Translation of Report from

SCHERING AG
PLANT PROTECTION RESEARCH
Analytical and Biochemistry

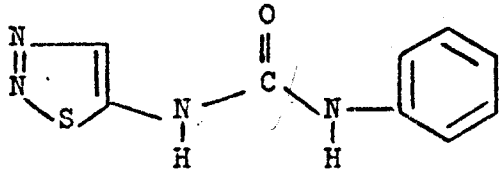
Berlin, 11/20/75
Dr. Ottnad/Wi
PA 49 537.51/5

49 537/1

Residue Determination of SN 49 537 in Cottonseed and Cotton Fiber (Preliminary Method)

1. Active Ingredient

SN 49 537



MW 220.25

5-N-Phenylcarbamoylamino-1,2,3-thiadiazole

2. Principle of the Method

Pulverized cottonseed or cotton fiber were subjected to an alkaline hydrolysis. Then the aniline formed from the active ingredient was isolated by continuous steam distillation and extraction with isooctane in a Bleidner Apparatus. The aniline was extracted with acid, brominated and quantitatively determined as 2,4,6-tribromo-aniline on a gas chromatograph with an electron capture detector.

3. Apparatus and Reagents

Bleidner distillation/extraction attachment, modified by Heizler (ref.: J. Agr. Food Chem. 19, 366 (1971)). A ground glass joint (NS 35) was used on the "water side".

Heating mantles for 250 ml (isooctane side) and 2 l (water side) round bottom flasks.

Centrifuge

Gas chromatograph, Hewlett-Packard 5710 A with Ni 63 electron capture detector.

3. Apparatus and Reagents (cont'd)

Antifoam A, Dow Corning

Isooctane, distilled

Potassium bromate solution, 0.2% with $KBrO_3$ AR (Riedel-de Haen, Seelze, Hannover) in water

Potassium bromide AR (Riedel-de Haen, Seelze, Hannover)

Sodium hydroxide AR (E. Merck, Darmstadt)

Sodium sulfate AR (Riedel-de Haen, Seelze, Hannover)

Sodium sulfite AR (E. Merck, Darmstadt)

Sodium hydroxide 5 N with NaOH AR (E. Merck, Darmstadt)

Hydrochloric acid 1 N with conc. HCl AR (Riedel-de Haen, Seelze, Hannover)

Toluene AR (E. Merck, Darmstadt)

2,4,6-Tribromoaniline, produced during the bromination of aniline with potassium bromide/potassium bromate in hydrochloric acid solution.

2,4,6-Trimethylaniline solution, 0.2% w/v in 1 N HCl, made from 2,4,6-trimethylaniline, pure (Fluka AG, Buchs, Switzerland), chromatographed on basic aluminum oxide, activity grade I (Camag).

Purity requirement: 10 ml of a solution of 200 mg 2,4,6-trimethylaniline in 100 ml 1 N HCl was brominated according to Section 4.2 and extracted with 10 ml toluene. 2 µl of the extract was injected into the gas chromatograph. The gas chromatogram may show only a very small peak at the retention time of 2,4,6-tribromoaniline. This must not exceed a peak area equivalent to 0.01 ng 2,4,6-tribromoaniline.

4.1 Hydrolysis and Distillation/Extraction

25 g cottonseed or 10 g finely cut cotton fiber were weighed into a 2 l RB flask. Add 1-2 g Antifoam A, 1000 ml water and 100 g sodium hydroxide AR. After addition of boiling chips the flask was attached to the water side of the Bleidner Apparatus.

A 250 ml RB flask containing 110 ml isooctane was attached to the isooctane side; the middle of the Bleidner Apparatus was filled with water; and finally, a water cooled reflux condenser was attached. Both RB flasks were heated to reflux temperatures with heating mantles. The distillation rate of each liquid should be adjusted to allow approximately the same rate of liquid flow back through the water and isooctane arms. The distillation-extraction was continued for 3 hours. After cooling the entire isooctane phase was transferred to a separatory funnel. By extracting three times with 1 N hydrochloric acid (10, 5 and 5 ml., respectively) the resulting aniline is transferred to a water phase which is suitable for the bromination reaction. The hydrochloric acid extract quantitatively transferred to a 100 ml erlenmeyer with ground glass stopper.

4.2 Bromination

To the aqueous extract from step 4.1 was added 12 g potassium bromide AR and 0.5 ml of trimethylaniline solution ($\hat{=}$ 1 mg trimethylaniline in 1 N HCl). Shake about 5 minutes to dissolve the potassium bromide and then add 0.5 ml potassium bromate solution ($\hat{=}$ 1 mg potassium bromate). The reaction mixture will become yellow colored. If decolorization occurs add another 0.5 ml potassium bromate solution until a stable yellow color is observed.

The reaction was stopped after 30 minutes by addition of sodium sulfite (about 100 mg) until decolorization of the solution is complete. The bromine atmosphere above the reaction mixture was purged with air and the reaction mixture was allowed to stand for 15 minutes.

Make alkaline by adding 5 ml 5 N sodium hydroxide and then pipet into the reaction mixture 10.0 ml toluene. Close the flask and shake vigorously for 2 minutes. Allow the reaction mixture to separate into layers and pipette off the toluene solution. 1 µl of toluene solution corresponds to 2.5 mg seed or 1 mg cotton fiber.

5. Gas Chromatographic Analysis of 2,4,6-Tribromoaniline

Instrument: Hewlett-Packard 5710 A GLC equipped with a Ni^{63} electron capture detector.

Column: 2.2 m (2.5 mm i.d) glass column filled with 10% SE-30 on Chromosorb W-AW-DMCS, 80-100 mesh, conditioned for 16 hours at 220°C.

Column temperature: 180°C

Injection port temperature: 250°C

Detector temperature: 350°C

Carrier gas: 90 ml/min purified nitrogen

Purge gas: 60 ml/min argon/methane 90/10

Retention time of 2,4,6-tribromoaniline: 3.6 min.

Detection limit: 2 pg 2,4,6-tribromoaniline

6. Evaluation

6.1 Reference Values

Evaluation of the chromatograms results from comparison of sample peak area with the peak area of the standard solution. The standard solution consists of 50 mg 2,4,6-tribromoaniline dissolved in 50 ml toluene and this solution is diluted with toluene to give concentrations of 0.1 ng/2 µl, 0.08 ng/2 µl, 0.06 ng/2 µl, 0.04 ng /2 µl and 0.02 ng/2 µl. Peak areas are plotted against the above amounts of injected 2,4,6-tribromoaniline, which gives a straight line plot. Two µl was usually injected.

6.2 Calculation of Residue and Recoveries

The amounts of 2,4,6-tribromoaniline in nanograms (b) corresponding to the total peak area was taken from the standard curve. 1 nanogram of 2,4,6-tribromoaniline is equivalent to 0.688 ng SN 49 537. The residue is

$$\frac{b \cdot 0.67}{5} \text{ ppm} \quad \text{or} \quad \frac{b \cdot 0.67}{2} \text{ ppm}$$

for a 2 µl injection, which corresponds to 5 mg seed or 2 mg cotton fiber.

Analyses using the above procedure resulted in the following recoveries from untreated cottonseed.

SN 49537, 4 ppm: 105 ± 8.5% (96-120%).

(These values are corrected for a bromination recovery of 100%).

7. Discussion

Check values in the range of 0.2 ± 0.01 ppm were obtained in the analysis of cottonseed. It appears that the determination limit of SN 49537 in cottonseed is 0.05 ppm.

Signed: Dr. Ottnad

NAJ/be
3/3/76

MODIFICATION OF RESIDUE METHOD 49537/1

(Evaluation of SN 49537 residues using a Hewlett-Packard 3380 A Recording Integrator)

As an alternate procedure to section 6.2 of method 49537/1 a Hewlett-Packard 3380 A Recording Integrator can be used to automatically integrate the 2,4,6-tribromoaniline peak and compute ppm of SN 49537 residue. The GLC procedure and operating conditions remain the same except for integration of the peaks.

Integrating recorder operating conditions are as follows: (see attached chromatograms)

Function:	Report as needed	Start Delay (Min)	Stop Timer (Min)	Area Reject	Chart Speed (cm/min)	Chart	Slope Sensitivity (MV/min)	Attenuation
Position:		4	10	10^3	0.5	Auto	0.3	256*

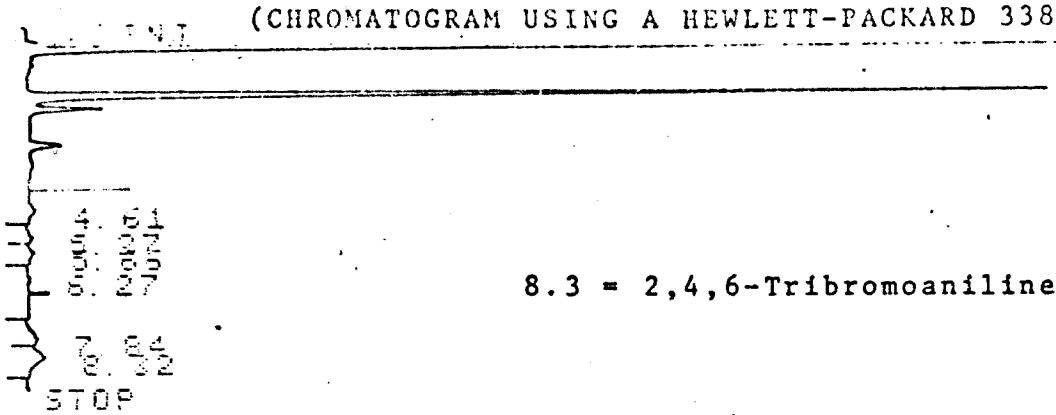
* Note: Attenuation is usually set at 256 but may be changed as needed.

Signed: Dr. N. Jenny

D-2

BAJ/jcf
7/25/76

COTTONSEED CHECK SAMPLE
(CHROMATOGRAM USING A HEWLETT-PACKARD 3380 A INTEGRATOR)



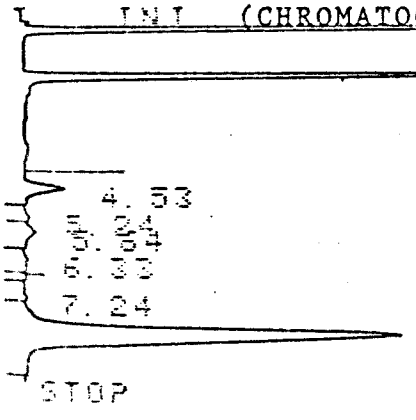
8.3 = 2,4,6-Tribromoaniline

RT	TYPE	AREA	ESTD ID#	AMT
4.61	TM	30295		.
5.27	TM	13769		.
5.66	TM	18279		.
5.27	TM	5631		.
7.84		41405		.
8.32	M	79240	1	.021 43
			TOTAL	.021 43

F 1.

P 3380A
LY 4. STOP 10 REJECT 1000
V/M .30 ATTN 256

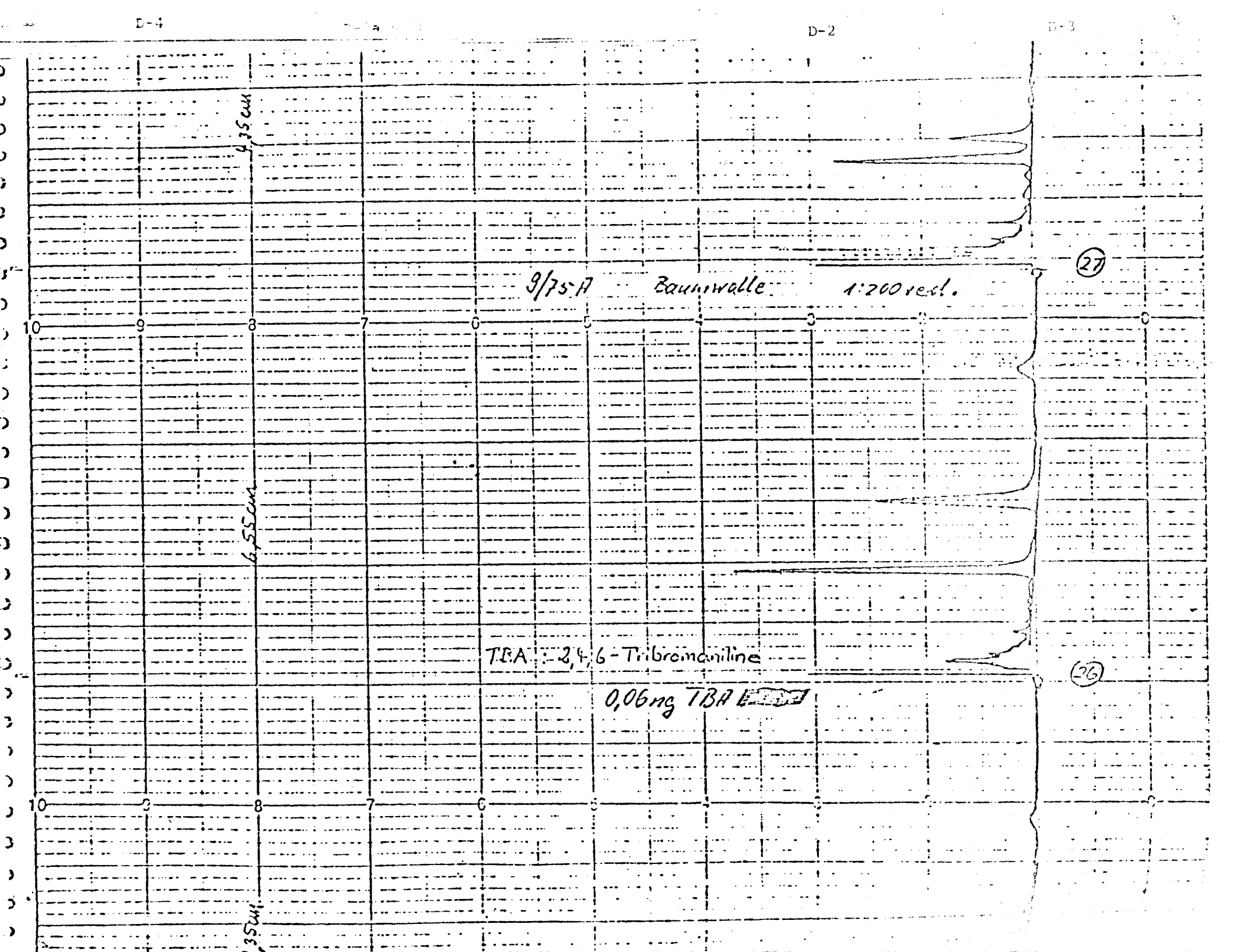
0.5 ppm SN 49537 STANDARD
INT (CHROMATOGRAM USING A HEWLETT PACKARD 3380A INTEGRATOR)



8.3 = 2,4,6-Tribromoaniline

RT	TYPE	AREA	ESTD ID#	AMT
[illegible]	TM	130574		.
[illegible]	TM	15898		.
[illegible]	TM	43858		.
[illegible]	TM	7416		.
[illegible]		4577		
[illegible]	M	1365572	1	.304 6
			TOTAL	.304 6

STOP 10 REJECT 1000
ATTN 256


D-4
D-2
D-3
9/75:A Baumwalle 1:200 red.
27
26
TBA: 2,4,6-Tribromanilin
0,06 mg TBA

NOR-AM Agricultural Products, Inc.
Woodstock, Illinois

May 17, 1976
Dr. NAJ/jcf

Blanks and Recoveries Using SN 49537 Residue Method 49537/1

A. Cottonseed Blanks (Calculated as SN 49537)

Rpt. No.	Origin	Dates of Analysis	Peak Area[1] Integrator Counts	Number of Tests	Average ppm SN 49537
49537/NA 1	Texas	4/7/76-4/19/76	63000 ± 2000	3	0.018 ± 0.001
49537/NA 3	Texas	4/8/76-4/19/76	70000 ± 27000	3	0.019 ± 0.007
49537/NA 7	Texas	4/9/76-4/20/76	93000 ± 33000	3	0.027 ± 0.008
49537/NA 8	Georgia	4/13/76-4/21/76	86000 ± 27000	3	0.024 ± 0.007
49537/NA 9	Georgia	4/13/76-4/21/76	73000 ± 6000	3	0.020 ± 0.002
49537/NA 10	California	4/14/76-4/23/76	81000 ± 16000	3	0.023 ± 0.004
49537/NA 11	California	4/15/76-4/26/76	39000 ± 17000	3	0.012 ± 0.005
49537/NA 12	California	4/15/76-5/3/76	81000 ± 8000	3	0.026 ± 0.002
49537/NA 13	Texas	4/30/76-5/5/76	62000 ± 24000	3	0.018 ± 0.007

1) Peak areas are recorded for aliquots of 2 microliter injections (=8 milligrams of plant material).

NOR-AM Agricultural Products, Inc.
Woodstock, Illinois

May 17, 1976
Dr. NAJ/jcf

Blanks and Recoveries Using SN 49537 Residue Method 49537/1

B. Cottonseed Recoveries

NOR-AM No.	Origin	SN 49537 Added ppm	Measured Values[1] Peak Area[2] Integrator Counts	Equivalent ppm SN 49537	Average Blank SN 49537 ppm	SN 49537 Recovery ppm	SN 49537 Recovery %
1098-190	Texas	0.2	643000	0.179	0.015	0.164	82%
1098-190	Texas	0.2	623000	0.172	0.015	0.157	79%
1098-190	Texas	0.5	1851000	0.514	0.015	0.499	100%
1098-190	Texas	0.5	1779000	0.494	0.015	0.479	96%
1098-190	Texas	0.5	1303000	0.398	0.015	0.383	77%
1098-190	Texas	1.0	3754000	1.149	0.015	1.134	113%
1098-190	Texas	1.0	3643000	1.012	0.015	0.997	100%
1098-190	Texas	1.0	3066000	0.851	0.015	0.836	84%
1098-190	Texas	1.0	2962000	0.823	0.015	0.808	81%
	Average	0.2-1.0					90±12%

1) Measured values are results of analysis of individual samples.

2) Peak areas are recorded for aliquots of 2 microliter injections (=8 milligrams of plant material).

[illegible] Products, Inc.
[illegible], Illinois

May 17, 1976
Dr. NAJ/jcf

Blanks and Recoveries Using SN 49537 Residue Method 49537/1

C. Cotton Lint Blanks (Calculated as SN 49537)

(Code No.) or Rpt. No.	Origin	Dates of Analysis	Peak Area[2] Integrator Counts	Number of Tests	Average ppm SN 49537
49537/NA 2	Texas	4/7/76-4/19/76	203000 ± 27000	3	0.212 ± 0.023
49537/NA 12	California	4/15/76-5/3/76	95000 ± 6000	3	0.100 ± 0.007
(1162-26)	California	4/22/76-5/3/76	190000 ± 47000	3	0.200 ± 0.044

D. Cotton Lint Recoveries

NOR-AM No.	Origin	SN 49537 Added ppm	Measured Values[1] Peak Area[2] Integrator Counts	Equivalent ppm SN 49537	Average Blank SN 49537 ppm	SN 49537 Recovery ppm	SN 49537 Recovery %
1162-26	California	1.0	1451000	1.46	0.20	1.26	126%
1162-26	California	1.0	1330000	1.35	0.20	1.15	115%
1162-26	California	1.0	1319000	1.34	0.20	1.14	114%
1162-26	California	1.0	1128000	1.17	0.20	0.97	97%
1162-26	California	5.0	5082000	5.34	0.20	5.14	103%
	Average	1.0 - 5.0					111 ± 11%

1) Measured values are results of analysis of individual samples.

2) Peak areas are recorded for aliquots of 2 microliter injections (=2 milligrams of plant material).

R-AM AGRICULTURAL PRODUCTS, INC.
)DSTOCK, ILLINOIS, USA

May 25, 1978
Dr. NAJ/mw

49537/NA 47

CONTROL VALUES AND RECOVERY OF THIDIAZURON RESIDUES FROM COW'S MILK AND TISSUES

ıtroduction

Thidiazuron (SN 49537) is a new non-phosphate cotton defoliant. The purpose of this study is to develop a suitable analytical method for monitoring thidiazuron residues in cow's milk and tissues.

aterials and Method

Cow's milk and tissues were obtained from various food outlets. The analytical method proposed for monitoring residues of N-phenyl-N'-1,2,3-thiadiazol-5-ylurea (thidiazuron) in animal materials is Schering AG residue method 49537/1, dated 11/20/75. Control values and recovery data obtained using this gas chromatographic method of analysis are presented in the SUMMARY TABLE and the attached residue report sheets. Recovery data was obtained by adding known amounts of thidiazuron to the animal product before hydrolysis and extraction.

Results

The data found in the SUMMARY TABLE demonstrates that the recoveries determined by this gas chromatographic procedure are satisfactory. Thus, Schering AG residue method 49537/1, dated 11/20/75, is an acceptable analytical method for monitoring residues of thidiazuron in cow's milk and tissues.

N. Jenny

SUMMARY TABLE

CONTROL VALUES AND RECOVERIES OF THIDIAZURON (GLC METHOD)

Commodity	No. of Analyses	Controls Calculated ppm Range	Mean + std.dev.	Amt. of Thidiazuron added (ppm)	Recoveries No. of Analyses	% Thidiazuron recovered Range	Mean + std. dev.
Cow's milk	12	0.004-0.018	0.010 ± 0.004	0.05	3	98-104	102 ± 3
				0.10	3	94-103	98 ± 5
Cow's liver	18	0.062-0.184	0.108 ± 0.048	0.05	3	78-120	93 ± 23
				0.10	4	96-124	108 ± 11
				0.20	5	96-115	105 ± 7
Cow's kidney	12	0.063-0.102	0.076 ± 0.011	0.10	7	72-102	81 ± 12
				0.20	6	97-119	111 ± 9
Cow's meat	3	0.094-0.104	0.097 ± 0.006	0.05	3	78-116	103 ± 21
				0.10	3	86-101	93 ± 8
Cow's fat	3	0.073-0.085	0.079 ± 0.006	0.10	2	72-78	75 ± 4
				0.20	3	67-77	71 ± 5

-AM AGRICULTURAL PRODUCTS, INC.
DSTOCK, ILLINOIS, USA

RESIDUE REPORT 49537/NA 47
DATE: May 25, 1978

MPOUND	COMMODITY	STORAGE	ANALYSIS SAMPLE WEIGHT
Thidiazuron	Cow's Milk	--	100 g
THOD 49537/1: Residue Determination of SN 49537 in Cottonseed and Cotton Fiber, November 20, 1975			DATES OF ANALYSIS 3/9/78 - 5/4/78
DIFICATIONS Hydrolysis with 300 ml 2.5N sodium hydroxide. Distillation-extraction time: 4 hours			

MMENTS
ntrols (5-122.5-.10)were fresh milk from Craig Rudi's farm,Woodstock,IL (3/8/78)
ntrols (5-128.10-.12)were pasteurized milk from Eagle Food Store,Woodstock,IL(3/28/78)
ntrols (5-129.10-.12)were pasteurized milk from Morton Chemical Co. Cafeteria, Wood-
stock, IL (3/30/78)

AMPLE MBER	TREATMENT	RESIDUES Thidiazuron PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
-122.5	Control	72200	0.009	--	--
-122.6	Control	48400	0.006	--	--
-122.7	Control	48000	0.006	--	--
-122.8	Control	59100	0.007	--	--
-122.9	Control	46700	0.006	--	--
-122.10	Control	37300	0.004	--	--
-128.10	Control	142100	0.018	--	--
-128.11	Control	106000	0.013	--	--
-128.12	Control	122700	0.015	--	--
-129.10	Control	78100	0.010	--	--
5-129.11	Control	82500	0.012	--	--
5-129.12	Control	75600	0.009	--	--

LABORATORY NAME	DATE	ANALYST	CHECK
Kappie 5	5/4/78	A.Kappie P.Niketas	N. Jenny
NOTEBOOK PAGES 122, 128-129			

-AM AGRICULTURAL PRODUCTS, INC.
DSTOCK, ILLINOIS, USA

RESIDUE REPORT 49537/NA 47
DATE: May 25, 1978

MPOUND idiazuron	COMMODITY Cow's Milk	STORAGE --	ANALYSIS SAMPLE WEIGHT 100 g
THOD 49537/1: Residue Determination of SN 49537 in Cottonseed and Cotton Fiber, November 20, 1975			DATES OF ANALYSIS 3/10/78-3/20/78
DDIFICATIONS Hydrolysis with 300 ml 2.5N sodium hydroxide. Distillation-extraction time: 4 hours			
OMMENTS Values of ppm NET corrected for corresponding check (0.006 ppm)			

RESIDUES Thidiazuron

AMPLE UMBER	TREATMENT	PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
-123.7	0.05 ppm	534900	0.055	0.049	98%
-123.8	0.05 ppm	561000	0.058	0.052	104%
-123.9	0.05 ppm	562700	0.058	0.052	104%
5-123.10	0.10 ppm	1092900	0.104	0.098	98%
5-123.11	0.10 ppm	1054600	0.100	0.094	94%
5-123.12	0.10 ppm	1145700	0.109	0.103	103%

LABORATORY NAME Kappie 5 NOTEBOOK PAGES 123	DATE 3/20/78	ANALYST A.Kappie P.Niketas	CHECK N.Jenny

NOR-AM AGRICULTURAL PRODUCTS, INC.
WOODSTOCK, ILLINOIS, USA

DATE: May 25, 1978

COMPOUND	COMMODITY	STORAGE	ANALYSIS SAMPLE WEIGHT
Thidiazuron	Cow's liver	0° F	10 g
METHOD 49537/1: Residue Determination of SN 49537 in Cottonseed and Cotton Fiber, November 20, 1975			DATES OF ANALYSIS 3/7/78 - 5/17/78
MODIFICATIONS Hydrolysis with 300 ml 2.5N sodium hydroxide. Distillation-extraction time: 4 hours			
COMMENTS Controls(5-120.1-.6) were purchased from Marengo Packing Plant (3/3/78) Controls (5-143.7-.9)were purchased from A & P Food Store,Woodstock, IL (3/28/78) Controls (5-148.1-.3)were purchased from IGA Food Store, Dundee, IL (5/13/78) Controls (5-148.4-.6)were purchased from Angels Food Store, Elgin, IL (5/13/78)			

		RESIDUES Thidiazuron			
SAMPLE NUMBER	TREATMENT	PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
5-120.1	Control	155600	0.184	--	--
5-120.2	Control	140000	0.166	--	--
5-120.3	Control	140000	0.166	--	--
5-120.4	Control	153000	0.181	--	--
5-120.5	Control	140600	0.166	--	--
5-120.6	Control	153800	0.182	--	--
5-143.7	Control	95800	0.084	--	--
5-143.8	Control	111600	0.097	--	--
5-143.9	Control	100400	0.088	--	--
5-148.1	Control	11300*	0.068	--	--
5-148.2	Control	11300*	0.068	--	--
5-148.3	Control	10300*	0.062	--	--
5-148.4	Control	12300*	0.074	--	--
5-148.5	Control	10200*	0.062	--	--
5-148.6	Control	12500*	0.075	--	--

LABORATORY NAME	DATE	ANALYST	CHECK
Kappie 5	5/17/78	A.Kappie/P.Niketas	N. Jenny
NOTEBOOK PAGES 120,143,148			

* Additional Modifications for analysis of samples 5-148.1-.6:
Used a 20m x 0.25mm i.d. glass capillary column coated with SE-30; oven temperature: 180°C; detector and injection port temperature: 250°C; split ratio: 1:10; carrier gas: 1.3 ml/min argon/methane 95/5.

COMPOUND	COMMODITY	STORAGE	ANALYSIS SAMPLE WEIGHT
Thidiazuron	Cow's liver	0° F	10 g
METHOD 49537/1: Residue Determination of SN 49537 in Cottonseed and Cotton Fiber, November 20, 1975			DATES OF ANALYSIS 3/7/78 - 5/17/78
MODIFICATIONS Hydrolysis with 300 ml 2.5N sodium hydroxide. Distillation-extraction time: 4 hours			
COMMENTS Controls (5-148.7-.9) purchased from Jewel Foods, Carpentersville, IL (5/13/78)			

Values of ppm NET corrected for corresponding check (0.183 or 0.090 ppm)
** Values of ppm a.i. calculated from new standard curve (5-145).

		RESIDUES Thidiazuron			
SAMPLE NUMBER	TREATMENT	PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
5-148.7	Control	11900*	0.072	--	--
5-148.8	Control	13800*	0.083	--	--
5-148.9	Control	13500*	0.084	--	--
5-120.9	0.05 ppm	188000	0.222	0.039	78%
5-120.10	0.05 ppm	205400	0.243	0.060	120%
5-129.2	0.05 ppm	189100	0.224	0.041	82%
5-129.3	0.10 ppm	251000	0.288	0.105	105%
5-144.4	0.10 ppm	240400**	0.189	0.096	96%
5-144.5	0.10 ppm	251200**	0.198	0.108	108%
5-144.6	0.10 ppm	271700**	0.214	0.124	124%
5-129.7	0.20 ppm	351100	0.384	0.201	100%
5-130.1	0.20 ppm	345200	0.376	0.193	96%
5-144.7	0.20 ppm	406600**	0.320	0.230	115%
5-144.8	0.20 ppm	388300**	0.305	0.215	107%
5-144.9	0.20 ppm	388500**	0.306	0.216	108%

LABORATORY NAME Kappie 5	DATE	ANALYST	CHECK
NOTEBOOK PAGES 120,129,130,144, 148	5/17/78	A.Kappie/P.Niketas	N.Jenny

* Additional Modifications for analysis of samples 5-148.7-.9:
Used a 20m x 0.25mm i.d. glass capillary column coated with SE 30; oven temperature: 180°C; detector and injection port temperature: 250° C; split ratio: 1:10; carrier gas: 1.3 ml/min. argon/methane 95/5.

NOR-AM AGRICULTURAL PRODUCTS, INC.
WOODSTOCK, ILLINOIS, USA

SEPTEMBER 12, 1978
Dr. NAJ/cal

49537/NA 48

CONTROL VALUES AND RECOVERY OF THIDIAZURON RESIDUES FROM CHICKEN EGGS

Introduction

Thidiazuron (SN 49537) is a new non-phosphate cotton defoliant. The purpose of this study is to develop a suitable analytical method for monitoring thidiazuron residues in chicken eggs.

Materials and Method

Chicken eggs were obtained from a food outlet. The analytical method proposed for monitoring residues of N-phenyl-N'-1,2,3-thiadiazol-5-ylurea (thidiazuron) in animal materials is Schering AG residue method 49537/1, dated 11/20/75. Control values and recovery data obtained using this gas chromatographic method of analysis are presented in the attached residue report sheets. Recovery data was obtained by adding known amounts of thidiazuron to the animal product before hydrolysis and extraction.

Results

The data found in the residue report sheets demonstrates that the recoveries determined by this gas chromatographic procedure are satisfactory. Thus, Schering AG residue method 49537/1, dated 11/20/75, is an acceptable analytical method for monitoring residues of thidiazuron in chicken eggs.

N. Jenny

-AM AGRICULTURAL PRODUCTS, INC.
)DSTOCK, ILLINOIS, USA

RESIDUE REPORT 49537/NA 48
DATE: September 12, 1978

)MPOUND	COMMODITY	STORAGE	ANALYSIS SAMPLE WEIGHT
Thidiazuron	Chicken Eggs	-	20 g

:THOD 49537/1: Residue Determination of SN49537 in Cottonseed and Cotton Fiber, November 20, 1975

DATES OF ANALYSIS 9/8/78 - 9/11/78

)DIFICATIONS Hydrolysis with 300 ml 2.5N sodium hydroxide. Distillation-extraction time: 4 hours. Add 4 ml Anti-Foam A, start with low heat and watch for foaming.

)MMENTS Controls (5-152.1-.6) were eggs from A&P Food Store, Fox Lake, IL. Six whole eggs were placed in a Waring Blender and blended for 1 minute.

AMPLE MBER	TREATMENT	RESIDUES Thidiazuron: PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
5-152.1	Control	85300	0.053	--	--
5-152.2	Control	77500	0.048	--	--
5-152.3	Control	77300	0.048	--	--
5-152.4	Control	65600	0.040	--	--
5-152.5	Control	66100	0.041	--	--
5-152.6	Control	69600	0.043	--	--
		Average Control:	0.045 ±	0.005	

BORATORY NAME	Kappie 5	DATE	ANALYST	CHECK
TEBOOK PAGES	152	9/11/78	A. Kappie P. Niketas	N. Jenny

R-AM AGRICULTURAL PRODUCTS, INC.
)ODSTOCK, ILLINOIS, USA

RESIDUE REPORT 49537/NA48
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METHOD 49537/1: Residue Determination of SN49537 in Cottonseed and Cotton Fiber, November 20, 1975

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COMMENTS
Values of ppm NET corrected for corresponding check (0.045 ppm).

		RESIDUES Thidiazuron			
SAMPLE NUMBER	TREATMENT	PEAK AREA INTEGRATOR COUNTS (HP 3380A INTEGRATOR)	ppm a.i.	ppm NET	% RECOVERY
-152.7	0.05 ppm	129300	0.080	0.035	70%
-152.8	0.05 ppm	143100	0.088	0.043	86%
-152.9	0.05 ppm	148900	0.092	0.047	94%
		Average recovery:	83 ± 12%		
-152.10	0.10 ppm	216400	0.134	0.089	89%
-152.11	0.10 ppm	227400	0.140	0.095	95%
		Average recovery:	92 ± 4%		

LABORATORY NAME Kappie 5	DATE	ANALYST	CHECK
NOTEBOOK PAGES 152	9/11/78	A. Kappie P. Niketas	N. Jenny