

Group 2 4/1/47 - 1/5/53  
Group 2 1/6/53 - 12/3/55  
Group 3 1/1/56 - 7/3/63  
Group 4 9/1/63 - 2/3/70

005079

**CONFIDENTIAL**

SUBJECT TO PROTECTIVE ORDER.

ATTORNEY WORK PRODUCT  
ATTORNEY-CLIENT PRIVILEGE

(NA = Not Applicable)

- 1 Operations eliminated by 1956.
- 2 Plows added by 1958. No centrifuging in Plant 3.
- 3 High exposure applies to Plant 1 only. Plant 3 filter was automated thus low exposure.
- 4 Mechanical arch breaker (vibrating device) added by 1956.
- 5 Conveyors eliminated by 1960.
- 6 Tray dryers used in Plant 1 only. Rotary dryer used in Plants 2 and 3 thus low exposure.
- 7 Concentrations are for inlet stream. Actual cleanout operations would have resulted in exposure to cakes/residues/tars with a much higher TCDD content.
- 8 Exposure levels for Plant 3 are pre-1969. For the last eight months of operation (Jan.-Aug., 1969) changes were made in processing conditions which dramatically lowered TCDD levels; most product lots in the 1-3 ppm range.

hygiene practices improved over the years.

005080

HCG/DLW  
7/28/83

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

- 3/8/49 A runaway reaction in the TCB hydrolysis autoclave at Nitro discharged its contents throughout the area.<sup>5,6,7</sup> Skin disorders appeared in persons working in the area within a few days.
- 5/8/49 A dermatology expert (L. Schwartz) visited the site and examined 25 cases of dermatitis which he diagnosed as chloracne.<sup>251</sup> He cited existing literature reports on such cases. He recommended treatments and suggested methods for further cleanup of the building. He also recommended patients be examined for liver disfunctions.
- 5/23/49 Report was issued by Nitro Research Dept. on lab investigation of the runaway reaction.<sup>201</sup> They were unable to duplicate the runaway but made recommendations for safer operation of the autoclave, particularly for feeding TCB over a period of time to the autoclave after it had been heated to the reaction temperature.
- 8/5/49 A second visit to the plant by Dr. L. Schwartz showed 52 new cases of chloracne since the May 8 visit.<sup>252</sup> All were less severe than the early cases. Most of the early cases showed improvement. He made recommendations for further building cleanup and a number of personnel protective steps: clothing, showering, medication, etc.
- Summer 1950 New TCB hydrolysis unit in Building 51.<sup>5,12</sup> went into operation. Design features reduced worker exposure to process streams (see Section VIII, Chronology of Events, this date).
- 6/16/50 Laboratory attempts were made to identify a tarry material, water insoluble, in NaTCP.<sup>253</sup> They noted that it was produced occasionally when autoclave temperature was on the high side. A portion of the tarry material was a very high melting solid.

Comment: This may well have been, at least in part, TCDD. It would have been partially removed from the process when NaTCP filtration was being practiced.

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

1950-1952 Samples were being taken for evaluation at Mellon Institute (see below).

10/7/52 Dr. R. E. Kelly feels a tarry component in TCB hydrolysis step is responsible for chloracne. 254 Engineering control, not medical control, is the important factor in controlling chloracne. Manufacturing equipment must be designed to avoid all exposure, or the tarry component must be removed from the process. It is his impression that Dow does the latter and has avoided chloracne.

11/52 Results of the "rabbit ear test" on 44 samples submitted in previous months to Mellon Institute were reported.<sup>23</sup> While there were anomalies, a reasonable summary of these results is:

Mild Reaction: NaTCP from distilled TCP or Dow TCP;  
2,4,5-T from regular production;  
2,4,5-T recrystallized.

Marked Reaction: Production or pilot plant NaTCP;  
TCA, some samples.

Severe Reaction: NaTCP from recovered TCP;  
Tar from NaTCP filter;  
Residues from TCP distillations;  
TCA, several samples;  
2,4,5-T from recovered TCP.

Comment: The Mellon studies showed that TCP distillation could remove much of the chloracne, which was concentrated in distillation residues. TCA was also implicated as the chloracne. Memos of transmittal of the samples to Mellon are not available, so exact sample preparations are not known. It is reasonable to assume that most of the TCA samples were prepared by extracting NaTCP solutions with TCE (trichloroethane or trichloroethylene, not specified) and removal of the solvent. Such a procedure would have also extracted the then-unrecognized TCDD which would be left in the TCA. This assumption makes the test results generally consistent.

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

Laboratory results at this time indicated that TCP distillation might be economically feasible since distilled TCP gave better 2,4,5-T yields than the current process, offsetting the distillation losses.<sup>22</sup> Various laboratory investigations aimed at TCA reduction were then proposed.<sup>23,24</sup> These studies were threefold:

- a) Acidifying NaTCP and distilling TCP for 2,4,5-T production with residue disposal to be determined;
- b) Extraction or steam distillation of TCA from NaTCP solution and return of TCA to the autoclave for further reaction to NaTCP;
- c) Further examine the autoclave reaction to reduce amount of TCA made.

Comment: Distillation of TCP would have removed most of the TCDD, and residue disposal would have been a problem. Extraction or steam distillation of TCA and return to the autoclave would also have returned TCDD to the process, and would not have been a forward step.

- 1/6/53 New 2,4,5-T facilities started up.<sup>6,27</sup> Closed operations and improved ventilation were designed to lessen worker exposure.
- 4/14/53 An environmental survey conducted at the plant identified areas for improvement of working conditions.<sup>30</sup>
- 1953 Literature reports of acute toxicity studies of 2,4-D and 2,4,5-T in dogs gave an oral LD<sub>50</sub> of approximately 100 mg/kg.<sup>246</sup>
- 10/3/53 Final report on lab investigations of TCB hydrolysis noted increase in NaOH:TCB molar ratio gave definite reduction in TCA content (TCA thought to be the chloracne).<sup>26</sup> No cases of chloracne in NaTCP production at W. G. Krummrich plant were noted in late 1952 and early 1953. Report recommended minimum contact with process materials.

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

- 12/31/53 Research report issued on 2,4,5-T yield improvement studies.<sup>36</sup> Report recognizes chloracne concentrated in recovered TCP, and work undertaken to increase yields sufficiently to permit economical discard of recovered TCP. Results were not successful.
- 6/8/54 State Department of Health air sampling defined some areas where 2,4,5-T concentrations were highest.<sup>34</sup>
- 7/1/54 Report from Kettering Laboratories of University of Cincinnati (Dr. R. S. Suskind) reported failure of TCA or 2,4,5-T to cause chloracne on rats or human skin.<sup>3</sup> Further work proposed. (The rabbit ear test was not used as it was not considered by Suskind to be realistic or reproducible.<sup>255</sup>)
- Comment: Failure of human skin tests to show chloracne may have been use of too low a concentration. See entries for 11/3/55 and 6/22/56 below.
- 1954 Literature report of acute toxicity of 2,4-D and 2,4,5-T and their various derivatives in rats, mice, guinea pigs and rabbits gave an oral LD<sub>50</sub> in the range of 300-1000 mg/kg.<sup>247</sup>
- 7/27/54 Dr. R. E. Kelly, recognizing further basic research (chloracne testing) will be lengthy and uncertain, recommends improvement in department's exposure potential be made.<sup>256</sup>
- 4/8/55 "Arch breaker" installation in 2,4,5-T dryer feed hopper eliminates exposure in manual rodding operation.<sup>39</sup>
- 11/3/55 Dr. Suskind (Kettering Labs) was able to induce chloracne in human skin tests with Halowax 1014 (penta- and hexachloro-naphthalenes) but not with recrystallized 2,4,5-T.<sup>257,258</sup> No liver disfunctions were noted with either substance.
- 11/17/55 Results of Dr. Suskind's work in attempting to identify chloracnogens were presented to the Workmen's Compensation Commission in Charleston, W.Va.<sup>260</sup>

VII. BASIC CHEMISTRY OF 2,4,5-T PRODUCTION - Page 23

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

11/18/55 Further testing by Suskind is proposed.<sup>259</sup>

12/7/55 Memo from R. E. Soden recognizes chloracne problem has not been solved at Nitro and is outstanding problem in dealing with hourly personnel.<sup>261</sup> He is convinced that Monsanto, Dow and Diamond Alkali experience indicates distillation of TCP will alleviate the problem. Recommends Dr. Suskind's laboratory be engaged to verify whether distillation will solve the problem. Samples were then set to Suskind's Lab.<sup>262</sup>

1/10/56 A second environmental survey of the Nitro plant identified further areas needing improvement in reducing worker exposure.<sup>40</sup>

6/12/56 E. P. Wheeler reported on a visit with Dr. H. Oettel and others at BASF (Badische Anilin-und Soda-Fabrik).<sup>263</sup> BASF had on 11/17/53, a TCB hydrolysis autoclave runaway reaction. Chloracne cases developed. The chloracne was accompanied by fatigue, vertigo, painful joints, etc. Cases seemed worse than those experienced at Nitro. Rabbits exposed in cages in the area died shortly of liver necrosis.

They found liver necrosis in rabbits exposed to the following doses of various substances:

15-20 mg. pentachloronaphthalene (e.g. Halowax);  
1 mg. chlorinated diphenyl ether;  
0.1 mg. residue from TCP distillation;  
0.01 mg. highest boiling fraction of the residue from TCP distillation.

Dr. Oettel had no faith in animal skin tests for chloracne, but BASF tested raw material TCP with animal liver function tests using bromsulfophthalein<sup>264</sup> before using it for 2,4,5-T production.

BASF was also able to reproduce the hydrolysis runaway reaction in the laboratory, but Wheeler did not get details.

Comment: These results apparently were published in 1957 as noted below.<sup>232</sup>

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

6/22/56 New Kettering Lab (Suskind) results were discussed.  
228 All tests used NaTCP applied to human skin compared to Halowax. Tests showed NaTCP produced from distilled TCP or a Diamond Alkali dilution - filtration - precipitation procedure were far less chloroacnegenic than plant NaTCP. No liver disfunction was discerned. The reference also stated that examination of Nitro people in 1949 showed minimal liver damage, and in 1953 none was noted in 35 people examined.

Comment: TCDD would have been present in the plant NaTCP. It would have been largely removed by acidifying, separating and distilling the TCP (remaining in the distillation residue). No adequate description of the Diamond Alkali procedure was found. Presumably the NaTCP solution was diluted and filtered (TCDD probably largely filtered out), and TCP then precipitated by acidification. Some of the remaining TCDD may not have been precipitated with the TCP. Unfortunately, this is speculative without further data.

1957 First published synthesis of TCDD was reported.<sup>265</sup> Dibenzo-p-dioxin and several other chlorinated isbenzo-p-dioxins had been synthesized previously, and generalized procedures for their preparation were known.

1957 Kimmig and Schulz published an investigation of chloracne cases resulting from TCB hydrolysis.<sup>222</sup> (Chemical Abstracts publication in early 1958). Using the rabbit ear test they showed that pure TCP was not chloracnegenic while the commercial product was. They isolated TCDD from the reaction by-products (details lacking) and showed it to be a potent chloracnegen.

1957 H. Th. Hofmann described (apparently) the BASF incident described previously<sup>223</sup> (Chemical Abstracts article in early 1958). Extracts from the carbonized residue of the runaway hydrolysis reaction, and from commercial TCP, produced severe liver damage in rabbits at low dosages. Of chlorine-containing compounds tested for comparison, various chlorinated dibenzo-p-dioxins showed similar toxicity.

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

- 1957 Steps taken in 1957 to reduce worker exposure included installation of a Fletcher centrifuge,<sup>38</sup> reducing fume problems by making a more dilute NaTCP solution prior to the condensation step,<sup>44</sup> and installing a centrifuge plow.<sup>46</sup>
- 4/17/59 Monsanto personnel are not aware of any publication of the BASF runaway reaction.<sup>266</sup> Reference is made to a Thompson-Hayward accident using the ethylene glycol low-pressure process.
- 6/16/59 E. P. Wheeler acknowledges receipt of a translation of the Kimmig and Schulz article,<sup>267</sup> regrets that Monsanto Research and Mellon and Kettering did not discover the TCDD causative agent.
- 3/60 Another industrial hygiene survey was done at Nitro.<sup>57</sup>
- 3/16/60 L. C. Weger describes results of Diamond Alkali runaway hydrolysis on 2/20/60.<sup>268</sup> After 11 days no new cases of chloracne had occurred; personnel entering the area took protective precautions. Process safety information, specifically TCB feed over a period of time, had been given Diamond Alkali by Monsanto the year before, but they had not taken the time to install such a system. Weger suggests points for safety review at Nitro.
- 7/22/60 Dust collector installed at packaging station to reduce dust exposure.<sup>65</sup>
- 9/60 Meeting of Monsanto personnel with Dr. Suskind (Kettering Labs) and Dr. H. Oettel (BASF) reviewed the investigations of the two companies' runaway reactions.<sup>255</sup> BASF's bromsulfophthalein retention test in rabbits as quality control for TCP is described more fully. Dr. Oettel stated Boehringer (in Germany) had experienced a decomposition in NaTCP production and had had many cases of chloracne for years; their TCP failed BASF's bromsulfophthalein test. Oettel and Suskind agreed that the rabbit ear test is unrealistic and non-reproducible.

B. HYDROLYSIS OF TCB

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- 9/8/60 L. C. Weger discussion with Diamond Alkali indicates their work on a chloracne indicator for chlorinated diphenyl ethers.<sup>269</sup>
- 1961 Another article, using rabbit ear and ingestion tests, relates TCDD to chloracne and liver damage; continuation of the 1957 Kimmig and Schulz article.<sup>270</sup>
- 8/63 New integrated facility for NaTCP and 2,4,5-T production started up.<sup>89</sup> A number of provisions for less worker exposure were incorporated.
- 10/22/64 Rabbit ear tests by Younger Laboratories (for Monsanto) showed by-product 2-methoxy-4,5-dichloro-phenol and 4-methoxy-2,5-dichlorophenol (structures in eq. 8, Section VI) to be non-chloracnegenic, and TCP distillation residue was again confirmed as chloracnegenic.<sup>271</sup>
- 2/24/65 In Dr. R. E. Kelly conversation with V. K. Rowe of Dow, Dow has 20 new cases of chloracne.<sup>118</sup> They found 3-10 ppm TCDD in five shipments of 2,4,5-T from Monsanto and were having a management meeting to decide whether to use them. They are convinced TCDD is a major chloracne impurity. They are confident of the rabbit ear test. Dow wants a "crash" meeting with other producers to reach a specification level for TCDD in 2,4,5-T and related products. Dow has had chloracne complaints from customers. Dow has heard that the U.S. Public Health Service is investigating chloracne complaints of Dow customers. Monsanto is unaware of any customer complaints of chloracne, but will investigate.
- 3/3/65 No severe chloracne cases were noted at Nitro 1960-1964; only four cases since new plant startup in mid-1963. No cases of chloracne found at J. F. Queeny Plant in ester manufacture.<sup>272</sup>
- 3/23/65 A TCDD sample and hints on analytical procedure were received from Dow by Monsanto Agricultural Division Research, and GLC method development was started.<sup>120</sup>

B. HYDROLYSIS OF TCB

5. Recognition of TCDD in NaTCP: A Chronology

e. Chronology of the Recognition of TCDD

- 3/25/65 First determination of TCDD in Na 2,4,5-T filter cake was about 10 ppm.<sup>225</sup>
- 4/1/65 TCDD in Na 2,4,5-T filter cake was not altered by NaOCl treatment.<sup>225</sup>
- 4/7/65 The TCDD sample received from Dow was analyzed for C, H, and Cl by Galbraith Laboratories.<sup>225</sup>
- Comment: Analyses do not reveal highest purity TCDD.
- 4/15/65 Filtration of NaTCP showed an 88% reduction in TCDD content.<sup>120</sup> A Dow 2,4,5-T sample had no detectable TCDD in it.<sup>120</sup>
- 6/22/65 This is the date on the Dow Analytical Method (GLC) for TCDD in 2,4,5-T.<sup>273</sup> It involves extraction, concentration, and GLC determination.
- 7/13/65 Distillation of TCP (from plant NaTCP) containing 15 ppm TCDD gave distillate with no detectable TCDD and residue with 400 ppm TCDD.<sup>225,226</sup>
- 9/14/65 Research data showed TCDD in NaTCP at Nitro plant had averaged 5-12 ppm over the period 1953-1964.<sup>125</sup> Some much higher values (to 60 ppm) in April 1965 were unexplained. Four autoclave batches were sampled and analyzed during the hold period. Disappearance of TCB, decrease in TCA and increase in TCP and TCDD contents were plotted. It is concluded that a detailed laboratory study of the autoclave reaction could lead to an appreciable diminution of TCDD formation, and such a study is in the planning stage.
- 10/65 Dow was contacted concerning removal of TCDD and its disposal, but any information obtained was not in the available documentation.<sup>274,275</sup>
- 8/11/66 A number of projects for reduction of dust and fumes in the Nitro plant were undertaken.<sup>140</sup>

B. HYDROLYSIS OF TCB

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e. Chronology of the Recognition of TCDD

- 12/1/66 Dr. R. E. Kelly states that chloracne, while mild, is a continuing problem at Nitro and has occurred at W. G. Krummrich and Luling ester operations.<sup>276</sup> A new industrial hygienist is being hired for Nitro. Although improved equipment and hygiene practices help, the eventual solution must be avoiding manufacture of the chloracnegen.
- 1967 Nitro laboratories were set up for routine analyses of TCDD in 2,4,5-T product, will try to analyze NaTCP also.<sup>277</sup> Research, Industrial Hygiene, and Manufacturing debate further approaches to chloracne problem.<sup>278, 279, 280</sup> Dust and fume elimination projects were completed.<sup>147, 239</sup>
- 2/13/68 Work was started on Research autoclave study to reduce TCDD.<sup>282, 283</sup>
- 6/28/68 Dr. R. E. Kelly urges vigorous pace on TCDD reduction program,<sup>85</sup> asks if it is true Dow and Hercules have no new cases of chloracne, and if an attempt should be made to purchase their knowhow.
- 10/68 Nitro Research process improvement plan calls for studies on TCDD removal by NaTCP filtration and flash distillation in addition to ongoing autoclave studies.<sup>173, 284</sup>
- 1968 Installation of equipment for charging methanol and NaOH under pressure to their autoclave reduced cycle time by two hours and halved the TCDD content.<sup>159, 160</sup>
- 1/69 Results of Agricultural Research autoclave study published.<sup>155, 285</sup> Recommended process called for essentially doubling the methanol: TCB molar ratio and some increase in NaOH:TCB ratio. Predicted TCDD in 2,4,5-T would be consistently below 2 ppm. Immediate testing in the plant autoclave was undertaken.<sup>286, 174, 233</sup> Reduction of TCDD in the NaTCP from 10.5 ppm to 3 ppm was shown in February.<sup>233</sup>

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e. Chronology of the Recognition of TCDD

- 2/10/69 Preliminary report from Bionetics Research Laboratories on teratology studies on 2,4,5-T commissioned by the National Institutes of Health<sup>289</sup> was received by Monsanto. The study showed teratogenic effects and liver damage in rats and mice.
- Comment: Sample used apparently contained 27 ppm TCDD (see below).
- 4/8/69 Dr. R. E. Kelly knows of no tests (animal or human) which would prove that current 2,4,5-T with its low level of TCDD was not chloracnegenic.<sup>287</sup> No letup in preventive measures should be permitted.
- 4/69 Agricultural Division Research monthly summary states TCDD content in NaTCP now 1-3 ppm, and TCDD in 2,4,5-T not detectable.<sup>288</sup>
- Comment: Lower detection limit of analytical method in use at the time was probably about 1 ppm.
- 10/13/69 Department of Defense was not given toxicity data in Fort Detrick's recommendations to use Orange.<sup>290</sup>
- 12/69 Process Amendment authorizes test of shorter TCB addition time and hold time (total autoclave cycle time).<sup>183, 184</sup> These could have further reduced TCDD formation. No documentation shows that tests were made.
- 2/12/70 Dow studies using 2,4,5-T with less than 1 ppm TCDD are showing no teratogenic effect.<sup>185</sup>
- 2/19/70 Meeting was held with Monsanto and Hooker represented.<sup>291</sup> Dow had analyzed 2,4,5-T used in Bionetics study, found 27 ppm, TCDD. It was agreed that a specification for TCDD in 2,4,5-T needs to be set with Federal agencies. GLC methods now in use by 2,4,5-T manufacturers (flame ionization detector) are accurate to about 1 ppm TCDD. A new method (FDA) involving an electron capture detector, much more sensitive, is being investigated.
- 2/24/70 New Monsanto specification was issued for 2,4,5-T including 1 ppm maximum TCDD content.<sup>292</sup>

B. HYDROLYSIS OF TCB

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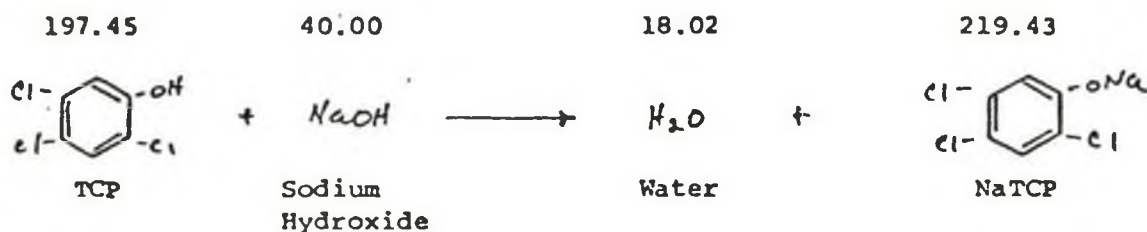
- 2/24/70 FDA and NIH are conducting new teratology studies with commercial 2,4,5-T containing less than 1 ppm TCDD.<sup>293</sup> V. K. Rowe of Dow stated rabbit ear tests showed 2,4,5-T used in Bionetics study (27 ppm TCDD) was chloracnegenic, whereas 2,4,5-T with less than 1 ppm TCDD was not.
- 1/29/71 Reduction of TCDD in a 2,4,5-T ester (specifically the isooctyl ester) from 1-3 ppm to 0.35 ppm by treatment with a specific activated carbon was shown.<sup>294</sup> It was successfully applied to 650,000 lbs. of material in stock.<sup>295</sup>

ATTORNEY WORK PRODUCT  
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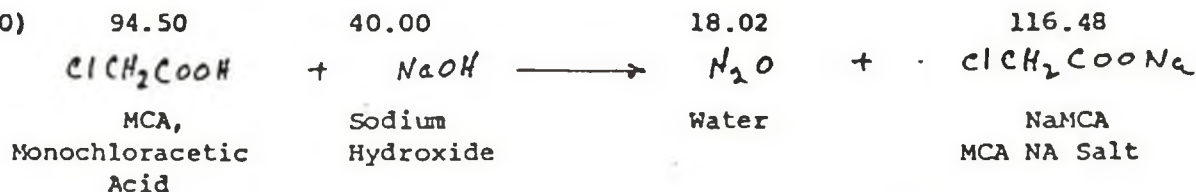
C. CONDENSATION REACTION

2,4,5-T was made by reacting NaTCP with MCA and acidifying to precipitate the solid 2,4,5-T. TCP was converted to Na TCP by equation 9. MCA was converted to NaMCA by reaction with NaOH (eq. 10) or with sodium carbonate (eq. 11). Solutions of the two salts were then reacted to give Na 2,4,5-T (eq. 12). A side reaction (eq. 13) was hydrolysis of some of the NaMCA to sodium glycolate.

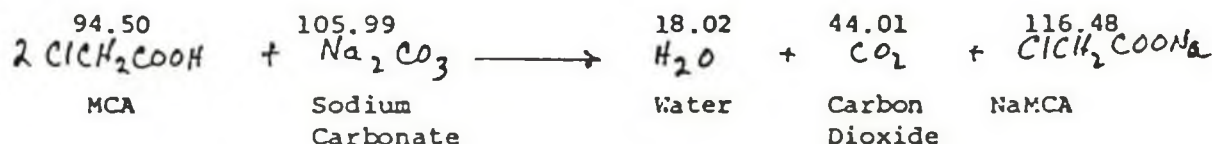
(9)



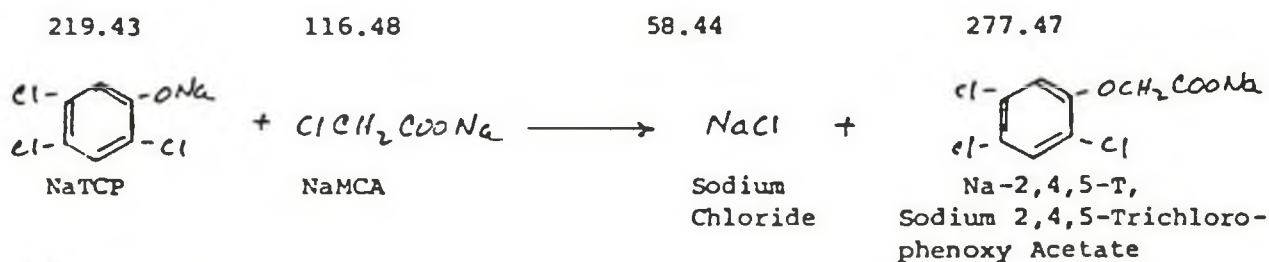
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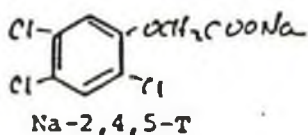
(13) Side Reaction:



D. ACIDIFICATION OF Na 2,4,5-T

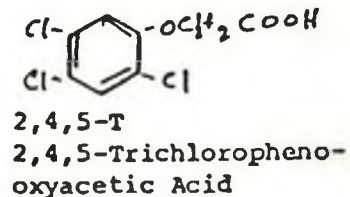
Na 2,4,5-T was acidified (eq. 14) with sulfuric (or hydrochloric) acid to precipitate 2,4,5-T, which was filtered out and dried.

(14)                      277.47                                      98.07                                      142.04                                      255.48



+ H<sub>2</sub>SO<sub>4</sub> →  
Sulfuric  
Acid

Na<sub>2</sub>SO<sub>4</sub> +  
Sodium  
Sulfate



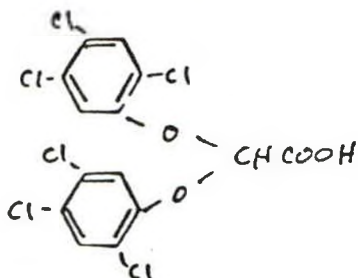
(15) Other Possible Impurities:

(a) In MCA raw material:

Cl<sub>2</sub>CHCOOH  
Dichloroacetic Acid

CH<sub>3</sub>COOH  
Acetic Acid

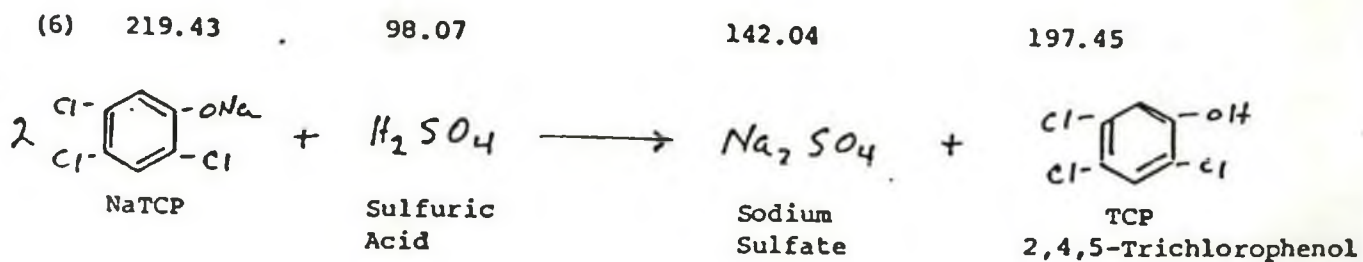
(b) In 2,4,5-T:



" bis-T "  
bis-(2,4,5-Trichlorophenoxy)-Acetic Acid

E. RECOVERY OF TCP

Unreacted TCP in the filtrate after separating out the 2,4,5-T was precipitated by reaction with additional sulfuric acid (eq. 6) and the recovered TCP was recycled to the process.



STURGEON LITIGATION

FROM THE FILES OF M. E. Strauss

THIS FILE HAS BEEN REVIEWED BY C. Conrad

FOR:

- Documents describing the presence or potential presence of dioxin in Monsanto products.
- Documents referring to the toxicity of any Isomer of dioxins.
- Documents referring to the toxicity of any Chlorophenol or Chlorophenol product.
- Documents referring to health effects as they relate to the above.

DATE 4/21/85