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

OFFICE OF PREVENTION,  
PESTICIDES AND TOXIC  
SUBSTANCES

September 12, 2002

DP Barcode: ~~D279318~~ 278734  
PC Code: 108801

MEMORANDUM

SUBJECT: S-Metolachlor Lab Method Validation

FROM: Mark Corbin, Environmental Scientist  
Environmental Risk Branch 1  
Environmental Fate and Effects Division (7507C)

*Mark Corbin* 9-15-02

TO: Joanne Miller, Product Manager 23  
Herbicide Branch  
Registration Division , 7505C

Attached is the Environmental Chemistry Laboratory Evaluation (ECME) review dated July 2, 2002 by the Biological and Economic Analysis Division (BEAD) Environmental Chemistry Laboratory (ECL) of the method validation study submitted by Syngenta Crop Protection, Inc. (MRID 454996-11) in support of the herbicide S-Metolachlor. The ECME review indicates that the method presented can be used to monitor surface water for the presence of S-Metolachlor. However, due to the potential for cross-reactivity, ECL recommends the necessity of confirmatory analysis by a conventional technique.

If you have any question please contact Mark Corbin at (703)-605-0033.





UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
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July 02, 2002

MEMORANDUM

DP Barcode: D279315

SUBJECT: S-Metolachlor Lab Method Validation  
Report No. ECM0200W1

FROM: Aubry E. Dupuy, Jr., Chief  
BEAD/Environmental Chemistry Lab

*Aubry E. Dupuy Jr.*

TO: Mark Corbin  
EFED/Environmental Risk Branch 1 (7507-C)

As requested ECL has completed an Environmental Chemistry Lab validation for S-metolachlor in water, MRID #454996-11, using a method submitted by Syngenta Crop Protection, Inc., entitled "Analytical Method for the Determination of S-Metolachlor in Water by Enzyme Immunoassay Including Validation Data".

This method was evaluated with reagent water fortified at three levels; 0.05 ppb, 0.075 ppb, and 0.75 ppb, and with quadruplicate analyses at each level.

The attached method lab evaluation report includes three parts:

Part I: Summary and Conclusions

In this section any problems encountered with the method and how they were handled are discussed. ECL's opinion of how well the method performed is also presented.

## Part II: Analytical Results

In this section the individual results of each sample at each spiking level are listed. The average percent recovery and relative standard deviation (RSD) for each spiking level is also presented here.

## Part III: Experimental Details

In this section any modifications that were made, instrument parameters, representative sample calculations and standard curve are listed and/or discussed.

If you have any questions concerning this report, please contact Henry Shoemaker at (228) 688-1222 or Aubry Dupuy at (228) 688-3212.

## Attachments

cc: Christian Byrne, QA Officer  
BEAD/Environmental Chemistry Lab

Henry Shoemaker, Chemist  
BEAD/Environmental Chemistry Lab

ENVIRONMENTAL CHEMISTRY METHOD LAB EVALUATION REPORT  
NUMBER ECM 0200W1

S-Metolachlor in Water

ENVIRONMENTAL CHEMISTRY LABORATORY  
BIOLOGICAL AND ECONOMIC ANALYSIS DIVISION

Prepared by: Henry Shoemaker  
Henry Shoemaker, ECL Chemist

Date: 6/19/02

Reviewed by: Christian Byrne  
Christian Byrne, ECL QA Officer

Date: 06/25/02

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## PART I

## SUMMARY AND CONCLUSIONS

ECL has completed an Environmental Chemistry Method Laboratory Evaluation of S-metolachlor in water. This method, MRID# 454996-11, submitted by Syngenta Crop Protection, Inc., is titled "Analytical Method for the Determination of S-Metolachlor in Water by Enzyme Immunoassay Including Validation Data".

In order to evaluate this method a water matrix was fortified with S-metolachlor at 0.05, 0.075, and 0.75 ppb. All samples were done in replicates of four at each level. The registrant's method limit of detection (LOD) of 0.05 ppb and the method limit of quantitation (LOQ) of 0.075 ppb of S-metolachlor in water were confirmed by this data. ECL found the precision to be within the target limits of  $\leq 20\%$  relative standard deviation (RSD) at or above the (LOQ). For example, the (RSD) is 14.3% at the (LOQ) level and 17.8% at the (10 x LOQ) level. The mean recoveries of 97.4% for S-metolachlor at the (LOQ) level and 104% at the (10 x LOQ) level are well within the target range of 70% to 120%.

ECL encountered no problems with the method and feel that it could be used as described by the registrant for low-cost monitoring of water for S-metolachlor. However, since there can be some potential for cross-reactivity from other compounds, ECL emphasizes the necessity of confirmatory analysis by a conventional technique.

## PART II

## ANALYTICAL RESULTS FOR S-METOLACHLOR

## EPA RECOVERIES IN WATER

Sample	Added (ppb)	Found (ppb)	Recovery (%)	Statistics
SM 01	0	0		
SM 02	0	0		
SM 03	0.05	0.04		
SM 04	0.05	0.05		
SM 05	0.05	0.04		
SM 06	0.05	0.02		
SM 07	0.075	0.058	77.3	mean(Rec) = 97.4%
SM 08	0.075	0.080	106.7	SD = 13.89
SM 09	0.075	0.080	106.7	RSD = 14.3%
SM 10	0.075	0.074	98.7	
SM 11	0.75	0.58	77.3	mean(Rec) = 104.0%
SM 12	0.75	0.83	110.7	SD = 18.53
SM 13	0.75	0.81	108.0	RSD = 17.8%
SM 14	0.75	0.90	120.0	

## PART III

## EXPERIMENTAL SUMMARY

(a) Principle of Method

A 0.50 mL aliquot of a water sample is added to a polystyrene culture tube coated with S-metolachlor antibody. The assay is carried out by sequential addition of enzyme conjugate, wash water, and color reagent. The reaction is terminated by acidification. Quantification is performed spectrophotometrically at 450 nm.

(b) Source of Analytical Reference Standard

Analytical standard of racemic metolachlor, lot# S98-2315, was obtained from the EPA National Pesticide Standard Repository, Fort Meade, Maryland, with certified purity of 98%. Refer to Appendix I for the chemical name and structure of S-metolachlor.

(c) Source of Sample Matrix

Matrix used was reagent grade water, Fisher Optima, lot 991185.

(d) Instrumentation for Quantitation

Photometer, fixed wavelength spectrophotometer RPA-1 RaPID Analyzer, Strategic Diagnostics Inc.

(e) Modification of Method

The RPA-1 spectrophotometer was set up to use a ln/logit B data transformation calculation, while the registrant used a log/linear regression function. A comparison of the two methods yields almost identical results.

(f) Time for Experiment

The time for preparing and analyzing a set of fourteen samples, including standards and controls, is one day for one person.



(g) Calculations

1. Standard Curve

The RPA-1 RaPID® Analyzer contains preprogrammed data reduction capabilities which calculates a standard curve for each analytical set using the absorbances of the calibration standards; which had concentrations of 0.05 ppb, 0.10 ppb, 0.40 ppb, and 1.00 ppb of S-metolachlor. The calibration curve is constructed using linear regression after performing a  $\ln(\text{conc.})$  and  $\text{Logit}(B/B_0)$  data transformation of the concentration and absorbance values respectively.

The calibration standard curve equation is:

$$\text{Logit}(B/B_0) = \text{slope}(\ln \text{ of conc.}) + Y \text{ intercept}$$

Where:

$$\text{Logit}(B/B_0) = \ln \left\{ \frac{B/B_0}{1-(B/B_0)} \right\}$$

B = the absorbance for a specific sample.

$B_0$  = the absorbance measured for the zero standard.

Conc. = the concentration of S-metolachlor (ppb).

2. Calculation of Analyte in Samples

Standards were processed and analyzed in duplicate. The RPA-1 Analyzer measured the absorbances of each sample and calculated the concentrations (ppb) of S-metolachlor using the regression equation generated from calibration standards in the sample set.

### 3. Example of manual calculation

Sample no. SM 12 for S-metolachlor

To manually calculate the concentration (ppb), use the following equation:

$$\text{Conc.} = e^a, \text{ where:}$$

$$a = [\text{Logit}(B/B_0) - (\text{Y intercept})] / \text{slope}$$

Mean absorbance of sample SM 12 = 0.728

Slope of calibration curve = - 0.643

Y intercept of calibration curve = - 0.987

Mean absorbance of zero standard = 2.460

$$B/B_0 = 0.728 / 2.460 = 0.296$$

$$\text{Logit}(B/B_0) = \ln[0.296/(1 - 0.296)] = - 0.868$$

$$\text{Conc} = e^{(-0.868 + 0.987)/(- 0.643)}$$

$$= e^{(- 0.185)}$$

$$= 0.83 \text{ ppb}$$

#### (h) Graphs and Data

Pages (8) and (9) contain a print-out of the calibration curve and data generated by the S-metolachlor standards and samples.

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ECM 0200W1

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\*\*\*\*\* OHMICRON \*\*\*\*\*

PROTOCOL : METOLACH

TECH ID : Amey Chomakey

LOT # : 31092

EXP DATE: JAN/03

Data Reduct:Lin.Regression  
Xformation: Ln/LgtB  
Read Mode : Absorbance  
Wavelength : 450 nm  
Units : PPB

EQUATION OF LINE :

Slope = -0.643  
Intercept = -0.987  
Corr (r) = 0.9996

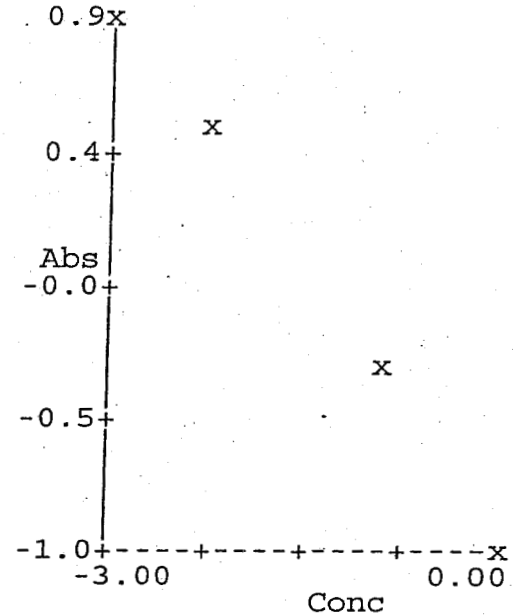
Transformed Data :

Conc	Abs
-3.00	0.925
-2.30	0.497
-0.92	-0.366
0.00	-1.011

Calibrator Data:

Conc	Abs Diff	%CV	Predic %Diff
0.00	2.477		
	2.443		
Mean	2.460	1.0	
0.05	1.811		0.04
	-0.006		-14.8
	1.712		0.06
	0.009		15.7
Mean	1.762	4.0	0.05
	0.001		2.0
0.10	1.532		0.10
	-0.001		-1.4
	1.526		0.10
	0.000		0.2
Mean	1.529	0.3	0.10
	-0.001		-0.6
0.40	1.000		0.39
	-0.012		-3.1
	1.015		0.37
	-0.027		-7.3
Mean	1.007	1.1	0.38
	-0.020		-5.2
1.00	0.637		1.11
	0.105		9.5
	0.676		0.97
	-0.026		-2.6
Mean	0.656	4.2	1.04
	0.037		3.6

Cal. Curve : †

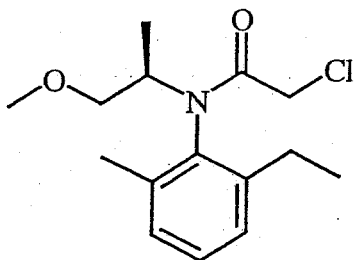


Samples Data :

Spl#	Abs	Conc
1	2.513	nd
ID: <u>SM-01</u>		
2	2.490	nd
ID: <u>SM-02</u>		
3	1.859	0.04
ID: <u>SM-03</u>		
4	1.801	0.05
ID: <u>SM-04</u>		
5	1.862	0.04
ID: <u>SM-05</u>		
6	2.025	0.02nd
ID: <u>SM-06</u>		
7	1.717	0.06
ID: <u>SM-07</u>		
8	1.611	0.08
ID: <u>SM-08</u>		
9	1.612	0.08
ID: <u>SM-09</u>		
10	1.636	0.07
ID: <u>SM-10</u>		
11	0.850	0.58
ID: <u>SM-11</u>		
12	0.728	0.83
ID: <u>SM-12</u>		
13	0.734	0.81
ID: <u>SM-13</u>		
14	0.702	0.90
ID: <u>SM-14</u>		

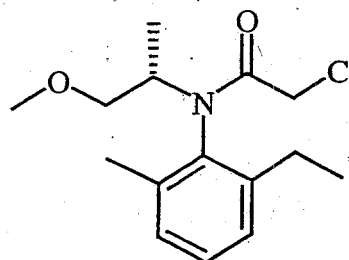
END OF RUN  
04-24-02 14:14:01

Acetamide, 2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-[(1*S*)-2-methoxy-1-methylethyl]-

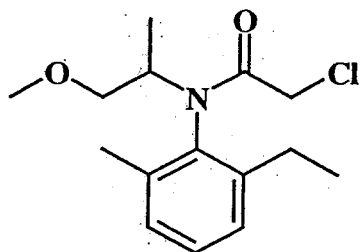


*S*-Metolachlor  
(CGA-77102)

Acetamide, 2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-[(1*R*)-2-methoxy-1-methylethyl]-



*R*-Metolachlor  
(CGA-77101)



Metolachlor

Acetamide, 2-chloro-*N*-(2-ethyl-6-methylphenyl)-*N*-[2-methoxy-1-methylethyl]-