



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

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

PC Code: 080801

MEMORANDUM

October 11, 1995

SUBJECT: Ametryn Method Evaluation - Report No. ECM0063S1-S4 - "Method Evaluation for the Determination of Ametryn and Three Metabolites in Soil." Method submitted by Ciba Geigy Corporation and validated by Science Applications International Corporation for BEAD/ACB/ECS.

TO: Kathryn Davis, PM 52
Special Review and Reregistration Division (7508W)

FROM: Dana S. Spatz, Chemist
Chemistry Review Section 2
Environmental Fate and Ground Water Branch
Environmental Fate and Effects Division (7507C)

THROUGH: Henry Jacoby, Chief
Environmental Fate and Ground Water Branch
Environmental Fate and Effects Division (7507C)

Mah T. Shamim, Ph.D., Section Head
Chemistry Review Section 2
Environmental Fate and Ground Water Branch
Environmental Fate and Effects Division (7507C)

The Environmental Chemistry Section of BEAD/ACB has completed the validation of the analytical method referenced above. In summary, it was found that method performance met project recovery and precision objectives (70-120% recovery, RSD \leq 20%) for ametryn and three metabolites of ametryn in soil samples at all fortification levels examined.

The registrant's HPLC solvent programs were not directly transferable to the validating laboratory's (SAIC) equipment. In order to retain the metabolites well beyond the solvent front, SAIC modified the solvent program by increasing the initial buffer content and adjusting the gradient. As a consequence, all three metabolites could be measured in a single run.

Please inform the registrant of the results of the method validation.



2006676

2.1 Data Summary from Soil Samples

2.1.1 Ametryn

Spike Level ($\mu\text{g}/\text{Kg}$)	Mean Rec.	SD ¹ Rec.	RSD ² Rec.	Mean Conc.	SD ² Conc.
LOQ (10.0)	74.7	10.9	14.6	7.5	1.1
10xLOQ (100)	110.8	7.9	7.1	110.8	7.9

¹ SD = Standard Deviation

² RSD = Relative Standard Deviation

2.1.2 Ametryn Metabolites

Spike Level ($\mu\text{g}/\text{Kg}$)	Mean Rec.	SD ¹ Rec.	RSD ² Rec.	Mean Conc.	SD ² Conc.
LOQ (10.0) GS-17794	90.9	2.8	3.1	9.09	0.28
LOQ (10.0) GS-11354	91.4	1.6	1.7	9.14	0.16
LOQ (10.0) G-34048	79.4	3.2	4.0	7.94	0.32
10xLOQ (100) GS-17794	86.6	11.1	12.9	86.6	11.1
10xLOQ (100) GS-1354	99.4	4.0	4.0	99.4	4.0
10xLOQ (100) G-34048	77.6	13.4	17.3	77.6	17.3

¹ SD = Standard Deviation

² RSD = Relative Standard Deviation

3.1.2 Analysis Method

The samples for ametryn detection were analyzed by gas chromatography using a Hewlett Packard 5890 Series II GC with a nitrogen-phosphorus detector. Chromatographic conditions were the same as those provided by the registrant except where noted; for any deviation from the registrant's conditions, the registrant's condition is given in parentheses after the actual experimental condition:

Ametryn

Column: 15-m x 0.53-mm i.d. DB-1, 1.5 μ m film thickness
Injector Temperature: 200°C (250°C)
Column Temperature: 180°C for 6 min. then 25°C/min. to 230°C for 4 min.
Detector Temperature: 250°C (330°C)
Flow Rate(s): Air @ 100 mL/min.
Carrier Helium @ 10 mL/min.
Makeup Helium @ 20 mL/min.
Hydrogen @ 4 mL/min.
Detector Type: Nitrogen Phosphorous Detector
Injection Volume: 1 μ L
Retention Time: ca. 5 min.

Registrant's HPLC Conditions

Column: 15-cm x 4.6-mm i.d. Supelco LC8 DB, 5 μ m packing material
Column Temperature: not specified
Detector: UV at 240 nm
Flow Rate: 2.0 mL/min.
Injection Volume: 100 μ L
Mobile Phase GS-11354: 25% acetonitrile/75% water (contains 4 g/L octane sulfonic acid sodium salt, and 4.08 g/L potassium phosphate, monobasic). Isocratic for 7 min., then ramp acetonitrile concentration to 60% in 2 min., hold for 4 min., ramp back to 25:75 acetonitrile/water in 1 min. and hold for 5 min.
Mobile Phase GS-17794: 15% acetonitrile/85% water (contains 4 g/L octane sulfonic acid sodium salt, and 4.08 g/L potassium phosphate, monobasic). Isocratic for 10 min., then ramp acetonitrile concentration to 60% in 1 min., hold for 4 min., ramp back to 15:85 acetonitrile/water in 1 min. and hold for 6 min.

SAIC Modified Program

Column: 15-cm x 4.6-mm i.d. Supelco LC8 DB, 5 μ m packing material
Column Temperature: 40°C
Detector: UV at 240 nm
Flow Rate: 2.0 mL/min.
Injection Volume: 100 μ L
Mobile Phase: 5% acetonitrile/95% water (contains 4 g/L octane sulfonic acid sodium salt, and 4.08 g/L potassium phosphate, monobasic). Isocratic for 2 min., then ramp acetonitrile concentration to 20% in 3 min., hold for 4 min., ramp to 60% acetonitrile in 4 min. and hold for 3 min., then return to 5% acetonitrile in 1 min. and hold for 3 min.
all metabolites: