GEH-22837

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3A 51.2

Issued: January 13, 1956

ALUMINUM: IRON
Colorimetric Method

[redacted]

References

F. D. Snell and C. T. Snell, "Colorimetric Methods of Analysis," 2nd Ed., Vol. II, p. 757, D. Van Nostrand Co., Inc., New York, 1951

Principle, Limitations, and Precision

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

Equipment

For Preparation of Standard

Refer to Standard Procedure 3S[illegible].1, Iron - Colorimetric, Thioglycollic Acid

For Analysis of Sample

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

APPROVED FOR
PUBLIC RELEASE
D J Brown
11-7-07

Reagents and Chemicals

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

Safety Precautions

1. Dilute ammonium hydroxide or hydrochloric acid may cause painful burns on prolonged or repeated contact with the skin. Work in a well ventilated area or hood when handling dilute ammonium hydroxide or dilute hydrochloric acid. If any of the solution contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3A 51.2

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ALUMINUM: IRON
Colorimetric Method

Procedure

A. Preparation of Standard Curve

Follow Part A, Preparation of Standard Curve, of Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

B. Analysis of Sample

Using a 5 ml aliquot of sample solution from Part B, step 5 of Essential Materials Method 3A 51.1b, Aluminum: Silicon, Colorimetric Method, follow Standard Procedure 3SP 5.1, Iron - Colorimetric Method, Thioglycollic Acid.

[redacted]

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Essential Materials Methods

Code: 3A 60.1

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ALUMINUM NITRATE: WATER INSOLUBLES
Gravimetric Method

References

SRP Standard Procedure 3SP 13.0, Water Insolubles - Gravimetric Method

HW-12866, Hanford Works Laboratory Manual, Essential Materials Method ESI-1a

Principle, Limitations, and Precision

Refer to Standard Procedure 3SP 13.0, Water Insolubles- Gravimetric Method

Equipment

Refer to Standard Procedure 3SP 13.0, Water Insolubles - Gravimetric Method

Safety Precautions

Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a clean, fine porosity, sintered-glass crucible for one hour in an oven at 105-110 C. Allow the crucible to cool in a desiccator for thirty minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	1. Inspect all glassware. Use tongs when placing the crucible in and removing the crucible from the oven. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport desiccators.

[redacted]

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ALUMINUM NITRATE: WATER INSOLUBLES
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Weigh 10.0 ± 0.001 g of sample into a 250 ml beaker.	
3. Add 100 ml of hot distilled water to the beaker.	3. Handle the container of hot water with asbestos gloves or beaker tongs.
4. Follow steps 4 through 7 of Standard Procedure 3SP 13.0, Water Insolubles, Gravimetric Method.	4. Same as steps 4 through 7 of Standard Procedure 3SP 13.0, Water Insolubles, Gravimetric Method.

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ALUMINUM NITRATE: ALUMINUM
Qualitative Method

References

N. H. Furman and W. W. Scott, "Standard Methods of Chemical Analysis," 5th Ed., Vol. I, p. 2, D. Van Nostrand Co., Inc., New York, 1952

F. P. Treadwell and W. T. Hall, "Analytical Chemistry," 9th Ed., p. 191, John Wiley and Sons, Inc., New York, 1949

Principle, Limitations, and Precision

Alizarin S (sodium alizarin sulfonate) reacts with solutions containing aluminum to produce a red precipitate or red color. The precipitate is produced when large amounts of aluminum are present; the red color when small amounts of aluminum are present. The red color is formed in ammoniacal solutions and is stable to acetic acid. Calcium, strontium, barium, zinc, and magnesium salts are readily soluble in cold, dilute acetic acid and do not interfere with the coloration.

Equipment

2 Beakers, Pyrex, 150 ml
2 Beakers, Pyrex, 50 ml
1 Transfer pipet, 1 ml
1 Transfer pipet, 5 ml
1 Dropping pipet

Reagents and Chemicals

Alizarin sodium monosulfonic indicator, 0.1%	(RA 23.0)
Hydrochloric acid, CP, HCl, 36%	
Ammonium hydroxide, CP, NH_4OH, 28% NH_3	
Acetic acid, 1:1	(RA 15.3)

Safety Precautions

1. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

ALUMINUM NITRATE: ALUMINUM
Qualitative Method

Safety Precautions (contd.)

2. Concentrated ammonium hydroxide is very corrosive and may cause painful burns on contact with the skin. The fumes of ammonium hydroxide are irritating to the eyes and respiratory tract. When handling concentrated ammonium hydroxide in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the ammonium hydroxide contacts the skin, flush the affected area immediately with water.

3. Acetic acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the acetic acid contacts the skin, flush the affected area immediately with water.

4. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh 1 ± 0.1 g of sample into a 150 ml beaker. Dissolve the sample in 100 ml of distilled water.	1. Inspect all glassware.
2. Pipet 5 ml of the dissolved sample into a 50 ml beaker.	
3. Pipet 1 ml of 0.1% alizarin sodium monosulfonate indicator solution inte the sample solution.	
4. Acidify the solution with concentrated hydrochloric acid, as indicated by the yellow color of the solution. Make the solution alkaline with concentrated ammonium hydroxide as indicated by the purple color of the solution.	4. Use concentrated ammonium hydroxide in a well ventilated hood.

ALUMINUM NITRATE: ALUMINUM
Qualitative Method

Procedure (contd.)	Hazards Breakdown
5. Boil the solution for 2-3 minutes, cool to room temperature, and acidify with 1:1 acetic acid.	5. Use beaker tongs when removing the beaker from the hot plate.
6. A remaining red coloration or precipitate is conclusive evidence of the presence of aluminum.	
7. Report the absence or presence of aluminum.	

[redacted]

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ALUMINUM SULFATE(LIQUID): ALUMINUM OXIDE
Gravimetric Method

References

I. M. Kolthoff and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., p. 318, The Macmillan Co., New York, 1952

Principle, Limitations, and Precision

Aluminum is precipitated with ammonium hydroxide as the hydrous aluminum oxide and then ignited to aluminum oxide at a high temperature. The precipitation of the hydrous oxide begins at pH 3 and is completed at or before pH 7. The addition of ammonium chloride minimizes the coprecipitation of divalent metals such as calcium and magnesium, since the ammonium ion has a replacing effect upon the other cations. A correction for iron in the sample is made in the calculations.

The precision of the method is unknown.

Equipment

2 Crucibles and covers, platinum
1 Smith weighing buret, 10 ml
4 Beakers, Pyrex, 250 ml
1 Graduated cylinder, 50 ml
Filter paper, Whatman No. 42 and No. 41
2 Funnels, filtering, 75 mm diam
1 Transfer pipet, 3 ml

Reagents and Chemicals

Hydrochloric acid, CP, 36%	
Hydrogen peroxide, 3% by weight	(RH 105.6)
Ammonium chloride, CP, NH_4Cl	
Methyl red indicator, 0.1%	(RM 40.0)
Ammonium hydroxide, 1:1	(RA 75.0)
Ammonium chloride, 2% by weight	(RA 71.0)

[redacted]

Code: 3A 70.0

Issued: November 21, 1955

ALUMINUM SULFATE(LIQUID): ALUMINUM OXIDE
Gravimetric Method

Safety Precautions

1. Concentrated ammonium hydroxide is very corrosive and may cause painful burns on contact with the skin. The fumes of ammonium hydroxide are irritating to the eyes and respiratory tract. When handling concentrated ammonium hydroxide in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the ammonium hydroxide contacts the skin, flush the affected area immediately with water.

2. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Place a platinum crucible in a muffle furnace at 900 C for two hours. Allow the crucible to cool for 30 minutes in a desiccator. Weigh the crucible to the nearest 0.0001 g and record the weight.	1. Wear asbestos gloves and use platinum tipped tongs when inserting and removing crucibles from the furnace. Cool the furnace below 900 C to remove the crucibles. Allow the crucibles to cool slightly before closing the desiccator completely. Use a Desi-guard to transport the desiccator.

[redacted]

ALUMINUM SULFATE(LIQUID): ALUMINUM OXIDE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Mix the sample thoroughly to obtain a representative sample. Using a Smith weighing buret, weigh 1.0 to 1.1 ± 0.0001 g of sample into a 250 ml beaker containing 50 ml of water and 5 ml of concentrated hydrochloric acid. Record the weight.	2. Inspect all glassware. If any of the acid contacts the skin, flush the affected area immediately with water.
3. Heat the sample on a steam bath for 30 minutes.	3. Use tongs or wear asbestos gloves when removing rings or beakers from the steam bath.
4. If a precipitate is present, filter the sample through Whatman No. 42 filter paper into a 250 ml beaker. Wash the filter paper with five small portions of hot distilled water. Adjust the volume of the filtrate to approximately 100 ml with distilled water.	4. Use tongs or wear asbestos gloves to handle the hot glassware.
5. Using a transfer pipet, add 3 ml of 3% hydrogen peroxide to the sample. Boil the sample for 10 minutes.	
6. Add 5 g of ammonium chloride crystals and 5 drops of 0.1% methyl red indicator to the sample. If the indicator color is destroyed, continue boiling the sample until the indicator color remains when the indicator is added.	

[redacted]

ALUMINUM SULFATE(LIQUID): ALUMINUM OXIDE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
7. With the sample near the boiling temperature, add 1:1 ammonium hydroxide dropwise, until the color of the sample solution is a distinct yellow. Maintain this temperature for three minutes.	7. Work in a hood or well ventilated area.
8. Filter the hot solution through Whatman No. 41 filter paper into a 250 ml beaker. Wash the filter paper with five small portions of 2% ammonium chloride. Place the filter paper in the tared crucible.	8. Use tongs or wear asbestos gloves to handle the hot beaker.
9. Ignite the contents of the crucible in a muffle furnace at 1100 C for 1 hour. Transfer the crucible from the oven to a desiccator to cool for 30 minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	9. Wear asbestos gloves and use platinum tipped tongs when inserting and removing the crucibles from the muffle furnace. Cool the furnace below 900 C to remove the crucibles. Allow the crucibles to cool slightly before closing the desiccator completely. Use a Desi-guard to transport the desiccator.
10. Repeat step 9 until two consecutive weighings differ by no more than 0.0005 g.	10. Same as step 9.

Calculations

$$\% \ Al_2O_3 = \left[\frac{A - B}{C} \times 100 \right] - D$$

where A = weight of the sample crucible and precipitate, g (step 9)
B = weight of empty sample crucible, g (step 1)
C = weight of the sample, g (step 2)
D = % Fe_2O_3 obtained from Essential Materials Method 3A 70.2

[redacted]

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Essential Materials Methods

Code: 3A 70.1

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ALUMINUM SULFATE (SOLID): ALUMINUM SULFATE
Gravimetric Method

References

I. M. Kolthoff, and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., p. 318, The Macmillan Co., New York, 1952

Principle, Limitations, and Precision

Aluminum is precipitated with ammonium hydroxide as the hydrous aluminum oxide and then ignited to aluminum oxide at high temperature. The precipitation of the hydrous aluminum oxide begins at pH 3 and is completed at or before pH 7. The addition of ammonium chloride minimizes the coprecipitation of divalent metals such as calcium and magnesium, since the ammonium ion has a replacing effect upon the other cations. A correction for iron in the sample is made in the calculations.

The precision of the method is unknown.

Equipment

2	Crucibles and covers, platinum
2	Volumetric flasks, 200 ml
1	Transfer pipet, 20 ml
4	Beakers, Pyrex, 250 ml
1	Transfer pipet, 3 ml
	Filter paper, Whatman No. 41
2	Funnels, filtering, 75 mm diam

Reagents and Chemicals

Hydrogen peroxide, 3% by weight	(RH 105.6)
Ammonium chloride, CP, NH_4Cl	
Methyl red indicator, 0.1%	(RM 40.0)
Ammonium hydroxide, 1:1	(RA 75.0)
Hydrochloric acid, CP, HCl, 36%	
Ammonium chloride, 2% by weight	(RA 71.0)

[redacted]

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ALUMINUM SULFATE (SOLID): ALUMINUM SULFATE
Gravimetric Method

Safety Precautions

1. Concentrated ammonium hydroxide is very corrosive and may cause painful burns on contact with the skin. The fumes of ammonium hydroxide are irritating to the eyes and respiratory tract. When handling concentrated ammonium hydroxide in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the ammonium hydroxide contacts the skin, flush the affected area immediately with water.

2. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	**Hazards Breakdown**
In the analysis of a single sample run duplicate determinations.	
1. **Place a platinum crucible in a muffle furnace at 900 C for two hours. Place the** crucible in a desiccator to cool for 30 minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	1. **Wear asbestos gloves and use platinum tipped tongs when inserting and removing the crucible from** the furnace. Cool the furnace below 900 C to remove the crucible. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport desiccators.

[redacted]

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ALUMINUM SULFATE (SOLID): ALUMINUM SULFATE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Weigh 5.0 to 5.2 ± 0.0001 g of sample. Transfer the sample to a 200 ml volumetric flask.	2. Inspect all glassware.
3. Dissolve the sample in distilled water, dilute to calibration mark, and mix well.	3. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
4. Pipet a 20 ml aliquot of the dissolved sample into a 250 ml beaker. Dilute to approximately 100 ml with distilled water.	
5. Using a transfer pipet, add 3 ml of 3% hydrogen peroxide to the sample. Boil the sample for 10 minutes.	
6. Add 5 g of ammonium chloride crystals and 5 drops of 0.1% methyl red indicator to the sample. If the indicator color is destroyed, continue boiling the sample until the indicator color remains when the indicator is added.	
7. With the sample near the boiling temperature, add 1:1 ammonium hydroxide, dropwise, until the color of the sample solution is a distinct yellow. Maintain this temperature for three minutes.	7. Work in a hood or well ventilated area.

[redacted]

ALUMINUM SULFATE (SOLID): ALUMINUM SULFATE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
8. Filter the hot solution through Whatman No. 41 filter paper into a 250 ml beaker. Wash the filter paper with five small portions of 2% ammonium chloride. Place the filter paper in the tared crucible.	8. Use tongs or wear asbestos gloves to handle the hot beaker.
9. Ignite the contents of the crucible in a muffle furnace at 1100 C for 1 hour. Transfer the crucible from the oven to a desiccator to cool for 30 minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	9. Wear asbestos gloves and use platinum tipped tongs when inserting and removing the crucible from the furnace. Cool the furnace below 900 C to remove the crucible. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport desiccators.
10. Repeat step 9 until two consecutive weighings differ by no more than 0.000[illegible] g.	

Calculations

$$\% \, Al_2O_3 = \left[\frac{(A - B) \times C \times 100}{D \times F}\right] - E$$

where A = weight of crucible plus precipitate, g (step 9)
B = weight of empty crucible, g (step 1)
C = volume to which the original sample was diluted = 200 ml (step 3)
D = volume of aliquot taken for analysis = 20 ml (step 4)
E = $\% \, Fe_2O_3$ obtained from Essential Materials Method 3A 70.2
F = weight of sample, g (step 2)

$$\% \, Al_2(SO_4)_3 = \% \, Al_2O_3 \times G$$

where G = factor for converting aluminum oxide to aluminum sulfate $= \frac{342.14}{101.94} = 3.36$

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Code: 3A 70.3

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ALUMINUM SULFATE (SOLID): IRON
Colorimetric Method

References

F. D. Snell and C T. Snell, "Colorimetric Methods of Analysis," 3rd Ed., Vol. II, p. 319, D. Van Nostrand Co., Inc., New York, 1951

Principle, Limitations, and Precision

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

Equipment

For Preparation of Standard Curve

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

For Analysis of Sample

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

Reagents and Chemicals

Refer to Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

Safety Precautions

1. Dilute ammonium hydroxide or hydrochloric acid may cause painful burns on prolonged or repeated contact with the skin. Work in a well ventilated area or hood when handling dilute ammonium hydroxide or dilute hydrochloric acid. If any of the solution contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

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ALUMINUM SULFATE (SOLID): IRON
Colorimetric Method

Procedure

A. Preparation of Standard Curve

Follow Part A, Preparation of Standard Curve, of Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

B. Analysis of Sample

Follow Part B, Analysis of Sample, of Standard Procedure 3SP 5.1, Iron - Colorimetric, Thioglycollic Acid

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Essential Materials Methods

Code: 3B 9.0

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BARIUM CHLORIDE: BARIUM
Qualitative Method

References

F. P. Treadwell and W. T. Hall, "Analytical Chemistry," Vol. I, 9th Ed., p. 457, John Wiley and Sons, Inc., New York, 1949

Principle, Limitations, and Precision

The sample is dissolved in distilled water and a small amount of the solution is introduced into a flame of a Bunsen burner by means of a platinum wire. The appearance of a greenish yellow flame indicates the presence of barium.

Equipment

2 Beakers, 150 ml
2 Stirring rods, Pyrex, 8-10 in. in length
1 Wire, platinum, 8-10 in. in length
1 Bunsen burner

Reagents and Chemicals

Hydrochloric acid, 1:1 (RH 97.0)

Safety Precautions

1. Hydrochloric acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the sulfuric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh, on the triple beam balance, approximately 1.0 g of sample into a 150 ml beaker.	1. Inspect all glassware.

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Issued: December 14, 1955

BARIUM CHLORIDE: BARIUM
Qualitative Method

Procedure (Contd.)	Hazards Breakdown
2. Dissolve the sample in approximately 50 ml of distilled water.	
3. Dip a clean platinum wire in the sample solution and then introduce the wire into the flame of the Bunsen burner. (Remark 1) Note the color of the flame.	
4. Dip the wire in hydrochloric acid to clean it, and then repeat step 3.	4. To prevent spattering, allow the platinum wire to cool before cleaning with the acid.
5. A greenish yellow flame confirms the presence of barium. Report barium as present or absent.	
6. Clean the platinum wire with hydrochloric acid before returning the wire to the proper place.	6. Same as step 4.

Remarks

1. The platinum wire must be clean and free from sodium and barium before the test can be performed. Clean the wire by dipping it into dulute hydrochloric acid and then introducing it into the flame. Repeat this cleaning until the wire produces no color when introduced into the flame.

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BARIUM CHLORIDE: CHLORIDE
Qualitative Method

References

I. M. Kolthoff and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., p. 303-308, The Macmillan Co., New York, 1952

Principle, Limitations, and Precision

Refer to Standard Procedure 3SP 2.5, Chloride: Qualitative Determination

Equipment

Refer to Standard Procedure 3SP 2.5, Chloride: Qualitative Determination

Reagents and Chemicals

Refer to Standard Procedure 3SP 2.5, Chloride: Qualitative Determination

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

2. Silver nitrate solution is caustic. It is poisonous when ingested and discolors the skin on contact.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3B 9.1

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BARIUM CHLORIDE: CHLORIDE
Qualitative Method

Procedure

Follow Standard Procedure 3SP 2.5, Chloride: Qualitative Determination.

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Essential Materials Methods

Code: 3C 1.0

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CARBON TETRACHLORIDE: ACIDITY

References

"Reagent Chemicals, ACS Specifications," p. 104, Washington, D. C., 1950

Principle, Limitations, and Precision

A water extract of the carbon tetrachloride is made slightly alkaline with sodium hydroxide and then tested for acidity with phenolphthalein indicator. The method is empirical and the prescribed conditions must be closely followed.

Equipment

1 Graduated cylinder, glass-stoppered, 50 ml
1 Transfer pipet, 10 ml
1 Erlenmeyer flask, 25 ml
1 Mohr measuring pipet, 1 ml

Reagents and Chemicals

Sodium hydroxide, 0.1 N	(RS 60.1)
Phenolphthalein indicator, 1%	(RP 33.0)
Water, deionized and distilled	(RW 8.4)

Safety Precautions

1. Carbon tetrachloride is toxic by inhalation, ingestion, or by prolonged contact with the skin. Carbon tetrachloride is dangerous to the eyes as a vapor or liquid. Wear rubber gloves and work in a hood or well ventilated area.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
1. Add 13 ml of the sample to a 50 ml glass-stoppered, graduated cylinder containing 20 ml of deionized distilled water.	1. Work in a well ventilated hood. Wear rubber gloves and flush any contacted areas immediately with water.

[redacted]

CARBON TETRACHLORIDE: ACIDITY

Procedure (contd.)	Hazards Breakdown
2. Stopper the cylinder and shake the solution vigorously for five minutes.	2. When shaking the cylinder, place the index and middle fingers on the glass stopper, allowing the ring and little fingers and the thumb to grasp the cylinder. Hold the cylinder securely with the other hand. Several times during the shaking period, remove the stopper to release the pressure.
3. Remove the stopper and allow the layers to separate. Pipet 10 ml from the top (water) layer into a 25 ml Erlenmeyer flask.	
4. Add 2 drops of 1% phenolphthalein indicator and 0.05 ml of 0.1 N sodium hydroxide to the flask. Swirl the flask to mix the solution.	
5. Observe the color of the solution. If the color is pink or red, report that the sample passes the test. If the solution is colorless, report that the sample fails to pass the test.	

[redacted]

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Essential Materials Methods

Code: 3C 1.1

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CARBON TETRACHLORIDE: DISTILLATION RANGE

References

ASTM Standards, Part 5, D 86-46, D 216-40, D 1078-49T, Philadelphia, Pa., 1949

Principle, Limitations, and Precision

Refer to SRP Standard Procedure 3SP 3.0, Distillation Range

Equipment

1 Engler flask, 100 ml
1 Thermometer, ASTM Solvents Distillation, E 1 (39C - 44T), Range 48 to 102 C
Distillation range apparatus (ED 44.0)

Safety Precautions

1. Do not use external flames or hot exposed heating elements near the receiver of the distillation apparatus. Work in a well ventilated area.

2. Carbon tetrachloride is toxic by inhalation, ingestion, or by prolonged contact with the skin. Wear rubber gloves, and flood any contacted areas with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure

Follow Standard Procedure 3SP 3.0, Distillation Range. Record only the initial boiling point (IBP) and the dry point temperatures corrected to standard barometric pressure.

[redacted]

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Essential Materials Methods

Code: 3C 1.2

Issued: November 21, 1955

CARBON TETRACHLORIDE: RESIDUE ON EVAPORATION
Gravimetric Method

References

SRP Standard Procedure 3SP 9.0, Residue on Evaporation: Gravimetric Method

"Reagent Chemicals, ACS Specifications," ACS, Washington, D. C., 1950

Principle, Limitations, and Precision

Refer to Standard Procedure 3SP 9.0, Residue on Evaporation, Gravimetric Method

Equipment

1 Smith weighing buret, 30 ml
2 Evaporating dishes, platinum, 50 ml

Safety Precautions

1. Carbon tetrachloride is toxic when inhaled or on prolonged or repeated contact with the skin. When handling carbon tetrachloride, work in a well ventilated area. Wash the hands thoroughly after handling the material.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure

Using a 40.0 to 50.0 ± 0.01 g sample (28.4 to 31.2 ml), follow Standard Procedure 3SP 9.0, Residue on Evaporation, Gravimetric Method.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3C 2.0

Issued: November 25, 1955

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. I. p. 209-211, D. Van Nostrand Co., Inc., New York, 1952

Principle, Limitations, and Precision

Calcium oxalate is precipitated from a mild ammoniacal solution with ammonium oxalate. The presence of ammonium chloride prevents the precipitation of magnesium but does not interfere with the precipitation of calcium. Calcium oxalate formed from hot solutions is crystalline or granular and filters more readily than the flocculant precipitate formed in cold solutions. Calcium oxalate is decomposed at red heat to calcium oxide; the calcium oxide is weighed and reported as percent calcium carbonate.

The precision of the method is unknown.

Equipment

3 Crucibles and covers, platinum
3 Beakers, Pyrex, 250 ml
1 Graduated cylinder, 50 ml
1 Steam bath
3 Stirring rods, Pyrex, 8-10 in. long
Filter paper, Whatman No. 41 and 42
3 Funnels, filtering, 75 mm diam
6 Beakers, Pyrex, 400 ml

Reagents and Chemicals

Hydrochloric acid, CP, HCl, 36%	
Bromine water, saturated	(RB 72.0)
Methyl red indicator, 0.1% by weight	(RM 40.0)
Ammonium hydroxide, CP, NH_4OH, 28% NH_3	
Ammonium chloride, 2% by weight	(RA 71.0)
Ammonium oxalate, saturated	(RA 81.0)

[redacted]

Code: 3C 2.0

Issued: November 25, 1955

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

Safety Precautions

1. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

2. When handling bromine water, work in a well ventilated, flame-free hood. The vapors of bromine are extremely irritating to the skin, eyes, and mucous membranes. If any of the bromine water contacts the skin, wash the affected area immediately with mild soap and water. Store bromine water in a secondary earthenware container in a well ventilated hood.

3. Concentrated ammonium hydroxide is very corrosive and may cause painful burns on contact with the skin. The fumes of ammonium hydroxide are irritating to the eyes and respiratory tract. When handling concentrated ammonium hydroxide in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the ammonium hydroxide contacts the skin, flush the affected area immediately with water.

4. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

[redacted]

Code: 3C 2.0

Issued: November 25, 1955

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Place two platinum crucibles in a muffle furnace at 900 C for two hours. Remove the crucibles from the furnace and allow them to cool for 30 minutes in a desiccator. Weigh the crucibles to the nearest 0.0001 g and record the weights.	1. Wear asbestos gloves and use platinum tipped tongs when inserting and removing crucibles from the furnace. Cool the furnace below 900 C to remove the crucibles. Allow the crucibles to cool slightly before closing the desiccator completely. Use a Desiguard to transport desiccators.
2. Weigh 0.15 to 0.16 ± 0.001 g of sample into a clean, dry, 250 ml beaker. Record the weight of the sample.	2. Inspect all glassware.
3. Add approximately 50 ml of distilled water and 10 ml of concentrated hydrochloric acid to dissolve the sample.	3. If any of the acid contacts the skin, flush the affected area immediately with water.
4. Prepare a blank by adding 50 ml of distilled water and 10 ml of concentrated hydrochloric acid to another clean, dry, 250 ml beaker. Treat the sample and the blank in the same manner throughout the remainder of the procedure.	4. Same as step 3.
5. Heat the sample on a steam bath with occasional stirring until the sample is completely dissolved.	5. Remove the rings from the steam bath with tongs.

[redacted]

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
6. Remove the sample from the steam bath and add 2-3 drops of saturated bromine water. Boil the sample on a hot plate until all traces of the bromine have disappeared.	6. Work in a well ventilated hood. Store the bromine water in a secondary earthenware container in the hood. Place a HOT sign in front of the hot plate.
7. Add 5 ml of concentrated hydrochloric acid and dilute the sample to approximately 200 ml with distilled water.	7. If any of the acid contacts the skin, flush the affected area immediately with water.
8. Add 2-3 drops of 0.1% methyl red indicator and heat the sample to boiling. Add concentrated ammonium hydroxide, dropwise, until the color of the solution is a distinct yellow.	8. Work in a well ventilated hood. Place a HOT sign in front of the hot plate.
9. Heat the sample at a simmering temperature for two minutes, and if a precipitate is present, allow it to settle and then filter the solution through Whatman No. 41 filter paper into a clean, dry, 400 ml beaker.	9. Wear asbestos gloves or use beaker tongs to handle the hot beaker.
10. Wash the precipitate three times with small portions of 2% ammonium chloride. Add the washings to the filtrate; discard the precipitate.	10. Same as step 9.
11. Add concentrated ammonium hydroxide, dropwise, until the color of the filtrate is a distinct yellow. Bring the solution to a boil and add, with constant stirring, 35 ml of boiling, saturated ammonium oxalate.	11. Same as step 9.

[redacted]

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
12. Allow the solution to stand 30 minutes and filter through Whatman No. 42 filter paper into a 400 ml beaker.	
13. Wash the precipitate ten times with 10-12 ml portions of hot, distilled water, adding the washings to the filtrate.	13. Same as step 9.
14. Transfer the filter paper and precipitate to the tared crucible. Cover the crucible and place it in a muffle furnace. Gently heat the crucible until the water is expelled and the paper is charred.	14. Use platinum tipped tongs when inserting and removing platinum crucibles from the furnace.
15. Increase the temperature to 900 C and ignite the crucible and precipitate for one hour.	
16. Remove the crucible from the furnace. Place the crucible in a desiccator to cool for 30 minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	16. Wear asbestos gloves and use platinum tipped tongs to remove the crucible from the furnace. Allow the furnace to cool below 900 C to remove the crucible. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport desiccators.
17. Repeat steps 15 and 16 until two consecutive weighings differ by not more than 0.0005 g.	

[redacted]

Code: 3C 2.0

Issued: November 25, 1955

CALCIUM CARBONATE: CALCIUM CARBONATE
Gravimetric Method

Calculations

$$\% \text{ Calcium carbonate} = \frac{[(A - B) - (C - D)] \times E \times 100}{F}$$

where A = weight of sample crucible plus residue, g (step 16)
B = weight of empty sample crucible, g (step 1)
C = weight of blank crucible plus residue, g (step 16)
D = weight of empty blank crucible, g (step 1)
E = factor for converting calcium oxide to calcium carbonate $= \frac{100.09}{56.08} = 1.7848$
F = weight of sample, g (step 2)

SHP Laboratory Manual
Essential Materials Methods

Code: 3D 20.0

Issued: December 20, 1955

DUPONOL, ME, DRY: FATTY ACIDS
Qualitative Method

References

F. P. Treadwell and W. T. Hall, "Analytical Chemistry", Vol. I, 9th Ed., p. 477, John Wiley and Sons, Inc., New York, 1949

Principle, Limitations, and Precision

A weighed sample is dissolved in distilled water and acidified with concentrated hydrochloric acid. The solution is heated on a hot plate and stirred frequently. The presence of fat near the surface of the solution confirms the presence of fatty acids.

Equipment

1 Beaker, Pyrex, 250 ml
1 Graduated cylinder, 100 ml

Reagents and Chemicals

Hydrochloric acid, CP, HCl, 37%

Safety Precautions

1. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3D 20.0

Issued: December 20, 1955

DUPONOL, ME, DRY: FATTY ACIDS
Qualitative Method

Procedure	Hazards Breakdown
1. Weigh, on a triple beam balance, approximately 5.0 g of the sample into a 250 ml beaker.	1. Inspect all glassware.
2. Add approximately 100 ml of distilled water to the sample and mix thoroughly.	
3. When the sample is completely dissolved, slowly add 25 ml of concentrated hydrochloric acid.	3. Wear rubber gloves, rubber apron, and work in a well ventilated hood. If any of the solution contacts the skin, flush the affected area immediately with water.
4. Boil the solution on a hot plate for 30 minutes.	4. Work in a hood. Place a HOT sign in front of the hot plate and use beaker tongs when handling the hot beaker.
5. The presence of fat globules near the surface of the solution confirms the presence of fatty acids. Report fatty acids as present or absent.	

Code: 3D 20.1

Issued: December 20, 1955

DUPONOL, ME, DRY: SULFATES
Qualitative Method

References

F. P. Treadwell and W. T. Hall, "Analytical Chemistry," Vol. I, 9th Ed., p. 477, John Wiley and Sons, Inc., New York, 1949

Principle, Limitations, and Precision

A weighed sample is dissolved in distilled water and acidified with concentrated hydrochloric acid. The formation of a fine, white precipitate, barium sulfate, after the addition of barium chloride indicates the presence of the sulfate ion.

Equipment

1 Beaker, 250 ml
1 Graduated cylinder, 25 ml
1 Graduated cylinder, 100 ml

Reagents and Chemicals

Barium chloride, 10% by weight (RB 2.2)
Hydrochloric acid, CP, HCl, 37%

Safety Precautions

1. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3D 20.1

Issued: December 20, 1955

DUPONOL, ME, DRY: SULFATES
Qualitative Method

Procedure	Hazards Breakdown
1. Weigh, on a triple beam balance, approximately 1.0 g of the sample into a 250 ml beaker.	1. Inspect all glassware.
2. Add approximately 100 ml of distilled water to the sample and mix the solution thoroughly.	
3. Using a graduated cylinder, add 25 ml concentrated hydrochloric acid and boil the solution on a hot plate for 30 minutes.	3. Wear rubber apron, rubber gloves, and work in a well ventilated hood. Place a HOT sign in front of the hot plate. If any of the acid contacts the skin, flush the affected area immediately with water.
4. Remove the beaker from the hot plate. Using a graduated cylinder, add 25 ml of 10% barium chloride.	4. Use beaker tongs or wear asbestos gloves to handle the hot beaker.
5. A fine, white precipitate confirms the presence of sulfates. Report sulfates as present or absent. (Remark 1)	

Remarks

1. The solution should be allowed to stand for at least one hour for complete precipitation.

SRP Laboratory Manual
Essential Materials Methods

Code: 3F 25.0

Issued: January 11, 1956

FERROUS SULFAMATE: REDUCING NORMALITY
Volumetric Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. I, p. 471, D. Van Nostrand Company, Inc., New York, 1952

Principle, Limitations, and Precision

Reducing normality of ferrous sulfamate is determined by titrating the sample with standard potassium permanganate to the pink end point. In acidic solutions, ferrous ions are oxidized to the ferric state. The reaction is as follows:

$$5Fe^{++} + MnO_4^{-} \longrightarrow 5Fe^{+++} + Mn^{++}$$

Other ions which act as reducing agents will interfere.

The precision of the method is ± 3% in the 45-50% concentration range of ferrous sulfamate.

Equipment

1	Smith weighing buret, 10 ml
3	Erlenmeyer flasks, 500 ml
1	Transfer pipet, 10 ml
1	Buret, dispensing, 50 ml

Reagents and Chemicals

Sulfuric acid solution, 10% by volume	(RS 88.0)
Potassium permanganate solution, 0.1 N, standardized	(RP 83.1)
o-Phenanthroline ferrous sulfate, 0.025 N	(RP 29.0)

Safety Precautions

1. Sulfuric acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the sulfuric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3F 25.0

Issued: January 11, 1956

FERROUS SULFAMATE: REDUCING NORMALITY
Volumetric Method

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh 0.9 to 1.0 ± 0.0001 g of ferrous sulfamate from a Smith weighing buret into a clean, dry, 500 ml Erlenmeyer flask. Record the weight of the sample.	1. Inspect all glassware.
2. Add approximately 150 ml of distilled water.	
3. Add 10 ml of 10% sulfuric acid. Gently swirl the flask to mix the contents.	3. If any of the acid contacts the skin, flush the affected area immediately with water.
4. Add 3 drops of 0.025 N o-phenanthroline ferrous sulfate and mix thoroughly.	
5. Titrate the contents of the flask with 0.1 N standard potassium permanganate to the disappearance of the rose red color. Record, to the nearest 0.01 ml, the volume of potassium permanganate used.	
6. Run a reagent blank with 150 ml of distilled water and proceed with steps 3 through 5.	

Calculations

$$\text{Reducing normality, me/ml} = \frac{(A - B) \times C \times D}{E}$$

where A = volume of potassium permanganate used to titrate the sample, ml (step 5)
B = volume of potassium permanganate used to titrate the blank, ml (step 5)
C = normality of potassium permanganate
D = specific gravity of the sample (From Essential Materials Method 3F 25.1, Ferrous Sulfamate: Specific Gravity)
E = weight of the sample, g (step 1)

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3F 30.0

Issued: December 5, 1955

FLUOSILICIC ACID: TOTAL ACIDITY
Volumetric Method

References

F. P. Treadwell and W. T. Hall, "Analytical Chemistry," 9th Ed., Vol. II, p. 512, John Wiley and Sons, Inc., New York, 1942

Principle, Limitations, and Precision

The sample is titrated with standard sodium hydroxide using phenolphthalein indicator. Percent total acidity is expressed as fluosilicic acid.

When the titration is performed at room temperature, a false end point appears because of the formation of sodium silicofluoride. The sodium silicofluoride reacts slowly with excess sodium hydroxide. To speed up this reaction, the solution is heated to 80-90 C and the neutralization is completed while the solution is hot. No allowance is made for other acid impurities.

The precision of the method is unknown.

Equipment

1 Smith weighing buret, 5 ml
1 Buret, 50 ml
2 Beakers, plastic, 200 ml
2 Erlenmeyer flasks, 250 ml

Reagents and Chemicals

Sodium hydroxide, 0.1 N, against primary standard	(RS 60.6)
Phenolphthalein indicator, 1%	(RP 33.0)

Safety Precautions

1. The hazards of fluosilicic acid closely resemble the hazards of hydrefluoric acid. Fluosilicic acid can cause burns to the skin and mucous membranes of the body. Work in a hood and wear rubber gloves when filling the Smith weighing buret. Keep a slurry of magnesium oxide or calcium hydroxide available in the working area where fluosilicic acid is being used. If any fluosilicic acid contacts the skin, flush the affected area with generous amounts of water and apply the slurry of magnesium oxide or calcium hydroxide to the affected area. Report to your supervisor immediately.

[redacted]

FLUOSILICIC ACID: TOTAL ACIDITY
Volumetric Method

Safety Precautions (contd.)

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a Smith weighing buret, weigh, by difference, 0.30 to 0.35 ± 0.001 g of the sample into a clean, dry, 200 ml plastic beaker containing 50 ml of distilled water. Record the weight of the sample. (Remark 1)	1. Inspect all glassware. Work in a hood and wear rubber gloves when filling the weighing buret. If any fluosilicic acid contacts the skin, flush the affected area immediately with generous amounts of water and apply a slurry of magnesium oxide or calcium hydroxide.
2. Add three drops of 1% phenolphthalein indicator to the contents of the beaker.	
3. Titrate the solution with 0.1 N sodium hydroxide to the first pink color.	
4. Quantitatively transfer the contents of the beaker into a 250 ml Erlenmeyer flask and heat the solution to 80 C. (Remark 2) Continue the titration while the solution is hot to the first pink color that persists for 30 seconds. Record, to the nearest 0.01 ml, the total volume of sodium hydroxide used.	4. Use flask holders or wear asbestos gloves when removing the flask from the hot plate. Place a HOT sign in front of the hot plate when it is in operation.

[redacted]

Code: 3F 30.0

Issued: December 5, 1955

FLUOSILICIC ACID: TOTAL ACIDITY
Volumetric Method

Calculations

$$\% \text{ Total acidity as fluosilicic acid} = \frac{A \times B \times C \times 100}{D}$$

where A = volume of sodium hydroxide used, ml (step 4)
B = normality of sodium hydroxide
C = gram-milliequivalent weight of fluosilicic acid
$= \frac{144.08}{6 \times 1000} = 0.02401$
D = weight of sample, g (step 1)

Remarks

1. Concentrated solutions (21 - 32%) of fluosilicic acid should be handled in either plastic, paraffin lined glass, or platinum. Dilute solutions of fluosilicic acid may be handled in ordinary Pyrex glass for short periods of time.

2. If the pink color does not disappear when the sample is heated, the end point has been passed and the determination must be run again.

SRP Laboratory Manual
Essential Materials Methods

Code: 3G 12.0

Issued: December 14, 1955

GLUCONIC ACID: GLUCONIC ACID
Volumetric Method

References

Maria Marconi, "Titration of the Anion in Soluble Salts," Chemical Abstracts, Vol. 46, 4427, 1952

Principle, Limitations, and Precision

An excess of standard sodium hydroxide is added to a weighed sample of gluconic acid. The excess sodium hydroxide is titrated to pH 8.3 (the equivalence point) with standard hydrochloric acid and the result is calculated as percent gluconic acid.

The precision of the method is ± 0.2% in the concentration range 48-52% gluconic acid.

Equipment

1 pH Meter, Beckman Model H-2 (EM 25.2)
1 Transfer pipet, 50 ml
2 Beakers, Pyrex, 250 ml
1 Smith weighing buret, 10 ml
1 Magnetic stirrer
2 Magnetic stirring bars, Teflon enclosed
1 Buret, 50 ml

Reagents and Chemicals

Buffer solution, pH 7.0
Sodium hydroxide, 0.2 N, against primary standard (RS 60.3)
Hydrochloric acid, 0.1 N, against primary standard (RH 97.9)

Safety Precautions

1. Dilute hydrochloric acid and dilute sodium hydroxide may cause painful burns to the skin on prolonged or repeated contact. If any of the solution contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

GLUCONIC ACID: GLUCONIC ACID
Volumetric Method

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a pH 7.0 buffer solution, buffer the model H-2 pH meter by following Laboratory Equipment Method EM 25.2, pH Meter, Beckman Model H-2.	
2. Pipet 50 ml of 0.2 N sodium hydroxide into a clean, dry, 250 ml beaker.	2. Inspect all glassware. If any of the solution contacts the skin, flush the affected area immediately with water.
3. Weigh 3.0 ± 0.001 g of sample from a Smith weighing buret into the beaker. Mix the contents of the beaker thoroughly.	3. Same as step 2.
4. Allow the solution in the beaker to stand 10 minutes. Place a magnetic stirring bar in the beaker and immerse the electrodes of the pH meter in the sample solution.	
5. Using a buret, add 0.1 N hydrochloric acid to the sample until the pH of the solution is 8.3, as indicated by the pH meter. Stir the solution during the titration. (Remark 1) Record, to the nearest 0.01 ml, the volume of hydrochloric acid added.	5. Same as step 2.

Code: 3G 12.0

Issued: December 14, 1955

GLUCONIC ACID: GLUCONIC ACID
Volumetric Method

Calculations

$$\text{Percent gluconic acid} = \frac{[(A \times B) - (C \times D)] \times E \times 100}{F}$$

where A = volume of sodium hydroxide added in step 2, ml
B = normality of sodium hydroxide
C = volume of hydrochloric required for the titration, ml (step 5)
D = normality of the hydrochloric acid
E = gram-milliequivalent weight of gluconic acid
$= \frac{196.2}{1000} = 0.1962$
F = weight of sample, g (step 3)

Remarks

1. For details of operation of the pH meter, refer to Laboratory Equipment Method EM 25.2, pH Meter, Beckman Model H-2.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3H 20.0

Issued: December 1, 1955

HYDROXYLAMINE SULFATE: HYDROXYLAMINE SULFATE
Volumetric Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., p. 647, D. Van Nostrand Co., Inc., New York, 1939

Principle, Limitations, and Precision

Hydroxylamine in hot acid solutions reduces ferric salts to the ferrous state. The amount of ferrous iron, which is a measure of the hydroxylamine originally present, is determined by titration with standard potassium permanganate.

Serious errors result if the titration is begun before the oxidation of the hydroxylamine is complete, if insufficient acid is present, or if the excess of ferric ammonium sulfate is too small.

The precision of the method is ± 0.2% in the 96-100% range.

Equipment

4 Erlenmeyer flasks, 500 ml
1 Transfer pipet, 15 ml
1 Transfer pipet, 10 ml
1 Buret, 50 ml

Reagents and Chemicals

Ferric ammonium sulfate, saturated	(RF 22.0)
Sulfuric acid, 1:4	(RS 86.0)
o-Phenanthroline ferrous sulfate, 0.025 N	(RP 29.0)
Potassium permanganate, 0.1 N, standardized	(RP 83.1)

Safety Precautions

1. Hydroxylamine sulfate is a fire hazard and may cause burns or irritations on prolonged or repeated contact with the skin. If any hydroxylamine sulfate contacts the skin, flush the affected area immediately with water.

[redacted]

HYDROXYLAMINE SULFATE: HYDROXYLAMINE SULFATE
Volumetric Method

Safety Precautions (contd.)

2. Sulfuric acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the sulfuric acid contacts the skin, flush the affected area immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh 0.1000 to 0.1500 g of sample and transfer it to a clean, dry, 500 ml Erlenmeyer flask. Record the weight of the sample.	1. Inspect all glassware. Work in a well ventilated, flame-free area. If any of the hydroxylamine sulfate contacts the skin, flush the affected area immediately with water.
2. Dissolve the sample in approximately 50 ml of distilled water. Prepare a blank by adding 50 ml of distilled water to another clean, dry, 500 ml Erlenmeyer flask. Treat the sample and the blank in the same manner throughout the remainder of the procedure.	
3. Using a transfer pipet, add 30 ml of cold, saturated, ferric ammonium sulfate to the sample.	

[redacted]

HYDROXYLAMINE SULFATE: HYDROXYLAMINE SULFATE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
4. Using a transfer pipet, add 10 ml of 1:4 sulfuric acid.	4. If any of the acid contacts the skin, flush the affected area immediately with water.
5. Boil the sample for 5 minutes on a hot plate.	5. Work in a hood. Place a HOT sign in front of the hot plate.
6. Remove the flask from the hot plate and dilute the solution to approximately 300 ml with distilled water.	6. Use beaker tongs or asbestos gloves to handle the hot flask.
7. Add 5 drops of 0.025 N o-phenanthroline ferrous sulfate. Titrate the contents of the flask with 0.1 N potassium permangante until the disappearance of the rose-red color. Record, to the nearest 0.01 ml, the volume of potassium permanganate used.	

Calculations

$$\% \text{ Hydroxylamine sulfate} = \frac{(A - B) \times C \times D \times 100}{E}$$

where A = volume of potassium permanganate used to titrate the sample, ml (step 7)

B = volume of potassium permanganate used to titrate the blank, ml (step 7)

C = normality of the potassium permanganate

D = gram-milliequivalent weight of hydroxylamine sulfate $= \frac{164.14}{4 \times 1000} = 0.04104$

E = weight of the sample, g

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3H 20.1

Issued: November 16, 1955

HYDROXYLAMINE SULFATE: RESIDUE ON IGNITION
Gravimetric Method

References

"Reagent Chemicals, ACS Specifications," ACS, p. 178, Washington, D. C., 1950

Principle, Limitations, and Precision

A sample of hydroxylamine sulfate is ignited in a tared platinum dish at 900 C. The dish is brought to constant weight and the weight of the residue is obtained. The result is expressed as percent residue on ignition.

The precision of the method is unknown.

Equipment

2 Crucibles and covers, platinum

Safety Precautions

Before inserting and removing crucibles from the furnace, the temperature of the furnace must be below 900 C. Do not stand in front of the open furnace door. Wear asbestos gloves and use proper length platinum tipped tongs to manipulate platinum crucibles in the furnace.

Procedure	Hazards Breakdown
1. Dry two platinum crucibles in a muffle furnace at 900 C for two hours. Remove the crucibles from the furnace and allow them to cool in a desiccator for 30 minutes. Weigh the crucibles to the nearest 0.0001 g and record the weights.	1. Wear asbestos gloves and use platinum tipped tongs to place the crucibles in and remove the crucibles from the furnace. Allow the furnace to cool below 900 C to remove the crucible. Allow the crucibles to cool slightly before closing the desiccator completely. Use a Desiguard to transport the desiccator.
2. Weigh 2.0 ± 0.001 g of the sample into each platinum crucible. Record the weight of each sample.	

[redacted]

Code: 3H 20.1

Issued: November 16, 1955

HYDROXYLAMINE SULFATE: RESIDUE ON IGNITION
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
3. Cover each crucible and place them in a cold muffle furnace. Slowly raise the temperature of the furnace to 850-900 C.	
4. Maintain this furnace temperature for two hours. Remove the crucibles from the furnace and place them in a desiccator to cool for 30 minutes. Weigh the crucibles to the nearest 0.0001 g and record the weights.	4. Same as step 1.
5. Repeat step 4 until two consecutive weighings differ by no more than 0.0005 g.	

Calculations

$$\% \text{ Residue on ignition} = \frac{(A - B)}{C} \times 100$$

where A = weight of crucible and residue, g (step 4)
B = weight of empty crucible, g (step 1)
C = weight of sample, g (step 2)

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3L 30.0

Issued: November 25, 1955

LIME: CALCIUM HYDROXIDE
Gravimetric Method

References

Refer to Essential Materials Method 3C 2.0 Calcium Carbonate: Calcium Carbonate, Gravimetric Method

Principle, Limitations, and Precision

Refer to Essential Materials Method 3C 2.0, Calcium Carbonate: Calcium Carbonate, Gravimetric Method. Calcium oxalate is decomposed at red heat to calcium oxide; the calcium oxide is weighed and reported as percent calcium hydroxide.

Equipment

Refer to Essential Materials Method 3C 2.0, Calcium Carbonate: Calcium Carbonate, Gravimetric Method

Reagents and Chemicals

Refer to Essential Materials Method 3C 2.0, Calcium Carbonate: Calcium Carbonate, Gravimetric Method

Safety Precautions

1. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

2. Calcium oxide (lime) is very caustic and causes dermatitis. Lime dust is irritating to the eyes, mucous membranes, and upper respiratory tract.

3. When handling bromine water, work in a well ventilated, flame-free hood. The vapors of bromine are extremely irritating to the skin, eyes, and mucous membranes. If any of the bromine water contacts the skin, wash the affected area immediately with mild soap and water. Store bromine water in a secondary earthenware container in a well ventilated hood.

[redacted]

Code: 3L 30.0

Issued: November 25, 1955

LIME: CALCIUM HYDROXIDE
Gravimetric Method

Safety Precautions (contd.)

4. Concentrated ammonium hydroxide is very corrosive and may cause painful burns on contact with the skin. The fumes of ammonium hydroxide are irritating to the eyes and respiratory tract. When handling concentrated ammonium hydroxide in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the ammonium hydroxide contacts the skin, flush the affected area immediately with water.

5. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure

Follow Essential Materials Method 3C 2.0, Calcium Carbonate: Calcium Carbonate, Gravimetric Method

Calculations

$$\% \text{ Calcium Hydroxide} = \frac{[(A - B) - (C - D)] \times E \times 100}{F}$$

where A = weight of sample crucible plus residue, g (step 16)
B = weight of empty sample crucible, g (step 1)
C = weight of blank crucible plus residue, g (step 16)
D = weight of empty blank crucible, g (step 1)
E = factor for converting calcium oxide to calcium hydroxide = $\frac{74.096}{56.08} = 1.3213$
F = weight of sample, g (step 2)

SRP Laboratory Manual
Essential Materials Methods

Code: 3M 18.1

Issued: January 12, 1956

MANGANOUS NITRATE (AQUEOUS): MANGANESE
Volumetric Method

References

HW-25375, Essential Materials Analytical Manual, Mn-1, 1952

Principle, Limitations, and Precision

The divalent manganese content of the sample is determined by oxidizing the sample to the permanganate with sodium bismuthate. The excess sodium bismuthate is removed by filtration and an excess of ferrous ammonium sulfate is added to the filtrate. The excess ferrous ammonium sulfate is titrated with standard potassium permanganate and the result calculated as percent manganese.

The precision of the method is unknown.

Equipment

1 Smith weighing buret, 10 ml
2 Volumetric flasks, 250 ml
1 Graduated cylinder, 100 ml
2 Transfer pipets, 20 ml
3 Beakers, Pyrex, 400 ml
3 Crucibles, sintered-glass, fine porosity, 30 ml
3 Filtering flasks, Pyrex, 500 ml
1 Transfer pipet, 3 ml
1 Buret, 50 ml

Reagents and Chemicals

Sulfuric acid, 3 N	(RS 88.0)
Sodium bismuthate, CP, $NaBiO_3$	
Ferrous ammonium sulfate, 0.1 N	(RS 46.0)
Phosphoric acid, CP, H_3PO_4, 85%	
Potassium permanganate, 0.1 N, standardized	(RP 83.1)

Safety Precautions

1. Sulfuric acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the sulfuric acid contacts the skin, flush the affected area immediately with water.

MANGANOUS NITRATE (AQUEOUS): MANGANESE
Volumetric Method

Safety Precautions (contd.)

2. Concentrated phosphoric acid is very corrosive and may cause painful burns on contact with the skin. When handling concentrated phosphoric acid in containers larger than one liter, wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the phosphoric acid contacts the skin, flush the affected area immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a Smith weighing buret, weigh 1.2 ± 0.0001 g of the sample into a 250 ml volumetric flask containing 200 ml of distilled water. Mix the solution thoroughly, dilute to the calibration mark with distilled water, and mix again.	1. Inspect all glassware. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
2. Pipet a 20 ml aliquot of the diluted sample into a 400 ml beaker. Using a graduated cylinder, add 80 ml of 3 N sulfuric acid.	2. If any of the acid contacts the skin, flush the affected area immediately with water.
3. Prepare a reagent blank by adding 20 ml of distilled water and 80 ml of 3 N sulfuric acid to another 400 ml beaker. Treat the sample and the blank in the same manner throughout the remainder of the analysis.	3. Same as step 2.

MANGANOUS NITRATE (AQUEOUS): MANGANESE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
4. Add approximately 1 g of sodium bismuthate to the sample. Swirl the sample solution for approximately 1 1/2 minutes to mix thoroughly.	
5. Allow the suspended sodium bismuthate to settle. Filter the solution through a fine porosity, sintered-glass crucible into a 500 ml filter flask connected to the vacuum line through a trap. (Remark 1)	
6. Wash the crucible with 3 N sulfuric acid until the washings are colorless. Pipet 20 ml of 0.1 N ferrous ammonium sulfate and 3 ml of concentrated phosphoric acid to the filtrate. (Remark 2) Mix the solution thoroughly.	6. Same as step 2.
7. Titrate the filtrate with 0.1 N potassium permanganate to the faint pink end point which persists for 30 seconds. Record, to the nearest 0.01 ml, the volume of potassium permanganate used.	

Calculations

$$\text{Percent Manganese} = \frac{(A - B) \times C \times D \times E}{F \times G} \times 100$$

where A = volume of potassium permanganate used to titrate the reagent blank, ml

B = volume of potassium permanganate used to titrate the sample, ml

C = normality of the potassium permanganate

D = gram-milliequivalent weight of manganese $= \frac{54.93}{5 \times 1000} = 0.01099$

E = volume to which the original sample was diluted = 250 ml

F = weight of the sample, g

G = volume of the diluted sample taken for analysis = 20 ml

Code: 3M 18.1

Issued: January 12, 1956

MANGANOUS NITRATE (AQUEOUS): MANGANESE
Volumetric Method

Remarks

1. The crucible stem should extend to within 1 inch of the bottom of the filter flask to prevent spattering. The titration is performed in the filter flask.
2. Add the some amount of ferrous ammonium sulfate to the blank and the sample.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3M 20.2

Issued: November 1, 1955

METHANOL: RESIDUE ON EVAPORATION
Gravimetric Method

References

SRP Standard Procedure 3SP 9.0, Residue on Evaporation - Gravimetric Method

Principle, Limitations, and Precision

A representative sample is evaporated to dryness in a platinum dish. The residue consisting of non-volatile solids, is then dried to constant weight in an oven. The weight of residue obtained is expressed as a percentage of the original material.

The precision of the method is unknown.

Equipment

2 Evaporating dishes, platinum, 100 ml
1 Transfer pipet, 100 ml

Safety Precautions

Methanol (methyl alcohol) is flammable and constitutes a fire hazard. Never use it near open flames or electric heaters having exposed heating elements. The material is also very irritating to the eyes, skin, and respiratory tract, and is toxic when ingested.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a 100 ml platinum evaporating dish for one hour in an oven at 105 to 110 C.	1. When the platinum dish is hot, handle it with platinum tipped tongs.
2. Allow the dish to cool in a desiccator and weigh to the nearest 0.0001 g. Record the weight.	
3. Pipet 100 ml of sample into the dish.	3. Pipet the sample in a flame-free hood.

[redacted]

Code: 3M 20.2

Issued: November 1, 1955

METHANOL: RESIDUE ON EVAPORATION
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
4. Evaporate the sample to dryness on a steam bath in a well ventilated hood.	4. Handle the dish with tongs to prevent steam burns.
5. Heat the residue one hour in an oven at 105 to 110 C.	5. When the platinum dish is hot, handle it with platinum tipped tongs.
6. Allow the dish to cool in a desiccator and weigh to the nearest 0.0001 g. Record the weight.	

Calculations

$$\% \text{ Residue} = \frac{(A - B) \times 100}{C \times D}$$

where A = weight of dish plus residue, g (step 6)
B = weight of empty dish, g (step 2)
C = specific gravity of sample (see Essential Materials Method 3M 20.1, Methanol: Specific Gravity and Methanol Content - Gravimetric Method)
D = volume of sample used, ml (step 3)

if D = 100

$$\text{then } \% \text{ Residue} = \frac{A - B}{C}$$

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3M 20.3

Issued: November 8, 1955

METHANOL: ACIDITY
Volumetric Method

References

"Reagent Chemicals, ACS Specifications," p. 216, ACS, Washington, D. C., 1950

Principle, Limitations, and Precision

A known quantity of sample is titrated with 0.01 N sodium hydroxide using phenolphthalein indicator. The acidity is calculated as percent by weight of acetic acid.

The precision of the method is unknown.

Equipment

1 Graduated cylinder, 10 ml
1 Graduated cylinder, 50 ml
2 Erlenmeyer flasks, 250 ml
1 Transfer pipet, 25 ml
1 Buret, 10 ml

Reagents and Chemicals

Methyl alcohol, CP, CH_3OH	
Phenolphthalein indicator solution, 1%	(HP 33.0)
Sodium hydroxide, 0.01 N, standardized	(RS 60.7)

Safety Precautions

1. Methyl alcohol (methanol) is a flammable liquid and a dangerous fire hazard. Never use methyl alcohol near open flames or hot exposed heating elements. In addition, the material is extremely toxic when ingested. When handling methyl alcohol, work in a well ventilated area.

[redacted]

METHANOL: ACIDITY
Volumetric Method

Safety Precautions (contd.)

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
1. Measure 50 ml of CP methyl alcohol into a graduated cylinder and transfer the alcohol to a 250 ml Erlenmeyer flask containing 100 ml of carbon dioxide-free water.	1. Inspect all glassware. Measure the required amount of methyl alcohol in a flame-free hood.
2. Add 2 ml of 1% phenolphthalein indicator to the flask.	
3. Using a 10 ml buret, add 0.01 N sodium hydroxide dropwise until a permanent faint pink color is obtained.	3. Never fill a buret above eye level. Stand on safety steps, or lower the buret below eye level.
4. Divide the neutral solution into two equal portions by pouring half of the solution into a second Erlenmeyer flask.	
5. Pipet 25 ml of the sample into one of the two solutions.	
6. Using a 10 ml buret, titrate the sample solution with 0.01 N sodium hydroxide to the color of the solution in the second flask. Record the volume of sodium hydroxide used to the nearest 0.01 ml.	6. Same as step 3.
7. Pipet 25 ml of the sample into the second flask.	

[redacted]

Code: 3M 20.3

Issued: November 8, 1955

METHANOL: ACIDITY
Volumetric Method

Procedure (contd.)	Hazards Breakdown
8. Titrate the solution in the second flask with 0.01 N sodium hydroxide to the color of the solution in the first flask. Record the volume of the sodium hydroxide used to the nearest 0.01 ml.	8. Same as step 3.

Calculations

$$\% \text{ Acidity (as acetic acid)} = \frac{A \times B \times C \times 100}{D \times E}$$

where A = volume of 0.01 N sodium hydroxide required to titrate a 25 ml sample, ml

B = normality of sodium hydroxide

C = gram-milliequivalent weight of acetic acid = $\frac{60.05}{1000}$ = 0.06005

D = volume of sample used, ml

E = density of the sample (Remark 1)

Remarks

1. The density of pure methanol is 0.79 g/ml. This value will be sufficiently accurate for the determination.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3M 20.4

Issued: October 31, 1955

METHANOL: DISTILLATION RANGE

References

ASTM Standards, Part 5, D 86-46, D 216-40, D 1078-49T, Philadelphia, Pa., 1949

Principle, Limitations, and Precision

Refer to SRP Standard Procedure 3SP 3.0, Distillation Range

Equipment

1 Engler flask, 100 ml
1 Thermometer, ASTM Solvents Distillation, E 1 (39C-44T)
Range: 48 to 102 C
Distillation range apparatus (ED 44.0)

Safety Precautions

1. Do not use external flames or hot exposed heating elements near the receiver of the distillation apparatus. Work in a well ventilated area.

2. Methanol is a flammable liquid and should be kept away from flames and exposed heating elements. It is toxic by inhalation, ingestion, or by prolonged contact with the skin. Wear rubber gloves and flood any contacted areas immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure

Follow Standard Procedure 3SP 3.0, Distillation Range. Record only the initial boiling point (IBP) and the dry point temperatures corrected to standard barometric pressure.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3N 35.1

Issued: November 18, 1955

NITRIC ACID: CHLORIDES
Turbidimetric Method

References

SRP Standard Procedure 3SP 2.0, Chlorides: Turbidimetric Method

"Reagent Chemicals, ACS Specifications," Washington, D. C., 1950

Principle, Limitations, and Precision

The sample is acidified with dilute nitric acid and the chloride content is determined semi-quantitatively by adding an excess of silver nitrate. The turbidity of the sample solution is compared with that of a control solution containing a known amount of chloride. Thiocyanates, cyanides, and other halogens interfere.

Equipment

2 Nessler tubes, matched, 50 ml
1 Smith weighing buret, 10 ml
2 Transfer pipets, 1 ml
1 Transfer pipet, 5 ml

Reagents and Chemicals

Nitric acid, CP, HNO_3, 70%	
Chloride standard solution, 0.1 mg Cl^-/ml	(RC 35.0)
Silver nitrate, 0.1 N	(RS 41.2)

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

2. Silver nitrate solution is caustic. It is poisonous and discolors the skin on contact. If any of the silver nitrate contacts the skin, flush the affected area immediately with water.

[redacted]

Code: 3N 35.1

Issued: November 18, 1955

NITRIC ACID: CHLORIDES
Turbidimetric Method

Safety Precautions (contd.)

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh, from a Smith weighing buret, 10.0 to 10.2 ± 0.0001 g of sample into a 50 ml Nessler tube containing about 30 ml of distilled water. The sample weight is equivalent to approximately 7.3 ml of 60% acid.	1. Inspect all glassware. Wear rubber gloves and work in a hood while filling the Smith weighing buret. Clean up any spills immediately with generous amounts of water.
2. To a second Nessler tube, add about 30 ml of distilled water, 1 ml of concentrated nitric acid, and 1 ml of the standard chloride solution, 0.1 mg Cl^-/ml.	2. Add concentrated nitric acid to water slowly to prevent spattering.
3. Add 5 ml of 0.1 N silver nitrate to each Nessler tube. Dilute the solutions to the calibration mark with distilled water and mix thoroughly.	3. If any of the silver nitrate solution contacts the skin, flush the affected area immediately with water. When inverting the Nessler tubes, place the index and middle fingers of one hand on the stopper allowing the ring and little fingers and thumb to grasp the tube.
4. Compare the turbidity of the sample solution with that of the standard chloride solution. Report the chloride content as less than, equal to, or greater than 0.001%.	

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3N 35.3

Issued: November 1, 1955

NITRIC ACID: RESIDUE ON EVAPORATION
Gravimetric Method

References

Rosin, "Reagent Chemicals and Standards," D. Van Nostrand Co., Inc., New York, 1939

Principle, Limitations, and Precision

A representative sample is evaporated to dryness in a platinum dish. The residue consisting of non-volatile solids is then dried to constant weight in an oven. The weight of residue obtained is expressed as a percentage of the original material.

The precision of the method is unknown.

Equipment

1	Weighing buret, Smith, 30 ml
2	Evaporating dishes, platinum, 50 ml

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a 50 ml platinum evaporating dish for one hour in an oven at 105 to 110 C.	1. When the platinum dish is hot, handle it with platinum tipped tongs.

[redacted]

Code: 3N 35.3

Issued: November 1, 1955

NITRIC ACID: RESIDUE ON EVAPORATION
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Allow the dish to cool in a desiccator and weigh to the nearest 0.0001 g. Record the weight.	2. Allow the dish to cool slightly before closing the desiccator completely. Use a Desiguard to transport the desiccator.
3. Weigh between 13.7 and 13.8 ± 0.01 g of sample, from a Smith weighing buret, into the dish. The sample weight is equivalent to 10.0-10.1 ml of 60% acid. Record the weight.	3. Inspect all glassware. Wear rubber gloves, acid type goggles, and a rubber apron and work in a hood. If any of the acid contacts the skin, flush the affected area immediately with water.
4. Evaporate the sample to dryness on a steam bath in a well ventilated hood.	4. Cover the dish with a watch glass while transferring it from the balance to the steam bath.
5. Remove the watch glass and heat the residue one hour in an oven at 105 to 110 C.	5. Handle the dish with tongs to prevent steam burns.
6. Allow the dish to cool in a desiccator and weigh to the nearest 0.0001 g. Record the weight.	6. When the platinum dish is hot, handle it with platinum tipped tongs. Allow the dish to cool slightly before closing the desiccator completely.

Calculations

$$\% \text{ Residue} = \frac{(A - B) \times 100}{C}$$

where A = weight of dish plus residue, g (step 6)
B = weight of empty dish, g (step 2)
C = weight of sample, g (step 3)

SRP Laboratory Manual
Essential Materials Methods

Code: 30 50.0

Issued: January 10, 1956

OXALIC ACID: OXALIC ACID
Volumetric Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis", 5th Ed., Vol. II, p. 2253, D. Van Nostrand Co., Inc., New York, 1952

Principle, Limitations, and Precision

A weighed portion of the sample is dissolved in carbon dioxide free water and then titrated with standard potassium permanganate to a faint, pink, end point. Oxalic acid acts as a reducing agent for strong oxidizing agents. The reaction is stoichiometric and can be used for the quantitative determination of oxalic acid. The presence of other reducing agents interferes.

The precision of the method is unknown.

Equipment

2	Volumetric flasks, 500 ml
1	Graduated cylinder, 100 ml
1	Graduated cylinder, 25 ml
2	Erlenmeyer flasks, 250 ml
1	Buret, calibrated, 50 ml
1	Thermometer, 0 to 100 C
1	Magnetic stirrer
2	Magnetic stirring bars, Teflon enclosed
1	Pipet, 50 ml

Reagents and Chemicals

Sulfuric acid, H_2SO_4, CP, 96%	
Potassium permanganate, 0.1 N, standardized	(RP 83.1)

Safety Precautions

1. Oxalic acid is irritating to the mucous membranes and has a strong, local, caustic action on the skin. If any of the oxalic acid contacts the skin, flush the affected area immediately with water.

OXALIC ACID: OXALIC ACID
Volumetric Method

Safety Precautions (contd.)

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh 2.4 to 2.6 ± 0.0001 g of sample into a 500 ml volumetric flask containing 100 ml of distilled water.	1. Inspect all glassware. If any of the oxalic acid contacts the skin, flush the affected area immediately with water.
2. Swirl the flask to dissolve the sample. Dilute to the calibration mark with distilled water. Invert the flask to mix the contents of the flask.	2. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
3. Pipet a 50 ml aliquot of sample into a 250 ml Erlenmeyer flask.	
4. Add 3 ml of concentrated sulfuric acid and 25 ml of distilled water to the flask.	4. If any of the acid contacts the skin, flush the affected area immediately with water.
5. Add, from a buret, 35 ml of standardized 0.1 N potassium permanganate. (Remark 1)	
6. Heat the solution on a hot plate to 90 C as indicated by a thermometer. Complete the titration while the solution is hot. The end point is a pink coloration which persists for about one minute. (Remark 2) Record, to the nearest 0.01 ml, the total volume of potassium permanganate added in steps 5 and 6.	6. Place a HOT sign in front of the hot plate. Use flask holders when removing the hot flask from the hot plate.

OXALIC ACID: OXALIC ACID
Volumetric Method

Calculations

$$\text{Percent oxalic acid} = \frac{A \times B \times C \times D \times 100}{E \times F}$$

where A = total volume of potassium permanganate used in the titration, ml (step 6)

B = normality of potassium permanganate

C = gram-milliequivalent weight of oxalic acid

$= \frac{126.07}{2 \times 1000} = 0.06303$

D = volume to which the original sample was diluted = 500 ml

E = weight of the sample, g (step 1)

F = volume of diluted sample taken for analysis = 50 ml

Remarks

1. A purple color will appear after the addition of 35 ml of potassium permanganate, but upon shaking and heating this color will fade and the titration can be completed.
2. The end point can be more easily detected by stirring the solution with a magnetic stirrer during the titration.

[redacted]

Code: 30 50.1

Issued: November 23, 1955

OXALIC ACID: WATER INSOLUBLES AT 25 C
Gravimetric Method

Procedure (contd.)

Hazards Breakdown

7. Repeat step 6 until two consecutive weighings differ not more than 0.0005 g.

Calculations

$$\% \text{ Water insolubles at 25 C} = \frac{(A - B) \times 100}{C}$$

where A = weight of crucible and residue, g (step 6)
B = weight of empty crucible, g (step 1)
C = weight of sample, g (step 2)

SRP Laboratory Manual
Essential Materials Methods

Code: 3P 32.1

Issued: December 5, 1955

PHOSPHORIC ACID: CHLORIDES
Turbidimetric Method

References

"Reagent Chemicals, ACS Specifications," Washington, D. C., 1950

Principle, Limitations, and Precision

The sample is acidified with dilute nitric acid and the chloride is determined semi-quantitatively by adding an excess of silver nitrate. The turbidity of the sample solution is compared with the turbidity of a control solution containing a known amount of chloride. Thiocyanates, cyanides, and other halogens interfere.

The range of the method is 0.001 to 0.1% chloride.

Equipment

1	Smith weighing buret, 10 ml
3	Nessler tubes, matched, 50 ml
1	Transfer pipet, 1 ml
1	Transfer pipet, 2 ml
1	Transfer pipet, 5 ml

Reagents and Chemicals

Nitric acid, CP, HNO_3, 70%	
Chloride standard solution, 0.1 mg Cl/ml	(RC 35.0)
Silver nitrate, 0.1 N	(RS 41.2)

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

PHOSPHORIC ACID: CHLORIDES
Turbidimetric Method

Safety Precautions (contd.)

2. Concentrated phosphoric acid is very corrosive and may cause painful burns on contact with the skin. When handling concentrated phosphoric acid in containers larger than one liter, wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the phosphoric acid contacts the skin, flush the affected area immediately with water.

3. Silver nitrate solution is caustic. It is poisonous when ingested and discolors the skin on contact.

4. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a Smith weighing buret, weigh 2.0 to 2.1 ± 0.01 g of sample into a 50 ml Nessler tube containing about 30 ml of distilled water.	1. Inspect all glassware. Wear rubber gloves and work in a well ventilated area to fill the weighing buret.
2. Add 1 ml of concentrated nitric acid to the Nessler tube.	
3. To a second Nessler tube, add about 30 ml of distilled water, 1 ml of concentrated nitric acid, and 2 ml of chloride standard solution, 0.1 mg Cl/ml.	

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 30 50.1

Issued: November 23, 1955

OXALIC ACID: WATER INSOLUBLES AT 25 C
Gravimetric Method

References

SRP Standard Procedure 3SP 13.0, Water Insolubles, Gravimetric Method

HW-12866, Hanford Works Laboratory Manual, Essential Materials Method ESI-1a

Principle, Limitations, and Precision

A weighed quantity of the sample is dissolved in a measured volume of 25 C distilled water. The residue remaining after filtration is a measure of water insoluble material in the sample at 25 C.

The precision of the method is unknown.

Equipment

2 Crucibles, sintered-glass, fine porosity, 30 ml
2 Beakers, Pyrex, 250 ml
1 Graduated cylinder, 100 ml
1 Filter adapter
2 Filtering flasks, 500 ml

Safety Precautions

1. Oxalic acid is irritating to the skin and mucous membranes. If any of the acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

[redacted]

Code: 30 50.1

Issued: November 23, 1955

OXALIC ACID: WATER INSOLUBLES AT 25 C
Gravimetric Method

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a clean, fine porosity, sintered-glass crucible for one hour in an oven at 105-110 C. Allow the crucible to cool in a desiccator for 30 minutes. Weigh the crucible to the nearest 0.0001 g and record the weight.	1. Inspect all glassware. Use tongs when placing the crucible in and removing the crucible from the oven. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport the desiccator.
2. Weigh 10 ± 0.001 g of sample into a clean, dry, 250 ml beaker. Record the weight.	2. If any of the acid contacts the skin, flush the affected area immediately with water.
3. Add 100 ml of 25 C distilled water. Stir the solution vigorously to dissolve the sample.	
4. Using vacuum, filter the solution through the tared, sintered-glass crucible into a 500 ml filtering flask connected to a trap.	
5. Wash the beaker with six 10 ml portions of 25 C distilled water. Filter the washings into the filtering flask.	
6. Dry the crucible and contents for one hour in an oven at 105 to 110 C. Allow the crucible to cool 30 minutes in a desiccator. Weigh the crucible to the nearest 0.0001 g and record the weight.	6. Use tongs to place the crucibles in and remove crucibles from the oven. Allow the crucible to cool slightly before closing the desiccator completely. Use a Desiguard to transport the desiccator.

PHOSPHORIC ACID: CHLORIDES
Turbidimetric Method

Procedure (contd.)

4. Add 5 ml of 0.1 N silver nitrate to each Nessler tube. Dilute the solutions to the calibration mark with distilled water and mix thoroughly.

4. If any of the silver nitrate contacts the skin, flush the affected area immediately with water. When inverting the tubes, place the index and middle fingers on the stopper, allowing the ring and little fingers and thumb to grasp the tube. Never attempt to invert more than one tube at a time.

5. Compare the turbidity of the sample solution with the turbidity of the chloride standard solution. Report the chloride content of the sample as less than, equal to, or greather than 0.01%.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3R 50.1

Issued: September 13, 1955

ION EXCHANGE RESINS: EXCHANGE CAPACITY
Volumetric Method

References

Memorandum to D. G. Koennack from C. J. Banick, ECA-Resins-4, August 2, 1954, Essential Materials File

Principles, Limitations, and Precision

Cation resins react with a sodium sulfate solution to form sodium resin salts and sulfuric acid; anion resins react with a similar sodium sulfate solution to form resin sulfate salts and sodium hydroxide.

$$2 \text{ Resin H} + Na_2SO_4 \rightleftharpoons 2 \text{ Resin Na} + H_2SO_4$$

$$2 \text{ Resin OH} + Na_2SO_4 \rightleftharpoons \text{Resin}_2SO_4 + NaOH$$

A change in pH results from such a metathetical reaction and this change is a measure of the exchange capacity of the resin under study. Titration with standard acid or base of the filtrate from a resin exhausted in this manner and weighing the dried resin salt gives the information necessary for expressing the exchange capacity as milliequivalents of salt per gram of dry resin.

The precision of the method is unknown.

Equipment

2 Resin weighing bottles, complete with stand and cap (Figure I)
1 Buret, 50 ml
2 Volumetric flasks, 1000 ml
2 Transfer pipets, 100 ml
3 Erlenmeyer flasks, 250 ml
1 Graduated cylinder, 100 ml

Reagents and Chemicals

Sodium sulfate solution, 4% by weight	(RS 66.2)
Phenolphthalein indicator, 1% by weight	(RP 33.0)
Sulfuric acid, 0.1 N, standardized	(RS 88.18)
Methyl orange indicator, 0.1% by weight	(RM 35.0)
Sodium hydroxide, 0.1 N, standardized	(RS 60.6)

[redacted]

Code: 3R 50.1

Issued: September 13, 1955

ION EXCHANGE RESINS: EXCHANGE CAPACITY
Volumetric Method

Safety Precautions

1. Dilute sodium hydroxide or dilute sulfuric acid may cause painful burns on prolonged or repeated contact. If any of the solution contacts the skin, wash it off immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a resin weighing bottle in an oven at 125 C for 30 minutes. Remove the weighing bottle from the oven and place it in a desiccator to cool for 1 hour.	1. Inspect all glassware. Wear asbestos gloves when handling the hot weighing bottle. Allow the weighing bottle to cool slightly before closing the desiccator.
2. Weigh the weighing bottle to the nearest 0.0001 g and record the weight.	
3. Weigh 8 ± 1 g of sample and transfer it to the tared weighing bottle.	
4. Position the resin weighing bottle in such a manner that the filtrate from the resin will drain into a 1000 ml volumetric flask. Pour 800 ml of 4% sodium sulfate solution through the resin. Keep the resin weighing bottle filled with solution.	

[redacted]

Code: 3R 50.1

Issued: September 13, 1955

ION EXCHANGE RESINS: EXCHANGE CAPACITY
Volumetric Method

Procedure (contd.)	Hazards Breakdown
5. Wash the resin with distilled water until the volumetric flask is filled to the calibration mark.	
6. Using distilled water, wash the exterior of the resin weighing bottle free of any sodium sulfate. Place the resin weighing bottle in an oven at 125 C for 4 hours.	
7. Remove the weighing bottle from the oven and place it in a desiccator to cool for 1 hour.	7. Wear asbestos gloves when handling the hot weighing bottle.
8. Weigh the weighing bottle to the nearest 0.0001 g and record the weight.	
9. Mix the contents of the 1000 ml flask and pipet 100 ml of filtrate into a 250 ml Erlenmeyer flask.	9. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
10. Add 80 ml of 4% sodium sulfate solution and 20 ml of distilled water to a 250 ml Erlenmeyer flask for the reagent blank.	
11. a. For cation resins: Add two drops of 1% phenolphthalein indicator to each flask and titrate the sample and the reagent blank with 0.1 N sodium hydroxide to the first pink color which persists for 30 seconds. Record, to the nearest 0.01 ml, the volume of sodium hydroxide used for each titration.	

[redacted]

Code: 3R 50.1

Issued: September 13, 1955

ION EXCHANGE RESINS: EXCHANGE CAPACITY
Volumetric Method

Procedure (contd.)

Hazards Breakdown

11. b. For anion resins:

Add two drops of 1% methyl orange indicator to each flask and titrate the sample and the reagent blank with 0.1 N sulfuric acid to the definite color change from yellow to orange. Record, to the nearest 0.01 ml, the volume of sulfuric acid used.

Calculations

$$\text{Milliequivalents per gram of dry resin} = \frac{(A - B) \times C \times 10}{(D - E) \times F}$$

where A = volume of reagent required to titrate the sample aliquot, ml (step 11)
B = volume of reagent required to titrate the reagent blank, ml (step 11)
C = normality of reagent used to titrate the sample aliquot and the reagent blank
D = weight of weighing bottle plus sample, g (step 8)
E = initial weight of weighing bottle, g (step 2)
F = resin salt conversion factor for cation resins = 0.89 or resin salt conversion factor for anion resins = 0.84 (Remark 1)

Remarks

1. The conversion factors are developed in the memorandum to D. G. Ebenhack from C. J. Banick, ECA-Resins-4, August 2, 1954, Essential Materials File. The factors apply only to the cation IRA-120 resin and the anion IRA-400 resin. If other type resins are analyzed, a new set of factors should be calculated.

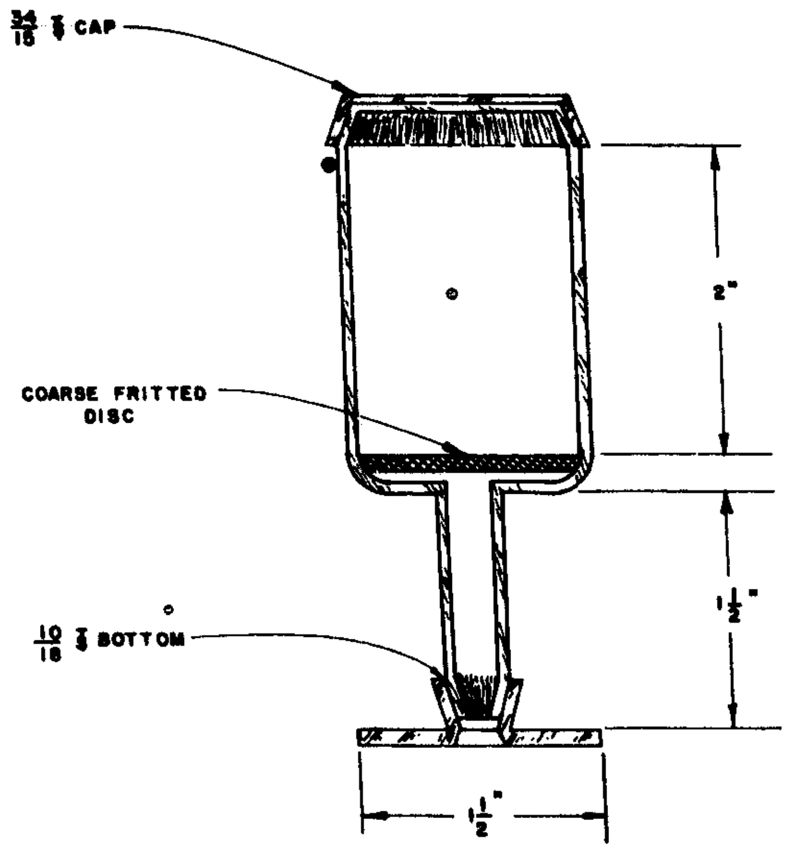

FIGURE 1

RESIN WEIGHING BOTTLE

SRP Laboratory Manual
Essential Materials Methods

Code: 3R 50.2

Issued December 21, 1955

ION EXCHANGE RESINS: DENSITY
Gravimetric Method

References

N. A. Lange, "Handbook of Chemistry," 8th Ed., p. 1767, Handbook Publishers, Inc., Sandusky, Ohio, 1952

Principle, Limitations, and Precision

A measured volume of resin is weighed and the density is expressed as grams per milliliter.

The precision of the method is unknown.

Equipment

1 Graduated cylinder, 100 ml

Reagents and Chemicals

None

Safety Precautions

Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
1. Weigh a clean, dry, 100 ml graduated cylinder to the nearest 0.01 g. Record the weight.	1. Inspect all glassware.
2. Fill the graduated cylinder to the 100 ml mark with resin. Do not pack the resin. Weigh the cylinder and resin to the nearest 0.01 g. Record the weight.	2. Clean up any spilled resins to remove the hazard of slipping.

Code: 3R 50.2

Issued: December 21, 1955

ION EXCHANGE RESINS: DENSITY
Gravimetric Method

Calculations

$$\text{Density} = \frac{A - B}{C}$$

where A = weight of graduated cylinder and resins, g
B = weight of empty graduated cylinder, g
C = volume of resin weighed = 100 ml

Code: 3R 50.3

Issued: December 21, 1955

ION EXCHANGE RESINS: MOISTURE CONTENT
Gravimetric Method

References

Memorandum to D. G. Ebenhack from C. J. Banick, ECA-Resins-4, August 2, 1954, Essential Materials File

Principle, Limitations, and Precision

A weighed sample of resin is dried in an oven at 125 C for four hours, cooled, and weighed again. From these data the percent moisture of the resin is calculated on a wet basis.

The resin should not be dried over four hours due to the possibility that the resin may be decomposed.

The precision of the method is unknown.

Equipment

2 Erlenmeyer flasks, glass-stoppered, 125 ml

Reagents and Chemicals

None

Safety Precautions

Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Dry a clean, 125 ml, Erlenmeyer flask in an oven at 125 C for 30 minutes. Do not stopper the flask during the drying period. Place the flask in a desiccator to cool to room temperature. Weigh the flask to the nearest 0.0001 g and record the weight.	1. Inspect all glassware. Wear asbestos gloves when handling hot glassware. Allow the flask to cool slightly before closing the desiccator completely. Transport the desiccator in a Desiguard.

ION EXCHANGE RESINS: MOISTURE CONTENT
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Place approximately 8 g of resin in the tared flask. Weigh and record, to the nearest 0.0001 g, the weight of the flask and moist resin.	
3. Dry the resin in an oven at 125 C for 4 hours. Do not stopper the flask during the drying period. Place the flask in a desiccator to cool to room temperature. Weigh and record, to the nearest 0.0001 g, the weight of the flask and dry resin.	3. Same as step 1.

Calculations

$$\text{Percent moisture} = \frac{(A - C)}{(A - B)} \times 100$$

where A = weight of the flask and moist resin, g (step 2)
B = weight of the empty flask, g (step 1)
C = weight of the flask and dry resin, g (step 3)

Code. 3R 50.4

Issued: December 21, 1955

ION EXCHANGE RESINS: REGENERATION
Method for Anion Resins

References

Laboratory Manual of the Permutit Co., 330 West 42nd St., New York, SRP Essential Materials File

Principle, Limitations, and Precision

In the determination of the exchange capacity of used ion exchange resins, it is necessary to first regenerate the resin. For anion resins, this is done by leaving the resin in contact with a 5% solution of sodium hydroxide; after a period of 1 1/2 hours, hydroxyl ions should have completely replaced all other anions in the resin. The resin is then filtered and washed free of excess base.

Equipment

4 Beakers, 600 ml
1 Graduated cylinder, 100 ml
2 Stirring bars, Teflon enclosed
2 Magnetic stirrers
2 Funnels, filtering, 75 mm diam
Filter paper, Whatman No. 41
Hydrion paper

Reagents and Chemicals

Sodium hydroxide, 5% by weight (RS 60.0)

Safety Precautions

1. Dilute sodium hydroxide may cause painful burns on prolonged or repeated contact. If any of the sodium hydroxide contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Code: 3R 50.4

Issued: December 21, 1955

ION EXCHANGE RESINS: REGENERATION
Method for Anion Resins

Procedure	Hazards Breakdown
Perform all regenerations in duplicate.	
1. Place approximately 10 g of resins in a clean, dry, 600 ml beaker. Using a graduated cylinder, add 200 ml of 5% sodium hydroxide.	1. Inspect all glassware. If any of the sodium hydroxide contacts the skin, flush the affected area immediately with water.
2. Place a stirring bar in the beaker and place the beaker on a magnetic stirrer. Gently stir the mixture for 1 1/2 hours.	
3. Filter the solution through Whatman No. 41 filter paper into a 600 ml beaker. Wash the resin with distilled water until the washings give a pH 6 reaction indicated by Hydrion paper.	3. Same as step 1.
4. Reserve the regenerated resins for exchange capacity determinations. (Essential Materials Method 3R 50.1, Ion Exchange Resins: Exchange Capacity)	

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Essential Materials Methods

Code: ES 40.0

Issued: January 10, 1956

SILICA GEL: DETERMINATION OF FINENESS
Sieve Test

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. II, p. 1314-[illegible]5-16, D. Van Nostrand Co., Inc., New York, 1952

Principle, Limitations, and Precision

A weighed sample is placed in the top of a nest of U. S. sieves equipped with a cover and a bottom pan. The sieves are placed in a Ro-Tap Sieve Shaker and shaken for a specified time. The residue on each screen is weighed and the percentage of sample passing that screen is calculated.

The precision of the method is unknown.

Equipment

1 Ro-Tap shaker (ES 34.0)
1 U. S. standard sieve, 8" diam., No. 14 mesh
1 U. S. standard sieve, 8" diam., No. 50 mesh
1 Cover for sieves, 8" diam
1 Receiver for sieves, 8" diam

Safety Precautions

Wear leather gloves when placing the sieves in position and when operating the Ro-Tap shaker. Do not wear dangling jewelry or loose clothing when working near the machine. Stand clear of the machine during the starting and operating periods. Turn off the shaker before attempting to uncover the nest of sieves.

Procedure	Hazards Breakdown
1. Weigh a clean, dry, undamaged, 8" U. S. Standard No. 14 mesh sieve and a No. 50 mesh sieve to the nearest 0.1 g. Record the weight of each sieve. (Remark 1)	

SILICA GEL: DETERMINATION OF FINENESS

Sieve Test

Procedure (contd.)	Hazards Breakdown
2. Nest the weighed sieves and receiver so that the No. 14 mesh sieve nests into the No. 50 mesh sieve.	
3. Weigh 50 ± 0.1 g of sample into the No. 14 mesh sieve. Record the weight.	
4. Attach a sieve cover to the No. 14 mesh sieve and place the nest of sieves, cover, and receiver in a Ro-Tap shaker. (Remark 2)	4. Wear leather gloves when placing the sieves in position. Position sieves, receiver, and cover in a secure manner in the Ro-Tap shaker. Raise or lower base plate with the adjustable bolts on each side so that a snug fit is obtained. Adjust the cover retainer and spring clip on the base plate to insure security of the nest.
5. Shake the sieves for ten minutes.	5. Wear leather gloves when operating the Ro-Tap shaker. Stand clear of the Ro-Tap shaker when starting it and while machine is in operation. Do not wear loose clothing around the machine.
6. Uncover the sieves and disengage the No. 14 mesh sieve.	6. Turn off the Ro-Tap shaker before attempting to uncover the nest of sieves.
7. Using a soft camel's hair brush, brush the particles adhering to the under surfaces of the sieve into the No. 50 mesh sieve.	
8. Weigh the No. 14 mesh sieve and contents to the nearest 0.1 g.	
9. Repeat steps 6, 7, and 8 for the No. 50 mesh.	

SILICA GEL: DETERMINATION OF FINENESS

Sieve Test

Procedure (contd.)	Hazards Breakdown
10. Repeat step 2, cover the No. 14 mesh sieve, place the nest of sieves, cover, and receiver into the shaker and shake the sieves for 5 minutes.	10. Same as steps 4 and 5.
11. Repeat steps 6, 7, 8. and 9	11. Turn off the Ro-Tap shaker before attempting to uncover the nest of sieves.
12. If the weight of either sieve changes more than 0.1 g after the 5 minute shaking period, continue to shake the sieves for 5 minute intervals until the weight of each sieve does not change more than 0.1 g.	

Calculations

I. Percent of the sample passing the No. 14 sieve = 100 - A

II. Percent of the sample passing the No. 50 sieve = 100 - (A + B)

$$A = \frac{(C - D) \times 100}{E}$$

$$B = \frac{(F - G) \times 100}{E}$$

where A = percent of residue on No. 14 mesh sieve

B = percent of residue on No. 50 mesh sieve

C = weight of residue and No. 14 mesh sieve after shaking, g (step 8)

D = weight of empty No. 14 mesh sieve, g (step 1)

E = original weight of sample, g (step 3)

F = weight of residue and No. 50 mesh sieve after shaking, g (step 8)

G = weight of empty No. 50 mesh sieve, g (step 1)

Remarks

1. Examine the screen of new and used sieves for minor defects and small tears. Do not use defective screens.
2. For details of operation of the shaker, refer to Laboratory Equipment method ES 34.0, Ro-Tap Shaker.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3S 52.0

Issued: November 4, 1955

SODIUM HYDROXIDE (AQUEOUS): SODIUM HYDROXIDE
Volumetric Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. I, p. 804, D. Van Nostrand Co., Inc., New York, 1939

Principle, Limitations, and Precision

A weighed quantity of sample is titrated with standard hydrochloric acid, using phenolphthalein as an indicator. Barium chloride is added to the sample before the titration to precipitate the carbonates. Other basic impurities cause erroneous results but these are not expected to be present in significant amounts.

The precision of the method is unknown.

Equipment

1 Smith weighing buret, 10 ml
2 Erlenmeyer flasks, 250 ml
1 Graduated cylinder, 25 ml
1 Buret, calibrated, 50 ml

Reagents and Chemicals

Barium chloride, 10% by weight	(RB 2.2)
Hydrochloric acid, 0.1 N, against primary standard	(RH 97.9)
Phenolphthalein indicator, 1%	(RP 33.0)

Safety Precautions

1. Concentrated sodium hydroxide solution is very corrosive and may cause painful burns on contact with the skin. When handling concentrated sodium hydroxide solution in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the sodium hydroxide solution contacts the skin, flush the affected area immediately with water.

[redacted]

Code: 3S 52.0

Issued: November 4, 1955

SODIUM HYDROXIDE (AQUEOUS): SODIUM HYDROXIDE
Volumetric Method

Safety Precautions (contd.)

2. Concentrated hydrochloric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of hydrochloric acid are irritating to the eyes and respiratory tract. When handling concentrated hydrochloric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Measure approximately 100 ml of distilled water into a 250 ml Erlenmeyer flask.	1. Inspect all glassware.
2. Weigh, from a Smith weighing buret, between 0.3 and 0.4 ± 0.0001 g of sample into the flask. The sample weight is equivalent to 0.20 to 0.26 ml of the liquid sample.	2. Wear rubber gloves, rubber apron, and acid type goggles when filling the Smith weighing buret. Clean up any spills immediately.
3. Add 5 ml of 10% barium chloride and 3 drops of 1% phenolphthalein indicator to the flask. Swirl the flask until the solution is thoroughly mixed.	

[redacted]

SODIUM HYDROXIDE (AQUEOUS): SODIUM HYDROXIDE
Volumetric Method

Procedure (contd.)

Hazards Breakdown

4. Titrate the contents of the flask with 0.1 N hydrochloric acid until the last trace of pink color has disappeared. Record the volume of hydrochloric acid used to the nearest 0.01 ml. Apply buret corrections to this reading.

Calculations

$$\% \text{ Sodium hydroxide} = \frac{A \times B \times C \times 100}{D}$$

where A = corrected volume of hydrochloric acid used in the titration, ml

B = normality of hydrochloric acid

C = gram-milliequivalent weight of sodium hydroxide $= \frac{40}{1000} = 0.04$

D = weight of sample, g

Code: 3S 59.0

Issued: January 9, 1956

SODIUM SULFITE: SODIUM SULFITE
Volumetric Method

References

"Reagent Chemicals, ACS Specifications," Washington, D. C., 1950

I. M. Kolthoff and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., p. 604, The Macmillan Co., New York, 1952

Principle, Limitations, and Precision

Sulfurous acid and its salts may be titrated directly with iodine, but the method is inaccurate because of air oxidation of the sulfurous acid or its salts during the titration. Accurate results are obtained when sulfurous acid or its salts are added to an excess of iodine solution and the excess iodine titrated with sodium thiosulfate.

The iodine solution should be stored in a closed container in a dark place. The iodine solution should be standardized before each determination.

The precision of the method is unknown.

Equipment

1	Automatic buret with reservoir, calibrated, 50 ml
2	Iodine flasks, 250 ml
1	Transfer pipet, 10 ml
1	Buret, calibrated, 50 ml
1	Graduated cylinder, 10 ml

Reagents and Chemicals

Iodine, 0.1 N, standardized	(RI 60.2)
Sodium thiosulfate solution, 0.1 N, standardized	(RS 70.1)
Starch indicator, 0.25%	(RS 82.1)
Hydrochloric acid, 10%	(RH 97.0)

Safety Precautions

1. Hydrochloric acid is corrosive and may cause painful burns on prolonged or repeated contact with the skin. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.

SODIUM SULFITE: SODIUM SULFITE
Volumetric Method

Safety Precautions (contd.)

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Measure 50 ± 0.1 ml of standard iodine solution from a calibrated buret into a 250 ml iodine flask. Record, to the nearest 0.01 ml, the volume of iodine added. (Remark 1) Stopper the flask immediately.	1. Inspect all glassware.
2. Weigh 0.25 ± 0.01 g of the sample and add it to the iodine in the flask. Record, to the nearest 0.0001 g, the weight of the sample. Replace the stopper and swirl gently until all the sample dissolves. Allow the sample to stand for 5 minutes.	
3. Pipet 10 ml of 10% hydrochloric acid into the flask and titrate with standard sodium thiosulfate to a faint straw color.	3. If any of the hydrochloric acid contacts the skin, flush the affected area immediately with water.
4. Add 5 ml of 0.25% starch indicator from a graduated cylinder, and continue the titration until the blue color disappears. Record, to the nearest 0.01 ml, the total volume of sodium thiosulfate used. (Remark 1)	

Code: 3S 59.0

Issued: January 9, 1956

SODIUM SULFITE: SODIUM SULFITE
Volumetric Method

Calculations

$$\text{Percent sodium sulfite} = \frac{[(A \times B) - (C \times D)] \times E}{F} \times 100$$

where A = volume of iodine added, ml (step 1)
B = normality of iodine
C = volume of sodium thiosulfate required for the titration, ml (step 4)
D = normality of the sodium thiosulfate
E = gram-milliequivalent weight of sodium sulfite
$= \frac{126.05}{2 \times 1000} = 0.0630$
F = weight of the sample, g (step 2)

Remarks

1. Apply buret corrections to all buret readings.

SRP Laboratory Manual
Essential Materials Methods

Code: 3T 51.1

Issued: **December 6, 1955**

TRICHLOROETHYLENE: (WATER SOLUBLE) CHLORIDES
Turbidimetric Method

References

"Reagent Chemicals, ACS Specifications," Washington, D. C., 1950

Principle, Limitations, and Precision

The chloride content is determined semi-quantitatively by extracting a weighed sample with water, adding an excess of silver nitrate to an aliquot of the water layer, and comparing the turbidity with a control solution containing a known amount of chloride. Thiocyanates, cyanides, and other halogens interfere.

Equipment

1	Weighing buret, Smith, 10 ml
2	Separatory funnel, 125 ml
1	Transfer pipet, 20 ml
2	Transfer pipets, 10 ml
3	Nessler tubes, matched, 50 ml
1	Graduated cylinder, 50 ml
1	Transfer pipet, 1 ml
1	Transfer pipet, 2 ml
1	Transfer pipet, 5 ml

Reagents and Chemicals

Nitric acid, CP, HNO_3, 70%	(RN 44.0)
Chloride standard solution, 0.1 mg Cl^-/ml	(RC 35.3)
Silver nitrate, 0.1 N	(RS 41.2)

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

TRICHLOROETHYLENE: (WATER SOLUBLE) CHLORIDES
Turbidimetric Method

Safety Precautions (contd.)

2. Silver nitrate solution is caustic. It is poisonous when ingested and discolors the skin on contact.

3. Trichloroethylene is toxic upon inhalation or prolonged or repeated contact with the skin. Work in a well-ventilated area. If any trichloroethylene contacts the skin, flush the affected area immediately with water.

4. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a Smith weighing buret, weigh 4.0 to 4.1 ± 0.01 g of sample into a 125 ml separatory funnel. 4.0 g is equivalent to approximately 2.7 ml.	1. Inspect all glassware. Work in a well ventilated area. Wash any contacted areas of the skin with mild soap and water.
2. Pipet 20.0 ml of distilled water into the funnel.	
3. Stopper the funnel and shake the mixture thoroughly for two minutes. Place the funnel in a ring, which is attached to a ring stand, and allow the mixture to stand for one minute.	3. When shaking the funnel, place the index and middle fingers of one hand on the glass stopper, allowing the ring and little fingers and the thumb to grasp the body of the funnel. Hold the stopcock securely in place with the other hand. At least twice during the shaking period invert the funnel and open the stopcock to release the pressure.

Code 3T 51.1

Issued: December 6, 19[illegible]

TRICHLOROETHYLENE: (WATER SOLUBLE) CHLORIDES
Turbidimetric Method

Procedure (contd.)	Hazards Breakdown
4. Draw off and discard the lower layer. Pipet 10 ml of the water layer into a 50 ml Nessler tube.	
5. Add about 20 ml of distilled water to the Nessler tube.	
6. Acidify the solution with nitric acid and then add about 1 ml in excess. Filter the solution if it is turbid.	6. Wear rubber gloves and a rubber apron. Work in a hood. Flood any contacted areas of the skin immediately with water.
7. To a second Nessler tube, add about 40 ml of distilled water, 1 ml of nitric acid, and 2 ml of the chloride standard solution, 0.1 mg Cl^-/ml.	7. If any of the acid contacts the skin, flush the affected area immediately with water
8. Add 5 ml of 0.1 N silver nitrate solution to each Nessler tube. Dilute the solutions to the mark with distilled water. Stopper the tubes and mix the solution thoroughly.	8. If any of the silver nitrate contacts the skin, flush the affected area immediately with water. When inverting the tubes, place the index and middle fingers on the rubber stopper, allowing the ring and little fingers and the thumb to grasp the tube. Never attempt to invert more than one tube at a time.
9. Compare the turbidity of the sample solution with that of the chloride standard solution. Report the chloride content of the sample as less than, equal to, or greater than 0.01%.	

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 3T 51.2

Issued: November 8, 1955

TRICHLOROETHYLENE: DISTILLATION RANGE

Reference

SRP Standard Procedure 3SP 3.0, Distillation Range

Principle, Limitations, and Precision

Refer to SRP Standard Procedure 3SP 3.0, Distillation Range

Equipment

1 Engler flask, 100 ml
1 Thermometer, ASTM solvents distillation, E 1 (40C-44T)
Range: 72 to 126 C
Distillation range apparatus (ED 44.0)

Safety Precautions

1. Do not use external flames or hot exposed heating elements near the receiver of the distillation apparatus. Work in a well ventilated area.

2. Trichloroethylene is toxic when inhaled or by prolonged contact with the skin. Wear rubber gloves and flood any contacted areas with water.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure

Follow Standard Procedure 3SP 3.0, Distillation Range. Record only the initial boiling point (IBP) and the 95% recovery temperatures corrected to standard barometric pressure.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 61.2a

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

References

Dana Method No. S 50.1-T-3, E. I. du Pont de Nemours and Co. (Inc.)

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. 2, p. 2181, D. Van Nostrand and Co., Inc., New York, 1939

"Analysis of Sodium Sulfahydrate," Barium Reduction Corporation, July 26, 1951

Principle, Limitations, and Precision

Using a portion of the sample from which carbonate, sulfite, and sulfate are removed, the volume of hydrochloric acid required to completely neutralize the sodium hydrosulfide and sulfide is determined. Another aliquot of this sample is then treated with a solution of hydrochloric acid sufficient to decompose the sulfide. An excess of iodine is added to oxidize the hydrogen sulfide which is thus liberated. The excess of iodine is determined by titration with sodium thiosulfate.

Since two moles of hydrochloric acid are required to neutralize one of sodium sulfide and only one is required for each mole of sodium hydrosulfide, and since each of the reactions liberates one mole of hydrogen sulfide, a convenient method for calculating the percent of each present is available.

This method is based on the reactions:

$$NaHS + HCl \longrightarrow NaCl + H_2S$$

$$Na_2S + 2HCl \longrightarrow 2NaCl + H_2S$$

$$H_2S + I_2 \longrightarrow 2HI + S$$

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

Sample sizes are selected for a concentration range of 38 to 41% sodium hydrosulfide. The precision of the method is approximately ± 1.0%.

[redacted]

Code: 4S 61.2a

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

Equipment

2 Weighing bottles, 25 ml
4 Volumetric flasks, 250 ml
2 Transfer pipets, 25 ml
1 Graduated cylinder, 25 ml
2 Filtering funnels, 75 mm diam
Filter paper, Whatman No. 40
2 Erlenmeyer flasks, 300 ml
2 Transfer pipets, 50 ml
4 Erlenmeyer flasks, 500 ml
3 Burets, calibrated, 50 ml
Glass beads
1 Transfer pipet, 10 ml
1 Buret, calibrated, 100 ml
1 Graduated cylinder, 10 ml

Reagents and Chemicals

Barium chloride, 10% by weight	(RB 2.2)
Hydrochloric acid, 0.1 N, against primary standard	(RH 97.9)
Lead acetate indicator paper	
Methyl red indicator, 0.1%	(RM 40.0)
Sodium hydroxide, 0.1 N, against primary standard	(RS 60.6)
Hydrochloric acid, 5% by weight	(RH 97.0)
Iodine, 0.1 N, standardized	(RI 60.2)
Sodium thiosulfate, 0.1 N, standardized	(RS 70.1)
Starch indicator, 0.25%	(RS 82.1)

Safety Precautions

1. Sodium hydrosulfide is highly corrosive and poisonous. Work only in a well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

2. The analysis of sodium hydrosulfide is not to be undertaken unless a second person is present. He should be working or stationed at a distance of not less than 10 feet and will immediately make known any unusual incident, then render aid as required, wearing proper safety devices. In event of hydrogen sulfide gas evolution, minimum equipment for rendering aid will be an escape mask.

[redacted]

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

Safety Precautions (contd.)

3. The hydrogen sulfide gas evolved on reaction of sodium hydrosulfide with an acid is toxic. Analysis must therefore be performed only in a well ventilated area.

4. Carefully label all sodium hydrosulfide bottles and working solutions.

5. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a weighing bottle, transfer approximately 30 g of sample to a clean 250 ml volumetric flask. Record the weight to the nearest 0.0005 g. (Remark 1)	1. Inspect all glassware. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.
2. Dilute the sample to the calibration mark with distilled water. Stopper the flask and invert it several times to mix the solution thoroughly. Label the flask Solution 1.	2. Follow the directions regarding the use of volumetric flasks. Hold the flask with both hands when inverting it.
3. Using a transfer pipet, add 25 ml of the diluted sample to a clean 250 ml volumetric flask. Label the flask Solution 2.	3. Use a rubber bulb for suction.

[redacted]

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE

Volumetric Method

Procedure (contd.)	Hazards Breakdown
4. Add 25 ml of 10% barium chloride from a graduated cylinder to Solution 2. Dilute to the calibration mark with distilled water and mix thoroughly.	4. Follow the directions regarding the use of volumetric flasks. Hold the flask with both hands when inverting it.
5. Save Solution 1 for the determination of sodium carbonate.	
6. Allow Solution 2 to stand at least three hours or until the barium carbonate, sulfite, and sulfate precipitates have settled.	
7. Filter approximately 150 ml of the material prepared in step 6 through Whatman No. 40 filter paper into a clean, dry 300 ml Erlenmeyer flask.	
8. Pipet 50 ml of this filtrate into a 500 ml Erlenmeyer flask containing approximately 75 ml of distilled water.	
9. Using a calibrated buret, add 50 ml of 0.1 N hydrochloric acid. Record, to the nearest 0.01 ml, the volume of acid added.	
10. Add several glass beads to the flask. Place the flask on a hot plate and boil the solution until it is free of hydrogen sulfide vapors as indicated by moist lead acetate paper suspended in the vapors. (Remark 2) Allow the solution to cool to room temperature.	10. Work in a hood. Place a HOT sign in front of the hot plate. Use gloves or tongs to remove the flask from the hot plate.

[redacted]

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

Procedure (contd.)

11. Add 2 or 3 drops of methyl red indicator. Using 0.1 N sodium hydroxide, titrate the excess hydrochloric acid to the pink end point which persists for thirty seconds. Record, to the nearest 0.01 ml, the volume of sodium hydroxide used.

12. Add approximately 50 ml of distilled water to a clean 500 ml Erlenmeyer flask.

13. Pipet 10 ml of 5% hydrochloric acid into the flask. Using a calibrated buret, add approximately 100 ml of 0.1 N iodine to the flask. Record, to the nearest 0.01 ml, the volume of iodine added.

14. Pipet 50 ml of the carbonate, sulfite, and sulfate free material prepared in step 7 into the flask. (Remark 3)

15. Titrate the excess iodine contained in the flask by the addition of 0.1 N sodium thiosulfate until a faint straw color is obtained.

Hazards Breakdown

[redacted]

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
16. Add 10 ml of a freshly prepared starch indicator solution and continue the titration to the first disappearance of the blue color. Record, to the nearest 0.01 ml, the volume of sodium thiosulfate used.	

Calculations

1. % Sodium hydrosulfide = $(A - B) \times C$

2. % Sodium sulfide = $(2B - A) \times D$

where $A = (E \times F) - (G \times H) - Q$

$B = (J \times K) - (L \times M)$

$$C = \frac{P \times 100}{N \times R}$$

$$D = \frac{S \times 100}{N \times R}$$

and E = volume of iodine used, ml
F = normality of iodine
G = volume of sodium thiosulfate used, ml
H = normality of sodium thiosulfate
J = volume of hydrochloric acid used, ml
K = normality of hydrochloric acid
L = volume of sodium hydroxide used, ml
M = normality of sodium hydroxide
N = original weight of the sample, g
P = gram-milliequivalent weight of sodium hydrosulfide $= \frac{56}{1000} = 0.056$
Q = correction factor for the thiosulfate content of the sample = 0.01 (Remark 4)
R = dilution factor for the sample aliquot = 0.02
S = gram-milliequivalent weight of sodium sulfide $= \frac{78}{2 \times 1000} = 0.039$

[redacted]

Code: 4S 61.2a

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM HYDROSULFIDE, SODIUM SULFIDE
Volumetric Method

Remarks

1. As soon as the analysis is satisfactorily completed, empty all unused technical 40% sodium hydrosulfide into the sodium hydrosulfide discard bottle located in a hooded sink. Carefully empty all other solutions, one at a time, into a sink, flushing with generous amounts of water before, during, and after the operation.
2. If the moist lead acetate paper darkens when held in the vapors, all of the hydrogen sulfide gas has not been evolved and further boiling is necessary.
3. Add the sample to the flask on a definite time schedule so the time interval between the addition of the sample and the titration will be the same for all determinations.
4. This correction is applicable only if the thiosulfate content is between 0.0 and 0.3%.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 61.2b

Issued: January 25, 1955

SODIUM HYDROSULFIDE: SPECIFIC GRAVITY
Pycnometer Method

References

Dana Method No. S 50.1a-T, E. I. du Pont de Nemours and Co. (Inc.)

SRP Laboratory Equipment Method EP 97.1, Pycnometer, Side Arm, 50 ml

Principle, Limitations, and Precision

A calibrated side arm pycnometer is used to determine the specific gravity of the sample. Values are reported as specific gravity at 25 C with reference to water at 15.6 C.

The precision of the method is unknown.

Equipment

1	Pycnometer, side arm, 50 ml, with thermometer	(EP 97.1)
1	Constant temperature bath	

Safety Precautions

1. Sodium hydrosulfide is highly corrosive and poisonous. Work in a well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

2. The analysis of sodium hydrosulfide is not to be undertaken unless a second person is present. He should be working or stationed at a distance of not less than 10 feet and will immediately report any unusual incident, then render aid as required, wearing proper safety devices. In the event of hydrogen sulfide gas evolution, minimum equipment will be an escape mask.

3. Clearly label the pycnometer containing sodium hydrosulfide.

4. The hydrogen sulfide gas evolved on reaction of sodium hydrosulfide with an acid is toxic. Work in a well ventilated, acid-free area.

[redacted]

SODIUM HYDROSULFIDE: SPECIFIC GRAVITY
Pycnometer Method

Safety Precautions (contd.)

5. As soon as the analysis is completed, carefully empty all unused technical 40% sodium hydrosulfide into the "NaHS Discard" bottle located in a hooded sink.

6. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards Breakdown
1. Calibrate a 50 ml, side arm pycnometer as outlined in Laboratory Equipment Method EP 97.1, Pycnometer, Side Arm, 50 ml, Part A, Calibration, using 25.0 C for the value of T and 15.6 C for the value of T_1.	1. Inspect all glassware. Follow the Hazards outlined in Part A, Calibration, of Laboratory Equipment Method EP 97.1, Pycnometer, Side Arm, 50 ml.
2. Rinse the pycnometer, thermometer, and cap with methyl alcohol or acetone, and dry them in a gentle stream of clean air. Follow this with several rinses using the sample to be tested. (Remark 1)	2. Work in a flame-free, well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.
3. Place a 5 cm length of 1/8 in. ID gum rubber tubing over the side arm to act as a reservoir.	
4. Fill the pycnometer with the sample to be tested.	4. Same as step 2.
5. Insert the thermometer carefully and seat it firmly, taking care that no air bubbles are entrapped.	5. Do not apply excess pressure when seating the thermometer.

[redacted]

Code: 4S 61.2b

Issued: January 25, 1955

SODIUM HYDROSULFIDE: SPECIFIC GRAVITY
Pycnometer Method

Procedure (contd.)

6. Immerse the pycnometer (until at least half of the thermometer joint is covered) in a constant temperature bath at 25.0 C.

7. Allow the pycnometer to remain in the bath until the pycnometer thermometer, after correction, shows the same temperature as that of the bath.

8. Grasp the pycnometer by the thermometer joint and lift it from the bath.

9. Quickly remove the gum rubber tubing and wipe off the tip and joint of the side arm with a dry cloth. Do not pull any liquid out of the side arm capillary.

10. Place the cap in position.

11. Observe to make sure that no air bubbles have collected around the thermometer.

12. Dry the outside of the pycnometer with a clean, lint-free cloth and allow the pycnometer to come to room temperature.

13. Weigh the pycnometer and sample and record the weight to the nearest 0.0001 g.

Hazards Breakdown

9. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

[redacted]

Code: 4S 61.2b

Issued: January 25, 1955

SODIUM HYDROSULFIDE: SPECIFIC GRAVITY
Pycnometer Method

Calculations

Specific gravity of the sample at 25.0 C with reference to water at 15.6 C $= \frac{A - B}{C}$

where A = weight of the pycnometer and sample, g
B = weight of the pycnometer, g
C = weight of water at 15.6 C (refer to Laboratory Equipment Method EP 97.1, Pycnometer, Side Arm, 50 ml)

Remarks

1. The pycnometer should never be heated above room temperature. To do so may cause temporary expansion of the glass with a return to normal only after long standing.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 61.4

Issued: January 20, 1955

SODIUM HYDROSULFIDE: SODIUM CARBONATE
Volumetric Method

References

Dana Method No. S 50.3-T-2, E. I. du Pont de Nemours and Co. (Inc.)

"Analysis of Sodium Sulfahydrate," Barium Reduction Corp., July 26, 1951

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. 2, p. 2181, D. Van Nostrand and Co., Inc., New York, 1939

Principle, Limitations and Precision

Sodium carbonate is determined by precipitation with barium chloride, isolation of the barium carbonate precipitate, and titration of this precipitate with hydrochloric acid.

For concentrations higher than 2%, the sample size should be reduced. The precision of this method is unknown.

Equipment

1	Transfer pipet, 100 ml
1	Graduated cylinder, 25 ml
2	Erlenmeyer flasks, 300 ml
	Filter paper, Whatman No. 40
2	Filtering funnels, 75 mm diam
2	Burets, calibrated, 50 ml
	Glass beads

Reagents and Chemicals

Barium chloride, 10% by weight	(RB 2.2)
Silver nitrate, 10% by weight	(RS 41.3)
Hydrochloric acid, 0.1 N, against primary standard	(RH 97.9)
Methyl red indicator, 0.1%	(RM 40.0)
Sodium hydroxide, 0.1 N, against primary standard	(RS 60.6)

[redacted]

Code: 4S 61.4

Issued: January 20, 1955

SODIUM HYDROSULFIDE: SODIUM CARBONATE
Volumetric Method

Safety Precautions

1. Sodium hydrosulfide is highly corrosive and poisonous. Work only in a well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.
2. The analysis of sodium hydrosulfide is not to be undertaken unless a second person is present. He should be working or stationed at a distance of not less than 10 feet and will immediately report any unusual incident, then render aid as required, wearing proper safety devices. In the event of hydrogen sulfide gas evolution, minimum equipment will be an escape mask.
3. Clearly label all sodium hydrosulfide bottles and working solutions.
4. The hydrogen sulfide gas evolved by the reaction of sodium hydrosulfide with an acid is toxic. Analysis must, therefore, be performed only in a well ventilated area.
5. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards [illegible]
In the analysis of a single sample run duplicate determinations.	
1. Using Solution 1 from Essential Materials Method 4S 61.2a, Sodium Hydrosulfide: Sodium Hydrosulfide, Volumetric Method, step 5, pipet 100 ml of the sample into a clean, 300 ml Erlenmeyer flask.	1. Inspect all glassware.

[redacted]

SODIUM HYDROSULFIDE: SODIUM CARBONATE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
2. Add 25 ml of neutral 10% barium chloride solution to the flask.	
3. Place the flask on a steam bath or an electric burner at low heat and digest for thirty minutes.	
4. After digesting, filter off the precipitated barium carbonate, sulfide, and sulfate on a Whatman No. 40 filter paper. Wash the precipitate and Erlenmeyer flask with warm distilled water, putting all washings through the filter paper, until the washings are free of sulfide when tested with 10% silver nitrate solution. (Remark 1)	4. Wear gloves or use tongs to handle the hot flasks.
5. Return the precipitate and the filter paper to the original flask and add 50-75 ml of hot distilled water.	5. Use gloves when handling the container of hot water.
6. Add, from a calibrated buret, 50 ml of 0.1 N hydrochloric acid. Record the volume to the nearest 0.01 ml.	
7. Add a few glass beads to the flask and boil the solution on a hot plate for 10 to 15 minutes to expel the dissolved carbon dioxide.	7. Place a HOT sign in front of the hot plate when it is in use. Wear gloves or use tongs to remove the hot flask from the hot plate.
8. Allow the solution to cool to room temperature and add four drops of methyl red indicator.	

[redacted]

Code: 4S 61.4

Issued: January 20, 1955

SODIUM HYDROSULFIDE: SODIUM CARBONATE
Volumetric Method

Procedure (contd.)

9. Using a calibrated buret, titrate the excess hydrochloric acid with 0.1 N sodium hydroxide to the pink end point which persists for 30 seconds. Record, to the nearest 0.01 ml, the volume of sodium hydroxide required.

Hazards Breakdown

9. Upon completion of the analysis, empty the solution into the sink. Flush the sink with copious amounts of water before, during, and after the operation.

Calculations

$$\% \text{ Sodium carbonate} = \frac{(A \times B) - (C \times D) \times E \times 100}{F \times G}$$

where A = volume of hydrochloric acid added, ml

B = normality of hydrochloric acid

C = volume of sodium hydroxide required for the titration, ml

D = normality of sodium hydroxide

E = gram-milliequivalent weight of sodium carbonate

$= \frac{105.99}{2 \times 1000} = 0.053$

F = weight of original sample, g (see Essential Materials Method 4S 61.2a, Sodium Hydrosulfide: Sodium Hydrosulfide, Volumetric Method, step 1)

G = dilution factor for sample aliquot $= \frac{100}{250} = 0.4$

Remarks

1. Collect a portion of the filtrate in a test tube and add a few drops of 10% silver nitrate solution. If a precipitate forms, the sulfide ion is present and further washing is necessary.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 61.5

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM THIOSULFATE
Volumetric Method

References

Dana Method No. S 50.4-T-1, E. I. du Pont de Nemours and Co. (Inc.)

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. 2, p. 2181, D. Van Nostrand and Co., Inc., New York, 1939

"Analysis of Sodium Sulfahydrate," Barium Reduction Corp., July 26, 1951

Principle, Limitations, and Precision

A portion of sample, from which carbonate, sulfite and sulfate are removed by precipitation with barium chloride, is boiled with magnesium chloride until the sodium sulfide and hydrosulfide are completely decomposed.

The sodium thiosulfate remaining in solution is then titrated directly using a standard iodine solution.

This method is based on the reactions:

$$MgCl_2 + Na_2S \longrightarrow 2NaCl + MgS$$

$$MgS + 2H_2O \longrightarrow Mg(OH)_2 + H_2S$$

$$MgCl_2 + 2NaHS \longrightarrow 2NaCl + Mg(HS)_2$$

$$Mg(HS)_2 + 2H_2O \longrightarrow Mg(OH)_2 + 2H_2S$$

$$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$$

The precision of the method is unknown.

[redacted]

Code: 4S 61.5

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM THIOSULFATE
Volumetric Method

Equipment

1	Transfer pipet, 25 ml
2	Volumetric flasks, 250 ml
2	Graduated cylinders, 25 ml
	Filter paper, Whatman No. 40
2	Filtering funnels, 75 mm diam
2	Erlenmeyer flasks, 300 ml
2	Transfer pipets, 100 ml
2	Erlenmeyer flasks, 500 ml
1	Buret, calibrated, 50 ml

Reagents and Chemicals

Barium chloride, 10% by weight	(RB 2.2)
Magnesium chloride, crystals, CP, $MgCl_2 \cdot 6H_2O$	
Lead acetate test paper	
Starch indicator, 0.25%	(RS 82.1)
Hydrochloric acid, HCl, CP, 36%	
Iodine, 0.025 N, standardized	(RI 60.1)

Safety Precautions

1. Sodium hydrosulfide is highly corrosive and poisonous, Work only in a well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

2. The analysis of sodium hydrosulfide is not to be undertaken unless a second person is present. He should be working or stationed at a distance of not less than 10 feet and will immediately report any unusual incident, then render aid as required, wearing proper safety devices. In the event of hydrogen sulfide gas evolution, minimum equipment will be an escape mask.

3. Clearly label all sodium hydrosulfide bottles and working solutions.

4. The hydrogen sulfide gas evolved on reaction of sodium hydrosulfide with an acid is toxic. Analysis must, therefore, be performed only in a well ventilated area.

[redacted]

Code: 4S 61.5

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM THIOSULFATE
Volumetric Method

Safety Precautions (contd.)

5. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Using a transfer pipet, add 25 ml of sample to a 250 ml volumetric flask containing approximately 100 ml of distilled water.	1. Inspect all glassware. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.
2. By means of a graduated cylinder, add 25 ml of 10% barium chloride to the flask. Dilute to the calibration mark with distilled water. Stopper the flask and invert it several times to mix the solution thoroughly.	2. Follow the directions regarding the use of volumetric flasks. Hold the flask with both hands when inverting it.
3. Allow the solution to stand at least three hours. Filter about 150 ml of the solution through a Whatman No. 40 filter paper into a clean, dry, 300 ml Erlenmeyer flask.	
4. Using a transfer pipet, add 100 ml of the filtrate to a 500 ml Erlenmeyer flask containing approximately 50 ml of distilled water.	

[redacted]

Code: 4S 61.5

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM THIOSULFATE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
5. Add 25 ± 0.5 g of magnesium chloride to the flask.	
6. Place the flask on a hot plate and boil the solution until it is free of hydrogen sulfide as indicated by moist lead acetate paper held in the boiling vapors. (Remark 1) Continue boiling for an additional five minutes.	6. Place a HOT sign in front of the hot plate when it is in use.
7. Remove the flask from the hot plate and allow the solution to cool to room temperature.	7. Use tongs or wear gloves when handling hot glassware.
8. Using a graduated cylinder, add 10 ml of starch indicator and 20 ml of concentrated hydrochloric acid to the flask. Titrate with 0.025 N iodine to the first faint blue color which persists for one minute. Record, to the nearest 0.01 ml, the volume of iodine consumed.	8. Wear rubber gloves when handling concentrated acids. If any of the acid contacts the skin, wash it off immediately with water. Under no circumstances is the solution from step 8 to be emptied into the "NaHS Discard" bottle.

Calculations

$$\% \text{ Sodium thiosulfate} = \frac{A \times B \times C \times 100}{D \times E \times F}$$

where A = volume of iodine used, ml

B = normality of iodine

C = gram-milliequivalent weight of sodium thiosulfate $= \frac{158.13}{1000} = 0.1581$

D = specific gravity of the sample

E = volume of sample used, ml (step 1)

F = dilution factor for sample aliquot $= \frac{100}{250} = 0.4$

[redacted]

Code: 4S 61.5

Issued: February 2, 1955

SODIUM HYDROSULFIDE: SODIUM THIOSULFATE
Volumetric Method

Remarks

1. If the moist lead acetate paper darkens when held in the boiling vapors, all the hydrogen sulfide gas has not been evolved and further boiling is necessary.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 61.6

Issued: February 15, 1955

SODIUM HYDROSULFIDE: SODIUM SULFITE
Volumetric Method

References

Dana Method No. S 50.5-T-1, E. I. du Pont de Nemours and Co. (Inc.)

"Analysis of Sodium Sulfide, Determination of Sodium Sulfite," Organic Chemicals Dept., Jackson Laboratory, E. I. du Pont de Nemours and Co. (Inc.)

Principle, Limitations, and Precision

Barium chloride is added to the sample to precipitate sulfites as barium sulfite. The precipitate, which also contains carbonates and sulfates, is reacted with excess standard iodine. The iodine is reduced in proportion to the amount of sulfite present and the residual iodine is then titrated with standard sodium thiosulfate. The result is reported as percent sodium sulfite.

This method is based on the reactions:

$$Na_2SO_3 + BaCl_2 \longrightarrow BaSO_3 + 2NaCl$$

$$BaSO_3 + I_2 + H_2O \rightarrow BaSO_4 + 2HI$$

The precision of the method is unknown.

Equipment

2 Weighing bottles
2 Iodine flasks, 500 ml
2 Graduated cylinders, 25 ml
2 Gooch crucibles, with asbestos mats
2 Burets, calibrated, 50 ml

Reagents and Chemicals

Barium chloride, 10% by weight	(RB 2.2)
Hydrochloric acid, CP, HCl, 36%	
Iodine, 0.1 N, standardized	(RI 60.2)
Sodium thiosulfate, 0.1 N, standardized	(RS 70.1)
Starch indicator, 0.25%	(RS 82.1)

[redacted]

Code: 4S 61.6

Issued: February 15, 1955

SODIUM HYDROSULFIDE: SODIUM SULFITE
Volumetric Method

Safety Precautions

1. The analysis of sodium hydrosulfide is not to be undertaken unless a second person is present. He should be working or stationed at a distance of not less than 10 feet and will immediately report any unusual incident, then render aid as required, wearing the proper safety devices. In the event of hydrogen sulfide gas evolution minimum equipment will be an escape mask.

2. Sodium hydrosulfide is highly corrosive and poisonous. Work only in a well ventilated area. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

3. Clearly label all sodium hydrosulfide bottles and working solutions as indicated in the procedure.

4. As soon as the analysis is completed, carefully empty all unused technical 40% sodium hydrosulfide into the "NaHS Discard" bottle located in a hooded sink. Carefully rinse all other solutions, one at a time, into a sink, flushing the sink with copious amounts of water before, during, and after the operation.

5. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh approximately 5 g of sample and add it to a clean, 500 ml glass-stoppered iodine flask containing approximately 50 ml of distilled water. Record the sample weight to the nearest 0.0005 g.	1. Inspect all glassware. If any of the sodium hydrosulfide contacts the skin, wash it off immediately with water.

[redacted]

SODIUM HYDROSULFIDE: SODIUM SULFITE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
2. Slowly add 25 ml of 10% barium chloride reagent. Stopper the flask and invert it several times to mix the solution thoroughly.	2. Follow the directions regarding the use of volumetric flasks. Hold the flask with both hands when inverting it.
3. Allow the solution to stand at least 3 hours or until the barium sulfite, sulfate and carbonate have settled.	
4. Filter off the precipitated barium sulfite, sulfate, and carbonate on a Gooch crucible containing a previously prepared fine asbestos mat.	
5. Wash the iodine flask with four 10 ml portions of hot distilled water, putting all washings through the crucible. (Remark 1)	
6. Return the precipitate and the asbestos mat to the iodine flask.	
7. Dilute the contents of the flask to approximately 250 ml with distilled water. Immediately add, from a buret, 50 ml of 0.1 N iodine and 25 ml of concentrated hydrochloric acid. Record, to the nearest 0.01 ml, the volume of iodine added.	7. Wear rubber gloves when handling concentrated acid. If any of the acid contacts the skin, wash it off immediately with water.

[redacted]

Code: 4S 61.6

Issued: February 15, 1955

SODIUM HYDROSULFIDE: SODIUM SULFITE
Volumetric Method

Procedure (contd.)	Hazards Breakdown
8. Stopper the flask and allow the solution to stand, with occasional swirling, in a cold water bath in the dark for one hour.	
9. Titrate the excess iodine in the flask with 0.1 N sodium thiosulfate until a straw color is obtained. Then add 10 ml of a freshly prepared starch indicator solution and continue the titration until the blue color disappears. Record, to the nearest 0.01 ml, the volume of sodium thiosulfate required.	9. When the titration is completed, empty the flasks into a sink, flushing the sink with copious amounts of water before, during, and after the operation. Keep this solution away from all other solutions containing sodium hydrodrosulfide.

Calculations

$$\% \text{ Sodium sulfite} = \frac{[(A \times B) - (C \times D)] \times E \times 100}{F}$$

where A = volume of iodine added, ml
B = normality of iodine
C = volume of sodium thiosulfate used, ml
D = normality of sodium thiosulfate
E = gram-milliequivalent weight of sodium sulfite
$= \frac{126.06}{2 \times 1000} = 0.063$
F = weight of sample, g

Remarks

1. Sodium sulfite is slightly water soluble. Therefore, washing of the precipitate must be kept to a minimum.

[redacted]

SRP Laboratory Manual
Essential Materials Methods

Code: 4S 62.1a

Issued: July 6, 1954
Revised: October 21, 1955

SULFURIC ACID: CONCENTRATION
Baumé Hydrometer Method

References

N. H. Furman and W. W. Scott, "Scott's Standard Methods of Chemical Analysis," 5th Ed., Vol. II, p. 2205, D. Van Nostrand Co., Inc., 1950

N. A. Lange, "Handbook of Chemistry," 8th Ed., p. 1109, 1178, Handbook Publishers Inc., 1952

Principle, Limitations, and Precision

The specific gravity of sulfuric acid is a convenient measure of its concentration. In this procedure the specific gravity in terms of degrees Baumé is measured by means of a Baumé hydrometer at 25 C. If desired, the percent sulfuric acid at 15.6 C can be determined from this reading by referring to Graph I, which relates concentration at 15.6 C with degrees Baumé at 25 C.

The precision of the method is unknown.

Equipment

1 Hydrometer jar, 250 ml
1 Hydrometer, Baumé, Fisher No. 11-540, range 54-65 degrees
or 1 Hydrometer, Baumé, Fisher No. 11-540, range 64-72 degrees
1 Thermometer, 0-110 C
1 Stirring rod, Pyrex, 12 in. long

Safety Precautions

1. Concentrated sulfuric acid is very corrosive and may cause painful burns on contact with the skin. When handling concentrated sulfuric acid in containers larger than one liter, wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the sulfuric acid contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

[redacted]

Code: 4S 62.1a

Issued: July 6, 1954
Revised: October 21, 1955

SULFURIC ACID: CONCENTRATION
Baume Hydrometer Method

Procedure	Hazards Breakdown
1. Add approximately 200 ml of sample to a clean, dry 250 ml hydrometer jar.	1. Inspect all glassware. Wear rubber gloves with roughened palms and fingers, a rubber apron, and forearm covers.
2. Immerse the hydrometer jar in a constant temperature water bath at 25.0 C so that the water level is above the sample level. At frequent intervals, slowly stir the sample with a glass stirring rod.	2. Do not allow water to come in contact with the sample. Support the hydrometer jar to prevent spilling.
3. Wait 15 minutes for equilibrium to be attained. Check the temperature of the sample with a clean, dry thermometer to make certain that it has reached 25.0 C.	3. The thermometer must be dry. Rinse the thermometer and stirrer with water after use.
4. Remove the jar from the bath.	
5. Slowly immerse a clean, dry Baume hydrometer in the liquid to a depth slightly greater than that at which it floats, and then allow the hydrometer to float freely. (Remarks 1 and 2)	5. The hydrometer must be dry. Clean up any spills immediately. Flush any affected areas of the skin immediately with water.
6. Center the hydrometer in the sample by spinning the hydrometer slightly. Do not read the hydrometer if it is touching the walls of the jar. Be sure there are no air bubbles in the sample.	

[redacted]

Code: 4S 62.1a

Issued: July 6, 1954
Revised: October 21, 1955

SULFURIC ACID: CONCENTRATION
Baumé Hydrometer Method

Procedure (contd.)	Hazards Breakdown
7. With the eyes level with the surface of the sample, read the value on the Baumé scale at the underside of the surface. Record this reading as degrees Baumé.	7. Rinse the hydrometer with water immediately after use.
8. Check the temperature again to be sure it is at 25.0 C.	8. The thermometer must be dry. Rinse the thermometer after use. After the analysis is complete, slowly pour the sample into a sink, flushing the sink with large amounts of water before, during, and after the operation. Allow the water to run for about 5 minutes after pouring.
9. If desired, the percent sulfuric acid at 15.6 C can be obtained from Graph I.	

Remarks

1. If a Baumé hydrometer is not available, a specific gravity hydrometer may be used. The specific gravity reading can be converted to a Baumé reading by means of the following equation:

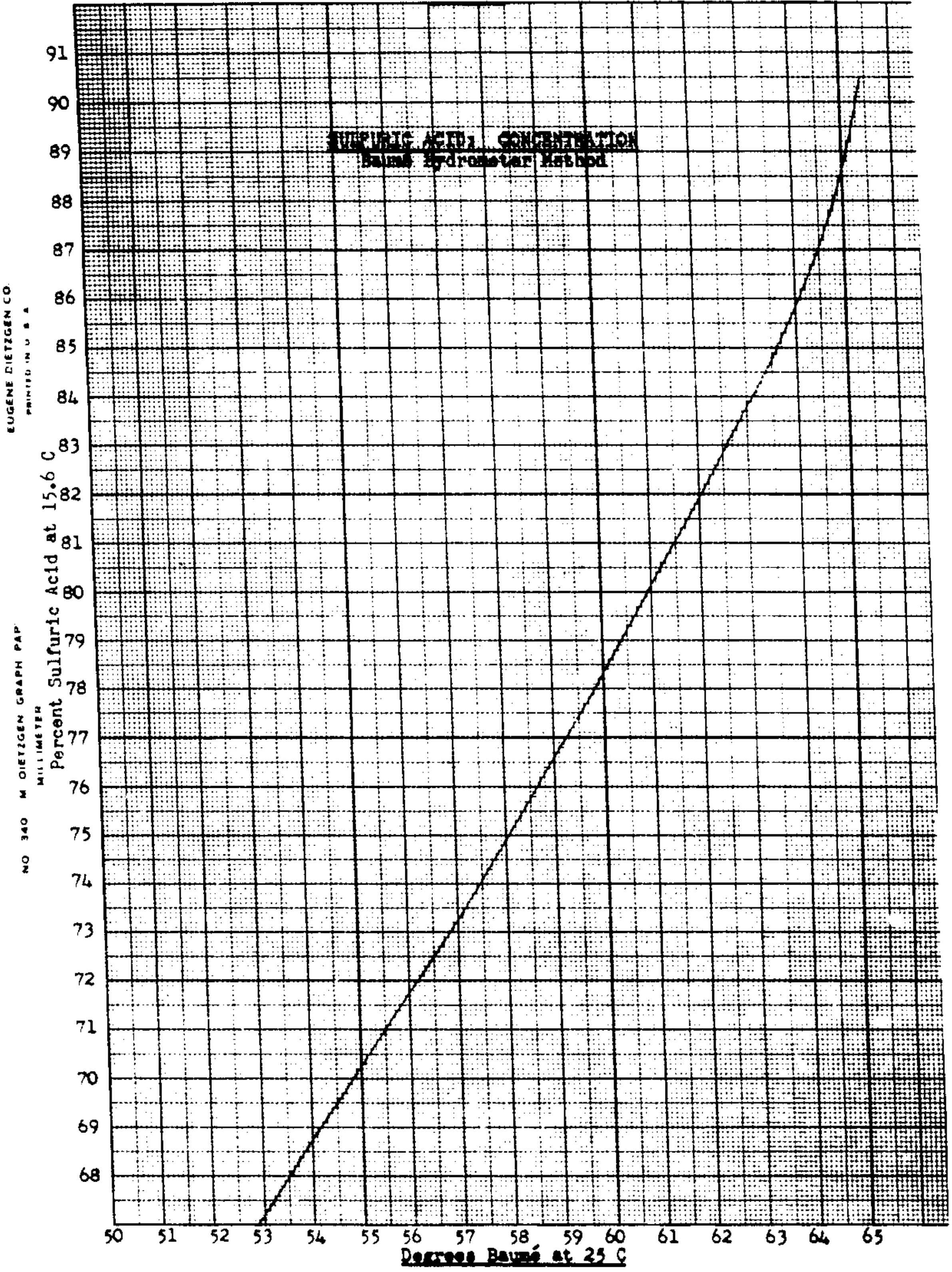
$$\text{Degrees Baumé} = 145 - \frac{145}{\text{specific gravity}}$$

2. If the expected concentration of the sample is less than 65 degrees Baumé, use a hydrometer with a 54-65 degree range. If the expected concentration exceeds 65 degrees Baumé, use a hydrometer with a 64-72 degree range.

[redacted]

Code: 4S 62.1a

GRAPH I



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[redacted]

SRP Laboratory Manual
Standard Procedures

Code: 3SP 2.5

Issued: November 3, 1955

CHLORIDE: QUALITATIVE DETERMINATION

References

I. M. Kolthoff and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., p. 303-308, The Macmillan Co., New York, 1952

Principle, Limitations, and Precision

The sample containing chloride is dissolved, acidified with nitric acid, and precipitated with an excess of silver nitrate. The formation of a white precipitate, silver chloride, indicates the presence of the chloride ion.

$$Ag^+ + Cl^- \longrightarrow AgCl$$

Anions such as bromide, iodide, thiocyanate, cyanide, ferrocyanide, ferricyanide, sulfide, and thiosulfate, must be absent, since these anions form insoluble silver salts in dilute nitric acid.

Equipment

2 Beakers, 100 ml
1 Graduated cylinder, 25 ml
1 Dispensing buret, 50 ml

Reagents and Chemicals

Nitric acid, CP, HNO_3, 70%
Silver nitrate, $AgNO_3$, 0.1 N (RS 41.2)

Safety Precautions

1. Concentrated nitric acid is very corrosive and may cause painful burns on contact with the skin. The fumes of nitric acid are irritating to the eyes and respiratory tract. When handling concentrated nitric acid in containers larger than one liter, work in a hood and wear safety glasses, rubber gloves, a rubber apron, and acid type goggles or a nitrometer mask. If any of the nitric acid contacts the skin, flush the affected area immediately with water.

[redacted]

Code: 3SP 2.5

Issued: November 3, 1955

CHLORIDE: QUALITATIVE DETERMINATION

Safety Precautions (contd.)

2. Silver nitrate solution is caustic. It is poisonous when ingested and discolors the skin on contact.

3. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
In the analysis of a single sample run duplicate determinations.	
1. Weigh, on a triple beam balance, approximately 1.0 g of the sample into a 100 ml beaker.	1. Inspect all glassware.
2. Dissolve the sample in 50 ml of distilled water.	
3. Add 5 ml of nitric acid, stir the solution, and then add 10 ml of silver nitrate.	3. Wear rubber gloves. If any of the solutions contact the skin, flush the affected area of the skin immediately with water.
4. A white precipitate confirms the presence of chloride. Report the chloride as present or absent.	

[redacted]

SRP Laboratory Manual
Standard Procedures

Code: 3SP 5.1

Issued: November 16, 1955

IRON
Colorimetric, Thioglycollic Acid

References

F. D. Snell and C. T. Snell, "Colorimetric Methods of Analysis," 3rd Ed., Vol. II, p. 757, D. Van Nostrand Co., Inc., New York, 1951

Principle, Limitations, and Precision

Thioglycollic acid reacts with ferrous and ferric iron to produce a reddish-purple color proportional to the concentration when the concentration is between 0.02 and 0.2 mg in a final volume of 50 ml. The color is stable in artificial light for 12 hours and is not affected by concentration of reagents or pH within wide limits. More than 10 ppm of copper in the final volume causes bleaching. Citric acid prevents the precipitation of aluminum in the ammoniacal solution. The range of the method is from 0.02 to 0.2 mg of iron.

The precision of the method is unknown.

Equipment

For Preparation of Standard Curve

9	Volumetric flasks, 50 ml	
1	Transfer pipet, 1 ml	
1	Transfer pipet, 2 ml	
2	Transfer pipets, 3 ml	
1	Transfer pipet, 4 ml	
3	Transfer pipets, 5 ml	
1	Spectrophotometer, Beckman Model B	(ES 68.0)
4	Absorption cells, 10 mm light path	

For Analysis of Sample

5	Volumetric flasks, 50 ml	
1	Transfer pipet, 10 ml	
2	Transfer pipets, 5 ml	
1	Transfer pipet, 1 ml	
	Hydrion paper	
1	Transfer pipet, 3 ml	
1	Spectrophotometer, Beckman Model B	(ES 68.0)
4	Absorption cells, 10 mm light path	

[redacted]

Code: 3SP 5.1

Issued: November 16, 1955

IRON

Colorimetric, Thioglycollic Acid

Reagents and Chemicals

Iron standard solution, 0.02 mg Fe/ml	(RI 70.5)
Ammonium citrate, 10% by weight	(RA 64.1)
Thioglycollic acid, 8% by volume, basic	(RT 20.1)
Ammonium hydroxide, 20% by volume	(RA 75.0)
Hydrochloric acid, 20% by volume	(RH 97.0)

Safety Precautions

1. Dilute ammonium hydroxide or hydrochloric acid may cause painful burns on prolonged or repeated contact with the skin. Work in a well ventilated area or hood when handling dilute ammonium hydroxide or dilute hydrochloric acid. If any of the solution contacts the skin, flush the affected area immediately with water.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
A. Preparation of Standard Curve	
1. Prepare a series of iron standard solutions containing 0.02, 0.04, 0.06, 0.08, 0.10, and 0.12 mg of iron by pipeting 1, 2, 3, 4, 5, and 6 ml of the iron standard solution, 0.02 mg Fe/ml, into six 50 ml volumetric flasks. These will be the standard reagent solutions.	1. Inspect all glassware.
2. Prepare a standard blank by pipeting 6 ml of the iron standard solution, 0.02 mg Fe/ml, into another 50 ml volumetric flask.	

[redacted]

Code: 3SP 5.1

Issued: November 16, 1955

IRON
Colorimetric, Thioglycollic Acid

Procedure (contd.)	Hazards Breakdown
A. Preparation of Standard Curve	
3. Prepare a reagent reference and a miscellaneous reagents blank by adding approximately 10 ml of distilled water to two, 50 ml volumetric flasks.	
4. Add 5 ml of 10% ammonium citrate to all flasks. (Remark 1)	4. If any of the solution contacts the skin, flush the affected area immediately with water.
5. Pipet 3 ml of 8% thioglycollic acid into the standard reagent solution and the reagent reference.	5. Same as step 4.
6. Dilute all flasks to approximately 30 ml with distilled water, add 5 ml of 20% ammonium hydroxide, and mix thoroughly.	6. Same as step 4. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
7. Using a spectrophotometer (Remark 2), measure the absorbancies of the standard reagent solutions against the reagent reference, using cells with a 10 mm light path, a wave length of 535 mμ, and a sensitivity of 2. Record the readings as the observed absorbancies of the standard reagent solutions.	
8. Using a spectrophotometer and the same cell arrangement and conditions stated in step 7, measure the absorbancy of the standard blank against the miscellaneous blank. Record the reading as the observed absorbancy of the standard blank.	

[redacted]

Code: 3SP 5.1

Issued: November 16, 1955

IRON
Colorimetric, Thioglycollic Acid

Procedure (contd.)	Hazards Breakdown
A. Preparation of Standard Curve	
9. Obtain the true absorbancies of the standard reagent solutions by subtracting the observed absorbancy of the standard blank from the observed absorbancies of the standard reagent solutions.	
10. Plot, on standard graph paper, the true absorbancies of the standard reagent solutions against the concentrations of the standard reagent solutions.	
B. Analysis of Sample	
In the analysis of a single sample run duplicate determinations.	
1. Prepare a sample reagent solution and a sample blank by pipeting an aliquot of sample containing 0.02 to 0.12 mg of iron into two, clean, dry, 50 ml volumetric flasks.	1. Inspect all glassware.
2. Prepare a reagent reference and a miscellaneous reagents blank by adding approximately 10 ml of distilled water to two, clean, dry, 50 ml volumetric flasks.	
3. Pipet 5 ml of 10% ammonium citrate into all flasks. (Remark 3)	

[redacted]

Code: 3SP 5.1

Issued: November 16, 1955

IRON
Colorimetric, Thioglycollic Acid

Procedure (contd.)	Hazards Breakdown
B. Analysis of Sample	
4. Dilute each flask to approximately 30 ml with distilled water. Using 20% ammonium hydroxide or 20% hydrochloric acid, adjust the pH of each solution to approximately 7, as indicated by Hydrion paper.	4. If any of the solution contacts the skin, flush the affected area immediately with water.
5. Add 3 ml of 8% thioglycollic acid to the sample reagent solution and the reagent reference. Mix well and add 5 ml of 20% ammonium hydroxide to all flasks to develop the color.	5. Same as step 4. Follow the directions regarding the use of volumetric flasks outlined in the Laboratory Safety Rules (Section IV-B) of the Safety Practices Manual, DPSOP 81.
6. Using a spectrophotometer (Remark 2), measure the absorbancy of the sample reagent solution against the reagent reference, using cells with a 10 mm light path, a wave length of 535 mμ, and a sensitivity of 2. Record the reading as the observed absorbancy of the sample reagent solution.	
7. Using a spectrophotometer and the same cell arrangement and conditions as stated in step 6, measure the absorbancy of the sample blank against the miscellaneous reagents blank. Record the reading as the observed absorbancy of the sample blank.	

[redacted]

IRON
Colorimetric, Thioglycollic Acid

Procedure (contd.)

Hazards Breakdown

B. **Analysis of Sample**

8. Obtain the true absorbancy of the sample reagent solution by subtracting the observed absorbancy of the sample blank from the observed absorbancy of the sample reagent solution.

9. Using the standard curve, determine the weight (mg) of iron corresponding to the true absorbancy of the sample reagent solution.

Calculations

$$\% \text{ Iron} = \frac{A \times B \times 100}{C \times D \times 10^3}$$

where A = weight of iron from standard curve, mg
B = volume to which the original sample was diluted, ml
C = weight of sample, g
D = volume of aliquot taken for analysis, ml (step 1)

Remarks

1. If the standard curve is to be used for samples that do not contain aluminum, omit the addition of ammonium citrate.

2. For details of operation of the spectrophotometer, refer to Laboratory Equipment Method ES 68.0, Spectrophotometer, Beckman Model B.

3. If the sample does not contain aluminum, omit the addition of ammonium citrate.

[redacted]

SRP Laboratory Manual
Standard Procedures

Code: 3SP 13.0

Issued: June 22, 1954

WATER INSOLUBLES
Gravimetric Method

References

HW-12866, Hanford Works Laboratory Manual, Essential Materials Method ESI-1a

Principle, Limitations, and Precision

A weighed quantity of sample is dissolved in a definite volume of water under specified conditions. The amount of residue remaining after filtration is a measure of water insoluble. material in the sample. The precision of the method is unknown.

Equipment

2 Beakers, Pyrex, 250 ml
1 Graduated cylinder, 100 ml
2 Watch glasses, 3.5 in. diam
2 Crucibles, sintered-glass, fine-porosity, 30 ml
1 Filter adapter for Gooch crucible
2 Filtering flasks, Pyrex, 500 ml

Safety Precautions

1. Observe all precautions pertaining to the material being analyzed.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is completed and place it in the proper drying area.

[redacted]

Code: SSP 13.0

Issued: June 22, 1954

WATER INSOLUBLES
Gravimetric Method

In the analysis of a single sample, run duplicate determinations.

Procedure	Hazards Breakdown
1. Dry a clean, fine porosity, sintered-glass crucible for one hour in an oven at 105-110 C. Allow it to cool for 30 minutes in a desiccator and then weigh to the nearest 0.0001 g.	1. Inspect all glassware for cracks and chips. Dispose of faulty equipment in the waste glass can. Use tongs when placing the crucible in and removing the crucible from the oven. Use a Desiguard when transporting the desiccator to the balance room.
2. Weigh the specified amount of sample into a 250 ml beaker. (Remark 1)	
3. Add the specified amount of hot distilled water to the beaker. (Remark 2)	3. Handle the container of hot water with asbestos gloves or with beaker tongs.
4. Cover the beaker with a watch glass and heat the solution on a steam bath for one hour. (Remark 3)	4. Use tongs when removing the rings from the top of the steam bath.
5. Insert a filter adapter in a filtering flask, which is connected to a vacuum line through a trap, and place the tared crucible in the adapter. Apply a gentle suction and pour the sample solution into the crucible.	5. Inspect all glassware to insure that it is fit for vacuum work.

GEH-22831

[redacted]

Code: 3SP 13.0

Issued: June 22, 1954

WATER INSOLUBLES
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
6. Rinse the sample beaker six times with 10 ml portions of hot distilled water and pour the rinsings into the crucible.	6. Handle the container of hot water with asbestos gloves or with beaker tongs.
7. Dry the crucible and contents for one hour in an oven at 105-110 C. (Remark 4) Allow it to cool [illegible]r 30 minutes in a desiccator and then weigh to the nearest 0.0001 g.	7. Use tongs when placing the crucible in and removing the crucible from the oven. Use a Desi-guard when transporting the desiccator to the balance room.

Calculations

$$\% \text{ Water insolubles} = \frac{(A - B) \times 100}{C}$$

where A = weight of crucible and residue, g
B = weight of empty crucible, g
C = weight of sample, g

Remarks

1. The accuracy with which the sample must be weighed will be given in the procedure for the particular essential material to be analyzed. All other weighings should be made to the nearest 0.0001 g.
2. The exact amount of water to be added will be given in the procedure for the particular material to be analyzed.
3. For many materials, a one hour digestion period is not necessary. Specific instructions will be given under the particular essential material involved if a different digestion period is necessary.
4. For some materials, other drying temperatures are desirable. Specific instructions will be given for these materials.

[redacted]

SRP Laboratory Manual
Standard Procedures

Code. 3SP 3.0

Issued: November 4, 1955

DISTILLATION RANGE

References

ASTM Standards, Part 5, D 86-46, D 216-40, D 1078-49T, Philadelphia, Pa., 1949

"Instruction Booklet For Use With Princo Fortin Type Barometers", catalog No. 453, Precision Thermometer and Instrument Co., 1434 Brandywine St., Philadelphia 30, Pa.

Principle, Limitations, and Precision

The temperature or temperature range at which the sample distills is determined under carefully standardized conditions. The given conditions are empirical and must be closely followed. The method is applicable to all solvents and thinners provided the proper equipment is used in accordance with the ASTM methods listed above. The method for the particular material to be tested specifies the proper flask, thermometer, and conditions to be used. This standard procedure outlines general conditions applicable to all ASTM distillations.

Equipment

Distillation range apparatus	(ED 44.0)

Safety Precautions

1. Observe all safety precautions involved in handling the particular material to be distilled. Work in a well ventilated, flame-free area.

2. Inspect all glassware before and after use for cracks and chipped or jagged edges. Remove from service any that is faulty and immediately dispose of it in the waste glass container if it cannot be salvaged. Carefully rinse all glassware as soon as the analysis is complete and place it in the proper drying area.

Procedure	Hazards Breakdown
1. Swab the inside of the condenser tube clean and dry with a soft, lint-free cloth attached to the end of a wire or rod.	1. Inspect all glassware. Wear leather gloves to clean the condenser.

[redacted]

Code: 3SP 3.0

Issued: November 4, 1955

DISTILLATION RANGE

Procedure (contd.)	Hazards Breakdown
2. Fill the condenser bath with enough distilled water to cover the condenser tube. Maintain a temperature of 10 to 20 C throughout the distillation by adding warm or cold distilled water as needed.	
3. Shake the sample until it is thoroughly mixed. Allow any separated water or cloudiness to settle. Filter approximately 200 ml of clear sample through rapid soft, filter paper into a clean 250 ml beaker. Cover the funnel with a watch glass while the liquid drains.	3. When mixing the sample remove the stopper or cap at intervals to allow expanding gases to escape.
4. Measure 100 ± 0.5 ml of the sample into a clean, dry 100 ml graduated cylinder. Transfer the sample to a cool, clean, dry distillation flask of the size specified in the procedure for the material to be tested. Do not permit any liquid to flow into the side arm of the flask.	
5. Place the graduated cylinder, which was used to measure the sample, under the lower end of the condenser tube to receive the distillate.	

[redacted]

DISTILLATION RANGE

Procedure (contd.)	Hazards Breakdown
6. Select the specified partial immersion thermometer. Insert the thermometer into the neck of the flask through a tight-fitting cork stopper so that the upper end of the thermometer bulb is on a level with the bottom of the vapor-outlet tube (side arm) at its junction with the neck of the flask. The thermometer should be vertical and centrally located in the neck of the flask.	6. Use leather gloves when boring the corks and when inserting the thermometer and side arm in the corks.
7. Mount the flask in the heater opening of the apparatus so that the neck of the flask is vertical and the side arm extends through a tight-fitting cork stopper 1 to 2 in. into the condenser tube. (Remark 1)	7. Same as step 6.
8. Apply heat to the flask at a uniform rate so that the first drop of the distillate falls from the condenser in not less than five and not more than 10 minutes. Avoid major changes in the heating rate. (Remark 2)	
9. Read, to the nearest degree, the temperature of the distillation thermometer when the first drop of distillate falls from the end of the condenser tube. Record this reading as the initial boiling point (IBP).	
10. When the distillation begins, position the inner wall of the receiver against the tip of the condenser and allow the condensate to flow down the walls of the receiver.	

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DISTILLATION RANGE

Procedure (contd.)	Hazards Breakdown
11. Adjust the rate of heating so that the distillation proceeds at a rate of 4 to 5 ml per minute (about 2 drops per second). Make no further adjustment in the rate of heating during the course of the distillation. The rate of heating may be increased only when more than 5 minutes are required to bring over the last 5 ml of the distillate and reach the end point (EP). (Remark 3)	
12. Record, to the nearest degree, the temperature readings of the distillation thermometer when 5%, 10%, each additional 10% up to and including 90% and 95% of the sample has distilled over. (Remark 4)	
13. Record, to the nearest degree, the temperature reading of the distillation thermometer at the instant the last drop of liquid evaporates from the bottom of the distillation flask. This is the dry point (DP). Disregard any liquid clinging to the side of the flask.	
14. When testing crude materials, continue the distillation and record, to the nearest degree, the temperature at the decomposition or end point (EP). When the temperature begins to fall, discontinue the heating. (Remark 3)	

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DISTILLATION RANGE

Procedure (contd.)	Hazards Breakdown
15. Record, to the nearest ml, the total volume of the distillate collected in the receiving graduate as the recovery. The recovery must not be less than 97% for materials with a boiling range of about 10 degrees or less, and not less than 95% for wide-boiling refined products and light oils. Otherwise, the test must be repeated.	
16. Allow any liquid remaining in the distillation flask at the end of the test to cool to room temperature. Transfer the cooled liquid to a small cylinder graduated in 0.1 ml divisions. Record the volume as residue.	16. Allow the distillation flask to cool before dismantling the set-up.
17. Using a barometer, record, to the nearest 0.1 mm, the barometric pressure. Record, to the nearest 0.5 C, the room temperature.	

Calculations

I. Using the following equations, correct the recorded temperature readings to standard barometric pressure. (Remark 5)

Corrected temperature, degrees centigrade = A + B

$$B = 0.00012 \times (760 - C) \times (273 + A)$$

$$C = D - (E) - (F)$$

where A = recorded temperature, degrees centigrade (Remark 6)

B = correction to be added algebraically to the recorded temperature, degrees centigrade

C = barometric pressure corrected for temperature and gravity, mm

D = recorded barometric pressure, mm (step 17)

E = temperature correction from table corresponding to the barometric pressure and the room temperature, degrees centigrade, (Remark 7)

F = gravity correction from table corresponding to the latitude and barometric pressure = 0.8 (Remark 8)

[redacted]

DISTILLATION RANGE

Calculations (contd.)

II. Using the following equation, calculate the distillation loss of the sample.

Distillation loss, ml $= 100 - (G + H)$

where G = volume of residue, ml (step 16)
H = volume of recovery, ml (step 15)

Remarks

1. For low boiling materials, cool the heating unit to room temperature before beginning the distillation.
2. The sample must be heated uniformly to prevent superheating. Application of uniform heat will give a more accurate initial boiling point (IBP).
3. The end point (EP) is the maximum temperature observed during the distillation.
4. In some cases, only certain readings need to be taken as directed in the procedure for the particular material being tested.
5. Temperature corrections to standard barometric pressure need not be made unless directed in the procedure for the particular material being tested.
6. Apply thermometer corrections to all temperature readings.
7. The tables used in the calculations may be found in the Instruction Booklet listed in the reference section of this procedure.
8. The gravity correction factor 0.8 applies only for latitude 34 and a barometric pressure of 740-760 mm. Should the barometric pressure fall below 740 mm, a new correction factor must be obtained from the table.

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Standard Procedures

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RESIDUE ON EVAPORATION
Gravimetric Method

References

"Reagent Chemicals, ACS Specifications," ACS, Washington, D. C., 1950

Rosin, "Reagent Chemicals and Standards," D. Van Nostrand Co., Inc., New York, 1939

Principle, Limitations, and Precision

A representative sample is evaporated to dryness in a platinum dish. The residue, consisting of non-volatile solids, is then dried to constant weight in an oven. The weight of residue obtained is expressed as a percentage of the original material.

The precision of the method is unknown.

Equipment

2 Evaporating dishes, platinum, 50 or 100 ml

Safety Precautions

1. Observe all precautions pertaining to the material being analyzed.
2. Steam produces severe burns. Be cautious when using the steam bath.

Procedure	Hazards Breakdown
In the analysis of a single sample, run duplicate determinations.	
1. Dry a 50 ml platinum evaporating dish (Remark 1) for one hour in an oven at 105-110 C. Allow the dish to cool in a desiccator for 15 minutes and weigh it to the nearest 0.0001 g.	1. Use platinum tipped tongs when placing the dish in and removing the dish from the oven. Use a Desiguard when transporting the desiccator to the balance room.

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RESIDUE ON EVAPORATION
Gravimetric Method

Procedure (contd.)	Hazards Breakdown
2. Weigh or transfer the specified amount of sample into the dish. (Remarks 2 and 3)	
3. Evaporate the sample to dryness on a steam bath in a well ventilated hood.	3. Use tongs when removing the rings from the top of the steam bath. Handle the dish with platinum tipped tongs when placing it on and removing it from the steam bath.
4. Heat the residue for one hour in an oven at 105-110 C. Allow the dish to cool in a desiccator for 15 minutes and weigh it to the nearest 0.0001 g.	4. Use platinum tipped tongs when placing the dish in and removing the dish from the oven. Use a Desiguard when transporting the desiccator to the balance room.

Calculations

$$\% \text{ Residue} = \frac{(A - B) \times 100}{C}$$

where A = weight of dish plus residue, g
B = weight of dish, g
C = weight of sample, g

Remarks

1. For details of care and use of platinum ware refer to the Laboratory Training Manual.
2. It is unnecessary to weigh the sample with an accuracy exceeding 0.01 g. All other weighings, however, should be made to the nearest 0.0001 g.
3. Specific instructions will be given where variations of the procedure are desirable.