3	A6	Guard House	.213	.98	6 ⁴	7.0	1.5	655
4	A12	NJDOT Inlet	6.249	.84	12.1	5.4	28.3	12,701
5	A11	SW Curb Cut	3.306 ¹	.88	8.1	6.4	18.6	8,356
			3.104 ²	.88	8.1	6.4	17.5	7,846
6	A14	RR Curb Cut	2.945	.91	18.5	4.4	11.8	5,292
5 plus 6	A11, A14	Curb Cut	6.251 ¹	.89	18.5	4.4	24.5	10,986
			6.049 ²	.89	18.5	4.4	23.8	10,686

Notes:

- 1 Calculation for preconstruction conditions, before construction of concrete diked area for proposed neutralization facility.
- 2 Calculation for post construction conditions, after construction of the concrete dike area for the proposed neutralization facility.
- 3 Based upon Rainfall Intensity Frequency Duration curves developed for Wayne Township, Passaic County, NJ.
- 4 Time of concentration not measurable, therefore used minimum time on Chart referenced in Note 2.

TABLE 4					
QUANTIFICATION OF RUNOFF RATES FROM SITE SUMMARY OF PEAK RATE OF RUNOFF BY DESTINATION					
DESTINATION	POINT NUMBERS	AREA NUMBERS	AREA (acres)	PEAK RATE OF RUNOFF, 25 YR STORM	
				CFS	GPM
Delawanna Avenue Stormsewer	1,2,3	A4,A3,A6	0.852	5.6	2,480
NJDOT Stormsewer	4	A12	6.249	28.3	12,700
Oak Street Stormsewer (via River Road Gutter)	5,6	A11,A14	6.251 ¹	24.5	10,986
			6.049 ²	23.8	10,686

Notes:

- 1 Calculation for preconstruction conditions, before construction of concrete diked area for proposed neutralization facility.
- 2 Calculation for post construction conditions, after construction of the concrete dike area for the proposed neutralization facility.

TABLE 5

CONCEPTUAL SOLUTIONS EVALUATION

TITLE	DESCRIPTION	FEASIBILITY FACTORS
<p>1 Retention Lagoon</p>	<p>Reroute portions of stormwater runoff from on-site areas A11 and A14 via new piping and inlets to the existing retention pond to reduce the rate of runoff and volume leaving the site to minimize icing on River Road near the Rail Road crossing.</p>	<ul style="list-style-type: none"> • Lagoon over topped in December 1992, capacity may not be adequate, could expand lagoon. • Rerouting will require that piping be installed on-site through areas with many buildings and existing underground utilities. • Pumping may be required, especially if rerouting stormwater from southern part of site. • Pumps add on going maintenance costs. • May have to modify NJPDES-DGW permit for lagoon. • Building permit, site plan approval, SCS certification.
<p>2 Detention Impoundment</p>	<p>Reroute portions of the runoff from areas A11 and A14 new piping and inlets to an aboveground or underground detention impoundment. Discharge point would be River Road curb cut. This structure would meter the rate at which the runoff leaves the site at the River Road curb cut.</p>	<ul style="list-style-type: none"> • Aboveground impoundment-berms must be less than 3 feet in height, top to outside toe, otherwise dam-additional permits and constraints. • If invert is too low, may require pumping to discharge. • Pumps add on going maintenance costs. • Underground impoundment consists of large pipes with small outlet point. These pipes could be perforated to allow pecculation into the ground-however preliminary review of soils data indicates high clay content. • May require NJPDES-DGW permit if impoundment is permeable, i.e., not lined or perforated. • Building Permit, site plan approval, NJDEPE approvals, SCS certification.
<p>3 Delawanna Avenue Stormsewer</p>	<p>Reroute portions of the runoff from areas A11 and A14 via new piping and inlets to the Delawanna Ave stormsewer to reduce the peak rate of runoff and volume leaving the site at the River Road curb cut.</p>	<ul style="list-style-type: none"> • Since the stormsewer is at the high end of the site, pumping would most likely be required. • Pumps add on going maintenance costs. • Delawanna Ave. is a Passaic County Road, therefore county approval would be required. • Rerouting requires moving stormwater from one subbasin to another. Proof that existing piping has adequate capacity or detention or replacement of piping may be required. • See <i>Detention Impoundment</i> for other constraints. • Building permit, site plan approval, County approval, SCS certification.

- NJPDES Permit - New Jersey Pollution Discharge Elimination System Permit (New Jersey Annotated Code 7:14A)
- DGW Permit - Discharge to Groundwater Permit, a subset of NJPDES.
- TWA Modification - Treatment Works Approval Modification, a subset of NJPDES.
- SCS Certification - Soil Erosion and Sediment Control Plan Certification from New Jersey Soil Conservation Service.

Note: In all cases the roadway in the area of the icing problem must be repaired to eliminate ponding of stormwater runoff.

TABLE 5		
CONCEPTUAL SOLUTIONS EVALUATION		
TITLE	DESCRIPTION	FEASIBILITY FACTORS
4 PVSC Sewer	Reroute portions of the runoff from areas A11 and A14 via new piping and inlets to the Givaudan-Roure Sanitary Sewer line that connects to the sewer line in River Road and eventually connects to the PVSC trunk line to reduce the rate of runoff and volume leaving the site.	<ul style="list-style-type: none"> Uncontaminated stormwater is prohibited unless specifically allowed by the PVSC. Stormwater would increase the hydraulic loading significantly during a storm. Not likely that PVSC would approve. Givaudan-Roure would have to pay for the volume of stormwater discharged to PVSC City endorsement, PVSC approval, TWA modification from NJDEPE, SCS certification, building permit.
5 NJDOT Inlet	Reroute portions of the runoff from areas A11 and A14 to the NJDOT Inlet in River Road via new piping and inlets to reduce peak rate of runoff and volume leaving the site at the River Road curb cut.	<ul style="list-style-type: none"> May require installation of piping in congested areas of the site. Based upon previous calculations, 16 " on-site connection pipe is currently inadequate. If this connection with the inlet in River Road is modified-NJDOT permits will be required. NJDOT will not allow increase in peak rate of runoff. Therefore, some form of on-site detention will be required. See <i>Detention Impoundment</i> for other constraints. SCS Certification, County approval, building permit
6 New River Road Stormsewer	<p>Collect stormwater runoff (17.5 cfs) from area A14 via new inlet near pine trees on-site and convey via new stormsewer pipe to existing stormsewer system beginning at Oak street to reduce stormwater in gutter in area of icing.</p> <p>See Sketch of Proposed Alignment.</p>	<ul style="list-style-type: none"> Requires little construction on-site. Requires no pump stations. Requires installation of stormsewer pipe and manholes for a minimum of 800 LF in River Road. The County requires a minimum size of 18" RCP. Collects stormwater not generated from Givaudan-Roure property. If the existing system is inadequate, an additional 500 LF of piping will require replacement or installation of a parallel system. (Alternative-construct some on-site detention. See <i>Detention Impoundment</i> for other constraints.) Once constructed, the County will accept the portion within River Road ROW as part of the County system, eliminating future ownership and maintenance by Givaudan-Roure. County approval, agreement with County Freeholders, City review, SCS Certification, building permit.

- NJPDES Permit - New Jersey Pollution Discharge Elimination System Permit (New Jersey Annotated Code 7:14A)
- DGW Permit - Discharge to Groundwater Permit, a subset of NJPDES.
- TWA Modification - Treatment Works Approval Modification, a subset of NJPDES.
- SCS Certification - Soil Erosion and Sediment Control Plan Certification from New Jersey Soil Conservation Service.

Note: In all cases the roadway in the area of the icing problem must be repaired to eliminate ponding of stormwater runoff.

TABLE 5

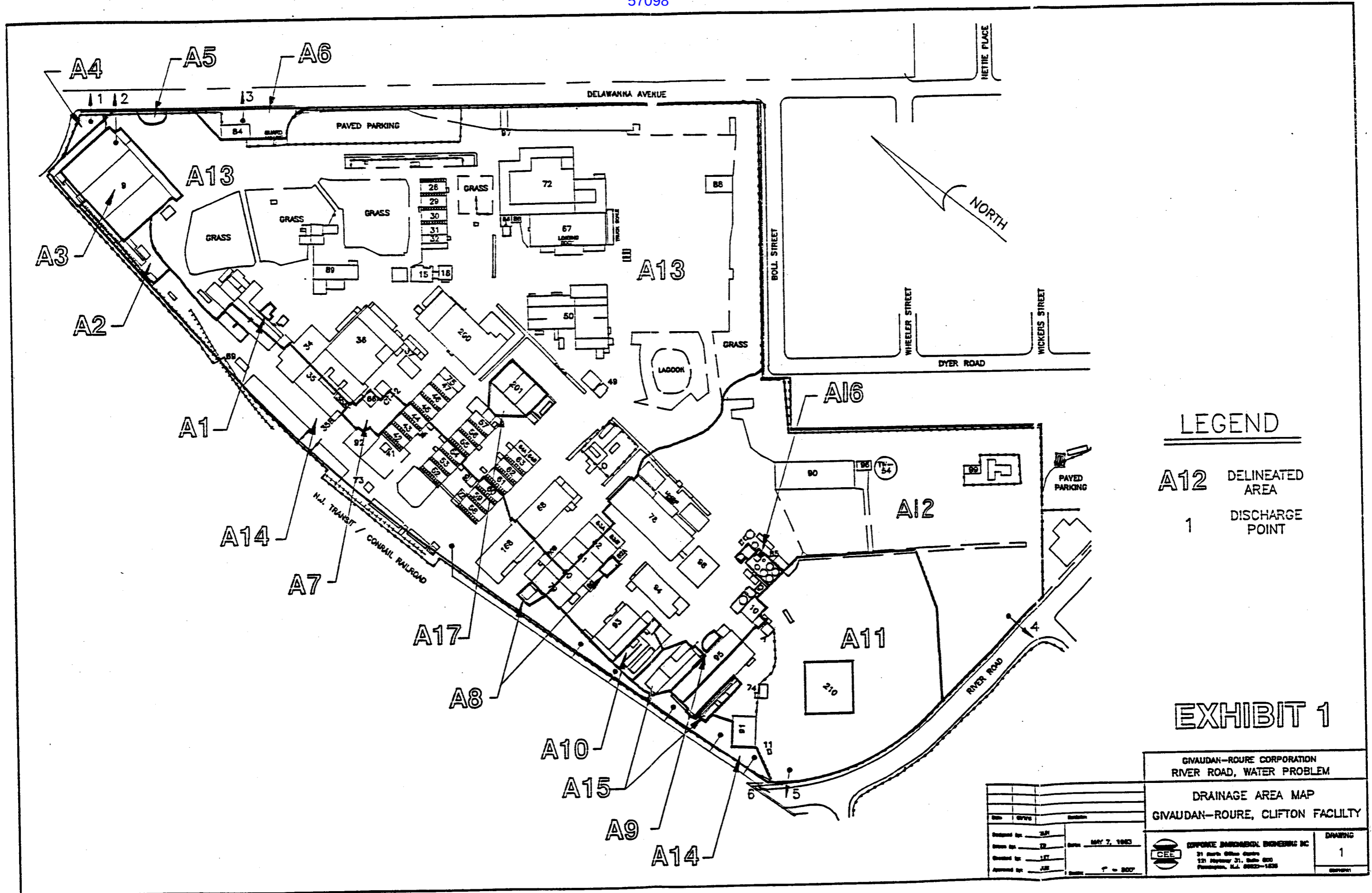
CONCEPTUAL SOLUTIONS EVALUATION

TITLE	DESCRIPTION	FEASIBILITY FACTORS
<p>7</p> <p>River Road Stormsewer Improvements</p>	<p>Collect stormwater runoff (23.8 cfs) from areas A11 and A14 via new inlet in River Road and convey via new stormsewer piping and inlets to existing stormsewer system beginning at Oak street to minimize stormwater in gutter in area of icing.</p> <p>See Sketch of Proposed Alignment.</p>	<ul style="list-style-type: none"> • Requires no construction on-site. Requires no pump stations. • Requires installation of stormsewer pipes and inlets for a minimum of 800 LF in River Road. The County requires a minimum size of 18" RCP. • Collects stormwater not generated from Givaudan-Roure property. • If the existing system is inadequate, an additional 500 LF of piping will require replacement or installation of a parallel system. (Alternative-construct some on-site detention. See <i>Detention Impoundment</i> for other constraints.) • Once constructed, the County will accept the system as part of the County system, eliminating future ownership and maintenance by Givaudan-Roure. • County approval, agreement with County Freeholders, City review, SCS Certification.

- NJPDES Permit - New Jersey Pollution Discharge Elimination System Permit (New Jersey Annotated Code 7:14A)
- DGW Permit - Discharge to Groundwater Permit, a subset of NJPDES.
- TWA Modification - Treatment Works Approval Modification, a subset of NJPDES.
- SCS Certification - Soil Erosion and Sediment Control Plan Certification from New Jersey Soil Conservation Service.

Note: In all cases the roadway in the area of the icing problem must be repaired to eliminate ponding of stormwater runoff.

EXHIBITS AND SKETCHES

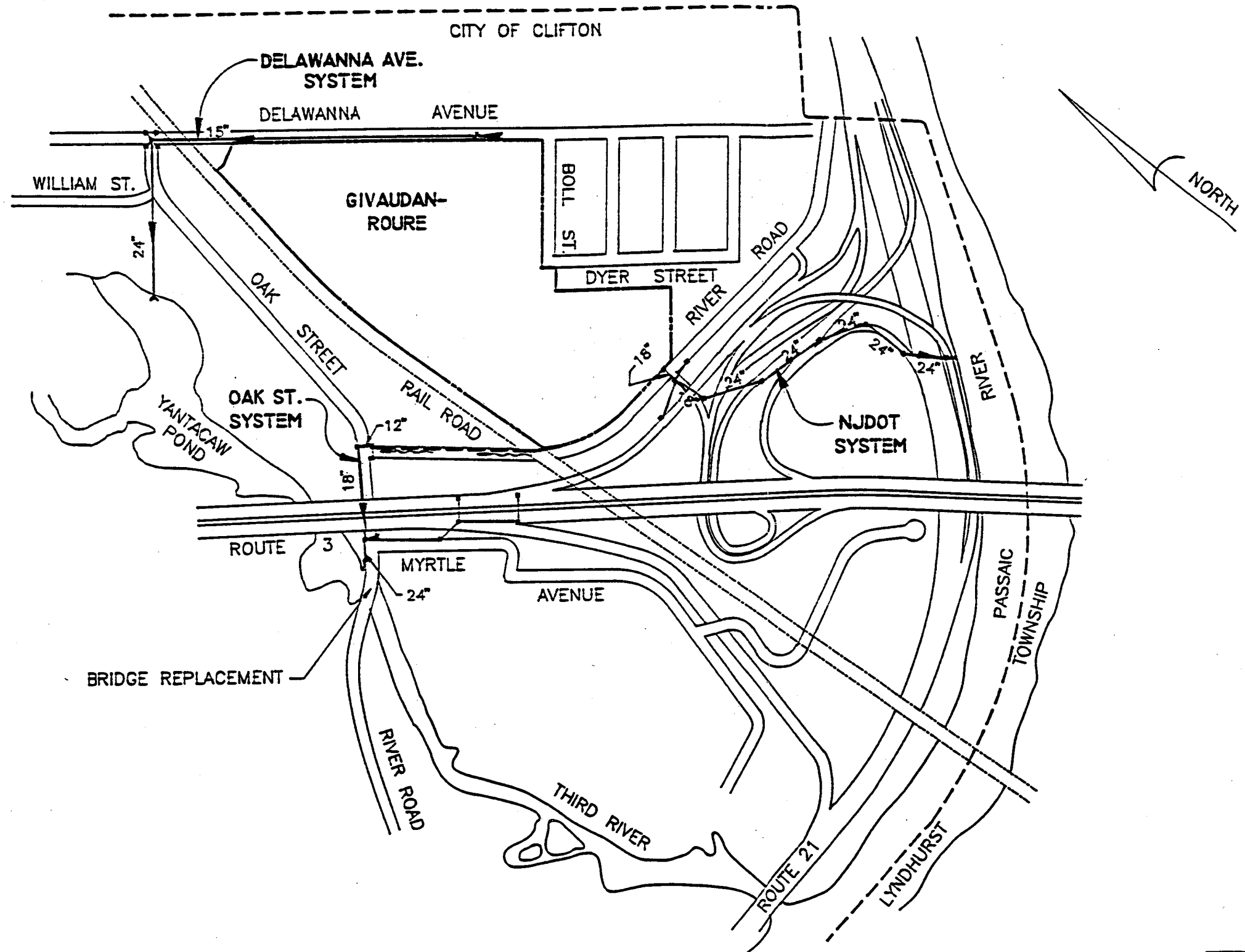


LEGEND

- A12 DELINEATED AREA
- 1 DISCHARGE POINT

EXHIBIT 1

GVAUDAN-ROURE CORPORATION RIVER ROAD, WATER PROBLEM	
DRAINAGE AREA MAP GVAUDAN-ROURE, CLIFTON FACILITY	
Drawn by: J.M. Check by: J.T. Approved by: J.M.	Date: MAY 7, 1983 Scale: 1" = 800' CORPORA ENVIRONMENTAL ENGINEERS INC. 31 North Ocean Drive 121 Highway 31, Suite 800 Farmington, N.J. 08842-1438
DRAWING 1	DATE



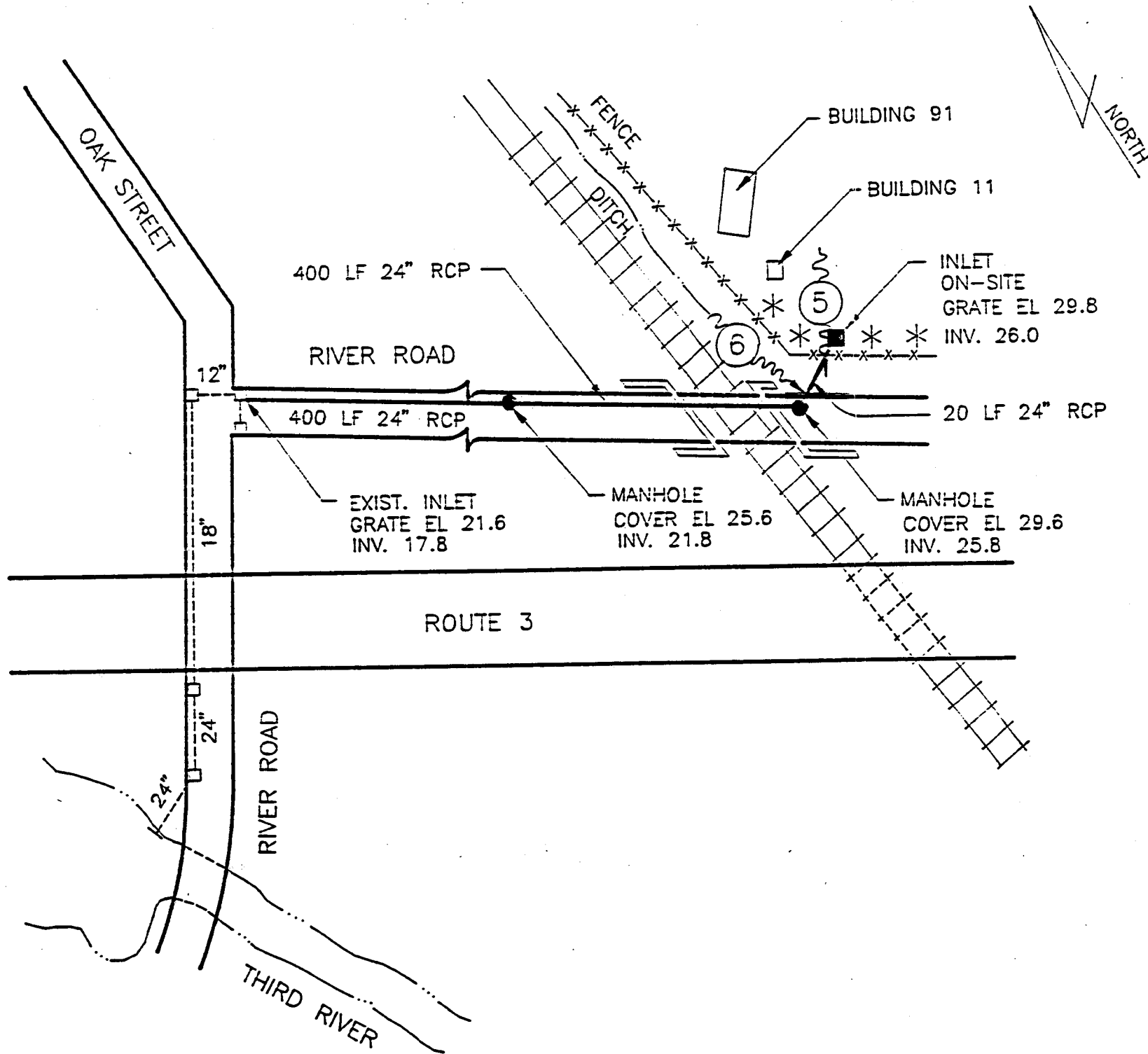
LEGEND

- INLET
- STORMSEWER
- ▶ FLOW DIRECTION
- 15" PIPE DIAMETER
- - - ROADWAY GUTTER

NOTE: ROADWAY GUTTER REFERS TO THE TRIANGULAR GUTTER FORMED BY THE CURB FACE AND THE CROSS SLOPED ROAD SURFACE INTERSECTION.

EXHIBIT 2

GIVAUDAN-ROURE CORPORATION RIVER ROAD, WATER PROBLEM	
DESTINATION LOCATION MAP GIVAUDAN-ROURE, CLIFTON FACILITY	
Date: 05/04 Drawn by: [initials] Checked by: [initials] Approved by: [initials]	Date: MAY 7, 1983 Scale: 1" = 800' CORPUS ENVIRONMENTAL ENGINEERING INC. 31 North 9th Street 121 Monmouth St., Suite 800 Freehold, N.J. 08822-1026
DRAWING	2




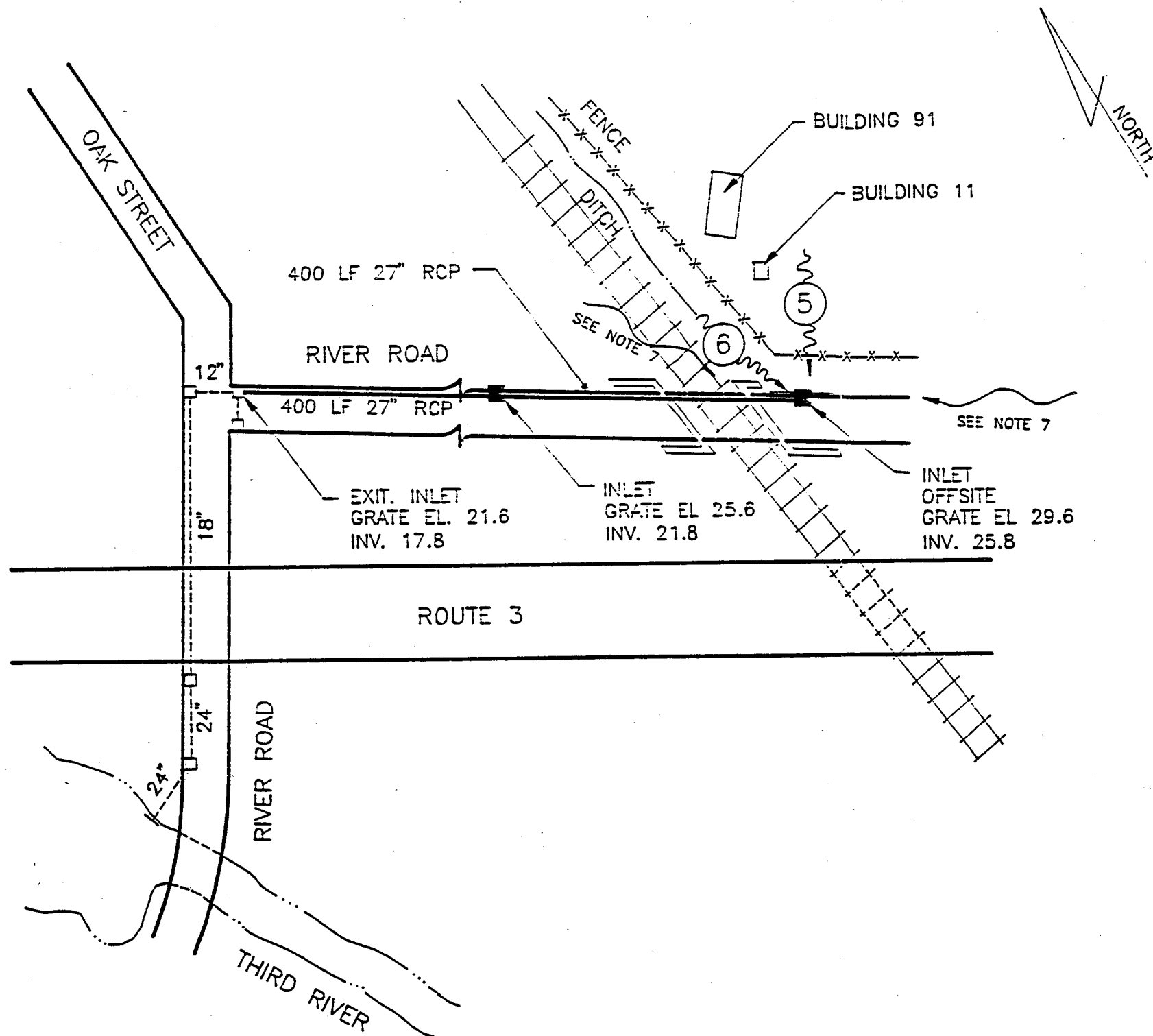
NOTES:

1. ALL LENGTHS, DEPTHS AND ELEVATIONS ARE BASED UPON LIMITED INFORMATION OR GENERAL ASSUMPTIONS WHERE NO INFORMATION AVAILABLE AND ARE FOR INFORMATIONAL PURPOSES ONLY. ALL DATA MUST BE VERIFIED BY FIELD SURVEY FOR ANY DESIGN PURPOSES.
2. PROPOSED PIPE SIZE OF 24" IS BASED UPON ASSUMPTION OF 1% PIPE SLOPE.
3. NJDOT TYPE "E" INLET. (NO CURB PIECE) (3.5' x 4')
4. NJDOT STANDARD MANHOLE (4' DIAMETER)
5. 400' MINIMUM SPACING BETWEEN DRAINAGE STRUCTURES.
6. DOES NOT CONSIDER SUBSURFACE CONFLICTS.

LEGEND

- DISCHARGE POINTS { (6) RR CURB CUT (A14)
 (5) SW CURB CUT (A11)
- REINFORCED CONC. PIPE - RCP

GIVAUDAN - ROURE CORPORATION	
RIVER RD. STORMSEWER IMPROVEMENT (OPTION 6) 17.5 CFS DESIGN FLOW	
Drawn by :	EB
Checked by :	LET
Approved by :	FSJ
Date :	MAY 1993
Scale :	NOT TO SCALE
Project No. :	0102-001-RT
 CORPORATE ENVIRONMENTAL ENGINEERING INC	SKETCH 1
	10201RT6




NOTES:

1. ALL LENGTHS, DEPTHS AND ELEVATIONS ARE BASED UPON LIMITED INFORMATION OR GENERAL ASSUMPTIONS WHERE NO INFORMATION AVAILABLE AND ARE FOR INFORMATIONAL PURPOSES ONLY. ALL DATA MUST BE VERIFIED BY FIELD SURVEY FOR ANY DESIGN PURPOSES.
2. PROPOSED PIPE SIZE OF 27" IS BASED UPON ASSUMPTION OF 1% PIPE GRADE SLOPE.
3. DOES NOT CONSIDER SUBSURFACE CONFLICTS.
4. NJDOT TYPE "B" INLET WITH CURB PIECE. (3.5' x 4')
5. NJDOT STANDARD MANHOLE (4' DIAMETER)
6. 400' MINIMUM SPACING BETWEEN DRAINAGE STRUCTURES.
7. DOES NOT INCLUDE FLOW FROM RIVER ROAD OR R.R. ROW - ONLY WHAT IS GENERATED FROM GIVAUDAN - ROURE PROPERTY.

LEGEND

- DISCHARGE POINTS { (6) RR CURB CUT (A14)
 (5) SW CURB CUT (A11)
- REINFORCED CONC. PIPE - RCP

GIVAUDAN - ROURE CORPORATION	
RIVER RD. STORMSEWER IMPROVEMENT (OPTION 7)	
23.8 CFS DESIGN FLOW (SEE NOTE 7)	
Drawn by :	EB
Checked by :	LET
Approved by :	FSJ
Date :	MAY 1993
Scale :	NOT TO SCALE
Project No. :	0102-001-RT
 CORPORATE ENVIRONMENTAL ENGINEERING INC	SKETCH 2
	10201RT5

APPENDIX A
LIST OF DRAWINGS REVIEWED

LIST OF DRAWINGS REVIEWED FOR THIS REPORT

1. Utilities Services, Property and Fence Lines For Bldgs. # 100, 102, 103,105 &106, Givaudan Corporation, Clifton, New Jersey, dated 7-12-78, Givaudan-Roure No. R-8564
2. Topographic Survey of Property for Givaudan Corporation, prepared by John G. Reutter Associates, dated August 30, 1982, Givaudan-Roure No. A-9267
3. Clifton South Side Site Plan, (Existing Plant Sewer System), Givaudan-Roure Corporation, Clifton, New Jersey, dated 12-23-91, Givaudan-Roure No. PLT-D-014-UT
4. Topographic Survey of Givaudan-Roure Corporation, prepared by GEOD Corp. and revised by Corporate Environmental Engineering, date April 30, 1992, last revised 3-15-93, Givaudan-Roure No. PLT-E-068-GN.
5. New Jersey State Highway Department Plans, Route S-3, 1927, Section 4C, from Passaic Ave. to Passaic River, Federal Aid Project Number F-186(10), fiscal year 1948, sheets 9, 10, 11, available from NJDOT, 2 Route 21, Newark, NJ (Region 2).
6. Clifton Inlet Plan, obtained from the Engineers Office, oversized drawing, no title block, shows schematically, stormsewer drainage, available from the Engineer's Office at Clifton City Hall.
7. Right of Way and Track Map, Morris and Essex Lines, Erie-Lackawanna Railroad Company as of October 17, 1960, station 481+20 to 544+80, dated June 27, 1932 and December 31, 1931, sheets V-1, 94 and 95, available from NJ Transit Rail Operations, 1160 Raymond Blvd., Newark, NJ 07102.
8. Utility Plan, River Road Bridge over the Third River,draft, November 4, 1992, New Jersey Department of Transportation, prepared by EBASCO Services Incorporated, Federal Project number BRM - 7676(101). (Furnished by Givaudan-Roure Corporation.)
9. New Jersey State Highway Department Construction Layout & Landscaping Plan, Route 21 Freeway Section 1C, from Highfield Lane to South Parkway, grading, paving, bridges and walls, Federal Project number U-126(12), various sheets, available from NJDOT, 1010 Parkway Avenue, Trenton, NJ (Design, Region 2).

APPENDIX B
STORMWATER RUNOFF CALCULATIONS

TO BE FURNISHED ON REQUEST

APPENDIX C
OTHER SUPPORTING DOCUMENTS

LIST OF OTHER SUPPORTING DOCUMENTS

1. Memo dated March 2, 1993 from D.J. Hogan, Givaudan-Roure, titled "Stormwater Drainage from Pretreatment Area Construction Site".
2. Letter dated March 4, 1993 from County of Passaic Department of Operations advising Givaudan-Roure of River Road water problem.
3. Letter dated April 8, 1993 from H. Kent-Smith, Esq. of Mason, Griffin & Pierson to Dr. R. Taveras of Givaudan-Roure advising Givaudan-Roure of legal liabilities associated with River Road water problem.
4. Letter dated November 4, 1992 from Corporate Environmental Engineering Inc (CEE) to Givaudan-Roure, titled "Conceptual Closure Plan for the Lagoon" (See p.p. 4-5 for discussion of NJDOT stormwater system status).

To: DISTRIBUTION LIST
From: D.J. HOGAN
Subj: STORMWATER DRAINAGE FROM PRETREATMENT AREA CONSTRUCTION SITE

LATE MONDAY EVENING, MARCH 1, A COMPLAINT WAS RECEIVED BY THE NIGHT SUPERINTENDANT, PHIL HITCHUK, FROM THE CLIFTON DPW THAT RUNOFF FROM THE PLANT WAS CREATING AN ICY CONDITION ON RIVER ROAD NEAR THE CONRAIL OVERPASS. THE DPW WORKER INFORMED THE NIGHT SUPER THAT THIS CONDITION HAD BEEN OCCURRING FOR SOME TIME. THE NIGHT SUPER WAS TOLD TO HAVE SOMEONE FROM GIVAUDAN CONTACT THE DPW OFFICE ABOUT THIS SITUATION.

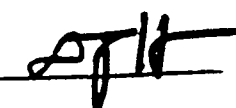
I INSPECTED THE AREA THE NEXT MORNING AT 10:30 AM AND OBSERVED RUNOFF FROM SNOW MELT EXITING THE PLANT IN A 12 INCH WIDE STREAM.

IT APPEARS THAT A PLAN WILL HAVE TO BE DEVELOPED TO HANDLE THIS RUNOFF SINCE IT MAY BE CREATING A DRIVING HAZARD DURING CERTAIN WEATHER CONDITIONS. THE IMPACT, IF ANY, OF THE DPW COMPLAINT ON THE PENDING SITE PLAN APPLICATION CAN NOT BE OVERLOOKED.

INASMUCH AS THERE ARE NO OBVIOUS SOLUTIONS TO THE RUNOFF PROBLEM, CEE WILL BE RETAINED TO REVIEW THE OPTIONS AVAILABLE TO IMPROVE DRAINAGE IN THIS AREA. CEE'S EVALUATION WILL INCLUDE PREPARATION OF CONCEPTUAL PLANS INCLUDING COSTS AND FEASIBILITY DATA.

I WILL MAKE THE FOLLOW-UP TELEPHONE CALL TO THE CITY DPW ON WEDNESDAY.

DISTRIBUTION LIST: R. EBI
H. KENT-SMITH
J. LOPEZ
R. SAUS
R. TAVARES
H. THOMPSON
J. ZGURZYNSKI

D.J. HOGAN 

MAR 2 1993



River Rd. ^{3/2/93} ~~Cliff~~
under tressel.
Bridge ice condition



Entrance to Plant
Complex - River Rd. Cliff
3/2/93



River Road ~~Cliff~~
Plant grounds
4. grounds in Background
3/2/93



River Rd. ~~Cliff~~
water - coming from
property - Linear Tressel
3/2/93

COPY

MASON, GRIFFIN & PIERSON

A PROFESSIONAL CORPORATION
COUNSELLORS AT LAW
625 WASHINGTON STREET
HOBOKEN, NEW JERSEY 07030

JAMES J. BURKE*
HENRY L. KENT-SMITH, OF COUNSEL
*ALSO ADMITTED IN NY

201/222-8000
201/222-3292 (FAX)

MAIN OFFICE:
101 FAVOR FARM ROAD
PRINCETON, NJ 08540
609/921-6543
609/683-7978 (FAX)

: ATTORNEY-CLIENT :
: PRIVILEGED COMMUNICATION :

April 8, 1993

Dr. Robert F. Tavares
GIVAUDAN-ROURE CORPORATION
100 Delawanna Avenue
Clifton, NJ 07015-5034

Re: Givaudan/Clifton
County Roadway Issue

Dear Dr. Tavares:

By letter dated March 15, 1993, you have requested that I research the issue of Givaudan's potential liability for a drainage condition existing on River Road, a Passaic County roadway, that apparently may be emanating from Givaudan's property.

I. GIVAUDAN-ROURE MAY BE LIABLE FOR A HAZARDOUS CONDITION WITHIN THE RIVER ROAD RIGHT-OF-WAY IF THE CONDITION WAS CAUSED BY GIVAUDAN.

The legal principles governing a property owner's liability for a hazardous condition on a public right-of-way emanating from the owner's property are based on well-established principles of common law liability for nuisances. The case of Durant v. Palmer, 29 N.J.L. 544 (E. & A. 1862), first recognized the common law liability of a property owner for a hazardous condition created within a public right-of-way:

By force of common law, every part of the street is so dedicated to the public that any act or obstruction which, when left unprotected, unnecessarily incommodes or impedes its lawful use by the public is a nuisance. . . . The traveling public have a right to suppose that there is no dangerous impediment or pitfall in any part of it [the street].

Durant v. Palmer, 29 N.J.L. at 547-548. The Court went on to hold that where an adjoining owner causes or permits a nuisance to remain within a public right-of-way, the owner is responsible for any injuries or damages sustained as a result of the nuisance. Id. The Durant case involved an obstruction (*i.e.* a hole) within the right-of-way created

COPY

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JAMES J. BURKE*
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Dr. Robert F. Tavares
April 8, 1993
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by the adjoining owner to access his basement, and therefore may be distinguishable from the case *sub judice*. However, the common law principle of liability for a nuisance within a public right-of-way created by an adjoining owner is valid today.

More recent expressions of adjacent property owner liability for hazardous conditions on or within a public right-of-way are found in the context of "sidewalk liability." These cases involve the liability of an adjoining property owner for injuries sustained by pedestrians on adjoining sidewalks. In Davis v. Pecarino, 69 N.J. 1 (1975), and Yanhko v. Fane, 70 N.J. 533 (1976), the Supreme Court enunciated the rule that an adjoining property owner is under no obligation to remedy a hazardous condition that naturally occurs within the public right-of-way; however, if the adjoining property owner has created or aggravated a hazardous condition within the right-of-way through improvements either in the right-of-way or on the owner's property, the property owner is under a duty to mitigate the hazardous conditions created by his use of the property. See Yanhko, 70 N.J. at 531. Note that an adjoining owner has no liability for hazardous conditions *occurring naturally* within a public right-of-way, or that result from a defective design in the road. Id.; Brown v. Brown, 86 N.J. 565 (1981).

Therefore, Givaudan's liability for injuries or damages sustained as a result of the icy condition on River Road would depend on whether or not the icy condition was the result of storm water flow from Givaudan's property into the right-of-way, or if this is a naturally occurring condition or the result of a defective design within the right-of-way. The key element in the analysis would be whether the re-grading and improvements undertaken on Givaudan's property so aggravated or otherwise altered storm water run-off so as to create an off-site condition that would cause an ice sheet to form on River Road. If the run-off from Givaudan's property caused the icy condition, then principles of common law liability attached to hazardous conditions within a public right-of-way created by off-site improvements would subject Givaudan to liability for damage sustained as a result of the ice sheet on River Road. Of course, the injured party must sustain the *prima facie* element of causation as it relates to Givaudan's improvements being the cause of the ice problem.

There are also statutory and regulatory provisions controlling storm water management that may impose potential liability on Givaudan for the River Road situation. The New Jersey Legislature has authorized the completion of the Uniform Site Plan Standards for Non-Residential Development. These standards will provide the basis for all local site plan design standards for non-commercial uses. Therefore, the standards as they relate to storm water management would be highly instructive as to the obligations and potential liabilities of Givaudan with regard to new construction and storm water management.

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Dr. Robert F. Tavares
April 8, 1993
Page 3

The State Storm Water Management Standard in the Uniform Site Plan Ordinance is found in Article VI, Section D. This provision requires all new construction to provide storm water management so that:

Detention will be provided such that after development the peak rate of flow from the site will not exceed the corresponding flow which would have been created by a similar storm prior to development.

State Uniform Site Plan Standards: Article VI-D(5)(d). Therefore, Givaudan's obligation is to manage storm water run-off from any new construction such that the storm water run-off would be the equivalent for the same storm for both pre-construction and post-construction conditions. In this regard, the proposed Phase Two Neutralization Facility should be analyzed as to pre-construction versus post-construction storm water run-off characteristics. The State standard governs not only volume of run-off, but rate of run-off as well, so that although the volume of run-off may be reduced, if the rate is increased, there is a requirement under the State Uniform Standards to provide on-site storm water management so as to reduce the run-off rate to pre-construction velocity.

Both the common law and the soon-to-be-adopted State Site Plan Standards recognize that a property owner has the obligation to provide storm water management so as not to create a hazardous condition on an adjoining right-of-way, and under the State Uniform Site Plan, there is an obligation on the part of an adjoining property owner to provide on-site storm water management so that the off-site storm water flow for pre-construction compared to post-construction conditions are equivalent. Given the above, the analysis of the Givaudan facility should focus on improvements made on Givaudan's property and their impact on the storm water flow as viewed on a *pre-* versus *post-* construction framework. If the difference between pre- and post-construction run-off results in the finding of an increased rate or volume of storm water run-off, then Givaudan has the obligation to mitigate the increased storm water flow by on-site storm water management.

II. PASSAIC COUNTY LIABILITY FOR THE DEFECTIVE DESIGN OF THE RIVER ROAD DRAINAGE SYSTEM.

Passaic County faces potential statutory liability for the River Road situation that may have an impact on Givaudan's liability. *N.J.S.A. 59:4-2* and *4-3* impose liability on public entities where the governmental authority has, or reasonably should have had, knowledge of a dangerous condition on a public right-of-way, which condition was the proximate cause of an injury which was a reasonably foreseeable result of the dangerous

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condition on the right-of-way. In the above instance, the County's liability may attach either by negligent or wrongful act or omission on the part of the County that is the cause of the dangerous condition, or the fact that the County had actual or constructive notice of a dangerous condition on the River Road right-of-way and refused to correct same.

In the case of Brown v. Brown, 86 N.J. 565 (1981), the Court recognized public entity liability for dangerous conditions existing on public roadways caused by the State's "palpably unreasonable" failure to maintain drainage structures. The Brown court recognized that the State is obligated to maintain its roadways so as not to create a dangerous condition. In this instance, the State failed to maintain a drainage swale within the right-of-way thereby causing flooding of the road, a situation that was conceded to create a hazardous condition. 83 N.J. at 570-575.

Of greater moment, the case of McGowan v. Borough of Eatontown infers that an adjoining owner may share liability with a public entity for icy conditions on a public road that emanate from an adjoining property. McGowan, 151 N.J. Super. 440 (App. Div. 1977). The McGowan case is factually analogous to Givaudan's situation. The injured plaintiff sued the adjoining property owner in this action. The property owner settled with the plaintiff and therefore, the court did not discuss or determine the liability of the owner for causing a hazardous condition within the right-of-way. In McGowan, the Court recognized that the State enjoys an absolute immunity for the design of the roadway drainage system. Id. at 446, 447, citing N.J.S.A. 59:4-6. The Court found that there was a question of fact as to whether the State had actual or constructive knowledge of the icy condition on the road and whether the State's failure to salt the ice or provide warning signage was "palpably unreasonable" within the meaning of N.J.S.A. 59:4-2. Id. at 448.

As applied to the situation at hand, Passaic County knows that the icy condition on River Road creates a hazardous condition on the roadway. Therefore, the County is under an obligation to remedy this condition *when resources allow*. See N.J.S.A. 59:4-2. The remedy may be reasonably related to the danger imposed by the condition, such that signage and/or other warning devices may be sufficient, and/or the spreading of salt or other de-icing chemicals on the roadway would be sufficient for the County to have fulfilled its obligations under N.J.S.A. 59:4-2. The County enjoys an absolute immunity for the design of the drainage system for River Road. See N.J.S.A. 59:4-6.

Therefore, in answer to your first question, the County will not be liable for the defective design or plan for handling the River Road drainage, but may be liable for its failure to maintain this drainage system or its failure to address the icy conditions on River Road if this failure is "palpably unreasonable." One means by which the County

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April 8, 1993
Page 5

may address this hazardous condition is to require adjoining property owners (*i.e.* Givaudan) to contribute to the remedy of the drainage condition affecting River Road, to the extent that the adjoining owners contribute to the problem. Therefore, there is an additional impetus to rectify any storm water run-off off-site in this situation.

III. GIVAUDAN MAY BE LIABLE FOR THE NEGLIGENT OR DEFECTIVE DESIGN AND/OR CONSTRUCTION OF ANY IMPROVEMENTS CONSTRUCTED BY GIVAUDAN WITHIN THE RIVER ROAD RIGHT-OF-WAY.

It is important to note that a private property owner who constructs improvements within the public right-of-way, which improvements were either negligently constructed and/or maintained, faces potential liability for any injury resulting from the above negligence. See Furness v. Siquett, 60 *N.J. Super.* 410 (Ch. Div. 1960) (*the negligent maintenance of improvements within a right-of-way by a private owner constitutes a public nuisance*). Essentially, a private property owner cannot create a nuisance within the public right-of-way and is under a duty to exercise reasonable care if the owner constructs a structure within a right-of-way for the purposes of storm water drainage. See Salo v. Hall, 1 *N.J.* 377, 382 (1949) (*owner liable for damages resulting from defective construction and negligent maintenance of a drain pipe in a public sidewalk*). Therefore, Givaudan should avoid constructing any storm water management facilities within the River Road right-of-way. If these facilities are required to adequately address the storm water management issue, I suggest that Givaudan contact the Passaic County Road Department and arrange to have these improvements constructed by the County, with Givaudan contributing its *pro rata* share to the cost of the improvements.

It is critical to the determination of potential liability to ascertain the genesis of the storm water problem on River Road. As the above legal analysis demonstrates, the issue of potential liability is related to the origin of the hazardous condition within the right-of-way, in particular as to whether the hazardous condition originated from the site plan improvements constructed by Givaudan or whether this is a naturally occurring condition on the property.

If you have any questions with regard to the above, please do not hesitate to contact me.

Very truly yours,


Henry L. Kent-Smith

HKS:av
cc:  Donald Hogan

THIS IS AN ATTORNEY-CLIENT
PRIVILEGED COMMUNICATION



CORPORATE ENVIRONMENTAL ENGINEERING INC

31 North Office Centre
121 Highway 31, Suite 600
Flemington, NJ 08822-1635

Phone: (908) 782-4393
Fax: (908) 782-5826

November 4, 1992

SEE PAGE 4 of 5 "STATUS OF 16 INCH CONNECTION"

Mr. Andrei Aroneanu
Project Manager
Givaudan-Roure Corporation
125 Delawanna Avenue
Clifton, NJ 07015-5034

**RE: Conceptual Closure Plan
for the Lagoon
Givaudan-Roure Corporation**

Dear Mr. Aroneanu:

Attached, please find the Conceptual Closure Plan for the Lagoon at the Givaudan-Roure Clifton Facility. This plan was prepared in accordance with our proposal to you dated August 7, 1992 and supplemented by a letter dated September 15, 1992. The order of magnitude Construction Cost Estimate will follow under separate cover.

Please note that the Conceptual Closure Plan was prepared assuming:

1. that the soil to be excavated is not contaminated;
2. that the soil can be disposed as non hazardous construction debris; and.
3. that no sampling of the soil or water will be required.

In the course of preparation, we investigated two issues that required a summary of findings. It may not be appropriate to discuss these issues in the Conceptual Closure Plan. We investigated:

1. The possible affect of the increase in peak rate of stormwater runoff to the existing off-site storm sewer connection as a result of the proposed rerouting and lagoon closure.
2. The permit status of the Givaudan-Roure connection to the existing inlet located in River Road along the southern property line.

CORPORATE ENVIRONMENTAL ENGINEERING INC

Andrei Aroneanu
November 4, 1992
Page 2 of 5

This letter conveys the Conceptual Closure Plan and provides a summary of findings for each of the issues referenced above.

SUMMARY OF FINDINGS

1. Increase in Peak Rate of Stormwater Runoff

During the meeting at your facility to initiate the Conceptual Closure Plan preparation, the rerouting of the storm sewer system was discussed. Accordingly, by closing the lagoon, the storm sewers outfalling into the lagoon would be rerouted to the existing 16 inch storm sewer connected to the inlet in River Road. A rough comparison of the existing drainage area to the lagoon and the existing drainage area to the 16 inch pipe showed almost a 2 to 1 ratio of size. It was expected that the peak rate of runoff at the 16 inch pipe would increase after the lagoon closure and pipe rerouting. As a result, the 16 inch pipe might become inadequate to convey the existing plus the proposed stormwater runoff.

Two options were be discussed. Givaudan-Roure could upgrade the 16 inch pipe. This would require permits from Passaic County and NJDOT in addition to providing support data that the system downstream has the capacity to convey the increase in peak rate of stormwater runoff. The other option would involve installing an inlet on the rerouted piping system at the low area in the southern portion of the Givaudan-Roure facility. The grate elevation of this inlet would be lower than the that of the existing inlet in River Road. In this manner, the 16 inch pipe would continue to convey the current maximum peak rate of flow. When the runoff to the rerouted storm sewer system exceeded this maximum, the excess would surcharge the new inlet at the low point on the Givaudan-Roure property and temporarily flood this area.

At the meeting the second option seemed to be the most feasible. Based upon the experience of the Givaudan-Roure, it was noted that the facility does not have flooding problems. The Lagoon has not overtopped recently and such an occurrence is very infrequent. Site flooding generally consists of large puddles in the relatively flat paved areas. Therefore, it was agreed that this strategy would be incorporated into the Conceptual Plan.

Order of magnitude calculations were prepared to support the selected option. These

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calculations were submitted with the conceptual rerouting of the storm sewers submitted to you and Mr. Frank Slaby on September 14, 1992. These calculations can be summarized as follows:

Drainage area to existing 16 inch pipe -	6.7 Acres	
Drainage area to existing lagoon -	14 Acres	
Total proposed drainage area -	20.7 Acres	
Peak rate of runoff to 16 inch pipe (existing)	- 35 cfs or 15,700 gpm	(25 yr)
	- 30 cfs or 13,500 gpm	(10 yr)
	- 17 cfs or 7,700 gpm	(1 yr)
Peak rate of runoff to lagoon (existing)	- 82 cfs or 36,800 gpm	(25 yr)
	- 73 cfs or 32,760 gpm	(10 yr)
	- 41 cfs or 18,400 gpm	(1 yr)
Peak rate of runoff to 16 inch pipe (proposed)	- 120 cfs or 53,850 gpm	(25 yr)
	- 105 cfs or 47,100 gpm	(10 yr)
	- 59 cfs or 26,500 gpm	(1 yr)
Maximum capacity of 16 inch pipe -	12.5 cfs or 5,600 gpm	

Area in southern portion of property required to temporarily store excess stormwater runoff:

	<u>Cubic Feet</u>	<u>Gallons</u>	<u>Time to Dissipate*</u>
25 year (340'x340'x1')	117,450	878,500	3.2 hours
10 year (315'x315'x1')	99,225	742,200	2.8 hours
1 year (215'x215'x1')	46,462	347,539	1.6 hours

- Duration of storm plus time to empty area when full at maximum flow rate. Actual time will be somewhat longer.

The 1 year storm has been substituted for the 2 year storm calculations discussed in the September 14, 1992. Status of the 16 Inch Connection The time to dissipate assumes that conditions downstream are favorable.

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2. Status of the 16 Inch Connection

The status of the 16 inch connection to the existing inlet in River Road was investigated because Givaudan-Roure did not have knowledge of whether or not NJDOT had written an NJDOT drainage permit for this connection.

The strategy consisted of determining the jurisdiction and trying to find existing plans on which the 16 inch connection is shown without outright asking the Permits Group if a permit exists. In this manner, if the a permit has never been requested for this connection, this information will not be come general knowledge until after Givaudan-Roure has a chance to study the issue.

River Road is a Passaic County Road. Therefore, we contacted the Office of the County Engineer and reviewed what plans the county has. The County has no plans of that area of River Road except for the NJDOT Plans for the construction of Route 21. (Federal Trust Project).

Clifton is part of NJDOT Region II. We contacted the NJDOT design group in Newark and reviewed the plans that group has available. The plans consisted of the NJDOT construction plans for Route 3. (Another Federal Trust Project).

As a result of this investigation, we were not able to find the connection on the plans reviewed. Therefore, we reviewed all the plans available on microfilm for this area at the NJDOT office in Trenton, beginning with the construction of Route 3 in 1938 to the most recent safety improvements in 1975. Again, the 16 inch connection did not appear on any of the reviewed plans. However, the inlet is part of the drainage system for Ramp Number 10 of the Route 21/Route 3 Highway system.

As a last effort, we contacted the permits group to ask if there were any permits for connections to the NJDOT system on Ramp Number 10 of the Route 21/Route 3 Highway system in Clifton. Mr. Tom Kaynak, specifically, was contacted. He immediately knew of the area and that Givaudan had a connection to the inlet in River Road. Mr. Kaynak remembers a connection being discussed about 8-10 years ago. The permit system was started in 1975. He thinks that a permit was issued for the connection. He also stated that if the inlet is not was touched, then he does not need to know about changes upstream.

In a follow up call, Mr. Kaynak stated that he was not able to find a permit and feels that one may not have been issued because the inlet, itself, may be under county jurisdiction even

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though the next inlet down the line is NJDOT's. However, a permit may or may not exist since the NJDOT Trenton office has undergone many changes and the paper work may have been lost.

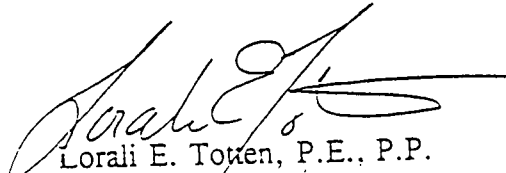
Mr. Kaynak remembers the connection because apparently, NJDEPE was trying to track down some contamination in the Passaic River and contacted NJDOT since the outfall is NJDOT's. He reported that the contamination was from up the line of NJDOT's system, and speculated possibly Givaudan-Roure.

In light of the above, NJDOT knows and has known that the connection exists. The advice is to leave the inlet and 16 inch pipe connection intact without improvements.

If you have any questions, please feel free to contact me. It has been a pleasure working on this project with you.

Sincerely,

CORPORATE ENVIRONMENTAL ENGINEERING INC



Lorali E. Token, P.E., P.P.
Project Manager

Enclosures

cc: File 0062-001-P3, 2.1.1

APPENDIX D

SOILS DATA

TO BE FURNISHED ON REQUEST