### **FINAL REPORT**

#### Crude MCHM

HAEL No.: 97-0216

EAN: 972790

PM No.: 18717-00

CAS: Not Available

## **BIOCHEMICAL OXYGEN DEMAND DETERMINATION**

## **AUTHOR**

M. Steinbugler

## TEST GUIDELINE

"Environmental Laboratory Certification Manual", New York State Department of Health, January 1987.

"OECD Principles of Good Laboratory Practice", [C(81)30(Final)], Annex 2.

#### **TESTING FACILITY**

Environmental Analytical Services
Chemicals Quality Services Division
Manufacturing Quality Assurance Organization
Eastman Kodak Company
Rochester, New York 14652-6276
USA

## STUDY SPONSOR

Eastman Chemical Company

Report No. BOD-00774

STUDY COMPLETION DATE

leptember 30, 1997

## ANALYTICAL QUALITY ASSURANCE INSPECTION STATEMENT (CFR 58.35(B)(7) 792.35(B)(7) 160.35(B)(7))

STUDY: 97-0216 STUDY DIRECTOR: BISHOPP, M.

ANALYTICAL DIRECTOR:

KAN: 972790 CQS JOB NUMBER:

STUDY TYPE: BIOCHEMICAL OXYGEN DEMAND DETERMINATION

\_ Syptember 26, 1997

THE FOLLOWING PHASES OF THIS STUDY WERE INSPECTED BY ONE OR MORE PERSONS OF THE QUALITY ASSURANCE UNIT ON THE DATES LISTED BELOW. WRITTEN STATUS REPORTS WERE SUBMITTED TO THE STUDY DIRECTOR AND APPROPRIATE MANAGEMENT.

INSPECT DATES	REQUEST NUMBER	PHASE (S) INSPECTED	STATUS REPORT DATES
08/06/97		PROTOCOL SUBMISSION ECHEM	08/06/97
09/26/97		TEST REPORT INSPECTION ECHEM	09/26/97

## GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This study was conducted according to:

United States Food and Drug Administration, Good Laboratory Practice for Nonclinical Laboratory Studies, 21 CFR Part 58;

United States Environmental Protection Agency, Toxic Substances Control Act, Good Laboratory Practice Standards, 40 CFR Part 792;

Annex 2, Organization for Economic Cooperation and Development, Guidelines for the Testing of Chemicals [C(81)30(Final)].

Mary Lee Bishopp

Mary Lee Bishopp

Study Director

Month/Day/Year

Kun R. miles October 9, 1997

Karen R. Miller Month/Day/Year

Eastman Contact Representative

# SIGNATURE PAGE

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## Crude MCHM

HAEL No.: 97-0216 EAN: 972790

PM No.: 18717-00 CAS: Not Available

#### ABSTRACT

The biochemical oxygen demand (BOD) of the test substance was measured in compliance with "OECD Principles of Good Laboratory Practice", [C(81) 30(Final)], Annex 2. The method of measurement used was Method C.5., "Degradation, Biochemical Oxygen Demand", Official Journal of the European Communities, No. L 251/212, 19.9.84. The results of the 5-day BOD for the test substance at 0.0020 % and 0.0030 % were 0.074 and 0.066 grams BOD / gram of test substance, respectively. The average result for the 5-day BOD was 0.070 grams BOD / gram of test substance. The test substance exhibited inhibitory effects in the 5-day BOD test. An inhibitory BOD result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The result of the 20-day BOD for the test substance at 0.0030 % was 1.30 grams BOD / gram of test substance. The test substance exhibited inhibitory effects in the 20-day BOD test, therefore only the most dilute solution was used to calculate the final 20-day BOD. An inhibitory BOD result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The result of the BOD5/COD was 0.028.

The density was determined by weighing the test substance contained in a 1 milliliter volumetric flask on a Mettler AT261 balance. The density was done in triplicate. The mean of the three density results was found to be 0.91 grams per milliliter.

## STUDY AND TEST SUBSTANCE INFORMATION

#### **Testing Facility**

Environmental Analytical Services Chemicals Quality Services Division Eastman Kodak Company Rochester, New York 14652-6276 USA

#### **Sponsor**

Eastman Chemical Company P.O. Box 431 Kingsport, Tennessee 37662-5280 U.S.A.

## **Sponsor Representative**

Karen Miller P.O. Box 431 Kingsport, Tennessee 37662-5280 U.S.A.

### **Study Dates**

Study Start Date: August 6, 1997 Experimental Start Date: August 6, 1997 Experimental Completion Date: August 26, 1997

## **Project Participants**

Study Director Mary Lee Bishopp
Principal Investigators Margaret Brazwell Tammy Kleiber
Joseph Donnelly Tracey Sanford
Deltris Dunn Mary Steinbugler

Report Author Mary Steinbugler

# Test Substance Characterization

Name: Crude MCHM

EAN: 972790

HAEL No.: 97-0216 PM#: 18717-00

### **Test Substance Storage**

The test substance was stored at room temperature in a locked cabinet before and after each analysis was performed.

#### **PURPOSE**

The purpose of this study was to determine the biodegradability of the test substance under standard conditions of the test.

#### MATERIALS AND METHODS

The method and material can be found in "Determination of Biochemical Oxygen Demand of Solid and Liquid Organic Chemicals". Method No. KPCQ-A-EA-G-M-3-1, Eastman Kodak Company.

#### **Calculations**

The 5-day BOD was calculated by subtracting the final D.O. (dissolved oxygen) reading and the 5-day SDW (seedwater) drop from the initial D.O. This is then multiplied by 100. The resulting answer is then divided by the percent concentration of the sample being analyzed and 1,000,000. The result is a value in units of grams of BOD per gram of test substance for a 5-day incubation.

The 20-day BOD was calculated by subtracting the final D.O. (dissolved oxygen) reading and the 20-day SDW (seedwater) drop from the initial D.O. This then multiplied by 100. The resulting answer is then divided by the percent concentration of the sample being analyzed and 1,000,000. The result is a value in units of grams of BOD per gram of test substance for a 20-day incubation.

The BOD/COD ratio was calculated by dividing the average 5-day BOD value in units of grams per gram by the mean of the COD results in units of grams per gram. The result is a value without units.

The density was calculated by dividing the weight of the test substance by the volume. The result is a value of grams per milliliter.

## **Protocol And Standard Operating Procedure Deviations**

The 5-day BOD result was calculated on dilutions that exhibited a dissolved oxygen drop less than the required drop of two milligrams per liter. This was necessary due to the inhibitory effects of this test substance. An inhibitory BOD result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The dissolved oxygen drop of the dilution water and the seeded dilution water was outside of the method requirements. The reference sample was within the accuracy requirements for the method. Therefore, this will not adversely affect the result of the test.

## **Data Storage And Record Retention**

All original protocols, raw data, and reports will be stored for at least ten years by the Chemicals Quality Services Division, Building 320 of the Eastman Kodak Company, Kodak Park, Rochester, New York 14652-6276.

#### **RESULTS**

The results of the 5-day BOD analysis are as follows:

Replicate 1 = 0.074 grams of BOD/ gram of test substance (at 0.0020% concentration). Replicate 2 = 0.066 grams of BOD/ gram of test substance (at 0.0030% concentration).

mean = 0.070 grams of BOD/ gram of test substance.

The test substance exhibited inhibitory effects in the 5-day BOD test and should be interpreted as an estimated result due to decreased activity of the microorganisms.

The results of the 20-day BOD analysis are as follows:

Replicate 1 = 1.30 grams of BOD/ gram of test substance (at 0.0030% concentration).

The test substance exhibited inhibitory effects in the 20-day BOD test, therefore only the most dilute solution was used to calculate the final 20-day BOD and the result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The result of BOD5/COD ratio is 0.028.

#### **CALCULATIONS**

5-day BOD

BOD grams/gram = 
$$\frac{(DO_1 - DO_2 - C) \quad (100)}{(\% concentration)(density)(1,000,000)}$$

Where:

C = DO drop of seeded dilution water after 5 days.

 $DO_1$  = initial DO of sample solution, mg/L.  $DO_2$  = Total DO drop after 5 days, mg/L.

% concentration = concentration of the incubated sample.

density = density of the incubated sample

Example Calculation:

Replicate One:

BOD grams / gram = 
$$\frac{(7.60-6.15-0.10)(100)}{(0.0020)(0.91)(1,000,000)} = 0.074$$

20-Day BOD

BOD grams/gram = 
$$\frac{(DO_{Total\ 20} - C_{20})(100)}{(\% conc.)(density)(1,000,000)}$$

Where:

DO<sub>Total20</sub> = Total DO drop over 20days. C<sub>20</sub> = Average seed water DO drop after 20 days. % conc. = concentration of incubated sample. 1,000,000 = conversion factor mg/L to g/g. density = density of the incubated sample Example Calculation:

Replicate One:

BOD grams/gram = 
$$\frac{(38.85 - 3.23)(100)}{(0.0030)(0.91)(1,000,000)} = 1.30$$

BOD/COD Ratio

BOD5/COD Ratio = 
$$\frac{Average\ BOD5day concentration}{Average\ COD\ concentration} = \frac{0.070\ grams/\ gram}{2.54\ grams/\ gram} = 0.028$$

**Density** 

Density grams/milliliter = 
$$\frac{\text{grams test substance}}{\text{milliliters test substance}}$$

Example Calculation:

Replicate One:

Density grams/milliliter = 
$$\frac{0.9084}{1}$$
 = 0.9084

#### **DISCUSSION**

The results of the 5-day BOD for the test substance at 0.0020 % and 0.0030 % were 0.074 and 0.066 grams BOD / gram of test substance, respectively. The average result for the 5-day BOD was 0.070 grams BOD / gram of test substance. The test substance exhibited inhibitory effects in the 5-day BOD test. An inhibitory BOD result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The result of the 20-day BOD for the test substance at 0.0030 % was 1.30 grams BOD / gram of test substance. The test substance exhibited inhibitory effects in the 20-day BOD test, therefore only the most dilute solution was used to calculate the final 20-day BOD and the result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The result of the BOD5/COD was 0.028.

#### **CONCLUSION**

The biochemical oxygen demand (BOD) of the test substance was measured in compliance with "OECD Principles of Good Laboratory Practice", [C(81) 30(Final)], Annex 2. The method of measurement used was Method C.5., "Degradation, Biochemical Oxygen Demand", Official Journal of the European Communities, No. L 251/212, 19.9.84. The mean of the two 5-day BOD results was found to be 0.070 grams per gram of the test substance. The 20-day BOD result was found to be 1.30 grams per gram of test substance. The test substance exhibited inhibitory effects in both the 5-day and 20-day BOD tests. An inhibitory BOD result should be interpreted as an estimated result due to decreased activity of the microorganisms.

The BOD5/COD ratio was found to be 0.028.

#### REFERENCES

- 1. "Biochemical Oxygen Demand", Method 405.1, U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979.
- 2. "Degradation, Biochemical Oxygen Demand", Method C.5., Official Journal of the European Communities, No. L 251/212, 19.9.84.

#### FIGURE I

## 5-day BOD Worksheet

#### **BIOCHEMICAL OXYGEN DEMAND**

Chemical HAEL #: 97-0216

EAN: 972 790

Concentration of Solution: | ML/1L

pH prior to adjustment:

5 - DAY - Standards , DW.& SDW:

Date Setup: 8 -6 -97

Date Read: 8-11-97

20-DAY - SDW: Date Setup: 8-6-97
Date Read: 8-26-97

	1.50%	1.50%	2.00%	2.00%	
Bottle#	64	337	59	339	
Initial DO	7.45	7.70	7.65	7.65	1
Final DO	3.90	4,45	3,45	3.50	
SDW Drop	0.0	0.10	040	O(O	1
Adj. Drop	3.10	3.15	4,10	4.05	
BOD. mg/L	230,0	210.12	205.0	202.5	Averaç

e= 211.9

20-Day 5-Day DW SDW SDW 7.55 Final DO 040 **Average Drop** 

5-day BOD Samples

Date Setup: 3-6-97

Date Read: 9-11-97

Bottle#
% Conc.
mis added
Initial DO
Final DO
5day SDW Drop
Adjusted Drop
BODyg/g pr g/ml
DON'S AND ALCOHOL

250	501	96	59	4	70	24	4%	61	205	19	369
0.00033	0.00050	0.0010	0.0020	0,0030	0.0050	0.010	0.020	0.030	0.060	0.090	0.10
1.0	1.5		6.1	9.1	15.2	30.0	61.0	91.0	182	273	Full Btl.
760	7.50	7.55	7.60		7,55	7,60	7.55	7,55	7.65	7.70	7.70
6.90	6.45	6,50			145.00		3.40	3,30	5.5/15	6.45	6.70
0.10	0.10	040	0,10	0,10	0,10	0,10	0.10	0.10	0.10	0.10	0.10
0.50	0.95	0.90	1.35	1.80	2.45	3.85	4.05	4.15	2.00	کایا	0.90
0.17	011-	- 2017	0.02	(ó.00G)	0.054	0,042	0.022				

ANALYST(S) / DATE(S) : 8-6-97

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# FIGURE II

# 20-day BOD Worksheet

# 20 - Day Biochemical Oxygen Demand

Bottle # # Date Set-up 9-6-97 pH Prior to setup	HAEL# 97-0216 KANGAN) 972790 Concentration of Stock Sample	(Circle one)		
Initial DO 5.70 Final DO 2.95 8.05 DO Drop 2.75 3.95 8.05 Prev.Drop 7.45 4.10 3.55 8.10 Tot. Drop 4.20 4.50 5.75 Day 7.12/476 8.30 8.30 2.35 13 7 12.80 12.80	2.42 2.12 8.92 . 2.70 8.90 2.42 30.	Bottle Init. E Final DO D Avera	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	
previous drop  Should be 190  Therefore 0.45 is added to 38.40 for a total drop  Percent Concentration: 0.0030  Analyst: Taning Klester 11.5  Date: (8-12-97, 8/13-97, 8-14-97, 8-15-9)  June Dunch Raymon 8-15-9  June Dunch 18/16/97 &  18 8/16/97 &  18 8/16/97 &	10 20.65 20.65 2.30 17 11 23 30 2330 07 38.85 12 25.60 19 15 10 20.65 20.65 2.30 17 11 23 30 2330 18 12 25.60 19 15	5:75   5:75   2:50 8:6 25:60 3:25 5;	5.85 15 5.45 9.25 20 31.00 4.60 16 32.40 3.65 23 17 32.40 24 18 36.05	8.35 6.00 2.35 36.05 78,40 26.20 3,23 1.29 44
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BOD-00744

## FIGURE III

# 20-day BOD Worksheet

# 20 - Day Biochemical Oxygen Demand

Bottle # 72- Date Set-up 8-6-97 pH Prior to setup	HAEL# 97-02/6 KANEAN 978.790 (Circle one) Concentration of Stock Sample /ml/L	
Initial DO	140 D/ 4.95 8.20	20- Day Seed Water  Bottle # 5 60  Init. DO 7.65 7.65  Final DO 4.15 4.60  DO Drop 3.40 3.05  Average 3.23
13 7 15,40 15,40 14 8 17,90 15 8	1190 3.50 5/0 0-4	7.90 5.40 5 2.50 \ 8.15
Percent Concentration: 0.005  Analyst: Tammy Kleiter, 4.5  Date: (8/297,8/397,8-14-97,8-1  Market Praywell 8-15-97  Journ Danels 9/14/97  18 8/24/97	1) 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	15 36.10 4.00 8.00  22 16 36.10 3.90  23 17 40.10 4.20  24 18 40.10  25 19 44.30  26 20  20-Day Seed Water Drop 3.23  20-Day BOD @gor g/mL) 0.90

# FIGURE IV

# 20-day BOD Worksheet

# 20 - Day Biochemical Oxygen Demand

Bottle # 24  Date Set-up 3-6-97  pH Prior to setup	HAEL# 97-02/6  KAN(EAN) 972 790 (Circle Concentration of Stock Sample 1.0 ~	one)	,
Tot. Drop 7,50 7,50 5,00 5.  Day 9/2/97 6 13,00 13.00 2.	65 4.75 8120	Bottle # Init. DO Final DO DO Drop Average	20- Day Seed Water  5
13 7 18.00 18 H 8 20	9 23.80 2380 2.70 5.80 5.80 16 10 27.30 27.30 2.25 / 17 11 30.00 30.00 / 15 12 32.25 32/25 19 13 /	5780 255 7.80	8.25
Percent Concentration: 0. C  Analyst: Tanimy, Klecker, M  Date: (8-12-97 1-13-97 8-14-97)  Maynet fragura 8-15-9  Jrept Donally 8/14  Bb 8/24/97	710 . Steinbigler 8-26 97 .8-6-41 9-2147,6-22-97) 7	21 15 37 65 22 16 22 20-Day	3.65 1.50 37.65 3.70 17 41.30 3.90 24 18 41.30 25 19 45.10 26 20 28 Seed Water Drop 3.25 BOD (g/g or g/mL) 0.46

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By M. Menluy a Date 9-26-97

## FIGURE V

# 20-day BOD Worksheet

## 20 - Day Biochemical Oxygen Demand

Bottle # 45	HAEL# 97-02/6		
Date Set-up 8-6-97		(Circle one)	
pH Prior to setup	Concentration of Stock Sample //O	nl/t	•
			20- Day Seed Water
Initial DO 3,40		Bottle	
Final DO 1, 85 8.05		Init. D	
DO Drop 1.55 5.75 7.80	<b>,</b> Q	Final I	
Prev.Drop 4,15 2.36 0,20 7.50	1 2	, bo b	
Tot. Drop 5.70 5.70 7.60 4.15	7.80	Avera	ge 3.23
Day 3/12/97 6 8 .00 8,00 3.35	4.50 8.10	4	<del></del>
13 7 15.60 15.60	3 30 4.20 8.45	1 8.21.97 8/20/17	
N4 8 18.95	18.95 3.90 5.55 7.95		•
15 9	22.25 22.25 2.90 5.35	5.35	
	16 10 26 .15 26.15 2.60	5.35	<del>-</del> 7
	17 1129.05 29.05	1.70 7.6	<del></del>
	13 12 31.65	31.65 3.65 5.3	
,	19 13	1	وتصافات فالمستبد والمستبد والمستبد والمستبد والمستبد والمستبد
Percent Concentration: 0,02	20 L	$\frac{20}{2}$ $\frac{14(33.30)}{21}$ $\frac{35}{15}$	
Analyst Tan my Klo Klo N.	He above 8-26-67	22	16 37,65 4,40
Date: (8-12 47 1-18-47, 8-18-57	8-21-97 8-22-97)	l	23 17 41.40 3.75
majoret Brazione 8-15-97			24 18 41.40
Percent Concentration: 0,02  Analyst: Taniny Klaber, M.  Date: (8-12471-18-47,8-14-97, 8-15-57)  Marguet Baywell 8-15-97  Joseph Donald P/K/97		4	25 19 45.15
Just the state of	2/2/2		عد 20
8/197	+ 8/11/87		20-Day Seed Water Drop 3.23
Joseph Denilly P/1/97 	•		20-Day BOD (g/g or g/ml.) 0.23

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# FIGURE VI

# 20-day BOD Worksheet

# 20 - Day Biochemical Oxygen Demand

Bottle #	HAEL# 97-0216 KANEAN 972 790 Concentration of Stock Sample	(Circle one)		
Initial DO 3,30 Final DO 1.75 8.05 DO Drop 1.55 6.90 6.90 7.70 Prev.Drop 4.25 1.15 4.90 6.90 Tot. Drop 5,80 5.80 2.00 6.50 Day 4/2/16 6.95 6/95 1.20	7 a 2	ir F	20- Da Sottle # 5 nit. DO 7.65 inal DO 4.26 DO Drop 3.46 werage	460
13 787.95 65.75. 4 8 11.15	1.85 6.20 6.20 1 11.15 1.90 3.90 7.85 13.00 13.00 2.30 5.90 16 10 14.90 14.90 185 17 11 17.20 17.20	5.90 < 8.15	7.60 P.C. 25 62-97	
Percent Concentration: 0,030  Analyst: Tampus Kabe M. G	18 12 19.15  19 1:  5 triphyels 8-26-97	2d 14 22 9 0 6 2d 14 22 9 0 6	6.25 6.25 135 5.30 2290 0.95 2325 2325 22 16 24.20	9,25 5,20
Analyst: Tamony Karber, M. 63 Date: 6-12-97 8-18-97 8-14-97, 8-15-97  The recent fragrance 8-15-97  Journ Donell 8/16/97 2	17/97	·	23 17	3,05 18 24.20 25 19 27,25 46 20 3,23

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# FIGURE VII

# **Density Worksheet**

	public reas			
9-24-97	DEMSite	deter mination for Method 24X	97-0216	
MS	* Not	- Method 24X		
			wt. of Ind volfask	density
	Run	wt. of Ind vol. flask	4 Inl sample	4
			· · · · · · · · · · · · · · · · · · ·	
		19.4314	20,3398	0.9084
	2	19.6354	20,5477	0.9123
	3	19.1314	20,0350	0.9036
		Arg. dens	sity = 0.9081 g/ml	
			7	

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