

Reviewed by: Srinivas Gowda, Microbiologist/Chemist, Team 1 S. Gowda, Date 3/13/03

DATA EVALUATION RECORD

STUDY TYPE: Anaerobic Aquatic Metabolism of Didecyl dimethyl ammonium chloride

DP BARCODE: D288350 & D287563

PC CODE: 069149

SUBMISSION CODE: S627084

CASE TYPE: Resubmission

TEST MATERIAL: Didecyl dimethyl ammonium chloride

SYNONYMS: DDAC, Bardac 22 (2250, 2280)

CITATION: "Anaerobic Aquatic Metabolism of ¹⁴C-Didecyl dimethyl ammonium chloride (¹⁴C-DDAC)" U.S. EPA-FIFRA, 40 CFR, Sec. 158.130 Guideline 162-3, by Walter Cranor, Manager, Environmental Fate, ABC Laboratories, Inc., 7200 East ABC Lane, P.O. Box 1097, Columbia, Missouri 65205, ABC Final Report #37007, dated August 6, 1991 (MRID Number 422538-02).

SPONSOR: Lonza Inc.

Data Evaluation Report on the anaerobic biotransformation of ¹⁴C-didecyldimethyl-ammoniumchloride in water/sediment system

PMRA Submission Number:

EPA MRID Number: 422538-02

Data Requirement: PMRA DATA CODE:
EPA DP Barcode:
OECD Data Point:
EPA Guideline: Chemistry Series 162-3/835.4400

Test material:
Common name
chemical name: ¹⁴C-didecyldimethyl-ammoniumchloride
IUPAC:
CAS name:
CAS No.: 7173-51-5
synonyms: ¹⁴C-DDAC

Primary Reviewer:
{EPA/OECD/PMRA}

Signature:

Date:

Secondary Reviewer(s):
{EPA/OECD/PMRA}

Date:

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

Date Evaluation Completed: {dd-mmm-yyyy}

CITATION:

Study Title: "Anaerobic Aquatic Metabolism of
¹⁴C-Didecyldimethylammoniumchloride (¹⁴C-DDAC)"
Year: August 6, 1991
Author: Walter Cranor
Manager, Environmental Fate
Laboratory Name: ABC Laboratories, Inc.
7200 East ABC Lane
P.O. Box 1097
Columbia, MD 65205
Laboratory Report No.: 37007
Sponsor: Lonza, Inc.
17-17 Route 208
Fair Lawn, New Jersey 07410

EXECUTIVE SUMMARY:

The anaerobic biotransformation of ¹⁴C-didecyldimethylammoniumchloride (¹⁴C-DDAC) was studied in a pond water/sediment system (water pH was 8.1; sediment texture was classified as sandy loam, pH was 8.0, organic matter content was 1.6%) from a pond located near Northwood, North Dakota, for 365 days, in a dark environment at a temperature of 25 °C. The sediment was treated with a sufficient amount of ¹⁴C-DDAC to achieve a nominal concentration of 10 ppm. The sediment/water ratio used was 10.0 grams (dry weight) of pond sediment and 20 ml of pond water. The experiment was conducted in accordance with the Environmental Protection Agency, Pesticide Assessment Guidelines, Subdivision N, Section 162-3. The study author stated that the study was conducted under EPA Good Laboratory Practice Standards (40 CFR Part 160), with one exception: the soil characterization data performed at A and L Midwest Laboratories, Inc. cannot be assured to have followed current U.S. EPA Good Laboratory Practice Standards. The registrant waived claims of confidentiality within the scope of FIFRA Section 10 (d)(1)(A), (B), or (C). The test system consisted of a standard tall form 3000 ml resin-pot (the incubation vessel) with traps attached for the collection of CO₂ and volatile organic compounds. Samples were analysed at 0, 1, 3, 7, 14, 31, 61, 92, 123, 182, 273, and 365 days of incubation. The water samples were extracted via mixing with an equal volume of 80:20 (v:v) dimethylformamide:acetic acid and then run through a 0.2-μ filter to remove any suspended solids. The sediment samples were extracted with 30 ml of 80:20 (v:v) dimethylformamide:acetic acid and then shaken for 1 hour, followed by centrifugation for 10 minutes. Quantification and identification of the ¹⁴C-DDAC residues was performed using TLC and/or HPLC.

Material balance averaged 103.1 ± 3.5 % of the applied amount for the year-long study. The test conditions outlined in the study protocol were maintained throughout the study. Extractable [¹⁴C]residues in sediment increased from a mean of 84.87 % at day 0, to a mean of 92.22 % of the applied amount, at study termination. Non-extractable [¹⁴C]residues in sediment decreased from a mean of 12.21 % at day 0, to a mean of 10.39 % of the applied amount, at study termination. At the end of the study, 0.30 % of the applied radioactivity was present as volatile compounds, and this activity was later confirmed to be ¹⁴C-carbon dioxide via precipitation with barium chloride.

The concentration of ¹⁴C-DDAC in water decreased from a mean of 100 % at day 0 to a mean of 68.65 % of the applied amount, at study termination. The concentration of ¹⁴C-DDAC in the sediment increased from a mean of 93.85 % at day 0 to a mean of 97.45 % of the applied amount, at the end of the study period.

The Registrant's calculated half-life of ¹⁴C-DDAC (for the entire system), based on a first-order degradation, was 6,218 days. Versar's calculated half-lives, based on first-order degradations of ¹⁴C-DDAC in aerobic water, sediment and in the entire system were 261; 4,594; and 6,217 days, respectively. No biotransformation of the parent compound was reported.

Results Synopsis:

Test system used: Anerobic aquatic metabolism with ¹⁴C-DDAC on floded pond sediment
Half-life in water: 261 days
Half-life in sediment: 4,594 days (about 12.5 years)
Half-life in the entire system: 6,217 days (about 17 years)
Major transformation products: None Reported
Minor transformation products: None Reported

Study Acceptability: This study is classified acceptable and mostly satisfies the guideline requirements for an anaerobic biotransformation study in a water/sediment system. The deficiencies and points of concern are noted in this study review in Sections III and IV.

I. MATERIALS AND METHODS

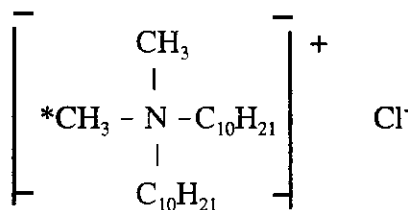
GUIDELINE FOLLOWED: The experiment was conducted in accordance with the Environmental Protection Agency, Pesticide Assessment Guidelines, Subdivision N, Section 162-3.

COMPLIANCE: The study was conducted in compliance with the U.S. EPA GLP Standards (40 CFR Part 160). A signed GLP statement was provided which confirmed compliance stating that one exception existed: the study stated that the soil characterization data performed at A and L Midwest Laboratories, Inc. cannot be assured to have followed current U.S. EPA Good Laboratory Practice Standards.

A. MATERIALS:

1. Test Material: ^{14}C -didecyldimethyl-ammoniumchloride

Chemical Structure:



(* denotes the labeled carbon)

Description: The test substance was a liquid.

Radiochemical purity: The purity stated on the vials containing the test substance label was >99%. A 100 ml stock solution was created using the contents of the vials and Millipore water. The radiochemical purity of the stock solution (990 $\mu\text{g}/\text{ml}$) was determined to be 90.0% by a triplicate TLC analysis.

Lot/Batch No.: 7499-E

Specific activity: The specific activity stated on the vial label was 9.01 mCi/mmol.

Locations of the radio label: The radio label was located on the methyl carbon.

Storage conditions of test chemicals: The primary stock solution was stored in the refrigerator when not in use.

Physico-chemical properties of ¹⁴C-DDAC: Water solubility, vapor pressure/volatility, UV absorption, pK_a, K_{ow}/log K_{ow}, and stability of compound at room temperature were not provided in the Study Report.

2. Water-Sediment Collection, Storage and Properties

Table 1: Description of Water Collection and Storage.

Description	Details
Geographic location	Northwood, North Dakota
Pesticide use history at the collection site	Pesticide use history was not provided.
Collection procedures for water and sediment:	Collection procedures were not discussed. The study states that the ABC laboratories received a 5-gallon Nalgene carboy filled to capacity with water that also contained 6 inches of pond sediment.
Sampling depth for water and sediment:	Sampling depth for water and sediment was not provided.
Storage conditions:	The water and sediment sample were both shipped to the laboratory in the same 5-gallon Nalgene carboy. At the laboratory, the water was decanted into a 5-gallon glass jar being careful not to disturb the sediment and the jar was then covered with aluminum foil. The sediment was wet-sieved, placed in a dishpan, covered with an inch of the pond water and covered with aluminum foil. Both the glass jar and the dishpan containing the sediment were stored in an environmental chamber at 25 °C under dark conditions until used in the study.
Storage length:	Sample storage length was not provided.
Preparation of water and sediment samples	Water samples were filtered through a 0.2- μ filter prior to HPLC analysis to remove suspended solids. Sediment samples were wet-sieved through a 2-mm screen.

Table 2: Properties of the Water.

Property	Details
Temperature (°C)	24.7°C
pH	8.1
Redox potential (mv)	Not provided.
Oxygen concentration (mg/L)	11.2
Dissolved organic carbon (%)	Not provided.
Hardness (CaCO ₃)	9.68 gr/gallon
Electrical conductivity	0.68 mmhos/cm
Biomass	Not provided.

Table 3: Properties of the Sediment.

Property	Details	
Textural classification	sandy loam	
% sand	62	
% silt	22	
% clay	16	
pH	8	
Organic matter (%)	1.6	
CEC (meq/100 g)	16.3	
Redox potential (mv)	Not provided.	
Bulk density (g/cm ³)	1.29	
Biomass (colony forming units per gram)	Initial	Final
	1.8x10 ⁵	7.6x10 ⁴

B. EXPERIMENTAL DESIGN:

- 1. Preliminary experiments:** A 14-day preliminary study was conducted prior to initiation of the definitive study. The purpose of this experiment was to evaluate the proposed experimental design and to determine an approximate half-life of ¹⁴C-DDAC under study conditions.

No evidence was observed that indicated degradation of ¹⁴C-DDAC during the preliminary experiment. This indicated that ¹⁴C-DDAC was stable in the presence of anaerobic bacteria and that the design of the study was appropriate.

2. Experimental conditions:

Table 4: Study Design.

Parameter		Details	
Duration of the test		365 days	
Water:		Water utilized in this study was filtered through a 0.2- μ filter.	
Amount of sediment and water/ treatment		10.0 g (dry weight) of sediment to 20 ml of water were placed in each sample tube.	
Application rates		A 137- μ l of the purified ¹⁴ C-DDAC dosing solution at 732 μ g/ml was added to 35 sample tubes.	
Control conditions		Controls were exposed to the same conditions as treated samples except that they were not dosed with the test substance.	
Number of Replications	Controls	35	
	Treatments	35	
Details of traps for CO ₂ and organic volatile, if any		The effluent was passed through a 250 ml ethylene glycol trapping solution, then a 250 ml 1.0 N sulfuric acid trapping solution, and finally, two 250 ml 1.0 N KOH trapping solutions.	
If no traps were used, is the system closed		N/A	
Identity and concentration of co-solvent		No co-solvent was identified.	
Any indication of the test material adsorbing to the walls of the test apparatus		Not specified.	
Microbial biomass/microbial population of the control		Initial	Final
	water: sediment:	Not provided 1.9x10 ⁵	Not provided 3.2x10 ⁵
Microbial biomass/microbial population of the treated		Initial	Final

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Parameter		Details		
		water:	Not provided	Not provided
		sediment:	1.8x10 ⁵	7.6x10 ⁴
Experimental conditions:	Temperature (°C):	25°C		
	Continuous darkness	Yes		

3. **Anaerobic conditions:** A nitrogen supply was used to flush ¹⁴C-volatiles from the test system.

4. **Supplementary experiments:** No supplementary experiments were performed.

5. **Sampling:** The information on sampling is provided in Table 5.

Table 5: Sampling Details.

Criteria	Details
Sampling intervals	¹⁴ C-residues in the sediment and water and volatile metabolite evolution sampling occurred on day-0, day-1, day-3, day-7, day-14, day-31, day-61, day-92, day-123, day-182, day-273, and day-365. Sampling for volatile metabolite evolution also occurred on day-151, day-212, day-243, day-304, and day-335.
Sampling method	Samples consisted of 10.0 g (dry weight) of sediment to 20 ml of water were placed in each sample tube. After the tubes were made, they were stored under dark conditions at 25 °C until utilized.
Method of collection of CO ₂ and organic volatile compounds	CO ₂ and volatile compounds were trapped using ethylene glycol solution, sulfuric acid solution, and KOH trapping solution
Sampling intervals/times for: -sterility check, if sterile controls are used: -redox potential:	 Sterile controls were not used in this study. Not provided.
Sample storage before analysis	Not provided.

C. ANALYTICAL METHODS: Briefly describe the methodology for:

1. Separation of the sediment and water:

The water and sediment sample were both shipped to the laboratory in the same 5-gallon Nalgene carboy. At the laboratory, the water was decanted into a 5-gallon glass jar being careful not to disturb the sediment and the jar was then covered with aluminum foil. The sediment was wet-sieved, placed in a dishpan, covered with an inch of the pond water, and covered with aluminum foil.

2. Extraction/clean up/concentration methods for water and sediment samples:

Sample tubes containing flooded sediment samples were removed from the metabolism vessel and centrifuged for approximately 10 minutes. After centrifuging the samples, the water was decanted and triplicate 1.0 ml aliquots were taken for LSC analysis. Prior to analysis of the water samples for ^{14}C -DDAC, samples were mixed with equal volumes of 80:20 (v:v) dimethylformamide:acetic acid. This mixture was then run through a $0.2\text{-}\mu$ filter. This step was performed to remove any suspended solids in the mixture prior to HPLC analysis.

Sediment extraction was accomplished by adding a 30 ml portion of 80:20 (v:v) dimethylformamide:acetic acid to the sediment. This mixture was then shaken for 1 hour followed by centrifuging for 10 minutes. After centrifuging, the liquid was decanted into a 100 ml graduated mixing cylinder. This procedure was repeated twice to give a composite extract. The composite extract was adjusted to a final volume of 100 ml with the addition of extraction solvent. Three 1.0 ml aliquots of the composite extract were taken for LSC analysis. The sample extract was transferred into an amber bottle and stored in a freezer until subsequent analysis.

3. Total ^{14}C measurement:

Total ^{14}C is taken to be the summation of total volatile ^{14}C -activity, total extractable ^{14}C -activity, total ^{14}C -aqueous residues, and total ^{14}C -non-extractable residues. The analysis methods for total extractable, total aqueous, and total non-extractable residues is provided above. For the analysis of volatiles, 1 ml aliquots of the trapping solutions were analyzed by duplicate LSC to determine the amount of ^{14}C -radioactivity. The presence of $^{14}\text{CO}_2$ was confirmed in composite mixtures of the first KOH trapping solutions using barium chloride which produces the insoluble precipitant barium carbonate. The composites were analyzed in triplicate 1.0 ml aliquots by LSC to determine the amount of ^{14}C -radioactivity. Then, a portion of barium chloride was added to a 10 ml aliquot of each composite. The composites underwent vortex mixing and centrifuging, triplicate 1.0 ml aliquots were taken of each supernatant for LSC analysis and the reductions of soluble ^{14}C -activity provided measures of $^{14}\text{CO}_2$ present.

Measurements of radioactivity were made using a benchtop microprocessor-controlled spectrometer (Beckman Model 3801 Liquid Scintillation Counting System). This system incorporates a daily self-calibration feature and successful calibration was reported throughout the study. Liquid samples were pipetted into scintillation vials where they received aliquots of scintillation fluid (Beckman Ready Gel MP[®]). All samples were counted for 5 minutes or to a 2 sigma (95%) confidence level using a single label dpm data calculation program.

4. Determination of non-extractable residues:

A post-extracted sediment sample from the 12-month sample point was used to analyze non-extractable residue. This sample (9.994 g) was previously analyzed to contain 1.182 ppm ^{14}C -DDAC equivalents. The sample was transferred into a 250 ml flask along with 50 ml of 80:20 (v:v) DMF:acetic acid and the mixture was vigorously boiled under reflux for three hours. A 10 g sample of control study sediment was spiked with ^{14}C -DDAC and processed concurrently to ensure no loss due to the extraction process. After the extraction mixtures cooled to room temperature, the samples were transferred to culture tubes and centrifuged and the extracts were decanted. The sediments were rinsed with two 20 ml aliquots of extraction solvent and the combined extract and solvent rinses were adjusted to a final volume of 100 ml and analyzed by LSC analysis. The sediment samples were allowed to air dry and were analyzed by combustion analysis to determine the levels of ^{14}C -activity remaining. Following storage for several days, the extract was noted to contain suspended solids and was passed through a 0.45- μ filter to remove this material.

4. Derivatization method, if used:

Not utilized in this experiment.

5. Identification and quantification of parent compound:

TLC and HPLC were both used for the quantification and identification of ^{14}C -DDAC during the anaerobic aquatic metabolism study and in addition, HPLC was used as a confirmational analytical method for levels of ^{14}C -DDAC quantified by TLC.

The TLC system was developed at ABC Laboratories. Merck, silica gel TLC plates, 60/Kieselguhr F-254, were employed for all TLC work. All TLC analyses were accomplished with overspotting of ^{14}C -DDAC non-radiolabeled analytical standard. TLC zones corresponding to ^{14}C -DDAC were located as a dark spot by irradiation with longwave ultraviolet light by scanning for ^{14}C -radioactivity using a Radiomatic[®] RTLC Multi-Scanner and by formation of autoradiograms. In all cases, the parent compound, didecyldimethylammoniumchloride, was the only unique TLC zone observed.

For HPLC, a Shimadzu LC-6A HPLC pump fitted with a Rheodyne 7- μ HPLC column was used for the analysis of all study water samples and sediment extract samples taken from the 6-month, 9-month and 12-month samples points. A flow rate of 1.5 ml/minute was used with a gradient system using methanol and 0.025 M KH_2PO_4 buffered at pH 4 with 0.005 M tetrabutylammonium dihydrogenphosphate (TBAP).

5. Identification and quantification of transformation products:

Transformation products were not identified and quantified.

6. Detection limits (LOD, LOQ) for the parent compound:

The limits of detection are directly related to the sensitivity of the counting. Twice the background is taken as the limit of detection. The limit of detection reported in the Study Report for bound residues, extractable residues, volatile residues and CO_2 , and HPLC analysis was $\leq 0.001 \mu\text{g}$.

7. Detection limits (LOD, LOQ) for the transformation products:

Transformation products were not identified and quantified.

II. RESULTS AND DISCUSSION:

A. TEST CONDITIONS:

The test system employed in this study was contained within an environmental control chamber maintained with dark conditions and regulated at a temperature 25 °C for the duration of the study. The initial microbial activity of the sediment was measured at 1.8×10^5 colony forming units per gram and the final microbial activity was measured to be 7.6×10^4 colony forming units per gram. The study stated that ¹⁴C-DDAC is stable to microbial degradation.

B. MATERIAL BALANCE:

Total recovery of radiolabeled material ranged from 95.6 to 110.8 % of the applied amount. Mean overall recovery was 103.1 ± 3.5 % of the applied amount (mean \pm std). Table 6 presents biotransformation as a percentage of applied radioactivity for extractable residues, non-extractable residues, organics, and total ¹⁴C recovery.

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Table 6: Biotransformation of ¹⁴C-DDAC, Expressed as Percentage of Applied Radioactivity (9.807 µg ¹⁴C-DDAC equivalents/g of soil) in Water-sediment System Under Anaerobic Conditions.

Sampling Day (days)	0	1	3	7	14	21	28	35	42	49	56	63	70	77	84	91	98	105	112	119	126	133	140	147	154	161	168			
Extractable residues	88.41	8.133	85.24	100.7	99.12	93.61	93.39	91.93	74.19	92.58	89.42	94.45	94.13	84.64	92.97	91.46														
Non-extractable residues	10.32	14.1	14.54	4.88	9.95	9.22	9.31	10.91	28.12	9.03	13.52	6.09	7.02	8.64	8.73	12.05														
Aqueous Residues	4.06	1.78	3.32	1.36	1.74	2.22	1.26	1	0.34	0.64	0.79	0.45	0.79	2.04	1.33	1.4														
Volatiles ^{cd}																														
Et	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.07	0.08	0.00														
H	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00														
K ₁	0.00	0.00	0.01	0.01	0.02	0.03	0.07	0.07	0.07	0.08	0.08	0.10	0.15	0.22	0.22	0.22														
K ₂	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00														
Total	0.00	0.00	0.01	0.01	0.02	0.03	0.07	0.07	0.07	0.08	0.08	0.10	0.15	0.29	0.30	0.30														
Total ¹⁴ C-Accountability ^b	102.8	97.22	103.1	107	110.8	105.1	104	103.9	102.7	102.3	103.8	101.1	102.1	95.61	103.3	105.2														

^a Initial Measured Dose = 9.807 µg ¹⁴C-DDAC equivalents/g of sediment

^b Mean ¹⁴C-accountability through study day 365 = 103.13 ± 3.48%

^c Note: Individual values for volatiles may be less than minimum quantifiable limit, whereas, summation of all observed values are reported.

^d Volatiles collected at days between sample points are included with the following analysis days.

- Et = Ethylene Glycol Trapping Solution
- H = Sulfuric Acid Trapping Solution
- K1 = First KOH Trapping Solution
- K2 = Second KOH Trapping Solution

C. TRANSFORMATION OF PARENT COMPOUND: The concentration of the parent compound in water decreased from a mean of 100 % at day 0 to a mean of 68.65 % of the applied amount at study termination. The concentration of the parent compound in the sediment increased from a mean of 93.85 % at day 0 to 97.45 % of the applied amount at the end of the study period.

1. Half-life:

The Registrant's calculated half-life of ¹⁴C-DDAC, based on a first-order degradation, was 6,218 days. Versar also performed calculations based on the information provided in the report. Using the ug/g values provided for extractable ¹⁴C residues, ¹⁴C-DDAC, and the applied dose, Versar calculated the percent recovered as ¹⁴C-DDAC and the percent of dose recovered. Using these numbers, Versar calculated a half-life of 6,217 days (see Table 7).

Table 7: Versar Calculated ¹⁴C-DDAC Half-lives

System	First order		
	Half-life (Days)	Regression equation	R ²
water	261	$y = 0.00266x + 0.1945$	0.074
sediment	4594	$y = 0.000151x + 4.4341$	0.0788
entire system	6217	$y = 0.000111x + 4.4545$	0.0474

2. Transformation Products

Transformation products were not reported in the Study Report.

3. Extractable and Non-Extractable Residues:

Extractable [¹⁴C]residues in sediment increased from a mean of 84.87 % at day 0 to a mean of 92.22 % of the applied amount at study termination. Non-extractable [¹⁴C]residues in sediment decreased from a mean of 12.21 % at day 0 to a mean of 10.39 % of the applied at the end of incubation period.

4. Volatilization:

At the end of the study, 0.30 % of the applied radioactivity was present as volatile compounds. This ¹⁴C-activity was later confirmed to be ¹⁴C-carbon dioxide via precipitation with barium chloride.

5. Transformation Pathway:

The biotransformation pathway was not provided in the Study Report. The Registrant reported that transformation products were not present in any significant amounts. Transformation products were not identified in the Study Report.

D. SUPPLEMENTARY EXPERIMENT RESULTS:

No supplementary experiments were conducted.

III. STUDY DEFICIENCIES:

The following study deficiencies were noted:

1. Half -life estimates were only calculated for the entire system in the Study Report and not for water and sediment separately.
2. The treatment rate were not provided. The guidelines state that the treatment rate should be at the highest proposed field use rate; however, the field use rate was not provided and could not be obtained by Versar. It is also unclear as to how the dosing solution was applied.
3. The Study Report did not provide data to show that anaerobic conditions were maintained throughout the experiment (e.g., redox potential).

IV. REVIEWER'S COMMENTS:

The following points of concern were noted:

- On Table IV, the Registrant lists the "% Recovered" for Day 0-2 as 87.7 %, however, when Versar performed the calculations, using the "dpm analyzed" (2759) and the "dpm Recovered" (2549) provided in the Study Report, the "% Recovered" was found to be 92.3 %.
- For the quality control samples (Table V), it was unclear how the Registrant calculated the "% Recovered as ¹⁴C-DDAC". Versar was unable to verify those values presented.
- The raw data were not included with the Study Report and therefore, the values presented could not be verified.

V. REFERENCES:

American Public Health Association. "Standard Methods for Examination of Water and Wastewater", 16 ed., 1985.