9-4-86

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

SEP 4 1986

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: 056227 - Triphenyl Tin Hydroxide,
Response to Registration Standard:
M&T Residue Analytical Method + Residue Data
EPA File Symbol 5204-Q
[Accession Nos. 263218, 263219, 263220, 263221,
263222; RCB No. 1096]

FROM: Susan V. Hummel, Chemist
Special Registration Section II
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

THRU: Edward Zager, Section Head
Special Registration Section II
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

TO: Henry Jacoby, PM #21
Herbicide Fungicide Branch
Registration Division (TS-767C)

M&T Chemicals, Inc. has submitted a residue analytical method for phenyltin species ($\phi_a SnX_{4-a}$) in sugarbeets, soybeans, peanut hulls, carrots, and potatoes, in response to the Registration Standard for the fungicide triphenyltin hydroxide (TPTH). M&T has submitted validation data using tetraphenyltin, triphenyltin hydroxide, diphenyltin oxide, monophenylstannoic acid, and tetrabutyl tin as standards. Similar methods were reviewed in our memos of 2/26/86 (S. Hummel, Accession No. 261251, RCB No. 127) and 7/9/86 (S. Hummel, Accession No. 260289, RCB No. 813). M&T does not have a registered product containing TPTH. M&T has applied for registration of their technical TPTH (EPA File Symbol 5204-AO).

The Registration Standard for TPTH was issued on 9/30/85. M&T has previously submitted Product Chemistry data, which were reviewed in our memo of 8/16/85 (A. Reiter), and updated in our memo of 8/30/85 (A. Reiter). No additional data have been submitted by M&T to fill the remaining product chemistry data gaps.

MRID 160465, 160466, 160467, 160468, 160469

1/18

Triphenyltin hydroxide is a fungicide registered for use on carrots, peanuts, pecans, potatoes, and sugarbeets. Tolerances have been established for residues of triphenyltin hydroxide, per se, in or on peanuts, pecans, and potatoes; and kidney and liver of cattle, goats, hogs, horses, and sheep; at 0.05 (N) ppm, carrots and sugarbeets at 0.1 ppm, and peanut hulls at 0.4 ppm (40 CFR 180.236). Tolerances for residues of TPTH on rice and soybeans are pending (PP#0F2340 and PP#3F2833/ FAP#3H5384, respectively). Tolerances are also pending for eggs, milk, meat, fat, and meat byproducts of cattle , goats, hogs, horses, and sheep (PP#0F2340).

According to the Registration Standard, the TPTH metabolites, diphenyltin oxide and monophenylstannoic acid should be included in the tolerance expression. We recommend that they be calculated as TPTH equivalents.

NOTE TO PM: This change involves review of a significant amount of residue data and should be filed as a petition.

DIRECTIONS FOR USE

Soybeans: A tolerance for residues of TPTH in soybeans is pending. (PP#3F2833/FAP#3H5384). The proposed use is 2 applications per season of 0.24 lb ai/A (1/2 lb of 47.5% WP). There is a 60 day PHI.

Carrots: A foliar spray of a 47.5% WP may be used at the rate of 1.9 to 3.8 oz ai/A. Applications may be made using ground or aerial equipment at 1 week intervals starting 6 weeks after planting, with a 14 day PHI There is no limit on the number of applications per season.

Potatoes: A foliar spray of a 47.5% WP may be used at the rate of 2.38 to 4.75 oz ai/A. Application may be made by ground or aerial equipment, or in sprinkler irrigation system. There is no limit on the number of applications/season. Typically, 5 applications per season would be made at 7 day intervals.

Sugar Beets: A full coverage spray of a 47.5% WP may be made at the rate of 1.9 to 4.75 oz ai/A using ground or aerial equipment. The maximum number of applications per season and the interval between applications is not specified. Normally, 3 to 5 applications would be made at 10-14 day intervals. Sugar beet tops may not be fed to livestock. There is also a grazing restriction.

Peanuts: A full coverage spray, using the 47.5% WP, may be used starting 6 weeks after planting. The registered rate is 2.4 to 3.8 oz ai/A. Applications may be made using ground or aerial equipment at 10 to 14 day intervals. The maximum

number of applications per season is not specified. There is a feeding restriction for the hulls and vines. The feeding restriction for hulls is impractical and should be removed from the label.

Pecans: Use as a delayed dormant spray when leaves are unfolding or as a foliar spray when small nuts are forming. Aerial application may be made at the rate of 0.36-0.71 lb ai/A. Ground application may be made at the rate of 1.5-4.5 oz/100 gal with a full coverage spray (approx. 7.5 - 22.5 oz ai/A). Applications may be repeated at two week intervals. There is no limit on the number of applications per year. Grazing of treated cover crops is restricted.

Information which may reveal the manufacturing process is not included.

PLANT AND ANIMAL METABOLISM

The metabolism of triphenyltin hydroxide in plants and animals is adequately understood. TPTH is not significantly absorbed or translocated in plants. The residue of concern is TPTH, per se, di- and mono- phenyltin oxides and hydroxides, and tetraphenyl tin. [redacted] residues of tetraphenyl tin could potentially concentrate on processing.

ANALYTICAL METHODOLOGY

This submission contains the following reports: "Validation of a Method for the Separation and Determination of Phenyltin Species ($\phi_a SnX_{4-a}$) in Soybeans, by Liquid Chromatography/Atomic Absorption Spectroscopy," No Author, May 15, 1986, M&T Chemicals, Inc. Method No. TA-46. (Accession No. 263222).

"Validation of a Method for the Separation and Determination of Phenyltin Species ($\phi_a SnX_{4-a}$) in Carrots, Sugar Beets, and Potatoes, by Liquid Chromatography/Atomic Absorption Spectroscopy," No Author, May 15, 1986, M&T Chemicals, Inc. Method No. TA-47, (Accession No. 263218, 263219, 263221).

"Validation of a Method for the Separation and Determination of Phenyltin Species ($\phi_a SnX_{4-a}$) in Peanut Hulls, by Liquid Chromatography/Atomic Absorption Spectroscopy," No Author, May 15, 1986, M&T Chemicals, Inc. Method No. TA-48. (Accession No. 263220).

The previously submitted methods were designated M&T method Nos. TA-43 and TA-45.

These methods are reported to be applicable for the analysis for TPTH and its degradation products (diphenyl tin oxide and phenyl stannoic acid) and tetraphenyl tin and inorganic tin from agricultural crops at levels down to 10 or 50 ppb. It was not stated whether this level was ppb Sn or ppb TPTH. However, this level appears to be ppb of the individual organotin compound.

The initial extraction of the three methods differs. In Method No. TA-46 for soybeans, the tin compounds are extracted into methanol/tartaric acid, then extracted into toluene containing tropolone as a chelating agent. In Method No. TA-47 for carrots, sugar beets, and potatoes, a sample is extracted with methanol/methyl isobutyl ketone containing tropolone as a chelating agent. In Method No. TA-48 for peanut hulls, a sample is extracted with methanol/tartaric acid, then extracted into hexane containing tropolone.

The extracts are washed with water. An aliquot of the upper organic phase is evaporated to near dryness. The residue is reconstituted with THF and hexane/tropolone. The tin compounds are then converted to the tetraorganotin compounds by reaction with butylmagnesium chloride (Grignard reagent). Triphenyltin hydroxide (ϕ_3SnOH) is converted to triphenylbutyl tin (ϕ_3SnBu), diphenyltin oxide (ϕ_2SnO) is converted to diphenyl-dibutyltin (ϕ_2SnBu_2), phenyl stannoic acid (ϕ_1SnOOH) is converted to phenyltributyltin ($\phi SnBu_3$). Any inorganic tin present would be converted to tetrabutyl tin ($SnBu_4$). We postulate that any butyl tins present would be converted to tetrabutyltin, as well.

After reaction, the extract is acidified and washed with water. An aliquot is evaporated to near dryness and reconstituted with THF/hexane/tropolone. The tin compounds are separated by liquid chromatography using a reverse phase C8 column and an acetonitrile/ water/tetrahydrofuran (THF) isocratic solvent system. The LC parameters were adequately described. The separation is monitored by UV absorbance at 218 nm. Fractions are collected every 60 seconds. (The previous methods collected three minute fractions.) These fractions are then quantitated by graphite furnace (flameless) atomic absorption spectroscopy at 286.3 nm using a Perkin Elmer Model 603 AA with D_2 background

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NOTE: D_2 background correction is a type of continuum source background correction used in a double beam atomic absorption instrument. A deuterium lamp is used as the continuum source for the reference beam. A hollow cathode lamp (HCL) of the element to be analyzed (Sn, in this case) is the analytical beam. These two beams are passed alternatively through the sample cell (in this case, the graphite tube) using a chopper. The power of the two beams is compared.

correction (see footnote) and an autosampler. The source is a tin hollow cathode lamp (HCL). With the autosampler, the AA trace becomes a chromatogram.

Tetraphenyl tin, triphenyltin hydroxide, diphenyltin oxide, monophenylstannoic acid, and tetrabutyl tin are used to prepare fortified samples and standards. The standards are dissolved in THF, reacted with butylmagnesium chloride to produce the phenylbutyltin compounds which are used as standards for the GF-AA. These standards are used to prepare a calibration curve to be used for quantitation. The sample calculations show that results are to be corrected for control samples. However, the residue data submitted do not appear to have been corrected for controls. The data indicate that both sample controls and solvent controls were analyzed.

The sensitivity of a method by atomic spectroscopy is defined as the concentration of an element which produces a signal of 1% absorption (0.0044 absorbance units) (Skoog and West, Principles of Instrumental Analysis, 2nd Ed., 1979). The sensitivity of this method was reported to be 0.0008, 0.00097, 0.00088, 0.00112, and 0.00076 mg/L for standard solutions of ϕ_4Sn, ϕ_3SnOH, ϕ_2SnO, $\phi SnOOH$, and Bu_4Sn, respectively. The limit of detection (concentration of an element that produces a signal of twice the standard deviation of the background signal) was reportedly tabulated with the recovery data. However, the limit of detection was not reported. Absolute detection limits for non-flame AA methods are typically 10^{-10} to 10^{-13} g (0.01 to 10 ppb for a typical 10 uL sample). Signal to noise ratios for the various crop matrices are tabulated below. The lowest spiking level is somewhat higher than the limit of detection.

		Signal to Noise Ratio (S/N)				
Matrix	Spike (ppb)	ϕ_4	ϕ_3	ϕ_2	ϕ_1	Bu_4
Soybeans	10	6/1	5/1	5/1	5/1	4/1
Potatoes	10	4/1	4/1	5/1	4/1	3/1
Sugar Beets	10	4/1	4/1	3/1	5/1	3/1
Carrots	10	2.5/1	3/1	3/1	6/1	5/1
Peanut Hulls[1]	50	2.5/1	4/1	4/1	3/1	5/1

1/ Note that a smaller size sample of peanut hulls is used

Recovery data are reported in a section labeled, "Validation Procedure," undated, no author. Recoveries were reported for each crop matrix. Control samples were spiked with ϕ_4Sn, ϕ_3SnOH, ϕ_2SnO, ϕ_1SnOOH, and Bu_4Sn. Fortifications are apparently expressed as ppm ϕ_4Sn, ϕ_3SnOH, ϕ_2SnO, ϕ_1SnOOH, and Bu_4Sn. Recoveries are not corrected for controls. Chromatograms

(tracings from the graphite furnace AA) were included, along with raw data sheets. Controls showed no residues above the limit of detection. Reported recoveries are tabulated below.

Crop/Fortification Level (ppm)	% Recovery ϕ_4Sn	ϕ_3Sn	ϕ_2Sn	ϕ_1Sn	Bu_4Sn
Sugar Beets (Accession No. 263218)					
3.0	101	102	109	108	108
1.0	108	92	98	100	89
0.05	130	102	136	118	112
0.01	84	90	84	105	97
Peanut Hulls (Accession No. 263220)					
3.0	104	90	87	95	88
1.0	90	96	99	90	126
0.05	98	124	106	109	94
Soybeans (Accession No. 263222)					
3.0	95	91	95	99	95
1.0	79	88	107	103	93
0.05	109	131	112	92	93
0.01	105	85	124	101	95
Carrots (Accession No. 263218)					
3.0	100	96	99	81	89
1.0	102	82	102	94	85
0.05	91	101	97	91	109
0.01	97	94	105	108	127
Potatoes (Accession No. 263219)					
3.0	107	103	117	110	98
1.0	91	93	98	86	107
0.05	107	87	108	129	91
0.01	104	82	109	89	91

These results are in sharp contrast to the previously submitted validation data which showed residues in control samples approximately five times the level of residues in samples fortified at 50 ppb. The major change in the method is the length of the fractions collected (reduced from three minute fractions to one minute fractions being collected). This major discrepancy should be explained.

No data were submitted to demonstrate that other organotin pesticides will not interfere in this determination. These data are needed. Vendex [hexakis(B,B-dimethylphenethyl)distannoxane] has a tolerance for residues on pecans. Both Vendex

and cyhexatin (Plictran) have tolerances for residues on meat commodities.

This method would not be suitable for the analysis of TPTH and its metabolites in meat, milk, poultry, and eggs, since a base hydrolysis step has not been included. An analytical method for the analysis of TPTH and its metabolites in meat, milk, poultry and eggs is still needed.

STORAGE STABILITY DATA

Soybeans were reportedly spiked with 6 ug TPTH and stored at room temperature. Elsewhere in the report, the soybeans were reportedly spiked with 410 uL of 10 ppm TPTH (4.1 ug). Results were reported as % recovered, rather than ug found, although the raw data sheets, showing calculations of ug found are submitted.

	% Recovery				
Time (days)	ϕ_4Sn	ϕ_3Sn	ϕ_2Sn	ϕ_1Sn	Bu_4Sn
0	-*	86%	-	-	-
3	-	33%	31%	29%	15%
7	-	-	28%	61%	-
30	-	-	-	-	-
90	-	-	-	-	-
180	-	-	-	-	-

*Less than the limit of detection found

Although we question the calculations, this study clearly shows that residues degrade rapidly when samples are stored at room temperature. The residue profile changes with time so that even if the residue analyses in this submission were done within three days of harvest, they would not be acceptable.

We suggest that samples be stored frozen from harvest until analysis. Another storage stability study will be needed using frozen samples. The conditions used in the storage stability study should be the same as those used for the storage of the samples from harvest until analysis. Storage stability data are needed from soybeans or peanuts and from a root crop.

RESIDUE DATA

Insufficient information on the residue data has been submitted. Since the same information is missing from all of the residue data, we will make some general comments on

residue data. The only information submitted about the residue data was the crop tested, the state in which the field trial was conducted, and that "Supertin" was applied. Neither the EPA Reg. No., nor the formula of "Supertin" was submitted.

Dates of planting, treatment, and sampling (harvest) are not given. Weather information was not given. Storage conditions of the samples from harvest to analysis were not given. Dates of analysis were not given. The submitted data cannot be evaluated without the above information and the EPA Reg. No. or formula of the product used.

Crop residues are apparently reported as ppb ϕ_4Sn, ϕ_3SnOH, ϕ_2SnO, ϕ_1SnOOH, and Bu_4Sn. We suggest that residues be reported as ppm TPTH equivalents (ϕ_3SnOH). The tolerance should be expressed as ppm TPTH equivalents.

We note that residues for some fractions, where the signal is clearly more than the limit of detection (twice the standard deviation of the background signal), have not been calculated. This discrepancy should be explained.

Complete residue data should include field trials from all major growing areas for the crop and represent all typical growing seasons. The pesticide should be applied at the maximum registered or proposed rate, with the maximum number of applications allowed per season. The crop should be harvested at the minimum PHI allowed on the label. Data for all types of applications allowed on the label should be made (ground, aerial, etc.). Exaggerated rate data may be needed.

Complete information on the field trials would include
- identification of responsible personnel from planting through writing of final report
- the type and variety of crop
- the formulation used, the formula, and the EPA Reg. No., the percent active ingredient, and the lb ai/gal if appropriate
- the type of formulation used (WP, EC, G, etc.)
- any adjuvants or other pesticides used
- size of field trial plots
- the developmental stage and general condition of the crop at harvest
- method of harvesting
- method of assuring random, representative sample
- dates of planting, pesticide application, number and timing of applications
- complete information on sample handling from harvest to the laboratory to analysis
    - details of any compositing or subsampling
    - Were samples trimmed, cleaned, etc.? Were procedures in PAM I §141-2 followed?

- conditions of storage from harvest until shipping (temperature, humidity, etc.)
- shipping container type, size, etc
- method of shipping, ambient or iced, etc.
- dates of shipping and receipt in laboratory
- dates samples entered storage in laboratory
- any compositing or subsampling

- description of quality control measures

Details needed for evaluation of residue data are outlined in "Hazard Evaluation Division Standard Evauation Procedure: Magnitude of the Residue: Crop Field Trials." We suggest that the registrant obtain a copy of this document through NTIS.

Soybeans (Accession No. 263222)

Applications of "Supertin" were made at 8 and 16 oz/A. There was no indication of the number of applications made.

Field trials were done in undisclosed locations in VA, AL, and TX. These states produce 1, 2, and 1% of the soybean acreage, respectively. Residue data are needed from geographically representative areas where soybeans are grown (refer to Agricultural Statistics). Data are needed from IL/IN, MN/IA, MO/AK, MI/OH, NE/KS, KY/TN, AL/MS/GA, and TX/LA.

We note that residues for some fractions, where the signal is clearly more than the limit of detection (twice the standard deviation of the background signal), have not been calculated. This discrepancy should be explained.

Maximum residues reported are tabulated below. As explained above, these data cannot be evaluated without more information.

	Residue (ppb)				
	ϕ_4	ϕ_3	ϕ_2	ϕ_1	Bu_4
Control	<10	<10	<10	22	<10
8 oz/A	195	190	883	3138	3194
16oz/A	440	349	1274	2559	2473

No data were submitted on soybean processed fractions. These data are needed since finite residues are found on soybean grain. These fractions are meal hulls, soapstock, and crude and refined oil.

Carrots (Accession No. 263218)

One trial was made in undisclosed locations in each of four states, CA, TX, MI, and WA at 7 oz "Supertin"/A. One trial was conducted in TX at 15.2 oz "Supertin"/A. No residues were reported. However, one AA trace shows residue unreported in the data sheets and summary sheet.

As explained above, these data cannot be evaluated without more information. The geographical representation of these data appears to be adequate. However, residue data are needed reflecting multiple applications made every seven days beginning 6 weeks after planting. Data reflecting both ground and aerial application are needed.

Potatoes (Accession No. 263219)

Four trials were conducted in undisclosed locations in ME, ND, WA, and ID. Applications of 9.5 oz and 19.0 oz "Supertin" were made. No residues were reported above a 10 ppb limit of detection. However, three AA traces showed unreported residue above the limit of detection.

As explained above, these data cannot be evaluated without more information. Even if these data are later determined to be acceptable, additional data are needed for spring/summer potatoes from FL, NC/VA, and CA. Data from ME, ND, WA, and ID will be sufficient for winter potatoes.

If finite residues are found in potatoes treated at exaggerated rate equal to the theoretical concentration factor for potatoes processing fractions, a potato processing study will be needed. Potato processed fractions are potato granules, potato chips, and dried potatoes.

Sugar Beets (Accession No. 263221)

Four trials were conducted in undisclosed locations in ND and MN, using 9.5 oz and 19.0 oz "Supertin"/A. These states reflect adequate geographic representation. No residues were reported. However, one AA trace shows unreported residue.

As explained above, these data cannot be evaluated without more information.

If finite residues are found in sugar beets treated at exaggerated rate equal to the theoretical concentration factor for sugar beet processed fractions, a sugar beet processing study will be needed. Sugar beet processed fractions are pulp, molasses, and refined sugar.

Peanut Hulls (Accession No. 261220)

Five trials were conducted in undisclosed locations in GA, AL, VA, and an unknown state. Applications were made at the rates of 9.6 and 19.2 oz "Supertin"/A.

The following residues were reported. As explained above, these data cannot be evaluated without more information.

	Residue (ppb)				
	ϕ_4	ϕ_3	ϕ_2	ϕ_1	Bu_4
Control	<50	<50	<50	<50	<50
9.6 oz/A	<50	<50	<50	<50	<50
19.2 oz/A	71	98	169	<50	<50

A number of AA traces show residues above the limit of detection which were not reported in the data sheets or in the summary sheets.

Even if these data are later determined to be adequate, additional data are needed from TX. For adequate geographical representation, residue data are needed from AL/GA, NC/VA, and TX.

No residue data for peanut nut meats were submitted. These data are needed. No peanut processing study was submitted. These data will be needed if finite residues are found in peanuts treated at an exaggerated rate equal to the theoretical concentration factor for peanut processed fractions. Peanut processed fractions are meal, soapstock, crude and refined oil.

Pecans

No residue data were submitted for pecans. These data are needed.

MEAT, MILK, POULTRY, AND EGGS

No conclusions can be made about residues in meat, milk, poultry, and eggs can be made until the deficiencies in the residue data are resolved.

OTHER CONSIDERATIONS

Most of our conclusions in previous memos on methodology are moot, since this submission supercedes these previous submissions.

The conclusions in our memo of 7/9/86 will be stated here verbatim, followed by the company response and our conclusions.

Deficiencies

We recognize that additional methodology and residue data have been submitted and will be reviewed shortly. Upon review of the additional data our conclusions may change.

1. It was not clear whether the stated limit of detection was expressed as ppb Sn or ppb TPTH. It appears that the limit of detection was intended to be expressed as ppb TPTH. This should be clarified. The stated limit of detection may or may not be adequate to enforce tolerances after they are reevaluated to include the mono-, di-, and tetra- phenyltin species.

2. The sample treatment of the control samples was not described. It is unclear if the controls were plant controls or reagent control. RCB normally does not accept residue analytical methods requiring correction for untreated control samples, since untreated control samples are not available for enforcement purposes. The recoveries calculated without correction for control samples are unacceptably high.

3. Diphenyltin oxide or hydroxide and monophenyl-stannoic acid should be used for the preparation of the standards and fortified samples. Alternatively, the registrant should demonstrate that the di- and mono-phenyltin chlorides have recoveries and responses comparable to those of the oxides and hydroxides. Sources of the standards for TPTH and mono- and di- phenyltin species should be stated. These standards should be made available from the EPA Pesticide Repository.

4. The registrant should demonstrate that the method is applicable to the determination of tetraphenyltin, since residue data reflecting analysis for tetraphenyltin are required for processed commodities of sugarbeets, potatoes, and peanuts.

Registrant Response

The registrant submitted a new analytical method incorporating all of these comments.

RCB Conclusion

These deficiencies are moot.

Deficiency

5. No data were submitted to demonstrate that other organotin pesticides will not interfere in this determination. These data are needed.

6. This method would not be suitable for the analysis of TPTH and its metabolites in meat, milk, poultry, and eggs, since a base hydrolysis step has not been included. An analytical method for the analysis of TPTH and its metabolites in meat, milk, poultry and eggs is still needed.

Registrant Response

These deficiencies were not addressed.

RCB Conclusion

These deficiencies remain outstanding.

Deficiency

7. Storage Stability data were not included in this submission. These data are needed.

Registrant Response

Storage Stability data were submitted.

RCB Conclusion

The submitted storage stability data are inadequate. For further discussion, see the Storage Stability data section of this review.

CONCLUSIONS

1. According to the Registration Standard, the TPTH metabolites, diphenyltin oxide and monophenylstanneic acid should be included in the tolerance expression. We recommend that they be calculated as TPTH equivalents.

2. The feeding restriction for peanut hulls is impractical and should be removed from the label.

3. The validation results included in this submission are in sharp contrast to the previously submitted validation data which showed residues in control samples approximately five times the level of residues in samples fortified at 50 ppb. The major change in the method is the length of the

fractions collected (reduced from three minute fractions to one minute fractions being collected). This major discrepancy should be explained.

4. No data were submitted to demonstrate that other organotin pesticides will not interfere in this determination. These data are needed. Vendex [hexakis(B,B-dimethylphenethyl)-distannoxane] has a tolerance for residues on pecans. Both Vendex and cyhexatin (Plictran) have tolerances for residues on meat commodities.

5. This method would not be suitable for the analysis of TPTH and its metabolites in meat, milk, poultry, and eggs, since a base hydrolysis step has not been included. An analytical method for the analysis of TPTH and its metabolites in meat, milk, poultry and eggs is still needed.

6. The storage stability study included in this submission clearly shows that residues degrade rapidly when samples are stored at room temperature, although we question the calculations. The residue profile changes with time so that even if the residue analyses in this submission were done within three days of harvest, they would not be acceptable.

We suggest that samples be stored frozen from harvest until analysis. Another storage stability study will be needed using frozen samples. The conditions used in the storage stability study should be the same as those used for the storage of the samples from harvest until analysis. Storage stability data are needed from soybeans or peanuts and from a root crop.

7. Complete residue data should include field trials from all major growing areas for the crop and represent all typical growing seasons. The pesticide should be applied at the maximum registered or proposed rate, with the maximum number of applications allowed per season. The crop should be harvested at the minimum PHI allowed on the label. Data for all types of applications allowed on the label should be made (ground, aerial, etc.). Exaggerated rate data may be needed.

Complete information on the field trials would include
- identification of responsible personnel from planting through writing of final report
- the type and variety of crop
- the formulation used, the formula, and the EPA Reg. No., the percent active ingredient, and the lb ai/gal if appropriate
- the type of formulation used (WP, EC, G, etc.)
- any adjuvants or other pesticides used
- size of field trial plots
- the developmental stage and general condition of the crop at harvest

° method of harvesting
° method of assuring random, representative sample
° dates of planting, pesticide application, number and timing of applications
° complete information on sample handling from harvest to the laboratory to analysis
    ° details of any compositing or subsampling
    ° Were samples trimmed, cleaned, etc.? Were procedures in PAM I §141-2 followed?
    ° conditions of storage from harvest until shipping (temperature, humidity, etc.)
    ° shipping container type, size, etc
    ° method of shipping, ambient or iced, etc.
    ° dates of shipping and receipt in laboratory
    ° dates samples entered storage in laboratory
    ° any compositing or subsampling
° description of quality control measures

Details needed for evaluation of residue data are outlined in "Hazard Evaluation Division Standard Evauation Procedure: Magnitude of the Residue: Crop Field Trials." We suggest that the registrant obtain a copy of this document through NTIS.

We note that residues for some fractions, where the signal is clearly more than the limit of detection (twice the standard deviation of the background signal), have not been calculated. This discrepancy should be explained.

7a. Residue data on soybeans are needed from geographically representative areas where soybeans are grown (refer to Agricultural Statistics). Data are needed from IL/IN, MN/IA, MO/AK, MI/OH, NE/KS, KY/TN, AL/MS/GA, and TX/LA.

No data were submitted on soybean processed fractions. These data are needed since finite residues are found on soybean grain. These fractions are meal hulls, soapstock, and crude and refined oil.

7b. The geographical representation of the carrot data appears to be adequate. However, residue data are needed reflecting multiple applications made every seven days beginning 6 weeks after planting. Data reflecting both ground and aerial application are needed.

7c. Even if the potato data are later determined to be acceptable, additional data are needed for spring/summer potatoes from FL, NC/VA, and CA. Data from ME, ND, WA, and ID will be sufficient for winter potatoes.

If finite residues are found in potatoes treated at exaggerated rate equal to the theoretical concentration factor for potatoes processing fractions, a potato processing study

will be needed. Potato processed fractions are potato granules, potato chips, and dried potatoes.

7d. The sugar beet data appear to have adequate geographic representation.

If finite residues are found in sugar beets treated at exaggerated rate equal to the theoretical concentration factor for sugar beet processed fractions, a sugar beet processing study will be needed. Sugar beet processed fractions are pulp, molasses, and refined sugar.

7e. Even if the peanut hull data are later determined to be adequate, additional data are needed from TX. For adequate geographical representation, residue data are needed from AL/GA, NC/VA, and TX.

No residue data for peanut nut meats were submitted. These data are needed. No peanut processing study was submitted. These data will be needed if finite residues are found in peanuts treated at an exaggerated rate equal to the theoretical concentration factor for peanut processed fractions. Peanut processed fractions are meal, soapstock, crude and refined oil.

7f. No residue data were submitted for pecans. These data are needed.

8. No conclusions can be made about residues in meat, milk, poultry, and eggs until the deficiencies in the residue data are resolved.

RECOMMENDATIONS

We recommend that the registrant be informed of our conclusions and advised to submit data to satisfy the deficiencies outlined in our conclusions.

cc: R.F., Circu., S. Hummel, Triphenyltin Hydroxide SF, PP#3F2833, Triphenyltin Hydroxide SRF (Hummel), Reg. Std. File (W. Boodee), PM#31, PMSD/ISB
RDI: EZ:9/03/86:RDS:9/03/86
TS-769C:RCB:SVH:svh:Rm.710A:CM#2:557-3045:9/03/86

JUN 24 1986

M & T Chemicals, Inc.
Rahway, NJ 07065-0970

Attention: A.W. Sheldon

Gentlemen:

Subject: Data Submitted Under the TPTH Registration Standard
Your Letter Dated June 6, 1986

The data submitted under your letter of June 6, 1986 were assigned the following EPA Accession Number(s):

EPA Accession Number(s)	Title of Report(s)
263222	Validation of a method for the separation of phenyltin species in soybeans by L.E./A.A.S.
263221	Validation of a method for the separation and determination of phenyltin species in sugar beets.
263220	Validation of a method for the separation and determination of phenyltin species in peanut hulls.
263219	Validation of a method for the separation and determination of phenyltin species in potatoes.
263218	Validation of a method for the separation and determination of phenyltin species in carrots.

88748:Forrest:J-10:KENCO:6/20/86:6/27/86:dej:LMF

CONCURRENCES

SYMBOL ▶								
SURNAME ▶								
DATE ▶								

EPA Form 1320-1 (4-81) OFFICIAL FILE COPY

In future correspondence regarding these data, you should reference the assigned EPA Accession Number to facilitate our retrieval of these data. The requested regulatory action must await completion of the review of the data submitted.

Sincerely yours,

Henry M. Jacoby
Product Manager (21)
Fungicide-Herbicide Branch
Registration Division (TS-767C)