

Monsanto

APR 26 1982



FROM : R. T. Parker - Nitro Plant - #507

DATE : April 22, 1982

cc. H. S. Scott - E2ND
T. M. Bistline - E2NJ
H. M. Galloway

SUBJECT : TCDD ANALYSES

REFERENCE : R. T. Parker memo, TCDD
Analyses, 4/13/82

TO : A. M. Ford - A2SA

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SUBJECT TO PROTECTIVE ORDER.

The TCDD levels (40 - 50 ppm) cited in the 1969 TSD Annual Summary do not refer to the amounts of TCDD in the 2,4,5-T product (although this is not entirely clear from the summary). These values refer to the TCDD concentration in the NaTCP mass taken from the autoclave. A progress report by J. C. Strum (Job No. 2982, Rept. 1) dated 2/21/69 is apparently the source for the TSD summary. In the Strum progress report, a value of 40 ppm is reported as typical for the TCDD concentration in NaTCP for the year 1968. The 40 ppm quotation is reported by Strum to be based on an Agricultural Research study on the NaTCP autoclave (it is possible Strum quoted figures from pilot plant material rather than actual Nitro Plant material).

The Agricultural Research study would have been conducted by W. R. Udell. I phoned Udell and he said he was in the process of gathering his information on 2,4,5-T. Upon inspecting some documents at hand, Udell stated the highest value for TCDD in NaTCP was 30 ppm for some plant samples run in January 1969. Udell said he could not remember values running as high as 40 to 50 ppm. Udell should be able to supply you with the documents needed to confirm or deny the information in the TSD Annual Report. Nitro lab data on TCDD levels both in 2,4,5-T and NaTCP were attached to my 4/13/82 memo, and these data do not support the 40 to 50 ppm citation in the TSD Annual Report.

C14883

As a result of looking into this matter, I turned up only three Monsanto chemists involved in TCDD analyses during the time of actual plant production. {1} These were Bill Udell, Phil Balderson and Marty Wilson. Bill Udell stated that he ran no TCDD analyses on 2,4,5-T plant product after the year 1965 (this does not apply to NaTCP intermediate). Also, both Udell and Balderson said they employed an analytical method for TCDD supplied by Dow (this method may have been adapted or modified to suit their particular instrumental/sample needs).

Since the majority of documents regarding 2,4,5-T have been taken to St. Louis, I would suggest these be looked at closely to insure I have not overlooked something.


R. T. Parker

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Attachment

- {1} Some questions arose as to whether Nitro quality control lab personnel ever ran TCDD analyses (see statement in Marty Wilson's notebook, copy attached). Both O. E. Dolin and Phil Balderson do not believe control lab analyst ever ran such analyses. O. E. Dolin kept the TCDD analytical standard (supplied by Bill Udell), and he can not remember giving out the standard for control lab use. W. C. Woods, the Control Lab Supervisor at that time (currently with Marketing), may be able to shed more light on this matter. If control lab personnel did run TCDD analyses, there may be some additional analytical data not recorded in the Nitro research notebooks. Also, T. N. Estep (a co-op student at that time, no longer with Monsanto) had plans to improve the TCDD analytical method (see attached notebook pages). Apparently he never ran any samples or followed up on this issue.

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C14884

Analysis of TDD in TACIL

Objective : To analyze TACIL for % TDD to confirm low level TDD. Question was raised when control lab reported 8 ppm TDD in TACIL.

Procedure

- ① Five gram sample in small vial; add H₂O (several ml) to dissolve sample, shake well.
- ② Add 1 ml CHCl₃ and shake for 10 mins. Let separate and inject. Get from CH layer.
- ③ Standard solution - 3rd size sample from

<u>Samples</u>	<u>TDD in ppm</u>
477	0.6
479	ND
489	0.2
490	0.5
495	0.5
510	0.5
517	0.2

[Signature]
6/13/67

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Statement
in Question
RVP

Copy

T. N. Estep

Notebook 2

1970

TDD in 2,5,4-7

Objective: Try to make improvements on Dan's method of analyzing 2,4,5-7 for TDD.

Background: Dan's method involves extracting TDD from 2,4,5-7 with chloroform and then neutralizing any 2,4,5-7 in the chloroform with 1N. NaOH. The salt subsequently formed is soluble in the water contained in the 1N. NaOH leaving TDD in CHCl_3 layer. After the NaOH ~~neutralizing~~ neutralization step the CHCl_3 layer will be on the bottom after sample centrifugation. This necessitates using a syringe with a long needle to withdraw the CHCl_3 layer. The presence of a solid phase above the CHCl_3 layer poses some difficulty. In order to facilitate the removal of the TDD containing layer benzene will be used instead of CHCl_3 . Benzene will remain on top of the water layer rather than the bottom. A comparison of the two

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170 34
method using CHCl_3 and benzene
was made:

Sample TDD(CHCl_3) TDD(C_6H_6)

Copy

T. N. Estep

Notebook 2

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