MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPTS. 2,3,4,5,7,
8,20, 44,
45,46,48,
56,60
METHOD 602.009 (WC)
Rev. 7/19/71

MATERIAL

Product SM, AN, MeOH, Bz, Toluene, EB and all other clear and colorless materials requiring an APHA color measurement, - DOP, DIDP, -711- Other Alcohols, Caustic.

ANALYSIS REQUIRED

Color

SAMPLE

One quart bottle

APPARATUS

Nessler (short) tubes 100 ml marked at 150 mm from inside bottom.
Lumetron Model 450 with color filter #B-420.

REAGENTS

Distilled water XXX
APHA 500 Standard

PROCEDURE

1. With switch off, set instrument to 0% transmission by means of the lever in the back.

2. Fill a tube to 150 mm mark with clear distilled water. Insert tube in machine and turn switch on.

3. Adjust the instrument to 100% transmission by menas of the adjustment level at thetop. Turn switch off and remove tube.

4. Fill a tube with sample and place in instrument. Turn switch on and read % transmission from the scale.

7/20/71

PREPARATION OF REAGENTS

None

STANDARDIZATION AND/OR CALIBRATION

Prepare standards by diluting APHA 500 stock at rate of 0, 1, 2, 3, 4, & 5 ml to 100 ml with distilled water. The standards represent 0, 5, 10, 15, 20 & 25 APHA colors. Read the % transmission of these vs. the distilled water used. Plot the %-transmissions thus obtained vs. the corresponding APHA colors. Draw in the best representative line. Use this curve for routine work or prepare a chart from it showing %T vs. APHA color.

SAFETY

Use normal precautions in handling chemicals, glassware and electrical equipment.

ANALYTICAL TIME

5 minutes

PRECISION OF RESULTS AND SIGNIFICANT FIGURES TO REPORT

95% confidence limits are $\pm$ 2 at a level of 10 APHA. Report results to the nearest 1 APHA.

SCOPE

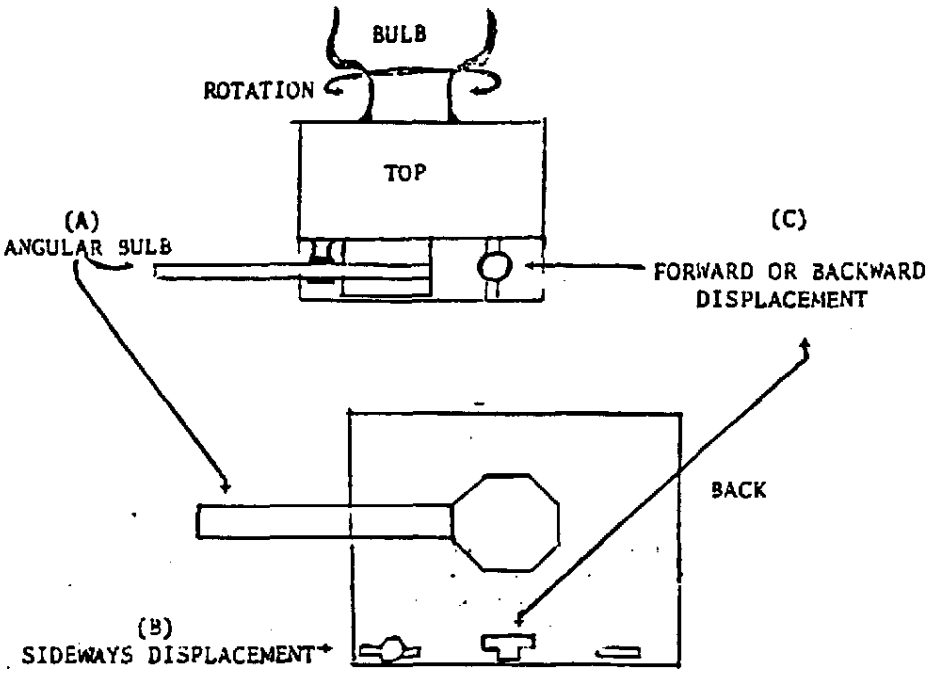
The color of the sample is compared to the APHA scale.

REFERENCES

Method 31-16-6

/jw
7/20/71

LIGHT ADJUSTMENT ON LUMETRON



----------STEPS IN ADJUSTING LIGHT SOURCE----------

1. When bulb* burns out; remove light housing and, replace bulb.
2. Adjust iris diaphragm to 50% transmittance.
3. Adjust Bar A for maximum deflection to the right.
4. Loosen thumb screw B and adjust housing sideways for maximum deflection to the right and retighten.
5. Loosen thumb screw C and adjust backward or forward for maximum deflection to the right.
6. Adjust dark current to zero.
7. The instrument is ready to use.
8. Follow usual procedures for adjusting light source on a water blank for 100% transmittance.

* The bulb used has a double contact on the base and is coded GE 1130.

/jw
7/20/71

MATERIAL

10-D7-OH, Gulf C_2H_4, Humble C_2H_4, Dept. 2 & and Dept. 3 Meter Runs, Dept. 2 & 3 C_2H_4 Composite, Dept. 23 C_2H_4 Composite, Dept. 41 C_2H_4

ANALYSIS REQUIRED

CH_4, C_2H_2, C_2H_4, C_2H_6

SAMPLE

1.7 liter oxygen bomb with positive sample pressure.

APPARATUS

Perkin-Elmer Vapor Fractometer, Model 154B/or equivalent
Gas charging valve with 1 cc sample loop
1/4" O.D. aluminum or stainless steel tubing

REAGENTS

30-60 mesh silica gel
Methane, Phillips Research Grade
Ethane, Phillips Research Grade
Ethylene, Phillips Research Grade
Acetylene, Commercial Grade - water scrubbed and dried.

PROCEDURE

A. Column Preparation

1. Pack a 1.5 meters column of 1/4" tubing with 30-60 mesh silica gel.

2. Install the column in the Fractometer. Adjust the temperature to 50°C., determine optimum detector current and set at this value, and adjust the pressure such that the ethylene peak appears in 3-4 minutes after charging the sample. Allow the instrument to reach equilibrium at these conditions.

B. Analysis

1. Turn recorder on and set recorder range to 1. Recorder trace should show no drift for five minutes before Fractometer is ready for use.

2. Purge 1 cc gas sample loop with sample to be analyzed, shut off valve from bomb, and enter 1 cc sample into Fractometer.

3. Record peaks for air, methane, ethane, ethylene, and acetylene or allow enough time for acetylene to appear as determined by retention times from a previous chart run under the same conditions.

CALCULATIONS

1. Draw base lines for all peaks except air.

2. Use rule with 50 divisions per inch and measure peak height and width at one-half peak height for all peaks. Measurements should be made to the nearest one-tenth of a division.

3. Calculate corrected area for all peaks as follows:

Corrected area = h x w x r x F

Where: h = peak height
w = width at one-half peak height
r = recorder range
F = calibration factor for that component

4. Calculate mol percentage of each component in the sample as follows:

$$\text{Mol \%} = \frac{A_{corr.} \times 100}{\sum (A_{corr.})}$$

Where: $A_{corr.}$ = corrected area from Step 3

$\sum (A_{corr.})$ = total of all corrected areas calculated in Step 3.

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare at least two synthetics containing methane, ethane, and acetylene in approximately 95% ethylene. Allow to stand overnight to insure thorough mixing.
2. Analyze as in the procedure under Analysis.
3. Measure peaks as in Steps 1 and 2 under Calculations.
4. Calculate area of each peak as follows:

   $A = h \times w \times r$

   Where: A = area
   h = peak height
   w = width at one-half peak height
   r = recorder range

5. Calculate factors for each component according to the following equation:

   $$F = \frac{\%}{A}$$

   Where: F = factor
   % = mol % of component in synthetic
   A = area of component peak calculated in Step 4 above

6. All factors should be normalized such that the factor for ethylene equals 1.000.

SAFETY PRECAUTIONS

No special precautions need be observed other than those normally observed when handling gaseous hydrocarbon samples.

ANALYTICAL TIME

The average time of analysis using this method should be 0.4 hours.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits using this method should be + or - 0.5%. Report results to nearest 0.01%.

SCOPE

This method is designed for samples containing only methane and the C_2's. Samples containing propane, but not propylene, may also be analyzed using this method as propane has a retention time slightly less than acetylene. If "acetylene" appears in a sample normally known not to contain this component, the sample should be analyzed by another method; eg., infrared, to determine if it is acetylene or propane.

/jw
7/20/71

MATERIAL

High Purity Ethylene

ANALYSIS REQUIRED

Carbon Dioxide

SAMPLE

Minimum of liter stainless steel bomb with positive sample pressure.

APPARATUS

Perkin-Elmer Vapor Fractometer, Model 154B/or equivalent.

Gas charging valve with 5 cc sample loop.

1/4" O.D. aluminum or stainless steel tubing.

REAGENTS

Activated Charcoal (Absorbent-A, used as purchased from Burrell Corp.)

Di-2-ethyl hexyl sebacate (Octoil "S" or Narcoil 20)

High purity ethylene - CO_2 free

CO_2

PROCEDURE

A. Column Preparation

1. Pack 6 feet of 1/4" aluminum or stainless steel tubing with activated charcoal modified with 4 wt.% di-2-ethyl hexyl sebacate.

2. Install the column in the chromatograph. Adjust the temperature to 70°C, determine optimum detector current and set at this value, and adjust the pressure such that the CO_2 peak appears in approximately 5 minutes after charging the sample. Allow the instrument to reach equilibrium at these conditions.

PROCEDURE (cont'd)

B. Analysis

1. Turn recorder on and set recorder range to 1. Recorder trace should show no drift for five minutes before chromatograph is ready for use.

2. Purge 5 cc gas sample loop with sample to be analyzed, shut off valve from bomb, and enter 5 cc sample into chromatograph.

3. Record all peaks up to and including CO_2 peak or allow enough time for CO_2 to appear as determined by retention time from a previous chart run under the same conditions.

CALCULATIONS

1. Draw base line for CO_2 peak only.

2. Use rule with 50 divisions per inch and measure peak height and width at one-half peak height for the CO_2 peak. Measurements should be made to the nearest one-tenth of a division.

3. Calculate ppm of CO_2 as follows:

ppm CO_2 = h x w x r x F

Where h = peak height
w = width at one-half peak height
r = recorder range
F = calibration factor for CO_2, expressed as ppm/unit area

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare at least two synthetics containing CO_2 in high purity ethylene. These synthetics should be in the range of 10 - 50 ppm CO_2. Allow to stand overnight to insure thorough mixing.

2. Analyze as in the procedure under Analysis.

3. Measure the CO_2 peak as in Steps 1 and 2 under Calculations.

4. Calculate area of CO_2 peak as follows:

   $A = h \times w \times r$

   Where A = Area
   h = peak height
   w = width at one-half peak height
   r = recorder range

5. Calculate CO_2 factor according to the following equation:

   $$F = \frac{C}{A}$$

   Where F = Factor
   C = Concentration of CO_2 in synthetic expressed in ppm
   A = Area of CO_2 peak calculated in Step 4 above.

6. Calibrations should be checked daily.

SAFETY PRECAUTIONS

No special precautions need be observed other than those normally observed when handling gaseous hydrocarbon samples.

ANALYTICAL TIME

The average time of analysis using this method should be 0.2 hour.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits using this method should be ≠ or -2ppm. Report results to nearest ppm.

SCOPE

This method is designed for trace CO_2 analysis of high purity ethylene, although it may be used for the determination of CO_2 in any inert or hydrocarbon gas mixture.

If used for analysis of streams containing larger concentrations of CO_2, it may be desireble to use a smaller sample size. If the sample size is changed, the factor should be adjusted for the new sample size. Samples containing large amounts of butanes and heavier will eventually "over-modify" the carcoal and cause a decrease in the retention time of CO_2. If this occurs, it may be necessary to replace column.

/jw
7/20/71

MATERIAL

All gas samples, hydrocarbon and inert samples.

ANALYSIS REQUIRED

Oxygen, Nitrogen and Carbon Monoxide

SAMPLE

Minimum of one liter stainless steel bomb with positive sample pressure.

APPARATUS

Perkin-Elmer Vapor Fractometer, Model 154B/ or equivalent.

Gas charging valve with 25 cc sample loop.

1/4" O.D. aluminum or stainless steel tubing.

REAGENTS

5 Angstrom Molecular Sieve/or 13 Angstrom Molecular Sieve

Air - scrubbed with caustic and dried.

CO

Helium

PROCEDURE

A. Column Preparation

1. Pack a 2 meter length of 1/4" aluminum or stainless steel tubing with 30 - 60 mesh molecular sieve which has been dried in an oven overnight at 110°C.

2. Install the column in the chromatograph. If a 5 Angstrom sieve is used, adjust temperature to 70°C; if a 13 Angstrom sieve is used, adjust temperature to 50°C. Determine optimum detector current and set at this value, and adjust the pressure such that the oxygen peak appears in one minute after charging the sample. Allow the instrument to reach equilibrium at these conditions.

PROCEDURE (Cont'd)

B. Analysis

1. Turn recorder on and set recorder range to 1. Recorder trace should show no drift for five minutes before chromatograph is ready for use.

2. Purge 25 cc gas sample loop with sample to be analyzed, shut off valve from bomb, and enter 25 cc sample into chromatograph.

3. Record peaks for oxygen, nitrogen, methane, and carbon monoxide or allow enough time for carbon monoxide to appear as determined by retention time from a previous chart run under the same conditions.

CALCULATIONS

1. Draw base lines for all peaks except methane.

2. Use rule with 50 divisions per inch and measure peak height and width at one-half peak height for all peaks. Measurements should be made to the nearest one-tenth of a division.

3. Calculate concentration of all components, except methane, in ppm as follows:

$$ppm = h \times w \times r \times F$$

Where h = peak height
w = width at one-half peak height
r = recorder range
F = calibration factor for that component expressed as ppm/unit area.

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare at least two synthetics containing air, which has been caustic-scrubbed and dried, and carbon monoxide in helium. 2.46 mm of air per 100 psia synthetic will be 100 ppm oxygen. Synthetics should be in the range of 25-100 ppm oxygen and carbon monoxide. Allow to stand overnight to insure thorough mixing.

CALIBRATION (Cont'd)

2. Analyze as in the procedure under Analysis.

3. Measure peaks as in Steps 1 and 2 under Calculations.

4. Calculate area of each peak as follows:

$$A = h \times w \times r$$

Where A = Area
h = peak height
w = width at one-half peak height
r = recorder range

5. Calculate factors for each component according to the following equation:

$$F = \frac{C}{A}$$

Where F = Factor
C = concentration of component in synthetic expressed in ppm
A = area of component peak calculated in Step 4 above.

6. Calibrations should be checked daily.

SAFETY PRECAUTIONS

No special precautions need be observed other than those normally observed when handling gaseous hydrocarbon samples.

ANALYTICAL TIME

The average time of analysis using this method should be 0.3 hour for a 5 Angstrom column and 0.2 hour for a 13 Angstrom column.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits using this method should be $\pm$ 4 ppm. Report results to nearest ppm.

SCOPE

This method is designed for high purity ethylene samples; however, it may be used on any inert or gaseous hydrocarbon mixture. If argon is present in the sample, it will be eluted with oxygen. Periodically, depending on usage, the column may need to be regenerated. This is done by purging with nitrogen or helium for 2 hours while the column is maintained at 500° to 550°F. If sample volume is changed, all factors should be adjusted to new sample volume.

GL/nv

MATERIAL

High Purity Ethylene

ANALYSIS REQUIRED

Hydrogen

SAMPLE

Minimum of one liter stainless steel sample bomb with positive-sample pressure.

APPARATUS

Loenco, Model 15-B - or equivalent.
Gas charging valve with a 25 cc sample loop.
1/4" O.D. aluminum or stainless steel tubing.

REAGENTS

13 Angstrom molecular sieve, 20-60 mesh.
Nitrogen (to be used as carrier gas)
Hydrogen, electrolytic
Ethylene, Phillips Research Grade

PROCEDURE

A. Column Preparation

1. Activate the molecular sieve by drying in an oven at 400 °F for 3 hours, cool in a desiccator and store in screw-cap bottles. Pack 12 feet of 1/4" aluminum or stainless steel tubing with the activated sieve.

2. Install the column in the chromatograph. With the instrument at room temperature, determine the optimum detector current and set at this value. Adjust the pressure such that the hydrogen peak appears in approximately 1.5 - 2.0 minutes after charging the sample. Allow the instrument to reach equilibrium at these conditions.

B. Analysis

1. Turn on recorder and line out equipment.
2. Purge 25 cc sample loop with sample for 2 to 3 minutes or until all air has been removed.
3. Stop purge and after pressure in sample loop has stabilized, enter ethylene sample into instrument.
4. The recorder pen will deflect off scale when sample is entered but should return to base-line point after one minute.
5. Record hydrogen peak.

CALCULATIONS

1. Read peak height and calculate ppm hydrogen in sample.

   ppm H_2 = Peak height x attenuation x factor

PREPARATION OF REAGENT SOLUTIONS

None necessary

CALIBRATION

1. Prepare standards samples of hydrogen in ethylene in the 1 to 50 ppm range.
2. Analyze these standard samples following the steps under procedure.
3. Read peak heights and calculate calibration factor.

   $$F = \frac{\text{Concentration of } H_2}{\text{Peak height x attenuation}}$$

SAFETY PRECAUTIONS

No special precautions need be observed other than those normally observed when handling gaseous hydrocarbon samples.

ANALYTICAL TIME

The average time of analysis using this method should be 0.2 hour.

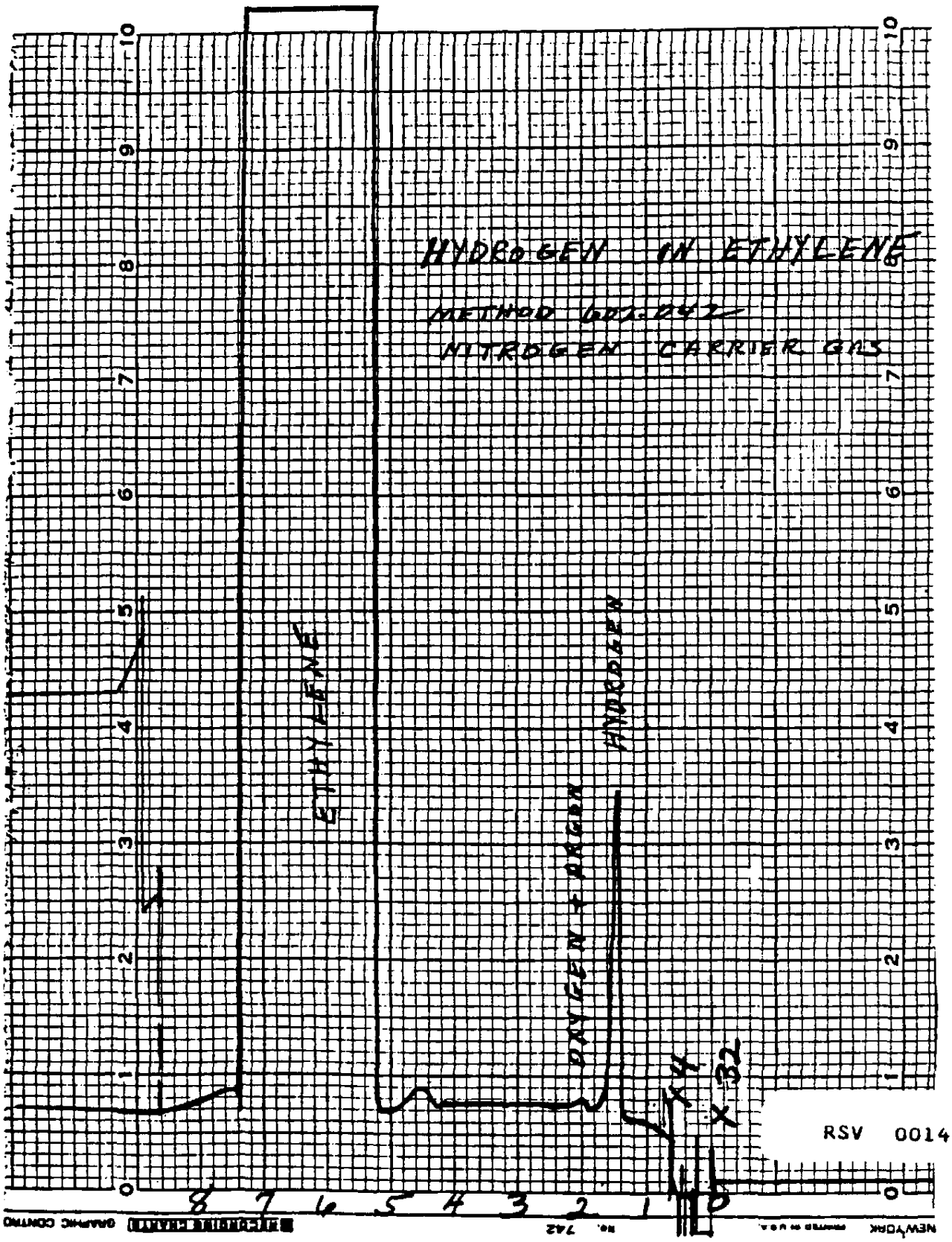
PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

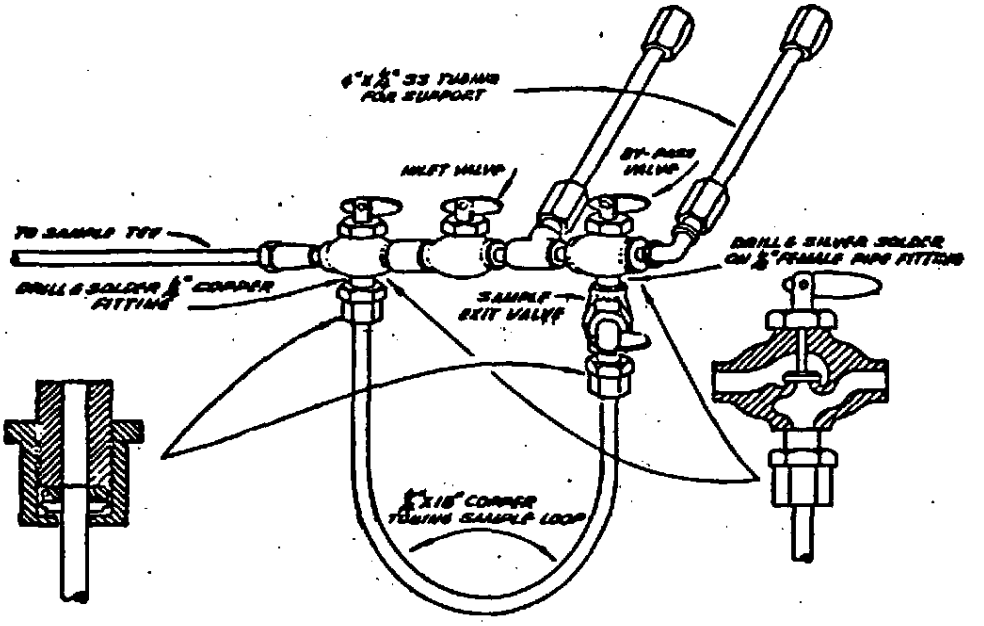
95% confidence limits using this method should be + or -.5 /ppm in the 0-50 ppm range. Report results to nearest 1 ppm.

SCOPE

This method is applicable to any gaseous mixture. Some changes may be desirable as to sample size depending on the concentration of hydrogen in the sample.

LYS:ff
10/19/64


HYDROGEN ON ETHYLENE
METHOD 602.042
NITROGEN CARRIER GAS
ETHYLENE
OXYGEN + ARGON
HYDROGEN
X4
X32
RSV 0014972



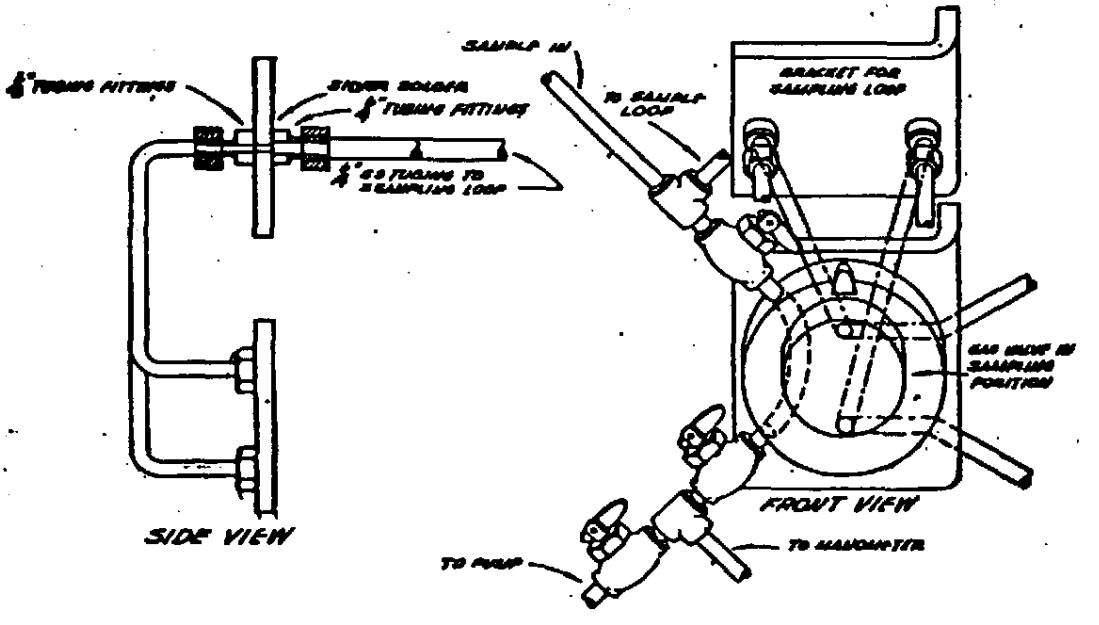


FIGURE I
CONCENTRATION & SAMPLING EQUIPMENT

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.043 (G.C.)
Rev. 7/20/71

MATERIAL

High Purity Ethylene

ANALYSIS REQUIRED

Propylene and other olefins.

SAMPLE

Minimum of one liter stainless steel bomb with positive-sample pressure.

APPARATUS

Perkin-Elmer Vapor Fractometer, Model 154B/or equivalent.

Gas charging valve with 1 cc. sample loop (modified for evacuating the sample loop)

¼" O.D. aluminum or stainless steel tubing.

Vacuum pump capable of producing 50 microns vacuum or better, with a free air capacity of at least 21 liters per minute.

Mercury manometer.

REAGENTS

Celite, 30-80 mesh; a satisfactory source is the Celite Division, Johns Manville Company, New York.

Diethyl Ether, for column preparation.

Helium.

Hexamethylphosphoramide, available from Monsanto Chemical Company, Organic Chemicals Division, 800 N. 12th Street, St. Louis, Missouri.

Propylene, Phillips Research Grade.

PROCEDURE

A. Column Preparation

1. Pack 25 feet of ¼" aluminum or stainless steel tubing with 30-80 mesh Celite aggregate containing 0.2 g of hexamethylphosphoramide per gram of aggregate and shape column to fit into chromatograph.

2. Install the column in the chromatograph and purge with helium (approx. 20 ml/min.) for at least 24 hours before using.

3. With the instrument at room temperature, determine the optimum detector current and set at this value, and adjust the pressure such that the propylene peak appears in approximately 7 to 8 minutes after charging the sample. Allow the instrument to reach equilibrium at these conditions.

B. Analysis

1. Turn recorder on and set recorder range to 1. Recorder trace should show no drift for five minutes before chromatograph is ready for use.

2. Purge 1 cc gas sample loop with sample to be analyzed, shut off valve from bomb, and enter 1 cc sample at one atmosphere pressure into chromatograph.

3. Record all peaks up to and including propylene on recorder attenuation of 1, or allow enough time for propylene peak to appear as determined by retention time from a previous chart run under the same conditions.

CALCULATIONS

1. Draw base line for propylene peak only.

2. Use rule with 50 divisions per inch and measure the peak height of the propylene peak. Measurements should be made to nearest one-tenth of a division.

CALCULATIONS (cont'd)

3. Calculate concentration of propylene as follows:

$$C = \frac{D}{S}$$

Where: C = concentration of propylene in sample expressed in mol per cent.
D = deflection of propylene peak in sample chromatogram measured in step 2 above.
S = sensitivity of propylene, expressed as deflection per mole per cent as determined from the calibration curve.

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare a sensitivity curve for propylene by adding pure propylene at 150, 100, 50, and 25 mm of pressure in the 1 cc sample loop.

2. Plot the peak height (scale of 50 divisions per inch) versus pressure and extrapolate the curve to zero pressure.

3. Calculate the sensitivity in scale divisions (50 divisions per inch) of deflection per mol per cent for a 1 cc sample from the slope of the curve below 20 mm pressure.

4. Before analyzing each set of samples, check the sensitivity by measuring a secondary standard and make suitable corrections to the calibrations.

SAFETY PRECAUTIONS

No special precautions need be observed other than those normally observed when handling gaseous hydrocarbon samples.

ANALYTICAL TIME

The average time of analysis using this method should be 0.3 hour.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits using this method should be + or - 0.0003 mol per cent in the 0-0.025 mol per cent range. Report results to nearest .001 mol per cent.

SCOPE

This method is for the determination of propylene and other olefins in high purity ethylene. It may be used on other gaseous hydrocarbon mixtures with possible expansion of calibration curves.

/jw
7/21/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPTS. 2,3,20, 33,60
METHOD 602.063 (WC)
July 21, 1971

MATERIAL

NaOH receipts, Circulating Caustic, Waste Caustic

ANALYSIS REQUIRED

NaOH content

SAMPLE SIZE

Pint bottle

APPARATUS

50 ml Burette
Oleum bulbs
300 ml Erlenmeyer flask

REAGENTS

Methyl red indicator
0.5 N HCl

PROCEDURE

Place about 1 gram of sample into a tared oleum bulb. Seal and reweigh bulb. Slide the oleum bulb into a 300 ml Erlenmeyer flask containing about 25 ml of distilled water. Cautiously, break the oleum bulb with a stirring rod which has been flattened on one end. Be sure that oleum bulb stem is also crushed with stirring rod. Add about 25 ml more of distilled water. Add a few drops of Methyl red indicator, swirl and titrate to a red end point with 0.5 N HCl. Analyze receipts (only) in duplicate. Single analysis for other caustic samples.

CALCULATIONS

$$\frac{(Vol_{HCl})\ (N_{HCl})\ (4.00)}{\text{Sample Weight}} = \%\ NaOH$$

On caustic receipts also report % Na_2O
(% NaOH)
(Average of) (1.0195 = % Na_2O @ 76%)
(Duplicates)

RSV 0014978

PREPARATION OF REAGENTS

Refer to solutions manual

STANDARDIZATION AND CALIBRATION

Refer to solutions manual for standardization of 0.5 N HCl.

SAFETY PRECAUTIONS

Caustic receipts are-approximately 50% NaOH. Caution should be used to prevent contact with skin, eyes or clothing. If 50% NaOH does come in contact with the body, wash affected part with copious quantities of water.

ANALYTICAL TIME

0.3 Hours

PRECISION AND SIGNIFICANT FIGURES

Report to the nearest .01%. Duplicate results should agree within 0.5%. Precision is $\leq$ 0.4% at 50% caustic level.

SCOPE

Method is applicable to any range of caustic by varying sample size and normality of HCl.

/jw
7/21/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPTS. 2,3,10,20,
33,60
METHOD 602.212 (WC)
7/21/71

MATERIAL

Specification Acrylonitrile and Product Methanol
Muriatic Acid - Caustic Receipts

ANALYSIS REQUIRED

Appearance

SAMPLE

Quart Bottle

APPARATUS

APHA Color Tube

REAGENTS

None

PROCEDURE

Observe the sample in a clean dry tube of size large enough to hold about 100 ml of sample. Sample should be clear and free of suspended matter. Report results accordingly.

CALCULATIONS

None

STANDARDIZATION AND/OR CALIBRATION

None

SAFETY PRECAUTIONS

Avoid skin contact with AN, Caustic, HCl, MeOH.
Avoid breathing AN and MeOH, HCl, Muriatic Acid and caustic soda vapors.
Keep out of eyes.

ANALYTICAL TIME

0.1 hour

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

Report appearance as clear and free of suspended matter, cloudy or suspended matter present.

SCOPE

This method is capable of detecting any foreign matter of large enough particle size to be observed visually.

REFERENCES

Method 20-29-3, MLO, 7-21-51, 10-22-51

/jw
7/21/71

MATERIAL

Acrylamide

ANALYSIS REQUIRED

Melting range

APPARATUS

(a) Bath - JFQ Standard Apparatus Drawing 5

(b) Bath Liquid - Use appropriate liquid as follows:

Bath Temperature	Liquid
40-85°C	Water
80-200°C	Dow Corning No. 550 Silicone Fluid
190-270°C	Dow Corning No. 550 Silicone Fluid

(c) Thermometer - Precision Thermometer and Instrument Company, Philadelphia, Pa., round front precision type thermometer, or equivalent, calibrated against a standard or platinum resistance thermometer, selected as follows:

Bath Temperature	
40-85°C	40-85°C range, 3" immersion 0.2°C division, 12" length
80-200°C	80-200°C range, 3" immersion 0.2°C division, 18" length
190-270°C	190-270°C range, 4" immersion 0.2°C division, 20" length

APPARATUS (cont'd)

(d) Magnifier - Surth Number UG-49-D without base, Safety Incorporated, St. Louis, Missouri, type magnifier containing a 2" x 4" lens with a 3" focal length.

(e) Melting Point Tubing - Friedrich and Dimmack, Inc., P.O. Box 230, Millville, New Jersey.

7" length, 1.4-1.6 mm OD purchased with both ends sealed. Score with a chip of carborundum and break off one end just prior to use. Since it is impractical to fire-polish the broken end, exercise CARE to prevent lacerations.

(f) Continuously Adjustable Transformer, such as No. V-5 Variac, General Radio Co.

(g) Variable Speed Stirrer, such as Eastern Industries, Model No. 3, New Haven, Conn.

DETERMINATION

1. Reduce a portion of the sample to a very fine powder if necessary, using a mortar and pestle. When specified, dry the sample at the time and temperature designated in the specification for the product. If the determination is to be made on the sample as is, transfer a portion to a clean piece of glassine paper for filling the melting point tube.
2. By tamping the open end in a small mound of sample, charge the tube with sufficient material to form a column in the bottom of the tube from 4.0 to 5.0 mm high when packed down as closely as possible by moderate reverse tapping on a solid surface. The filling of the tube can be aided by carefully scratching the side of the tube with a round file during the process. The remainder of the sample in the mortar or on the glassine paper should be discarded since it may contain slivers of glass.

DETERMINATION (cont'd)

3. Select the appropriate bath and make certain that it is filled to the proper height. The liquid should be filled so that it is within 1/2" of the top of the bath when at the melting point temperature. Adjust the thermometer to the proper immersion point, making certain that the thermometer is as close to the center of the bath as possible.

4. Start the stirrer and heat the bath rapidly to within approximately 15°C of the expected melting point. Regulate the rise with a Variac to 1°C per minute ± 10 seconds and maintain at this rate throughout the remainder of the test.

5. Insert the melting point tube exactly 10°C below the expected melting point so that the sample portion of the tube is adjacent to the bulb of the thermometer and clearly visible through the bath window.

6. Read the temperature of the melt at the following two points:

   a. Meniscus Point - When the liquid phase of the sample forms a distinct meniscus at any level within the sample.

   b. Clear Point (End of Melt) - When the last evidence of solid disappears and the sample is completely liquid.

7. Apply the thermometer calibration correction and report to 0.1°C the corrected temperatures as the melting point range.

DISCUSSION

The material must be in a fine powdered state to facilitate proper packing of the melting point tube. Granular or crystalline material will allow air voids, thus affecting the amount of material used. The volume of material is critical since amounts varying from the recommended 4.0-5.0 mm can affect the malting range by 0.2°C or more.

DISCUSSION (cont'd)

The position of the melting point tube and the thermometer are critical. They should be placed adjacent to each other and centered in the bath to minimize variations.

It is essential that the proper liquid level be maintained in order to facilitate efficient stirring of the bath. To counteract the greater convection currents in the highest temperature bath, this bath has been made 1" deeper than the other baths. The same liquid level should be maintained, namely 1/2" from the top.

Table 1 lists the significant differences in procedure between this method, the USP XVI, and the BP (1948) methods.

TABLE 1

COMPARISON OF THE MONSANTO MELTING POINT METHOD, THE USP XVI, AND THE BP (1948) METHODS (1)

Method	Visual Aid	Rate of Rise in Temp., °C per Minute	Alteration in Rate of Temp. Rise	Immersion Point in °C Below Expected Melt	Preheating Time From Immersion to Expected Melt in Minutes
Monsanto	Magnifier	1°	None	10	10
USP XVI	None	3°C	Change rate to 1°C per minute at 3° below the expected melt	30	12
BP (1948)	None	3°	None	10	3-1/3

DISCUSSION (cont'd)

Following are the five possible observable points during the transition from solid to liquid with notations of the points taken in each of the above three methods:

1. Sintering Point - The sample shows evidence of shrinking in volume and its appearance is altered.

2. Liquefaction Point - The sample shows pronounced shrinkage in volume, and the first distinct minute droplet of liquid forms at any level in the sample or on the wall of the capillary. This point closely approximates, or is identical with, the "Beginning of Melting" of the USP XVI method.

3. Flow Point - The sample sorms droplets which readily begin to flow together and droplets adhering to the wall of the capillary begin tc spread and flow. Shrinkage of the sample is virtually complete.

4. Meniscus Point - The liquid phase of the sample forms a distince meniscus at any level within the sample. This point is identical with the "Meniscus Point" of this method and the BP (1948).

5. Clear Point (End of Melt) - The point where the last evidence of solid disappears and the sample is completely liquid. This point is identical with the "End of Melt" of this method and the USP XVI.

SCOPE

This method is intended for the determination of the melting point (range) of essentially pure organic compounds. It involves a capillary tube technique similar to a method proposed by Felker (1) for adoption as a standard procedure by the United States Pharmacopeia.

PRECISION AND ACCURACY

Results by this method, compared to those by the USP XVI and the BP (1948) methods will differ product for product since the transition from the solid to liquid phase varies as the complexity of the molecular structure varies. Products such as caffeine and acetophenetidin will respond as follows:

1. The meniscus point will be approximately 0.4°C higher than the USP XVI liquefaction point and 0.2°C lower than the BP (1948) meniscus point, owing primarily to the different rates of heating.

2. The end of melt will be essentially the same in this method and the USP XVI.

The precision of this method is dependent on the purity of the material being tested. Duplicate results on essentially pure material should agree within 0.3°C.

SAFETY

Mercury is a cumulative poison with a relatively high vapor pressure. In the event that a thermometer is broken, collect the mercury and place in a waste jar under water.

REFERENCES

Felker, W. S., Drug Standards. 20, 169 (1952).

/jw
7/22/71

MATERIAL

Acrylamide

ANALYSIS REQUIRED

Solution appearance.

SCOPE

This method is intended to provide an indication of the completeness of solution of materials at specified concentrations in specific solvents. It involves comparison with an equivalent solution of an approved standard, or subjective description of the solution when no standard is available.

EQUIPMENT

a) Clark Turbidimeter, Catalog No. T-11315, The Chemical Rubber Co., Cleveland, Ohio. (Lamp replaced with a 150 watt showcase lamp.)

b) Appropriate comparison glassware such as test tubes, Nessler tubes, flasks, or bottles, matched as to volume and tint of glass; thoroughly cleaned, rinsed with distilled water, and dried.

c) Solvent as designated in the specification for the product being tested. The solvent must be clear and colorless and should be filtered or distilled if necessary.

DETERMINATION

By Comparison with Standards

1. Read the specification for the product and, using a balance of 0.1 g. sensitivity, weigh the designated amount of sample and transfer to the appropriate glassware.

DETERMINATION (cont'd)

2. Add the specified volume of clear, colorless solvent and dissolve the sample by agitation at room temperature, or at the temperature designated in the specification.

3. Compare the color of the sample solution with the prescribed standard for color, referring to Method 38-D, E if an APHA color is designated, and noting any abnormality in hue.

4. Compare the turbidity of the sample solution under the turbidimeter in parallel with the prescribed standard, recording the appropriate degree of comparison as: better, slightly better, very slightly better, equivalent, very slightly more turbid, slightly more turbid, or more turbid, and note any difference in particle size, type or opalescence.

   NOTE: When product minimum standards selected by production departments are designated, solutions of them are prepared in parallel with the sample, observing the same precautions.

By Description

1. Prepare the specified sample solution as directed in Method 112-A.

2. Describe the color of the solution.

3. Observe the solution under the turbidimeter and describe the degree of turbidity, opalescence, particles or sediment.

SAFETY

Take cognizance of any potential hazards in the solvents and samples, such as flammability, corrosiveness, lachrymatory or dermatological properties, or reactivity, and exercise adequate precautions.

/jw
7/22/71

MATERIAL

Acrylamide

ANALYSIS REQUIRED

Moisture

SCOPE

This method is intended as a general directive for the determination of water, titratable with Karl Fischer reagent, in various organic and inorganic substances. It involves the solubilization or dispersion of a sample of the material to be analyzed in a suitable solvent and the titrimetric determination of the moisture present with Karl Fischer reagent. The end point may be detected colorimetrically in colorless or nearly colorless solutions, or electrometrically in colored solutions.

EQUIPMENT

a) N/100 methanolic iodine - Dilute 10 ml. of N/10 iodine to 100 ml. with methanol.

b) Standardized regular and low alcohol Karl Fischer reagents in automatic burettes protected with anhydrous silica gel traps. For details of preparation see Method 1 - Section VII.

c) Auto-Aquatrator, Catalog No. S-29742, Precision Scientific Co., Chicago, Ill.

d) Common laboratory apparatus and reagents.

APPLICATION

Solids are measured by weight. Liquids of known gravity may be measured from an appropriate volumetric pipette, multiplying volume by specific gravity to obtain weight.

Expected Moisture	Sample Size
0-0.5%	10 ± 0.1 g.
0.5-1.0%	5 ± 0.1 g.
1.0-5.0%	2 ± 0.01g.
5.0-12.0%	1 ± 0.01g.

DETERMINATION

A-Electrometric

1. Turn the line switch ON.
2. Set the selector to "DIRECT ADJUST".
3. Adjust the meter to full scale with the "DIRECT ADJUST" knob.
4. Set the selector to "DIRECT TITRATION".
5. Set the meter Low Set Point (left red needle) to 8 microamperes. (Where the moisture level is 0.05% or less, low set point must be set lower.)
6. Set the meter High Set Point (right red needle) to 15 microamperes.
7. Set the "End Point Hold" to 30 seconds.
8. Fill the K.F. burette.
9. To the titration vessel add a sufficient quantity of the solvent designated on the product specification sheet, to cover the electrode tip.
10. Place the titration vessel under the electrode, swing the support under the vessel and tighten the support until the vessel is snug against the cover. (The system must be air-tight.) Put the cover stopper in place.

DETERMINATION (cont'd)

11. Start the stirrer (stir slowly), apply pressure to the cover stopper and evacuate moist air from the system by pulling on the vacuum switch for 15 seconds.

12. Depress the "Start" button and allow the unit to titrate to the instrument end point.

13. While the solvent is being titrated, weigh-in a suitable container - an appropriate amount of the sample for moisture determination.

14. When the instrument indicates the end of the solvent titration, turn off the stirrer and remove the cover stopper.

15. Carefully add the sample to the titration vessel.

16. Replace the stopper, turn on the stirrer (stir slowly) and evacuate the system for 15 seconds.

17. Fill the K.F. burette, depress the start button, and while the unit is titrating to the instrument endpoint; reweigh the sample container to determine the weight of the sample used.

18. Read the burette and calculate.

$$\% \text{ Moisture} = \frac{\text{ml. titre} \times \text{K.F. factor} \times 100}{\text{sample weight}}$$

B-Visual

1. Measure about 25 ml. of the solvent designated in the specification for the product (or see Discussion) into a dry 125 ml. Erlenmeyer flask.

2. Titre with Karl Fischer reagent to an end point that matches the color of the N/100 methanolic iodine solution, sweeping moisture out of the flask by swirling the solution up the walls of the flask.

DETERMINATION (cont'd)

3. Zero the Karl Fischer burette.

4. Add the prescribed weight of sample to the blanked solvent, swirl to dissolve, and immediately titrate to the same endpoint.

   NOTE: If the material to be analyzed is an insoluble porous solid, disperse finely divided sample in the solvent and allow to stand in a stoppered flask with occasional shaking for periods up to one hour at room temperature before titrating with Karl Fischer reagent.

5. Calculate:

$$\% \text{ Moisture} = \frac{\text{ml. titre} \times \text{K.F. factor} \times 100}{\text{sample weight}}$$

   NOTE: In cases where pre-treatment of the sample is necessary to eliminate interaction of the material to be analyzed and Karl Fischer reagent, a separate solvent or reagent blank may be necessary. The calculation then becomes:

$$\% \text{ Moisture} = \frac{(\text{Sample ml.} - \text{Blank ml.}) \times \text{K.F. factor} \times 100}{\text{sample weight}}$$

PRECISION AND ACCURACY

a) Sensitivity, precision, and accuracy depend on several factors, for example, concentration of the Karl Fischer reagent, titration technique, apparatus, quantity of water titrated, and nature of material being analyzed.

b) The sensitivity is about 0.1 mg of water for visual titrations. Less than 0.02 mg can be measured by electrometric titration.

PRECISION AND ACCURACY (cont'd)

c) The following is an example of the precision attained in an interlaboratory study on two samples of acetone containing 0.1 percent and 0.4 percent water and two samples of methyl ethyl ketone containing 0.05 percent and 0.17 percent water:

Repeatability - Two results (each the average of duplicate determinations) obtained by the same analyst should be considered suspect if they differ by more than 0.013 percent, absolute (95 percent confidence level). Duplicate determinations which agree within 0.008 percent are acceptable for averaging.

Reproducibility - Two results (each the average of duplicate determinations) obtained by analysts in different laboratories should be considered suspect if they differ by more than 0.028 percent, absolute (95 percent confidence level).

NOTE: The interlaboratory study was carried out by ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Product, Subcommittee V on Solvents, Plasticizers, and Chemical Intermediates. Sevel laboratories participated with a single analyst performing duplicate determinations on each of two days, using two methods on the four samples described above. The Method of Test for Water in Lacquer Solvent and Diluents (Fischer Reagent Titration Method) (ASTM Designation: D1364)[5] was the subject of the test program being compared with each laboratory's own version of a Karl Fischer method. As neither the means nor the variances of the two sets of data proved significantly different, all of the results were pooled to give estimates of the repeatability based on 55 degrees of freedom and reproducibility based on 47 degrees of freedom.

DISCUSSION

The stoichiometry of the reaction between water and Karl Fischer reagent is as follows:

$$C_5H_5N.I_2 + C_5H_5N.SO_2 + C_5H_5N + H_2O \longrightarrow 2C_5H_5N.HI + C_5H_5N\begin{matrix}SO_2\\O\end{matrix}$$

and

$$C_5H_5N\begin{matrix}SO_2\\O\end{matrix} + CH_3OH \qquad C_6H_5N\begin{matrix}H\\SO_4CH_3\end{matrix}$$

Pyridine is used to combine with acidic products, which will cause a reversal in the following reaction:

$$I_2 + SO_2 + 2H_2O \qquad 2HI + H_2SO_4$$

It has the added advantage of combining with the sulfur dioxide, thereby reducing the vapor pressure of the latter.

Most substances are inert to Karl Fischer reagent and may be analyzed without special treatment. However, a number of compounds and classes of compounds react stoichiometrically with one or more of the components of Fischer reagent. Included among these are: - Ascorbic Acid, Hydrazine Salts, Substituted Hydrazine Salts, Mercaptans, Alkali Becarbonates, Alkali Carbonates, Alkali Sulfites, Alkali Pyrosulfites, Boric Acid and Oxides, Carbonyl Compounds, Cupric Salts, Ferric Salts, Metal Hydroxides, Metal Oxides, Sodium Arsenate, Sodium Arsenite, Sodium Tetraborate, Sodium Thiosulfate, and Stannous Chloride.

In general these reactive compounds either may be rendered inert (the use of glacial acetic acid as a solvent eliminates amine and hydrzaine interference) or may be estimated by an independent method and a stoichiometric correction applied to the water determination.

DISCUSSION

Although the cyanohydrin technique is the only general method for the determination of water in the presence of carbonyl compounds, low alcohol Karl Fischer reagent and/or special solvents may be used to inhibit interfering side reactions of many ketones and some aldehydes. By greatly reducing the methanol and increasing the pyridine in the regular Fischer solution, the tendency of carbonyls to form acetals and ketals by reacting with the methanol of the standard reagent is reduced. Appreciable quantities of lower alcohols in the sample will permit the interfering acetal of ketal reaction even with the use of modified reagent.

Details of other method variations are beyond the scope of this method and will be given in the methods of analysis for the specific compounds.

In general, methanol may be used as a solvent for the determination of water in inert materials. Amines stronger than benzyl amine ($K_B = 2.4 \times 10^{-5}$) and other basic compounds are preferably treated with or dissolved in an excess of glacial acetic acid or spent Fischer reagent, which in both cases combines with the amines. Some organic acids, formic, acetic, adipic, etc., tend to give slightly high results when methanol is used as a diluent, because of the ease with which they esterify. When equal parts of dry methanol and pyridine are used as a diluent, the inhibiting influence of the amine is sufficient to discourage any esterification of the acid at room temperature.

SAFETY

No unusual hazards are involved in this operation when cognizance of the inflammability and toxicity of the reagents and solvents is taken.

REFERENCE

1. "Aquametry", Mitchell and Smith, Interscience Publishers, N.Y., 1948.

2. ASTM E203-64, 31 pp. 567-578, 1965.

7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.247
May 31, 1971

MATERIAL

Acrylamide, Sodium lauryl sulfate

ANALYSIS REQUIRED

Sulfate (as $SO_4^{=}$)

SCOPE

This method is intended for the determination of soluble sulfates in concentrations around 0.1% in essentially water soluble materials. It involves gravimetric determination as barium sulfate.

EQUIPMENT

a) Usual laboratory apparatus and reagents.

b) 20 ml. fine porosity Selas filtering crucible, Catalog No. 8-227, Fisher Scientific Co.

DETERMINATION

1. Dissolve 10 grams of sample, weighed on a prescription balance, in 100ml. of water in a 600ml. beaker.

2. Neutralize the solution to methyl orange with 10% HCl and add 4 ml. in excess.

   NOTE: If the solution is not completely clear at this point, filter by gravity through Whitman 41 paper into a 600 ml. beaker.

3. Dilute the solution to about 400 ml. and heat to boiling.

4. While boiling gently, add 25 ml. and heat to boiling.

5. Cover the beaker with a watch glass and digest the solution on a steam bath for one hour, or until the supernatent liquid is clear.

DETERMINATION (cont'd)

6. Filter through an ignited and tared fine porosity Selas crucible, transferring the precipitate quantitatively with a stream of water from a wash bottle, policing the beaker thoroughly, and washing down the inside walls of the crucible.

7. Ignite the crucible in a muffle furnace at 800-900°C for 20 minutes, cool in a desiccator for 30 minutes and accurately weigh.

8. Calculate the sulfate content as the specific sulfate designated in the specification for the product:

$$\% \text{ Specified Sulfate} = \frac{\text{net wt. of ppt. x Factor}}{\text{sample weight}}$$

Table of Sulfate Factors

Specified Sulfates	$BaSO_4$ Factor
$CaSO_4$	58.3
$Fe_2(SO_4)_3$	57.1
H_2SO_4	42.0
K_2SO_4	74.7
$MgSO_4$	51.6
$MnSO_4$	64.7
Na_2SO_4	60.9

PRECISION AND ACCURACY

Duplicate determinations should agree to within 0.002%.

SAFETY

Exercise due care in the use of the high temperature muffle furnace. Handle crucible with long muffle tongs, preferably held with an asbestos glove.

/jw
7/22/71

MATERIAL

Ammonia Persulfate

ANALYSIS REQUIRED

Active Oxygen

APPARATUS

a) Analytical balance

b) 250 ml Erlenmeyer flash

c) Titrating burette

d) Usual laboratory equipment

REAGENTS

a) 1N H_2SO_4

b) 0.5N ferrous ammonium sulfate solution

c) 0.5N $KMnO_4$

PROCEDURE

The active oxygen content of the persulfates can be conveniently determined by the following method:

Weigh approximately 1 gram of the sample to the nearest milligram and dissolve it in 50 ml of approximately 1 normal H_2SO_4 in an Erlenmeyer flask. Add 40 ml of ferrous ammonium sulfate solution (0.5N). Swirl constantly while adding the iron solution. Let stand for one minute and titrate with 0.5N $KMnO_4$. Run a blank titration on 40 ml of ferrous ammonium sulfate solution, as used above, in 50 ml of the 1 normal H_2SO_4.

CALCULATIONS

$$\text{Percent active oxygen} = \frac{(A-B)C \times 0.8}{D}$$

$$\text{Percent Ammonium Persulfate} = \frac{(A-B)C \times 11.4}{D}$$

A = ml $KmnO_4$ solution used for titrating the blank

B = ml $KmnO_4$ solution used for titrating sample

C = Normality of the $KmnO_4$ solution used

D = Weight of the sample in grams

/jw
7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.249
May 31, 1971

MATERIAL

Ferric Ammonium Sulfate
Sodium Formaldehyde Sulfoxylate
Tetrasodium ethylene diamine tetraacetate

ANALYSIS REQUIRED

Odor

SCOPE

This method is intended for the characterization of the odors of materials by olfactory perception.

DETERMINATION

1. Remove sufficient material to allow about two inches of head room in the sample bottle above the contents to permit adequate trapping of the odor evolved.

2. If the nature of the sample permits, agitate the contents of the bottle.

3. Let the bottle stand closed at least 30 minutes before performing the odor evaluation.

4. If unfamiliar with the odor of the product being tested, carefully open a previously approved sample and cautiously smell the air in the neck of the bottle.

5. Describe the sample odor with respect to the specification requirement.

/jw
7/22/71

MATERIAL

Sodium Lauryl Sulfate
Tetrasodium ethylene diamine tetraacetate

ANALYSIS REQUIRED

pH

SCOPE

This method is intended to be a guide to proper techniques in determining the pH of aqueous solutions by pH meter with the glass electrode. The manufacturer's instruction manual should be consulted for proper operation of the pH meter used.

EQUIPMENT

1. pH meter, such as the Leeds and Northrup pH meter 7664 - Leeds and Northrup Co., 4901 Stention Avenue, Philadelphia 44, Pa., or the Beckman Model H2 pH meter, Scientific Instruments Division, Beckman Instruments, Inc., Fullerton, California.

2. Suitable calomel reference electrode.

3. Suitable glass electrode.

4. Brushless stirring motor such as Fultork Labmotor 14-502, Fisher Scientific Co.

STANDARDIZATION

1. Ascertain that the meter is connected to the proper line voltage, the electrodes are properly plugged into the proper electrode connections, and that the meter is set for reading pH.

STANDARDIZATION

2. With the meter in a "Zero" or "Neutral" position, remove the water bath in which the electrodes are immersed and check visually to see that they are clean and unbroken and that the salt bridge is filled. Oils, etc. that adhere to the electrodes may be removed with a suitable solvent and the electrodes rinsed with water.

3. Immerse the electrodes in a suitable standard buffer, start the stirrer and check the temperature of the buffer. Adjust the temperature compensator on the meter to this temperature.

4. Switch the meter from "Neutral" to "Read" position and adjust the standardization control so that the meter reads the exact pH of the buffer solution.

5. After two minutes, read the meter again. If it has drifted from the original setting, adjust the meter to the buffer pH value with fresh buffer solution and once more allow two minutes to check for drift. Continue until a stable reading is obtained.

6. Switch the meter from "Read" to "Neutral" position and turn off the stirrer. Remove the buffer solution from the electrodes, rinse them with water, and proceed to measurement of the sample solutions.

pH MEASUREMENT

1. After standardization of the meter, rinse the electrodes with a portion of the sample solution and immerse the electrodes in a fresh portion of the sample solution.

2. Turn on the stirrer and allow to stir several minutes; check the sample temperature and adjust the temperature compensator if necessary. Then switch the meter from "Neutral" to "Read"position.

3. Observe the pH reading. Wait two minutes and read again. If the second reading does not vary more than $\pm$ 0.05 pH units from the first reading, the pH value is acceptable.

pH MEASUREMENT (cont'd)

4. If the second reading varies more than ± 0.05 ph units from the first reading, shut off the stirrer, switch the meter to "Neutral" and repeat steps 2 and 3 with a fresh portion of the sample solution.

5. Continue as in steps 2, 3 and 4 until a reading is obtained that does not vary more than ± 0.05 pH units within two minutes.

6. Turn off stirrer, switch meter to "Neutral" position, remove sample solution from electrodes and clean them with rinses of water or a solvent that will effectively clean them.

7. Leave the electrodes immersed in distilled water.

DISCUSSION

The meter should be standardized with a buffer, the pH of which is within two pH units of the pH of the sample solution to be measured. Glass electrodes will occasionally lose response, so it is good practice to check the pH reading of a second buffer solution, bracketing the anticipated pH of the sample solution. If the buffers are known to be accurate, and the meter does not accurately relate them, then either the meter or the electrode system is not functioning properly, and an accurate pH value cannot be obtained.

It should be noted that pH meters are often used with non-aqueous systems that remove water from the bridge in the calomel reference cell making a high resistance junction with resultant change in reference potential and instability. The pH response of the glass electrode can also be impaired by dehydration by non-aqueous solutions. The electrodes may be restored by soaking in distilled water.

SAFETY

Line-operated pH meters should be grounded.

REFERENCES

1. Willard, Merritt, and Dean, "Instrumental Methods of Analysis", 3rd. ed., 1958. D. Van Nostrand Co., New York, N.Y.

2. Manov, G. G., and Acree, S. F., "The pH of Some Standard Buffer Solutions from 0 to 60°C, and the Calibration of Glass Electrode pH Meters", ASTM Bulletin No. 137, Dec. 1945.

3. Pharmacopeia of the United States XV, "Potentiometric Determination of pH", 1. 933.

/jw
7/21/71

MATERIAL

Reactor Overhead Gases

ANALYSIS REQUIRED

Vinyl chloride

APPARATUS

Perkin-Elmer Vapor Fractometer, Model 154B or equivalent
Gas charging valve with 5 cc sample loop
1/4" O.D. stainless steel tubing

PROCEDURE

A. Column Preparation

1. Pack 16 ft of 1/4" stainless tubing with 33% dimethyl sulfolane on 45-60 mesh chromosorb.

2. Install column in the Fractometer. Adjust the temperature to 30°C and helium flow to 50 ml/min. Determine the optimum detector current and set at this value. Allow instrument to reach equilibrium at these conditions.

B. Analysis

1. Turn recorder on and set recorder range to 1. Recorder trace should show no drift for five minutes before Fractometer is ready for use.

2. Purge 5 cc sample loop with sample to be analyzed; shut off valve from sample container, and enter 5cc sample into Fractometer.

3. Record peaks for ethylene and vinyl chloride.

CALCULATIONS

1. Draw base lines for both peaks.

2. Use rule with 50 divisions per inch and measure peak height and width at one-half peak height for all peaks. Measurements should be made to the nearest one-tenth of a division.

3. Calculate areas for both peaks:

   $A = H \times w$

4. Calculate mole percent VCM:

   $$\text{mol \%} = \frac{\text{Area VCM}}{\text{Area VCM + Area Ethylene}}$$

/jw
7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.252
July 13, 1971

MATERIAL

Caustic

ANALYSIS REQUIRED

Assay

APPARATUS

a) Hydrometers capable of reading gravity to 0.001 ranging from 1.010 to 1.525.

b) Constant temperature both @ 20°C.

DETERMINATION

a) Cool 250 ml of sample to 1-2°C below the specified temperature and fill a hydrometer jar to within 2-3 inches of the top.

b) Place the specified hydrometer in the liquid and quickly adjust the sample temperature to the specified temperature.

c) Allow the hydrometer to float to rest.

d) Read the bottom of the meniscus and record.

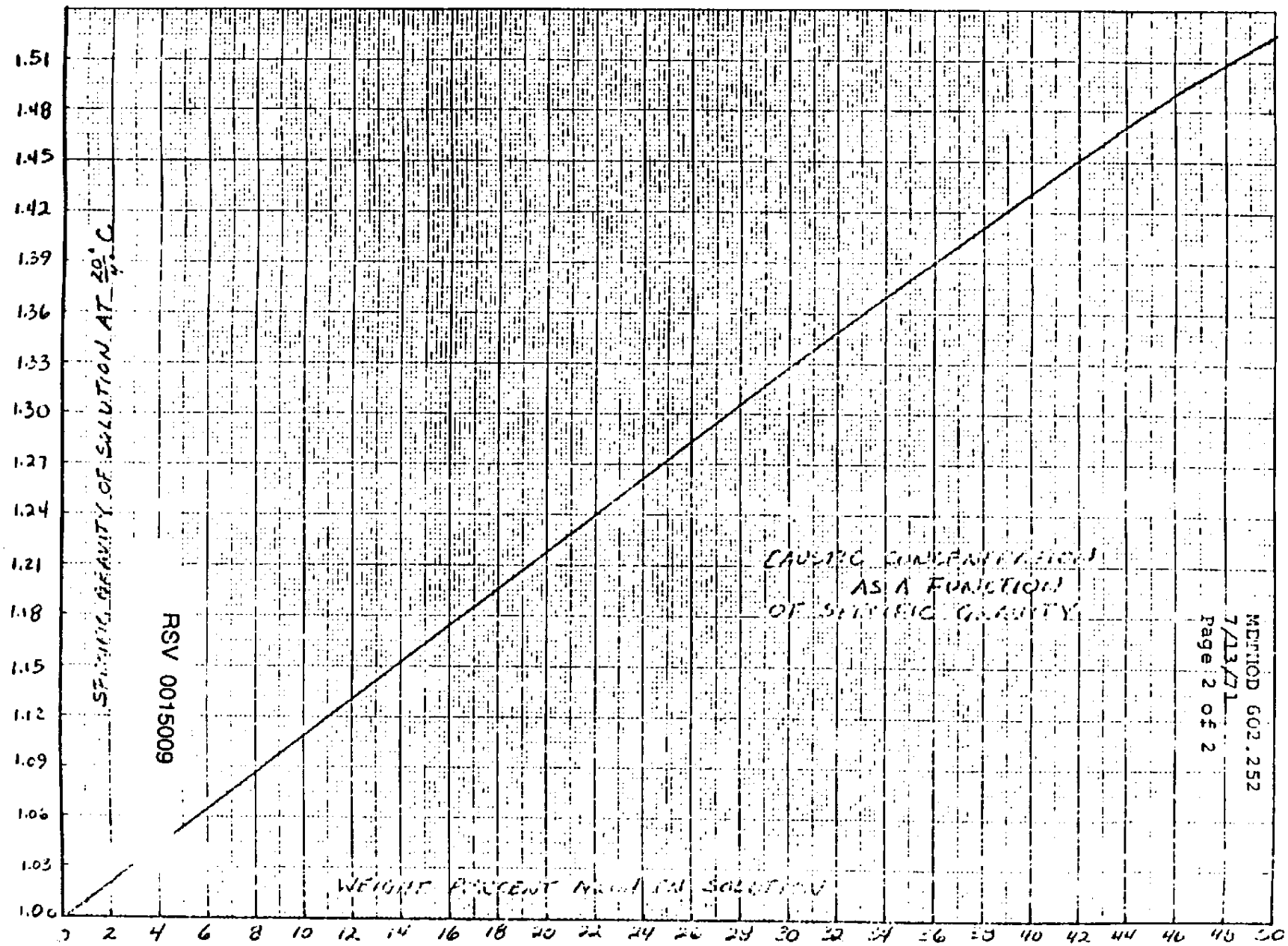
CALCULATION

Convert specific gravity to percent NaOH by following graph.

/jw
7/22/71

K&E 10 X 10 TO THE CENTIMETER 46 1512
KEUFFEL & ESSER CO.

METHOD 602.252



MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.253
July 13, 1971

MATERIAL

Acrylamide - Soap Solution

ANALYSIS REQUIRED

% Acrylamide

APPARATUS

a) Erlenmeyer flask, 250 ml heavy wall, fitted with a 24/40 Standard Taper joint (Ace Glass Co., No. 2671).

b) Filling funnel, 50 ml cylindrical, open top with Standard Taper stopcock and 24/40 Standard Taper joint on bottom (Kontes Glass Co., No. K-63125). In the top of the funnel insert a rubber stopper fitted with a short glass tube for connection to vacuum line.

REAGENTS

a) 0.1 N $KBrO_3$ - KBr solution (2.79 g potassium bromate and 10 g of potassium bromide per liter)

b) 6 N H_2SO_4

c) 20% KI solution

d) Std. 0.1 N $Na_2S_2O_3$

e) 1% Starch solution

PROCEDURE

Weigh accurately a sample containing 3-4 g Stream #4 of acrylamide into a 500 ml volumetric flask, dilute to volume with water and mix. Pipet a 25.00 ml aliquot of this solution and 10 ml of bromate-bromide reagent into the 250 ml iodine. Draw 5 ml of 6N sulfuric acid into the flask and seal with water. Allow the solution to stand in the dark for 20 min. and shake frequently. Add 15 ml of potassium iodide solution, mix and then break the vacuum. Titrate the solution immediately with standard thiosulfate using starch indicator. Run a blank determination.

CALCULATION

$$\% \text{ Acrylamide} = \frac{(\text{blank titer} - \text{sample titer})\ (N\ Na_2S_2O_3)\ (3.554)}{\text{Weight of sample in aliquot}}$$

/jw
7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.254
July 13, 1971

MATERIAL

Acrylamide - Soap Solution

ANALYSIS REQUIRED

% Alkyl Sulfate

REAGENTS

p-toluidine hydrochloride solution 6.8%
Formula 30 Alcohol
Carbon tetrachloride (reagent grade)
Meta-cresol purple indicator (0.1% solution)
Phenol red indicator (0.1% solution)
0.1 N NaOH

APPARATUS

150 ml beaker
250 ml volumetric falsk
25 ml pipette
500 ml separatory funnel
Analytical balance

SCOPE

This procedure employes the extraction of the organic sulfates by p-toluidine hydrochloride precipitation.

PROCEDURE

Using an anlytical balance, transfer 25 g on Stream #4 sample to a 150 ml beaker. Dissolve in distilled water and transfer to a 250 ml volumetric flask (a small amount of F-30 Alcohol may be used to dissipate foam) and dilute to 250 ml mark. Mix well and withdraw a 25 ml aliquot portion (equivalent to 0.5 g of sample).

PROCEDURE (cont'd)

Transfer the aliquot portion to a 500 ml separatory funnel. Add 2 drops of phenol red indicator (red=alkaline, yellow=acid). If solution is alkaline, adjust to neutral with .1 N HCl. If solution is acid, adjust to neutral with .1 N NaOH. To neutral solution add 30 ml of Formula 30 Alcohol, 30 ml of 6.8% p-toluidine hydrochloride solution and 30 ml of carbon tetrachloride. Shake mixture for three minutes (occasionally releasing gas from separatory funnel). Allow to stand and separate. Cut lower (carbon tetrachloride) layer into 250 ml containing 50 ml of Formula 30 Alcohol and 3 cc H_2O adjusted to the alkaline (purple) end-point of meta cresol purple. Add 30 ml carbon tetrachloride to contents of separatory funnel and repeat extraction. Allow to stand and separate and again cut lower layer into same receiving flask.

Repeat extraction once more. These combined extractions in the alcohol medium contain all the organic sulfates and are titrated directly with standard 0.1 N NaOH to determine the alkyl sulfate content. The end point is taken as the first change in color from gray to purple that remains for 15 seconds.

CALCULATION

$$\frac{\text{ml NaOH} \times N \times 300 \times 100}{\text{Wt. of sample in aliquot} \times 1000} = \% \text{ Alkyl Sulfate as sodium lauryl sulfate}$$

/jw
7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.255
July 13, 1971

MATERIAL

Product Latex B, BH and E
Spent THF Solvent

ANALYSIS REQUIRED

Percent Total Solids

APPARATUS

Analytical balance capable of weighing to nearest 0.1 mg.
Aluminum weighing dish
Oven
Dessicator
200 mesh sample screen

PROCEDURE

Screen latex sample through 200 mesh screen. Tare duplicate aluminum weighing dishes to 0.1 mg. Transfer 3-4 g of sample to each dish and obtain gross weight to 0.1 mg. Place the dishes in an oven at 130°C for 3 hours. Cool dish in a dessicator and weigh to 0.1 mg. Report % solids. If two samples differ by more than 0.3%, repeat.

CALCULATIONS

$$\% \text{ Solids} = \frac{\text{net weight after drying}}{\text{net weight before drying}} \times 100$$

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MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.256
July 13, 1971

MATERIAL

Product Latex B, BH and E

ANALYSIS REQUIRED

Latex Viscosity

APPARATUS

Brookfield viscometer, Model LVT with #2 spindle or Model RVT with #2 spindle.
400 ml beaker
200 mesh sample screen (Stainless Steel)

PROCEDURE

Screen latex sample through 200 mesh screen. Sample temperature must be 25° + 1°C. Pour 300 ml sample into a 400 ml beaker. Place a #2 spindle in the Brookfield viscometer model LVT and lower spindle into the sample to the level indicated by the indentation in the spindle shaft. Set the RPM selector at 6 and turn on the viscometer. After the spindle has completed 3 revolutions, depress the clutch and record the viscometer reading. While the clutch is still depressed turn the RPM selector to 60, turn on the viscometer, release the clutch and allow the spindle to complete 10 revolutions. Depress the clutch and record the viscometer reading.

CALCULATIONS

Convert the readings to centipoise.

Reading at 6 RPM x 50 = centipoise (LVT)

Reading at 60 RPM x 5 = centipoise (LVT)

Reading at 100 RPM (RVT)

/jw
7/22/71

MATERIAL

Product Latex B, BH, and E

ANALYSIS REQUIRED

pH

APPARATUS

a) pH Meter, such as the Leeds and Northrup pH meter 7664 - Leeds and Northrup Co., 4901 Stention Avenue, Philadelphia 44, Pa., or the Beckman Model H2 pH meter, Scientific Instruments Division, Beckman Instruments, Inc., Fullerton, California.

b) Suitable calomel reference electrode.

c) Suitable glass electrode.

d) Brushless stirring motor such as Fultork Labmotor 14-502, Fisher Scientific Co.

STANDARDIZATION

a) Ascertain that the meter is connected to the proper line voltage, the electrodes are properly plugged into the proper electrode connections, and that the meter is set for reading pH.

b) With the meter in a "Zero" or "Neutral" position, remove the water bath in which the electrodes are immersed and check visually to see that they are clean and unbroken and that the salt bridge is filled. Oils, etc. that adhere to the electrodes may be removed with a suitable solvent and the electrodes rinsed with water.

c) Immerse the electrodes in a suitable standard buffer, start the stirrer and check the temperature of the buffer. Adjust the temperature compensator on the meter to this temperature.

STANDARDIZATION (cont'd)

d) Switch the meter from "Neutral" to "Read" position and adjust the standardization control so that the meter reads the exact pH of the buffer solution.

e) After two minutes, read the meter again. If it has drifted from the original setting, adjust the meter ot the buffer pH value with fresh buffer solution and once more allow two minutes to check for drift. Continue until a stable reading is obtained.

f) Switch the meter from "Read" to "Neutral" position and turn off the stirrer. Remove the buffer solution from the electrodes, rinse them with water, and proceed to measurement of the sample solutions.

pH MEASUREMENT

a) Screen the latex sample through a 200 mesh stainless steel screen. After standardization of the meter, rinse the electrodes with a portion of the sample solution and immerse the electrodes in a fresh portion of the sample solution.

b) Turn on the stirrer and allow to stir several minutes; check the sample temperature and adjust the temperature compensator if necessary. Then switch the meter from "Neutral" to "Read" position.

c) Observe the pH reading. Wait two minutes and read again. If the second reading does not vary more than $\pm$ 0.05 pH units from the first reading, the pH value is acceptable.

d) If the second reading varies more then $\pm$ 0.05 pH units from the first reading, shut off the stirrer, switch the meter to "Neutral" and repeat steps b and c with a fresh portion of the sample solution.

e) Continue as in steps b, c and d until a reading is obtained that does not vary more than $\pm$ 0.05 pH units within two minutes.

pH MEASUREMENT (cont'd)

f) Turn off stirrer, switch meter to "Neutral" position, remove sample solution from electrodes and clean them with rinses of water or a solvent that will effectively clean them.

g) Leave the electrodes immersed in distilled water.

SCOPE

This method is intended to be a guide to proper techniques in determining the pH of aqueous solutions by pH meter with the glass electrode. The manufacturer's instruction manual should be consulted for proper operation of the pH meter used.

DISCUSSION

The meter should be standardized with a buffer, the pH of which is within two pH units of the pH of the sample solution to be measured. Glass electrodes will occasionally lose response, so it is good practice to check the pH reading of a second buffer solution, bracketing the anticipated pH of the sample solution. If the buffers are known to be accurate, and the meter does not accurately relate them, then either the meter or the electrode system is not functioning properly, and an accurate pH value cannot be obtained.

It should be noted that pH meters are often used with non-aqueous systems that remove water from the bridge in the calomel reference cell making a high resistance junction with resultant change in reference potential and instability. The pH response of the glass electrode can also be impaired by dehydration by non-aqueous solutions. The electrodes may be restored by soaking in distilled water.

SAFETY

Line-operated pH meters should be grounded.

REFERENCES

a) Willard, Merritt, and Dean, "Instrumental Methods of Analysis", 3rd ed., 1958, D. Van Nostrand Co., New York, N.Y.

b) Manov, G. G., and Acree, S. F., "The pH of Some Standard Buffer Solutions from 0 to 60°C, and the Calibration of Glass Electrode pH Meters", ASTM Bulletin No. 137, Dec. 1945.

c) Pharmacopeia of the United States XV, "Potentiometric determination of pH", 1. 933.

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7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.258
July 13, 1971

MATERIAL

Product Latex B, BH, and E

ANALYSIS REQUIRED

% Grit

APPARATUS

325 mesh stainless steel screen
Analytical balance capable of weighing to nearest 0.1 mg
Oven
Evaporating dish

PROCEDURE

Take 500 g of latex and filter through a 325 mesh screen. Wash the latex on the screen with a gentle stream of distilled or deionized water until no further latex can be washed through. Collect the retained grit by washing onto a weighed evaporating dish. Dry the grit at 120°C and determine the weight.

CALCULATIONS

$$\% \text{ grit} = \frac{\text{wt. grit}}{500 \text{ g}} \times 100$$

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7/22/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.259
July 13, 1971

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Surface Tension

APPARATUS

Fisher tensiomat
Shallow sample container
200 mesh stainless steel screen

PROCEDURE

Screen sample through 200 mesh screen.
In making a determination of surface tension, careful preparation of the sample and the Surface Tensiomat must precede the actual manipulation of the instrument.

The sample should be placed in a glass beaker or cylindrical vessel with a diameter of at least 45 millimeters. For testing oil samples according to ASTM Method D-971, the glassware should be cleaned according to a definite procedure. Any residual oil from the previous sample is removed with petroleum naphtha or benzene followed by several washes with methyl ethyl ketone and water, then the glassware is immersed in a hot cleaning solution of chromic acid. The glassware also should be rinsed thoroughly with tap water, then with distilled water. It should be drained in an inverted position over a clean cloth, unless it is to be used immediately.

The platinum-iridium ring should be cleaned by rinsing it in petroleum naphtha or benzene, then by rinsing in methyl ethyl ketone. The ring should then be heated in the oxidizing portion of a gas flame.

PROCEDURE (cont'd)

The preceding instructions apply particularly to preparation for determinations in oil samples. A comparable degree of cleanliness should be maintained for other samples.

Since surface tension is dependent upon temperature, consideration must be given to this factor. For theoretical work, temperatures must be specified; however, 25°C is the temperature most commonly used. For control work, the operator should use the same temperature for each type of measurement.

Since the Surface Tensiomat may be employed as a manual or semi-automatic instrument, separate procedures are described below for both operating modes.

MANUAL DETERMINATIONS

Measuring Surface Tension

The cleaned platinum-iridium ring should first be attached to the hook at the end of the lever arm. The arrest mechanism should be holding the arm at this time.

The liquid to be measured is transferred to the clean glass vessel and placed on the sample table. The sample table is moved around until it is directly beneath the platinum-iridium ring. Raise the sample table until the ring is immersed in the test liquid. The ring should be in the liquid, beneath the surface so that the entire ring will be wetted. About 1/8 inch immersion is generally considered sufficient.

The torsion arm is now released and the instrument adjusted to a zero reading. Adjust the knob on the right side of the case until the index and its image are exactly in line with the reference mark on the mirror. Be careful to keep the ring in the liquid during this manipulation, raising or lowering the sample table if necessary by means of the knob adjustment underneath of the table. Now turn the knob beneath the main dial on the front of the case until the vernier reads zero on the outer scale of the dial.

MANUAL DETERMINATIONS (cont'd)

Lower the sample table until the ring is in the surface of the liquid, while at the same time adjusting the knob on the right side of the case to keep the index lined up with the reference mark on the mirror. The surface of the liquid will become distended but the index must be kept on the reference. Continue the two simultaneous adjustments until the distended film at the surface of the liquid breaks. The scale reading at the breaking point of the distended film is the apparent surface tension.

This reading is the one obtained after the sample has been adjusted for cross hairs mesh. Make only one repeat determination after this adjustment. Repeat determinations without cleaning the loop will give higher and higher readings due to the latex coating the wire.

SEMI-AUTOMATIC DETERMINATIONS

The semi-automatic operation of the FISHER Surface Tensiomat is basically similar to the manual mode of operation - the only essential difference being the use of samll electric motor to twist the torsion wire and thus raise or lower the ring in the test solutions. No adjustments or complicated procedures are involved in changing from eigher mode of operation to the other, the use of a switch to activate the semi-automatic feature and a slightly different technique in regard to the level of the surface or interface are the sole differences. The motor is connected to the semi-automatic mechanism by means of a clutch which does not interfere with the manual mode.

The semi-automatic drive mechanism actuates the torsion arm and consequently the ring; the position of the sample table is not changed or controlled by the drive mechanism. As a consequence, the simultaneous adjustment of torsion arm and sample table is not feasible. Therefore, it is necessary to determine the level of the surface or interface at the instant the film is broken by means of a manual determination and to maintain this level of surface or interface at the instant the film is broken by means of a

SEMI-AUTOMATIC DETERMINATIONS (cont'd)

manual determination and to maintain this level of surface or interface for subsequent determinations of similar samples. Provided the subsequent samples are of the same character (approximately the same density and surface or interfacial tension) no appreciable error is introduced by presetting the level of the surface or interface. Since a major advantage of the Surface Tensiomat is its ability to make repetitive determinations on similar samples rapidly, this determination of a level is a simple and worthwhile operation.

A practical method for attaining a reproducible level for each sample is to use a sample container with a mark showing the level. More than one sample container can be so marked and used in succession. Also, it is usually practical to introduce reproducible volumes of sample into containers of reasonably uniform size. For example, Petri dishes with identical volumes of sample will produce levels which are for all practical purposes the same.

Once the level of the surface has been determined and the approximate reading for the type of sample ascertained, the semi-automatic mode of operation is very simple. The sample is placed on the sample table and the ring immersed in the proper liquid. The knob on the right side of the case is put in the UP position for surface tension measurements or interfacial tension measurements where the ring is pulled up through the interface, and in the DOWN position for interfacial tension determinations where the ring is forced down through the interface. When the surface or interface film ruptures, the level arm actuates a contact (No. 5 of Figure 1) which in turn actuates a relay and stops the drive motor. The value is then read from the proper scale on the dial.

A pilot light on the front of the case indicates when the Surface Tensiomat is running. When the film ruptures, the motor stops and the light goes out. The switch must be returned to the NEUTRAL position and the lever arm returned to the proper position prior to making the next determination.

SEMI-AUTOMATIC DETERMINATIONS (cont'd)

Experience has shown that readings obtained using semi-automatic operation will be slightly different from those obtained manually. However, with any given set of conditions, these variations can be determined by manual and semi-automatic experiments, and if necessary, the variations may then be taken into account by the user.

/jw
7/22/71

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Glass Transition Temperature (Tg)

APPARATUS AND MATERIALS

duPont 900 Differential Thermal Analyzer or its equivalent
Vacuum Pump
Liquid nitrogen and reservoir
Dry nitrogen gas
Bunsen burner

TEST SPECIMEN

The test specimens are prepared by air drying a film of latex on a glass plate.

PROCEDURE

A condensed operating procedure is described here. For detailed operating instructions, refer to duPont 900 DTA instruction manual.

a) Preparation for the Run

1) Cold junction thermocouples should be at room temperature.

2) Fill macro sample tube with the sample to depth of 5 mm. Brush the loaded sample tube through a small Bunsen flame two or three times, and pack the sample tightly by pushing the centering ceramic sleeve to the bottom of the tube. A thermocouple is then placed in the sample so that the junction touches the bottom of the tube. The ceramic sleeve should be positioned over the thermocouple to center it in the macro tube.

PROCEDURE (cont'd)

3) Fill control and reference tubes with quartz powder to depth of 5 mm. Insert thermocouples and centering sleeves in tubes.

4) Wrap glass wool around thermocouple connections at the receptable in cell assembly in order to shield from drafts during cooling.

5) Place tubes in heating block, bend leads so junctions bottom in tubes, and put cell assembly in place.

6) Place chart paper on recorder.

b) Set the Recorder Controls

1) Switch POWER to RECORD (5 minutes warm-up).

2) Set T ZERO SHIFT at +5.

3) Set T SCALE at 10°C/in. (Scale on chart: -50°C to + 50°C)

4) Turn Δ T ZERO SHIFT until pen is near top of chart.

5) Set Δ T SCALE at 0.2°C/in.

6) Set BASELINE SLOPE to 0.

c) Set Sample Atmosphere Controls

1) Connect nitrogen gas supply to PURGE, liquid nitrogen to COOL and vacuum to VACUUM connections.

2) Open VACUUM and COOL valves fully, this introduces liquid nitrogen into the cell.

3) When pen reaches chart reading of -20°C, close COOL valve and adjust the VACUUM gauge to read approximately 15. Set GAS FLOW of purge nitrogen at 2 SCFH.

PROCEDURE (cont'd)

d) Set Temperature Program Controls

1) Push RESET.

2) Set TEMPERATURE RATE at 20°C/min.

3) Turn STARTING TEMPERATURE clockwise until heater voltage indicator reads minimum value. Then turn counterclockwise until voltage indicator just begins to move upscale.

e) Start the Run

1) Set PROGRAM MODE at HEAT.

2) Set POWER at RECORD.

3) The thermogram will not be recorded on the chart until the pen reaches on the right hand edge of the paper. The Δ T SCALE or Δ T ZERO SHIFT controls may be adjusted at any time during the run to keep strong endotherms or exotherms on the chart paper. Similarly, the BASELINE SLOPE control may be adjusted at any time during the run to make the baseline more nearly horizontal.

f) Conclude the Run

1) Turn PROGRAM MODE to HEATER OFF.

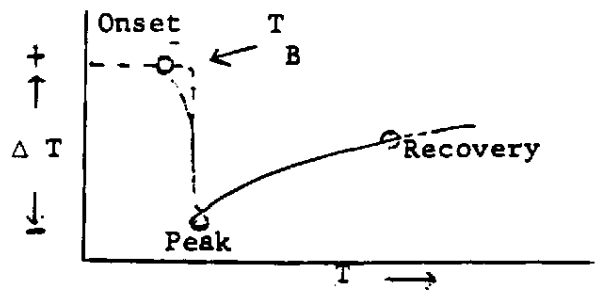
2) Turn POWER to STAND BY.

3) After each run the sample tube may be discarded. The sample thermocouple and the centering ceramic sleeve should be cleaned by burning over a small Bunsen flame to burn all organic compounds, they can be reused for twenty or more runs.

PROCEDURE (cont'd)

g) Definition of Transition Temperature

The literature of DTA contains many definitions for the location of significant transition temperatures. This procedure defines the transition temperature as a temperature of extrapolated onset as shown in the figure.



Unlike first-order thermodynamic transitions, the glass transitions of amorphous polymers may not exhibit a peak but only show a shift in baseline. Similarly, the Tg is defined as the temperature of the extrapolated onset in the thermogram.

h) Report

The report shall include the following:

1) Complete identification of the material tested.

2) Size of sample tube.

3) Reference material.

4) Heating, rate, °C/min.

5) Nitrogen purge rate, SCFH.

6) Temperature scale and shift factor.

7) Δ T of sensitivity, °C/in.

SCOPE

This method describes a test procedure for the determination of Tg of the polymer using a differential thermal analyzer.

SIGNIFICANCE

Differential thermal analysis (DTA) is a technique for studying the thermal behavior of materials as they undergo physical and chemical changes during heating or cooling. The glass transition temperature (Tg) of amorphous polymer is only one of many thermal fingerprints of polymer obtainable with DTA. Determination of Tg, which is one of the most important fundamental properties of polymers, by DTA represents one of the simplest and fastest methods.

/jw
7/23/71

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Intrinsic Viscosity

APPARATUS

1. Viscometers:
   a. Cannon-Ubbelohde Dilution Type, Size 50, uncalibrated
   b. Cannon-Fenske #50
2. Constant temperature water bath, $\pm$ 0.05°C
3. Volumetric pipettes: 10 ml, 20 ml, 100 ml
4. Aluminum weighing dishes
5. Forced air oven
6. Stopwatch
7. 1 Liter volumetric flask
8. Tetrahydrofuran (THF), Fisher T-397)

PROCEDURE

1. Measure the solids content of the latex sample and dilute it to 45% solids.
2. Weigh 2 grams of the 45% solids latex into a 200 ml jar.
3. Add 100 ml of pure THF (Tetrahydrofuran) to the jar, cap tightly, and shake. Heat the sample at 40-50°C for 1 hour with intermittent shaking. The solution may still appear hazy.

PROCEDURE (cont'd)

4. Prepare the dilution solvent by adding 10.9 ml H_2O to a 1000 ml volumetric flask and making up to the mark with pure THF.

5. Obtain tare weights on two aluminum weighing dishes. Pipette 10 ml of the polymer solution into each dish and dry to constant weight at 40-50°C. This will take approximately one hour. Record the weight of residue.

6. Determine the concentration of the polymer solution.

   Wt. residue x 10 equals the concentration expressed as grams per deciliter. The initial concentration should be just below 1 gm/deciliter.

7. With a small size coarse glass filter adapted to a 10 ml pipette, draw up 10 ml of the dilution solvent and deliver to a viscometer. The viscometer water bath should be maintained at 30°C.

8. Record the flux time of the dilution solvent. A viscometer should be chosen which gives a 100-150 sec. flux time for the dilution solvent. Make three determinations and record the average.

9. Replace the solvent in the viscometer with the polymer solution as in No. 7. Determine the flux time at this concentration.

10a. If a dilution type viscometer is used, simply add 10 ml of the dilution solvent to the polymer solution already in the viscometer and determine the flux time for this solution. Calculate the actual concentration of the solution.

   b. Make a second dilution by adding 20 ml of dilution solvent to the viscometer after Step 10a. Determine the flux time for this solution and calculate the exact concentration.

11. If a dilution type viscometer is not available, the two additional solutions can be made up separately by adding 10 ml and 30 ml of dilution solvent to each of two 10 ml samples of the original polymer solution.

PROCEDURE (cont'd)

12. For each of the three solution concentrations, calculate the reduced viscosity according to the following formula:

$$\text{Reduced viscosity} = \frac{\left(\frac{\text{Flux time of solution}}{\text{Flux time of solvent}} - 1\right)}{\text{Actual concentration in gm/deciliter}}$$

13. Plot the reduced viscosity versus the actual concentration. The data should fall on a straight line which is extended to zero concentration.

    The intrinsic viscosity is given by the value of the reduced viscosity at zero concentration and will generally be in the range of 0.6-0.8 deciliters/gm. (See attached Figure.)

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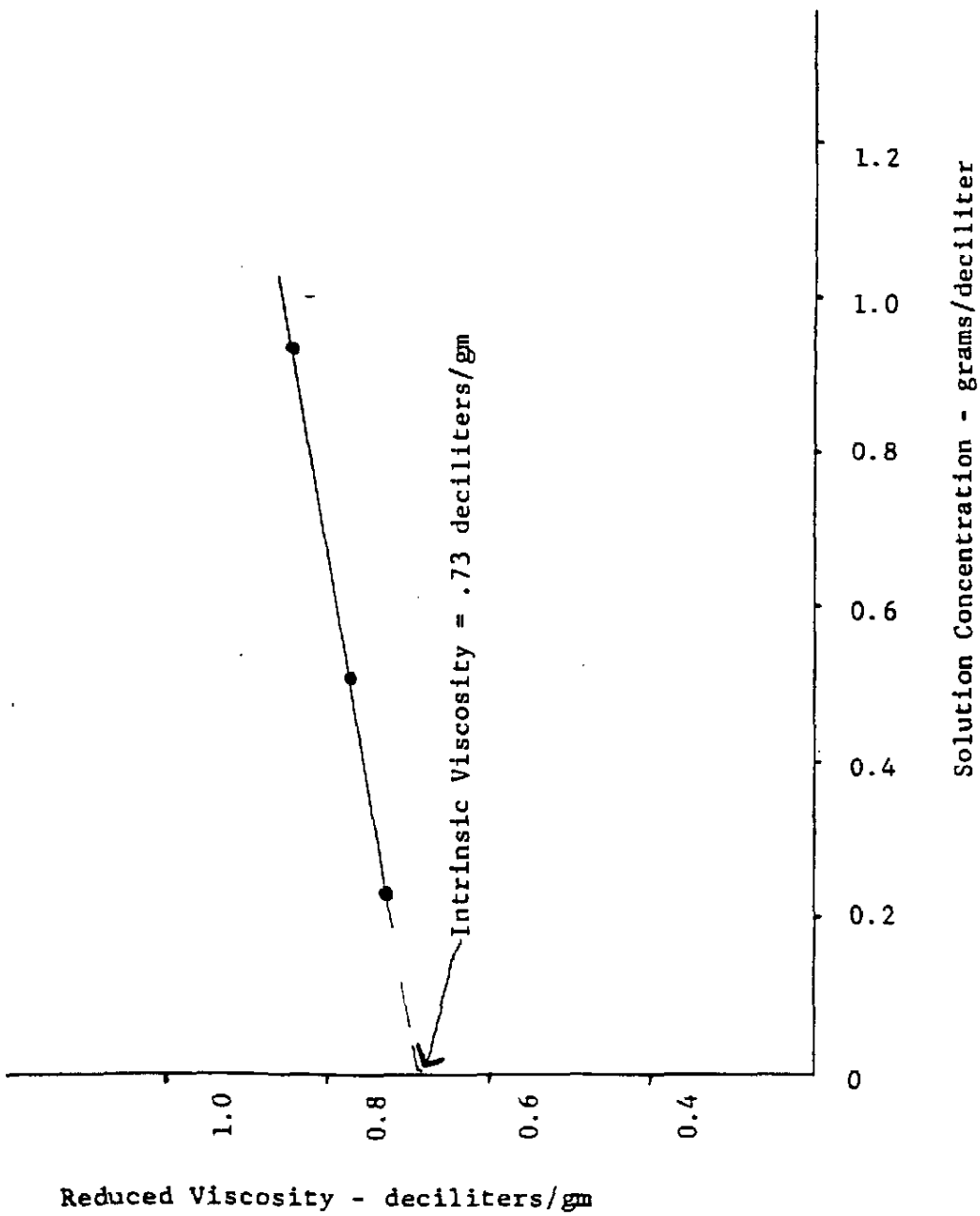
Intrinsic Viscosity = .73 deciliters/gm
1.2
1.0
0.8
0.6
0.4
0.2
0
Solution Concentration - grams/deciliter
1.0
0.8
0.6
0.4
Reduced Viscosity - deciliters/gm

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Particle Size

APPARATUS

Amberlite XAD-2 resin
Analytical balance
Tumbler
Buchner funnel, monofilament nylon filter cloth
Aluminum weighing dish
Fisher Tensiomat

REAGENTS

Sodium lauryl sulfate (sipon WD)

PROCEDURE

A. Wash Amberlite XAD-2 absorbent with distilled water. Remove excess water from washed Amberlite and store Amberlite in closed jar to keep moist.

B. Weigh 150 g of screened (200 mesh) latex (40-50% T.S.) and 300 g wet Amberlite into 16 oz. wide mouth bottle.

C. Cap bottle, shake to mix well, and keep mixed by slow tumbling motion for one hour.

D. Filter out latex from mixture with Buchner funnel and monofilament nylon filter cloth under suction.

E. Determine surface tension of latex. Repeat Steps B-D if surface tension is less than 70 dynes/cm.

PROCEDURE (cont'd)

F. Determine total solids of the Amberlite treated latex stock:

Weigh out 1-2 g of stock in aluminum dish. Add enough water to thin out latex and barely cover dish bottom. Dry in oven at 120°C to constant weight (approx. 1 hour). Calculate % T.S. from dry weight.

G. Prepare two latex samples in 4 oz. bottles with following compositions:

Sample 1:	60 g	Amberlite treated latex stock
	11 g	10% SLS (Sipon WD)
	39 g	Distilled water
	110 g	latex with 1% SLS

Sample 2:	60 g	Amberlite treated latex stock
	50 g	Distilled water
	110 g	latex without SLS

Shake to mix well.

H. Mix Samples 1 and 2 in 1 oz. bottles at various proportions as shown in Column I of Table I. Shake to mix well. The corresponding SLS concentration of each sample is shown in Column II of Table I.

I. Determine surface tension of the 11 samples. A hypothetical set of surface tension values is given in Column III of Table I for illustrative purposes.

J. Plot surface tension vs. SLS concentration of sample in semi-log graph paper as shown in Fig. I. The resulting graph will typically show a break which separates the two rather distinct portions of the graph, i.e., the initial portion where the surface tension decreases rapidly with increasing SLS concentration, and the latter portion where surface tension practically remains at a constant low value.

PROCEDURE (cont'd)

K. Compute the particle size using the following equation:

$$\text{Particle size (Å)} = 0.24 \frac{\text{\% T.S. of latex stock} \times (60/110)}{\text{Titration end point in } \frac{\text{g SLS}}{\text{g latex}}}$$

L. When particle size is much larger than 1000 Å or much smaller than 500 Å, the titration end point will move too close to the low or high end of the covered SLS concentration range, making a precise determination of the end point difficult.

When the end point is too low ($<0.002 \frac{\text{g SLS}}{\text{g latex}}$), repeat the determination using the same procedure except change the composition of Sample I of Step G as follows:

60.0 g	Amberlite treated latex stock
5.5 g	10% SLS
44.5 g	Distilled water
100.0 g	latex with 0.5% SLS

and correct the computed particle size of Step K by a factor of 2.

When the end point is too high ($>0.008 \frac{\text{g SLS}}{\text{g latex}}$), repeat determination. Change composition of Sample I of Step G to:

60 g	Amberlite treated latex stock
22 g	10% SLS
28 g	Distilled water
110 g	latex with 2% SLS

and then correct particle size computed in Step K by a factor of 0.5.

Table I

I	II	III
gr Sample I / gr Sample II	gr SLS / gr latex	Surface tension ≠ (dynes/cm)
0/20	0	71.5
2/18	0.001	64.8
4/14	0.002	56.7
6/14	0.003	49.6
8/12	0.004	43.5
10/10	0.005	38.2
12/8	0.006	34.5
14/6	0.007	33.3
16/4	0.008	33.1
18/2	0.009	33.1
20/0	0.010	33.0

≠ Hypothetical set of values for illustrative purposes.

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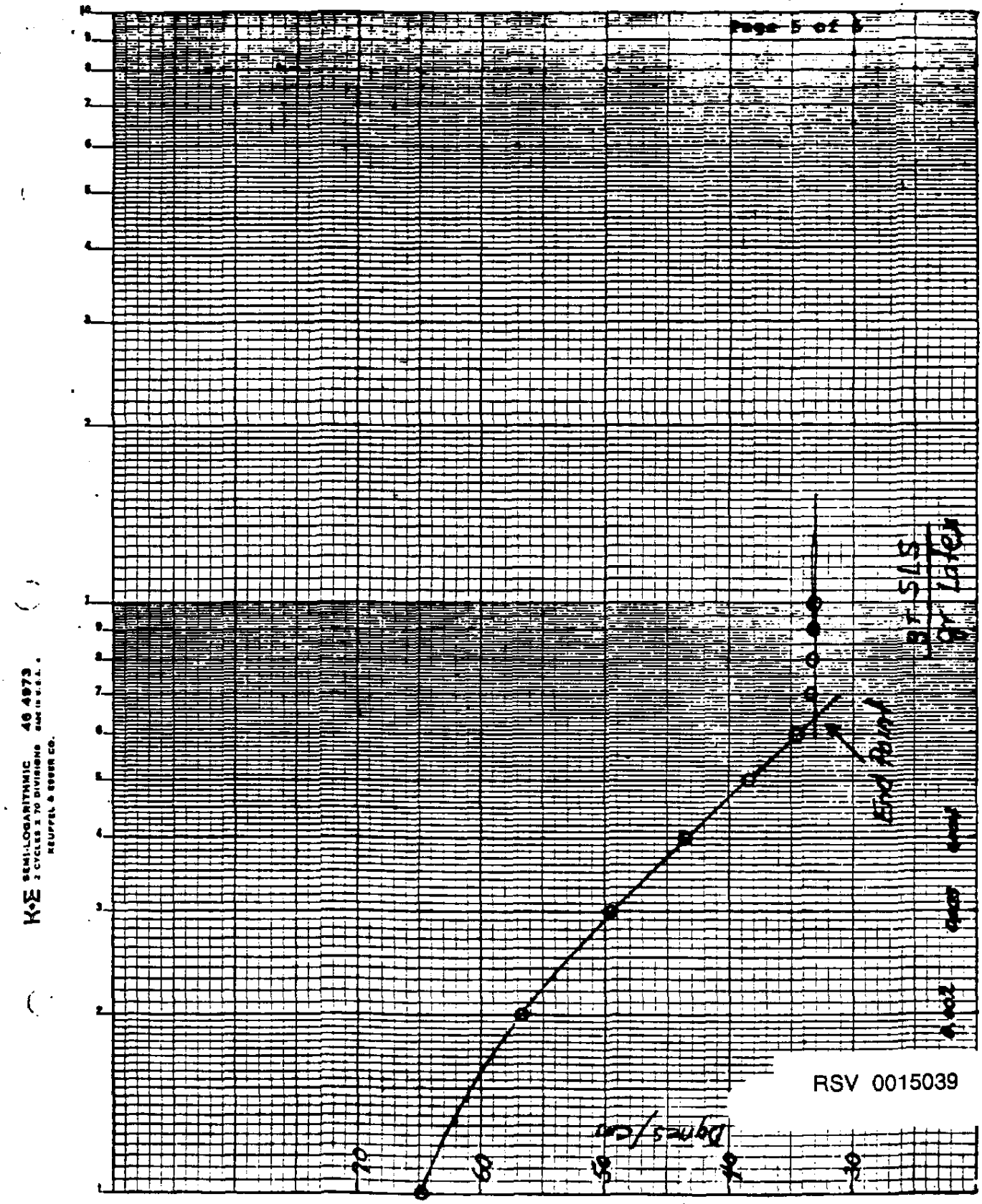

K+E SEMI-LOGARITHMIC 46 4873
2 CYCLES x 70 DIVISIONS
KEUFFEL & ESSER CO.
End Point
at 515
or later
dyne/cm
RSV 0015039

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Instron tests for tensile stress, elongation, and 100% modulus.

APPARATUS

Instron Tester
Glass Plate and Razor

PROCEDURE

1. Draw down a wet film of screened (200 mesh) latex using an 8-9 mil bar.

2. Slowly air dry the film. If drying proceeds too fast, the film will crack and craze excessively. Drying can be slowed down by placing a flat box over the wet film immediately after it is drawn down. Drying will take 2-3 hours.

3. Remove the film from the glass plate and place it onto a Teflon covered plate.

   If the film adheres too tightly to the glass plate, it may be necessary to slightly moisten it to remove it. Also, it is usually better to cut small sections of film with a razor and remove these from the glass plate rather than trying to remove the entire film intact.

4. Allow the film to air dry completely, then cure in a hot air circulating oven for 5 minutes at 150°C.

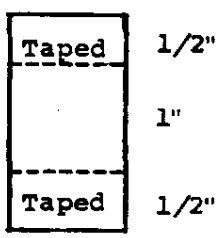
5. Accurately cut the cured film into pieces 1/2" x 2" for testing. Use a sharp razor for cutting the test pieces and avoid any edge defects. Also avoid using sections of the cured film with obvious defects, such as air bubbles or inclusions of foreign particles.

   If a specimen cutting die is available as described in ASTM D882-GIT, it can be used.

6. Determine the thickness of each test strip, using any convenient calipering device which can measure and read out to ± 0.05 mil.

Procedure (cont'd)

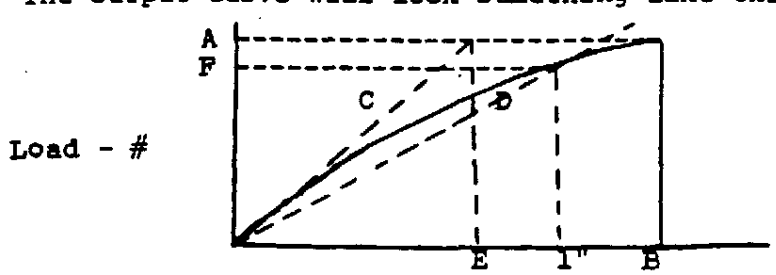
7. Reinforce the ends of each test specimen, using Scotch tape. This allows more secure clamping of the specimen and helps to minimize slippage in the jaws. One-half inch of each end should be taped.



8. Place the specimen in the Instron with a jaw span set at 1" and strain the specimen at a rate of 1"/min. until it fails.

9. Discard samples which show slippage in the jaws or within the taped ends. Also discard the results if an obvious defect develops upon straining.

10. The output curve will look something like this:



CALCULATIONS

a. Nominal tensile stress at break = (psi)

$$\frac{\text{Load at break (A)}}{\text{(Sample width in inches) x (Initial thickness in inches)}}$$

b. % Elongation at break =

$$\frac{\text{Extension in inches at break (B)}}{\text{Initial sample length (1")}} \times 100$$

Calculations (cont'd)

c. Initial tensile modulus =
(psi) - from line C

$$\frac{\text{Nominal tensile stress at break (A)}}{\text{Extension E in inches/initial span (1")}}$$

d. Secant modulus at 100% extension =
(psi) - from line D

$$\frac{\text{Load at elongation of 1" (F)}}{\text{(Sample width in inches) x (Initial thickness in inches)}}$$

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MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.264
July 17, 1971

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

% Acrylamide

APPARATUS

Kjeldahl apparatus
2000 ml beaker
Laboratory agitator for 2000 ml beaker
Buchner funnel
Vacuum source
Vacuum desiccator

REAGENTS

Isopropanol

PROCEDURE

Twenty ml latex are slowly added to a stirred beaker containing 1200 ml isopropanol. The coagulated latex and isopropanol are then stirred for 5 minutes. The polymer is filtered out using two isopropanol rinses. The vacuum to the filter is released before the polymer packs into the filter paper. The polymer is shredded up and stirred in 1500 ml of H_2O for 2 hours. It is then filtered using five 300 ml portions of H_2O as rinses. The polymer is dried in a vacuum desiccator for 24 hours at room temperature or until completely dry.

Total nitrogen in the dry polymer is obtained by the standard Kjeldahl method. Total nitrogen is assumed to be present as acrylamide. Therefore,

$$\% \text{ acrylamide} = \frac{(71)}{(14)} (\%N)$$

The total acrylamide put into the latex based on solids content may be calculated.

$$\% \text{ Acryl.} = \frac{\left[\text{lbs. Termonomer Soln. Added}}{(\text{lbs. Latex Out})(\% \text{ Solids})} (\% \text{ Acryl. in Soln})(100)\right] + 0.$$

NITROGEN DETERMINATION - KJELDAHL

EQUIPMENT

1. Kjeldahl digestion rack - gas fired.
2. Kjeldahl digestion flask - 100 ml capacity.
3. Distillation unit - JFQ Plant standard apparatus drawing 2.
4. Kjeldahl digestion catalyst - thoroughly mix 150 g. of potassium sulfate anhydrous and 10 g. of yellow mercuric oxide powder.
5. Bromcresol green methyl red mixed indicator solution - mix 50 ml of bromcresol green solution (0.1% in 95% ethanol) and 10 ml of methyl red solution (0.1% in 95% ethanol).
6. Zinc metal, 20 mesh.
7. Phenolphthalein solution - 1.0% in Formula 30 alcohol.
8. Boric acid solution - 4% in distilled water.
9. Sodium thiosulfate - 44% aqueous solution.
10. Sodium hydroxide - 50% aqueous solution.
11. Volhard absorbers - JFQ Plant standard apparatus drawing 22.
12. Usual laboratory reagents and equipment.

DETERMINATION

1. Accurately weigh 0.20 to 0.25 g. of sample into a 100 ml Kjeldahl flask, add 15 g. of the digestion catalyst and 20 ml of concentrated sulfuric acid and swirl to mix. Carry through a blank digestion omitting the sample.
2. Add several glass beads or boiling chips and place the flask in a 110°C oven for over night charring.
3. Remove the sample from the oven and digest slowly, gradually increasing the heat until the mixture boils briskly. Continue to heat until the mixture becomes clear.
4. Continue the digestion for an additional hour.

Nitrogen Determination (cont'd)

5. Cool to room temperature and cautiously add approximately 75 ml of distilled water with mixing.

6. Transfer quantitatively with the aid of distilled water to a 500 ml round bottom flask containing 10 g. of zinc metal. Add several drops of phenolphthalein solution and attach the flask to the distillation assembly. Carry through the blank in a similar manner.

7. Add sufficient 4% boric acid solution to a Volhard absorber so that the bottom overflow exit is completely covered and add 4-5 drops of the mixed indicator.

8. Attach the absorber tightly to the condenser exit.

9. Add to the round bottom flask, via the distillation side arm, sufficient 50% sodium hydroxide to make the sample alkaline to phenolphthalein. Add 5 ml of 44% sodium thiosulfate in the same manner.

10. Make sure all connections are tight, apply heat to the flask and distill approximately 100 ml of distillate into the Volhard absorber.

11. At the end of the distillation, disconnect the Volhard absorber and titrate its contents with standardized N/10 HCl solution.

12. Calculate:

$$\frac{\text{(ml titer for blank} - \text{ml titer for sample)} \times 0.14\%}{\text{sample weight}} = \%\ \text{Nitrogen}$$

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7/19/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.266
July 17, 1971

MATERIAL

Product Latex B and E

ANALYSIS REQUIRED

Film Clarity

APPARATUS

Glass plate
Drawdown bar

PROCEDURE

1. Draw down a latex film from screened latex (200 mesh) on a clean glass plate, using an 8 mil applicator bar. The exact thickness of the film is not critical.

2. Allow the wet film to slowly dry at room temperature. The film should be about 3 mils thick. If the film is too thick, it will show excessive crazing upon drying.

3. Observe the film and note the following points:

   a. Continuity of the film formed.

   b. Presence of grit.

   c. Presence of incompatible deposits in the film.

   d. Excessive roughness or graininess in the film.

   e. Clarity and color of the film.

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MATERIAL

Product Latex BH

ANALYSIS REQUIRED

I.G.T. Test

APPARATUS

Brookfield LVT or RVT viscometer
Holder for wire wound coating rods
Analytical balance
Supply of paper and board substrates
Paper cutter
I.G.T. Printability Tester, consisting of:
Printmaker equipped with pendulum and one inking disc
Ink distributing apparatus with a set of two polymethane rollers.
I.G.T. Spring Drive Device to increase speed to the 0-650 ft/min range.
Four double width inking discs (20 mm), aluminum
I.G.T. ink pipette
Extra mouthpiece for I.G.T. ink pipette
Set of two glue-glycerine composition rollers
10 sets of 6-paper packings 25 mm wide for I.G.T. Tester
Mylar film - 1 mil thick (Herculene or Stabiline Drawing film from Keuffel and Esser).
Solvent for clean-up. 2-5 gal cans of Varn Wash V-120 from Vain Products Co., Flushing, N.Y.
Two, 1/2# tubes of #4, #5, #6 black tack graded inks from Interchemical Corp., I.P.I. Printing Ink Division, 4168 Meramec, St. Louis, Mo. 63105
Three, 50# bags of Lustra Coating Day from Freeport Kadin Co., 405 Lexington Ave., N.Y., N.Y 10017.
A binocular microscope with a 15-20X magnification.
One - Series 2000 standard Premier laboratory Dispersator, 1/2 HP single phase, universal type motor, powerstat, complete with stand and mounting bracket, B/M 2053 equipped with 1-1/2" Hi-Vis head and stub shaft assembly, with shaft 3/4" dia. x 11" long constructed of 316 stainless steel. Order from Premier Mill Corp., 224 Fifth Ave., New York, N.Y.
Pyrex radiant panel heater, cat. no. 604001, 250 volts, 3500 watts, Corning Glass Works, Corning, N.Y.

Apparatus (cont'd)

One - ATC percentage input timer, type 304B-07-B-O-XX, 230 volts, 60 cycle, 20 amp., Automatic Timing & Controls, Inc., King of Prussia, Pa. 19406.
3 each of the following wire wound drawdown coating rods: #8, #10, #12, R.D. Specialties Co., P.O. Box 6, Webster, N.Y. 14580.
One - #162D case for surface mounting of type 304 ATCOTROL manual set percentage timer, general purpose sheet steel (Order with #15 above)

PROCEDURE FOR MEASURING PIGMENT BINDING STRENGTH OF LATEX - IGT PICK STRENGTH

The procedure can be broken down into four parts: 1) preparation of the coating formulation, 2) coating and drying of the substrate, 3) printing of the samples on the tester, 4) determination of end-point.

1. Preparation of the Coating Formulation

The coating formulation is a simple mixture of latex plus coating clay diluted to a total solids content of 60%. The amount of latex used is such as to give a mixture of 18 parts dry latex solids per 100 parts dry clay solids.

A 70% solids clay slip is prepared as follows:

a. Add 1.5 grams tetrasodium pyrophosphate (TSPP) to 215 grams of deionized or distilled water and stir until the TSPP is dissolved.

b. Place the solution under the Premier Dispersator and slowly add in 500 grams of Lustra Coating Clay. It will be necessary to increase the speed of the agitator as the clay is being added.

c. After all the clay has been added, disperse at the highest speed possible for 10 minutes.

d. Determine the solids content of the clay slurry.

To make up the coating formulation, add 286 grams of the clay slurry (equal to 200 grams of oven dry clay) to a 250 ml beaker. Add latex to the beaker to give the equivalent of 36 grams of oven dry latex.

Procedure (cont'd)

The amount of latex to add can be found from the following table:

% Solids of Latex	Grams of Latex to Add To Clay Slurry
45	80.0
46	78.3
47	76.7
48	75.0
49	73.5
50	72.0

After the latex has been added, add water to make the solids content of the total coating formulation equal to 60%. The amount of water needed will depend on the solids content of the latex, assuming that the clay slurry has been made up properly at 70% solids. The following table can be used as a guide for the water addition:

% Solids of Latex	Grams of Water to Add To Formulation
45	27.0
46	28.7
47	30.3
48	32.0
49	33.5
50	35.0

The coating mixture should be stirred for 10 minutes to insure proper mixing of all components. Filter the coating through a conical 200 mesh stainless steel screen to remove any aggregates of dried material and to remove excess entrained air.

The viscosity of the coating formulation should be measured with a Brookfield viscometer, Model RVT, using a #2 spindle at 10 RPM, room temperature in the 250 ml beaker.

2. Coating and Drying of the Substrate

The substrate to be used is a bleached solid sulfate board (15 point Fold-Brite White Tag Bleached Board from Riegel Paper Corp., Riegelwood, N.C.). The coating is applied to the "wire" side of the sheet. The wire side of the sheet can be discerned by the faint screen-like markings on the

Procedure (cont'd)

sheet when viewed with glancing light. It is important that the proper side be coated since each side may give different results.

A dry coat weight of 10-12#/3300 ft.^{2} is applied using the appropriate wire wound rod. This is equivalent to a dry coat weight of .00957-.01148 gm./in.^{2}.

The uncoated base sheets are supplied cut to 12" x 15" with the grain direction in the long dimension. The base sheet should be weighed and the base sheet weight calculated as follows:

$$\frac{\text{grams}}{12" \times 15"} = \text{base sheet weight in gm./in.}^2$$

Immediately after the coating is applied, the sheet is dried in the infrared oven for one minute at a temperature of 120°C. If the sheet is not properly dried, the results may be too low.

After drying, the sheet is allowed to come to equilibrium in a constant temperature and humidity room and trimmed to a size of 10" x 11". It is important to keep track of the grain direction. A good convention to follow is to keep the long dimension the grain direction.

Weigh the coated sheet and calculate the weight per unit area:

$$\frac{\text{weight of coated sheet in grams}}{10" \times 11"} = \text{Coated sheet wt. in gm/in.}^2$$

The coat weight is the difference between the coated sheet weight and base sheet weight.

3. Printing of the Samples

The samples to be tested should be allowed to equilibrate to constant temperature and humidity. Control of both room temperature and humidity is very important in obtaining reproducible results. T.A.P.P.I. standard conditions of 73°F. and 50% relative humidity are recommended if at all

Procedure (cont'd)

possible to achieve with tolerable temperature variations of ± 3.5°F and relative humidity variations of ± 2%.

The samples should be cut as strips one inch wide by ten inches long; in this case with the grain direction being the short dimension. Avoid touching the coated surface or in any other way contaminating the coated surface.

The tester should be set up and inked with 2 ml of #5 ink according to the instructions supplied with the tester. The B scale spring drive is used to give a velocity range of from 0 to 630 fpm. A loading of 70 kg. should be used on the printing nip to insure good contact of inked roll and coated sample.

At least two strips from each sample should be printed. In addition, a control latex should be run with each set of samples to insure reproducibility of results from one day to the next.

After every four strips, the ink supply on the distribution system should be replenished with 0.16 ml of ink. The glue-glycerine roll should be used as the top roll of the ink distribution device. After two hours the ink distribution system should be cleaned up and a fresh supply of ink used.

4. Determination of the End-Point

The test is designed to measure the tack stresses on the surface of the sheet which cause rupture of the coating film. These stresses are a function of the ink viscosity and the speed of printing. In the I.G.T. test the ink viscosity is kept constant by the use of a special tack graded ink. The strip which is printed is accelerated through the printing nip so that the speed of the sample through the nip is a logarithmic function of distance along the nip.

The end point measured is that speed which just causes rupture of the coating film. In order to define this point the sample is removed from the tester and examined under a low power microscope. Starting with the lowest speed end (the clamped end) draw the sample under the microscope and look for areas

Procedure (cont'd)

where the coating has been torn away. These will show up as distinct white spots against the black ink printed surface. Do not confuse white spots caused by the ink not covering the surface with picked areas. A little practice will be needed to tell the difference between a non-inked area and a pick. As the strip is scanned, the severity of picking should increase. The end point is marked as the point at which picking appears to increase in severity. Occasionally, a few isolated will be observed on the early part of the sample. These should be ignored if the picking does not increase in severity with distance. There is a considerable judgment factor involved in consistently calling the end point so it is often advisable to have only one person perform the test.

The end point is marked on the strip and converted to a speed reading (f.p.m.) using the scale supplied with the instrument.

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MATERIAL

Aqueous Charge

ANALYSIS REQUIRED

SLS Concentration

APPARATUS

100 ml, 200 ml volumetric flasks
20 ml pipet
125 ml separatory funnel
50 ml pipet
Cotton
Beckman Spectrophotometer

REAGENTS

Methylene Blue Solution
Chloroform

PROCEDURE

Accurately weigh 1 g sample into a 200 ml volumetric flask. Dissolve in distilled water and make up to volume.

Pipette 20 ml into a 125 ml separatory funnel.

Add 30 ml of distilled water and 25 ml of methylene blue solution. Mix well by swirling.

Extract with 15 ml $CHCl_3$, then 25 ml $CHCl_3$ collecting the fractions in a 100 ml volumetric flask.

NOTE: Filter the fractions through a cotton plug that has been previously wetted with $CHCl_3$. Bring to volume with $CHCl_3$.

Read at 652 mμ on the Beckman vs. $CHCl_3$ for blank.

Settings Required: Phototube in Load
Resistor #3
Slit width 0.04 to 0.1
Tungsten lamp

CALCULATIONS

$$\% \text{ SLS} = \frac{(A632)(0.38)}{\text{Sample Weight}}$$

SCOPE

This method is for small concentrations of S.L.S. (<0.5%) in process streams such as the initial aqueous charge. It involves complexing the SLS with methylene blue; extraction with $CHCl_3$, and subsequent U.V. reading at 652 mμ

DISCUSSION

1. Samples should be read within 90 min.

2. Sample size may have to be varied with stream charge variations.

3. Methylene blue, N.F. - Matheson, Coleman and Bell.

   Methylene Blue Solution - dissolve 0.1 g methylene blue, N.F. contained in a 100 ml volumetric flask in distilled water and dilute to 100 ml. Transfer 30 ml of this solution to a 1 liter volumetric flask, containing 500 ml of distilled water, 6.8 ml of concentrated sulfuric acid, and 50 g of sodium sulfate anhydrous, A.R. grade. Shake until solution is complete and dilute to volume with distilled water.

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MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.289-A (WC)
July 17, 1971

MATERIAL

Vinyl Chloride

ANALYSIS REQUIRED

Determination of Iron in Vinyl Chloride

SAMPLE

100 ml glass pressure sample bomb

APPARATUS

Beckman Spectrophotometer
Dry Ice Chest
Two, 250 ml Erlenmeyer Flasks
100 ml Graduated Cylinder
10 ml Graduated Cylinder
100 ml Volumetric Flask

REAGENTS

-10% Aqueous solution of Hydroxylamine hydrochloride
-2 molor solution of C.P. sodium acetate (164 ± 0.01 g in 1 liter of distilled water)
-0.1% solution of brom phenol blue (0.1 g dissolved in 100 ml of 50% ethyl alcohol)
-0.12% aqueous solution of o-phenanthroline
-Alcohol water solution (25 parts 2B alcohol and 30 parts water)
-Concentrated C.P. HCl

PROCEDURE

Place 25 ml of the alcohol-water solution in a 250 ml erlenmeyer flask. From the sample bomb, previously cooled for two hours in the dry ice chest, measure out 100 ml of vinyl chloride into a chilled graduated cylinder. Add the vinyl chloride to the 250 ml erlenmeyer flask and allow to evaporate. Then swirl the flask to remove the last trace of vinyl chloride. A vented hood is needed for the above evaporation.

Procedure (cont'd)

Quantitatively transfer the residue to a 100 ml volumetric flask.

Acidify the solution in the volumetric flask by adding two drops of concentrated hydrochloric acid. Add two drops of brom phenol blue to the flask and then add the 2 molar sodium acetate solution dropwise until the solution turns a faint blue. Next, add four ml of 10% hydroxylamine hydrochloride solution, and then 10 ml of 0.12% o-phenathrcline. Dilute to the mark with distilled water; mix well, and allow the solution to stand for at least 20 minutes.

With the Beckman spectrophotometer measure the light transmission at 510 mu, with visible light.

CALCULATIONS

Refer to the attached graph and report the iron concentration to the nearest 0.05 ppm.

PREPARATION OF REAGENTS

10% aqueous hydroxylamine HCl - dissolve 100 g of the salt in 900 ml of distilled water. Be sure water is iron free. Store in P.E. bottle.

2 molar sodium acetate - dissolve 164 gms of C.P. salt in one liter of distilled water. Mix. Store in clean bottle.

.1% solution of brom phenol blue - dissolve .1 gm of the indicator in 50 ml of EtOH. Dilute to 100 ml with distilled water.

.12% of o-phenanthroline - dissolve .12 gms of o-phenanthroline in 100 ml distilled water.

Alcohol water solution - Mix 455 ml of 2B alcohol and distilled water up to one liter.

1000 ppm Fe Standard - dissolve .1000 gms of C.P. iron wire in 10 ml of 20% HCl (iron free). Cool and dilute to 100 ml with distilled water. Mix. Keep for stock reagent.

10 ppm Standard - dilute 1 ml of 1000 ppm standard to 100 ml with demineralized water. Mix. Keeps only one or two days.

STANDARDIZATION AND/OR CALIBRATION

To five 100 ml volume flasks add 0, 3, 5, 7, and 10 ml of the 10 ppm standard. These correspond to 0, 30, 50, 70 and 100 µgms of Fe.

Follow the procedure outlined above from paragraph three on.

Determine absorbance in 20 mm cells against the zero standard above at 510 mµ.

Plot the absorbances so obtained against the corresponding µgms of Fe. Draw in the best average line. Use this as a working curve or calculate a factor f from it such that f x absorbance = µgms Fe.

SAFETY

Handle VCM with caution. Keep vapors away from sparks and flames. Guard against cold burns, handle with gloves. Use normal precautions with chemicals, glassware, and electrical apparatus.

ANALYTICAL TIME

1/2 hour

PRECISION OF RESULTS & SIGNIFICANT FIGURES TO REPORT

95% confidence limits are ± .1 ppm at a level of 1 ppm or ± 10% of the level found. Report results to nearest .05 ppm.

SCOPE

VCM sample is evaporated leaving any iron in a non-freezing mixture of water and alcohol. The iron is reduced to valence two and reacted with 1,10 phenanthroline to a red product analytical to iron at 510 mµ. These are known interferences.

REFERENCES

Plastics Division Control Lab. procedure 112-70-3.
Texas City C.L. Method 22-18-10, G.L. 4/22/54.

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IRON IN VCM CALIBRATION CURVE

Wave Length: 510 mu
Cell Length: 20 mm
Standard: Blank (with reagents)

ppm Fe	Optical Density
0-0.1	0-0.05
0.2	0.05-0.09
0.3	0.090-0.112
0.4	0.112-0.150
0.5	0.150-0.19
0.6	0.19-0.23
0.7	0.23-0.27
0.8	0.27-0.31
0.9	0.31-0.34
1.0	0.34-0.40

MATERIAL

Product ethylene

ANALYSIS REQUIRED

Methanol

SAMPLE

200 ml stainless steel flow thru bomb

APPARATUS

Gas chromatograph equipped as follows:

1. Hydrogen flame ionization detector which should have a sensitivity of approximately 1 coulomb per mole for a C_4 hydrocarbon.
2. Amplifier and recorder with sensitivity sufficient to yield a full scale response to 1 x 10-11 amps of ion current and a noise level equivalent to less than 1 x 10-13 amps.
3. Gas sampling valve with a 5CC sample loop.
4. Temperature controller for operation at 80°C.
5. Column consisting of 20 feet of 1/8 inch O.D. aluminum tubing packed with 30-60 mesh red chromosorb coated with 20% LAC-1-R- 296 polyester (available from Cambridge Industries Co., Inc., Cambridge, Mass.) Methylene chloride is the solvent used in coating the chromosorb with the polyester.

REAGENTS

1. Ethylene - Pure grade from Phillips Petroleum Co.
2. Methanol - Reagent grade
3. Hydrogen - Electrolytic grade
4. Helium - Commercial
5. Air - Free of water, organic compounds and particulate matter.

PROCEDURE

1. Install the column in the instrument and adjust oven temperature to 80°C and helium carrier flow to 45 cc per minute.

2. Adjust the hydrogen and air flow to the detector for maximum signal-to-noise ratio. Vary hydrogen flow first then air.

3. Allow instrument to "line out" at these conditions as indicated by a stable baseline on the recorder for five minutes or more using maximum sensitivity on the amplifier.

4. Using the gas sample valve, introduce 5cc of sample into the instrument.

5. Record the chromatogram. Methanol is eluted in approximately 7 minutes.

CALCULATIONS

1. Measure the area of the methanol peak.

2. Calculate percent methanol as follows:

Peak area x attenuation x calibration factor = % MeOH

CALIBRATION

1. Prepare synthetic mixtures of methanol in ethylene to cover concentration range of interest. The importance of using clean bombs for preparation of synthetic mixtures cannot be over emphasized.

2. Analyze the synthetic mixtures according to the above procedure.

3. Calculate the calibration factor.

SAFETY PRECAUTIONS

Normal safety precautions on handling hydrocarbon gases should be observed.

ANALYTICAL TIME

Average time required for this analysis is 0.25 hours.

PRECISION OF ANALYSIS

The precision of this method is ±5 ppm in the range of 5 to 50 ppm methanol in ethylene.

SCOPE

This is a gas chromatographic procedure for the determination of methanol in the concentration range of 1-500 ppm in ethylene. The minimum detection limit is approximately 5 ppm of methanol. C_4's and up to 1% C_5's will not interfer with this analysis. Heating bomb above ambient temperature before injecting a sample into the chromatograph was found unnecessary.

REFERENCES

1. Research Method TC - 1180 (GC)

2. Research Notebook HC No. 1, pp. 24-33.

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MATERIAL

Vinyl Chloride Monomer

ANALYSIS REQUIRED

Acidity as HCl

SAMPLE SIZE

Glass Bomb

APPARATUS

500 ml Iodine Flasks

5 ml Micro Burette .01 ml Graduations

Mettler Balance

REAGENTS

0.1% Thymol Blue in Methanol

C.P. Methanol

Ethylene Dichloride (Neutral)

Methanolic Caustic (.003 N)

Solvent Mixture Prepared from above.

PROCEDURE

1. Add 100 ml solvent mixture to each of two 500 ml iodine flasks. To the "blank" flask add 60 ml of neutral EDC.

2. Neutralize both flasks to an orange-yellow end point with .003 N MeOH-NaOH. Bring "sample" flask and "blank" flask to the same color, Check the color by looking down through the liquid from the top. Stopper flasks.

3. Place flasks in dry ice chest for 2-3 minutes. Remove and match colors again. Adjust, if necessary, with MeOH-NaOH to the same color again.

PROCEDURE (Continued)

4. Attach bomb to 1/8" SS coil in cooling bath (-35°C). Place sample flask under outlet of tubing. Open valve on bomb and drain VCM into sample flask. Tubing should be under the surface of the liquid. When sample stops bubbling, close valve and remove bomb. Purge a small amount of nitrogen through the SS tubing.

5. Remove flask, swirl to mix, and titrate to end point of the blank by matching the colors. Note volume of MeOH-NaOH used.

   NOTE: Obtain sample weight by weighing bomb on Mettler balance before and after entering sample into titration flask.

CALCULATION

$$\text{ppm HCl} = \frac{\text{V MeOH - NaOH} \times \text{N MeOH-NaOH} \times 36{,}500}{\text{Sample Weight}}$$

PREPARATION OF REAGENTS

Titration solvent: 920 ml neutral EDC, 30 ml 0.1% thymol blue and 50 ml CP. Methanol per liter of solution.

Neutral EDC: Refer to Solutions Manual.

.003 MeOH-NaOH: Refer to Solutions Manual.

0.1% Thymol Blue in Methanol: Refer to Solutions Manual.

STANDARDIZATION AND CALIBRATION:

Refer to Solutions Manual for standardization of .03 MeOH-NaOH. This in turn is diluted 1:10 with methanol for .003 N MeOH-NaOH.

SAFETY PRECAUTIONS

When disposing of material in sample flask, be sure that all VCM has been evaporated from the solution before the EDC is put in solvent drain.

ANALYTICAL TIME

0.3 Hours.

PRECISION AND SIGNIFICANT FIGURES

± 0.1 ppm at 1 ppm level. Report to the nearest 0.1 ppm.

SCOPE

This is a non-aqueous acid-base titration capable of detecting about 10 micrograms of HCl.

REFERENCE

Research Notebook #2271, pp. 148746-53.

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MONSANTO COMPANY

TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.344 (WC)
May 14, 1964

MATERIAL

Product vinyl chloride and 22E11

ANALYSIS REQUIRED

Determination of aldehydes

SAMPLE SIZE

Quart thermos bottle

APPARATUS

Beckman Model "B" Spectrophotometer

100 ml volumetric flask

Coca-cola bottle and cap

Bottle opener

Ice pick

250 ml Erlenmeyer flask

100 ml graduated cylinder

25 ml graduated cylinder

1 ml pipette

Triple beam balance

REAGENTS

Alpha - methyl indole solution

Ammonium hydroxide (NH_4OH), (CP conc.)

Acetaldehyde (CH_3CHO)

PROCEDURE

Select a clean, dry Coca-cola bottle free from large scratches. Pipette 1.0 ml of concentrated NH_4OH into the bottle and add 10 ml H_2O.

Pour 100 ml of vinyl chloride into a 100 ml graduate which has been cooled in dry ice for at least 15 minutes. Transfer to the Coca-cola bottle and cap the bottle immediately.

Shake the sealed bottle vigorously and place in the cabinet for one hour (CAUTION: Wear face shield while handling vinyl chloride). Puncture the cap with an ice pick and then remove cap. Allow the vinyl chloride to evaporate in the hood.

When all of the vinyl chloride has evaporated, wash the contents of the bottle into a 100 ml volumetric flask with several portions of distilled water. The volume of distilled water shall not exceed 50 ml. Add 20 ml of α- methylindole solution to the washings. Shake to mix thoroughly and allow the solution to stand for 10 minutes. Dilute to 100 ml with distilled water and again shake thoroughly.

Read the optical density of this solution on the Beckman "B" Spectrophotometer at 550 mu using 20 mm cells and distilled water as the blank.

If the turbidity exceeds the range of the chart, repeat the analysis using a smaller volume of vinyl chloride.

CALCULATIONS

The results are read directly from the chart to the nearest part per million. Duplicate determinations should check within 2 ppm. See chart on last page of procedure.

PREPARATION OF REAGENT SOLUTIONS

Prepare fresh solutions daily by adding 60 ml concentrated CP HCl to 30 ml of distilled water in a 250 ml Erlenmeyer flask. Then dissolve in this 0.15 ± 0.05 grams of alpha methyl indole.

STANDARDIZATION AND/OR CALIBRATIONS

Chill a 5 ml Mohr pipette graduated in 0.1 ml increments to a temperature of 0°C by placing in an ice chest. Wrap pipette with a paper towel to avoid moisture contamination. After ten minutes, remove and pipette 5 ml of technical grade acetaldehyde. Introduce 1.25 ml of this chilled acetaldehyde into a 1000 ml chilled volumetric flask containing distilled water. Make up to volume with distilled water and shake thoroughly. The contents of this flask contains 1000 ppm acetaldehyde. Call this Solution A.

10 millileters of A diluted to 1 liter water = 10 ppm AcH

5 millileters of A diluted to 1 liter water = 5 ppm AcH

1 millileter of solution A diluted to 1 liter = 1 ppm AcH

Intermediate parts are obtained by diluting various parts of A to 1 liter. These aliquot portions are then run in a similar manner to that of the sample and the various scale readings recorded. Duplicate determinations should be run for a check.

These scale readings will relate to the concentration of the acetaldehyde present.

SAFETY PRECAUTIONS

When depressurizing the VCM in the Coca-cola bottle, be sure that the ice pick is removed slowly and the bottle is kept in a safety cage and wearer has on a face shield.

ANALYTICAL TIME

0.2 hour

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING

Report to nearest 1 ppm; duplicate determinations should check each other ± 1 ppm at 1 ppm level.

SCOPE

Alpha methyl indole reacts with aldehydes to form a turbidity. A measure (spectrophotometer) of turbidity determines the amount of aldehydes present.

/jw
7/29/71

ACETALDEHYDE IN SPECIFICATION VCM

20 mm Cells

Beckman "B" Spectrophotometer

Optical Density	ppm AcH
0 - 0.027	0
0.028 - 0.082	1
0.083 - 0.136	2
0.137 - 0.190	3
0.191 - 0.245	4
0.246 - 0.299	5
0.300 - 0.354	6
0.355 - 0.409	7
0.410 - 0.463	8
0.464 - 0.518	9
0.519 - 0.572	10

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.431
May 23, 1971

MATERIAL

Acrylamide
Ferric Ammonium Sulfate
Sodium Formaldehyde Sulfoxylate
Tetrasodium Ethylene diamine Tetraacetate

ANALYSIS REQUIRED

Appearance of Solids

SAMPLE SIZE

8-ounce bottle

APPARATUS

None

REAGENTS

None

PROCEDURE

1. If the material consists of free flowing relatively stable non-hazardous particles, spread a small amount on a white card. Otherwise observe in the sample container.
2. Describe the color of the sample.
3. Describe the physical size, form, and uniformity of particles such as powder, crystals, flakes, briquettes, lumps, fused mass, etc., with respect to the specification requirement.
4. Note the presence of any discolored areas or particles and/or the presence of extraneous materials.

SCOPE

This method is intended to provide a general physical description of solid materials.

:ff
7/20/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.432
May 23, 1971

MATERIAL

Sodium Lauryl Sulfate
Tetrahydrofuran

ANALYSIS REQUIRED

Appearance of Liquids

SAMPLE SIZE

8-ounce bottle

APPARATUS

Source of strong oblique illumination shaded from the observer's eye, such as Turbidimeter, Clark, Catalog No. T11315, The Chemical Rubber Co., Cleveland, Ohio.

REAGENTS

None

PROCEDURE

1. Rinse the outside of the sample container, a clear glass bottle, with methanol and water, wipe it with a towel and place it on a white card or opal glass background.
2. Describe the color of the sample.
3. Describe the clarity with respect to haze, sediment, globules or layers of immiscible liquids or other insolubles.
4. Swirl the sample, avoiding the introduction of air bubbles, and observe under the Clark Turbidimeter, describing any turbidity, opalescence, or sediment.
5. Describe the fluidity with respect to the specification requirement.

SCOPE

This method is intended to provide general physical description of liquid materials when viewed under a constant set of conditions, and to define any visibly apparent contamination.

:ff
7/20/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.433
May 23, 1971

MATERIAL

Acrylamide

ANALYSIS REQUIRED

Assay

SAMPLE SIZE

8-ounce bottle

APPARATUS

1. Erlenmeyer flask, 250 ml heavy wall, fitted with a 24/40 Standard Taper joint (Ace Glass Co., No. 2671).
2. Filling funnel, 50 ml cylindrical, open top with Standard Taper stop-cock and 24/40 Standard Taper joint on bottom (Kontes Glass Co., No. K-63125). In the top of the funnel insert a rubber stopper fitted with a short glass tube for connection to vacuum line.

REAGENTS

1. 0.1 N $KBrO_3$ - KBr solution (2.79 g. potassium bromate and 10 g. of potassium bromide per liter).
2. 6 N H_2SO_4
3. 20% KI solution
4. Std. 0.1 N $Na_2S_2O_3$
5. 1% starch solution

PROCEDURE

Weigh accurately a sample containing 1.0 - 1.5 g. of acrylamide into a 500 ml volumetric flask, dilute to volume with water and mix. Pipet a 25.00 ml aliquot of this solution and 25.00 ml of bromate-bromide reagent into the 250 ml Erlenmeyer flask. Attach the filling funnel and rubber stopper, evacuate the flask, then close the stopcock and remove the stopper. Draw 5 ml of 6N sulfuric acid into the flask while preserving the vacuum. Allow the solution to stand in the dark for twenty minutes and shake frequently. Add 15 ml of potassium iodide solution, mix and then break the vacuum. Titrate the solution immediately with standard thiosulfate using starch indicator. Run a blank determination.

CALCULATION

$$\% \text{ Acrylamide} = \frac{(\text{blank titer} - \text{sample titer})(N\ Na_2S_2O_3)(3.554)}{\text{Weight of sample in aliquot}}$$

SCOPE

Acrylamide may be determined by bromination of the double bond. This method, which is used in the laboratories of the American Cyanamid Company, is applicable to solids and solutions.

:ff
7/20/71

MATERIAL

Sodium Formaldehyde Sulfoxylate

ANALYSIS REQUIRED

Ash

SAMPLE SIZE

8-ounce bottle

APPARATUS

a. Porcelain wide form crucible, such as Coors No. 2, Catalog No. 7-955, Fisher Scientific Company.

b. Platinum dish, 25 ml. American Platinum Works

c. Burner, such as Catalog No. 3-902, Fisher Scientific Company.

d. Nichrome Triangles, such as Catalog No. 15-260, Fisher Scientific Company.

e. Nichrome Triangles with Fused Silica Tubes, such as Catalog No. 15-280, Fisher Scientific Company.

f. Desiccator, such as Catalog No. 8-631, Fisher Scientific Co.

g. Desiccant, such as anhydrous 4-mesh calcium chloride.

h. Furnace - muffle, such as Catalog No. 10-526, Fisher Scientific Co.

REAGENTS

Sulfuric Acid, T.S., Diluted - Cautiously add 57 ml of sulfuric acid (95-98%) to sufficient water to make 1000 ml.

PROCEDURE

NOTE: This procedure specifies a platinum crucible for general applicability. A porcelain crucible may be used if previous experience with the product being ashed indicates no attack on porcelain.

Procedure (cont'd)

1. Sand polish and mold the platinum crucible.
2. Heat the crucible on a silica sleeved Nichrome triangle for 5-10 minutes being careful not to allow the blue reducing flame of the burner to come in contact with the crucible.
3. Allow the crucible to pre-cool for 10 minutes on the silica covered triangle, place in a desiccator for 30 minutes if using platinum or 1 hour if using porcelain, and weigh accurately.
4. Add 2.00 ± 0.01 grams of sample and 10 ml of diluted sulfuric acid T.S. to the crucible.
5. Place the crucible on a porcelain top hot plate and heat gently until the sample is dry and thoroughly charred, then continue heating until the sample has been volatilized or nearly all the carbon has been oxidized.
6. Allow the crucible to cool and then carefully moisten the residue with 0.1 ml of H_2SO_4.
7. Heat as in Step 5 until the remainder of the sample and any excess sulfuric acid have been volatilized.
8. Finish the ignition in a muffle adjusted to 800 ± 25°C.
9. Pre-cool the crucible for 10 minutes on a silica covered triangle, cool in a desiccator for 30 minutes if using platinum or 1 hour if using procelain, and weigh accurately.
10. Calculate:

$$\% \text{ Ash} = \frac{\text{net weight of residue x 100}}{\text{sample weight}}$$

DISCUSSION

To obtain constant weight of the porcelain crucible within a reasonable time, it is imperative that it be pre-cooled on a porcelain or aluminum block for 10-15 minutes prior to placing in a desiccator and then further cooled in the desiccator for one hour. Elimination of the pre-cooling step necessitates a three hour or more cooling cycle in the desiccator. One hour cooling periods without the pre-cooling period could incur errors of 5-10 milligrams, depending on the size and number of crucibles and the size of the desiccator.

The initial combustion should be made with as low a temperature as possible. Only when the carbon has fully disappeared from the

Discussion (cont'd)

sides of the crucible should it be placed in a muffle furnace. A further consideration to be kept in mind is that if the flame is allowed to envelop the crucible fully, the atmosphere within the crucible is not air but largely a mixture of nitrogen and the products of combustion, of which water vapor is an important one. The chief effect of the water vapor is to raise the temperature necessary for complete dehydration. This is the reason long ignition periods are often required for the conversion of a metal salt to the oxide, whereas under proper conditions the conversion could be made in minutes.

Once the carbon is completely removed from the wall of the crucible, it is only a matter of expedience as to whether the muffle or the Meker burner is used to burn the carbonaceous matter from the residue (Ref. 1).

If the residue after ignition contains sodium or potassium carbonate, it may fuse and occlude carbon, making complete removal of the latter difficult. Increasing the sample weight only increases the tendency toward false higher answer.

Results will always be low in the presence of easily reducible oxides of volatile metals.

Due to the fluffiness of some ash residues, extreme care must always be exercised to prevent loss by air currents.

PRECISION

The following criteria should be used for judging the acdeptability of results (95% confidence):

a. Repeatability. Duplicate results by the same operator should be considered suspect if they differ by more than the following amount - 0.0 to 0.15 Ash, %; 0.003 Repeatability.

b. Reproducibility. The results submitted by each of two laboratories should be considered suspect if the two results differ by more than the following amount - 0.0 to 0.15 Ash, %; 0.005 Reproducibility.

SAFETY

Handle hot crucibles with tongs.

Operate in a fume hood, and behind a safety shied, if there is any possibility of the material having explosive tendencies.

Safety (cont'd)

Make sure the ground glass flange of the desiccator is greased to prevent sticking.

REFERENCES

1. "Applied Inorganic Analysis", Hillebrand, Lundell, Bright, and Hoffman, John Wiley & Sons, London, 1953.

2. 1968 Book of ASTM Standards, part 17, page 195.

3. Food Chemicals Codex I.

4. USP XVIII.

SCOPE

This method is intended for the determination of Ash contents of less than 1.0% in pharmaceuticals, oils, etc. The scope is similar to that of the ASTM D-482-63 method for "Ash From Petroleum Products." The method does not necessarily give a true measure of the metallo-organic compounds or the inorganic compounds present because of possible losses by vaporization or reduction of the metal compounds present.

:ff
7/20/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.435
May 23, 1971

MATERIAL

Ammonium Persulfate

ANALYSIS REQUIRED

Assay

SAMPLE SIZE

8-ounce bottle

APPARATUS

a. Analytical balance
b. 250 cc glass stoppered flask
c. Titrating burets
d. Usual laboratory equipment

REAGENTS

a. Standard Acid Ferrous Sulfate solution (0.2 N)
b. 0.1 N potassium permanganate

PROCEDURE

Add 0.5 gm of sample, accurately weighed, to 25.0 ml of Standard Acid Ferrous Sulfate solution (about 0.2 N) in a glass-stoppered flask. Stopper the flask, allow it to stand for 1 hour with frequent shaking and titrate the excess ferrous sulfate with 0.1 N potassium permanganate. Titrate 25.0 ml of the Standard Acid Ferrous Sulfate solution with 0.1 N potassium permanganate. From the volume of 0.1 N permanganate required, subtract the volume of 0.1 N permanganate required in the titration of the sample solution. Each ml of the difference, consumed by the sample, is equivalent to 0.01141 gm of $(NH_4)_2S_2O_8$.

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7/20/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.436
May 23, 1971

MATERIAL

Ferric Ammonium Sulfate

ANALYSIS REQUIRED

Assay

SAMPLE SIZE

8-ounce bottle

APPARATUS

a. Analytical balance
b. 250 cc glass stoppered flask
c. Titrating burets
d. Usual laboratory equipment

REAGENTS

a. 0.1 N hydrochloric acid
b. 0.1 N potassium iodide
c. 0.1 N sodium thiosulfate
d. Starch indicator

PROCEDURE

Dissolve about 1.5 gm of sample, accurately weighed, in 50 ml of water in a glass-stoppered flask. Add 3 ml of hydrochloric acid and 3 gm of potassium iodide and let stand for 30 minutes in the dark. Dilute with 100 ml of water and titrate the liberated iodine with 0.1 N sodium thiosulfate adding starch indicator solution toward the end of the titration. Correct for a blank. Each ml of 0.1 N sodium thiosulfate consumed is equivalent to 0.04822 gm of $FeNH_4(SO_4)_2 \cdot 12H_2O$.

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7/20/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.437
May 23, 1971

MATERIAL

Sodium Formaldehyde Sulfoxylate

ANALYSIS REQUIRED

Assay

SAMPLE SIZE

8-ounce bottle

APPARATUS

a. Analytical balance
b. 1000 ml glass flask; 250 ml glass flask
c. Titrating buret
d. Usual laboratory equipment

REAGENTS

a. 0.10 N iodine
b. 10% formaldehyde solution
c. Starch indicator

PROCEDURE

Weigh out 10 grams of SODIUM FORMALDEHYDE SULFOXYLATE, dissolve in 150 cc of warm water, add distilled water to make a total volume of 500 cc. Take a 4 cc aliquot in 100 cc distilled water and titrate with 10 N Iodine (adding first 5 cc of 10% Formaldehyde solution) and Starch Indicator.

CALCULATIONS

$$\frac{\text{cc .10N Iodine} \times .00385 \times 100}{\text{Wt. of sample is 0.08 grams}} = \% \text{ Sodium Formaldehyde Sulfoxylate}$$

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7/20/71

MATERIAL

Sodium Lauryl Sulfate

ANALYSIS REQUIRED

Assay

SAMPLE SIZE

8-ounce bottle

APPARATUS

150 ml beaker
250 ml volumetric flask
25 ml pipet
500 ml separatory funnel
Analytical balance

REAGENTS

p-toluidine hydrochloride solution, 6.8%
3-A Alcohol, Form #30
Carbon Tetrachloride (reagent grade)
Meta-cresol purple indicator (0.1% solution)
Phenol red indicator (0.1% solution)
0.1 N NaOH

PROCEDURE

Using an analytical balance, transfer 5.00 grams of SIPON WD to a 150 ml beaker. Dissolve in distilled water and transfer to a 250 ml volumetric flask (a small amount of 3-A Alcohol may be used to dissipate foam) and dilute to 250 ml mark. Mix well and withdraw a 25 ml aliquot portion (equivalent to 0.5 grams of sample).

Transfer the aliquot portion to a 500 ml separatory funnel. Add 2 drops of phenol red indicator (red-alkaline, yellow-acid). If solution is alkaline, adjust to neutral with .1N HCl. If solution is acid, adjust to neutral with .1N NaOH. To neutral solution add 30 ml of 3-A Alcohol, 30 ml of 6.8% p-toluidine hydrochloride solution and 30 ml of carbon tetrachloride. Shake mixture for

Procedure (cont'd)

three minutes (occasionally releasing gas from separatory funnel). Allow to stand and separate. Cut lower (carbon tetrachloride) layer into 250 ml wide-mouth Erlenmeyer flask, containing 50 ml of 3-A Alcohol and 3 cc H_2O adjusted to the alkaline (purple) end-point of meta cresol purple. Add 30 ml carbon tetrachloride to contents of separatory funnel and repeat extraction. Allow to stand and separate and again cut lower layer into same receiving flask.

Repeat extraction once more. These combined extractions in the alcohol medium contain all the organic sulfates and are titrated directly with standard 0.1N NaOH to determine the alkyl sulfate content. The end-point is taken as the first change in color from gray to purple that remains for 15 seconds.

CALCULATION

$$\frac{\text{ml NaOH} \times N \times 300 \times 100}{\text{Wt. of sample in the aliquot} \times 1000} = \% \text{ Alkyl Sulfate as sodium lauryl sulfate}$$

SCOPE

This procedure employs the extraction of the organic sulfates by p-toluidine hydrochloride precipitation.

:ff
7/20/71

MATERIAL

Tetrasodium Ethylenediamine Tetraacetate

ANALYSIS REQUIRED

Chelation value

SAMPLE SIZE

8 ounce bottle

APPARATUS

a. Analytical balance
b. 250 ml Erlenmeyer flask
c. Titrating burette
e. Usual laboratory equipment

REAGENTS

a) Ammonium oxalate, saturated solution. Dissolve approximately 60 grams of ACS reagent grade (calciul-free) ammonium oxalate $(NH_4)_2C_2O_4 \cdot H_2O$ in a liter of water and allow to cool to room temperature.

b) Calcium chloride, standard solution.
Dissolve 0.5 molar of primary standard calcium carbonate (Mallincrodt No. 4071) in 300 ml of distilled water by slowly adding 86 ml of concentrated hydrochloric acid to completely dissolve the calcium carbonate. Heat to boiling to remove carbon dioxide and completely dissolve the sample. Cool to room temperature and dilute this solution to exactly one liter. One ml of this solution contains the equivalent of 50 mg of calcium carbonate.

c) Ammonium hydroxide, 1:1 solution. Add one volume of concentrated ammonium hydroxide to one volume of distilled water.

PROCEDURE

Weigh, to the nearest milligram, a 10 gram sample of chelating agent into a clean 250 ml Erlenmeyer flask, and add 85 ml of distilled water and 5 ml of saturated ammonium oxalate solution. Titrate the sample to the first faint permanent turbidity with standard calcium chloride. The pH of the solution should be checked by using pH paper after the endpoint has been reached; if the pH is below 11, a dilute solution of ammonium hydroxide should be added to raise the pH above 11. After the pH has been adjusted, the titration should be completed.

CALCULATION

$$\frac{\text{ml } CaCl_2 \text{ solution} \times 50}{\text{gms of sample}} = \text{mg } CaCO_3 \text{ per gm of chelating agent}$$

SCOPE

The total chelation value is determined by titrating with a solution of calcium salt in the presence of oxalate ion as an indicator at a pH of 11 or slightly higher. The pH is important because the chelating ability decreases when the pH of the solution drops below 11 and consequently low results are obtained.

/jw
7/23/71

MATERIAL

Ferric Ammonium Sulfate, Sodium Lauryl Sulfate

ANALYSIS REQUIRED

Chlorides

SAMPLE SIZE

8 ounce bottle

APPARATUS

a) Usual laboratory apparatus

b) Potentiometer sensitive to potential changes of 5 mv. or less, coupled to the system:

Ag::Solution::2M $MgSO_4$:Hg_2SO_4:Hg

REAGENTS

a) 10% H_2SO_4

b) Methyl orange indicator

c) 0.01 N $AgNO_3$

PROCEDURE

1. Dissolve 5 grams of sample, weighed on a prescription balance, in 40 ml of water in a 150 ml beaker.

2. Slowly neutralize to methyl orange with 10% H_2SO_4 and add 0.5 ml in excess.

3. Cool the solution to 0-5°C in an ice bath and stir mechanically under the silver electrode system.

   NOTE: If a precipitate forms, as with some organic salts, add just sufficient acetone to redissolve.

PROCEDURE (cont'd)

4. Titrate fairly rapidly with N/100 $AgNO_3$ until the potential begins to decrease markedly with small increments of titrant.

5. Continue the titration in 0.1 increments, recording the potential after each, then underscore the total volume of titrant at the point corresponding to the largest potential change per increment of titrant.

6. Calculate the chloride content as the specific chloride designated in the specification for the product:

$$\% \text{ Specified chloride} = \frac{\text{ml titre x Factor}}{\text{sample weight}}$$

Table of Chloride Factors

Specified Chloride	Factor for N/100 $AgNO_3$
Cl^-	0.0355
$CaCl_2$	0.0555
HCl	0.0365
$MgCl_2$	0.0476
$MnCl_2$	0.0629
NaCl	0.0585
NH_4Cl	0.0535

PRECISION AND ACCURACY

Duplicate determinations should agree to within 0.02%.

SAFETY

No unusual hazards are involved in this operation.

SCOPE

This method is intended for the determination of 0.01 to 0.5% ionizable chloride in water soluble materials. It involves a potentiometric titration with silver nitrate solution.

MATERIAL

Sodium Lauryl Sulfate

ANALYSIS REQUIRED

Cloud point Determination

SAMPLE SIZE

8 ounce bottle

APPARATUS

a) ASTM cloud and pour point kerosene cooling baths adjusted to 30°F, 0°F and -30°F with dry ice.

b) Test jars, gaskets and ASTM Cloud and Pour Point thermometers.

c) Usual laboratory equipment.

REAGENTS

None

PROCEDURE

1. Bring the fluid to be tested to a temperature at least 25°F above the specification cloud point in the bath adjusted to the next lower temperature.

2. If moisture is present, remove by filtration thru dry filter paper until the fluid is perfectly clear. The temperature at which the filtration is made must be at least 25°F above the cloud point.

3. Fill a test jar to a point slightly above the mark with a portion of the sample.

PROCEDURE (cont'd)

4. Close the jar tightly with a stopper carrying a cloud and pour point test thermometer in a vertical position in the center of the jar, with the bulb touching the bottom of the jar.

5. Adjust a gasket around the jar one inch from the bottom and place in the jacket of the bath adjusted to the first temperature below the temperature of the sample.

6. As the temperature falls at each reading that is a multiple of 2, remove the test jar from the jacket, quickly without disturbing the sample, observe against a good light for the appearance of a cloudy haze, and replace the jar in the bath within 3 seconds.

7. If the sample does not develop a cloud when it has cooled to within 20°F of the bath temperature, place the jar in the next lower bath and continue observing at 2°F intervals.

8. Record as the cloud point the temperature at which the first distinct cloudiness or haze appears in the bottom of the test jar.

SAFETY

Kerosene is a flammable liquid. Avoid open flames or excessive heat. Take cognizance of any dermatological, lachrymatory, or corrosive properties of samples and avoid skin contact and inhalation of vapors.

SCOPE

This method is intended for the determination of the cloud point of fluids which are transparent in layers 1-1/2" in thickness. It has the same general scope as ASTM D97-57 as applied to the determination of cloud points in petroleum products.

/jw
7/23/71

MATERIAL

Tetrahydrofuran
Tetrasodium ethylenediamine tetraacetate

ANALYSIS REQUIRED

Color of Liquids, Melts, and Solutions (APHA)

APPARATUS AND REAGENTS

a) Matched short form Nessler tubes of 50 ml capacity and tall form Nessler tubes of 100 ml capacity.

b) 0.05 M Periodic Acid - Dissolve 11.4 g. of $HIO_4 \cdot 2H_2O$ in water and dilute to 1000 ml.

c) Color Standards - Prepare APHA 500 standard by dissolving 1.0 g of cobaltous chloride ($CoCl_2 \cdot 6H_2O$) and 1.245 g of potassium chloroplatinate (K_2PtCl_6) in water, adding 100 ml of conc. HCl, and diluting to 1000 ml with water.

NOTE: The absorbance of the 500 platinum-cobalt stock solution must fall within the limits given below. The measurements shall be made in a cell having a 10 mm light path using a Beckman Model B Spectrophotometer at a sensitivity setting of one, or in another spectrophotometer having equivalent resolution and sensitivity. Reagent water in a matched cell shall be used as the reference solution.

Wavelength, mu	Absorbance
430	0.110 to 0.120
455	0.130 to 0.145
480	0.105 to 0.120
510	0.055 to 0.065

APPARATUS AND REAGENTS (cont'd)

From a burette add the amount of APHA 500 standard indicated in the following table to the respective tube, add 0.5 ml of 0.05 M periodic acid and dilute to the mark with double distilled water. If the marks on a series of tubes very in depth, a final adjustment of all standards to the depth of the shortest should be made. All tubes should be kept covered when not in use and wrapped in black paper so that no light enters from the side.

APHA No.	Short (50 ml.) Tubes APHA 500	Tall (100 ml.) Tubes APHA 500
5	--	1.0 ml.
6	--	1.2 ml.
7	--	1.4 ml.
8	--	1.6 ml.
9	00	1.8 ml.
10	1.0 ml.	2.0 ml.
11	--	2.2 ml.
12	--	2.4 ml.
13	--	2.6 ml.
14	--	2.8 ml.
15	1.5 ml.	3.0 ml.
16	--	3.2 ml.
18	--	3.6 ml.
20	2.0 ml.	4.0 ml.
25	2.5 ml.	5.0 ml.
30	3.0 ml.	6.0 ml.
35	3.5 ml.	7.0 ml.
40	4.0 ml.	8.0 ml.
45	4.5 ml.	9.0 ml.
50	5.0 ml.	10.0 ml.
60	6.0 ml.	12.0 ml.
70	7.0 ml.	14.0 ml.
80	8.0 ml.	16.0 ml.
90	9.0 ml.	18.0 ml.
100	10.0 ml.	20.0 ml.
125	12.5 ml.	
150	15.0 ml.	
175	17.5 ml.	
200	20.0 ml.	
250	25.0 ml.	
300	30.0 ml.	
350	35.0 ml.	
400	40.0 ml.	
450	45.0 ml.	

PROCEDURE

1. Fill a (black paper-wrapped) Nessler tube, using a 50 ml low form tube for off-white fluids or a 100 ml tall form tube for water-white fluids, to the depth of the standards with a portion of the sample, melt, or specified solution.

   NOTE: If the sample has any visible turbidity, filter it. (Note this on the analysis ticket).

2. Compare with the APHA standards viewing vertically against a white glass plate.

3. Record the number of the APHA standard which most nearly matches the sample, making a note of any abnormality in tint of clarity of the sample. In the event the color lies midway between two standards, report the darker of the two.

SAFETY

Take cognizance of any potential hazards in the material being tested, such as the temperature melts, corrosiveness, lachrymatory of dermatological properties, and exercise appropriate precautions.

PRECISION

Results should not differ from the mean by more than the following amounts:

Platinum-Cobalt Color	Repeatability One Operator & Apparatus	Reproducibility Different Operators & Apparatus
Under No. 50	2.5	5
No's. 50 to 100	5	10
Over No. 100	10	15

DISCUSSION

The function of the periodic acid is to stabilize the colors of the standards. It will not, however, protect against contamination. Hence, care must be exercised against splashing samples into the standards. In any event, the standards should be renewed monthly, unless obvious discrepancies dictate more frequent renewal.

The chart on page four shows some comparisons of color intensities as designated in various systems. In the comparison of color intensities of the ASTM D-1500 and the NPA systems, the asterisk (*) indicates the colors are not matched but they are close approximations. See also, references 3, 4.

SCOPE

This method is intended for the estimation of the color of liquids in terms of platinum-cobalt (Hazen) standards established by the American Public Health Association (ref. 1). It involves visual comparisons of the sample with prepared standards in matched Nessler tubes.

REFERENCES

1. "Standards Methods for the Examination of Water, Sewage, and Industrial Wastes", American Public Health Association, Inc., New York, 1955.

2. 1968 ASTM Standards, part 20, pages 555 and 817.

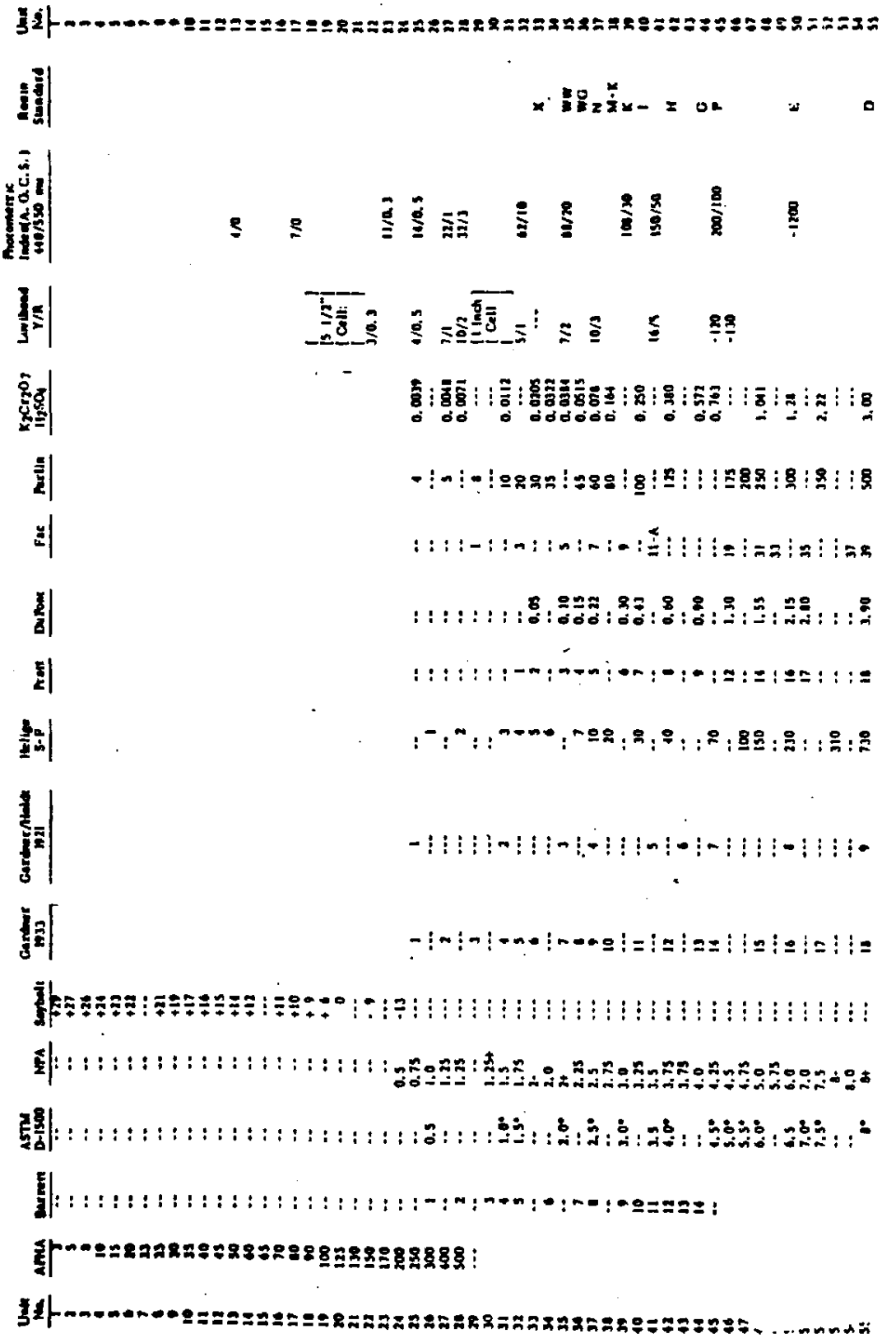
3. Federal Standard Stock Catalog, Section IV, Part 5, TT-P-141b, Method 458.1.

4. Quality Assurance Register Directory/Handbook and Specifications Catalog, Hitchcock Publishing Company, Hitchcock Building, Wheaton, Illinois 60187.

/jw
7/26/71

COLOR OF LIQUIDS, MELTS, AND SOLUTIONS (APHA)

COLOR INTENSITY COMPARISONS



MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.443
May 31, 1971

MATERIAL

Tetrahydrofuran

ANALYSIS REQUIRED

Distilling range

APPARATUS

a. 100 ml Engler flask
b. + 50°C to +70°C, grade AA, 76 mm immersion thermometer graduated in 0.1°C
c. Asbestos ring with 1 1/4 inch diameter
d. 100 ml graduated cylinder
e. 9/16" O.D. No. 20GA seamless brass tubing 2 feed long for condenser
f. Bunsen burner, ring stand and steam bath
g. Usually laboratory equipment
h. Apparatus setup pictured in Figure 1

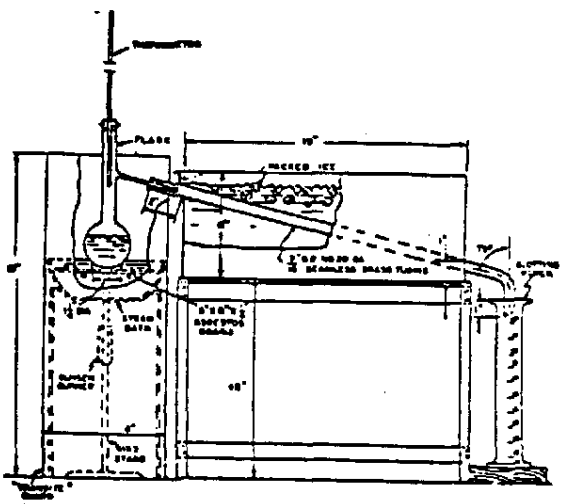


Figure 1
BOILING RANGE APPARATUS

PROCEDURE AND CALCULATION

Prior to conducting the boiling range test, use Method 602.446 to check the THF sample for THF-peroxide. If more than trace amounts of peroxide are present, add a few caustic pellets to the distillation flask to be used in the determination and proceed with the following test.*

Measure a 100 ml sample of THF at approximately 20°C, into a standard 100 ml Engler flask. Insert a +50°C to +70°C, grade AA, 76 mm immersion thermometer graduated in 0.1°C. (Wm. Hiergesell & Sons Catalog No. 6536) into the neck of the Engler flask so that the top of the mercury in the expansion bulb is level with the inside of the bottom of the flask vapor outlet tube. Support the flask by an asbestos ring or a "Transite"[2] plate having a 1 1/4 inch diameter hole and assemble the distillation apparatus as shown in Figure 1. Place a 100 ml graduated cylinder at the outlet of the condenser tube with the tube extending at least one inch into, but not below the 100 ml mark of, the cylinder. Cover the top of the graduated cylinder closely with blotting paper cut to fit the condenser tube tightly.

Heat the contents of the flask over an enclosed steam bath, taking care that the water in the steam bath is actively boiling before the flask is placed in position.

Place a "Transite" board guard on all sides around the legs of the steam bath except that facing away from the condenser assembly.

Record as the initial boiling point the temperature at which the first drop of condensate falls from the end of the condenser tube. Adjust the receiving graduate so that the end of the condenser tube touches the side of the cylinder and continue distilling. Maintain the rate of distillation between 3 ml and 5 ml per minute and record the temperature at which 95 ml has been collected, halt the distillation.

Record the barometric pressure and room temperature at the time of distillation and correct the recorded boiling range temperatures to 760 mm of mercury according to the following formula:

temperature correction (°C) = (760 - barometric reading reduced to 0°C) x 0.041

* Based on ASTM D 1078-58 test method.

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.444
May 31, 1971

MATERIAL

Tetrahydrofuran

ANALYSIS REQUIRED

Specific Gravity

APPARATUS

Westphal balance

PROCEDURE

Determine the specific gravity at 20°C by means of a Westphal balance which has been standardized with distilled water at 4°C.

/jw
7/26/71

MONSANTO COMPANY
TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.445
May 31, 1971

MATERIAL

Tetrahydrofuran

ANALYSIS REQUIRED

Moisture

APPARATUS

Usual laboratory equipment including 100 ml titrating buret.

REAGENTS

Anhydrous methanol
Karl Fisched reagent

PROCEDURE AND CALCULATIONS

Titrate 50 ml of anhydrous methanol to a permanent deep-red color with Karl Fischer reagent. Add a 10 ml sample of tetrahydrofuran and titrate immediately to the same endpoint with Karl Fischer reagent, using an "Aquameter" or the visual endpoint. Calculate percent moisture by use of the following equation.

$$\% \text{ moisture} = \frac{(\text{ml reagent})\ (\text{water equivalent}) \times 100}{(\text{specific gravity}) \times 10}$$

/jw
7/26/71

MATERIAL

Tetrahydrofuran

ANALYSIS REQUIRED

Peroxide

APPARATUS

a. 2 x 250 ml Erlenmeyer flasks and stoppers
b. 25 ml graduated cylinders
c. Titrating buret and equipment

REAGENTS

a. 1:4 concentrated sulfuric acid to water
b. 10% potassium iodide solution
c. 0.02 N sodium thiosulfate

PROCEDURE AND CALCULATION

Unstabilized tetrahydrofuran upon exposure to air may slowly form THF hydroperoxide, also designated as THF-peroxide. A simple, accurate analysis for THF-peroxide may be made as follows:

1. Place 100 ml of distilled water in each of two Erlenmeyer flasks.

2. To each flask add 25 ml of a 1:4 mixture of concentrated sulfuric acid and water, and then add 25 ml of a 10% potassium iodide solution.

3. Add a 25 ml sample of tetrahydrofuran to one flask, stopper both flasks, shake, and place in the dark for 15 - 20 minutes.

4. Titrate the contents of each flask with 0.02 N sodium thiosulfate until the solutions become colorless.

PROCEDURE AND CALCULATION

5. Calculate the THF-peroxide content of the THF sample by by use of the following formula:

$$\% \text{ THF peroxide} = \frac{(\text{ml thio in sample} - \text{ml thio in blank}) \times \text{N of thio} \times 0.23}{}$$

/jw
7/26/71

MATERIAL

Tetrahydrofuran

ANALYSIS REQUIRED

Carbonyl and hydroxyl compounds

APPARATUS

a. Vapor fractometer equipped with one millivolt recorder
b. 6 ft column filled with 15% carbowax 1500 on 80-100 mesh chromosorb W
c. 60 ml serum bottle
d. 0.05 ml Hamilton syringe fitted with Chaney adapter
e. Usual laboratory equipment

REAGENTS

a. Helium
b. Acetone
c. Isopropyl alcohol
d. n-propyl alcohol
e. n-butyl alcohol

PROCEDURE AND CALCULATIONS

The amounts of acetone, isoproply alcohol, n-propyl alcohol, and n-butyl alcohol in tetrahydrofuran may be determined by gas chromatography, using a 6-ft column filled with 15% "Carbowax" 1500[4] on 80-100 mesh "Chromosorb" W.[2] Apparatus suitable for this determination includes a vapor "Fractometer" (equipped with a 1-millivolt recorder)[3] or an equivalent gas chromatograph. The column may be purchased as a complete unit (Perkin-Elmer type "K" column or equivalent) or prepared in accordance with instructions outlined below.

To prepare the column, dissolve 3.5 g of "Carbowax" 1500 in 75 ml of methylene chloride and mix with 20 g of 80-100 mesh "Chromosorb" W to make a slurry. Evaporate the methylene chloride on a steam plate with occasional stirring to obtain the dried adsorbent. Fill a 6 ft (1/4" o.d.)

PROCEDURE AND CALCULATIONS (cont'd)

thin-walled tube of stainless steel with the dried adsorbent, tapping the tube continually while filling to insure uniform packing. Plug the ends of the tube with glass wool and insert into the "Fractometer", bending the tube as required to fit.

Prepare a standard sample by adding 50 ml of THF known to contain only minute quantities of other components to a tared serum bottle (60 ml) fitted with a rubber stopper. Into the THF standard, measure 0.01 ml acetone, 0.01 ml isopropyl alcohol, 0.01 ml n-propyl alcohol, and 0.02 ml n-butyl alcohol. Calculate the percentage by weight of each constituent added to the THF standard.

Before operating the "Fractometer", adjust the detector to 8.0 volts and sensitivity 1, and allow the "Fractometer" to attain a temperature of 73°C. Apply a helium gas pressure of 15 psi at a flowmeter reading of 5.5 (70 ml/min of helium), and inject 0.01 ml of the THF standard into the "Fractometer" by means of a Hamilton syringe (0.05 ml) fitted with a Chaney adapter. As the carrier gas passes through the "Fractometer", the components of the sample appear at the detector after various lengths of time according to their partition coefficients. The times of elution of the components in the sample are plotted automatically against recorder response on the recorder chart (Figure 2). Deflections in recorder response are indicated on the chart as peaks characteristic of each component.

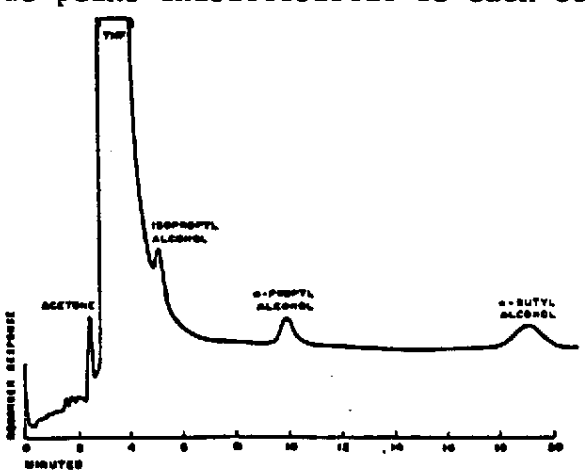


Figure 2
TYPICAL CHROMATOGRAPH FOR THF

PROCEDURE AND CALCULATIONS (cont'd)

Repeat the chromatographic procedure, injecting a 0.01 ml sample of the original THF into the "Fractometer". Measure the heights of the acetone, isopropyl alcohol, n-propyl alcohol, and n-butyl alcohol peaks and compare with the THF standard. Calculate the percentage by weight of each component in the standard in accordance with the following equation:

$$\% \text{ in std} = \frac{\% \text{ added} \times \text{peak height (std)}}{\text{peak height (std)} - \text{peak height (sample)}}$$

Chromatograph the samples of THF to be analyzed under the conditions specified above, and measure the peak heights corresponding to each component. Calculate the percentage by weight of component in the unknown by means of the following equation:

$$\% \text{ in sample} = \frac{\% \text{ (std)} \times \text{peak height (sample)}}{\text{peak height (std)}}$$

/jw
7/26/71

MATERIAL

Tetrasodium ethylene diamine tetraacetate

ANALYSIS REQUIRED

Heavy metals (Pb)

APPARATUS

A. Matched 100 ml low form Nessler tubes.

B. pHydrion test papers, 3.0 to 5.5 (Micro Essential Laboratory) or glass electrode pH indicator.

REAGENTS

A. 6% acetic acid
B. Saturated hydrogen sulfide water
C. Concentrated ammonium hydroxide
D. 10% hydrochloric acid
E. Redistilled acetone
F. Lead stock solution (100 mg/l) - Dissolve exactly 0.1598 gram of lead nitrate in 100 ml of water containing 1 ml of conc. nitric acid and dilute to 1 liter in a volumetric flask.
G. Standard lead solution (10 mg/l) - Accurately pipette 10.00 ml of the 100 mg/l stock solution into a 100 ml volumetric flask and dilute to volume with water. Prepare fresh daily.
J. 0.1% p-Nitrophenol indicator solution

DETERMINATION

1. Ascertain into which the following classifications the product being tested falls and treat as indicated:

   a. Soluble Products: Weigh 2.0 grams (see note on page 2) into a 100 ml Nessler tube and treat as directed in the table.

DETERMINATION (cont'd)

If the product under test is not listed but is soluble in water or acetone, dissolve in 25 ml of acetone or water; if acid or basic adjust to pH 3.5 with NH_4OH of 10% HCl, or if neutral add 4 ml of 6% HOAc and dilute to 50 ml with the alternate solvent to obtain an approximate 50% aqueous-acetone solution. (Some products may tend to separate on dilution. This difficulty can sometimes be overcome by a judicious selection of acetone-water ratio, taking care to duplicate this ratio in preparation of the standard (Step 4). Alternately, the product may be treated as described below under b, c, d or e.)

NOTE: With strong buffers such as the glycerophosphates, start with 1.0 gram of sample and 50 ml of water, omit acetone from both sample and standard (Step 4) and adjust to pH 3.5 by glass electrode.

Product	Dissolve In	Add	Dilute to 50 ml with
Aspirin	25 ml acetone	water	water
Aminoacetic Acid (Glycine)	25 ml water	4.0 ml 6% HOAc	acetone
Amobarbital	25 ml acetone	water	water
Benzoic Acid	25 ml acetone	water	water
Caffeine	20 ml hot H_2O	4.0 ml 6% HOAc	acetone
Caffeine Citrate	25 ml water	0.5 ml NH_4OH	acetone
Calcium Carbonate	20 ml 10% HCl	NH_4OH to pH 3.5	acetone
Calcium Chloride	20 ml water	4.0 ml 6% HOAc	acetone
Calcium Cyclamate	25 ml water	10% HCl to pH 3.5	acetone
Calcium Saccharin	25 ml water	10% HCl to pH 3.5	acetone
Chloramphenicol	25 ml acetone	4.0 ml 6% HOAc	water
Chlorothenyl-pyramine Citrate (Chlorothen Citrate)	25 ml water	1.0 ml 10% HCl	acetone

Product	Dissolve In	Add	Dilute to 50 ml with
Maleic Anhydride	25 ml water	NH_4OH to pH 3.5	acetone
Phenacetin	25 ml acetone	4.0 ml 6% HOAc	water
Saccharin Insoluble	25 ml acetone	0.8 ml NH_4OH	water
Salicylamide	25 ml acetone	4.0 ml 6% HOAc	water
Salicylic Acid	25 ml acetone	1.0 ml NH_4OH	water
Sodium Acetate	20 ml water	4.25 ml 10% HCl	acetone
Sodium Benzoate	25 ml water	3.5 ml 10% HCl	acetone
Sodium Carbonate	15 ml water	10.0 ml 10% HCl	acetone
Sodium Cyclamate	25 ml water	10% HCl to pH 3.5	acetone
Sodium Saccharin	25 ml water	0.25 ml 10% HCl	acetone
Sodium Salicylate	25 ml water	1.0 ml 10% HCl	acetone
Sodium Secobarbital	25 ml water	2.5 10% HCl	acetone
Sulfanilamide	25 ml acetone	4.0 ml 6% HOAc	water
Sulfathiazole	25 ml acetone	4.0 ml 6% HOAc	water
Sulfuric Acid	25 ml water	NH_4OH to pH 3.5	acetone
Thenylpyramine Fumarate (methylpyrilene Fumarate)	25 ml water	4.0 ml 6% HOAc	acetone
Thenylpyramine Hydrochloride (Methapyrilene HCl)	25 ml water	4.0 ml 6% HOAc	acetone
Urea	25 ml water	4.0 ml 6% HOAc	acetone

b. Insoluble Powders - (such as Actamer, Phthalyl Sulfathiazole, Succinyl Sulfathiazole, Theobromine): Moisten 2.0 grams with 10 ml of 10% HCl in a 100 ml beaker, hold on the steam bath for 5 minutes, cool to about 20°C, let stand until the supernatent liquid is clear, filter by vacuum and wash the residue with a small amount of water.

To the filtrate add 1 drop of 0.1% p-nitrophenol, titrate to a faint yellow endpoint with NH_4OH, add 4.0 ml of 6% HOAc and 25 ml of acetone, and dilute to 50 ml with water.

c. Oils (such as Methyl Salicylate): Extract 2.0 grams with 10 ml of 10% HCl in a 60 ml separatory funnel and filter the aqueous layer through a small circle of wet filter paper.

To the extract add 1 drop of 0.1% p-nitrophenol, titrate to a faint yellow endpoint with NH_4OH, add 4.0 ml of 6% HOAc and 25 ml of acetone, and dilute to 50 ml with water.

d. Colored Products (such as MHA or Kalcolor Acid): Moisten 2.0 grams with 4 ml sufluric acid in a 250 ml beaker. Hold the beaker with tongs and swirl until any initial reaction subsides. With constant swirling, heat to boiling over a moderate flame to carbonize. Remove the flame and add 30% hydrogen peroxide ((Superoxol) DROP-WISE from a 15 ml pipette, allowing the reaction to subside somewhat between drops. When there is noticeable diminution in the vigor of the reaction upon addition of hydrogen peroxide, heat cautiously to fumes, remove the heat and resume addition of hydrogen peroxide, cool the beaker in ice, add 10 ml of water and 1 drop 0.1% p-nitrophenol. Neutralize to a faint yellow endpoint with ammonium hydroxide, add 4 ml of 6% acetic acid and dilute to 50 ml with water.

e. Volatile Products (such as Acetic Anhydride): Weigh 2.0 grams into a clean evaporating dish and take to dryness on the steam bath. Dissolve the residue in 4 ml of 6% acetic acid, add 25 ml acetone and dilute to 50 ml with water.

2. Add (an additional) 25 ml of acetone to the prepared solution.

3. Verify the pH by a spot test on pHydrion paper, adjusting to 3.5 with NH_4OH or 10% HCl if necessary.

DETERMINATION (cont'd)

4. Prepare a standard by pipetting exactly 1.00 ml of 10 mg/l standard lead solution for each 5 ppm of the heavy metals specification, into a second Nessler tube containing 20 ml of water, 4 ml of 6% acetic acid, and the same quantity of acetone as is in the sample solution. (NOTE: if a 1.00 gram sample was taken, use 0.5 ml of standard lead solution for each 5 ppm of the heavy metals specification).

5. Dilute the standard and sample solutions to 75-80 ml with water.

6. Add 20 ml of saturated hydrogen sulfide water to the standard and to the sample solution, dilute both solutions to 100 ml with water, mix well and note the time.

7. Exactly 10 minutes after mixing the solutions compare longitudinally over a white, well lighted background.

8. Record the sample as containing less, equal to, or more than the ppm of heavy metals represented by the comparison standard, depending on whether the sample solution is lighter, equivalent to, or darker than the standard solution.

SAFETY

Prepare hydrogen sulfide water in an efficient fume hood and avoid inhalation during use. Conduct wet digestions (Determination, step 1, part d) in an efficient fume hood and behind a safety shield.

DISCUSSION

This method is essentially equivalent to the USP limit test, with no provision for compensating for inherent sample color. It is not directly applicable to ferric and manganese glycerophosphates owing to complex precipitation and oxidation of the sulfides.

DISCUSSION (cont'd)

Although some indication is obtained for most metals of the copper and arsenic groups, the method is most sensitive to lead. Thus, for example, the absorptivity of an equivalent amount of copper sulfide is about half that for lead. Since the test is intended primarily for the detection of toxic metals, the inclusion of iron is avoided by conducting the test in acetate solution below pH 5.

Glassware used in this test should be rinsed with concentrated hydrochloric acid and distilled water before use. Even reagent grade acetone should be redistilled. Oxidizing agents, such as traces of chlorine in the diluent water used, will cause the precipitation of elemental sulfur and invalidate the results.

REFERENCES

1. Stewart, F. N. and Strode, C. W., J. Am. Pharm. Assoc., Sci. Ed., 41, 242 (1952)

2. U.S.P. SVII.

SCOPE

This method is intended for the detection of apparent heavy metal contamination beyond a designated amount in fine chemicals. It involves formation of the sulfides at pH 3.5 and visual comparison with a standard.

/jw
7/26/71

MATERIAL

Acrylamide
Sodium Lauryl Sulfate

ANALYSIS REQUIRED

Iron

APPARATUS

a. Usual laboratory apparatus and reagents including matched 50 ml short form Nessler tubes.

b. Colorimeter such as the Bausch and Lomb Spectronic 20, with matched 1" test tube cells.

REAGENTS

a. 15% Ammonium Thiocyanate - Dissolve 75 g of ammonium thiocyanate A.R. in water and dilute to 500 ml.

b. 100 mg/l Iron - Clean primary standard iron wire with emery paper and cleansing tissue, holding with tweezers. Accurately weigh 0.1000 g into a 1000 ml volumetric flask, add 10 ml HCl, heat on a steam bath until dissolved, add 1.0 g. ammonium persulfate, and dilute to volume with distilled water at room temperature.

c. 5 mg/l Iron - Pipette 25 ml of 100 mg/l iron into a 500 ml volumetric flask and dilute to volume with water.

d. Acetone, A.R. Grade

CALIBRATION

1. Pipette 0, 5, 10, 20, 25, and 30 ml aliquots of 5 mg/l iron solution into separate 50 ml glass stoppered cylinders, add 2 ml of conc. HCl and dilute each solution to about 35 ml with distilled water. (After treatment and dilution to volume, these solutions will represent concentrations of 0, 0.5, 1.0, 1.5, 2.0, 2.5, and 3.0 mg/l.)

CALIBRATION (cont'd)

2. To each solution add 0.05 g of ammonium persulfate, 5.0 ml of 15% ammonium thiocyanate, and 5 ml of acetone; then dilute to volume with water and mix well.

3. Using 1" test tube cells in the Spectronic 20 colorimeter and a wavelength setting of 475, immediately measure the absorbance of the 1.5 mg/l solution against the 0, 0.5, and 1.0 mg/l solutions; measure the absorbances of the 2.0, 2.5, and 3.0 mg/l solutions against the 1.5 mg/l solution.

4. Plot the absorbances against concentrations on coordinate paper to obtain a "V" shaped curve with the apex at 1.5 mg/l Divide (1.5 - respective solution concentration) by the absorbances of the first three solutions, divide (respective solution concentration - 1.5) by the absorbances of the last three solutions, and average the results as calibration constant K.

   NOTE: For JFQ Lab Spectronic 20 Colorimeter, Serial No. RD 8233, calibration of 12-2-55, K = 4.7.

DETERMINATION

A-1 - Spectrophotometrically

1. Prepare 35 ml of clear, colorless, oxidized extract or solution of the sample or ash containing the equivalent of 2 ml of free conc. HCl in a 50 ml glass stoppered cylinder, as directed for the product being tested.

   NOTE: The optimum sample size in grams is between 25 and 125 divided by the expected iron content in ppm.

2. Add 0.5 g of ammonium persulfate, 5.0 ml of 15% ammonium thiocyanate, and 5 ml acetone; then dilute to 50 ml with water and mix well.

3. Concurrently prepare a separate mixture of 15.0 ml of 5 mg/l iron solution, 2 ml of conc. HCl, 0.05g of ammonium persulfate, 5.0 ml of 15% ammonium thiocyanate, and 5 ml acetone; then dilute to 50 ml with water and mix well.

DETERMINATION

4. Immediately measure the absorbance of the darker of the two solutions against the lighter solution in the Spectronic 20 colorimeter, using 1" test tube cells and a wavelength setting of 475.

5. Calculate:

$$\text{ppm Fe} = \frac{50\ (1.5 \pm KA)}{\text{sample wt. in solution}}$$

(% - ppm/10,000)

where:

K = the calibration constant, which has a positive value when the sample solution is darkest and a negative value when the 1.5 mg standard solution is darkest.

and

A = the absorbance found

A-2 - Visually

1. Prepare 35 ml of clear, colorless oxidized extract or solution of the sample or ash containing the equivalent of 2 ml of free conc. HCl in a 50 ml Nessler tube, as directed for the product being tested.

2. Add 0.05 g of ammonium persulfate and 5.0 ml of 15% ammonium thiocyanate, and 5 ml of acetone.

3. To a blank of 35 ml of water and 2 ml of conc. HCl in a second Nessler tube add 0.05 g of ammonium persulfate, 5.0 ml of 15% ammonium thiocyanate and 5 ml of acetone.

4. Using a Mohr pipette, titrate the blank with 5 mg/l iron solution, observing the tubes transversely over a white background, until the color of the blank matches that of the sample.

DETERMINATION (cont'd)

5. Calculate:

$$\text{ppm Fe} = \frac{\text{ml iron solution} \times 5}{\text{sample wt. in solution}}$$

(% = ppm/10,000)

DISCUSSION

In condentrations between approximately 0.1 and 100 ppm of iron in various materials the precision and accuracy of this method may vary from 5 to 10% of the amount present in the absence of interferences.

Fluoride, pyrophosphate and oxalate interfere, as may colored ions, metals such as mercury or zinc which react with thiocyanate, or substances present in sufficient amount to exhibit color before the addition of thiocyanate. High salt concentration may inhibit color formation, and strong light may cause fading. The color is not stable for more than 30 minutes.

SCOPE

This method is intended for the determination of small amounts of iron in aqueous solutions of soluble colorless materials, ashes or extracts. It involves formation of the ferric thiocyanate complex and colorimateic estimation of the concentration.

SAFETY

With proper regard to observance of handling instructions on bottles of ammonium persulfate, no unusual hazards are involved in this operation.

REFERENCE

Snell and Snell, "Colorimetric Methods of Analysis", Vol. II, D. Van Nostrand Company, Inc., New York, 1954.

/jw
7/26/71

MATERIAL

Vinyl Chloride Monomer (VCM)

ANALYSIS

Heavy-end Impurities:

MVA

Ethyl Chloride

2-CPY

Vinylidene Chloride (1, 1-DCY)

t-1,2-DCY

CB

SAMPLE

Liquid Sample in VCM Bomb

APPARATUS

P. E. Model 154-C Vapor Fractometer equipped with gas inlet valve, or equivalent, valved for multi-column operation (See attached drawing.)

Hydrogen Flame Ionization Detector - capable of detecting 2×10^{-10} moles of chlorinated compounds.

Meeco Vaporizing Valve, Type 1S, or equivalent, for flashing liquid VCM into gas sample loop of chromatograph.

Flow-meter for determining rate of purge on sample loop.

Recorder - L. & N. Speedomax Type G, or equivalent, with 1 second pen and 5 mv full-scale deflection. Recorder should be modified to switch to either the thermistor detector or the flame detector.

REAGENTS AND SUPPLIES

Aluminum Tubing - 3/16 inch O.D.

Helium - 2 regulated supplies.

Chromosorb - red, 30/60 mesh.

Narcoil 40 - dinonyl phthalate from National Research Corp.

Hydrogen - electrolytic grade.

Air - well-filtered, clean supply.

Ethyl chloride - commercial grade.

2-Chloropropene - Columbia Organic Chemical Co., purified by gas chromatography.

2-Chloro-1,3-butadiene - Monomer-Polymer, Inc., purified by gas chromatography.

1,1-Dichloroethylene - Monomer-Polymer, Inc., purified by gas chromatography.

Vinylacetylene - E. I. DuPont Co., purified by gas chromatography.

Vinyl Chloride - Free of, or very low concentration of, impurities.

Acetone - for column packing preparation.

PROCEDURE

A. Column Preparation

1. Mix 15 grams of Narcoil 40 with approximately 100 ml of acetone.

2. Pour mixture over 85 grams of 30/60 mesh chromosorb and add more acetone if necessary to make a slurry.

3. Mix continuously while evaporating the acetone. If available, use rotary-film evaporator with baffled flask for mixing. Apply heat from hot air gun and vacuum to flask to aid in evaporation.

PROCEDURE (cont'd)

4. When the reaction is completed, re-screen to remove fines and pack one four-foot length and one 35-foot length of 3/16-inch tubing with the 30/60 mesh fraction.

5. Install the columns in the instrument as shown on the attached drawing.

6. Adjust the two helium pressures and restrictor valve to give the following flows:

   a. Column I: 75 ml/minute.

   b. Column II (with restrictor in flow path): 85 ml/min.

   c. Columns I & II (in backflush position): 85 ml/min.

7. Adjust temperature to 50° C. and allow instrument and columns to reach equilibrium.

8. Light the hydrogen burner and adjust the hydrogen and air flows so as to yield maximum signal-to-noise ratio when MVA passes through it.

9. Turn on amplifier and recorder and set for maximum sensitivity. The instrument should show no drift for at least five minutes before it is ready for use.

B. Analysis

1. Purge the 5 cc sample loop for at least one minute with sample flashed through the Meco Vaporizing Valve, then inject the sample into the flowing carrier stream with the columns in Position A and the recorder turned to the thermal conductivity detector. The attenuator switch of the detector should be set on an attenuation of 2. The valve between the thermal detector and the flame detector should be in the vent position to prevent VCM from going to the flame detector.

2. Allow the sample to flow in Column I as shown in Position A for approximately three minutes (valve should be switched when recorder pen reaches 50% of full scale coming down on VCM peak). This allows the components lighter than VCM and most of the VCM to be vented from Column I.

PROCEDURE (cont'd)

3. Just before MVA is eluted from Column I, switch the valve to Position B. This allows everything behind the VCM peak to be backflushed onto Column II where they are separated and then flow to the detector. Sometime after backflushing and prior to the backflushed VCM being eluted from Column II, switch the recorder to the flame detector and the valve between the thermal detector and the flame detector to the position such that the flow is through the flame detector.

4. Record all peaks eluted from Column II. A typical chromatogram is attached.

CALCULATIONS

The concentration of each component is calculated as follows:

1. Measure the peak height and width at one-half the peak height using a scale of 50 divisions to 1 inch.

2. Calculate the area of each peak according to the following equation:

   area =

   peak height x peak width at one-half peak height x attenuation

3. Calculate the concentration of each component as follows:

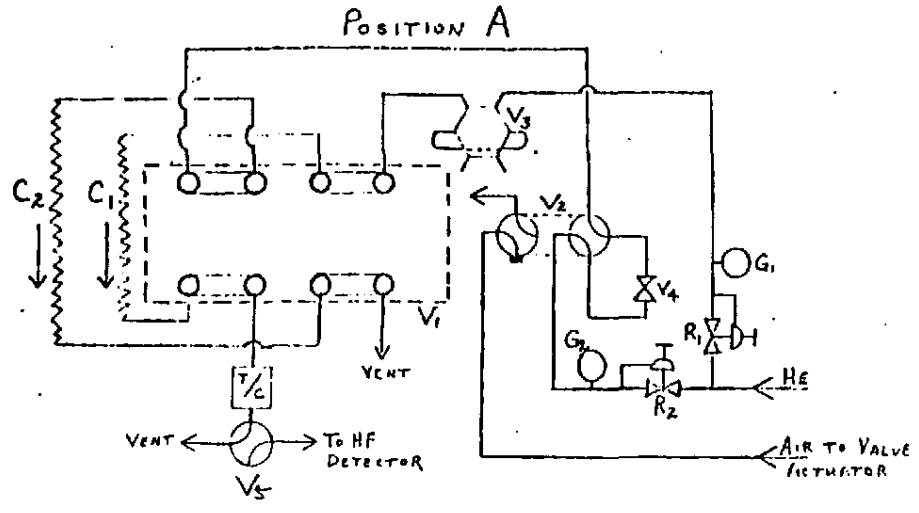
   $\text{Concentration}_{(ppm)}$ = peak area x factor

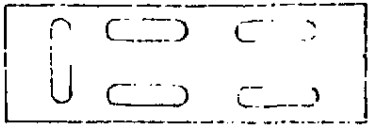
PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare synthetics of MVA, ethyl chloride, 2-CPY, 1,1-DCY. trans-1,2-DCY, and CB in VCM to cover the concentration range of intrest. At least two synthetics should be prepared in each concentration range. Synthetics should be prepared in clean, stainless steel bombs of any convenient size. The pressure should be sufficient to permit extended use of the mixtures. The blending system used must be completely free of leaks and all kinds of foreign materials.





View of Channel Locations in Teflon Slide for Wall 8-Port Valve

V_1 - Wall 8-Port Valve
V_2 - Circle Seal Double 4-Port Valve
V_3 - 6-Way Gas Chromatic Valve
V_4 - Needle Valve
C_1 - Column I
C_2 - Column II
$R_{1,2}$ - Helium Regulators
$G_{1,2}$ - Pressure Gauges, 0-60 psi
V_5 - Circle Seal 4-Port Valve

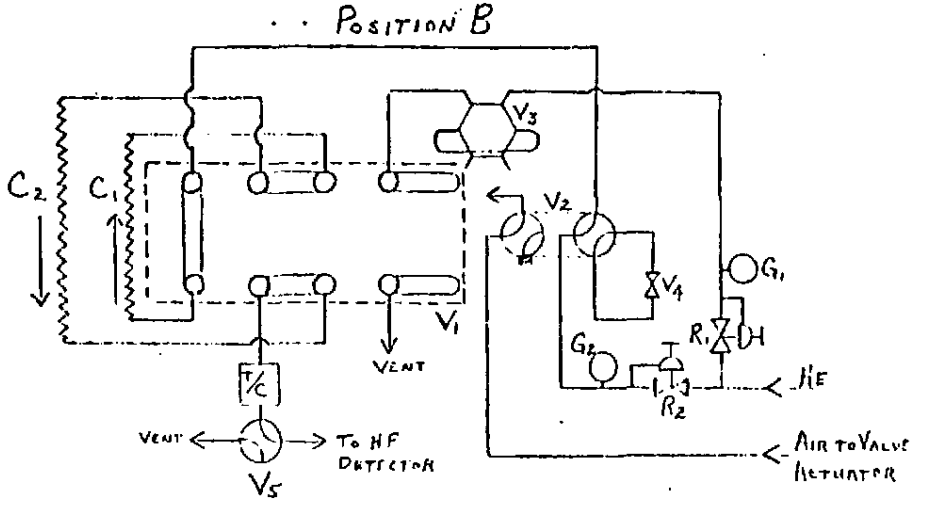
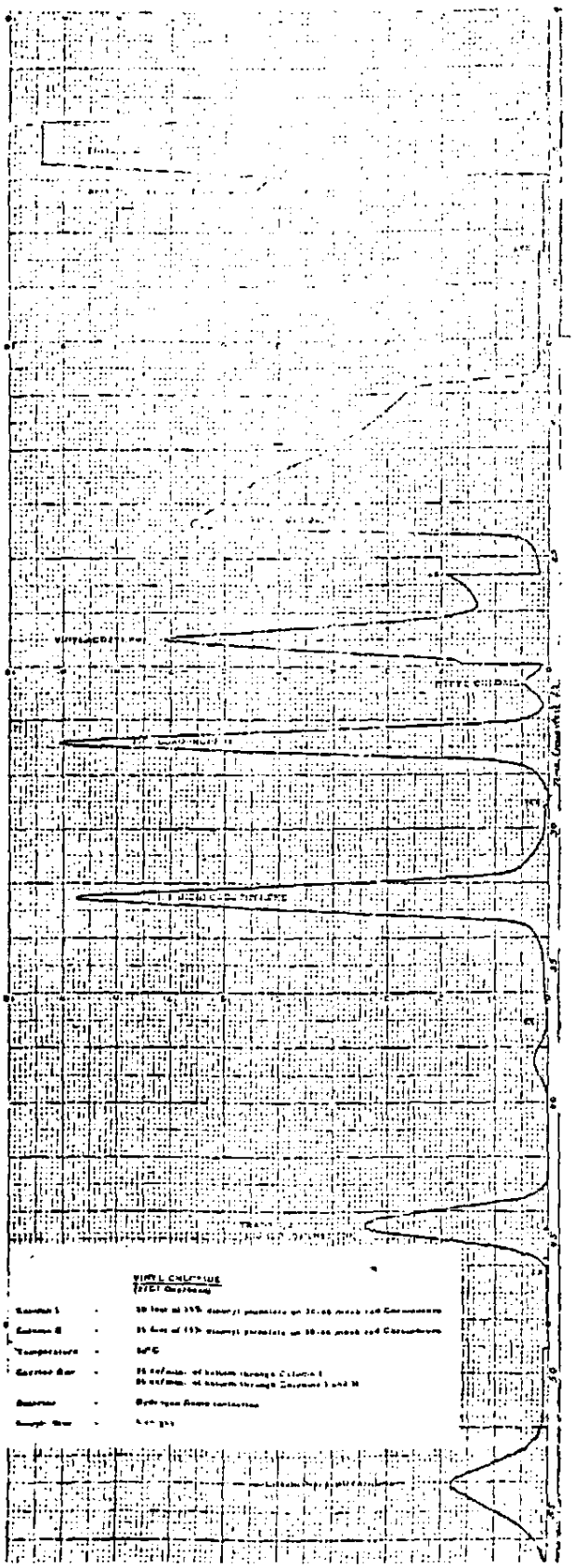


Figure II



MONSANTO COMPANY
TEXAS CITY, TEXAS

MATERIAL

Vinyl Chloride Monomer (VCM).

ANALYSIS

Light-end Impurities:

C_1 - C_4 Hydrocarbons
Methyl Chloride

SAMPLE

Liquid sample in VCM bomb.

APPARATUS

Gas chromatograph equipped with valve(s) for multi-column operation and two regulated helium supplies. (See Note)

Hydrogen flame ionization detector, Barber-Colman Model A-4504 or equivalent: sensitivity should be approximately one coulomb per mole.

Amplifier and Recorder: Over-all sensitivity should be sufficient to yield full-scale deflection for 1×10^{-11} amp with a noise level of less than 1×10^{-13} amp. Barber-Colman Electrometer for above flame detector is recommended.

Integrator: Perkin-Elmer Printing Integrator, or equivalent.

Tubing: 3/16" O.D. Aluminum.

Pressure Regulators: Two for helium, one for hydrogen, and one for air.

Meeco Vaporizing Valve, Type 1S, or equivalent, for flashing liquid VCM into gas sample loop of chromatograph.

REAGENTS

Helium - Two regulated supplies.

Chromosorb - red, 30/60 mesh.

Trioctyl Phosphate: Union Carbide Chemical Company.

REAGENTS (cont'd)

Tributyl Phosphate: Fisher Scientific.

C_1 - C_4 Hydrocarbons: Phillips Research Grade.

Methyl Chloride: The Matheson Company.

Vinyl Chloride Monomer: Free of, or very low concentration of, impurities.

Hydrogen: Electrolytic Grade.

Air: Purified source, such as cylinder breathing air.

Acetone: For column packing preparation.

PROCEDURE

A. Column and Instrument Preparation

1. Prepare two columns as follows:

   a. Column I: 20 feet of 15% w/w Trioctyl Phosphate on 30/60 mesh red chromosorb packed in 3/16" O.D. tubing.

   b. Column II: 45 feet of 15% w/w Tributyl Phosphate on 30/60 mesh red chromosorb packed in 3/16" O.D. tubing.

2. Install the columns in the gas chromatograph as shown in Position A and adjust the helium pressure on Column I to give a flow of 100 cc/minute. When the valve is switched to Position B, the flow through Columns I and II should be 100 cc/minute. The columns are operated at room temperature.

3. Light the hydrogen burner and adjust the hydrogen and air flows so as to yield maximum signal-to-noise ratio when a C_3 or C_4 hydrocarbon passes through it.

4. Turn on amplifier and recorder and set at maximum sensitivity. The instrument should show no drift for at least five minutes before it is ready for use.

5. Prepare the integrator for use.

PROCEDURE (cont'd)

b. Analysis

The analysis of light-ends may be made using the following procedure:

1. Line-out instrument in Position B, then switch to Position A for entering sample.

2. Purge the 5 cc sample loop for at least one minute with sample flashed through the Meeco Vaporizing Valve, then inject the sample into the flowing carrier stream with the columns in Position A.

3. Fifteen (15) seconds after entering the sample, switch the valve to diagram Position B. This allows the entire sample to be backflushed onto Column II where the components are separated and then flow to the detector. Using the integrator, record areas for all peaks eluted ahead of VCM.

4. When VCM begins to be eluted, switch the valve to diagram Position A. This allows the VCM to be vented instead of passing through the detector.

CALCULATIONS

The concentration of each light-end component is calculated as follows:

ppm = attenuation x peak area x factor

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare synthetics of the light-end impurities, hydrocarbons and methyl chloride, in VCM to cover the concentration range of interest. At least two synthetics should be prepared. Synthetics should be prepared in clean stainless steel bombs of any convenient size. The pressure should be sufficient to permit extended use of the mixtures. The blending system must be clean and completely free of leaks.

CALIBRATION (cont'd)

2. Analyze synthetic mixtures as described under the Procedure for Analysis.

3. Calculate factors for each of the light-end impurities according to the following equation:

$$\text{factor} = \frac{\text{concentration of component in synthetic}}{\text{integrated area x attenuation}}$$

SAFETY PRECUATIONS

VCM is a toxic, volatile, and highly flammable material and should be treated as such. Sample used to purge sample loop should be vented outside of building.

ANALYTICAL TIME

The average analytical time for this analysis should be 0.5 hour.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits using this method is ± 10% of the amount present.

Report results to nearest 0.1 ppm.

SCOPE

This method is designed for light-end impurities in VCM; however, it may be used for the analysis of light-end impurities in volatile pure grade hydrocarbons as long as the impurities are hydrocarbons in the C_1 - C_4 range.

REFERENCE

Monsanto Research Notebook - No. 2330, pp. 154660-154665.

NOTE:

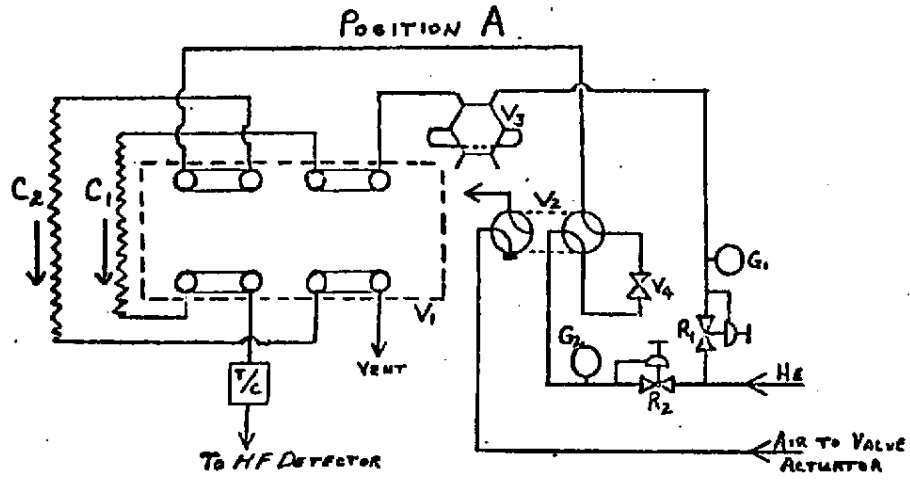
Valving recommended for this procedure is as follows:

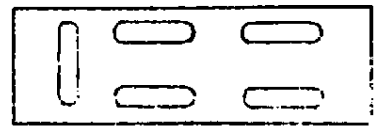
a. Backflushing and column switching may be done with a Wall 8-Port Valve purchased from Tycam Engineering and Manufacturing Co., Inc., Houston, Texas. A drawing (may be a rough sketch) should be furnished to indicate number and location of channels in teflon slide.

NOTE (cont'd)

b. A double 4-Port Circle Seal Valve may be used for the control of air to the air actuator of the Wall Valve as well as switching the flow of the second helium supply when backflushing. (See attached drawing.)

/jw
7/29/71





VIEW OF CHANNEL LOCATIONS IN TEFLON SLICE FOR WALL 6-PORT VALVE

V_1 - WALL 6-PORT VALVE
V_2 - CIRCLE SEAL DOMINO 4-PORT VALVE
V_3 - 6-WAY GAS CHECKING VALVE
V_4 - NEEDLE VALVE
C_1 - COLUMN I
C_2 - COLUMN II
$R_{1,2}$ - HELIUM REGULATORS
$G_{1,2}$ - PRESSURE GAUGES, 0-60 #

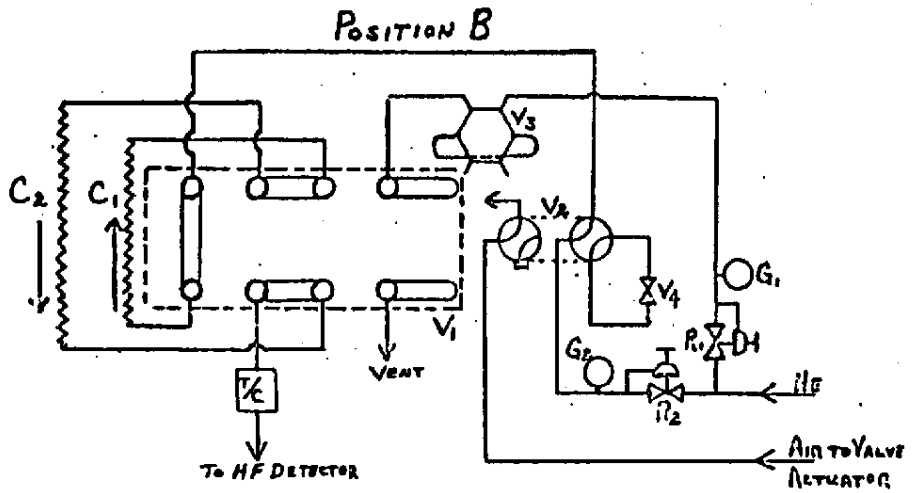
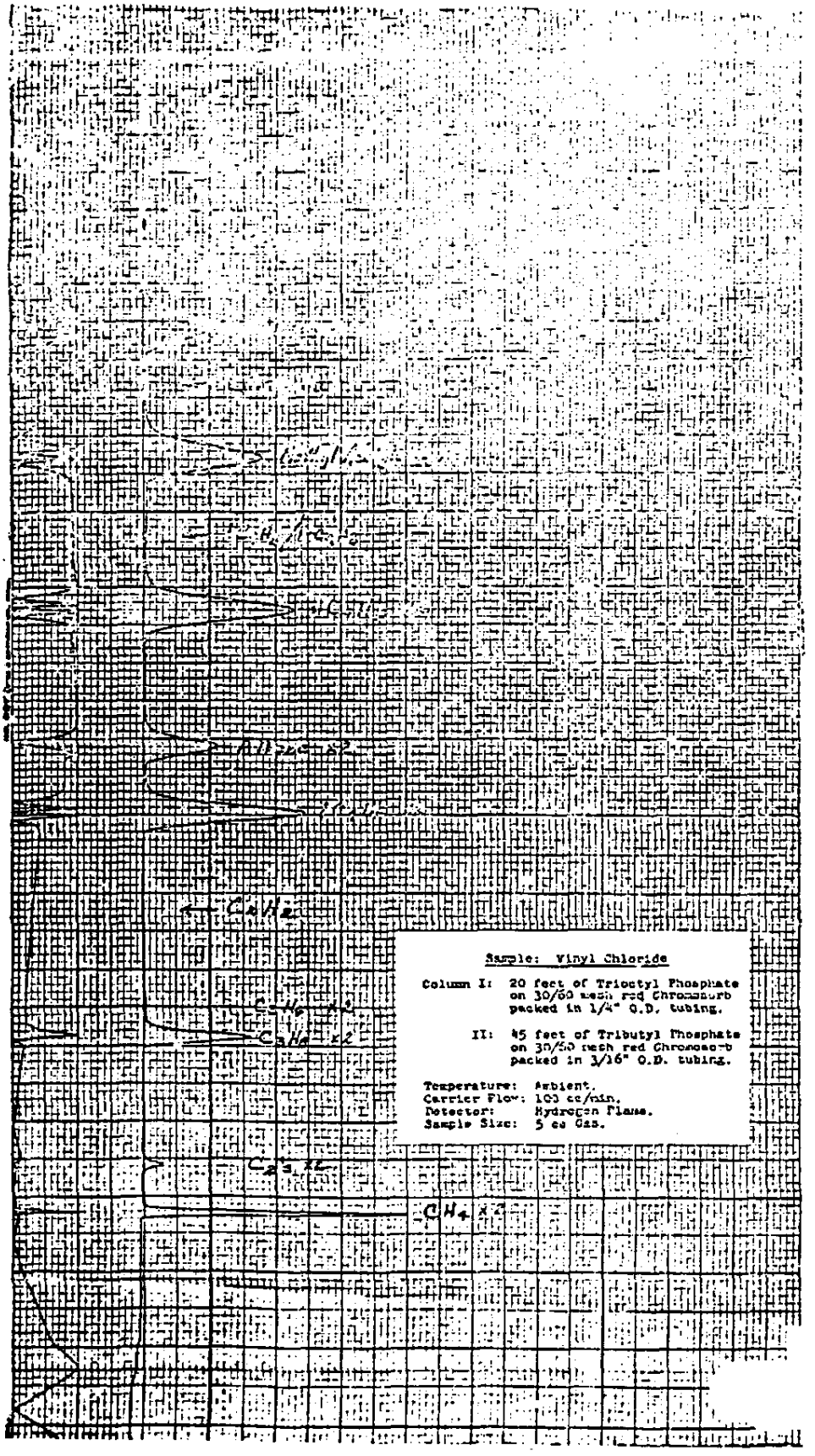


FIGURE I


Sample: Vinyl Chloride
Column I: 20 feet of Trioctyl Phosphate on 30/60 mesh red Chromosorb packed in 1/4" O.D. tubing.
II: 45 feet of Tributyl Phosphate on 30/60 mesh red Chromosorb packed in 3/16" O.D. tubing.
Temperature: Ambient.
Carrier Flow: 103 cc/min.
Detector: Hydrogen Flame.
Sample Size: 5 cc Gas.
CH4 x2

MONSANTO COMPANY

TEXAS CITY, TEXAS

DEPT. 60
METHOD 602.639 (VC)
May 28, 1964

MATERIAL

Product vinyl chloride monomer

ANALYSIS REQUIRED

Phenol

SAMPLE SIZE

Quart thermos jug

APPARATUS

250 ml Erlenmeyer flask

50 ml beaker

2 ml pipette

100 ml graduate

Beckman Model "B" Spectrophotometer

50 mm absorption cells (matched pair)

REAGENTS

Millons reagent -

Prepare in well ventilated hood.

PROCEDURE

Evaporate 100 mls of sample in a 250 ml Erlenmeyer flask. When sample is completely evaporated, add 25 ml distilled H_2O. Pipette 2 ml of Millons Reagent into flask. Boil for 1 minute on hot plate. Prepare a blank in the same manner, omit the sample. When solution has cooled, filter and read sample against blank on Beckman Model "B". Use 59 mm cells, sensitivity of 3, and wavelength of 440 mu.

CALCULATIONS

Multiply O.D. by 10 to obtain ppm phenol. Report to nearest 0.1 ppm.

Note:

Use of sensitivity of 3 is not normal for our procedures. Use setting of 3 to adjust instrument on blank. This will allow the use of much smaller slit and will give some improvement in reproducibility of results.

PREPARATION OF REAGENT SOLUTIONS

To 100 grams of fuming nitric acid, add 100 grams of mercury. Allow sufficient time for mercury to react and go completly in solution. Add 20 mls of distilled H_2O while stirring mixture. Place in a glass stoppered container.

STANDARDIZATION AND/OR CALIBRATIONS

See Solutions Manual.

SAFETY PRECAUTIONS

Handle mercury with care, avoid breathing vapors and dust spilled mercury with sulfur. Mercury reacts vigorously with nitric acid liberating large quantities of toxic NO_2. Perform the preparation of this reagent in the hood with good ventilation.

ANALYTICAL TIME

.2 hour

PRECISION AND SIGNIFICANT FIGURES

95% confidence limit ± 1 ppm at 5 ppm level.
Report to nearest 1 ppm level.

SCOPE

Mercuric nitrate reacts with the phenol in the sample to produce a color complex.

/jw
7/29/71

MONSANTO CHEMICAL COMPANY
ORGANIC DIVISION
TEXAS CITY, TEXAS

DEPT. 33, 60
METHOD 602.657 (W.C.)
February 4, 1964

MATERIAL

Caustic receipts

ANALYSIS REQUIRED

Iron

SAMPLE

1 pint

APPARATUS

Beckman "B" spectrophotometer

50 mm matched cells

5 ml pipettes

100 ml volumetric flasks

Analytical balances

Phydrion paper 4.0-6.0 range

Small weighing bottle

REAGENTS AND SOLUTIONS

1. 0.15% 1, 10 - phenanthroline (aqueous)
2. 10% hydroxylamine hydrogenchloride (aqueous)
3. Iron standard 100 γ gm/ml (aqueous)

4. A. R. grade ammonium hydroxide

5. .5N NaOH

6. .5N HCl

7. 1-1 HCl solution (iron free)

PROCEDURE

1. Fill a small weighing bottle with sample and weigh.

2. Place 2 gm of sample (40-50 drops) in a 100 ml beaker and dilute with 10-15 ml of water. Reweigh weighing bottle to obtain sample weight.

3. Add 10 ml of 1-1 HCl to the beaker and boil down until approximately 5 ml remains.

4. Pipette 5 ml of 10% NH_2OH . HCl and 5 ml of .15% O-phenanthroline to the beaker. Then pipette 4 ml of concentrated NH_4OH into the mixture.

5. Adjust the pH to 6-7 using phydrion paper with .5N HCl or .5N NaOH.

6. Transfer contents of the beaker to a 100 ml volumetric, rinsing the beaker several times to insure removal of all the sample. Adjust the volume with distilled water and mix.

7. Prepare a reagent blank.

8. Wait 20 minutes and then determine the absorbance of the sample at 510 wavelength in a 50 mm cells against a reagent blank. Obtain net absorbance.

CALCULATIONS

$$\text{PPM Fe} = \frac{\text{abs. X 526}}{\text{Weight of sample X 5}}$$

The 5 in the calculation denotes the cell length in CM.

PREPARATION OF REAGENTS

1. 0.15% 1, 10 phenanthroline - dissolve .75 μgm of reagent grade material in 5 ml of glacial acetic acid. Dilute to 500 ml with distilled water.

2. 10% hydroxylamine HCl - dissolve 50 μgms of AR grade material in 500 ml of distilled water.

3. Iron standard 100 μgm/ml - weigh 1000 gms of standard iron wire Dissolve in 10 ml HCl, dilute to 1 liter.

4. Iron standard 20 μgm/ml - dilute 20 ml of the standard 100 μgm/ml to 100 ml iron free distilled water.

5. Iron standard 5 μgm/ml - dilute 5 ml of the 100 μgm/ml standard to 100 ml with iron free distilled water.

STANDARDIZATION AND/OR CALIBRATIONS

Pipette 5, 10, 15, and 20 ml of the 5 μgm/ml standard to each of 4-100 ml volumetric flasks. These will contain 25, 50, 75 and 100 μgm/ml of iron. Similarly pipette 10, 15, 20 and 25 ml of the 20 μgm/ml standard to each of 4-100 volumetric flasks. These will continue 200, 300, 400 and 500 μgm of Fe. Prepare a reagent blank. Analyze following the method in the procedure. Plot the absorbances found against the corresponding μgm Fe. Draw in the best straight line through the points obtained. Re-check any points that are over 10% from the line value.

From this line, find the μgm of Fe corresponding to unit absorbance.

The factor 526 μgm Fe/unit absorbance was determined by reading the absorbance at 500 μgm from the curve and dividing 500 by the unit absorbance at this point (.95 abs.)

PRECISION AND SIGNIFICANT FIGURES

95% confidence limits are $\pm$ 0.1 PPM at a level of 1.05 PPM. Report results to two significant figures.

ANALYTICAL TIME

0.5 hours

SAFETY

Use normal precautions in handling laboratory chemicals, glassware and electrical equipment.

Strong caustic can cause severe burns if spilled on the skin. If such a spill does occur, wash with large amounts of water and report to first aid.

SCOPE

Iron is reduced to the divalent state with hydroxylamine at a pH 4 to 7. Fe(II) forms a red complex with 1.10 orthophenthroline. This red color is analytical W.R.T. iron.

REFERENCES

J.F.Q. General Method 70-F

/jw
7/26/71

MONSANTO CHEMICAL COMPANY
ORGANIC DIVISION
TEXAS CITY, TEXAS

MATERIAL

Caustic Receipts

ANALYSIS REQUIRED

Sodium hydroxide and sodium carbonate

SAMPLE

1 pint bottle

APPARATUS

50 ml acid burette

Small weighing bottle with dropper attached

250 ml Erlenmeyer flask

Analytical balance

REAGENTS

0.5 N standard HCl

Distilled Water

Phenolphthalein Indicator

Methyl Orange Indicator

PROCEDURE

1. Fill a small weighing bottle with the sample and weigh. This sample bottle can be used for all subsequent analyses of the caustic.

2. Place approximately 50 ml of distilled water in a 250 ml Erlenmeyer flask and add 2 gms of sample from the weighing bottle (approx. 40-50 drops). Reweigh to obtain the weight of sample.

3. Add 2-3 drops of phenolphthalein indicator and titrate with 0.5 N HCl until the solution is just colorless. Record this volume of HCl as V_1.

4. Refill HCl burette to zero. Add 2-3 drops of methyl orange indicator to the titration flask and titrate with 0.5 HCl until the solution is red orange. Record this volume of HCl as V_2.

CALCULATIONS

$$\frac{(V_1 - V_2)\ (N_{HCl})\ 4.0}{\text{Weight of Sample}} = \% \text{ NaOH}$$

$$\frac{(2V_2)\ (N_{HCl})\ 5.3}{\text{Weight of Sample}} = \% \text{ } Na_2CO_3$$

PREPARATION OF REAGENTS

Prepare indicator solutions and normal acid solution outlined in Reagent Manual.

STANDARDIZATION AND/OR CALIBRATIONS

Standardize HCl solution according to Reagent Manual.

SAFETY

Use care in handling strong caustic solutions, as caustic can cause severe burns if spilled on the skin. If a spill should occur, flush the area with large amounts of water and report to first aid. Use normal precautions with chemicals, glassware, and electrical equipment.

ANALYTICAL TIME

0.5 hours

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES

No precision has been determined, but duplicate analysis should agree within 0.5%.

SCOPE

In titrating to the phenolphthalein end point, the following two reactions occur:

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

$$Na_2CO_3 + HCl \longrightarrow NaHCO_3 + NaCl$$

The subsequent titration to the methyl orange end point completes the titration of the $NaHCO_3$ by the following reaction.

$$Na\ HCO_3 + HCl \longrightarrow NaCl + CO_2 + H_2O$$

REFERENCES

Method 22-10-1, Revised 3/17/53

GK/mlw

MONSANTO CHEMICAL COMPANY
ORGANIC DIVISION
TEXAS CITY, TEXAS

DEPT. 33,60
METHOD 602.659
February 4, 1964

MATERIAL

Caustic Receipts

ANALYSIS

Chlorides

SAMPLE

1 pint

APPARATUS

Small weighing bottles

250 ml beaker

50 ml burette

pH meter

Magnetic stirrer with stirrer bar

Silver electrode

$Hg-HgSO_4$ - K_2SO_4 electrode

REAGENTS

Concentrated nitric acid

0.1 N silver nitrate solution

PROCEDURE

1. Weigh 2 gms of sample (40-50 drops) and place in a 250 ml beaker. Add 50 ml of distilled water and 5 ml of concentrated nitric acid.

2. Place on magnetic stirrer and immerse the electrodes in the solution.

3. While solution is being agitated, titrate with 0.1 $AgNO_3$ to the first break, which will be about 100 MV.

CALCULATIONS

$$\frac{(\text{Volume of } AgNO_3)\ (\text{Normality})\ (3.55)}{\text{Weight of Sample}} = \%\ Cl^-$$

PREPARATION OF REAGENTS

Prepare 0.1 $AgNO_3$ according to Reagents Manual

STANDARD AND/OR CALIBRATIONS

Standard $AgNO_3$ by procedure in Reagents Manual

SAFETY

Handle all chemicals, glassware, and electrical equipment with normal precautions.

Caustic can cause severe burns to the skin. Handle with caution and if a spill should occur, wash the area with large amounts of water and report to first aid.

ANALYTICAL TIME

0.5 hours

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES

No precision has been determined, but should be accurate to ± .01%. Report 2 significant figures.

SCOPE

Chlorides in an acid solution precipitate as AgCl. An excess of chlorides in $AgNO_3$ causes a sharp change in potential, which is taken as the end point of the titration.

REFERENCES

Method 20-29-50, Rev. 7/27/58 (Modified)

GK/mlw

MONSANTO CHEMICAL COMPANY
ORGANIC DIVISION
TEXAS CITY, TEXAS

MATERIAL

Caustic Receipts

ANALYSIS REQUIRED

Sulfates

SAMPLE

1 pint

APPARATUS

100 ml beaker

Small weighing bottle

50 ml stoppered graduated cylinders

Pyrex hydrosol filter holders, Cat. #XX1002500

Millipore Filter Corp., Bedford, Massachusetts

Millipore filter discs., 25 mm dia. type AA 0.8μ

Scoop, glass, Cat. #1H-12. Laboratory Equipment Company

St. Joseph, Michigan

Beckman Model B

50 mm cells (matched)

REAGENTS

1. Barium chloride 20-30 mesh parr turbidimetric grade, J. T. Baker Chemical Company.
2. Dispersing reagent
3. Concentrated HCl
4. Phenolphthalein indicator

PROCEDURE

1. Weigh 2 gms of sample (40-50 drops from a weighing bottle) into a 100 ml beaker. Add 10 ml of distilled water and 1-2 drops of phenolphthalein indicator to the beaker. Carry a reagent blank along with the sample.

2. Drop-wise, add concentrated HCl until the solution is just colorless, then filter the sample through a millipore filter type AA. Wash the beaker several times with 5 ml portions of distilled water, passing the wash water through the filter after each washing. Transfer the contents of the filter flask to a 50 ml stoppered graduate cylinder, again washing the filter flask several times with 5 ml portions of distilled water.

3. Add 5 ml of dispersing reagent to the graduate and dilute to 50 ml with distilled water. Mix well. Then add 1 scoop (0.3 gm) of $BACl_2$ and shake for 1 minute.

4. Allow the graduate to stand for five minutes, and then transfer to a 50 mm cell and read against the blank at 420 mμ on the Beckman "B."

CALCULATIONS

From the attached calibration chart, determine the amount of SO_4 present.

$$\frac{\mu gm \text{ of } SO_4}{\text{Sample weight}} = PPM\ SO_4$$

PREPARATION OF REAGENTS

1. Dispersing reagent - Mix 25 ml concentrated HCl, 75 ml of deeminac water, 300 ml Cp glycerine, and 600 ml of 95% ethanol.

STANDARDIZATION AND/OR CALIBRATION

1. Measure .148 gm of AR Na_2SO_4 (Sodium sulfate) into a liter volumetric flask and dilute to a liter. This gives a 100 μgm/ml of SO_4 solution.

2. Pipette 10, 30, 50, 70 ml of the 100 ✓gm/ml standard solution into a 100 ml volumetric flask and dilute to volume. This gives a 10, 30, 50, 70 ✓gm/ml solution.

3. Use 1 ml of each solution and carry through the standard procedure for SO_4 determination. Plot the absorbance for each sample against the ✓gm SO_4 present. Draw a straight line connecting these values for an absorbance versus ✓gm chart.

SAFETY

Use normal laboratory safety precautions in handling chemicals, glassware and electrical equipment.

Caustic can cause a severe burn if spilled on the skin. If a spill should occur, wash with large amounts of water and report to first aid.

ANALYTICAL TIME

0.5 hours

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES

Report to the nearest PPM SO_4. 95% confidence limits are ±5PPM at a 50 PPM level.

SCOPE

The SO_4 present reacts with Ba to produce a tubidity, which is read as absorbance on the Beckman "B."

REFERENCES

Method 602.503 (W.C.) (Modified)

Micrograms of SO_4, versus absorbance on Beckman Model "B" spectrophotometer at 420 millimicrons with a 50 mm cell.

Absorbance	µgm SO_4	Absorbance	µgm SO_4
.002	3	.049	102
.003	6	.054	117
.004	9	.059	126
.005	12	.062	135
.007	15	.068	144
.009	18	.075	153
.010	21	.080	162
.012	24	.085	171
.013	27	.090	180
.014	30	.095	189
.015	33	.101	198
.017	36	.106	207
.019	39	.110	216
.020	42	.115	225
.021	45	.120	234
.022	48	.125	243
.023	51	.130	252
.024	54	.135	261
.026	57	.140	270
.027	60	.146	279
.028	63	.155	288
.030	66	.160	297
.031	69	.166	306
.032	72	.172	315
.034	75	.180	324
.035	78	.186	333
.036	81	.194	342
.038	84	.199	351
.040	87	.209	366
.042	90	.219	381
.043	93	.230	396
.044	96	.240	411
.045	99	.251	426

GK/mlw

800

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60N2 — Page 1 of 2
TANK NAME: Caustic-Finished Latex Mix Tank — MMS/ff
TANK CONTENTS: Caustic & Hydrolyzed Latex — Rev. 2/25/72
METHOD OF GAUGING: LI 214
TANK DIMENSIONS: 4' OD x 6'6"
TYPE: Vertical
HEADS: Bottom Flat; Top Dished
EQUIPPED WITH: An agitator
TRANSMITTER RANGE: 82.5 inches H_2O
SP. GR.: 1.145 **GALLONS/INCH:** 7.83

Instrument Reading (%)	Gallons	△
0	47	
1	53	5.6
2	58	
3	64	
4	70	
5	75	
6	81	
7	86	
8	92	
9	98	
10	103	
11	109	
12	115	
13	120	
14	126	
15	132	
16	137	
17	143	
18	149	
19	154	
20	160	
21	165	
22	171	
23	177	
24	182	
25	188	
26	194	
27	199	
28	205	
29	211	
30	216	
31	222	
32	228	
33	233	
34	239	
35	244	
36	250	

Instrument Reading (%)	Gallons	△
37	256	5.6
38	261	
39	267	
40	273	
41	278	
42	284	
43	290	
44	295	
45	301	
46	307	
47	312	
48	318	
49	323	
50	329	
51	335	
52	340	
53	346	
54	352	
55	357	
56	363	
57	369	
58	374	
59	380	
60	386	
61	391	
62	397	
63	402	
64	408	
65	414	
66	419	
67	425	
68	431	
69	436	
70	442	
71	448	
72	453	
73	459	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60N2	Page 2 of 2
TANK NAME:	Caustic-Finished Latex Mix Tank	MMS/ff
TANK CONTENTS:	Caustic & Hydrolyzed Latex	Rev. 2/25/72
METHOD OF GAUGING:	LI 214	
TANK DIMENSIONS:	4' OD x 6'6"	
TYPE:	Vertical	
HEADS:	Bottom Flat; Top Dished	
EQUIPPED WITH:	An agitator	
TRANSMITTER RANGE:	82.5 inches H_2O	
SP.GR.: 1.145	GALLONS/INCH: 7.83	

Instrument Reading (%)	Gallons	Δ
74	464	5.6
75	470	
76	476	
77	481	
78	487	
79	493	
80	498	
81	504	
82	510	
83	515	
84	521	
85	527	
86	532	
87	538	
88	543	
89	549	
90	555	
91	560	
92	566	
93	572	
94	577	
95	583	
96	589	
97	594	
98	600	
99	605	
100	611	

Instrument Reading (%)	Gallons	Δ

RSV 0015145

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60S1-1 & 2	Page 1 of 1
TANK NAME:	Blowdown Separator #1 & #2	MMS/ff
TANK CONTENTS:	Latex	Rev. 2/25/72
METHOD OF GAUGING:	LI 201-1&2	
TANK DIMENSIONS:	11' dia. x 15' SS	
TYPE:	Vertical	
HEADS:	Flanged & Dished	
EQPT. WITH:	Impingement Plate	
TRANSMITTER RANGE:	16.5 to 80.5 inches H_2O	
SP. GR.: 1.10	GALLONS/INCH: 59	

Instrument Reading (%)	Gallons	Δ
0	0	
37.5-Top of Dish	807	807
38	824	17
39	858	34.4
40	892	
41	927	
42	961	
43	996	
44	1030	
45	1064	
46	1099	
47	1133	
48	1168	
49	1202	
50	1236	
51	1271	
52	1305	
53	1340	
54	1374	
55	1408	
56	1443	
57	1477	
58	1512	
59	1546	
60	1580	
61	1615	
62	1650	
63	1684	
64	1718	
65	1752	
66	1787	
67	1821	
68	1856	

Instrument Reading (%)	Gallons	Δ
69	1890	34.4
70	1924	
71	1959	
72	1993	
73	2028	
74	2062	
75	2096	
76	2131	
77	2165	
78	2200	
79	2234	
80	2268	
81	2303	
82	2337	
83	2372	
84	2406	
85	2440	
86	2475	
87	2509	
88	2544	
89	2578	
90	2612	
91	2647	
92	2681	
93	2716	
94	2750	
95	2784	
96	2819	
97	2853	
98	2888	
99	2922	
100	2956	

RSV 0015146

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60S2 — Page 1 of 1
TANK NAME: THF Recovery Column Still Pot — MMS/ff
TANK CONTENTS: THF/Water/Latex Solids — Rev. 2/16/72
METHOD OF GAUGING: LIC 305
TANK DIMENSIONS: 6'4" I.D. x 7'8" SS
TYPE: Vertical
HEADS: Bottom double w/one inverted; Top F&D
EQPT. WITH: Steam Jacket and Agitator
TRANSMITTER TYPE: Displacer
SP. GR.: 1.0 GALLONS/INCH: 17.6

Instrument Reading (%)	Gallons	Δ
0	88	—
2	113	25.4
4	138	
6	164	
8	189	
10	215	
12	240	
14	265	
16	291	
18	316	
20	342	
22	367	
24	392	
26	418	
28	443	
30	469	
32	494	
34	519	
36	545	
38	570	
40	596	
42	621	
44	646	
46	672	
48	697	
50	723	

Instrument Reading (%)	Gallons	Δ
52	748	25.4
54	773	
56	799	
58	824	
60	850	
62	875	
64	900	
66	926	
68	951	
70	977	
72	1002	
74	1027	
76	1053	
78	1078	
80	1104	
82	1129	
84	1154	
86	1180	
88	1205	
90	1231	
92	1256	
94	1281	
96	1307	
98	1332	
100	1358	

RSV 0015147

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60S3
TANK NAME: THF Separator
TANK CONTENTS: THF
METHOD OF GAUGING: LIC 304
TANK DIMENSIONS: 30" OD x 39"
TYPE: Vertical
HEADS: Flanged and Dished
EQPT. WITH:

JSM/ff
7/4/71

Instrument Reading (%)	Gallons	Δ
0	21.7	.43
1	22.2	
2	22.6	
3	23.0	
4	23.4	
5	23.9	
6	24.3	
7	24.7	
8	25.2	
9	25.6	
10	26.0	
11	26.4	
12	26.9	
13	27.3	
14	27.7	
15	28.1	
16	28.6	
17	29.0	
18	29.5	
19	29.9	
20	30.3	
21	30.8	
22	31.2	
23	31.6	
24	32.0	
25	32.5	
26	32.9	
27	33.3	
28	33.8	
29	34.2	
30	34.6	
31	35.0	
32	35.5	
33	35.9	
34	36.3	

Instrument Reading (%)	Gallons	Δ
35	36.8	.43
36	37.2	
37	37.6	
38	38.1	
39	38.5	
40	38.9	
41	49.4	
42	39.8	
43	40.2	
44	40.6	
45	41.1	
46	41.5	
47	41.9	
48	42.4	
49	42.8	
50	43.2	
51	43.6	
52	44.1	
53	44.5	
54	44.9	
55	45.4	
56	45.8	
57	46.2	
58	46.7	
59	47.1	
60	47.5	
61	47.9	
62	48.4	
63	48.8	
64	49.2	
65	49.7	
66	50.1	
67	50.5	
68	51.0	
69	51.4	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60S3 (continued)

JSM/ff
7/4/71

Instrument Reading (%)	Gallons	Δ
70	51.8	.43
71	52.3	
72	52.7	
73	53.1	
74	53.5	
75	54.0	
76	54.4	
77	54.8	
78	55.3	
79	55.7	
80	56.1	
81	56.6	
82	57.0	
83	57.4	
84	57.8	
85	58.3	

Instrument Reading (%)	Gallons	Δ
86	58.7	.43
87	59.1	
88	59.6	
89	60.0	
90	60.4	
91	60.9	
92	61.3	
93	61.7	
94	62.1	
95	62.6	
96	63.0	
97	63.4	
98	63.9	
99	64.3	
100	64.7	

TANK CALIBRATION TABLE

TANK NUMBER: 60T1-1 & 2
TANK NAME: Vinyl Chloride Storage
TANK CONTENTS: Vinyl Chloride
METHOD OF GAUGING: LI 111-1&2
TANK DIMENSIONS: 10' dia x 40'
TYPE: Horizontal
HEADS: Elliptical
TRANSMITTER RANGE: 98 Inches H_2O
SP. GR.: .919

MMS/ff
Rev. 2/25/72

Instrument Reading (%)	Gallons	Δ
0	453	
1	572	124
2	712	135
3	857	145
4	1011	154
5	1171	160
6	1336	165
7	1506	170
8	1686	180
9	1876	190
10	2071	195
11	2271	200
12	2476	205
13	2686	210
14	2901	215
15	3122	221
16	3345	223
17	3572	227
18	3801	229
19	4034	233
20	4274	240
21	4517	243
22	4763	246
23	5012	149
24	5263	254
25	5520	257
26	5780	260
27	6042	262
28	6306	264
29	6573	267
30	6840	267
31	7113	273
32	7387	274
33	7662	275
34	7938	276

Instrument Reading (%)	Gallons	Δ
35	8218	280
36	8499	281
37	8782	283
38	9066	284
39	9353	287
40	9640	287
41	9928	288
42	10216	288
43	10504	288
44	10793	289
45	11083	290
46	11373	290
47	11663	290
48	11954	291
49	12245	291
50	12540	295
51	12833	291
52	13124	291
53	13414	290
54	13704	290
55	13994	291
56	14285	291
57	14576	291
58	14867	291
59	15157	290
60	15447	290
61	15735	288
62	16021	286
63	16305	284
64	16587	282
65	16867	280
66	17147	280
67	17426	279
68	17704	278
69	17979	275

TANK CALIBRATION TABLE

TANK NUMBER: 60T1-1 & 2 (continued)

MMS/ff
Rev. 2/25/72

Instrument Reading (%)	Gallons	Δ
70	18254	275
71	18527	273
72	18798	271
73	19067	269
74	19334	267
75	19598	264
76	19858	260
77	20114	256
78	20368	254
79	20618	250
80	20866	248
81	21110	244
82	21350	240
83	21586	236
84	21818	232
85	22046	228
86	22270	224
87	22490	220
88	22706	216
89	22918	212
90	23126	208
91	23326	200
92	23520	194
93	23708	188
94	23890	182
95	24065	175
96	24235	170
97	24397	162
98	24551	154
99	24698	147
100	24837	139

TANK CALIBRATION TABLE

TANK NO.: 60T2-1&2
TANK NAME: THF Storage Tank 1 & 2
TANK CONTENTS: Tetrahydrofuran
METHOD OF GAUGING: LI 301-1&2
TANK DIMENSIONS: 94.8" O.D. x 30' Str. Side
TYPE: Horizontal Cylindrical
HEADS: ASME Flanged & Dished
TRANSMITTER RANGE: 75 inches H_2O
SPECIFIC GRAVITY: 0.89

MMS/ff
6/21/72

Instrument Reading (%)	Gallons	Δ
0	495	0
1	570	75
2	647	77
3	720	80
4	802	82
5	898	86
6	987	89
7	1078	91
8	1161	93
9	1266	95
10	1363	97
11	1462	99
12	1564	102
13	1668	104
14	1773	105
15	1880	107
16	1989	109
17	2099	110
18	2210	111
19	2322	112
20	2436	114
21	2552	116
22	2669	117
23	2787	118
24	2906	119
25	3026	120
26	3147	121
27	3269	122
28	3393	124
29	3518	125
30	3643	125
31	3769	126
32	3895	126
33	4022	127
34	4149	127
35	4276	127
36	4404	128

Instrument Reading (%)	Gallons	Δ
37	4533	129
38	4662	129
39	4792	130
40	4922	130
41	5053	131
42	5184	131
43	5315	131
44	5447	132
45	5579	132
46	5711	132
47	5843	132
48	5975	132
49	6107	132
50	6239	132
51	6370	131
52	6500	130
53	6629	129
54	6757	128
55	6885	128
56	7013	128
57	7141	128
58	7269	128
59	7396	127
60	7523	127
61	7649	126
62	7774	125
63	7898	124
64	7921	123
65	8193	122
66	8264	121
67	8384	120
68	8503	119
69	8621	118
70	8737	116
71	8852	115
72	8966	114
73	9079	113

6/26/72

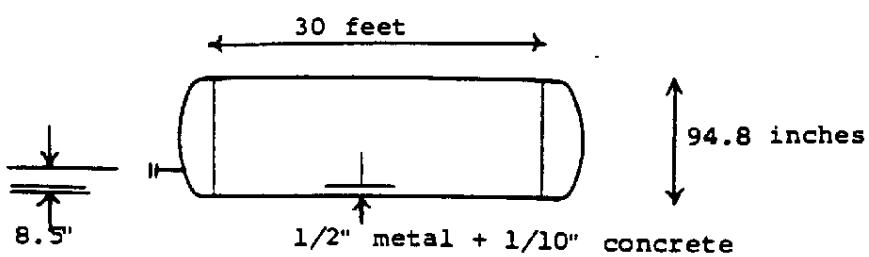
TANK CALIBRATION TABLE

TANK NO.: 60T2-1&2

MMS/ff
6/21/72

Instrument Reading (%)	Gallons	Δ
74	9191	112
75	9300	109
76	9408	108
77	9515	107
78	9620	105
79	9723	103
80	9824	101
81	9924	100
82	10022	98
83	10119	97
84	10215	96
85	10300	95
86	10390	90
87	10475	85
88	10557	82
89	10636	79
90	10711	75
91	10783	72
92	10853	70
93	10921	68
94	10987	66
95	11050	63
96	11107	57
97	11158	51
98	11200	42
99	11233	33
100	11258	25

Instrument Reading (%)	Gallons	Δ



6/26/72

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T3 — Page 1 of 1
TANK NAME: SLS Storage Tank — MMS /ff
TANK CONTENTS: Sodium Lauryl Sulfate — Rev. 2/25/72
METHOD OF GAUGING: LI 102
TANK DIMENSIONS: 12' OD x 15' SS
TYPE: Vertical
HEADS: Top - Conical; Bottom - Flat
EQPT. WITH: Side-mounted agitator and heating coil
TRANSMITTER RANGE: 175 inches H_2O
SP. GR.: 1.031 GALLONS/INCH: 70.46

Instrument Reading (%)	Gallons	Δ
0	846	119
1	965	
2	1085	
3	1205	
4	1325	
5	1444	
6	1564	
7	1684	
8	1804	
9	1924	
10	2043	
11	2163	
12	2283	
13	2403	
14	2523	
15	2642	
16	2762	
17	2882	
18	3002	
19	3121	
20	3234	
21	3354	
22	3474	
23	3594	
24	3713	
25	3833	
26	3953	
27	4073	
28	4192	
29	4312	
30	4432	
31	4552	
32	4672	
33	4791	
34	4911	

Instrument Reading (%)	Gallons	Δ
35	5031	119
36	5151	119
38	5390	239
40	5630	
42	5869	
44	6109	
46	6348	
48	6588	
50	6828	
52	7067	
54	7307	
56	7546	
58	7779	
60	8018	
62	8258	
64	8498	
66	8737	
68	8977	
70	9216	
72	9456	
74	9695	
76	9935	
78	10174	
80	10414	
82	10654	
84	10893	
86	11133	
88	11372	
90	11541	
92	11851	
94	12091	
96	12324	
98	12563	
100	12803	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T5
TANK NAME: Ammonia Make-up Tank
TANK CONTENTS: NH_4OH (22.2%)
METHOD OF GAUGING: LG 121
TANK DIMENSIONS: 4' dia x 6' SS
TYPE: Horizontal
HEADS: Flanged & Dished
EQPT. WITH: Cooling Coil

JSM/ff
7/4/71

Height	Gallons	Δ
0' 0"	45	12
1	57	12
2	69	12
3	82	13
4	95	13
5	110	15
6	125	15
7	140	15
8	155	15
9	170	15
10	185	15
11	200	15
1' 0"	216	16
1	232	16
2	249	17
3	267	18
4	285	18
5	303	19
6	322	19
7	341	19
8	359	18
9	377	16
10	393	16
11	409	16

Height	Gallons	Δ
2' 0"	425	16
1	441	16
2	457	15
3	472	15
4	487	15
5	502	15
6	517	15
7	532	14
8	546	13
9	559	13
10	572	12
11	584	12
3' 0"	596	11
1	607	9
2	616	8
3	624	7
4	631	6
5	637	6
6	641	4

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T6
TANK NAME: 20% Caustic Storage Tank
TANK CONTENTS: 20% Sodium Hydroxide
METHOD OF GAUGING: LG 210
TANK DIMENSIONS: 6'6" OD x 15'
TYPE: Horizontal
HEADS: Flanged & Dished
EQPT. WITH:

JSM/ff
7/4/71

Height	Gallons	Δ
0' 0"	135	35
1	170	37
2	207	39
3	246	40
4	286	44
5	330	48
6	378	48
7	426	50
8	476	50
9	526	51
10	577	52
11	629	54
1' 0"	683	56
1	739	57
2	796	59
3	855	59
4	914	60
5	974	63
6	1037	64
7	1101	64
8	1165	64
9	1220	64
10	1293	65
11	1358	65
2' 0"	1423	65
1	1488	65
2	1553	65
3	1618	65
4	1683	66
5	1749	66
6	1815	71
7	1886	71
8	1957	72
9	2029	72
10	2101	71
11	2172	71

Height	Gallons	Δ
3' 0"	2243	66
1	2309	66
2	2375	65
3	2440	65
4	2505	65
5	2570	65
6	2635	65
7	2700	65
8	2765	64
9	2829	64
10	2893	64
11	2957	64
4' 0"	3021	63
1	3084	60
2	3144	59
3	3203	59
4	3262	57
5	3319	56
6	3375	54
7	3429	52
8	3481	51
9	3532	50
10	3582	50
11	3632	48
5' 0"	3680	48
1	3728	44
2	3772	40
3	3812	39
4	3851	37
5	3888	35
6	3923	

RSV 0015156

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T7
TANK NAME: Condensate Storage Tank
TANK CONTENTS: Condensate
METHOD OF GAUGING: LIC 101
TANK DIMENSIONS: 12' ID x 19'11" SS
TYPE: Vertical
HEADS: Top - Conical; Bottom - Flat
TRANSMITTER RANGE: 154 inches H_2O
SP. GR.: 1.00 GALLONS/INCHES: 70.5

MMS /ff
Rev. 2/25/72

Instrument Reading (%)	Gallons	Δ
0	846	543
5	1388	
10	1956	
15	2473	
20	3016	
25	3558	
30	4101	
35	4643	
40	5186	
45	5728	
50	6271	

Instrument Reading (%)	Gallons	Δ
55	6814	543
60	7356	
65	7899	
70	8441	
75	8984	
80	9526	
85	10069	
90	10611	
95	11154	
100	11696	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T8
TANK NAME: Aqueous Make-Tank
TANK CONTENTS: Aqueous Solution
METHOD OF GAUGING: None
TANK DIMENSIONS: 90" OD x 8'6" SS
TYPE: Vertical
HEADS: Flanged & Dished
EQPT. WITH: An agitator

JSM/ff
7/4/71

Liquid Level % or Inches	Gallons	Δ
0	256	
5	396	140
10	536	
15	676	
20	816	
25	956	
30	1096	
35	1236	
40	1376	
45	1516	
50	1656	

Liquid Level % or Inches	Gallons	Δ
55	1796	140
60	1936	
65	2076	
70	2216	
75	2356	
80	2496	
85	2636	
90	2776	
95	2916	
100	3056	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T9
TANK NAME: Aqueous Storage Tank
TANK CONTENTS: Aqueous Mix
METHOD OF GAUGING: LI 110
TANK DIMENSIONS: 11' dia. x 15' SS
TYPE: Vertical
HEADS: Top - Conical; Bottom - Flat
EQPT. WITH: Internal Coils
TRANSMITTER RANGE: 157 inches H_2O
SP. GR.: 1.00 GALLONS/INCH: 59.2

MMS/ff
Rev. 2/25/72

Instrument Reading (%)	Gallons	Δ
0	710	
2	894	184
4	1083	
6	1267	
8	1456	
10	1640	
12	1823	
14	2013	
16	2196	
18	2386	
20	2569	
22	2753	
24	2942	
26	3126	
28	3309	
30	3499	
32	3682	
34	3872	
36	4055	
38	4245	
40	4428	
42	4612	
44	4801	
46	4985	
48	5174	
50	5358	

Instrument Reading (%)	Gallons	Δ
52	5541	184
54	5731	
56	5914	
58	6104	
60	6287	
62	6471	
64	6661	
66	6844	
68	7033	
70	7217	
72	7400	
74	7589	
76	7773	
78	7962	
80	8146	
82	8329	
84	8519	
86	8702	
88	8892	
90	9075	
92	9259	
94	9448	
96	9632	
98	9821	
100	10005	

RSV 0015159

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T10
TANK NAME: APS Make-Up Tank
TANK CONTENTS: APS Solution
METHOD OF GAUGING: None
TANK DIMENSIONS: 4'6" OD x 5'3"
TYPE: Vertical
HEADS: Flanged and Dished
EQPT. WITH: An agitator and Heating Coils

JSM/ff
7/4/71

Liquid Level %	Gallons	Δ
0	55	31
5	86	31
10	117	31
15	148	31
20	179	31
25	210	31
30	241	31
35	272	31
40	303	31
45	334	31
50	365	31
55	396	31
60	427	31
65	458	31
70	489	31
75	520	31
80	551	31
85	582	31
90	613	31
95	644	31
100	675	31

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T11
TANK NAME: APS Feed Tank
TANK CONTENTS: APS Solution
METHOD OF GAUGING: LG 118
TANK DIMENSIONS: 36" OD x 3'0"
TYPE: Vertical
HEADS: Flanged & Dished

JSM/ff
7/4/71

LG Height Ft.	In.	Gallons	Δ
0	0	34	4.4
	1	39	
	2	43	
	3	47	
	4	52	
	5	56	
	6	61	
	7	65	
	8	69	
	9	74	
	10	78	
	11	83	
1	0	87	
	1	91	
	2	96	
	3	100	
	4	105	
	5	109	
	6	113	
	7	118	
	8	122	
	9	127	
	10	131	
	11	135	

LG Height Ft.	In.	Gallons	Δ
2	0	140	4.4
	1	144	
	2	149	
	3	153	
	4	157	
	5	162	
	6	166	
	7	171	
	8	175	
	9	179	
	10	184	
	11	188	
3	0	193	

RSV 0015161

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T12	Page 1 of 1
TANK NAME:	SFS Make-Up Tank	JSM/ff
TANK CONTENTS:	SFS Solution	7/4/71
METHOD OF GAUGING:	None	
TANK DIMENSIONS:	42" OD x 4'0" SS	
TYPE:	Vertical	
HEADS:	Flanged & Dished	
EQPT. WITH:	Agitator and Heating Coils	

Liquid Level %	Gallons	Δ
0	26	14.4
5	40	
10	55	
15	69	
20	84	
25	98	
30	112	
35	127	
40	141	
45	156	
50	170	

Liquid Level %	Gallons	Δ
55	184	14.4
60	199	
65	213	
70	228	
75	242	
80	256	
85	271	
90	285	
95	300	
100	314	

RSV 0015162

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T13
TANK NAME: SFS Feed Tank
TANK CONTENTS: SFS Solution
METHOD OF GAUGING: LG 114
TANK DIMENSIONS: 36" OD x 3'0"
TYPE: Vertical
HEADS: Flanged & Dished

JSM/ff
7/4/71

LG Height Ft.	In.	Gallons	Δ
0	0	34	4.4
	1	39	
	2	43	
	3	47	
	4	52	
	5	56	
	6	61	
	7	65	
	8	69	
	9	74	
	10	78	
	11	83	
1	0	87	
	1	91	
	2	96	
	3	100	
	4	105	
	5	109	
	6	113	
	7	118	
	8	122	
	9	127	
	10	131	
	11	135	

LG Height Ft.	In.	Gallons	Δ
2	0	140	4.4
	1	144	
	2	149	
	3	153	
	4	157	
	5	162	
	6	166	
	7	171	
	8	175	
	9	179	
	10	184	
	11	188	
3	0	193	

RSV 0015163

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T14	Page 1 of 1
TANK NAME:	Acrylamide Make-Up Tank	JSM/ff
TANK CONTENTS:	Acrylamide/SLS Solution	7/4/71
METHOD OF GAUGING:	None	
TANK DIMENSIONS:	60" x 63"	
TYPE:	Vertical	
HEADS:	Flanged & Dished	
EQPT. WITH:	Agitator and Heating Coil	

Liquid Level %	Gallons	Δ
0	131	82
5	213	
10	295	
15	377	
20	459	
25	541	
30	623	
35	705	
40	787	
45	869	
50	951	

Liquid Level %	Gallons	Δ
55	1033	82
60	1115	
65	1197	
70	1279	
75	1361	
80	1443	
85	1525	
90	1607	
95	1689	
100	1771	

RSV 0015164

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T15 — Page 1 of 1
TANK NAME: Acrylamide Feed Tank — MMS /ff
TANK CONTENTS: Acrylamide/SLS Solution — Rev. 2/25/72
METHOD OF GAUGING: LI 122
TANK DIMENSIONS: 5' ID x 6'2"
TYPE: Vertical
HEADS: 5' radius
EQPT. WITH: External Jacket (not used).
TRANSMITTER TYPE: Displacer
SP. GR.: 1.04 GALLONS/INCH: 12.24

Instrument Reading (%)	Gallons	Δ
0	7	
2	15	8
4	28	13
6	42	14
8	59	17
10	76	18
12	93	
14	111	
16	129	
18	147	
20	164	
22	181	
24	200	
26	217	
28	235	
30	252	
32	269	
34	288	
36	305	
38	323	
40	340	
42	357	
44	376	
46	392	
48	411	
50	428	

Instrument Reading (%)	Gallons	Δ
50	428	18
52	446	
54	464	
56	481	
58	499	
60	577	
62	534	
64	552	
66	569	
68	588	
70	605	
72	622	
74	640	
76	657	
78	676	
80	693	
82	710	
84	728	
86	746	
88	764	
90	781	
92	798	
94	816	
96	834	
98	852	
100	869	

RSV 0015165

TANK CALIBRATION TABLE

TANK NUMBER: 60T16-1 & 2, 60T17
TANK NAME: Hydrolyzer #1 & 2, Filter FD. TK
TANK CONTENTS: Product Latex
METHOD OF GAUGING: LI 203-1&2 (0-110" WC)
TANK DIMENSIONS: 7.5' dia. x 8'9"
TYPE: Vertical
HEADS: Flanged & Dished
EQPT. WITH: Agitator and Heating Coils (#1 only)

AKS/ff
Rev. 2/11/72

Instrument Reading (%)	Gallons	Δ
0	411	
1	438	27.3
2	466	
3	493	
4	520	
5	548	
6	575	
7	602	
8	629	
9	657	
10	684	
11	711	
12	739	
13	766	
14	793	
15	821	
16	848	
17	875	
18	902	
19	930	
20	957	
21	984	
22	1012	
23	1040	
24	1066	
25	1094	
26	1121	
27	1148	
28	1175	
29	1203	
30	1230	
31	1257	
32	1285	
33	1312	
34	1339	

Instrument Reading (%)	Gallons	Δ
35	1367	27.3
36	1394	
37	1421	
38	1448	
39	1476	
40	1503	
41	1530	
42	1558	
43	1585	
44	1612	
45	1640	
46	1667	
47	1694	
48	1721	
49	1749	
50	1776	
51	1803	
52	1831	
53	1858	
54	1885	
55	1913	
56	1940	
57	1967	
58	1994	
59	2022	
60	2049	
61	2076	
62	2104	
63	2131	
64	2158	
65	2186	
66	2213	
67	2240	
68	2267	
69	2295	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T16-1 & 2,60T17 (continued)

AKS/ff
Rev. 2/11/72

Instrument Reading (%)	Gallons	Δ
70	2322	27.3
71	2349	
72	2377	
73	2404	
74	2431	
75	2459	
76	2486	
77	2513	
78	2540	
79	2568	
80	2595	
81	2622	
82	2650	
83	2677	
84	2704	
85	2732	

Instrument Reading (%)	Gallons	Δ
86	2759	27.3
87	2786	
88	2813	
89	2841	
90	2868	
91	2895	
92	2923	
93	2950	
94	2977	
95	3005	
96	3032	
97	3059	
98	3086	
99	3114	
100	3141	

TANK CALIBRATION TABLE

TANK NUMBER: 60T18
TANK NAME: Latex Receiver
TANK CONTENTS: Filtered Latex
METHOD OF GAUGING: LIC 206 (0-50" WC)
TANK DIMENSIONS: 3'6" OD x 4'3"
TYPE: Vertical
HEADS: Flanged and Dished
EQPT. WITH:

AKS/ff
Rev. 2/11/72

Instrument Reading (%)	Gallons	Δ
0	62.0	
1	64.7	2.7
2	67.4	
3	70.1	
4	72.8	
5	75.5	
6	78.2	
7	80.9	
8	83.6	
9	86.3	
10	89.0	
11	91.7	
12	94.4	
13	97.1	
14	99.8	
15	102.5	
16	105.2	
17	107.9	
18	110.6	
19	113.3	
20	116.0	
21	118.7	
22	121.4	
23	124.1	
24	126.8	
25	129.5	
26	132.2	
27	134.9	
28	137.6	
29	140.3	
30	143.0	
31	145.7	
32	148.4	
33	151.1	
34	153.8	

Instrument Reading (%)	Gallons	Δ
35	156.5	2.7
36	159.2	
37	161.9	
38	164.6	
39	167.3	
40	170.0	
41	172.7	
42	175.4	
43	178.1	
44	180.8	
45	183.5	
46	186.2	
47	188.9	
48	191.6	
49	194.3	
50	197.0	
51	199.7	
52	202.4	
53	205.1	
54	207.8	
55	210.5	
56	213.2	
57	215.9	
58	218.6	
59	221.3	
60	224.0	
61	226.7	
62	229.4	
63	232.1	
64	234.8	
65	237.5	
66	240.2	
67	242.9	
68	245.6	
69	248.3	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T18 (continued) Page 2 of 2

AKS /ff

Rev. 2/11/72

Instrument Reading (%)	Gallons	Δ
70	251.0	2.7
71	253.7	
72	256.4	
73	259.1	
74	261.8	
75	264.5	
76	267.2	
77	269.9	
78	272.6	
79	275.3	
80	278.0	
81	280.7	
82	283.4	
83	286.1	
84	288.8	
85	291.5	

Instrument Reading (%)	Gallons	Δ
86	294.2	2.7
87	296.9	
88	299.6	
89	302.3	
90	305.0	
91	307.7	
92	310.4	
93	313.1	
94	315.8	
95	318.5	
96	321.2	
97	323.9	
98	326.6	
99	329.3	
100	332.0	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T19
TANK NAME: Latex B Storage Tank
TANK CONTENTS: Latex B
METHOD OF GAUGING: LI 208 (0-366.5"WC)
TANK DIMENSIONS: 13' dia. x 28' SS
TYPE: Vertical
HEADS: Top - Conical; Bottom - Flat
EQPT. WITH: An eductor

AKS/ff
Rev. 2/14/72

Instrument Reading (%)	Gallons	Δ
0	910	273
1	1183	
2	1456	
3	1729	
4	2002	
5	2275	
6	2548	
7	2821	
8	3094	
9	3367	
10	3640	
11	3913	
12	4186	
13	4459	
14	4732	
15	5005	
16	5278	
17	5551	
18	5824	
19	6097	
20	6370	
21	6643	
22	6916	
23	7189	
24	7462	
25	7735	
26	8008	
27	8281	
28	8554	
29	8827	
30	9100	
31	9373	
32	9646	
33	9919	
34	10192	

Instrument Reading (%)	Gallons	Δ
35	10465	273
36	10738	
37	11011	
38	11284	
39	11557	
40	11830	
41	12103	
42	12376	
43	12649	
44	12922	
45	13195	
46	13468	
47	13741	
48	14014	
49	14287	
50	14560	
51	14833	
52	15106	
53	15379	
54	15652	
55	15925	
56	16198	
57	16471	
58	16744	
59	17017	
60	17290	
61	17563	
62	17836	
63	18109	
64	18382	
65	18655	
66	18928	
67	19201	
68	19474	
69	19747	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T19 (continued)

AKS /ff
Rev. 2/14/72

Instrument Reading (%)	Gallons	Δ
70	20020	273
71	20293	
72	20566	
73	20839	
74	21112	
75	21385	
76	21658	
77	21931	
78	22204	
79	22477	
80	22750	
81	23023	
82	23296	
83	23569	
84	23842	
85	24115	

Instrument Reading (%)	Gallons	Δ
86	24388	273
87	24661	
88	24934	
89	25207	
90	25480	
91	25753	
92	26026	
93	26299	
94	26572	
95	26845	
96	27118	
97	27391	
98	27664	
99	27937	
100	28210	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T20	Page 1 of 2
TANK NAME:	Latex BH Storage Tank	RTH /ff
TANK CONTENTS:	Latex BH Product	Rev. 1/25/72
METHOD OF GAUGING:	LI 207 (0-218.5"WC)	
TANK DIMENSIONS:	17' ID x 18'	
TYPE:	Vertical	
HEADS:	Bottom Flat; Top Conical	
EQPT. WITH:	An Agitator	

Instrument Reading (%)	Gallons	Δ
0	707	
1	987	279
2	1266	
3	1545	
4	1824	
5	2103	
6	2382	
7	2661	
8	2940	
9	3219	
10	3498	
11	3777	
12	4056	
13	4335	
14	4614	
15	4893	
16	5172	
17	5451	
18	5730	
19	6009	
20	6288	
21	6567	
22	6846	
23	7125	
24	7404	
25	7683	
26	7962	
27	8341	
28	8520	
29	8799	
30	9078	
31	9357	
32	9636	
33	9915	
34	10194	

Instrument Reading (%)	Gallons	Δ
35	10473	279
36	10752	
37	11031	
38	11310	
39	11589	
40	11868	
41	12147	
42	12426	
43	12705	
44	12984	
45	13263	
46	13542	
47	13821	
48	14100	
49	14379	
50	14658	
51	14937	
52	15216	
53	15495	
54	15774	
55	16053	
56	16332	
57	16611	
58	16890	
59	17169	
60	17448	
61	17727	
62	18006	
63	18285	
64	18564	
65	18843	
66	19122	
67	19401	
68	19680	
69	19959	

TANK CALIBRATION TABLE

TANK NUMBER: 60T20 (continued)

RTH/ff
Rev. 1/25/72

Instrument Reading (%)	Gallons	Δ
70	20238	279
71	20517	
72	20796	
73	21075	
74	21354	
75	21633	
76	21912	
77	22191	
78	22470	
79	22749	
80	23028	
81	23307	
82	23586	
83	23865	
84	24144	
85	24423	

Instrument Reading (%)	Gallons	Δ
86	24702	279
87	24981	
88	25260	
89	25539	
90	25818	
91	26097	
92	26376	
93	26655	
94	26934	
95	27213	
96	27492	
97	27771	
98	28050	
99	28329	
100	28608	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T21	Page 1 of 2
TANK NAME:	Misc. Product Storage	AKS/ff
TANK CONTENTS:	Latex Product	Rev. 2/14/72
METHOD OF GAUGING:	LI 209 (0-323.9"WC)	
TANK DIMENSIONS:	13' ID x 25'	
TYPE:	Vertical	
HEADS:	Top - Conical; Bottom - Flat	
EQPT. WITH:	An agitator	

Instrument Reading(%)	Gallons	Δ
0	703	
1	944	241.3
2	1186	
3	1427	
4	1668	
5	1910	
6	2151	
7	2392	
8	2633	
9	2875	
10	3116	
11	3357	
12	3599	
13	3840	
14	4081	
15	4323	
16	4564	
17	4805	
18	5046	
19	5288	
20	5529	
21	5770	
22	6012	
23	6253	
24	6494	
25	6736	
26	6977	
27	7218	
28	7459	
29	7701	
30	7942	
31	8183	
32	8425	
33	8666	
34	8907	

Instrument Reading(%)	Gallons	Δ
35	9149	241.3
36	9390	
37	9631	
38	9872	
39	10114	
40	10355	
41	10596	
42	10838	
43	11079	
44	11320	
45	11562	
46	11803	
47	12044	
48	12285	
49	12527	
50	12768	
51	13009	
52	13251	
53	13492	
54	13733	
55	13975	
56	14216	
57	14457	
58	14698	
59	14940	
60	15181	
61	15422	
62	15664	
63	15905	
64	16146	
65	16388	
66	16629	
67	16870	
68	17111	
69	17353	

TANK CALIBRATION TABLE

TANK NUMBER: 60T21 (continued)

AKS/ff
Rev. 2/14/72

Instrument Reading (%)	Gallons	Δ
70	17594	241.3
71	17835	
72	18077	
73	18318	
74	18559	
75	18801	
76	19042	
77	19283	
78	19524	
79	19766	
80	20007	
81	20248	
82	20490	
83	20731	
84	20972	
85	21214	

Instrument Reading (%)	Gallons	Δ
86	21455	241.3
87	21692	
88	21937	
89	22179	
90	22420	
91	22661	
92	22903	
93	23144	
94	23385	
95	23627	
96	23868	
97	24109	
98	24350	
99	24592	
100	24833	

TANK CALIBRATION TABLE

TANK NUMBER:	60T22	Page 1 of 2
TANK NAME:	Waste Latex & Vent Tank	AKS/ff
TANK CONTENTS:	Off-Spec. Latex	Rev. 2/11/72
METHOD OF GAUGING:	LT 123 (0-304"WC)	
TANK DIMENSIONS:	9'0" ID x 27' SS	
TYPE:	Vertical	
HEADS:	8'6" Radius	
EQUIPPED WITH:	Impingement Plate	

Instrument Reading (%)	Gallons H_2O	Δ gal/%
0	795	120.5
1	916	
2	1036	
3	1157	
4	1277	
5	1398	
6	1518	
7	1639	
8	1759	
9	1880	
10	2000	
11	2121	
12	2241	
13	2362	
14	2482	
15	2603	
16	2723	
17	2844	
18	2964	
19	3085	
20	3205	
21	3326	
22	3446	
23	3567	
24	3687	
25	3808	
26	3928	
27	4049	
28	4169	
29	4290	
30	4410	
31	4531	
32	4651	
33	4772	
34	4892	

Instrument Reading (%)	Gallons H_2O	Δ gal/%
35	5013	120.5
36	5133	
37	5254	
38	5374	
39	5495	
40	5615	
41	5736	
42	5856	
43	5977	
44	6097	
45	6218	
46	6338	
47	6459	
48	6579	
49	6700	
50	6820	
51	6941	
52	7061	
53	7182	
54	7302	
55	7423	
56	7543	
57	7664	
58	7784	
59	7905	
60	8025	
61	8146	
62	8266	
63	8387	
64	8507	
65	8628	
66	8748	
67	8869	
68	8989	
69	9110	

TANK CALIBRATION TABLE

TANK NUMBER: 60T22 (continued)

AKS/ff
Rev. 2/11/72

Instrument Reading (%)	Gallons H_2O	Δgal/%
70	9230	120.5
71	9351	
72	9471	
73	9592	
74	9712	
75	9833	
76	9953	
77	10074	
78	10194	
79	10315	
80	10435	
81	10556	
82	10676	
83	10797	
84	10917	
85	11038	

Instrument Reading (%)	Gallons H_2O	Δgal/%
86	11158	120.5
87	11279	
88	11399	
89	11520	
90	11640	
91	11761	
92	11881	
93	12002	
94	12122	
95	12243	
96	12363	
97	12484	
98	12604	
99	12725	
100	12845	

TANK CALIBRATION TABLE

TANK NUMBER: 60T24
TANK NAME: Waste Tank
TANK CONTENTS: Water/Latex Solids
METHOD OF GAUGING: LI 303 (0-100"WC)
TANK DIMENSIONS: 8'6" dia. x 8'8" SS
TYPE: Vertical
HEADS: 8'6" Radius
EQPT. WITH:

AKS/ff
Rev. 2/14/72

Instrument Reading(%)	Gallons H_2O	Δ
0	460	35.4
1	495	
2	531	
3	566	
4	602	
5	637	
6	672	
7	708	
8	743	
9	779	
10	814	
11	849	
12	885	
13	920	
14	956	
15	991	
16	1026	
17	1062	
18	1097	
19	1133	
20	1168	
21	1203	
22	1239	
23	1274	
24	1310	
25	1345	
26	1380	
27	1416	
28	1451	
29	1487	
30	1522	
31	1557	
32	1593	
33	1628	
34	1664	

Instrument Reading(%)	Gallons H_2O	Δ
35	1699	35.4
36	1734	
37	1770	
38	1805	
39	1841	
40	1876	
41	1911	
42	1947	
43	1982	
44	2018	
45	2053	
46	2088	
47	2124	
48	2159	
49	2195	
50	2230	
51	2265	
52	2301	
53	2336	
54	2372	
55	2407	
56	2442	
57	2478	
58	2513	
59	2549	
60	2584	
61	2619	
62	2655	
63	2690	
64	2726	
65	2761	
66	2796	
67	2832	
68	2867	
69	2903	

TANK CALIBRATION TABLE

TANK NUMBER: 60T24 (continued)

AKS/ff
Rev. 2/14/72

Instrument Reading (%)	Gallons	Δ
70	2938	35.4
71	2973	
72	3009	
73	3044	
74	3080	
75	3115	
76	3150	
77	3186	
78	3221	
79	3257	
80	3292	
81	3327	
82	3363	
83	3398	
84	3434	
85	3469	

Instrument Reading (%)	Gallons H_2O	Δ
86	3504	35.4
87	3540	
88	3575	
89	3611	
90	3646	
91	3681	
92	3717	
93	3752	
94	3788	
95	3823	
96	3858	
97	3894	
98	3929	
99	3965	
100	4000	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T25
TANK NAME: Seal Pot
TANK CONTENTS: Filtered Water
METHOD OF GAUGING: LG 403
TANK DIMENSIONS: 4' dia. x 10' SS
TYPE: Horizontal
HEADS: 4' Radius
EQPT. WITH:

JSM/ff
7/4/71

Height Ft.	Height In.	Gallons	Δ
0	0	37	32
	2	69	37
	4	106	41
	6	147	44
	8	191	45
	10	236	48
1	0	284	52
	2	336	52
	4	388	54
	6	442	54
	8	496	54
	10	550	54

Height Ft.	Height In.	Gallons	Δ
2	0	604	52
	2	656	52
	4	708	48
	6	756	45
	8	804	44
	10	845	41
3	0	886	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T26
TANK NAME: Chilled Water Tank
TANK CONTENTS: Chilled Condensate
METHOD OF GAUGING: LI 501 (0-114"WC)
TANK DIMENSIONS: 10' dia. x 40'
TYPE: Horizontal
HEADS: Elliptical
EQPT. WITH:

JSM/ff
7/4/71

Instrument Reading (%)	Gallons	Δ
0	448	133
1	581	144
2	725	152
3	877	160
4	1037	169
5	1206	179
6	1385	189
7	1574	196
8	1770	201
9	1971	206
10	2177	218
11	2395	222
12	2617	227
13	2844	231
14	3075	235
15	3310	242
16	3552	244
17	3796	249
18	4045	256
19	4301	264
20	4565	270
21	4835	273
22	5108	274
23	5382	276
24	5658	277
25	5935	280
26	6215	281
27	6496	285
28	6781	286
29	7067	296
30	7363	297
31	7660	300
32	7960	302
33	8262	304
34	8566	305

Instrument Reading (%)	Gallons	Δ
35	8871	305
36	9176	306
37	9482	308
38	9790	308
39	10098	309
40	10407	311
41	10718	313
42	11031	316
43	11347	316
44	11663	316
45	11979	316
46	12295	316
47	12611	317
48	12928	317
49	13245	316
50	13561	316
51	13877	316
52	14193	314
53	14507	312
54	14819	312
55	15131	311
56	15442	309
57	15751	309
58	16060	308
59	16368	306
60	16674	302
61	16976	300
62	17276	299
63	17575	298
64	17873	296
65	18169	294
66	18463	288
67	18751	285
68	19036	282
69	19318	280

TANK CALIBRATION TABLE

TANK NUMBER: 60T26 (continued)

Page 2 of 2
JSM/ff
7/4/71

Instrument Reading (%)	Gallons	Δ
70	19598	277
71	19875	274
72	20149	272
73	20421	269
74	20690	266
75	20956	263
76	21219	257
77	21476	252
78	21728	247
79	21975	241
80	22216	236
81	22452	230
82	22682	224
83	22906	219
84	23125	212
85	23337	205
86	23542	198
87	23740	193
88	23933	185
89	24118	178
90	24296	167
91	24463	156
92	24619	146
93	24765	137
94	24902	130
95	25032	127
96	25159	100
97	25259	89
98	25348	73
99	25421	39
100	25460	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T36
TANK NAME: Catch Tank
TANK CONTENTS: Filtered Water
METHOD OF GAUGING: None
TANK DIMENSIONS: 8' I.D. x 14'0"
TYPE: Horizontal
HEADS: Dished Heads
EQPT. WITH:

JSM/ff
7/4/71

Height Ft.	In.	Gallons	Δ
0	0		
	1	16	16
	2	32	17
	3	49	29
	4	78	30
	5	108	31
	6	139	38
	7	177	39
	8	216	39
	9	255	44
	10	299	44
	11	343	48
1	0	391	50
	1	441	50
	2	491	53
	3	544	53
	4	597	54
	5	651	55
	6	706	61
	7	767	61
	8	828	63
	9	891	63
	10	954	63
	11	1017	65
2	0	1082	65
	1	1147	67
	2	1214	68
	3	1282	69
	4	1351	69
	5	1420	70
	6	1490	71
	7	1561	72
	8	1633	72
	9	1705	73
	10	1778	73
	11	1851	74

Height Ft.	In.	Gallons	Δ
3	0	1925	74
	1	1999	74
	2	2073	75
	3	2148	75
	4	2223	75
	5	2298	76
	6	2374	76
	7	2450	77
	8	2527	77
	9	2604	78
	10	2682	78
	11	2760	78
4	0	2838	78
	1	2916	78
	2	2994	78
	3	3072	77
	4	3149	77
	5	3226	76
	6	3302	76
	7	3378	75
	8	3453	75
	9	3528	75
	10	3603	74
	11	3677	74
5	0	3751	74
	1	3825	73
	2	3898	73
	3	3971	72
	4	4043	72
	5	4115	71
	6	4186	70
	7	4256	69
	8	4325	69
	9	4394	68
	10	4462	67
	11	4529	65

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER: 60T36 (continued)

JSM/ff
7/4/71

Height Ft.	Height In.	Gallons	Δ
6	0	4594	65
	1	4659	63
	2	4722	63
	3	4785	63
	4	4848	61
	5	4909	61
	6	4970	55
	7	5025	54
	8	5079	53
	9	5132	53
	10	5185	50
	11	5235	50
7	0	5285	48
	1	5333	44
	2	5377	44
	3	5421	39
	4	5460	39
	5	5499	38
	6	5537	31
	7	5568	30
	8	5598	29
	9	5627	17
	10	5644	16
	11	5660	16
8	0	5676	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T38	Page 1 of 2
TANK NAME:	Chemlime Storage Tank	JSM/ff
TANK CONTENTS:	Chemlime	7/4/71
METHOD OF GAUGING:	LI 402	
TANK DIMENSIONS:	12' OD x 14'0"	
TYPE:	Vertical	
HEADS:	Top - Conical; Bottom - Flat	
EQPT. WITH:	Agitator and Internal Coils	

Instrument Reading (%)	Gallons	Δ
0	846	110
1	956	
2	1066	
3	1176	
4	1286	
5	1396	
6	1506	
7	1616	
8	1726	
9	1836	
10	1946	
11	2056	
12	2166	
13	2276	
14	2386	
15	2496	
16	2606	
17	2716	
18	2826	
19	2936	
20	3046	
21	3156	
22	3266	
23	3376	
24	3486	
25	3596	
26	3706	
27	3816	
28	3926	
29	4036	
30	4146	
31	4256	
32	4366	
33	4476	
34	4586	

Instrument Reading (%)	Gallons	Δ
35	4696	110
36	4806	
37	4916	
38	5026	
39	5136	
40	5246	
41	5356	
42	5466	
43	5576	
44	5686	
45	5796	
46	5906	
47	6016	
48	6126	
49	6236	
50	6346	
51	6456	
52	6566	
53	6676	
54	6786	
55	6896	
56	7006	
57	7116	
58	7225	
59	7335	
60	7445	
61	7555	
62	7665	
63	7775	
64	7885	
65	7995	
66	8105	
67	8215	
68	8325	
69	8435	

TANK CALIBRATION TABLE

TANK NUMBER: 60T38 (continued)

JSM/ff
7/4/71

Instrument Reading (%)	Gallons	Δ
70	8545	110
71	8655	
72	8765	
73	8875	
74	8985	
75	9095	
76	9205	
77	9315	
78	9425	
79	9535	
80	9645	
81	9755	
82	9865	
83	9975	
84	10085	
85	10195	

Instrument Reading (%)	Gallons	Δ
86	10305	110
87	10415	
88	10525	
89	10635	
90	10745	
91	10855	
92	10965	
93	11075	
94	11185	
95	11295	
96	11405	
97	11515	
98	11625	
99	11735	
100	11845	

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T40	Page 1 of 2
TANK NAME:	Latex E Storage Tank	AKS/ff
TANK CONTENTS:	Latex E Product	Rev. 1/25/72
METHOD OF GAUGING:	LI 216 (0-230.2"WC)	
TANK DIMENSIONS:	20'0" ID x 18'	
TYPE:	Vertical	
HEADS:	Bottom - Flat; Top - Conical	
EQPT. WITH:	An Eductor	

Instrument Reading (%)	Gallons	Δ	Instrument Reading (%)	Gallons	Δ
0	2153	405.9	35	16360	405.9
1	2559		36	16765	
2	2965		37	17171	
3	3371		38	17577	
4	3777		39	17983	
5	4183		40	18389	
6	4588		41	18795	
7	4994		42	19201	
8	5400		43	19607	
9	5806		44	20013	
10	6212		45	20419	
11	6618		46	20824	
12	7024		47	21230	
13	7430		48	21636	
14	7836		49	22042	
15	8242		50	22448	
16	8647		51	22854	
17	9053		52	23260	
18	9459		53	23666	
19	9865		54	24072	
20	10271		55	24478	
21	10677		56	24883	
22	11083		57	25289	
23	11489		58	25695	
24	11895		59	26101	
25	12301		60	26507	
26	12706		61	26913	
27	13112		62	27319	
28	13518		63	27725	
29	13924		64	28131	
30	14330		65	28537	
31	14736		66	28942	
32	15142		67	29348	
33	15548		68	29754	
34	15548		69	29754	

RSV 0015187

WS-10
MCC 930930

Monsanto

FROM (NAME & LOCATION) Texas City, Texas

DATE

CC

SUBJECT Additional Material for Dept.(s) 60
Operating Manual/Specification Manual

REFERENCE

TO : M E Gibbs - SL

Please insert the attached material into its correct location in your Department(s)_____ Operating Manual/Specification Manual, Copy No._____.

Freida Fredericks
Standards Section

The attached material is
COMPANY-CONFIDENTIAL

ANY MATERIAL THAT IS BEING SUPERSEDED BY THE ATTACHED SHOULD BE DESTROYED OR RETURNED TO THE STANDARDS SECTION

RSV 0015188

60-801.00

TANK CALIBRATION TABLE

TANK NUMBER:	60T41	Page 1 of 2
TANK NAME:	LATEX STORAGE TANK	JSM/ff
TANK CONTENTS:	LATEX PRODUCT (Sp.Gr. 1.11)	9/7/73
METHOD OF GAUGING:	LI (0-360" W.C.)	
TANK DIMENSIONS:	16'0" OD x 30'	
TYPE:	VERTICAL	
HEADS:	BOTTOM-FLAT; TOP-CONICAL	
EQPT. WITH:	BAFFLES FOR FUTURE AGITATOR	

Instrument Reading (%)	Gallons	Δ
0	1,467	404.9
1	1,872	
2	2,277	
3	2,682	
4	3,087	
5	3,492	
6	3,897	
7	4,302	
8	4,706	
9	5,111	
10	5,516	
11	5,921	
12	6,326	
13	6,731	
14	7,136	
15	7,540	
16	7,946	
17	8,351	
18	8,756	
19	9,161	
20	9,566	
21	9,970	
22	10,375	
23	10,780	
24	11,185	
25	11,590	
26	11,995	
27	12,400	
28	12,805	
29	13,210	
30	13,615	
31	14,020	
32	14,425	
33	14,830	
34	15,234	

Instrument Reading (%)	Gallons	Δ
35	15,639	404.9
36	16,044	
37	25,357	
38	16,854	
39	17,259	
40	17,664	
41	18,069	
42	18,474	
43	18,879	
44	19,284	
45	19,689	
46	20,094	
47	20,498	
48	20,903	
49	21,308	
50	21,714	
51	22,118	
52	22,523	
53	22,928	
54	23,333	
55	23,738	
56	24,143	
57	24,548	
58	24,953	
59	25,357	
60	25,762	
61	26,167	
62	26,572	
63	26,977	
64	27,382	
65	27,787	
66	28,192	
67	22,597	
68	29,002	
69	29,407	

TANK CALIBRATION TABLE

TANK NUMBER: 60T41 (continued)

JSM/ff
9/7/73

Instrument Reading (%)	Gallons	Δ
70	29,812	404.9
71	30,217	
72	30,621	
73	31,026	
74	31,431	
75	31,836	
76	32,241	
77	32,646	
78	33,051	
79	33,459	
80	33,861	
81	34,266	
82	34,671	
83	35,676	
84	35,481	
85	35,885	

Instrument Reading (%)	Gallons	Δ
86	36,290	404.9
87	36,695	
88	37,100	
89	37,505	
90	37,910	
91	38,315	
92	38,920	
93	39,125	
94	39,530	
95	39,935	
96	40,340	
97	40,745	
98	41,149	
99	41,554	
100	41,959	

MONSANTO COMPANY

MONSANTO INDUSTRIAL CHEMICALS DIVISION

TEXAS CITY PLANT

STANDARD MANUFACTURING PROCESS FOR ETHYLENE VINYL CHLORIDE (Dept. 60)

Prepared by: M. E. Gibbs
R. T. Hammann
G. A. Hart
M. M. Siegel
A. Shadensack
J. Morgan

Approved by: J. L. Rasmussen
J. L. Rasmussen
Manufacturing Superintendent

R. W. Flint
Guest Superintendent

M. E. Gibbs
Research Group Leader

J. R. Savage
Manufacturing Manager

Copy 1 of 20 Issued: OCT 1 1972

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   c. Ammonium Persulfate
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   h. Mono Methyl Ether Hydroquinone
   i. Sodium Formaldehyde Sulfoxylate
   j. Caustic
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FM TO
MCC 9500

Monsanto

NAME & LOCATION G. A. Danner - Texas City

DATE March 12, 1974 CC

SUBJECT EVC Tentative Amendment B

REFERENCE

TO : Ethylene Vinyl Chloride SMP Holders #1

Attached is a copy of Amendment B to the Ethylene Vinyl Chloride SMP. Please add this to your copy of the SMP.

G. A. Danner jp

G. A. Danner

/jp
Attach.

Date: January 24, 1974

Tentative Amendment No. B

STANDARD MANUFACTURING PROCESS FOR ETHYLENE VINYL CHLORIDE

Product Code No.'s
840.61, 840.62, 840.63, 840.64

ISSUANCE OF CURRENT PROCESS DOCUMENT 1972

TITLE OF AMENDMENT: Autoclave Heatup for Hydrolyzing EVC-BH

PRESENT PRACTICE:

At the end of the reaction period when producing EVC-BH, the batch is heated from 55 to 60°C by the use of steam on the autoclave coils so that the product in the hydrolyzer will be greater than 45°C.

SUGGESTED CHANGE:

When the VCM addition is 90% complete, the steam valve on the autoclave will be manually blocked in and the temperature controller will be increased from 55 to 60°C letting the heat of reaction increase the temp. Due to a period of low VCM addition because of pressure control on the autoclave, the reaction cycle will be increased from 4 1/3 to 4 2/3 hours per batch.

JUSTIFICATION FOR CHANGE:

There is some indication that heating the latex using steam on the coils may have contributed to fouling of the autoclave coils. This suggested approach will allow the department to make hydrolyzed EVC-BH without using steam on the autoclave coils.

SAFETY:

No new property or personnel hazards will be introduced by the proposed change. All temperature and pressure shutdown devices will continue to be in service.

QUALITY:

No quality problems are expected from this change.

EXTENT OF DEMONSTRATION:

Permission is requested to run for three months. If successful this amendment will become part of the Standard Manufacturing Process.

Originated by: _____
R. T. Hammann

Endorsed by: _____
J. L. Rasmussen

APPROVED BY:

_____
R. W. Flint — Supt., Mfg.

_____
M. E. Gibbs — Research Group Leader

_____
W. R. Richard — Research Manager

_____
J. R. Savage — Mfg. Manager

mm

Monsanto

(NAME & LOCATION) J. S. Morgan - Texas City

DATE February 23, 1973 CC

SUBJECT EVC Tentative Amendment A

REFERENCE

TO : Ethylene Vinyl Chloride SMP Holders #2

Amendment A to the Ethylene Vinyl Chloride SMP was successfully demonstrated in October and November, 1972. Attached is a copy of the amendment to be included in your SMP.

J. S. Morgan

/jp
Attach.

Tentative Amendment A
to the
Ethylene Vinyl Chloride
Tentative Process

DATE: March 20, 1972

TENTATIVE AMENDMENT: A

TENTATIVE PROCESS REPORT FOR PRODUCTION OF POLVIN PIGMENT BINDER, MONSAT, AND MONFLEX

January 7, 1970. Report No. P-1556.

TITLE: Eliminate Second Water Wash

PRESENT PRACTICE:

The EVC reactors are washed with 55°C steam condensate twice after batch blowdown to remove any residual latex and grit particles that will foul the cooling coils. Every fifteenth reaction batch also includes a solvent (tetrahydrofuran - THF) wash between the first and second condensate washes to dissolve any coagulated latex that has built up on the cooling coils. The wash cycle consists of the following steps:

1.	Fill reactor (2,150 gallons) with condensate (or THF)	- 20 min.
2.	Agitate reactor contents	- 15 min.
3.	Pump out reactor contents to sewer (or THF storage)	- 10 min.
TOTAL		- 45 min.

PROPOSED CHANGE:

This amendment proposes the elimination of the second condensate wash - retaining the first condensate wash on each batch and the THF wash every 15 batches. Each THF wash will be followed by a second condensate wash to remove residual THF from the reactor.

JUSTIFICATION

The primary justification for eliminating the second water wash is the reduction of 2,150 gallons H_2O/batch (15.9 M gallons/day at standard production rate) to the sewer. This represents a

JUSTIFICATION (cont'd.)

raw material savings of $5 M/year and $16 M in proposed secondary waste treating capital. These figures are for standard product rate - 23.7 M lbs./yr. (January, 1972 Monthly Report - M. E. Gibbs). The wash elimination will result in an individual batch cycle reduction of 45 minutes (8 1/2%). No savings due to increased unit capacity can be claimed at this time.

The first water wash contains approximately 20 lbs. (1,100 ppm) of dissolved polymer solids and approximately 1/2 lb. of grit. The color of this stream is chalky white. The second water wash contains approximately 2 lbs. (110 ppm) polymer solids and has been observed to be clear with some grit (settleable) present. Each wash is pumped to the sewer which flows through the Primary Waste Treatment Area (startup 6/1/72) and the proposed secondary treatment facility and eventually to Galveston Bay.

Any latex left in the reactor prior to batch start will result in the production of some larger than normal particle sizes. The 2 lbs. of latex from the second wash will be left in the reactor prior to the next batch start. The amount of latex remaining, however, is slight and will probably not noticeably affect latex particle size.

BACKGROUND

The low solids content of the second water wash has been observed on every batch since the unit startup in November, 1971. The Tentative Process gives approximately 100 ppm as the solids concentration of the second wash. It has been observed the amount of solids in the washes can be reduced by optimizing the 2 low pressure (100 psig) reactor blowdowns.

SAFETY

Safety will not be affected.

QUALITY

Product quality will be monitored through regular analysis throughout the demonstration. The solids content of the first wash will also be monitored to determine if the solids concentration increases. Reactor heat transfer data will be taken to assure that the THF wash frequency is not increased to greater than 1/15 ba due to coil fouling. Particle size will be obtained every fifth batch during the demonstration.

EXTENT OF DEMONSTRATION

Permission is requested to run for one month - in order to get at least 15 batches on one reactor - with the second water wash eliminated. If in this period no "off-spec" product is produced and the THF wash frequency is not increased to greater than 1/15 batches (U overall should remain > 100 BTU/hr. ft.^{2}°F at max. heat load), the Tentative Amendment will be considered permanent.

Originated by: A. K. Shadensack

Endorsed by: R. T. Hammann

Approved by:

W. L. Smull 4/4/72 / W. L. Smull

J. L. Rasmussen / J. L. Rasmussen

R. W. Flint / R. W. Flint

M. E. Gibbs / M. E. Gibbs

W. R. Richard / W. R. Richard

Safety Review Committee

R. W. Radue 5/1/72 / ~~J. Fowler~~

J. F. Quinn 5/1/72 / J. F. Quinn

M. A. Terpstra 5/1/72 / M. A. Terpstra

J. R. Savage / J. R. Savage

/jp
4/7/72

BN 10
MCC 950930

Monsanto

NAME & LOCATION: J. S. Morgan - Texas City

DATE : February 20, 1973 CC:

SUBJECT : EVC Tentative Amendment A

REFERENCE : Plant Demonstration

TO : Ethylene Vinyl Chloride SMP Holders

Tentative Amendment A was successfully demonstrated October 28 - November 8, 1972. Objective was to eliminate the second autoclave water wash from the normal batch cycle. The results are shown below.

Objectives and Results
For Plant Demonstration

Performance Criteria	Objective	Results
THF Wash Frequency	15	15
Heat Transfer Coefficient (BTU/hr. F^2 °F)	>100	>178
Product Particle Size (A°)	650-900	685-827
Off-Spec Product Produced (lbs.)	0	0
Condensate Savings @ 23.7 $\overline{M}$ lbs./yr. ($/yr.)	5 M	5 M
Secondary Treatment Capital Savings ($)	16 M	16 M
Batch Cycle Reduction (%)	8.5	8.5

A tabulation of the complete test results is attached.

Conclusions

The second water wash can be eliminated from the batch cycle without effecting product quality or the frequency of the washes. The $5 M/yr. condensate savings (at 23.7 $\overline{M}$ lbs./yr.) and the 8 1/2% reduction in batch cycle time will be achieved.

This amendment does not include:

1. Elimination of the two water washes during the THF wash sequence.

2. New products with significantly different heat transfer requirements and fouling characteristics as compared to Latex B or E.

Disposition of Tentative Amendment D

The amendment is accepted. No modifications will be required to permanently implement the new procedure. The sequencer currently

halts after the first water wash and must be manually advanced to the subsequent step. Under Amendment A the sequencer is advanced to Step 25 and held until the next batch is started.

J. S. Morgan

/jp
Attach.

Date	Batch Number	Heat Trans. Coefficient*	Particle Size	Since THF Wash
10/28/72	223	238		1
10/29/72	225	236	795	2
10/29/72	227	211		3
10/30/72	229	205		4
10/30/72	230	213		5
10/31/72	233	206		6
10/31/72	235	185	827	7
11/1/72	237	188		8
11/2/72	239	182		9
11/3/72	241	196		10
11/4/72	243	192	822	11
11/5/72	246	181		12
11/6/72	248	169		13
11/7/72	250	182	685	14
11/8/72	252	178		15

* Assuming Coil Area = 468 ft.^{2}
H_2O Flow = 340 GPM

JSM/jp
2/20/73

I. SYNOPSIS OF PROCESS

I. SYNOPSIS OF PROCESS

The EVC department located at the Texas City plant was designed to produce 19 M pounds of EVC latex (weight on basis of dry polymer) per year via a Monsanto developed batch process. Three different latices are made, denoted as Latex B, Latex BH and Latex E. Capacity for Latex B and Latex E has been demonstrated at 22-23 $\overline{M}$ pounds per year. Capacity of BH is as yet not defined but is somewhat lower at present.

The basic raw materials include the three monomers: vinyl chloride, ethylene, acrylamide, and a surfacant, sodium lauryl sulfate. Ethylene is obtained via pipeline from the Polymers and Petrochemical Division facilities at both the Texas City and Chocolate Bayou plants. Vinyl chloride is shipped by rail car from Dow. Sodium lauryl sulfate is received in bulk and acrylamide in 50 pound bags.

The manufacture of EVC latex is centered in the polymerization step and accomplished in 2150 gallon, 2500 psi, stainless steel reactors. The reactors are equipped with high intensity agitation and internal coils which provide the ability to remove high heat loads. The reaction is a semi-batch type whereby initial charges of aqueous solution, vinyl chloride and ethylene are made batchwise and continuous charges of vinyl chloride and other aqueous streams are made on a programmed basis during ensuing reaction. Two basic reactions occur simultaneously: one, the redox catalyst reaction including reduction of ferric to ferrous ion by sulfoxylate and the coupling oxidation reaction regenerating ferric ion and decomposing persulfate ion to a free radical;and two, the subsequent free radical emulsion polymerization forming molecular weight chains of about 35,000 from the vinyl chloride, ethylene and acrylamide monomers. Molecular weight data and simplified equations follow:

Persulfate Redox Catalyst System

Reduction

$$HSO_2^- + Fe^{+3} \rightarrow Fe^{+2} + X$$

Sulfoxylate

Oxidation

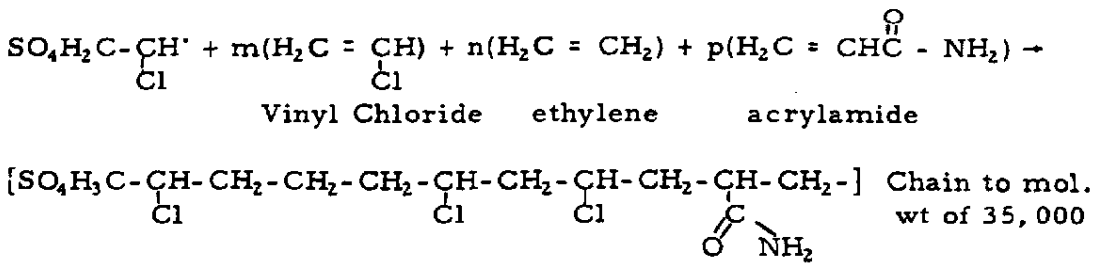
$$S_2O_8^{=} + Fe^{+2} \rightarrow Fe^{+3} + SO_4^{-\cdot} + SO_4^{=}$$

Polymerization Reaction

Initiation

$$SO_4^{-\cdot} + H_2C = \underset{Cl}{CH} \rightarrow SO_4H_2C - \underset{Cl}{CH}^{\cdot}$$

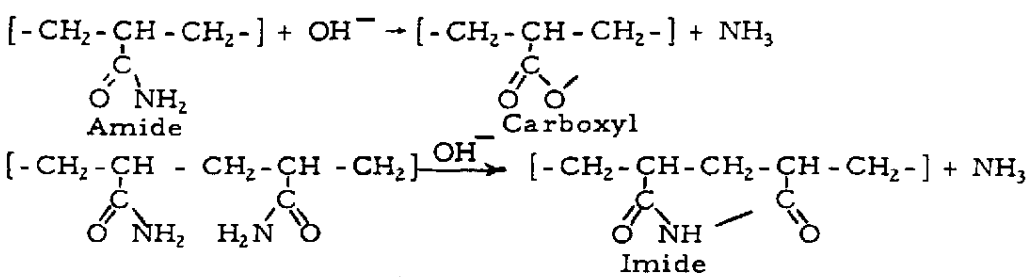
Chain Growth



The reaction is carried out at 55°C and 1750 - 1850 psi. Vinyl chloride is added to maintain constant pressure, as a pressure fall is associated with reaction of less dense monomers to more dense polymer. Surfactant addition maintains appropriate charge distribution on polymer particle surface to prevent coagulation. Acrylamide is added directly in proporation to degree of reaction. Two catalyst streams generate necessary free radicals. When reaction mass reaches a solids content of about 50%, reaction is terminated and blowdown to atmospheric pressure during which a gas/liquid phase separation occurs. The unreacted monomers are vented to an incineration system and the residual liquid latex is sent forward for further processing.

Primary difference in Latex B and Latex E is polymer composition which is determined during polymerization by feed rates and compositions. B type latices contain about 76% VCM, 21% ethylene and 3% acrylamide. E type latices contain more ethylene (29%) and less vinyl chloride (68%). No by-product, as such is made but some solid coagulum must be filtered out of the liquid latex before storage as final product. This is accomplished by a special type of vibrating filter screen.

Latex BH is produced by a further chemical processing step. B latex is hydrolyzed with caustic already diluted by previously hydrolyzed latex such that the caustic concentration is not too severe for virgin latex. High caustic concentrations can result in undesirable amounts of coagulation. The amide group contributed to the polymer of acrylamide is partially converted to carboxyl and imide as follows:



Hydrolysis is a relatively slow reaction requiring temperature, time and agitation to reach completion in a satisfactory batch cycle.

Following filtration, the latex is stored in bulk storage tanks. Loadout of the product is made from the storage facilities into tankcar, tank trucks, or into 55 gallon fibre drums.

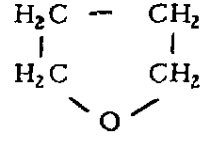
A tetrahydrofuran solvent system is provided to solvent wash reactors at a given frequency (currently after every 15 batches). Between solvent washes heat transfer coefficients decrease due to a solid film deposition on the cooling coils. When the coefficients reach a minimum value, solvent washing is instituted. A solvent recovery system is also provided to remove water and polymer solids from spent solvent. The recovery system consists primarily of a 26 foot packed, distillation tower.

EVC yields of major components are as follows:

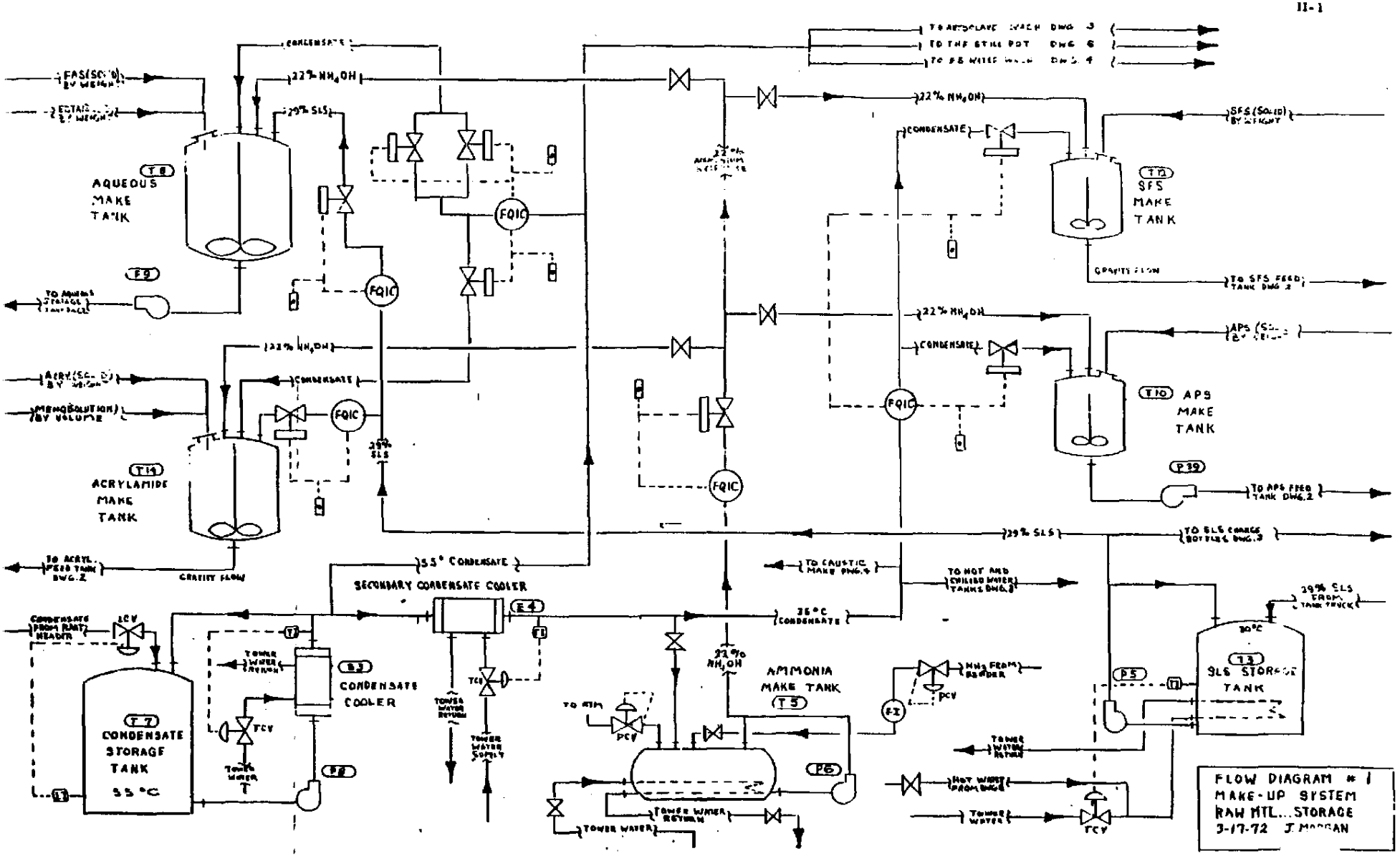
	Theory Usage		Design Usage	
Materials	Lbs/Cwt B	Lbs/Cwt E	Lbs/Cwt B	Lbs/Cwt E
VCM	72.4	65.2	82.7	79.5
Ethylene	21.0	28.0	36.1	42.8
Acrylamide	3.15	2.62	3.58	3.07
Sodium Lauryl Sulfate	7.29	7.11	8.35	8.06

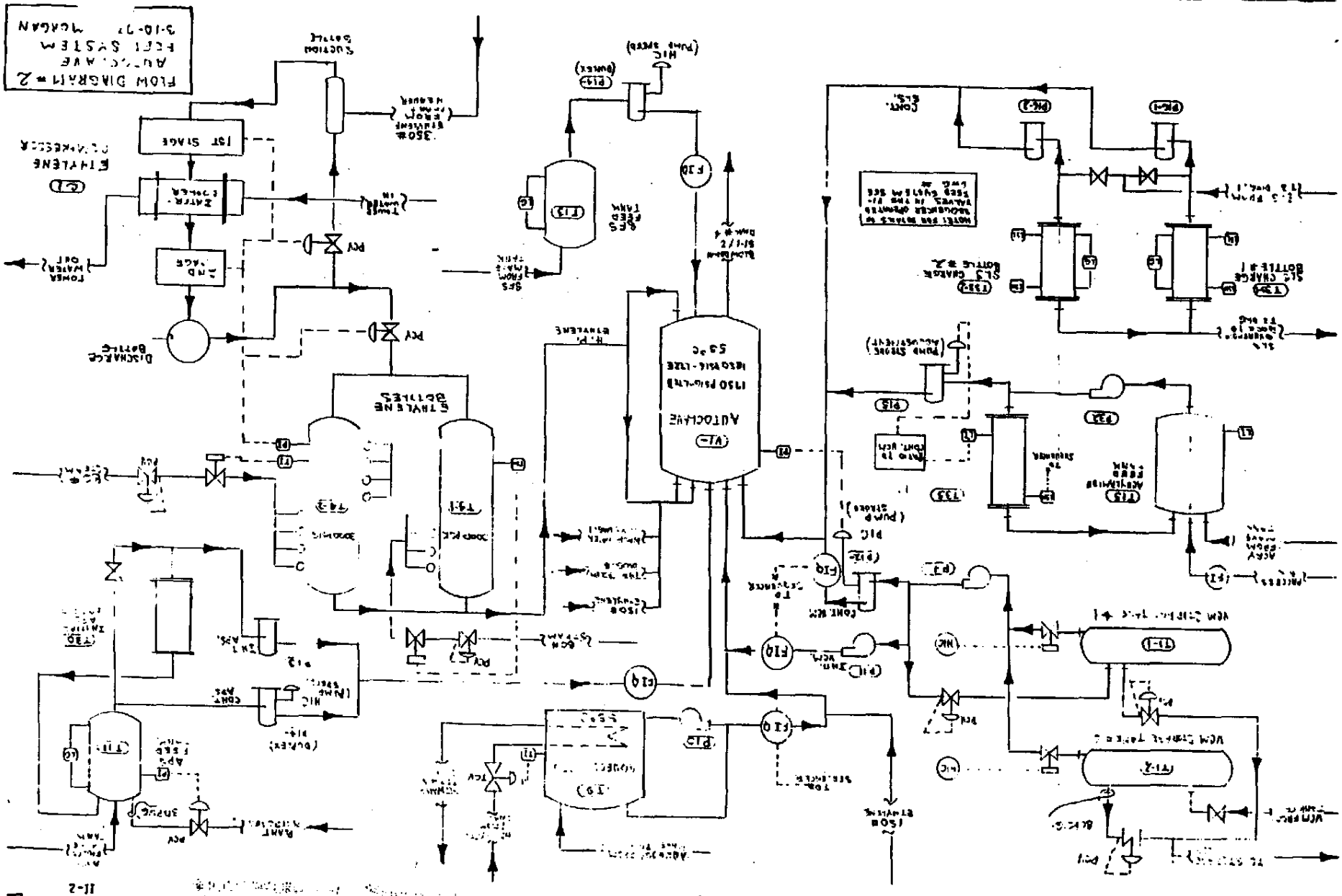
TABLE I

ABBREVIATIONS AND MOLECULAR WEIGHTS

Compound	Formula	Abbreviation	Molecular Weight
Acrylamide	$CH_2 = CHCONH_2$	A	71.08
Ammonium hydroxide	NH_4OH	NH_4OH	35.05
Ammonium persulfate	$(NH_4)_2S_2O_8$	APS	228.21
Ethylene	$H_2C=CH_2$	E	28.05
Ferric Ammonium Sulfate	$Fe(NH_4)(SO_4)_2 \cdot 12\ H_2O$	FAS	482.21
Sodium formaldehyde sulfoxylate	$NaHSO_2 \cdot CH_2O \cdot 2\ H_2O$	SFS	154.12
Sodium hydroxide	NaOH	NaOH	40.01
Tetrasodium Ethylene-diamine tetraacetate	$NaOOCCH_2$ CH_2COONa $N-CH_2-CH_2-N$ $NaOOCCH_2$ CH_2COONa	Na_4EDTA	380.18
Sodium lauryl sulfate	$C_{12}H_{25}OSO_3Na$	SLS	288.49
Vinyl Chloride	$H_2C=CHCL$	VCl	62.50
Water	HOH	H_2O	18.02
Tetrahydrofuran		FHF	72.10
Monomethyl ether hydroquinone	$CH_3OC_6H_4OH$	MEHQ	124.14

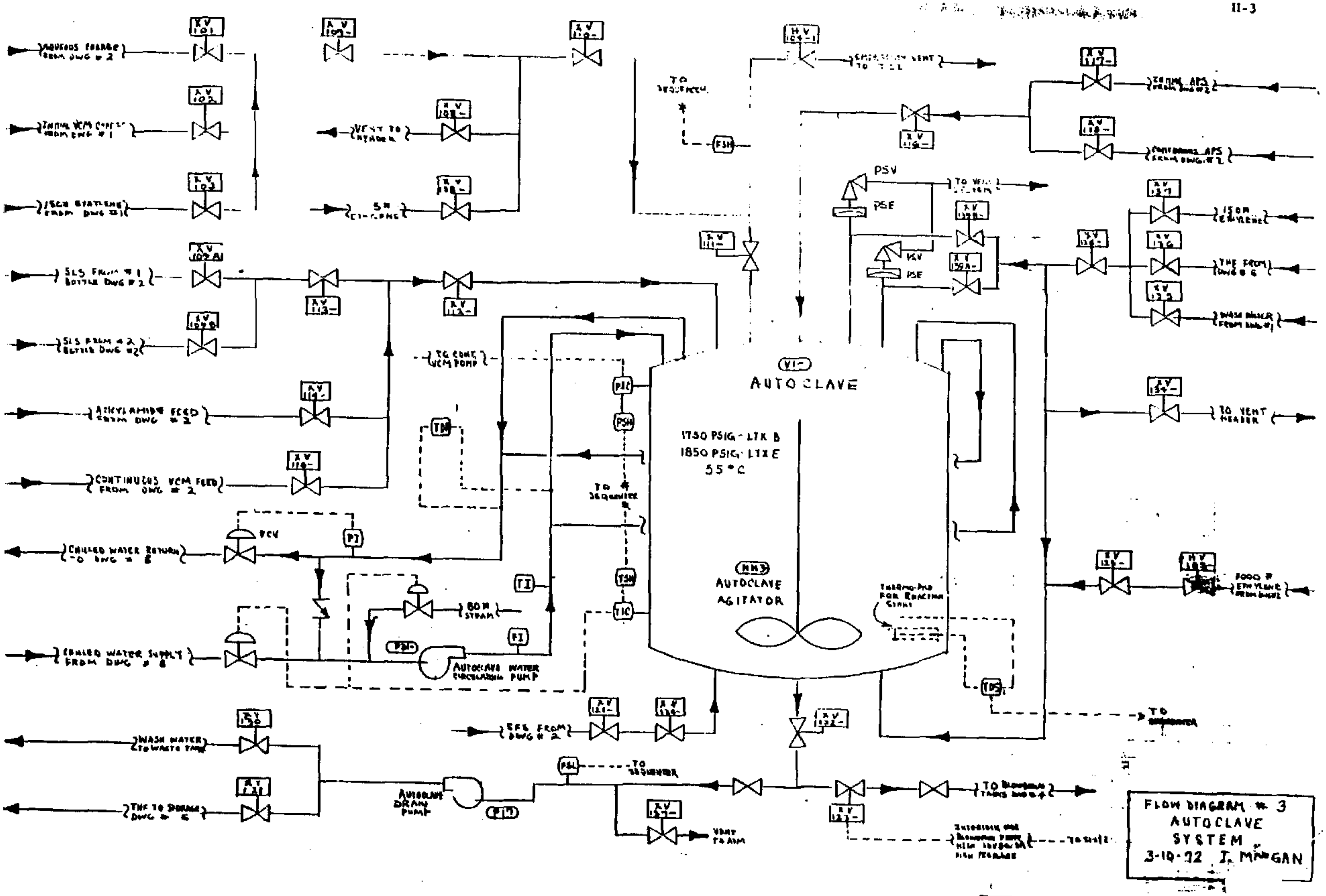
II. FLOW SHEETS


AQUEOUS MAKE TANK
T8
ACRYLAMIDE MAKE TANK
T14
SFS MAKE TANK
T11
APS MAKE TANK
T10
AMMONIA MAKE TANK
T5
CONDENSATE STORAGE TANK
55 °C
T7
SLS STORAGE TANK
T3
SECONDARY CONDENSATE COOLER
E4
CONDENSATE COOLER
E3
22% NH4OH
29% SLS
CONDENSATE
FQIC
P8
P39
P6
P5
FLOW DIAGRAM # 1
MAKE-UP SYSTEM
RAW MTL... STORAGE
3-17-72 J. MORGAN

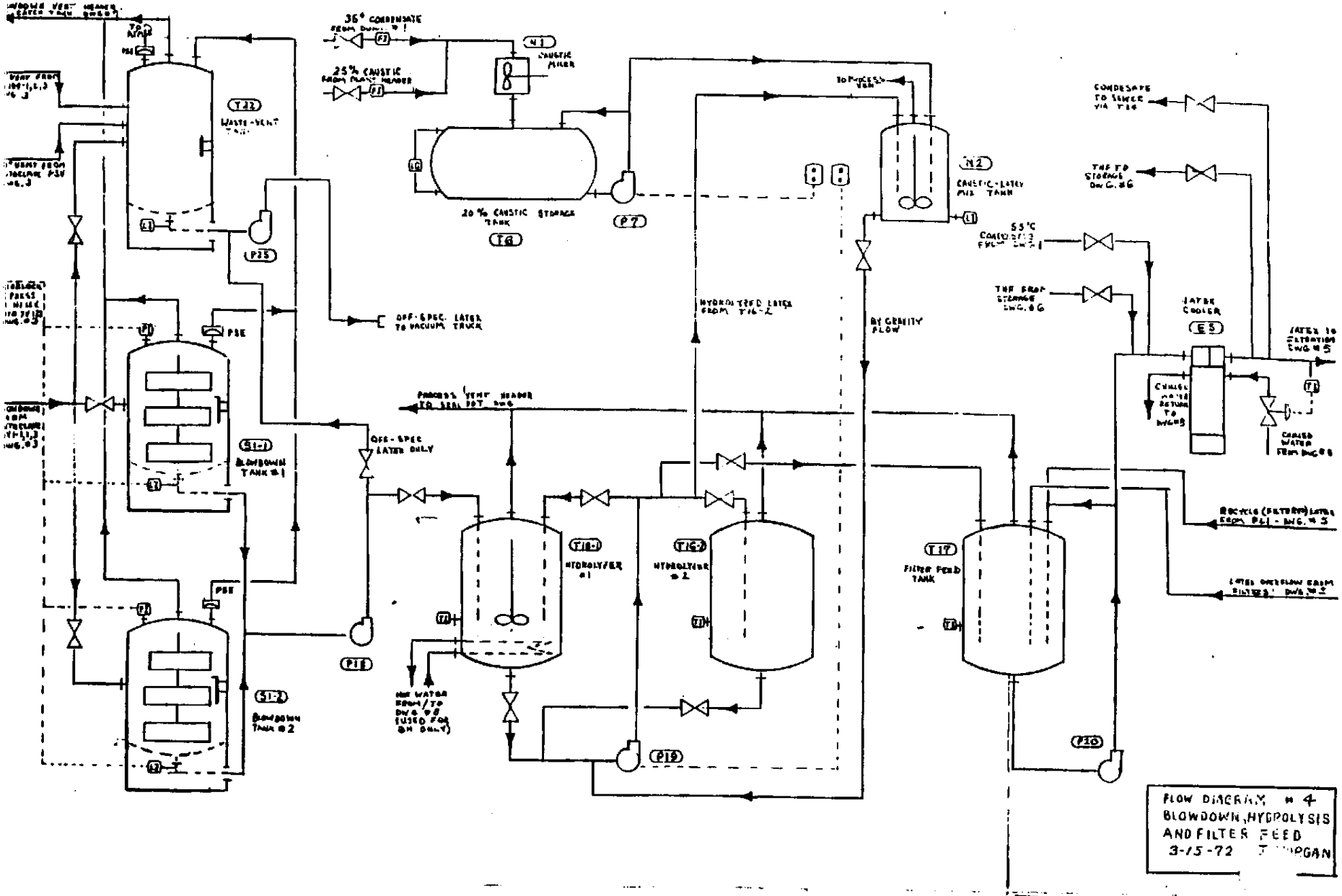


RSV 0015218

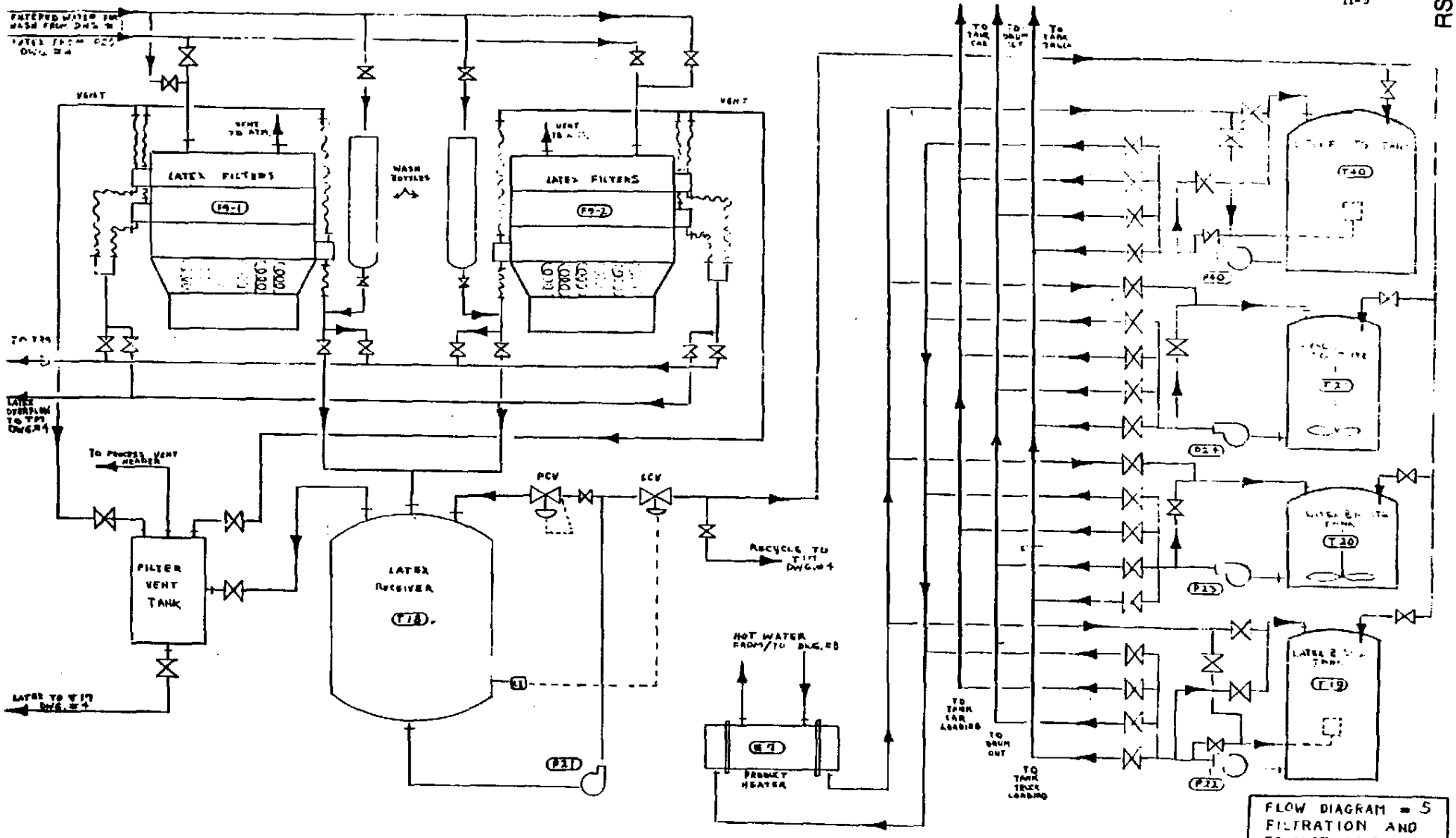
II-3

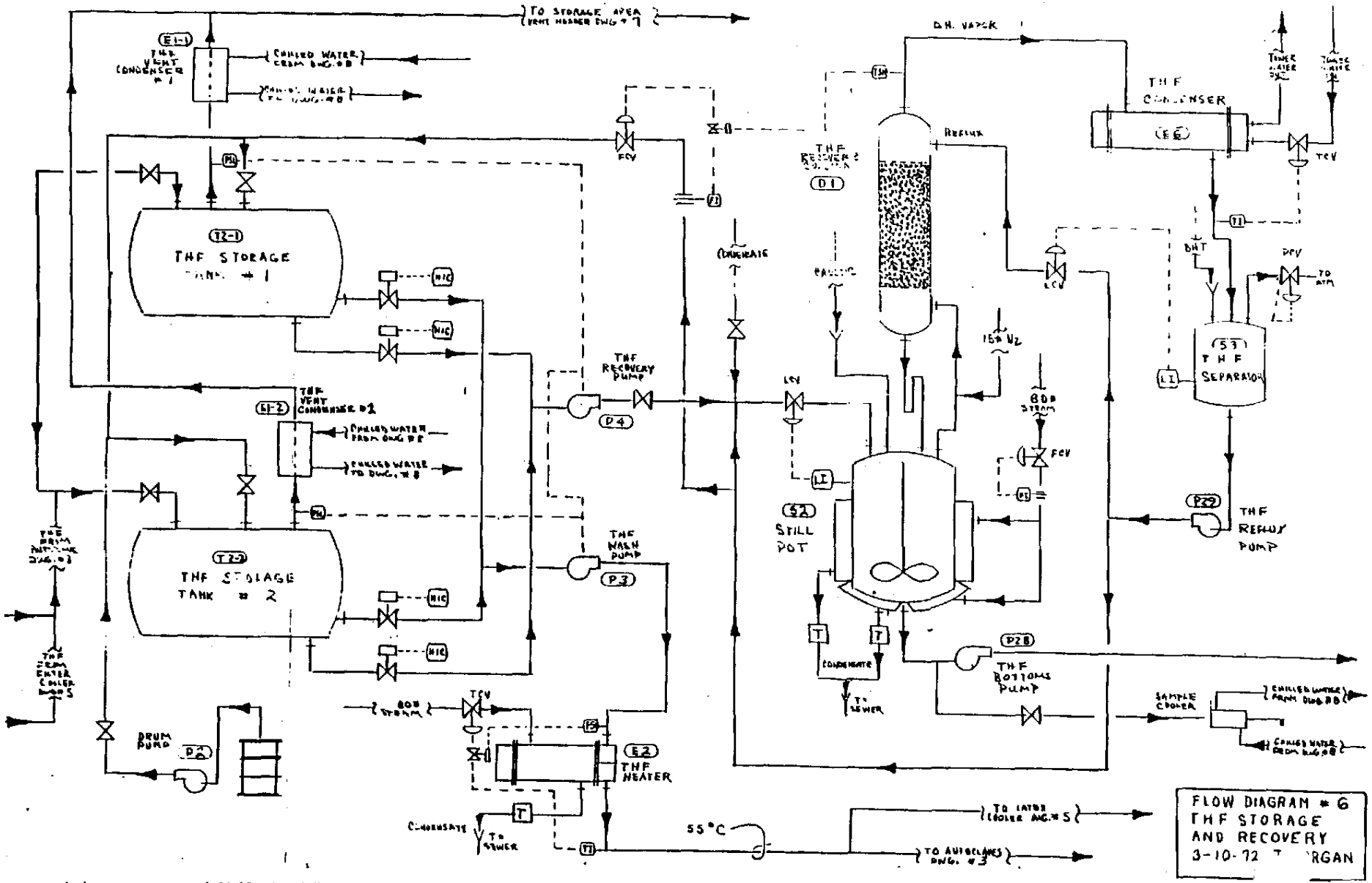


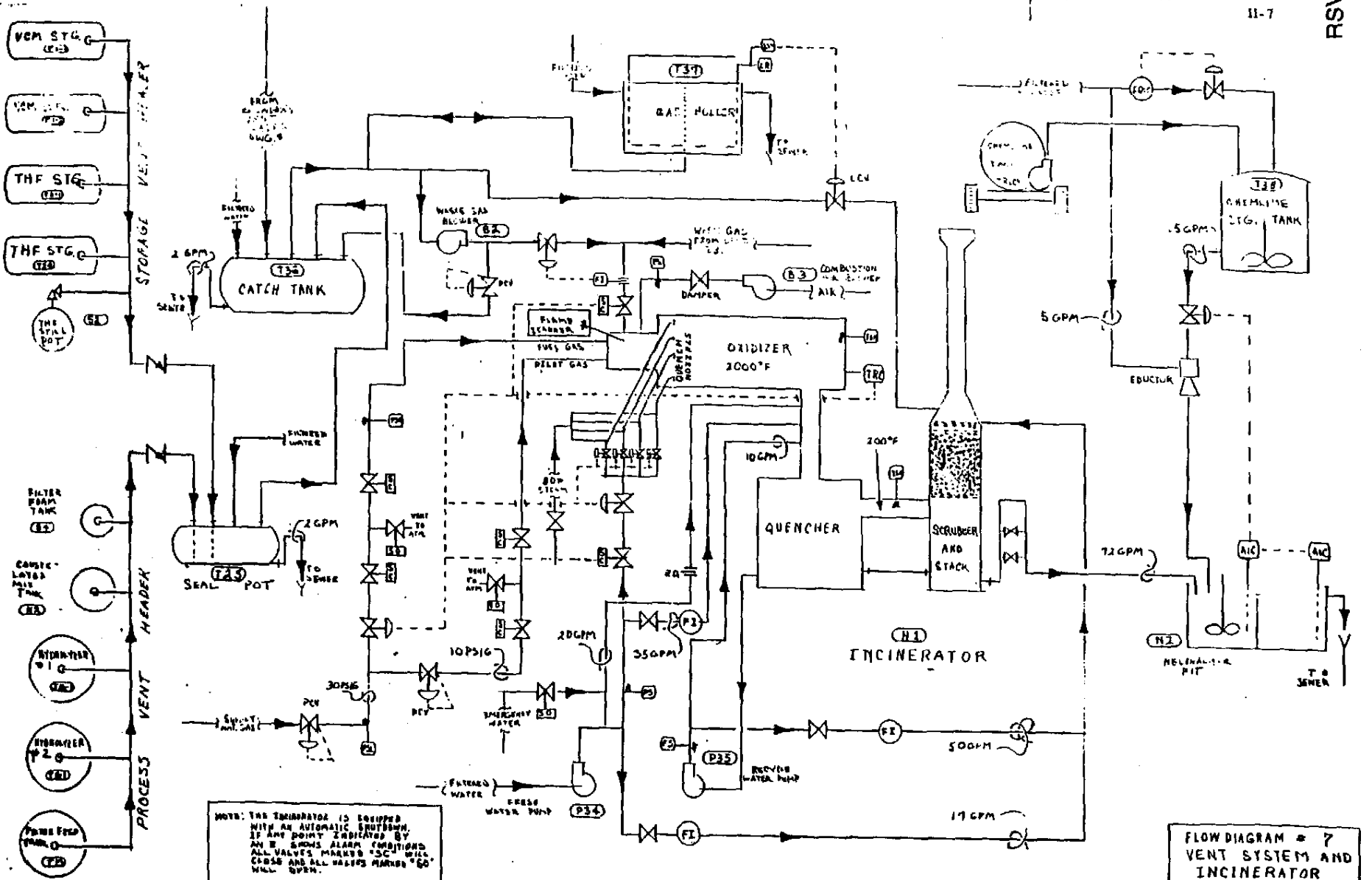
RSV 0015219



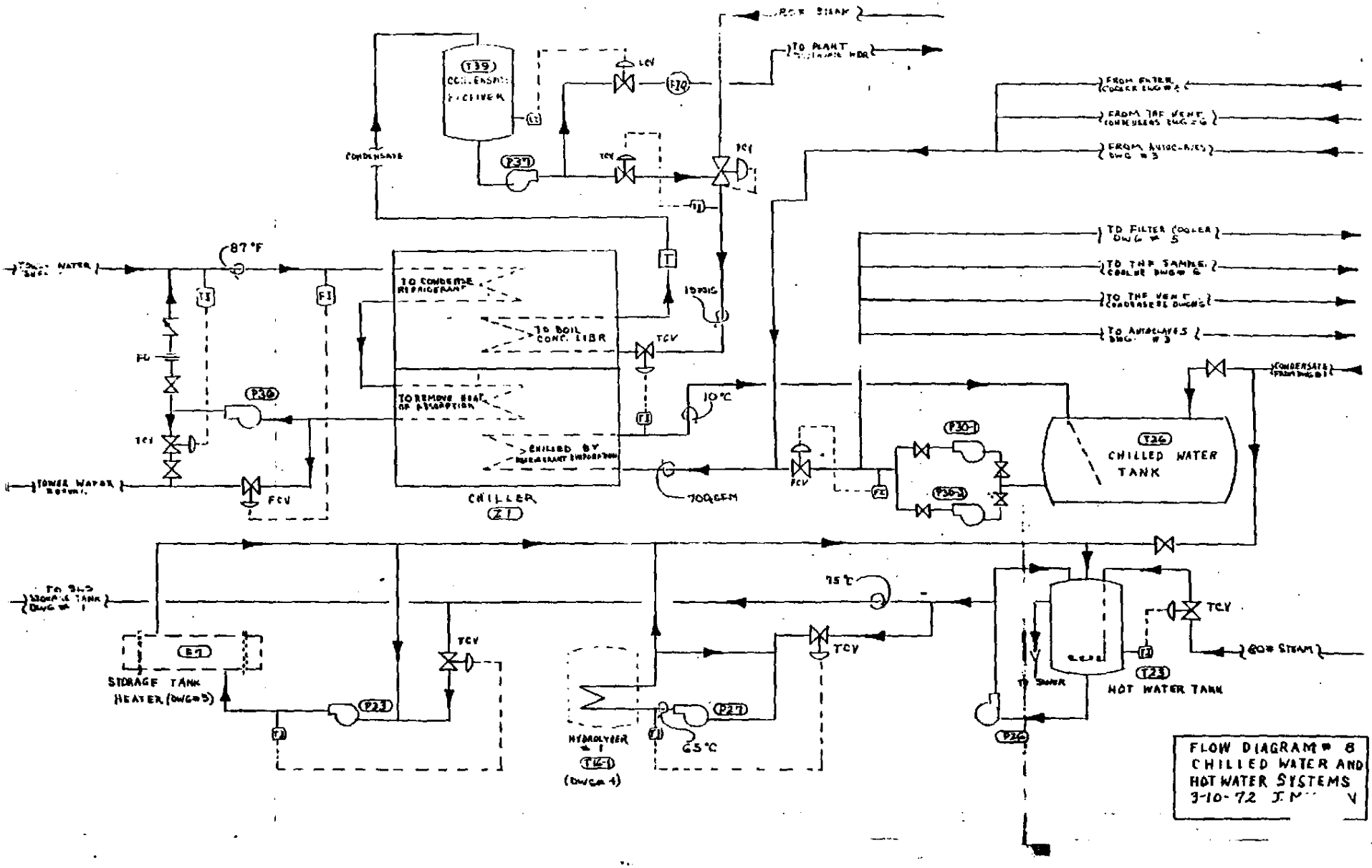
FLOW DIAGRAM # 4
BLOWDOWN, HYDROLYSIS AND FILTER FEED
3-15-72 J. HOGAN


LATEX FILTERS
F9-1
LATEX FILTERS
F9-2
WASH BOTTLES
VENT
VENT TO ATM.
FILTER VENT TANK
LATEX RECEIVER
T18
PCV
LCV
RECYCLE TO
HOT WATER
PRODUCT HEATER
E7
P21
TO TANK CAR
TO DRUM OUT
TO TANK TRUCK LOADING
T40
T2
T20
T19
P40
P22
P23
FLOW DIAGRAM = 5
FILTRATION AND
PRODUCT STORAGE
2-17-72


TO STORAGE AREA
E1-1
THF VENT CONDENSER #1
CHILLED WATER FROM BLDG #8
D.N. VAPOR
THF CONDENSER
E6
REFLUX
D1
T2-1
THF STORAGE TANK #1
CONDENSATE
PASSIVE
T1
BAT
PRV
TO ATM
TCV
FCV
LCV
15# N2
S3
THF SEPARATOR
LI
E1-2
THF VENT CONDENSER #2
P4
THF RECOVERY PUMP
T2-2
THF STORAGE TANK #2
P3
THF MAIN PUMP
S2
STILL POT
P29
THF REFLUX PUMP
P28
THF BOTTOMS PUMP
TO SEWER
SAMPLE COOLER
P2
DRUM PUMP
E2
THF HEATER
55 °C
TO LATEX COOLER BLDG. #5
TO AUTOCLAVES BLDG. #3
FLOW DIAGRAM # 6
THF STORAGE
AND RECOVERY
3-10-72 T. ORGAN



FLOW DIAGRAM # 7
VENT SYSTEM AND
INCINERATOR
3-10-72



FLOW DIAGRAM # 8
CHILLED WATER AND
HOT WATER SYSTEMS
3-10-72 J.M. V

III. PROCESS IN DETAIL

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III. PROCESS IN DETAIL

A. RAW MATERIALS SECTION

The following raw materials are used in theproduction of EVC Latexes:

1. Vinyl Chloride Monomer (VCM)

a.	Product:	B, BH, E
b.	Received:	Received from Dow in 20,000 gallon tank cars
c.	Storage: -	Stored in two (2) 24,000 gallon, horizontal, carbon steel storage tanks under 90 psig pressure

2. Ethylene (C_2H_4)

a.	Product:	B, BH, E
b.	Received:	Received from a plant header at 350 psig
c.	Storage:	Stored in two (2) - 372 cubic foot bottles under 3,000 psig of pressure

3. Acrylamide

a.	Product:	B, BH, E
b.	Received:	Received from American Cyanamid in 50 lb bags
c.	Storage:	The bags of acrylamide are stored in a solids storage shed until ready for use.

4. Sodium Lauryl Sulfate (SLS)

a.	Product:	B, BH, E
b.	Received:	Received from Proctor and Gamble in 4,500 gal. trucks
c.	Storage:	Stored in a 12,000 gallon, 304 SS storage tank.

5. Ammonium Persulfate (APS)

a.	Product:	B, BH, E
b.	Received:	Received from FMC Corp. in 250 lb fibre drums.
c.	Storage:	The drums of APS are stored in a solids storage shed until ready for use.

A. RAW MATERIALS SECTION (cont'd)

6. Ferric Ammonium Sulfate (FAS)

a.	Product:	B, BH, E
b.	Received:	Received from Mallinckrodt in 25 lb fibre drums
c.	Storage:	The drums of FAS are stored in a solids storage shed until ready for use.

7. Sodium formaldehyde Sulfoxalate (SFS)

a.	Product:	B, BH, E
b.	Received:	Received from Rohm and Haas in 325 lb. fibre drums.
c.	Storage:	The drums of SFS are stored in a solids storage shed until ready for use.

8. Tetrasodium Ethylenediamine Tetraacetate (EDTA)

a.	Product:	B, BH, E
b.	Received:	Received from Dow in 100 lb fibre drums
c.	Storage:	The drums of EDTA are stored in a solids storage shed until ready for use.

9. Ammonia

a.	Product:	B, BH, E
b.	Received:	Received from a plant header at 50 psig
c.	Storage:	Stored as a solution with water at 22.2%.

10. Caustic

a.	Product:	BH
b.	Received:	Received from a $\approx$ 25% plant header.
c.	Storage:	Stored at 20% concen. in a 4,000 gallon, horizontal, C.S. storage tank.

11. Tetrahydrofuran (THF)

a.	Product:	B, BH, E
b.	Received:	Received from DuPont in 500 lb drums and 6,000 gallon tank trucks
c.	Storage:	Stored in two (2) 11,200 gallon, horizontal storage tanks.