

Data Evaluation Report on the adsorption-desorption of BAS 510F Metabolite M510F47 in soil

PMRA Submission Number {.....}

EPA MRID Number 45405217

Data Requirement: PMRA Data Code:
EPA DP Barcode: D278387
OECD Data Point:
EPA Guideline: 163-1

Test material:

Common name: BAS 510 F Metabolite M510F47

Chemical name

IUPAC: 2-Chloronicotinic acid

CAS name:

CAS No: Not provided

Synonyms: M510F47

SMILES string:

Primary Reviewer: Dana Worcester
Dynamac Corporation

Signature: *Dana Worcester*
Date: 1/15/02

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Dynamac Corporation

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Date: 1/15/02

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Date: 1/15/02

Company Code: [for PMRA]
Active Code: [for PMRA]
Use Site Category: [for PMRA]
EPA PC Code: 128008

CITATION: Seher, A. 2001. Adsorption/desorption - study of 107371 (M510F47) on 3 US Soils. Unpublished study performed by BASF Aktiengesellschaft, Limburgerhof, Germany. Sponsored by BASF Corporation, Research Triangle Park, NC. BASF Registration Document No. 2001/1000967. Study completed February 5, 2001.



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EXECUTIVE SUMMARY:

The adsorption/desorption characteristics of the BAS 510 F metabolite [pyridine-3-¹⁴C]M510F47 (2-chloronicotinic acid) was studied in three U.S. soils: (a) silt loam soil [pH- 5.6, organic matter - 2.1%], (b) sandy loam soil [pH - 7.8, organic matter - 4.8%], and (c) loamy sand soil [pH - 7.0, organic matter - 1.3%], in a batch equilibrium experiment. The experiment was conducted in accordance with the U.S. EPA Pesticide Guidelines Subdivision N, 163-1 and OECD Guidelines for Testing of Chemicals, "Adsorption/Desorption", Guideline 106 (January, 2000), and in compliance with the GLP standard 40 CFR Part 160 and Federal Republic of Germany-GLP (1994). The adsorption phase of the study was carried out by equilibrating air-dried soil with M510F47 at nominal concentrations of 5, 1, 0.2, and 0.04 µg/mL at 21 ± 1°C for 24 hours in the dark. The equilibrating solution used was 0.01 M CaCl₂, with a soil/solution ratio of 1:1 (w:v) for all three soils. The desorption phase of the study was carried out by replacing the adsorption solution with an equivalent volume of sterilized, pesticide-free 0.01 M CaCl₂ solution and equilibrating for 16 hours. The desorption phase was repeated once.

The supernatant solution after adsorption and desorption was separated by centrifugation and duplicate aliquots were analysed for total radioactivity using LSC.

Mass balances were determined at the highest concentration (5 µg/mL) using only the test substance concentration in the solution from the adsorption and two desorption steps; residues in the test soils were not analyzed. Mass balance for the loamy sand soil was 99.3% of the applied at the end of the adsorption phase; no desorption could be measured. Mass balance for the silt loam soil was 95.2% of the applied at the end of the desorption phase; no adsorption could be measured. No adsorption or desorption could be measured for the sandy loam soil.

After 24 hours of equilibration, 6.70-8.46% and 0.76-1.00% of the applied M510F47 was adsorbed to the silt loam and loamy sand soils, respectively (reviewer-calculated). No adsorption could be measured for the sandy loam soil. Freundlich K_{ads} values were 0.078 and 0.009 mL/g for the silt loam and loamy sand soils, respectively; corresponding K_{oc} values were 1 and 6 mL/g for the silt loam and loamy sand soils. The coefficients of determination (r^2) for the relationships K_{ads} vs. organic carbon, K_{ads} vs. pH, and K_{ads} vs. clay content were 0.20, 0.96 and 0.02, respectively. At the end of the two desorption steps, 0.00-27.68% of the adsorbed amount was desorbed from the silt loam soil (reviewer-calculated). For the silt loam soil, the Freundlich K_{des} value was 2.36 mL/g, and corresponding K_{oc} value was 190 mL/g. No desorption could be measured for the sandy loam and loamy sand soils.

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Results Synopsis: Adsorption and desorption values determined using Freundlich isotherm equations

Soil type: Silt loam

Amount adsorbed: 6.70-8.46% of the applied

Adsorption K_d : 0.078 (very mobile)

Adsorption K_{oc} : 6

Amount desorbed: 0.00-27.68% of the adsorbed compound

Desorption K_d : 2.36

Desorption K_{oc} : 190

Soil type: Sandy loam

Amount adsorbed: No adsorption could be measured (very mobile)

Adsorption K_d : Not determined

Adsorption K_{oc} : Not determined

Amount desorbed: Not determined

Desorption K_d : Not determined

Desorption K_{oc} : Not determined

Soil type: Loamy sand

Amount adsorbed: 0.76-1.00%

Adsorption K_d : 0.009 (very mobile)

Adsorption K_{oc} : 1

Amount desorbed: No desorption could be measured

Desorption K_d : Not determined

Desorption K_{oc} : Not determined

Study Acceptability: This study is classified as supplemental and provides information on the mobility (adsorption/desorption) of a degradate of BAS 510 F. However, data indicating that acceptable material balances were achieved at all test concentrations were not submitted. Such data are required to confirm the scientific validity of the study. It is noted, however, that the test compound was determined to be very mobile in all test soils.

I. MATERIALS AND METHODS

GUIDELINE FOLLOWED: The study was conducted according to U.S. EPA Pesticide Assessment Guidelines Subdivision N, Series 163-1 (October 1982) and the OECD Guideline for Testing of Chemicals, "Adsorption/Desorption", Guideline 106 (January 2000). Deviations from Subdivision N 163-1 are:

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Material balances were not determined; residues associated with the soil were not measured. This does not allow for a definitive conclusion concerning the scientific validity of the study.

Only three soils were tested. None of the test soils had an organic matter content of $\leq 1\%$. This does not affect the validity of the study.

It was not stated whether desorption equilibration was conducted in the dark. This does not affect the validity of the study.

COMPLIANCE:

This study was conducted in compliance with 40 CFR Part 160, EPA GLP and Federal Republic of Germany-GLP (1994). Signed and dated GLP, Quality Assurance, Data Confidentiality, and Study Certification statements were provided.

A. MATERIALS:

1. Test Material

M510F47 (BAS 510 F Metabolite)

Chemical Structure:

Description:

Not reported

Purity:

Analytical purity: 99.9%

Lot/Batch No. 01174-216

Radiochemical purity: >99%

Lot/Batch No. 640-2039

Specific activity: 11.3 MBq/mg

Locations of the label: Pyridine-3-¹⁴C

Storage conditions of test chemicals:

Not reported

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Physico-chemical properties of BAS 510 F:

Parameter	Values	Comments
Water solubility	ca. 1.6 g/L in water at 20°C	
Vapour pressure	Not provided	
UV absorption	Not provided	
pK _a	Not provided	
K _{ow}	Not provided	
Stability of Compound at room temperature	Not provided	

Data obtained from p. 10 of the study report.

2. Soil Characteristics

Table 1: Description of soil collection and storage.

Description	Silt loam	Sandy loam	Loamy sand
Geographic location	Clinton County, IL	Santa Maria, CA	Seven Springs, NC
Pesticide use history at the collection site	Not provided	Not provided	Not provided
Collection procedures	Not provided	Not provided	Not provided
Sampling depth (cm)	Not provided	Not provided	Not provided
Storage conditions	Not provided	Not provided	Not provided
Storage length	Not provided	Not provided	Not provided
Soil preparation	Sieved 2 mm	Sieved 2 mm	Sieved 2 mm

Data obtained from p. 17 of the study report.

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Table 2: Properties of the soils.

Property	Clinton County, IL	Santa Maria, CA	Seven Springs, NC
Soil Texture	Silt loam	Sandy loam	Loamy sand
% sand	28	54	80
% silt	56	26	16
% clay	16	20	4
pH (CaCl)	5.6	7.8	7
Organic matter (%)	2.1	4.8	1.3
CEC (meq/100 g)	10.2	25.3	7.2
Moisture at 1/3 atm (%)	30.2	24.4	10.2
Bulk density (lb/cu ft ³)	Not provided	Not provided	Not provided
Biomass (mg microbial C/100 g or CFU or other)	Not provided	Not provided	Not provided
Soil taxonomic classification	Not provided	Not provided	Not provided
Soil mapping unit (for EPA)			

Data obtained from pp. 29-31 of the study report.

B. STUDY DESIGN:

1. Preliminary study:

To determine adsorption to glassware, M510F47 (5 µg/mL) in 0.01 M CaCl₂ solution was placed in glass centrifuge tubes, shaken for 24 hours, and analyzed by LSC (p. 12).

To determine whether the test substance adsorbed to centrifuge glass tubes, an aliquot (25 mL) of a solution containing 5 µg/mL of [¹⁴C]M510F47 in 0.01M CaCl₂ was placed in glass centrifuge tubes, shaken for 24 hours, and analyzed by LSC (p. 14).

To determine the equilibration time, samples of each of the three soils were equilibrated with a nominal 5 µg/mL concentration of M510F47 in 0.01M CaCl₂. The soil:solution (1:1, w:v) slurries were covered, shaken and tested at 2, 4, 8, 16, 24, and 48 hours. All samples were centrifuged and an aliquot was analyzed by LSC.

2. Definitive study experimental conditions:

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Table 3: Study design for the adsorption phase.

Parameters		Silt loam	Sandy loam*	Loamy sand
Condition of soil (air dried/fresh)		Air dried	Air dried	Air dried
Have these soils been used for other laboratory studies ? (specify which)		No	No	No
Soil (g/replicate)		20 g	20 g	20 g
Equilibrium solution used (name and concentration; eg: 0.01N CaCl ₂)		0.01M CaCl ₂	0.01M CaCl ₂	0.01M CaCl ₂
Control used (with salt solution only) (Yes/No)		None	None	None
Test material concentrations	Nominal application rates (µg/mL)	5, 1, 0.2, 0.4	5, 1, 0.2, 0.4	5, 1, 0.2, 0.4
	Analytically measured concentrations (µg/mL)	5.014, 1.003, 0.201, 0.040	5.014, 1.003, 0.201, 0.040	5.014, 1.003, 0.201, 0.040
Identity and concentration of co-solvent, if any		None	None	None
Soil:solution ratio		1:1	1:1	1:1
Initial pH of the equilibration solution, if provided		Not provided	Not provided	Not provided
No. of replications	Controls	2	2	2
	Treatments	2	2	2
Equilibration	Time (hours)	24	24	24
	Temperature (°C)	21 ± 1	21 ± 1	21 ± 1
	Darkness (Yes/No)	Yes	Yes	Yes
	Shaking method	Mechanical shaker	Mechanical shaker	Mechanical shaker
	Shaking time (hours)	24	24	24
Method of separation of supernatant (eg., centrifugation)		Centrifugation	Centrifugation	Centrifugation
Centrifugation	Speed (rpm)	Not reported	Not reported	Not reported
	Duration (min)	Not reported	Not reported	Not reported

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Parameters		Silt loam	Sandy loam*	Loamy sand
	Method of separation of soil and solution	Centrifugation	Centrifugation	Centrifugation

Data obtained from pp. 11-12 of the study report.

* No adsorption could be measured.

Table 4: Study design for the desorption phase.

Parameters		Silt loam	Sandy loam*	Loamy sand
Were the soil residues from the adsorption phase used? If not, describe the method for adsorption using a separate adsorption Table		Yes	Yes	Yes
Amount of test material present in the adsorbed state/adsorbed amount (mg a.i./kg soil)	5.014 mg/kg	0.336	Not determined	0.038
	1.003 mg/kg	0.068		0.009
	0.201 mg/kg	0.017		0.002
	0.040 mg/kg	0.003		0.0004
No. of desorption cycles		2	2	2
Equilibration solution and quantity used per treatment for desorption (eg., 0.01M CaCl ₂)		0.01M CaCl ₂	0.01M CaCl ₂	0.01M CaCl ₂
Soil:solution ratio		1:1	1:1	1:1
Replications	Controls	2	2	2
	Treatments	2	2	2
Desorption equilibration	Time (hours)	16	16	16
	Temperature (°C)	21 ± 1	21 ± 1	21 ± 1
	Darkness	Not provided	Not provided	Not provided
	Shaking method	Mechanical shaker	Mechanical shaker	Mechanical shaker
	Shaking time (hours)	16	16	16
Centrifugation	Speed (rpm or g)	Not reported	Not reported	Not reported
	Duration (min)	Not reported	Not reported	Not reported

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Parameters		Silt loam	Sandy loam*	Loamy sand
	Method of separation of soil and solution	Centrifugation	Centrifugation	Centrifugation
Second desorption	Indicate if the method is same as the first desorption cycle.	Same	Same	Same

Data obtained from pp. 11, 13 and Table 3, p. 19 of the study report.

*No desorption could be measured.

3. Description of analytical procedures:

Extraction/clean up/concentration methods:

Total ^{14}C : Aliquots of the test solutions were analyzed for total radioactivity using LSC.

Non-extractable residues, if any: Not determined.

Derivatization method, if used: A derivatization method was not employed.

Identification and quantification of M510F47 (briefly describe HPLC/GC/TLC/MS conditions; eg., column, mobile phase, detector etc.): For the three test soils, HPLC was conducted using the 5.0 $\mu\text{g/mL}$ solution before and at the end of the study, and the aqueous phase from the 5.0 $\mu\text{g/mL}$ solution following adsorption (p. 13). Identification and quantification of M510F47 were performed by radio-HPLC using the following operating conditions: Nucleosil, 100-5-C-18 column (250 x 4 mm), mobile phase of acetonitrile:water:phosphoric acid (85%) (300:700:5, v:v:v), flow rate 1.0 mL/minute. The identity of M510F47 was confirmed by chromatographic comparison of the HPLC retention time of a reference standard.

Identification and quantification of transformation products, if appropriate (briefly describe HPLC/GC/TLC/MS conditions; eg., column, mobile phase, detector etc.): Transformation products were not identified or quantified.

Detection limits (LOD, LOQ) for M510F47 (indicate the criteria/reference, if provided): Detection limits for M510F47 were not provided.

Detection limits (LOD, LOQ) for the transformation products, if appropriate (indicate the criteria/reference, if provided): Transformation products were not identified or quantified.

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II. RESULTS AND DISCUSSION

A. TEST CONDITIONS: [¹⁴C]M510F47 was stable during the study, based on chromatograms of the solutions at study initiation and completion (pp. 24-28).

B. MASS BALANCE: Mass balances were determined at the highest concentration (5 µg/mL) using only the test substance concentration in the solution from the adsorption and two desorption steps; residues in the test soils were not analyzed. Mass balance for the loamy sand soil was 99.3% of the applied at the end of the adsorption phase; no desorption could be measured. Mass balance for the silt loam soil was 95.2% of the applied at the end of the desorption phase; no adsorption could be measured. No adsorption or desorption could be measured for the sandy loam soil.

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Table 5: Recovery of M510F47, expressed as percentage of applied radioactivity, in soil after adsorption/desorption (mean $\pm$ s.d.).

At the end of the adsorption phase (mean $\pm$ S.E.).			
Matrices	Silt loam	Sandy loam*	Loamy sand**
At the end of the adsorption phase			
Supernatant solution ¹	90.93 $\pm$ 1.0	Not determined	98.35 $\pm$ 0.7
Solid phase (total ¹⁴ C) [†]	7.36 $\pm$ 0.8		0.92 $\pm$ 0.1
Total recovery ³	Not determined		99.3 $\pm$ 0.1
At the end of the desorption phase			
Supernatant solution ²	1.72 $\pm$ 0.5	Not determined	Not determined
Solid phase [†]	5.95 $\pm$ 1.3		
Non-extractable residues in soil, if measured	Not determined		
Total recovery ³	95.2 $\pm$ 0.1		

* No adsorption or desorption could be measured.

** No desorption could be measured.

¹ Reviewer calculated using data obtained from Table 3, p. 19 of the study report. (e.g., Divide aqueous phase 4.606 μ g/mL by initial present 5.014 μ g/mL $\times$ 100 = 91.86%. Means and standard deviations were calculated by the reviewer using Excel.

[†] Soils were not combusted. Reviewer-calculated from data obtained in Table 3, p. 19, in the study report (Divide amount adsorbed by initial $\times$ 100. Means and standard deviations were calculated by the reviewer using Excel.

² Reviewer calculated from data obtained from Table 4, p. 20, of the study report. (e.g., Divide aqueous phase two 0.064 μ g/mL by initial present 5.014 μ g/mL $\times$ 100 = 1.28%.

³ Total recovery for the 5 μ g/mL test solution obtained from Table 5, p. 21 of the study report.

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Table 6: Concentration of M510F47 in the solid and liquid phases at the end of adsorption equilibration period (mean $\pm$ s.d.).

Concentration ($\mu\text{g a.i./mL}$)	Silt loam			Sandy loam*			Loamy sand		
	on soil (mg a.i./kg)	in solution ($\mu\text{g a.i./mL}$)	% adsorbed ¹	on soil (mg a.i./kg)	in solution ($\mu\text{g a.i./mL}$)	% adsorbed	on soil (mg a.i./kg)	in solution ($\mu\text{g a.i./mL}$)	% adsorbed
5.014	0.336 $\pm$ 0.0	4.606 $\pm$ 0.1	6.7 $\pm$ 0.1	Not determined			0.038 $\pm$ 0.0	4.948 $\pm$ 0.0	0.76 $\pm$ 0.1
1.003	0.068 $\pm$ 0.0	0.921 $\pm$ 0.0	6.78 $\pm$ 1.1				0.009 $\pm$ 0.0	0.995 $\pm$ 0.0	0.9 $\pm$ 0.0
0.201	0.017 $\pm$ 0.0	0.181 $\pm$ 0.0	8.46 $\pm$ 0.8				0.002 $\pm$ 0.0	0.197 $\pm$ 0.0	115 $\pm$ 0.1
0.04	0.003 $\pm$ 0.0	0.036 $\pm$ 0.0	8.7 $\pm$ 0.3				0.0004 $\pm$ 0.0	0.039 $\pm$ 0.0	1.0 $\pm$ 0.3

Data obtained from Tables 4-5, pp. 21-22 and Attachment 5-6, pp. 33-34, of the study report. Standard deviations were calculated by the reviewer using Excel.

* No adsorption could be measured.

¹ % adsorbed as the % of the applied; reviewer calculated by dividing total adsorbed by total applied $\times 100$ (e.g., $0.336 \div 5.014 \times 100$)

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Table 7: Concentration of M510F47 in the solid and liquid phases at the end of desorption (total following both desorption steps).

Concentration ($\mu\text{g a.i./mL}$)	Silt loam			Sandy loam*			Loamy sand**		
	on soil (mg a.i./kg)	in solution ($\mu\text{g a.i./mL}$)	% desorbed as % of the adsorbed [†]	on soil (mg/kg)	in solution ($\mu\text{g a.i./mL}$)	% desorbed as % of the adsorbed	on soil (mg/kg)	in solution ($\mu\text{g a.i./mL}$)	% desorbed as % of the adsorbed
5.014	0.243	0.064	27.68	Not determined	Not determined	Not determined	Not determined	Not determined	Not determined
1.003	0.05	0.016	26.47						
0.201	0.013	0.003	23.53						
0.04	0.003	0.001	0						

Data obtained from Tables 6.1-6.5, pp. 26-30, of the study report.

* No desorption could be measured.

[†] Reviewer-calculated by subtracting the amount adsorbed after desorption from the initial adsorbed and dividing by initial adsorbed x 100 (e.g., $0.336 - 0.243 = 0.93 \div 0.336 \times 100 = 27.68\%$)

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Table 8: Freundlich adsorption and desorption constants of M510F47 in the soils.

Soil	Adsorption				Desorption			
	K_{ads}	1/n	r^2	K_{oc}	K_{des}	1/n	r^2	K_{oc}
Silt loam	0.078	0.935	0.9993	6	2.36	0.889	0.9874	190
Sandy loam*	Not determined				Not determined			
Loamy sand**	0.01	0.93	0.9943	1	Not determined			

Data obtained from Tables 3 and 4, pp. 19-20 and pp. 33-35 of the study report.

K - Freundlich adsorption and desorption coefficients; 1/N -Slope of Freundlich adsorption/desorption isotherms.

K_{oc} - Coefficient adsorption per organic carbon (K_d or $K \times 100/\%$ organic carbon).

R^2 - Regression coefficient of Freundlich equation.

* No adsorption or desorption could be measured.

** No desorption could be measured.

C. ADSORPTION: After 24 hours of equilibration, 6.70-8.46% and 0.76-1.00% of the applied M510F47 was adsorbed in the silt loam and loamy sand soils, respectively (Table 3, p. 19). No adsorption could be measured in the sandy loam soil. Freundlich K_{ads} values were 0.078 and 0.009 mL/g for the silt loam and loamy sand soils, respectively; corresponding adsorption K_{oc} values were 6 and 1 mL/g for the silt loam soil and loamy sand soil. The coefficients of determination (r^2) for the relationships K_{ads} vs. organic carbon, K_{ads} vs. pH, and K_{ads} vs. clay content were 0.88, 0.09 and 0.09, respectively.

D. DESORPTION: At the end of the desorption phase, 0.00-27.68% of the adsorbed ^{14}C was desorbed from the silt loam soil (Table 4, p. 20). No desorption could be measured in the sandy loam and loamy sand soils. For the silt loam soil, the Freundlich K_{des} value was 2.36 mL/g and corresponding K_{oc} value was 190 mL/g.

III. STUDY DEFICIENCIES: The objectives of this study were: (i) to study the sorptive behavior of M510F47, in three soils; (ii) to provide data for determining the leaching and runoff potential of the test substance in soil; and (iii) to determine the Freundlich adsorption isotherm in all test soils with >10% adsorption. The study does provide useful supplemental information on the mobility of M510F47 in three soils. However, data indicating that acceptable material balances were achieved at all test concentrations were not submitted. Such data are required to confirm the scientific validity of the study. It is noted, however, that the test compound was determined to be very mobile in all test soils, and a new study is not likely to provide additional useful information.

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IV. REVIEWER'S COMMENTS:

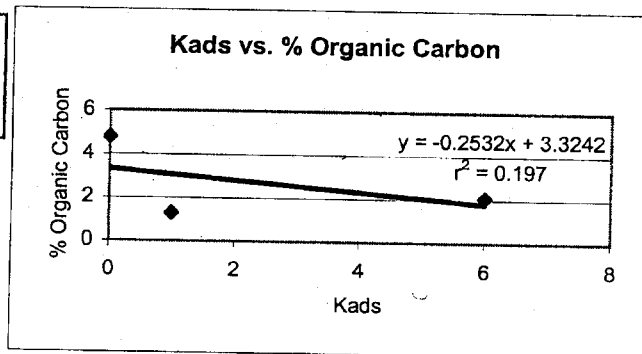
1. The study author failed to analyze soil to confirm adsorption and to provide a complete material balance for the single test concentration for which material balances were determined. Instead, the concentration of residues associated with the soil was determined by calculation. For other test concentrations, material balances were not determined.
2. The study was conducted using three soils. Subdivision N guidelines require that four soils be tested in unaged soil mobility studies. None of the test soils had an organic matter content of $\leq 1\%$.
3. It was not stated whether desorption of the test samples was conducted in the dark.
4. The length of time and conditions during storage prior to analysis were not reported. This does not affect the validity of the study.

V. REFERENCES: References were not cited.

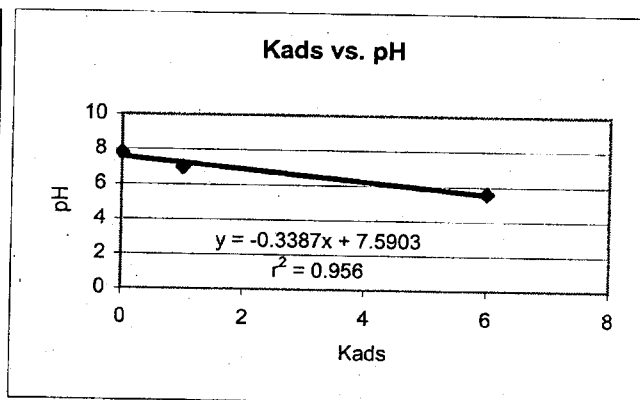
Attachment 1
Excel Workbook

Chemical Name M510F47
 PC Code 128008
 MRID 45405217
 Guideline No. 163-1

Soil	Kads	% Organic Carbon
Silt loam	6	2.1
Sandy loam	0	4.8
Loamy sand	1	1.3



Soil	Kads	pH
Silt loam	6	5.6
Sandy loam	0	7.8
Loamy sand	1	7



Soil	Kads	% Clay
Silt loam	6	16
Sandy loam	0	20
Loamy sand	1	4

