

FINAL REPORT

Crude MCHM

HAEL No.: 97-0216

EAN: 972790

PM No. : 18717-00

CAS : Not Available

CHEMICAL 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. COD-00775

STUDY COMPLETION DATE

October 2 , 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: CHEMICAL OXYGEN DEMAND

Karen H. Patterson
(AUDITOR, QUALITY ASSURANCE UNIT)

October 1, 1997
DATE

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 |
| 10/01/97 | | TEST REPORT INSPECTION ECHEM | 10/01/97 |

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 Bishop

Mary Lee Bishop
Study Director

October 2, 1997

Month/Day/Year

Karen R. Miller

Karen R. Miller
Eastman Contact Representative

October 9, 1997

Month/Day/Year

SIGNATURE PAGE

M. Steinbugler September 30, 1997
Mary Steinbugler Month/Day/Year
Report Author

Mary Lee Bishopp October 2, 1997
Mary Lee Bishopp Month/Day/Year
Study Director

Karen R. Miller October 9, 1997
Karen R. Miller Month/Day/Year
Eastman Contact Representative

TABLE OF CONTENTS

| | Page |
|--|------|
| ABSTRACT | 6 |
| STUDY AND TEST SUBSTANCE INFORMATION | |
| Testing Facility | 7 |
| Sponsor | 7 |
| Sponsor Representative | 7 |
| Study Dates | 7 |
| Project Participants | 7 |
| Test Substance Characterization | 7 |
| Test Substance Storage | 8 |
| PURPOSE | 8 |
| MATERIALS AND METHODS | 8 |
| Calculations | 8 |
| Protocol And Standard Operating Procedure Deviations | 8 |
| Data Storage And Record Retention | 8 |
| RESULTS | 9 |
| CALCULATIONS | 9 |
| DISCUSSION | 10 |
| CONCLUSION | 10 |
| REFERENCES | 10 |
| FIGURES | |
| I. COD Worksheet | 11 |

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ABSTRACT

The chemical oxygen demand (COD) 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 is identical to Method C.6., "Degradation, Chemical Oxygen Demand", Official Journal of the European Communities, No. L383A / 227,29 December 1992. The mean of two measurements was found to be 2.54 grams of COD per gram of the test substance. The BOD5/COD ratio was found to be 0.028.

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 7, 1997 |
| Experimental Completion Date: | August 8, 1997 |

Project Participants

| | |
|------------------------|------------------|
| Study Director | Mary Lee Bishopp |
| Principal Investigator | Tammy Kleiber |
| Report Author | Mary Steinbugler |

Test Substance Characterization

Name: Crude MCHM
EAN: 972790
HAEL No. : 97-0216
PM# : 18717-00
CAS : Not Available

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 oxidizability of the test substance under standard conditions of the oxidizing agent, temperature, and time.

MATERIALS AND METHODS

The test was performed in duplicate and 0.500 N potassium dichromate ($K_2Cr_2O_7$) solution was used to standardize the ferrous ammonium sulfate (FAS) titrant. Mercuric sulfate was added to minimize chloride interference, if any.

The method used was CQS-EAS/CLAS-QOD-0053.

Calculations

The COD concentration is calculated by subtracting the mLs of standardized FAS needed to titrate the test substance from the mLs required to titrate the blank, multiplying by the normality of the FAS, and then multiplying by the milliequivalent weight of oxygen. This value is then divided by the grams of test substance used in the analysis. The result is a value in units of grams of COD per grams of test substance.

All calculations are performed manually using a calculator.

Protocol And Standard Operating Procedure Deviations

The Chemical Oxygen Demand (COD) result, which is also used in the calculation of the BOD_5 /COD ratio, was performed in duplicate instead of triplicate. One sample setup used a sample weight that was too large. This had no adverse affect on the BOD_5 /COD ratio or the COD result.

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 COD analysis are as follows:

Replicate 1 = 2.48 grams COD/ gram of test substance
Replicate 2 = 2.60 grams COD / gram of test substance

mean = 2.54 grams COD / gram of test substance

CALCULATIONS

CALCULATION

$$\text{grams COD / gram of test substance} = \frac{(B - S)(N)(0.008)}{\text{sample weight (grams)}}$$

Where: B = mean volume of FAS used to titrate reagent blank (mL)

S = volume of FAS used to titrate sample (mL)

N = normality of FAS (meq/mL) = 0.239 meq/mL

0.008 = milliequivalent weight of oxygen

sample weight (grams) = grams of test substance used for analysis

e.g. grams COD / gram of test substance for Replicate 1

$$\text{grams COD / gram of test substance} = \frac{(41.3\text{mL} - 5.9\text{mL})(0.239\text{meq / mL})(0.008)}{0.0273\text{ grams}}$$

$$= 2.48 \text{ grams COD / gram of test substance}$$

$$\text{BOD5/COD Ratio} = \frac{\text{Average BOD}_5 \text{ day concentration}}{\text{Average COD concentration}} = \frac{0.070 \text{ grams / gram}}{2.54 \text{ grams / gram}} = 0.028$$

DISCUSSION

The results of the COD analysis for the test substance were 2.48 and 2.60 grams COD / gram test substance. The average result was 2.54 grams COD / gram test substance.

CONCLUSION

The chemical oxygen demand (COD) 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 is identical to Method C.6., "Degradation, Chemical Oxygen Demand", Official Journal of the European Communities, No. L383A / 227,29 December 1992. The mean of two measurements was found to be 2.54 grams of COD per gram of the test substance. The BOD₅ /COD ratio was found to be 0.028.

REFERENCES

1. "Chemical Oxygen Demand", Method 410, U.S. Environmental Protection Agency, EPA-600/4-79-020, March 1979.
2. "Degradation, Chemical Oxygen Demand", Method C.6., Official Journal of the European Communities, No. L 383 A / 227,29 December 1992.

FIGURE I

COD WORKSHEET

Attachment I: COD Worksheet

TMS # _____ KAN: 972790
 HAEL # 97-0216 SRID No. 6-97
 crude NICHM
 Date Set-up 8-7-97 Date Titrated 8-8-97
 Analyst Tammy Kleiber Analyst Tammy Kleiber

Replicate Analyses

| | | | |
|--|----------------|------------------------------|----------------|
| Sample Size | <u>0.0273g</u> | <u>0.0324g</u> | <u>0.0244g</u> |
| K ₂ Cr ₂ O ₇ , mL | <u>20.0</u> | <u>20.0</u> | <u>20.0</u> |
| Ave. FAS Blank, mL | <u>41.3</u> | <u>41.3</u> | <u>41.3</u> |
| FAS Sample, mL | <u>5.9</u> | <u>Sample Size too large</u> | <u>8.1</u> |
| Net FAS, mL | <u>35.4</u> | | <u>33.2</u> |
| g COD/ g | <u>2.479</u> | | <u>2.602</u> |
| Average COD | <u>2.54</u> | | |

Standardization of FAS

| | | | |
|--|--------------|--------------|--------------|
| K ₂ Cr ₂ O ₇ , mL | <u>20.0</u> | Normality | <u>0.500</u> |
| FAS titrated, mL | <u>41.80</u> | <u>41.85</u> | <u>41.80</u> |
| Normality of FAS | <u>0.239</u> | <u>0.239</u> | <u>0.239</u> |

Comments: Average Normality of FAS 0.239
 Blank Titrations ① 41.3
 ② 41.3
 ③ 41.3
 Average Blank Titration 41.3
 Analyst(s) Tammy Kleiber Date 8-8-97

This is an exact reduced copy of the original document.

By M. Blankley Date September 30, 1997