

## I. Introduction

The purpose of this report is to summarize the results of gas chromatography-mass spectrometry (GC/MS) confirmation of selected soil samples from metolachlor field dissipation study, California-Site C. Samples for GC/MS confirmation were provided by Agriseach, Inc., Frederick, Maryland after gas chromatography analyses using a flame ionization detector (GC/FID). Representative sample extracts showing positive detections of metolachlor or any of the metabolites of metolachlor: CGA-50720, CGA-40919, CGA-40172 or CGA-51202, were analyzed by GC/MS using single ion monitoring (SIM) approach. The screening level for GC/MS confirmation was 0.1ng/ul. This report describes the procedure used and summary of data for GC/MS confirmation of samples requested by Ciba-Geigy Corporation, Greensboro, North Carolina. Questions regarding the GC/MS confirmations mentioned in this report can be directed to James E. Whetzel, EMS Laboratories, phone number 704/393-1853.

## II. Experimental

### A). Materials

- 1) Autosampler vials, 100 ul
- 2) Hexane (pesticide grade)
- 3) Gas Chromatographic Standards which were prepared from an Agriseach, Inc. 25 ug/ml stock solution of metolachlor, metabolites: CGA-40172, CGA-40919, and methylated metabolites: CGA-51202, CGA-50720.

### B). Shipping

- 1) All extracts were shipped (in dry ice) over night in coolers from Agriseach, Inc. to EMS Laboratories.
- 2) Extracts were accompanied by a sample transmittal form (chain-of-custody) that had been filled out by the person relinquishing the extracts. A signed copy of this form was returned to Agriseach, Inc.
- 3) Extracts were logged into the LIMS (Laboratory Information Management System) and given unique identification numbers. Extracts were stored under refrigeration until removal for analysis. All retainer extracts were kept under refrigeration for 90 days after issuance of final report.

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c). Analytical Method

Analyses were performed using a gas chromatograph/mass spectrometer (GC/MS) in single ion monitoring (SIM) mode to specifically detect and confirm the presence of metolachlor and metabolites: CGA-50720, CGA-40172, CGA-40919 or CGA-51202.

Ions used for SIM analysis were chosen from mass spectra produced by scanning from 35 to 500 AMU each compound of interest. For each compound, the five most abundant ions greater than 100 AMU were chosen as the group to monitor. The most abundant ion of the group was chosen to be the quantitation ion. (Ions less than 100 AMU were disqualified to minimize interferences). Due to the close elution pattern and low sensitivity of CGA-40172 and CGA-51202, one group of four ions was selected to monitor both metabolites.

1) Gas Chromatographic Conditions

Instrument: Hewlett Packard 5890 Gas Chromatograph  
Column: Capillary J & W DB-5, 0.25mm ID, 30m length, 0.25um film  
Injector Temperature: 285°C  
Oven Temperature: 180°C  
Analysis Time: 19 minutes  
Carrier Flow: Helium at 1-2ml/min. at 10 psi

2) Detector Conditions

Detector: Hewlett Packard 5970 Mass Selective Detector in SIM mode.  
Detector Temp: 285°C  
Multiplier Voltage: 2200 volts  
Number of groups: 4

Group 1 start/stop scan: 5.00/7.50

	M/Z	Dwell (MSEC)
Quant ion CGA-50720	134.1	100
	146.0	100
	162.1	100
	163.1	100
	221.1	100

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Group 2 start/stop scan: 7.50/10.00

	M/Z	Dwell (MSEC)
	146.0	100
	160.1	100
Quant ion CGA-40919	161.1	100
	188.1	100
	233.1	100

Group 3 start/stop scan: 10.00/13.00

	M/Z	Dwell (MSEC)
	146.1	125
Quant ion CGA-40172	162.1	125
	220.1	125
Quant ion CGA-51202	248.1	125

Group 4 start/stop scan: 13.00/19.00

	M/Z	Dwell (MSEC)
	146.1	100
Quant ion Metachlor	162.1	100
	211.1	100
	238.1	100
	240.1	100

3. Instrument Calibration

Prior to the analysis of extracts, known quantities of metolachlor and the metabolites of interest were injected to construct an initial calibration curve for each compound. Calibration curves for concentrations between 0.10ng and 2.00ng were prepared by plotting the concentration of the standard versus the peak area of quantitation ion. Second degree equations of the curves were determined and used in all low level calculations. Standards containing metolachlor and each of the metabolites of interest were analyzed throughout the run to check the calibration.

4. Detection Limits

The method detection limit (MDL) was determined by the method listed in 40 CFR Part 136, App. B. The detection limit reported for the analysis is 0.1ng/ul.

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5. Calculation Method

The ng/ul values found for each extract were determined by the external standard method. The area of the quantitation ion of each compound found was substituted into the appropriate second degree equation as determined by the initial calibration, and ng/ul value was generated.

D. Quality Control and Archiving of Data

- 1) All analyses were performed in compliance with Good Laboratory Practices (40CFR, Part 160). All raw data was audited and approved by the Quality Assurance Officer.
- 2) All original raw data has been stored in the archives at EMS Laboratories, 4132 Pompano Rd., Charlotte, NC 28216. Copies of raw data have been provided to Ciba-Geigy Corporation.