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GERAGHTY & MILLER, INC.
Ground-Water Consultants

ORIGINAL (Red)

July 31, 1987

Ms. Ruth Rzepski (3HW16)
Compliance Officer
U.S. Environmental Protection Agency
Region III
841 Chestnut Building
Philadelphia, PA 19107

DOC. CONTRL. #
M0679FR6-GM-7-31-87-1

and

Ms. Diana Pickens (3ES23)
Chemist-Quality Assurance
U.S. Environmental Protection Agency
Region III
839 Bestgate Road
Annapolis, MD 21401

Dear Ms. Rzepski and Ms. Pickens:

In accordance with the conversation of July 31, 1987, between Diana Pickens and Michele Ruth, Geraghty & Miller, Inc., (G&M) submits this revision of responses to Comments 1 and 2. QC Documentation for Avtex Laboratory, which was originally submitted on July 1, 1987 (Doc. Control #M0679FR6-GM-7-1-87-3). We appreciate your cooperation and timely response. If you have any further questions or comments, please let us know.

Sincerely,

GERAGHTY & MILLER, INC.

Michele C. Ruth
Project Engineer

Jeffrey P. Sganbat, C.P.G.
Project Consulting Coordinator

MCR/JPS:gph

AR301112

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Comments 1 and 2. QC Documentation for Avtex Laboratory

With further review and planning for the performance of the bench-scale treatability study in support of the CERCLA action at Avtex Fibers, Inc., Front Royal, Virginia, Geraghty & Miller, Inc., (G&M) has modified the study and laboratories involved in the analysis of wastewater. The following discussion answers concerns raised by EPA with regard to QC documentation and the intended use of generated chemical data. The proposed changes in performing the actual bench-scale study are presented as subsequent sections within this document.

Avtex Fibers' "in-house" laboratory will perform the following analyses for trend-analysis of the bench-scale treatability study:

- $BOD_5$
- COD
- Suspended Solids
- Mixed Liquor Solids (Total and Volatile)
- Zinc
- pH
- Dissolved Oxygen
- Oxygen Uptake
- Flow
- Sludge Settling
- Temperature.

These analyses are done on a routine basis at the Avtex lab, and conducting these tests "in-house" should provide adequate data for trend analysis. In addition, proper maintenance of



AR301113

ORIGINAL (Red)

the test reactors requires immediate information on many of the above parameters and can be effectively managed only through "in-house" analyses. The data generated by Avtex's laboratory will only be used for trend-analysis, and is not intended for comparision with the results from Cambridge Analytical Associates, Inc. (CAA).

The methods which are routinely used by the Avtex laboratory are described in the attached information, and will be employed for the treatability study analytical work; many of the methods used are those described in Standard Methods. The analytical procedures for volatile solids is not included in the attached information, but is identical to that described in Standard Methods, 13th Edition. The employment of methods which are not in accordance with Standard Methods, but are routinely used by the Avtex laboratory personnel, are most likely to provide the most consistent data, due to the familiarity of the lab personnel with those procedures.

CAA will analyze samples of the feed and the effluent for each of the test reactors on a weekly basis during the testing portion of the study. These analyses will include:

- Sulfides
- Chlorides
- TOC
- Sulfate

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- T.K.N
- Ammonia as Nitrogen
- Phosphate
- Phenol
- $CS_2$
- COD
- Zinc.

Samples which are sent to the CAA for the above analyses will be prepared, preserved, and analyzed in accordance with Table 1.

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# ANALYTICAL PROCEDURES EMPLOYED AT AVTEX FIBERS' LABORATORY

# Total Digested Zinc

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1. In Zinc free 500 ml flask add 50mls of Composite 100 mls River Sample Sample + 5mls Conc. nitric acid

2.- Heat slowly on hot plate to dryness or almost dryness.

3.- Cool, add 5mls more nitric, Cover with glass dis + heat to dryness

4- Cool until warm, add ± 2mls more HCL or nitric. Wash down walls of flask w distilled $H_2O$. There will be a white precipitate. Let it settle and decant fluid into zinc free 500 ml volumetric. Rinse digestion flask 2 or 3 more times decanting into 500ml Volumetric. Fill to level line + use AA as before. Multiply answer by 10.

5- For River total zinc sample follow step 1-3 Cool, add 1-2 mls more nitric, wash down walls of flask with distilled $H_2O$. Decant into 100 ml Volumetric + fill to level line. Use AA as before - Report answer directly from AA

AR301117

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# COD (Jeri's Method)

1) (1 dipper) 0.3g of $HgSO_4$ into 125ml Florence flask.
2) Add boiling stones
3) Add 25ml of dichromate acid mixture to each flask + add required amount of sample
4) Put thermometer in flask marked blank and place samples on preheated hot plate Heat contents to 165+C. Remove from heat. Allow contents to cool to about 80°C. Remove thermometer from sample rinse tip with distilled $H_2O$.
5) Have 500 ml wide mouth Erlenmeyer Flask set up with distilled $H_2O$. Transfer the contents of small flask to the large flask. Add stirrer bar + about 5 drops of ferroin indicator and titrate with Ferrous Ammonium Sulfate.

Blank make up
1) 10 mls of distilled $H_2O$
2) 25 mls of dichromate Acid mixture.

STd. make up
1) 250 mls of distilled $H_2O$
2) 25 mls of 0.50N $K_2Cr_2O_7$
3) 20 mls of Sulfric Acid

AR301118

CALC. $\frac{.050N \times 25}{STD.} =$

CALC. (Jeri's) ____ $N\,Fe(NH_4)_2(SO_4)_2$ ____ STD. FACTOR

BLANK - SAMPLE tit. X S.Td. FACTOR =

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# SATURATED AIR CALIBRATION OF THE YSI MODEL 54 DISSOLVED OXYGEN METER

1. Check all wiring for good connection.

2. Fill the DO probe storage bottle half full with room temperature water. Seat the DO probe in the bottle, making sure that the probe membrane is free of any water droplets.

3. Switch the DO Meter to the Red Line Position. Allow the meter to "warm up" for 10 minutes.

4. Adjust meter to "Red Line" with the Red Line Control knob.

5. Switch to "Temperature" setting and read the temperature.

6. Switch to "Zero" and adjust the meter with the Zero Control knob.

7. Switch to the appropriate range for DO measurement of the sample (0-10 or 0-20) and read the DO in the saturated airspace of the probe storage bottle. Do not operate the motorized stirrer for this measurement.

8. Refer to Table I below to read the concentration of DO in the saturated airspace, at a barometric pressure of 29.92 inches, at the measured temperature. Adjust the "Calibration" knob to the value as read from Table I.

9. Transfer the probe to the solution to be measured and agitate. Allow to come to temperature.

10. If changing the membrane is required, refer to the YSI Model 54 Instruction Manual.

Reference: Yellow Springs Instrument Company
Instructions for YSI Model 54 Oxygen Meter

4-21-86 MRP

Calibration Table

Table I

SOLUBILITY OF OXYGEN IN WATER Saturated with Air IN PPM
– AT VARIOUS TEMPERATURES AND PRESSURES

| P mm | 775 | 760 | 750 | 725 | 700 | 675 | 650 | 625 |
|---|---|---|---|---|---|---|---|---|
| P inches | 30.51 | 29.92 | 29.53 | 28.54 | 27.56 | 26.57 | 25.59 | 24.61 |
| 15 | 10.4 | 10.2 | 10.0 | 9.7 | 9.5 | 9.3 | 9.1 | 8.9 |
| 16 | 10.1 | 9.9 | 9.8 | 9.5 | 9.3 | 9.1 | 8.9 | 8.7 |
| 17 | 9.9 | 9.7 | 9.6 | 9.3 | 9.0 | 8.8 | 8.7 | 8.5 |
| 18 | 9.7 | 9.5 | 9.4 | 9.1 | 8.8 | 8.6 | 8.4 | 8.2 |
| 19 | 9.5 | 9.3 | 9.2 | 8.9 | 8.6 | 8.5 | 8.3 | 8.1 |
| 20 | 9.3 | 9.2 | 9.1 | 8.7 | 8.4 | 8.3 | 8.1 | 7.9 |
| 21 | 9.2 | 9.0 | 8.9 | 8.6 | 8.3 | 8.1 | 7.9 | 7.7 |
| 22 | 9.0 | 8.8 | 8.7 | 8.4 | 8.1 | 7.9 | 7.8 | 7.6 |
| 23 | 8.8 | 8.7 | 8.6 | 8.3 | 8.0 | 7.8 | 7.6 | 7.4 |
| 24 | 8.7 | 8.5 | 8.4 | 8.1 | 7.9 | 7.7 | 7.5 | 7.3 |
| 25 | 8.5 | 8.4 | 8.3 | 8.0 | 7.7 | 7.6 | 7.4 | 7.2 |
| 26 | 8.4 | 8.2 | 8.1 | 7.9 | 7.6 | 7.5 | 7.3 | 7.1 |
| 27 | 8.2 | 8.1 | 8.0 | 7.7 | 7.5 | 7.3 | 7.2 | 7.0 |
| 28 | 8.1 | 7.9 | 7.8 | 7.6 | 7.3 | 7.2 | 7.0 | 6.8 |
| 29 | 7.9 | 7.8 | 7.7 | 7.4 | 7.2 | 7.0 | 6.9 | 6.7 |
| 30 | 7.8 | 7.7 | 7.6 | 7.3 | 7.0 | 6.9 | 6.8 | 6.6 |

AR301119

ORIGINAL
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$BOD_5$ PROCEDURE

Reference: Standard Methods
13th Edition

1. Dilution of sample

The dilutions are made for final effluent at 10, 15, and 25. Dilution water is siphoned into a 400 ml graduated cylinder to half full. For each dilution suitable aliquot is added:

10 - 40 mls
15 - 50 mls
25 - 100 mls

The cylinder is filled to 400 ml mark with dilution water. Mix well without entrapping air. Syphon into BOD bottle. A BOD bottle of dilution water is also prepared.

2. Using the saturated air method, the oxygen meter is standardized to a known concentration of dissolved oxygen. The dissolved oxygen is measured on the dilution blank and all samples. Extra drops of dilution water can be added to BOD bottle to make up for sample lost on probe. Make certain there are no bubbles trapped when sealing and stoppering.

3. Incubate blank and samples for five days in dark at 20°C. The D.O. meter is used after five days to determine remaining dissolved oxygen. The blank is used to check the quality of the dilution water and should not be more than 0.2 mg/l. Do not subtract blank from sample.

4. Calculations

Subtract D.O. after five days from D.O. at start and divide by decimal fraction of sample used.

$$\text{mg/l BOD} = \frac{DO_1 - DO_5}{\text{Decimal Fraction of Sample}}$$

either 0.5, 0.25, 0.15, based on the following

Initial D.O. $\geq$ 7.0 mg/l
D.O. Depletion $\geq$ 2.0 mg/l
Final D.O. $\geq$ 1.0 mg/l

AR301120

AR301120

Location: _____ Record on: _____

| Dilution ORIGINAL (Red) | Dissolved Oxygen Init. | Dissolved Oxygen Final | Dissolved Oxygen Diff. | % Depletion | Conc. Factor | Mg/L $BOD^5$ | Flow Mgd | BOD Lbs/Day | Date In / Date Out / Analyst |
|---|---|---|---|---|---|---|---|---|---|
| | | | | | | | | | |

AR301121

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TOTAL ZINC PROCEDURE

Reference: Standard Methods for the Examination
of Water and Wastewater
Section 301 AI.1.

Sample Storage

Immediately after sampling, acidify sample for zinc analysis to pH ∠2 with nitric acid and refrigerate. Samples can be stored for six months using above procedure. Digestion pretreatment of samples will normally occur on Fridays.

Digestion Pretreatment

1. In 500 ml (zinc-free) flask add 50 mls sample and 5 mls concentrated nitric acid.
2. Heat slowly on hot plate to dryness.
3. Cool, add 5 mls more nitric, cover with glass disc and heat to dryness.
4. Cool until warm, add 1-2 mls more ~~nitric~~ HCL or nitric acid. Wash down walls of flask with demin. distilled water. Let the white precipitate settle and decant liquid into zinc-free 500 ml volumetric flask. Rinse digestion flask two or three more times decanting into flask. The precipate can be filtered out as well, then transfer filtrate into volumetric flask. Fill volumetric flask to level line and use AA as described for zinc. Multiply answer by 10.

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## pH MEASUREMENT

1. Standardize the pH meter using two buffers, one of which is of the same pH (± 0.5 units) as the sample to be tested.

2. Rinse the electrode with distilled water and blot dry.

3. Measure the temperature of the sample. Set the temperature knob on the meter to the temperature of the sample.

4. Immerse the pH probe into the sample. (Best results are obtained when the sample is being stirred during pH measurement.)

5. When needle (analog) or readout (digital) has stopped moving, read and record pH.

6. Return the probe to a neutral aqueous solution. Return operating switch of the pH meter to "Standby".

# TOTAL DISSOLVED SOLIDS - BY CALCULATION

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1. Sample Preservation

   a. Begin analysis as soon as possible

2. On sample run total suspended solids as usual.

3. Total Residue - Dried at 103-105°

   a. Ignite clean evaporating dish in muffle furnace for one hour.

   b. Cool, place in desiccator to store and weight dish.

   c. Transfer sample to preweighed dish and evaporate in drying oven, set at 98°C. for Wells No. 1, 2, 5 - 50 mls sample Well No. 3 - 10 mls sample; Wells No. 4 and 6 - 25 mls sample

   d. Reset drying oven to 103-105° and dry evaporated sample for one hour.

   e. Cool dish in desiccator and weigh.

   f. Calculation:

$$\text{mg/l Total Residue} = \frac{(\text{Weight of Sample + Dish} - \text{Weight of Dish}) \times 1{,}000}{\text{Volume of Sample (ml)}}$$

4. Calculation of Total Dissolved Solids:

   Total Residue (mg/l) - Total Suspended Solids (mg/l) = Total Dissolved Solic

5. Reference: Standard Methods for the Examination of Water and Wastewater Section 208

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# TOTAL SUSPENDED SOLIDS
# or
# TOTAL NONFILTERABLE RESIDUE

1. Measure 200 ml of sample to be tested.

2. Weigh a properly prepared, empty Gooch crucible.

3. Place the crucible in the vacuum apparatus, being sure that the wrinkled surface of the filter disk is facing upward. With the vacuum applied, wet the disk to seat it against the holder.

4. With the vacuum applied, filter the sample.

5. Leaving the suction on, wash the apparatus three times with 10 ml portions of distilled water, allowing complete drainage between washings.

6. Stop vacuum, remove the Gooch crucible and dry in an oven at 103 - 105° C for 1 hour. After drying, cool to room temperature in a dessicator.

7. Reweigh the cooled Gooch crucible on the analytical balance.

Calculation: $\text{mg/l TSS} = \frac{(\text{wt. of sample} - \text{wts. of empty crucible})(1000)}{\text{ml of sample}}$

## Preparation of Gooch crucibles

1. Rinse each crucible with 100 ml of distilled water;

2. Place a Whatman glass microfibre filter, 934-AH, in the crucible with the wrinkled side up;

3. Dry in an oven at 103° - 105° C for 1 hour;

4. Store crucibles in a dessicator until further use.

Reference: STANDARD METHODS 13th edition

## Weekly check of drying time

1. After weighing the 1 hour dried sample, place the sample back in the drying oven (103 - 105° C) for an additional 30 minutes. Remove and cool in a dessicator.

2. Reweigh on the analytical balance. If the additional weight loss is 4 percent or 0.5 mg (whichever is less), the initial drying time should be increased to 1-1/2 hours.

# TECHNICAL INSTRUCTIONS

NUMBER ORIGINAL (Red)

DATE 6-18-86

TO: [illegible]
[illegible]
[illegible]
[illegible]

STAFF CHEMIST
TECH. SUPVR. - STAPLE - YARN
SECTIONAL ENGINEERS
PROCESS CONTROL SUPVR.

## SUSPENDED SOLIDS

### (For Sample With High Amount of Solids)

A properly prepared gooch is removed from dessicator and weighed. Record this weight.

Pipette exactly 10 ml of mixed liquor into the center of the tared gooch. Apply suction to dryness and then wash three times with 10 ml of distilled water. Dry in electric oven at 103°C for one hour. Cool in dessicator and reweigh. Subtract from first weight to obtain solids weight.

CALCULATIONS:

mg/l MLSS = Weight of solids in grams x 100,000

## SUSPENDED SOLIDS

### (For Clear Samples)

A properly prepared gooch is removed from dessicator and weighed. Record this weight.

Using a 250 ml graduated cylinder transfer 250 ml of sample to the gooch. Use stirring rod to direct flow to center of gooch. Apply suction to dryness and wash three times with 10 ml of distilled water. Dry in electric oven at 103°C for one hour. Cool in dessicator and reweigh. Subtract weights.

CALCULATIONS:

mg/l Eff. SS = Weight of solids in grams x 4,000

AR301126

Michael R. Pisarcik

TECHNICAL DEPT. APPROVAL [signature]

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# TECHNICAL INSTRUCTIONS

NUMBER 1404-2

DATE 6-18-86

## SETTLEABLE SOLIDS

### (30 Minute Settling Test)

Fill a one liter Imhoff cone to the one liter mark with mixed liquor from the aeration chamber. Record time and allow to settle, undisturbed, for 30 minutes. Record the ml of solids at the base of the cylinder.

## ZONE SETTLING

Same procedure as above but readings are recorded at five minute intervals for 30 minutes.

## SETTLEABLE SOLIDS

### (Sanitary Sewer Raw Wastes and Effluents)

Fill a one liter Imhoff cone to the one liter mark with raw waste or effluent. Record time and allow to settle, undisturbed, for 45 minutes. Then, using a stirring rod, lightly scrape the solids from the inner wall of the cone. Allow to settle 15 minutes more - total settling time - one hour.

AR301127

PREPARED BY Michael R. Pisarcik TECHNICAL DEPT. APPROVAL C.D. Ritter

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# TECHNICAL INSTRUCTIONS

NUMBER 1405-2

DATE 6-18-80

## YSI MODEL 54 OXYGEN METER – $O_2$ UPTAKE CALCULATION

With this instrument you measure the temperature and dissolved oxygen and oxygen uptake when recorder is attached.

Dissolved oxygen is measured with a YSI Model 54 Oxygen Meter in the following man

A. Calibration - Calibrate the DO meter with a BOD bottle of distilled water saturated with air. Initially the zero and red line adjustments are made. Then measure the temperature of the water and refer to the calibration table on P. 26-27 of the YSI Meter Instruction Manual. Adjust the DO reading with the calibration knob for the table valve at the temperature of the water.

B. DO Measurement - Immerse the tip of the DO Probe in the DO bottle that has been filled with sample. Turn knob to temperature and read °C. Then turn knob to 0-10 and read PPM. Recheck calibration after test.

C. Oxygen Uptake - Same procedure as in Step A. Record the DO initially and again after five minutes.

D. Calculation of $O_2$ Uptake from recorder charts -

$$DO_i - DO_{5\ Min.} \times 12 = \text{Oxygen Uptake } (mgO_2/l/hr.)$$



Michael P. Pizzarik