Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Dimethenamide

MRID #:   440832-07

Matrix: Soil

Analysis: TLC/RIS

This method is provided to you by the Environmental Protection Agency's (EPA) Environmental Chemistry Laboratory (ECL). This method *is not* an EPA method but one which was submitted to EPA by the pesticide manufacturer to support product registration. EPA recognizes that the methods may be of some utility to state, tribal, and local authorities, but makes no claim of validity by posting these methods. Although the Agency reviews *all* Environmental Chemistry Methods submitted in support of pesticide registration, the ECL evaluates only about 30% of the currently available methods. Most methods perform satisfactorily but some, particularly the older methods, have deficiencies. Moreover, the print quality of the methods varies considerably because the methods originate from different sources. Therefore, the methods offered represent the best available copies.

If you have difficulties in downloading the method, or further questions concerning the methods, you may contact Elizabeth Flynt at 228-688-2410 or via e-mail at flynt.elizabeth@epa.gov.

STUDY TITLE

Soil Thin Layer Chromatography of SAN 582H

DATA REQUIREMENT

Canada Pesticide Registration Requirement as listed in
Environmental Chemistry and Fate Guidelines, leaching
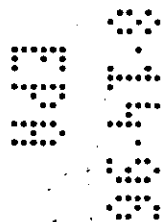(Page 29), 1987

DATE COMPLETED

February 28, 1991

Author: Tsun-Min Rosa Tong
Supervisor: Paul Moore
Department Head: Y.H. Atallah

TESTING FACILITY

Sandoz Agro, Inc.
Analytical Sciences and Formulations
1300 E. Touhy Ave.
Des Plaines, IL 60018

Report No. 414105-18

APPENDIX I

PROTOCOL AND AMENDMENT

PROTOCOL

SOIL THIN-LAYER CHROMATOGRAPHY

A. OBJECTIVE:

The objective of this study is to use thin layer chromatography to
estimate the leaching potential of a test material and its
degradates through various test soils. The present protocol is
designed to meet environment (Canada) test requirements, in
accordance with Sandoz Crop Protection Corporation's Good
Laboratory Practices.

B. TEST COMPOUND:

The preferred radiolabeled test compound would have a
radiochemical purity of greater than 95% and specific activity
greater than 10 mCi/mmole. Information required to characterize
the radiolabeled test compound includes: (1) compound name, (2)
structure, (3) radiolabel position, (4) radiochemical purity, (5)
source, and (6) specific activity. If it is necessary to dilute
the labelled test compound with non-labeled test compound, the
non-labeled compound should be analytical reference grade. All
other chemicals will be reagent grade quality or better; solvents
will be glass distilled.

C. GENERAL PROCEDURE AND TEST CONDITIONS:

(1) Soil Types

This test will be conducted using three types of soils. One of the soils utilized in the study will be the same as that used for the aerobic soil metabolism study. Three replicate plates will be prepared for each soil type.

(2) Soil Treatment and Aging

The aerobic aging portion of the study will be conducted for 30 days or for one half-life of the test compound in soil, whichever is shorter. The aging will be conducted at 25°C ± 1°C in darkness with the test soil at 75% of its 0.33 bar moisture level. Soil moisture will be maintained by weighing the vessel on a weekly basis and adding dionized water if weight loss is observed.

The concentration of test chemical in the soil will be equal to the maximum single application rate. This will generally be in the range of 0.1 to 10 ug/g of soil. The vessel containing the treated soil will be equipped with glass inlet and outlet tubes. The inlet tubes will be connected to an air supply; this air supply passes through a gas washing bottle containing 1.5 N KOH followed by a gas washing bottle containing distilled water, in order to provide 10 ml/min of CO_2-free moist air. The outlet tubes will be connected in series to a gas washing bottle containing ethylene glycol, followed by another gas washing bottle containing 1.5 N KOH. Volatile organic compounds are captured by the ethylene glycol, while the 1.5 N KOH traps respired CO_2. The contents of the CO_2 and organic trapping bottles will be radioassayed and replaced at weekly intervals.

An initial zero-time sample of the treated soil will be
immediately frozen and 0.5 g subsample in triplicate will be
radioassayed by combustion in a Packard 306 Sample Oxidizer.
The remaining treated soil will be incubated until the
termination of the aging period (not more than 30 days).

(3) Radiocarbon Extraction and Characterization

Radiolabeled compounds in the aged soil sample will be
extracted with appropriate solvent(s) according to
established procedures. Unextractable residues will be
determined in the extracted sample by combustion/radioassay
of triplicate 0.5 g subsamples.

A material balance will then be calculated for the soil at
the end of the aging portion of the study; the balance will
include $^{14}CO_2$, volatile organic fraction, extractable
residues and unextractable residues. The extractable
residues will then be identified by chromatographic and/or
spectrometric methods. All radiocarbon fractions
(degradates) greater than or equal to 10% of the applied
dosage at zero time will be identified, if possible.

The collected ^{14}C-residue fraction(s) (extractable parent
and degradate fractions) are now tested for the mobility
within the three test soils.

(4) TLC Soil Sample Preparation

Each test soil sample will be individually air dried at
20-30°C and gently ground to reduce aggregate size. Then the
soil will be sieved through a 500 μm sieve. Deionized water
at pH 7.0 is added to the sieved soil until a smooth
moderately fluid slurry is attained.

(5) Preparation of TLC Plates

The soil slurry is evenly and quickly across the clean glass
20 x 20 cm glass plates by using a variable thickness plate
spreader. The soil layer thickness should be between 0.5 to
1.0 mm.

After achieving a uniform slurry application, the plates are
air dried at 25°C for a minimum of 24 hours. After the
drying period a horizontal line is scribed 12.0 cm above the
glass plate bottom edge through the soil layer, exposing the
plate surface. (This channel provides a consistent stopping
point for solvent migration.)

(6) Chemical Mobility

The ^{14}C labeled test compound (0.1 to 1.0 ug) and a
pesticide standard, whose soil mobility characteristics are
already known, are spotted 2.0 cm above the bottom edge of
the plate. After spotting, the soil plate is immersed with
its bottom edge down, at some angle different from vertical,
in a closed chromatographic chamber containing deionized
water at a depth of 0.5 cm. Upon immersion, the plates are
only removed after the water front reaches the scribed line,
at which time they are air-dried.

(7) Determination of R_f Value

An AMBIS TLC radiographic imaging system is used to determine
the location of the radioactive zones on the soil plate. The
R_f value is calculated by dividing the migration distance
of the test compound by the migration distance of the solvent
front. The identify of the radioactive compounds is then
established and reported.

D. CALCULATIONS

The R_f value is the ratio of the distance traveled by the test material to the distance traveled by the solvent front. The mobility of the test substance and its degradates will be reported according to the following table of mobility classes, which are based on the calculated R_f values for the respective compounds.

Table 1

R_f	Class Number	Soil Mobility Class
0.00 to 0.09	1	Immobile
0.10 to 0.34	2	Low
0.35 to 0.64	3	Intermediate
0.65 to 0.89	4	Mobile
0.90 to 1.00	5	Very Mobile

The relationship between R_f and the soil/water distribution coefficient, K_d, is given by:

$$R_f = [1 + k_d(d_s)(1/\theta^{2/3}-1)]^{-1}$$

where, d_s is bulk density of soil, and θ is the soil porosity.

K_d will be estimated using the known soil characteristics and the R_f values determined during the soil thin layer chromatography study.

E.   DATA REPORTING

All raw data and calculations obtained from the soil thin-layer
chromatography study will be reported. In addition, the following
general information will be included with the report: (a) study
initiation and termination dates; (b) test location; (c) names of
principal investigators; (d) this protocol and any amendments; and
(e) test chemical description. (See Section "B", Test Compound",
of this protocol.) The soil characteristics used to estimate
K_d will also be incorporated with the report. (See "D.
Calculations" above.)

The soil thin-layer chromatography study will be conducted in
accordance with Sandoz Crop Protection's Good Laboratory Practice
Procedures. The completed report for this study is signed by the
scientist(s) responsible for its completion and evaluation.

For Test Chemical: _____SAN 582H_____

APPROVALS:

Tsun-Min Rosa Tong   _9/14/90_
Study Director   Date

J. H. Atallah   _Sept. 14, 90_
Director of Analytical Sciences   Date
and Formulations

SANDOZ CROP PROTECTION CORPORATION
INTER-OFFICE CORRESPONDENCE

DATE: September 19, 1990   COPIES TO: Y. H. Atallah
                                                       D. C. Judson

TO: Project File

FROM: Tsun-Min Rosa Tong

SUBJECT: Amendment to Protocol

The Protocol "Soil Thin-Layer Chromatography" is amended as follows:

Amendment

1. In Section B. Test Compound: add information as follows:

    SAN-582H, 2-chloro-N-[(1-methyl-2-methoxy)ethyl]-N-(2,4-dimethyl-3-^{14}C-thien-3-yl)acetamide

    CAS # 87674-68-8

    Purity: 99.3%

    Specificity: 43.2 mCi/mmole

2. Add this information: Study Sponsor

    This study will be carried out for:
            Sandoz Crop Protection Corporation
            1300 East Touhy Avenue
            Des Plaines, Illinois 60018

3. Add this information: Study Site

    This study will be carried out at:
            Sandoz Crop Protection Corporation
            1300 East Touhy Avenue
            Des Plaines, Illinois 60018

4. Add this information: Study Period

            Estimated Starting Date: September 14, 1990
            Estimated Completion Date: December 30, 1990

5. Add this information: Statistic Methods:

            A simple average will be used for this study.

Reason for Change:

The original protocol does not include Amendment 1. to 5. information.

Amendment:

6. Section C. General Procedure and Test Conditions: (2) Soil treat-
    ment and aging will not be performed.

Reason for Change:

The 30 day aged SAN-582H soil has been collected from the Aerobic Soil
Metabolism of SAN-582H study.

Tsun-Min Rosa Tong
_____
T.M. R. Tong

TMRT/rg

APPENDIX II

SAMPLE CALCULATION FOR K_d AND Koc

Kenyon Loam (SAN-582H)

Calculated according to Hamaker (1975):

$$R_f = \frac{1}{1 + \{K_{oc}(\% \text{ org. carbon}/100)(\text{bulk density})[(1/\text{vol. } H_2O)^{2/3}-1]\}}$$

where: bulk density is g/cm^3 and vol. H_2O content is cm^3/cm^3

$$k_d = K_{oc}\frac{(\% \text{ organic carbon})}{100}$$

$$0.21 = \frac{1}{1 + \{K_{oc}(2.2/100)(1.2)[(1/0.451)^{2/3}-1]\}}$$

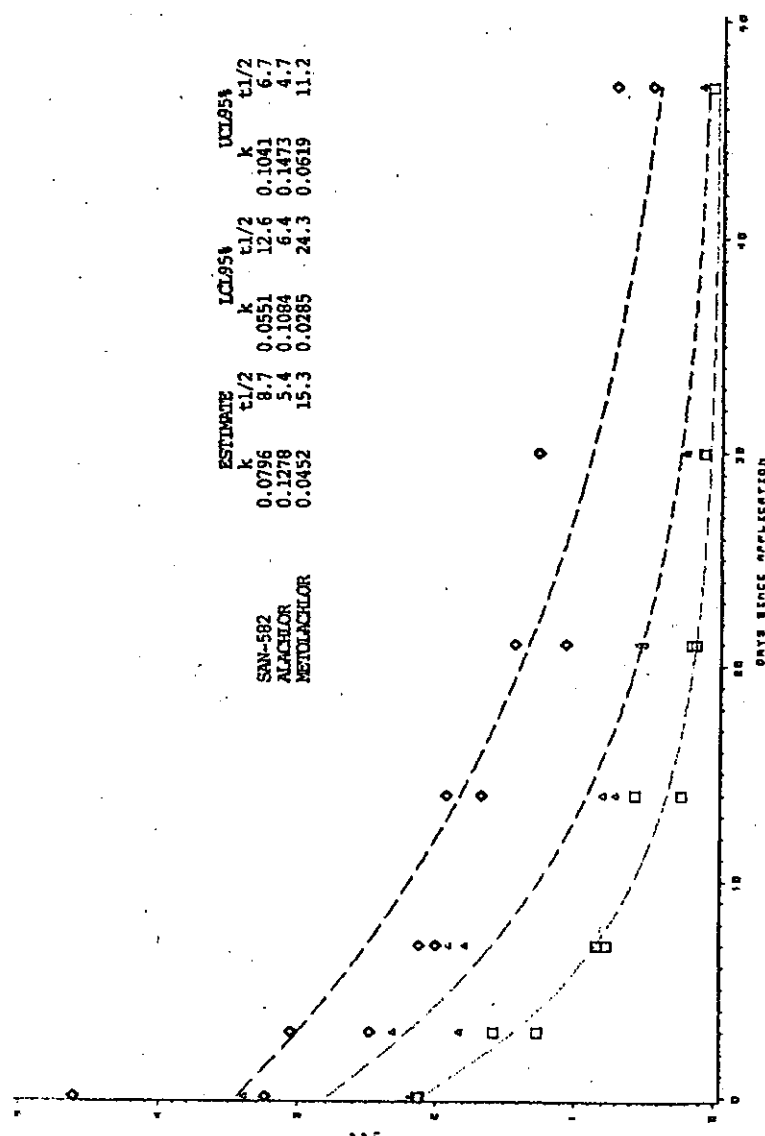$K_{oc} = 83.80$

$k_d = 83.80 \times \frac{(2.2)}{100}$

$ = 1.84$

$R_f = 0.21$ for SAN-582H in Kenyon Loam

% org. Carbon $= 2.2$ in Kenyon Loam

bulk density $= 1.2$ g/cm^3 in Kenyon Loam

Vol. H_2O Content $= 0.451$ cm^3/cm^3 for loam soil

FIGURE 1.

RATE OF DISSIPATION OF SAN-582, ALACHLOR, AND METOLACHLOR

Project 414105-Soil Degradation of SAN 582H, Alachlor and Metolachlor