

US EPA ARCHIVE DOCUMENT

GREENSBORO, N.C.	PAGE 1 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
	EDITION 12/7/82		
	SUBMITTED BY: K. Balasubramanian, R. Perez		

*K. Balasubramanian**R. Perez*

APPROVED BY:

W. B. Taylor

1.0 SCOPE

This total residue method is used for the determination of the combined residues of metalaxyl [N-(2,6-dimethylphenyl)-N-(methoxyacetyl)alanine methyl ester] and its metabolites which contain the 2,6-dimethylaniline moiety in crop samples. (See Figure 1 for structures). This method is a modification of the Analytical Method AG-348 (1) developed earlier for the analysis of crop substrates. The limit of detection for the method is 0.05 ppm expressed in metalaxyl equivalents.

2.0 PRINCIPLE

Wet crop samples are extracted by blending with a Polytron Homogenizer for one minute using 80% (V/V) methanol/water. Dry crops are extracted by refluxing with 80% (V/V) methanol/H₂O for two hours. A 2-g aliquot of the sample is evaporated to dryness. One or one and a half ml of water is added to dissolve the residue, depending on the substrate, and the sample is refluxed for 15 minutes after addition of 10 ml of methanesulfonic acid. The extract is basified after addition of water. The 2,6-dimethylaniline formed in the reaction is steam distilled using a modification of the steam distillation apparatus of Veith and Kiwus (2).

The steam distilled product is cleaned up with a silica Sep-Pak® cartridge prior to analysis by capillary gas chromatography using a nitrogen/phosphorus detector (NPD) operating in the nitrogen-specific mode.

3.0 APPARATUS

- HP-5880 capillary gas chromatograph, with nitrogen/phosphorus detector and autosampler.
- Polytron (Brinkman Instruments PCU-1) or equivalent.
- Rotary evaporator, Buchi or equivalent.
- Bottle, Boston round, narrow mouth, 16-oz, with Polyseal cap.
- Bottle, Boston round, wide mouth, 16-oz, with Polyseal cap.

GREENSBORO, N.C.

PAGE 2 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

- Syringe, Luer Lok.
- Filter paper, Whatman 2V, 32-cm.
- Funnel, 12.5-cm size; 6.5-cm size.
- Variable transformer, Powerstat.
- Round bottom flask, 250 ml, 100 ml.
- Condenser, Allihn bulb-type, 500-mm, water cooled.
- Glascol heating mantle, 250 ml.
- Hobart food cutter.
- Scintillation vial.
- Test tube with polyseal cap.
- Wiley Mill.
- Modified steam distillation apparatus (See Figure 3 for details).
- Separatory funnel, 125-ml, with Teflon stopcock.
- Automatic pipette, 100- μ l, Fisher or equivalent.

4.0 REAGENTS

- Methanol, HPLC grade.
- Dichloromethane, HPLC grade.
- Hexane, HPLC grade.
- Methanesulfonic acid, Practical, Eastman Kodak Chemicals, #P-6320.
- 25% sodium hydroxide solution [mixture of equal amounts of distilled water and 50% sodium hydroxide solution (Fisher)].

CIBA-GEIGY Corporation

BIOCHEMISTRY DEPARTMENT

66

GREENSBORO, N.C.

PAGE 3 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		

APPROVED BY:

- 0.05% aqueous sodium hydroxide solution.
- Analytical standard metalaxyl.
- Absorbent cotton.
- Glass wool.
- Standard, 2,6-dimethylaniline trifluoroacetic acid salt (DMA-TFA).
- Sep-Pak silica cartridge, Waters Associates.
- Trifluoroacetic acid, Eastman Kodak Chemicals.
- Deionized water.

5.0 ANALYTICAL PROCEDURE

5.1 Sample Preparation

A representative sample of 300-400 g of crop is chopped in a Hobart Food Cutter. Green crops are mixed with dry ice before chopping. Grain samples are ground using a Wiley Mill.

5.2 Sample Preparation for Determining Recovery Values

Recovery values are determined for each set of samples analyzed by including an untreated control sample and one or more control samples fortified with 0.05 ppm or more of metalaxyl. Samples are fortified prior to extraction. A 0.5 ml aliquot of a 1- μ g/ml metalaxyl standard in methanol is added to 10 g of control sample in step 5.3 or 5.4 for a 0.05-ppm fortification. Allow the sample to absorb the standard. Use appropriate standard concentrations for higher fortifications. The fortification volume should not exceed 2 ml.

5.3 Extraction - Wet Crop

5.3.1 Weigh a 10-g representative sample of a wet crop (e.g., lettuce, cabbage, fruit crop, broccoli, cucumbers, onions, etc.) into an 8-oz. amber, square, wide-mouth bottle. Nut-meats and grains may also be extracted by this procedure. Add 100 ml of 80% (V/V) methanol/water.

PAGE 4 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

5.3.2 Extract the sample by homogenizing with a Polytron® homogenizer for approximately one minute.

5.3.3 Filter the sample through a Whatman 2V filter paper into an 8-oz. bottle (Boston round, narrow mouth).

5.4 Extraction - Dry Crop

5.4.1 Weigh 10-g of the finely chopped sample of dry crop (e.g., cottonseed, grains, dry straw, etc.) into a 250-ml round bottom flask. Add 100 ml of 80% (V/V) methanol/water (use 200 ml for dry straw) and add a few boiling chips.

5.4.2 Place the flask in a 250-ml heating mantle, attach a reflux condenser to the flask and reflux for two hours.

5.4.3 Allow the solution to cool and filter through a Whatman 2V filter paper.

5.5 Methanesulfonic Acid Reflux

5.5.1 Transfer a 2-g aliquot (20 ml) of the extract (correct for moisture) in step 5.3.3 or 5.4.3 (40 ml for dry straw) to a 250 ml round bottom flask.

5.5.2 Evaporate the sample to dryness using a rotary evaporator (bath temperature 30-40°C). Add 1 ml (1.5 ml to dry substrates) of distilled water to the residue and swirl to dissolve.

5.5.3 Add carborundum chips and then add 10 ml of methanesulfonic acid reagent to the flask, swirl to completely dissolve residue, and place in a 250-ml heating mantle. Attach a reflux condenser. Heat the flask to reflux and continue refluxing for 15 minutes. Remove the heating mantle promptly after refluxing.

PAGE 5 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

Note: Refluxing the sample for more than 20 minutes may cause losses through degradation of 2,6-dimethylaniline.

5.5.4 Allow the solution to cool for about 15 minutes. Add 100 ml of distilled water. For nut meats go to step 5.5.5. Add 15 ml of hexane and 25 ml of 25% sodium hydroxide solution through the top of the condenser. Swirl the flask to facilitate mixing. Go to Section 5.6.

5.5.5 For nut meats, wait until the solution is completely cool, and then transfer to a 250 ml separatory funnel. Partition once with 50 ml of hexane and place the aqueous phase in a 250-ml round bottom flask. Add 15 ml of hexane, and 25 ml of 25% sodium hydroxide solution through the top of the condenser. Swirl the flask to facilitate mixing.

Note: The pH of the solution may be checked with pH paper to ensure that it is basic (>8.0).

5.6 Steam Distillation

5.6.1 Place the flask containing the basified extract in a 250-ml heating mantle and attach the steam distillation apparatus shown in Figure 3. Make sure that cold water is circulating through the cooling jacket and a glass wool plug is placed at the top of the condenser to prevent losses.

5.6.2 Heat the flask to reflux. When a bubbling effect is seen in the distilled hexane/H₂O reservoir, continue refluxing for one hour. The total heating time is about 1 1/4 hours.

5.6.3 Allow to cool for 15 minutes and then withdraw the hexane phase from the steam distillation apparatus through the solvent withdrawal tube into a scintillation vial. The hexane solution containing some water may be capped and stored in a freezer overnight, or

GREENSBORO, N.C.

PAGE 6 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		

APPROVED BY:

for about two hours, until the aqueous phase is completely frozen for easy separation of the phases.

The hexane phase containing 2,6-dimethylaniline is next cleaned-up by a Silica Sep-Pak cartridge.

5.7 Silica Sep-Pak Cartridge Cleanup

- 5.7.1 Fit a Silica Sep-Pak cartridge to the Luer Lok end of a 20-ml syringe with the plunger removed.
- 5.7.2 Rinse the Sep-Pak with about 5 ml of hexane, forcing the solvent through at a flow rate of less than 5 ml/min with air pressure. Remove the air pressure just before the top of the solvent penetrates the Sep-Pak cartridge.
- 5.7.3 If the sample was stored in the freezer, the aqueous phase will be frozen and the hexane layer can be easily poured into the syringe. Rinse the vial with 2 to 5 ml of hexane, and pour into the syringe barrel. Alternatively, pour the hexane solution into a 60-ml separatory funnel, separate the phases and discard the aqueous phase. Transfer the hexane phase to the syringe. Rinse separatory funnel with 5 ml of hexane and pour into syringe in the same way.
- 5.7.4 Force the sample through the Sep-Pak as in 5.7.2. Do not allow the packing to go dry. Discard eluate.
- 5.7.5 Add 18 ml of dichloromethane to the syringe and force through as in 5.7.2, but remove all the dichloromethane. Collect the sample in a 100-ml round bottom flask.
- 5.7.6 Add 200 μ l of trifluoroacetic acid to the sample and evaporate to dryness using a rotary evaporator (use ice to get bath temperature to 15°C).

CIBA-GEIGY Corporation

GREENSBORO, N.C.

PAGE 7 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

Note: Traces of the trifluoroacetic acid will cause losses. Sample should be brought to complete dryness. Bath temperatures higher than 18°C will cause losses.

5.7.7 For gas chromatographic detection, transfer the sample with 2.0 ml of toluene to a test tube. Wash the sample by shaking (or vortexing) for a half minute with 5 ml of 0.05% NaOH. Pipette out the toluene phase and transfer to an autosampler vial.

6.0 GAS CHROMATOGRAPHIC ANALYSIS OF 2,6-DIMETHYLANILINE (DMA)

Analyze samples by capillary gas chromatography using an NPD, operated in the nitrogen specific mode. The gas chromatographic conditions are given in Table I.

6.1 Standardization

6.1.1 Prepare a stock solution containing 100 mg of 2,6-dimethylaniline-trifluoroacetic acid salt (DMA-TFA) analytical standard in 100 ml of methanol. Serial dilutions should be made with toluene until a working range, 0.02 to 1.0 ng/μl, is achieved.

6.1.2 Standardize the analytical instrument, operating under the conditions specified in Table I for capillary GC analysis, or Table II for GC/MS analysis, by injecting 2-μl aliquots of the working standards during the analysis, covering a concentration range of 0.04 ng to 2.0 ng. Typical standard chromatograms are shown in Figure 4 for capillary GC and in Figure 5 for GC/MS.

6.1.3 Determine the peak heights for the injected standards and prepare a standard curve by plotting peak height versus nanograms of standard injected. Alternatively, enter the standardization data into an appropriate electronic calculator (e.g., Texas Instrument Model TI55) if detector response is linear,

CIBA-GEIGY Corporation

GRENSBORO, N.C.

PAGE 8 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

or a computer (e.g., HP1000 Lab Automation System) to calculate a least square standard curve. Typical standard curves for GC-NPD and GC-MS are presented in Figure 6.

6.2 Detection of Sample Residues

6.2.1 Inject a 2- μ l aliquot of the sample in Step 5.7.7 into the capillary gas chromatograph. Make appropriate dilutions of the sample to have the sample peak height within the range of the standard curve. Compare peak heights of unknown samples with the standard curve to obtain the amounts of DMA-TFA in the injected aliquot. If using a calculator or Lab Automation System, follow calculator or automation system instructions. Typical chromatograms of selected samples using GC-NPD detection are shown in Figures 7 through 8.

6.2.2 Calculate residue results as ppm equivalents of metalaxyl using the following equations:

$$\text{PPM Found} = \frac{(\text{Ng DMA-TFA Found})}{(\text{Mg Crop Injected})}$$

$$\text{Actual Metalaxyl PPM Found} = \text{PPM Found} \times 1.188$$

Correct the measured metalaxyl ppm found in recoveries by subtracting the measured metalaxyl ppm found, real or apparent, in the controls. Calculate the recovery factor by the following equation:

$$R = \frac{\text{Corrected PPM Found in Fortified Sample}}{\text{PPM Added}}$$

$$\text{Corrected PPM} = \frac{\text{Actual Metalaxyl PPM Found}}{R}$$

where R is the recovery factor based on fortified controls carried through the procedure and is expressed as a decimal (100% = 1.0, 80% = 0.80, etc.). If R > 1.0, use 1.0.

CIBA-GEIGY Corporation

PAGE 9 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

The conversion factor 1.188 is used to convert residues of DMA-TFA found into metalaxyl equivalents (molecular weight (MW) of metalaxyl/MW of DMA-TFA = $279.3/235.2 = 1.188$).

7.0 DISCUSSION

- 7.1 Loss of DMA has been experienced when samples are taken to dryness. Addition of TFA to DMA forms a salt complex or ion pair which can then be taken to dryness without appreciable losses. TFA is used in the method, therefore, solely to act as a keeper. Excess TFA is removed by partitioning with dilute aqueous base prior to gas chromatographic determination to avoid adsorption effects in the injection port. DMA-TFA chromatographs as DMA and, thus, is detected by GC/MS in the CI mode at m/e 122, the M + 1 ion of DMA.
- 7.2 Samples and standards in this method were analyzed using an HP-1000 computer with a Laboratory Automation System (LAS) software. A customized software called "Barebone System" was used for analyzing the samples automatically and generating a final summary report.
- 7.3 Average recovery of metalaxyl from peanuts, cucumbers, asparagus, avocados, cantaloupe, beans, broccoli, cabbage, cauliflower and squash fortified at 0.05 to 5.0 ppm was 89 ± 17 (N = 89).

GREENSBORO, N.C.

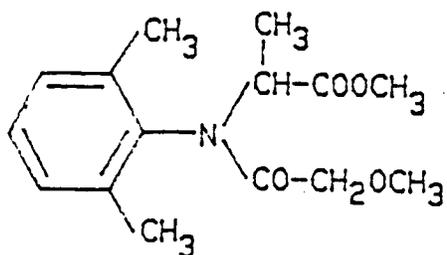
PAGE 10 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramania, R. Perez		

APPROVED BY:

8.0 REFERENCES

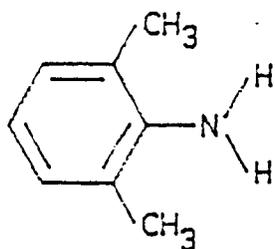
1. K. Balasubramanian, AG-348, "Analytical Method for the Determination of Total Residues of Metalaxyl, in Crops as 2,6-Dimethylaniline."
2. G. D. Veith and L. M. Kiwus, "An Exhaustive Steam Distillation and Solvent-Extraction Unit for Pesticides and Industrial Chemicals," Bulletin of Environmental Contamination and Toxicology, Vol. 17, 1977, p. 631-636.

PAGE 11 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 1 CHEMICAL NAMES AND STRUCTURES

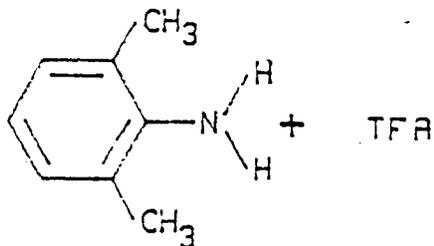
Metalaxyl, CGA-48988
N-(2,6-Dimethylphenyl)-N-
(methoxyacetyl)-alanine
 methyl ester
 $C_{15}H_{21}O_4N$

MW: 279.3



CGA-72649
2,6-dimethylaniline
 $C_8H_{11}N$

MW: 121.0



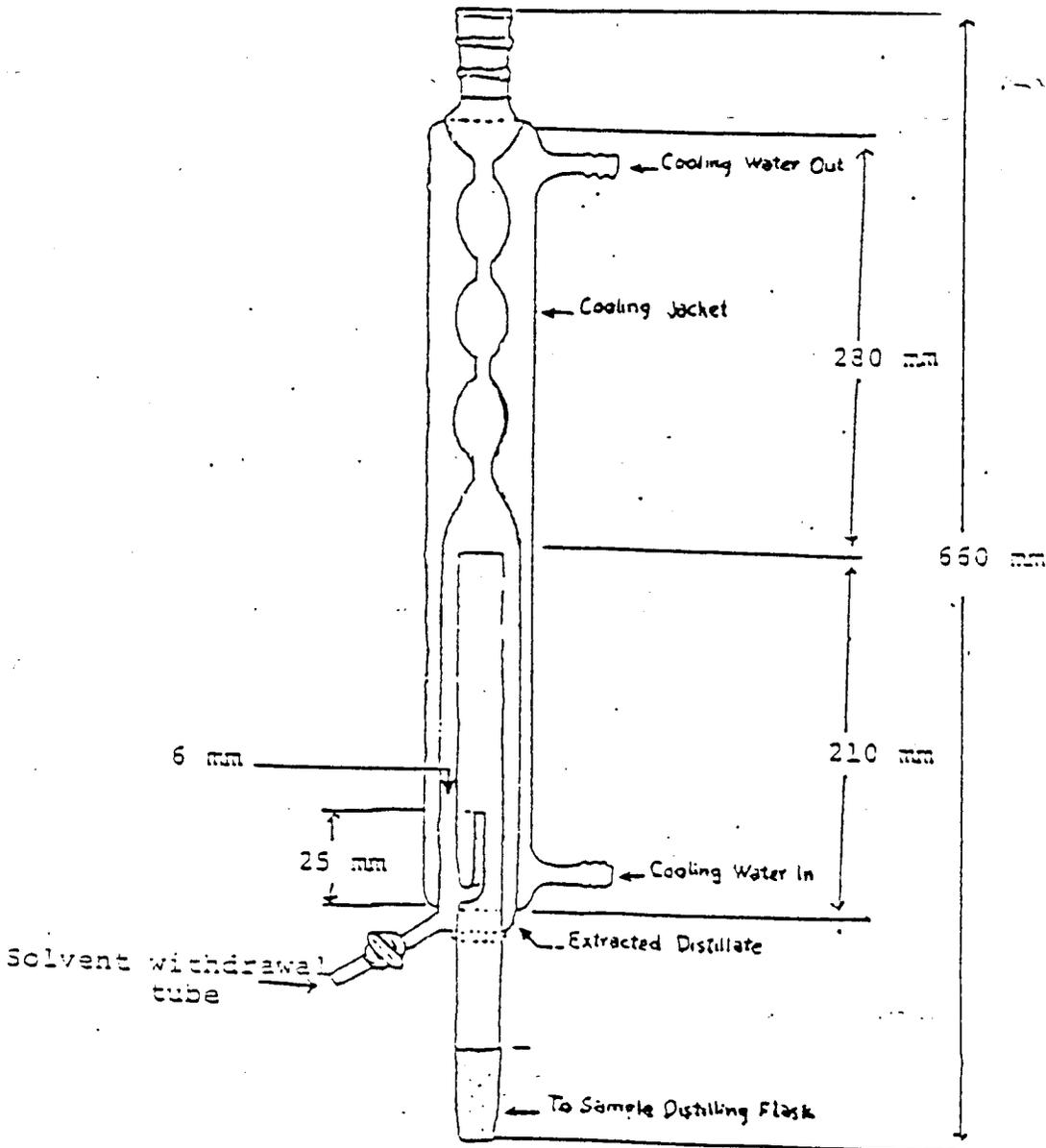
DMA-TFA
2,6-dimethylaniline-
trifluoroacetic acid salt

MW: 235.2

GREENSBORO, N.C.

PAGE 13 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 3: EXHAUSTIVE STEAM-DISTILLATION AND SOLVENT-EXTRACTION APPARATUS



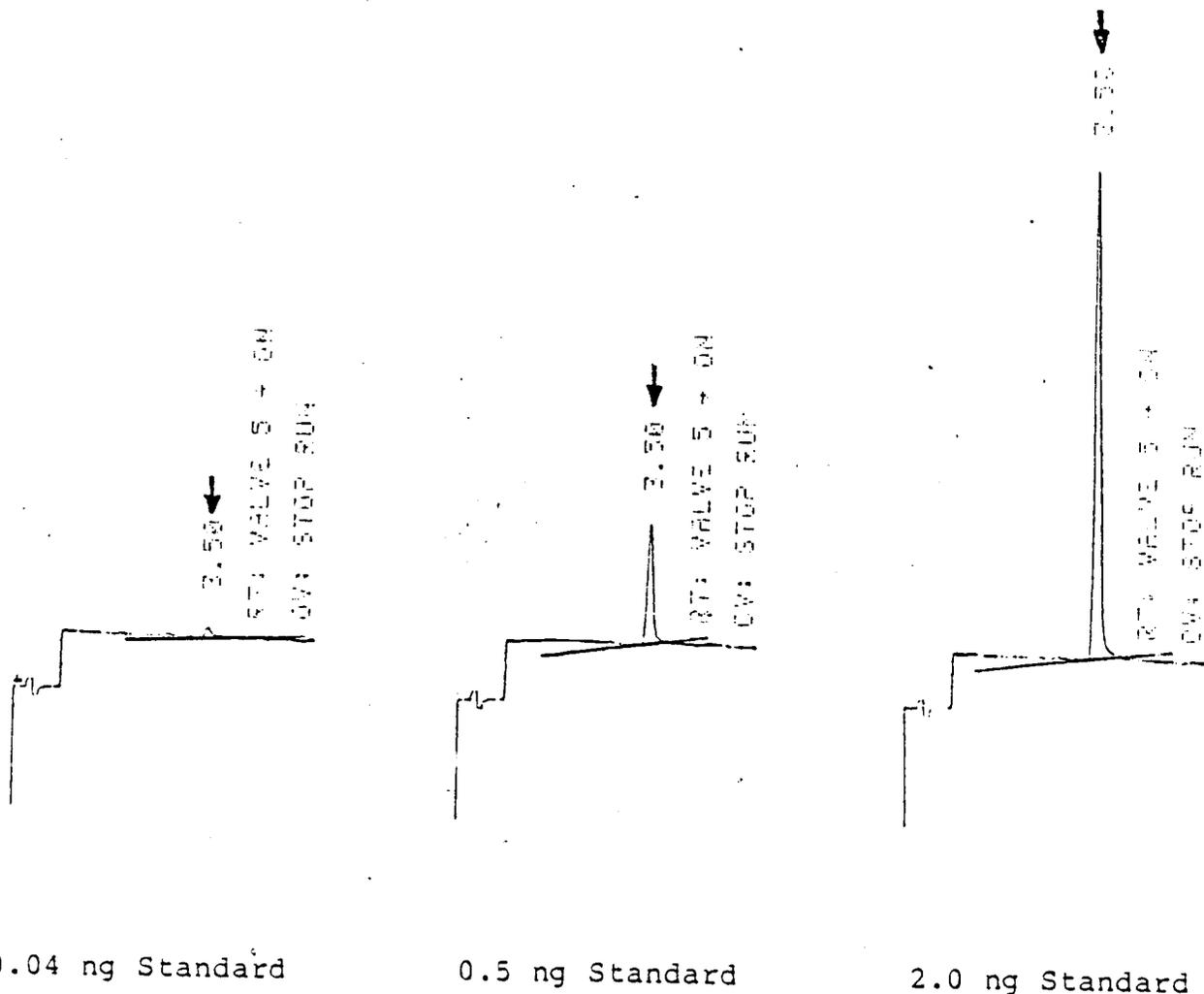
Note: The length of the tube (25 mm) and the space between the jackets (6 mm) are important. These dimensions assure that approximately 15 ml of organic solvent is contained in the well between the jackets.

CIBA-GEIGY Corporation

GREENSBORO, N.C.

PAGE 14 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 4: TYPICAL STANDARD CHROMATOGRAMS OF DMA-TFA FOR CAPILLARY GC-NPD

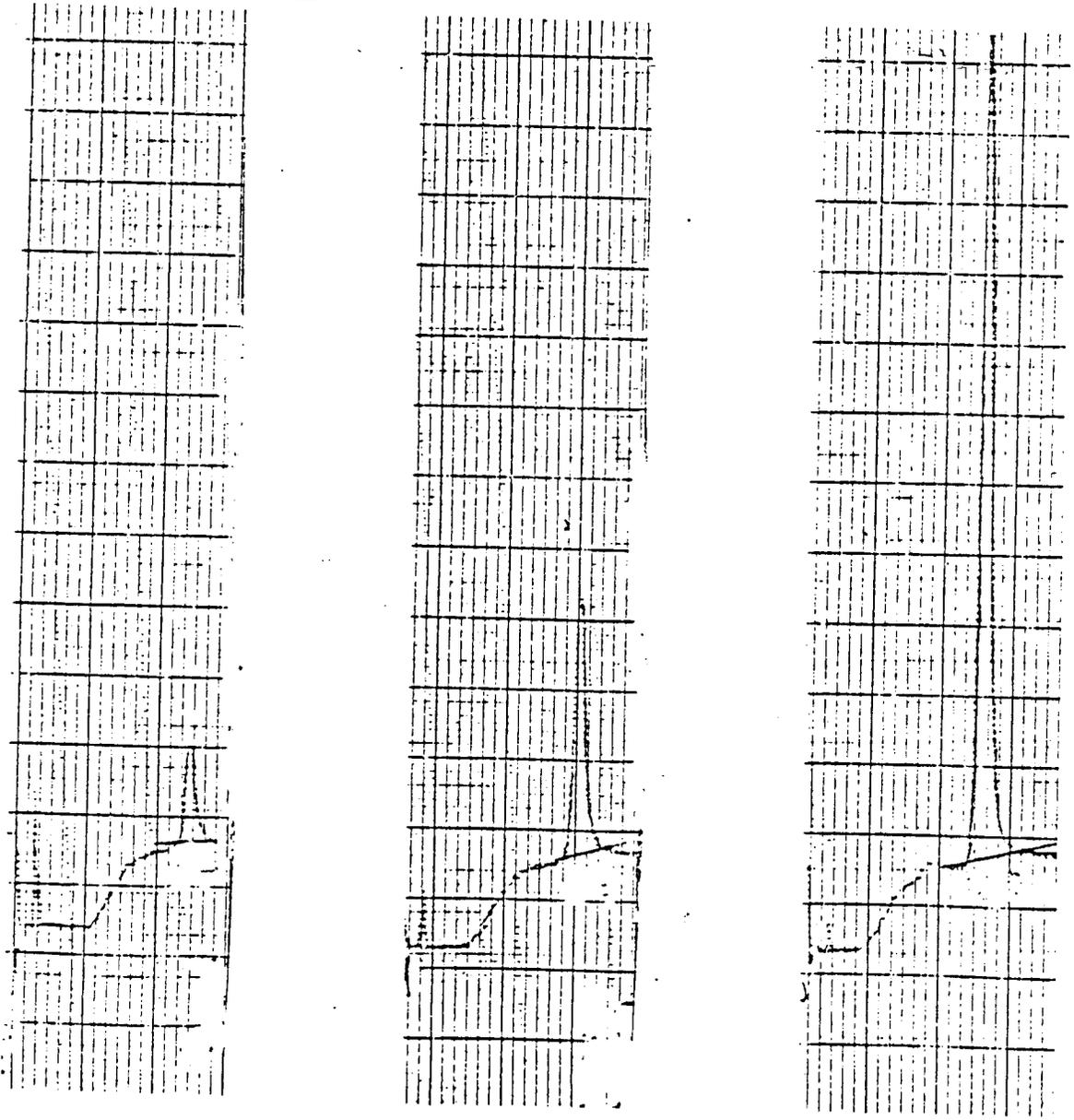


CIBA-GEIGY Corporation

GREENSBORO, N.C.

PAGE 15 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 5: TYPICAL STANDARD CHROMATOGRAMS OF DMA-TFA FOR GC/MS



0.4 ng Standard

1.0 ng Standard

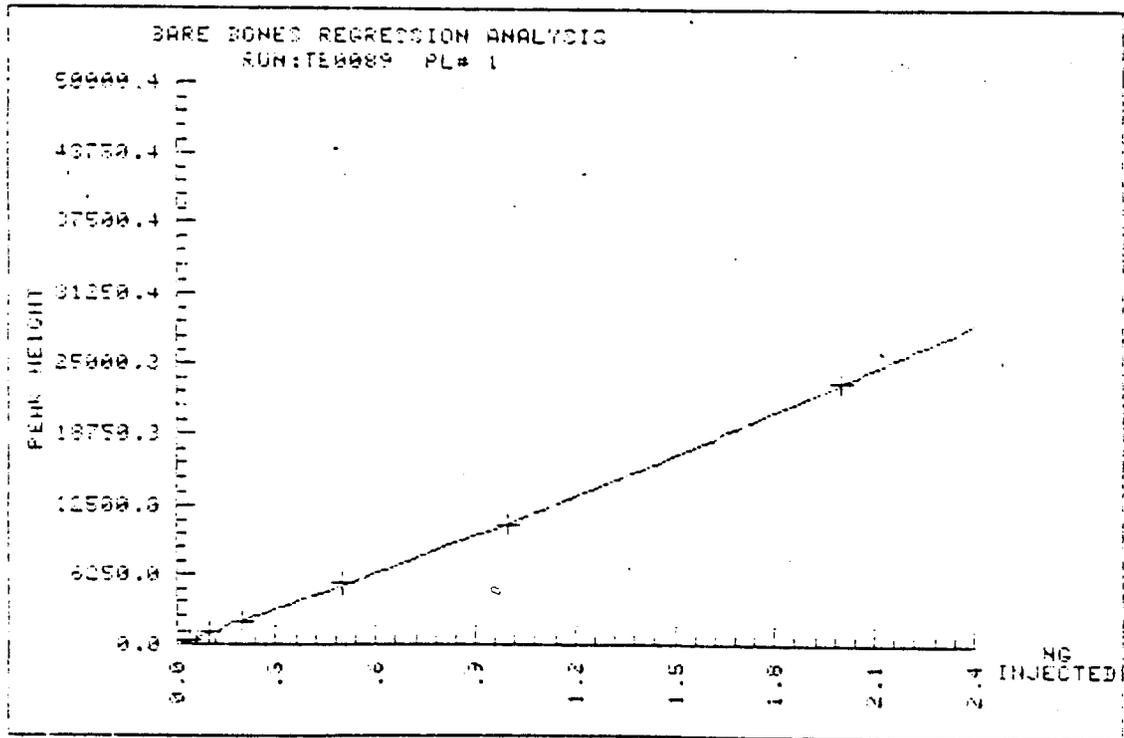
3.0 ng Standard

BIOCHEMISTRY DEPARTMENT

GREENSBORO, N.C.

PAGE 16 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 6: TYPICAL STANDARD CURVE OF DMA-TFA

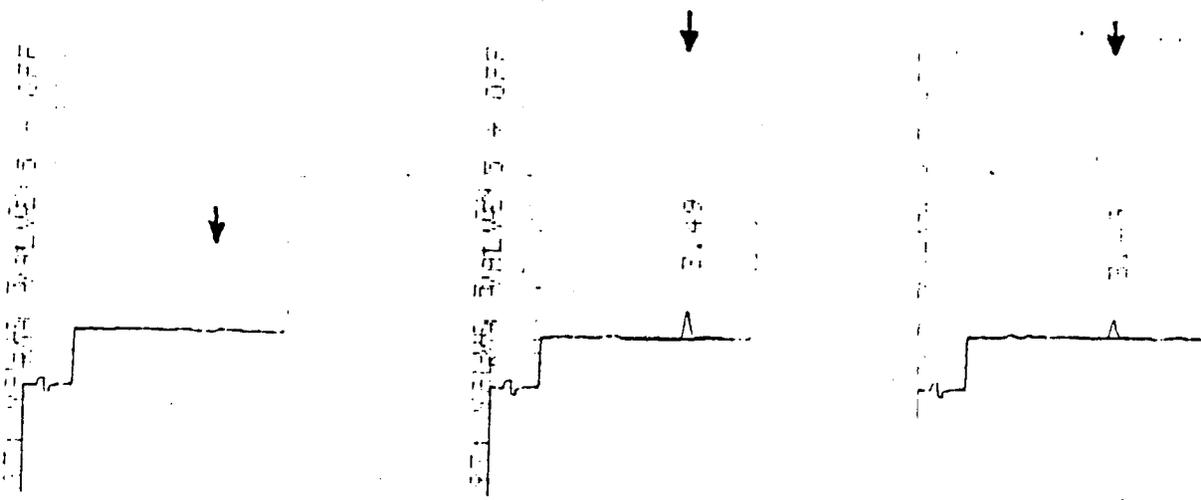


CIBA-GEIGY Corporation

GREENSBORO, N.C.

PAGE 17 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

FIGURE 7: TYPICAL CHROMATOGRAMS FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN LETTUCE



Check Sample
2.0 mg injected
Found: <0.04 ng
DMA-TFA
(<0.05 ppm*)

Check + 0.1 ppm
2.0 mg injected
Found: 0.134 ng
DMA-TFA
(0.08 ppm*)
Recovery: 80%

Treated Sample
2.0 mg injected
Found: 0.08 ng
DMA-TFA
(0.06 ppm*)

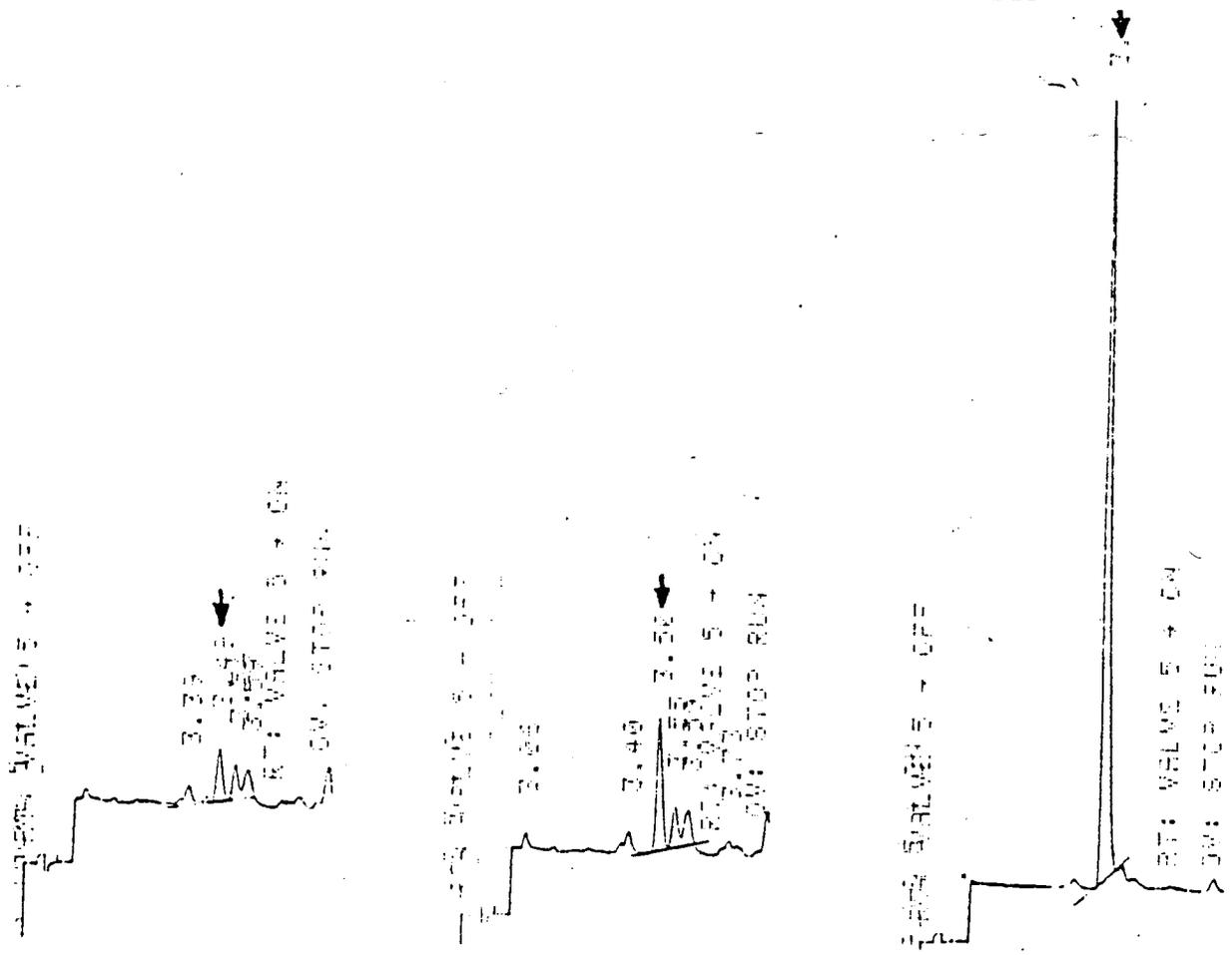
*Metalaxyl Equivalent

CIBA-GEIGY Corporation

GRENSBORO, N.C.

PAGE 18 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
		EDITION 12/7/82
		SUBMITTED BY: K. Balasubramanian, R. Perez
		APPROVED BY:

FIGURE 8: TYPICAL CHROMATOGRAMS FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN SPINACH



Check Sample
2.0 mg injected
Found: 0.2 ng
DMA-TFA
(0.13 ppm*)

Check + 0.2 ppm
2.0 mg injected
Found: 0.52 ng
DMA-TFA
(0.19 ppm*)
Recovery: 95%

Treated Sample
0.4 mg injected
Found: 0.6 ng
DMA-TFA
(1.87 ppm*)

*Metalaxyl Equivalent

CIBA-GEIGY Corporation

GREENSBORO, N.C.

PAGE 19 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

TABLE I: CAPILLARY GAS CHROMATOGRAPHY CONDITIONS

Instrument: Hewlett-Packard 5880 GC equipped with a capillary injector port and a nitrogen/phosphorus detector (NP detector).

Column: Fused silica capillary 0.25 μ m coating.

Oven Temperature:	SE-54 .2 mm X 25 Meter	DX-4 .32 mm X 30 Meter
Initial value -	60°C	60°C
Initial time -	1.0 min.	1.0 min.
Program rate -	30°C/min.	30°C/min.
Final value -	100°C	170°C
Final time -	2.0 min.	1.5 min.
Post value -	250°C	250°C
Post time -	4.0 min.	4.0 min.

Temperatures:
 Detector - 300°C
 Injector - 220°C

Detectors: Nitrogen-Phosphorus Ionization (Element = 140)

Run Table:	SE-54	DX-4
Valve 5 on	0.00	0.00
Valve 5 off	0.50	0.50
Chart Speed 4	3.00	5.00
Attenuation 2 + 2	3.01	4.99
Run time annotation on	3.02	5.01
Valve 5 on	4.00	6.10

Device #2: GC Terminal 1
 Signal - B
 Chart speed - 0.2 cm/min
 Attenuation - 2 + 6
 % Offset - 10

Device #5 HP Loop 1 (for Computer Data Acquisition)
 Signal - B
 Attenuation - 2 + 0
 % Offset - 0
 Signal on Device #5

Minimum Detection Limit: 0.04 ng

Volume Injected: 2 μ l

Retention Time (min.):	SE-54	DX-4
	3.59	5.56

CIBA-GEIGY Corporation

33

BIOCHEMISTRY DEPARTMENT

GREENSBORO, N.C.

PAGE 20 of 20	METHOD No. AG-395	SUBJECT IMPROVED METHOD FOR THE DETERMINATION OF TOTAL RESIDUES OF METALAXYL IN CROP AS 2,6-DIMETHYLANILINE
EDITION 12/7/82		
SUBMITTED BY: K. Balasubramanian, R. Perez		
		APPROVED BY:

TABLE II: GC/MS CONDITIONS

Instrument Finnigan Model 3200 gas chromatograph/mass spectrometer operating in the chemical ionization mode with methane as the reactant and carrier gas. (A "T" between the column and the MS interface is equipped with a solenoid valve or manual toggle valve which is used to vent solvent to a vacuum line)

Carrier Gas Methane flow adjusted to give 1000 microns pressure in the ion source

Column 3% Dexsil 300 on Gas Chrom Q (80/100 mesh), 4' x 2 mm i.d.

Temperatures

Column	100°C
Inlet	230°C
Interface	250°C
Transfer	250°C
MS Manifold	100°C

MS Settings

Electron energy	120 EV
Emission	0.50 ma
Ion energy	15V
Collector	30V
Lens	40V
Extractor	10V
Electron multiplier	2.0 KV
Mass range	122 + 0.2 amu
Sensitivity	10 ⁻⁷
Ion current integrator	30
Recorder	1V FS
Chart speed	1 cm/sec
Vent time	1.0 min
Minimum detection limit	0.2 ng
Volume injected	4-8 µl

CIBA-GEIGY Corporation