

**Processing
KODAK
Motion Picture Films,
Module 4**

**Reagent Preparation
Procedures**



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4 Reagent Preparation Procedures

QUALITY CONTROL FOR ANALYTICAL REAGENTS

Volumes of Reagents

The instructions are generally written for the preparation of 1-litre quantities, but larger volumes should be prepared if they can be consumed before the reagent expiration date.

Volumetric Flask Measurements

Volumetric flasks are to be used only when specified. Approximate volumes can be measured with graduated cylinders or tip-up pipets.

Weight Measurements

When weighing a sample to a specified weight, the tolerance is ± 1 unit in the last decimal place to the right. For example, 15.0 grams is understood to mean $\pm 15.0 \text{ g} \pm 0.1 \text{ g}$ and 15.000 grams is understood to mean $\pm 15.000 \text{ g} \pm 0.001 \text{ g}$.

When the weight of a sample, in grams, is specified to two or more decimal places (e.g., 15.75), use an analytical balance. A torsion balance or a triple-beam balance may be used for weighings which require less accuracy.

Water Quality

Use only distilled water in the preparation of standard reagents. Deionized water may be used if data are obtained that indicate reagents prepared with it are satisfactory.

Expiration Date

When no expiration date is given, it is implied either that the reagent is stable or that its proposed use is such that a critical concentration is not required.

Standardization of Reagents

Quality standardization of reagents is based on the use of the recommended methods, equipment, etc., and the ability of the analyst to replicate his standardizations well enough so that the standard deviation does not exceed 0.2 percent of the nominal normality. Rather than actually calculating the standard deviation from a series of successive standardizations, conformance to this precision requirement may be approximated sufficiently well by obtaining the range of the standardizations. The maximum permissible ranges between the highest and lowest values obtained in a series of replications are shown in the table below.

Table 4-1 Maximum Permissible Range of Normalities of Replicate Standardizations

Cumulative Number of Standardizations	0.0500 N Reagent	0.1000 N Reagent	1.000 N Reagent	2.500 N Reagent
2	0.00035	0.0007	0.007	0.0175
3	0.00040	0.0008	0.008	0.0200
4	0.00045	0.0009	0.009	0.0225
5	0.00045	0.0009	0.009	0.0225
6	0.00050	0.0010	0.010	0.0250

If the range of the standardizations does not fall within these values, the assignable cause for the “excessive” variability should be sought and corrected. Unless the range conforms to the table of *Maximum Permissible Range of Normalities of Replicate Standardizations*, the “out-of-control” values are useless for deciding whether the normality of the reagent is within the defined satisfactory limits. A careful analyst, well trained in the standardization procedures, should have no difficulty in obtaining results within the relatively broad tolerances.

Analyst Procedure

1. Run duplicate analyses for the standardization of the reagent and express results as normality.
2. Determine the range of the duplicates.
3. Refer to the table above to determine whether the duplicates are within the permissible range.
4. If within the permissible range, calculate the reagent normality using the replicate values.
5. If not within the permissible range, search for assignable causes for the excessive variation. If the cause cannot be found and corrected, discard both values and repeat the standardizations until the resulting range is within the permissible range.
6. Label the reagent stock bottle with the normality value to be used in all calculations.

PREPARATION OF SPECIFIC REAGENTS

This section alphabetically lists all reagents required by the analytical methods/procedures used in chemical control of the EASTMAN Color Processes ECN-2, ECP-2B, VNF-1 and RVNP.

For difficult-to-obtain chemicals, we have tried to provide a current source and catalog number in the reagent preparation write-up, or the last section of this module *Chemical Supply Sources*. A less extensive list of chemicals required for analytical laboratory use and processing solution preparation is given in each “Process Specifications” module, and includes several suppliers. The list of suppliers is not intended to be exhaustive, nor a recommendation by Eastman Kodak Company. For additional suppliers, consult the following publications available in many public libraries: *Chemical Week Buyer's Guide*, *PD Chemical Buyer's Directory*, *CPI Purchasing Directory* or *Chemyclopedia*.

Reagent Preparation Format

An explanation of the format for reagent preparation write-ups follows. The example on the next page illustrates each feature in the format. The features are described as they occur from top to bottom in the example.

Reagent Title—identifies a reagent made up of several chemicals [e.g. CITRATE BUFFER], or a single chemical that is used in an analytical method either in its concentrated form or after dilution [e.g., ACETIC ACID].

Chemical Name and Formula—identifies a chemical used in this preparation.

Purity Descriptor—identifies the chemical purity requirement for the preparation; usually it is for Reagent Grade Chemicals that meet American Chemical Society (ACS) specifications, unless specified otherwise.

Method Number—when a number is referenced [e.g., 1205G], it identifies an analytical method that requires use of the chemical identified by the “Chemical Name” This method number will also appear after the last “step” if that dilution is required.


Hazard Notice—when a chemical constitutes a health or safety hazard in its concentrated form, a chemical hazard notice preceded by the word or words DANGER, WARNING or CAUTION is given. If the diluted chemical is also hazardous, the hazard notice is displayed where the dilution procedure is described.

Dilution—identifies the solution concentration being prepared.

Steps—actions and measurements required to complete the reagent preparation. (Sometimes the “method number” explained above appears after the last preparation step. When it does, it identifies a method [e.g., ECN-2-1570C], that requires the dilution being prepared to make the referenced reagent, or to perform the method.)

Standardization—when a reagent must be standardized, the next section will list any additional reagents needed to carry out the standardization along with the steps required to perform the standardization. The equations for calculations needed to arrive at the exact solution concentration will also be given.

Table 4-2 Reagent Preparation Example

<i>Reagent Title</i>	ACETIC ACID
<i>Chemical Name and Formula</i>	• Glacial Acetic Acid, CH ₃ COOH
<i>Purity Description</i>	Reagent Grade, ACA Specification
<i>Method Numbers</i>	ECN-2-ML-3255, ECN-0009/1 ECP-0009/1 ECR-440B
<i>Hazard Notice</i>	<div> Warning</div> <p>CAUSES SEVERE EYE AND SKIN BURNS. Do not get in eyes, on skin, on clothing. Wear eye protection and impervious gloves when handling. Vapor extremely irritating. Avoid breathing vapor. Use with adequate ventilation. An exhaust hood is recommended. COMBUSTIBLE. Keep away from heat and flame.</p>
<i>Dilution</i>	Preparation 2.0 N Acetic Acid
<i>Steps</i>	<ol style="list-style-type: none">1. Place about 500 mL of distilled water in a 1-litre volumetric flask.2. Using a graduated cylinder, add slowly, while stirring, 115 mL of reagent-grade glacial acetic acid.3. Allow the solution to cool to room temperature and then dilute to volume with distilled water ad mix.
<i>Method Numbers</i>	ECN-2 -1570C, ECN-0002/1 ECP-0002/1 ECR-925A

REAGENT PREPARATIONS

Note: Information presented herein is accurate and reliable to the best of our knowledge and belief, but it is furnished without warranty of any kind. Customers must make their own determination of the suitability or completeness of any product, material, and/or procedure for a specific purpose and adopt such safety precautions as may be necessary.

Note: Observe precautionary information on chemical labels and on the Material Safety Data Sheets.

Accelerator Reagent

(6.0 N NaOH – 0.014 M EDTA)

- *Ethylenedinitrilotetraacetic Acid (EDTA)*

Also: Ethylenediamine Tetraacetic Acid

Reagent Grade, ACS Specifications

- *Sodium Hydroxide*, NaOH

Reagent Grade, ACS Specifications



Warning

CORROSIVE. Avoid contact with skin, or clothing. Do not weigh in an aluminum dish.

Note: Sodium hydroxide pellets are deliquescent. The supply container should only be open long enough to weigh the required amount of reagent. Keep the container tightly closed at other times

Preparation

6.0 N Accelerator Reagent

1. Weigh 240 grams of reagent grade sodium hydroxide.
2. Weigh 4.0 grams of reagent grade EDTA.
3. Place 1 litre of distilled water in a 2 litre beaker using a graduated cylinder.
4. Cautiously add and dissolve the sodium hydroxide and then add and dissolve the EDTA. Mix thoroughly,

Note: Sodium hydroxide solutions should be stored in rubber-stoppered Pyrex bottles.

1209D

Acetic Acid

- Glacial Acetic Acid, CH_3COOH

Reagent Grade, ACS Specifications

ECN-2-ML-3255, ECN-0009/1

ECP-0009/1, ECP-0022/1

ECR-440B



Warning

CAUSES SEVERE EYE AND SKIN BURNS. Do not get in eyes, on skin, on clothing. Wear eye protection and impervious gloves when handling. Vapor extremely irritating. Avoid breathing vapor. Use with adequate ventilation. An exhaust hood is recommended. COMBUSTIBLE. Keep away from heat and flame.

Preparation

2.0 N Acetic Acid

1. Place about 500 mL of distilled water in a 1-litre volumetric flask.
2. Using a graduated cylinder, add slowly, while stirring, 115 mL of reagent-grade glacial acetic acid.
3. Allow the solution to cool to room temperature and then dilute to volume with distilled water and mix.

ECN-2-1570C, ECN-0002/1

ECP-0002/1

ECR-925A

Acetone

- Acetone, $(\text{CH}_3)_2\text{CO}$

Reagent Grade, ACS Specifications

ULM-0004/1

ECR-930E



Warning

EXTREMELY FLAMMABLE. Keep away from heat, sparks, and flame. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with skin.

Aluminum

- Aluminum Wire, 20 gauge

Reagent Grade, ACS Specifications

900

Ammonium Hydroxide

- Ammonium Hydroxide, NH_4OH , conc., approx. 15 N
Reagent Grade, ACS Specifications



Warning

CORROSIVE. Avoid contact with solution and breathing of vapor. Use an exhaust hood. Wear eye protection and impervious gloves. Store in a cool place to prevent pressure build-up in the container.

Ammonium Molybdate

- Ammonium Molybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$
Reagent Grade, ACS Specifications



Caution

Use only with adequate ventilation. Use of a hood is preferable.

Ammonium Nitrate

(1.0 M)

- Ammonium Nitrate, NH_4NO_3
Reagent Grade, ACS Specifications



Warning

Causes eye and skin irritation. STRONG OXIDIZER. Forms explosive mixtures with reducing agents such as hydrocarbons, cellulose, and metals.

Preparation

1.0 M Ammonium Nitrate

- Weigh 80.0 g of reagent-grade ammonium nitrate.
- Dissolve in about 800 mL of distilled water in a 1-litre volumetric flask.
- When the solution is at room temperature, dilute to volume with distilled water and mix.

ECN-2-926C

ECP-2-926C

ECR-929C, ECR-930E

Ammonium Persulfate

- Ammonium Persulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$
Reagent Grade, ACS Specifications

RA-4-3300A



Warning

STRONG OXIDIZER. Contact with other material can cause fire. Harmful if swallowed. Keep dry, cool and protected from organic matter.

Ammonium Thiocyanate

- Ammonium Thiocyanate, NH_4CNS
Reagent Grade, ACS Specifications



Caution

IRRITANT. Harmful if absorbed through skin. Avoid contact with eyes and skin. Wear impervious gloves and eye protection when using.

Preparation

200 g/L Ammonium Thiocyanate

- Add 600 mL of distilled water to a 1-litre volumetric flask, stir on a magnetic stirrer.
- Add 200.0 g of ammonium thiocyanate to the volumetric flask; stir to dissolve.
- Dilute to volume with distilled water.
- Filter the reagent through a Whatman 2V filter paper.

ECN-2-ML-3260, ECN-2-3263

ECP-0006/1

Anti-Calcium, No. 4, KODAK

- KODAK Anti-Calcium, No. 4
Eastman Kodak Company, Rochester, NY

ECR-0001-1

Arsenic Trioxide

- Arsenic Trioxide, As_2O_3

Also: Arsenous Oxide, Arsenous Acid Anhydride, Arsenous Acid, Arsenic Sesquioxide or White Arsenic

NIST Oxidimetric Primary Standard SRM-83d
(or subsequent lot of SAM-83)



Warning

POISON. HARMFUL IF INHALED. IRRITANT. Avoid contact with eyes and skin. Wear impervious gloves and eye protection when using. Do not inhale vapors. Use only with adequate ventilation.

Borax Buffer (0.01M)

Please refer to ULM 191-2

for instructions on preparing buffers for pH Measurement of Photographic Processing Solutions.

Borohydride Reagent

- Potassium Borohydride, KBH_4

Reagent Grade, ACS Specifications



Caution

IRRITANT. Avoid contact with eyes and skin. Wear impervious gloves and eye protection when using. Avoid inhalation. Use with adequate ventilation only.

- 0.20 N *Sodium Hydroxide*, NaOH

Preparation

1. Carefully add 100 mL of 0.20 N sodium hydroxide to a 250 mL beaker; stir on a magnetic stirrer.
2. Add 3.0 g of potassium borohydride to the beaker; stir to dissolve.

ULM-0004/1

Note: Store in polyethylene bottles. This solution should be prepared fresh weekly.

Butyl Acetate

- n-Butyl Acetate, $\text{CH}_3\text{COO}(\text{CH}_2)_3\text{CH}_3$

Technical Grade

ECR-125F

Reagent Grade, ACS Specifications

ECN-0003/1

ECP-0003/1

ECR-1603E



Warning

FLAMMABLE. CAUSES EYE IRRITATION. Keep away from heat, sparks and flame. Avoid breathing vapor. Avoid contact with eyes and skin. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with skin.

Cadmium Nitrate

- Cadmium Nitrate, $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$

Reagent Grade, ACS Specifications



Warning

OXIDIZER. Keep separate from oxidizable materials. Poison. Avoid inhalation. Use with adequate ventilation. Avoid contact with eyes, skin, and clothing.

- *Nitric Acid*, HNO_3 , Concentrated

Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Preparation

1.0 M Cadmium Nitrate (also referred to as 300 g/L)

1. Dissolve 308 grams of cadmium nitrate in about 800 mL of distilled water contained in a 1-litre volumetric flask.
2. Dilute to volume with distilled water and mix.

Acidified Cadmium Nitrate

1. Dissolve 200 grams of cadmium nitrate in about 800 mL of distilled water contained in a 1-litre volumetric flask.
2. Using a graduated cylinder, carefully add 20 mL of concentrated nitric acid.
3. Dilute to volume with distilled water and mix.

Calcium Chloride/Calcium Hydroxide Buffer

Please refer to ULM 191-2

for instructions on preparing buffers for pH Measurement of Photographic Processing Solutions.

Calcium Hydroxide

- Calcium Hydroxide, $\text{Ca}(\text{OH})_2$

Reagent Grade, ACS Specifications



Warning

Causes eye and skin burns. Do not get in eyes, on skin, or on clothing. Avoid inhalation of dust. Use with adequate ventilation. Wear impervious gloves and eye protection when using.

Celite

- Celite—analytical filter aid
Reagent Grade, ACS Specifications

ECN-2-926C, ECN-0004/1

ECP-2-926C, ECP-0004/1

ECR-929C, ECR-930E, ECR-1611D, ECR-1612,

ECR-0004/1

Ceric Ammonium Nitrate

- Ceric Ammonium Nitrate, $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$
GFS Certified No. 15 (NIST Working Standard), GFS
Chemicals or equivalent

Cetyltrimethylammonium Bromide (CTAB)

(10 g/L)

- Cetyltrimethylammonium Bromide,
 $\text{CH}_3(\text{CH}_2)_{15}\text{N}(\text{CH}_3)_3\text{Br}$
Also: Hexadecyltrimethylammonium Bromide
Technical Grade

Preparation

1. Add approximately 200 mL of distilled water to a 250 mL volumetric flask.
2. Add and dissolve 2.50 g of cetyltrimethylammonium bromide.
3. Dilute to volume with distilled water and mix.

ECN-2-1570C

Chloroform

- Chloroform, CHCl_3
Reagent Grade, ACS Specifications

ECR-925A

Spectro Grade, Washed



Warning

DANGER! Suspected carcinogen. WARNING! Harmful if inhaled. Can cause embryo-fetal injury Avoid breathing vapor. Keep container closed Use with adequate ventilation. Avoid prolonged or repeated contact with skin.

Washed Chloroform

1. From a graduated cylinder, add 250 mL of spectro grade chloroform to a separatory funnel.
2. Add 125 mL of distilled water from a graduate
3. Stopper and shake the funnel for 30 seconds.
4. Allow the layers to separate for 10 minutes before decanting the chloroform (lower) layer.

ECP-2-936A

Citrate Buffer

- Citric Acid 1-Hydrate, $\text{HOC}(\text{COOH})(\text{CH}_2\text{COOH})_2 \cdot \text{H}_2\text{O}$
Reagent Grade, ACS Specifications



Warning

Causes eye burns and skin irritation. Do not get in eyes, on skin, on clothing. Wear impervious gloves and eye protection when using.

- Sodium Citrate 2-Hydrate,
 $\text{NaOC}(\text{COOH})(\text{CH}_2\text{COONa})_2 \cdot 2\text{H}_2\text{O}$
Reagent Grade, ACS Specifications

Preparation

2 M Citrate Buffer

1. Add approximately 600 mL of distilled water to a 1-litre volumetric flask on a magnetic stirrer.
2. Add and dissolve 210.14 g of citric acid 1-hydrate.
3. Add and dissolve 294.11 g of sodium citrate 2-hydrate.
4. Dilute to volume with distilled water.
5. The pH of this solution is 3.92 ± 0.03 at 77°F (25°C). If not, adjust this solution by adding small amount of sodium hydroxide to raise the pH or citric acid to lower the pH.

ECP-2-936A

Citric Acid

- Citric Acid 1-Hydrate, $\text{HOC}(\text{COOH})(\text{CH}_2\text{COOH})_2 \cdot \text{H}_2\text{O}$
Reagent Grade, ACS Specifications



Warning

Causes eye and skin burns. Do not get in eyes, on skin, or on clothing. Avoid inhalation of dust. Wear impervious gloves and eye protection when using.

Cleaning Solutions

(Sulfuric/Dichromate and Acid/Alcohol)

- Sulfuric Acid, H_2SO_4 , Conc., approx. 36 N
Technical or Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor.

- Sodium Dichromate, $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ or
- Potassium Dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$
Technical Grade

Note: The use and discharge of Cr(VI) containing chemicals may not be allowed in your locality due to State or Local regulations.



Warning

POISON. May be fatal if swallowed. Causes severe skin and eye burns. Suspected cancer hazard. May cause kidney damage. Harmful if inhaled or absorbed through the skin. Dust irritating to eyes and respiratory tract. May cause allergic skin reaction.

STRONG OXIDIZER. Contact with other material can cause fire. Do not breathe dust. Do not get in eyes, on skin, on clothing. Wash thoroughly after handling. Use with adequate ventilation. Keep from contact with clothing and combustible materials. Remove and wash contaminated clothing promptly. Before using, read Material Safety Data Sheet.

Note: The sodium salt is preferred because of its greater solubility.

- 3.0 N *Hydrochloric Acid*, HCl
- *Methanol*, CH₃OH

Practical or Reagent Grade, ACS Specifications



Warning

POISON. May be fatal or cause blindness if swallowed. Can not be made non-poisonous. Harmful if inhaled. Harmful if absorbed through the skin. Causes skin and eye irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly after handling. Avoid breathing vapor. Use with adequate ventilation. **FLAMMABLE.** Keep away from heat, sparks, and flame. Store in original tightly closed container.

Preparation

Sulfuric/Dichromate

1. Weigh about 30 g of sodium or potassium bichromate.
2. Add, while stirring, to 1 litre of concentrated sulfuric acid.

Note: Excess dichromate will separate from the liquid as chromium trioxide. To prevent clogging buret or pipet tips with the crystals while cleaning, decant the clear cleaning solution before using.

3. Use the solution undiluted.

ULM-0005/1



Caution

Wear impervious gloves and safety goggles when handling. The solution may be reused. Discard when it turns green.

Acid/Alcohol

1. Add carefully one volume 3.0 N hydrochloric acid to one volume methanol, while stirring.
2. Use undiluted.
3. Discard when the solution becomes highly colored.

ULM-0001/1

ULM-0005/1

m-Cresol Purple Indicator

Color change: Yellow to purple

pH7.4 to 9.0

- Meta Cresol Purple, m-cresolsulfonphthalein
Reagent Grade
- 0.01 N *Sodium Hydroxide*

Preparation

1. Grind 0.1 g of m-cresolsulfonphthalein in a mortar with 26.2 mL of 0.01 N sodium hydroxide.
2. Dilute to 250 mL with distilled water and mix.

ECR-1612

n,n-Dimethyl-p-phenylenediamine Sulfate Reagent

- n,n-Dimethyl-p-phenylenediamine Sulfate, 97%
minimum assay
Reagent Grade, ACS Specifications



Warning

POISON. Harmful if inhaled. Use an exhaust hood. **IRRITANT.** Avoid contact with eyes, skin, and clothing. Wear impervious gloves when handling.

- *Florisil*, 100- to 200-mesh
- *Sulfuric Acid*, H₂SO₄, Conc., 95 to 98%, sp gr 1.84, approx. 36 N
Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor.

Preparation

1. Measure and pour into a 250 mL beaker 89 mL of distilled water. Stir on a magnetic stirrer.
2. Carefully add 15 mL of concentrated sulfuric acid.

3. Add and dissolve 1.0 g n,n-dimethyl-p-phenylenediamine sulfate.
4. Add 5 g of Florisil and stir the mixture until all on material is absorbed.
5. Allow the adsorbant to settle and decant the supernatant solution.

ULM-0004/1

Ethylenedinitrilotetraacetic Acid (EDTA)

- Ethylenedinitrilotetraacetic Acid
Also: (Ethylenedinitrilo)tetraacetic Acid or Ethylenediaminetetraacetic Acid
Reagent Grade, ACS Specifications

Ethyl Acetate

- Ethyl acetate, $\text{CH}_3\text{COOCH}_2\text{CH}_3$
Spectro Grade, Reagent Grade, ACS Specifications



Warning

FLAMMABLE. Keep away from heat, sparks and flame. Avoid breathing vapor. Keep container closed. Use with adequate ventilation. Avoid prolonged or repeated contact with skin.

Absorbance Check

When ethyl acetate is used in a spectrophotometric method, check it for ultraviolet absorbance before it is used. If the absorbance of a 1 cm silica cell filled with ethyl acetate exceeds 0.150, measured against an air blank at 295 and 315 nm, the ethyl acetate is not suitable for use.

Preparation

Note: If large volumes of these reagents are needed, a large mixing container may be used. If a power stirrer is used, it must be air-driven or electrically driven (sparkless) to avoid the possibility of a spark igniting the flammable vapor.

Water-Saturated Ethyl Acetate

Note: Anhydrous ethyl acetate absorbs water very rapidly. Variations in its water content produce changes in volume when it is shaken with an aqueous sample. Since ethyl acetate cannot be assumed to be anhydrous, and since the water content is unimportant, provided it is always the same from one batch to another, it is recommended that it be saturated with water prior to use (3.3 mL of water will dissolve in 90 mL of anhydrous ethyl acetate).

1. Add 100 mL of distilled water to 900 mL of reagent (or spectro-reagent) grade ethyl acetate.
2. Mix well in a container large enough to allow for expansion.

3. Decant the water-saturated ethyl acetate or withdraw the lower (aqueous) layer by using a length of glass tubing attached to an aspirator pump.

ECN-2-1570C

ECP-2-407, ECP-2-2010A

ECR-440B

Extractant Solution

- Potassium Iodide*, KI
Reagent Grade, ACS Specifications
- Potassium Bromide*, KBr
Reagent Grade, ACS Specifications
- Potassium Dihydrogen Phosphate*, KH_2PO_4
Also: Potassium Phosphate, Monobasic
Reagent Grade, ACS Specifications

Preparation

1. Place a 1-litre volumetric flask containing 800 mL of distilled water on a magnetic stirrer.
2. Add and dissolve 1.0 g of potassium iodide, 20.0 g of potassium bromide, and 1.0 g of potassium dihydrogen phosphate.
3. Dilute to volume with distilled water; mix thoroughly.

ULM-0004/1

Ferric Ammonium Sulfate

- Ferric Ammonium Sulfate Dodecahydrate
 $[\text{Fe}(\text{NH}_4)(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$
Reagent Grade, ACS Specifications
Note: Observe precautionary information on product labels and on the Material Safety Data Sheets.
- Concentrated *Hydrochloric Acid*, HCl
Reagent Grade, ACS Specifications



Caution

Avoid contact with acid and fumes.

- Potassium Iodide*, KI
Reagent Grade, ACS Specifications
- Sodium Thiosulfate* Pentahydrate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$, standardized 0.1 N
Reagent Grade, ACS Specifications

Preparation

0.1 N Ferric Ammonium Sulfate Dodecahydrate

1. Add approximately 800 mL of distilled water to a 1-litre volumetric flask containing a Teflon stir bar.
2. Weigh 49 g of Ferric Ammonium Sulfate Dodecahydrate.

- Quantitatively transfer to the 1-litre volumetric flask while stirring.
- Remove stir bar and dilute to full volume with distilled water. Replace stir bar and stir the saturated solution thoroughly for approximately 5 minutes.
- Filter the saturated solution using a MILIPORE apparatus with HA 0.45 µm filter membrane, or equivalent.

Standardization

0.1 N Ferric Ammonium Sulfate Dodecahydrate (standardized to 4 decimal places)

- Pipet 15.0 mL of the 0.1 N Ferric Ammonium Sulfate Dodecahydrate solution to be standardized (4 decimal places) into a 250-mL beaker containing 50 mL of distilled water and a Teflon stir bar.
- While stirring, add 3 mL of Concentrated Hydrochloric Acid.
- Weigh 3.0 g ± 0.1 g of potassium iodide and Quantitatively transfer it to the 250-mL beaker. Stir to dissolve.
- Cover beaker with a watch glass and allow it to stand in the dark for 30 minutes.
- Add 50 mL of distilled water to the beaker and place on a magnetic stirrer.
- Immerse the Platinum indicator electrode (Beckman 39273 or equivalent) and double-junction reference electrode (Orion 90-02 or equivalent) into the solution.
- Titrate the sample with 0.1 N sodium thiosulfate (standardized to 4 decimal places) using a Mettler E536 Potentiograph, or equivalent, and a 20 mL buret assembly.

Horizontal Chart Span	500 mV
Titration Speed	15 min/100% volume
Vertical Chart Span	400 mm/100% volume
Titration Mode	mV/pH
Auto Control	Off
Auto Titration Stop	Off

- The titration end point is determined using a concentric arcs template.
- Repeat this procedure two more times
- Restandardize on a monthly basis.
- Calculation:

$$\text{Normality of Ferric Ammonium Sulfate Dodecahydrate} = \frac{(\text{vol Sodium Thiosulfate in mL})(\text{Normality of Sodium Thiosulfate})}{(15.0)}$$

Where:

$$15.0 = \text{Volume (in mL) of 0.1 N Ferric Ammonium Sulfate solution taken.}$$

Calculate the mean normality and standard deviation (s):

$$\text{Mean, } \bar{x} = \frac{\sum x}{n}$$

$$\text{Standard deviation, } s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

where: x = individual measurement
 n = the number of measurements
 \bar{x} = x - bar

From laboratory experience at Kodak, the standard deviation (1s) should be ≤ 0.0003. This should be determined in each laboratory.

ECN-2-ML-3255

Ferric Chloride

- Ferric Chloride, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

Reagent Grade, ACS Specifications

ULM-0004/1



Warning

IRRITANT. Use with adequate ventilation. An exhaust hood is recommended. Avoid contact with eyes, skin and clothing. Wear impervious gloves and eye protection when handling.

Ferric Nitrate



Caution

Acidified ferric nitrate is very corrosive to eyes, skin and metals.

- Ferric Nitrate, $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$

Reagent Grade, ACS Specifications

ECN-2-3263, ECN-2-ML-3260

- Nitric Acid, HNO_3 , Concentrated

Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Preparation

0.10 M Acidified Ferric Nitrate

- Add cautiously 40 mL of colorless, reagent-grade concentrated nitric acid to 400 mL of distilled water

contained in a 1-litre volumetric flask. If the acid is colored, bubble nitrogen or air through it, before diluting, until the brown color has entirely disappeared.

2. Dissolve 40.0 g of reagent-grade ferric nitrate, in the diluted acid; dilute to 1 litre with distilled water and mix.

ECR-125F
D94-0003/1

Ferriin Indicator

(Indicator for Iron)

Color change: Red to green

- 1,10-Phenanthroline Ferrous Sulfate Solution, ($C_{12}H_8N_2$)₃ FeSO₄ (0.025 M)
Also: Ferriin Indicator Solution, Ferrous Phenanthroline or Tris (1,10-Phenanthroline) Iron(II) Sulfate Solution

Reagent Grade, ACS Specifications

ECN-2-1125, ECN-2-3275-2, ECN-0003/1, ECN-0007/1, ECN-0024/1

ECP-2-407, ECP-2-1125A, ECP-2-2010A, ECP-2-2020, ECP-0003/1, ECP-0007/1, ECP-0026/1

ECR-125F, ECR-1113D, ECR-1125B

Ferrous Ammonium Sulfate

- Ferrous Ammonium Sulfate, 6-Hydrate, Fe(NH₄)₂(SO₄)₂•6H₂O
Also: Ferrous Ammonium Sulfate Hexahydrate, (FAS)

Reagent Grade, ACS Specifications

ECN-2-3275-2, ECN-0007/1

ECP-0007/1

- *Sulfuric Acid*, H₂SO₄

Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor.

Solution Strengths

0.25 N **ECN-0024/1**

ECP-0026/1

ECR-1125B

0.10 N **ECR-1113D**

0.001 N **ECN-2-ML-3255**

Preparation

0.25 N Ferrous Ammonium Sulfate

1. Add about 800 mL of distilled water to a 1-litre volumetric flask; stir on a magnetic stirrer.

2. Add and dissolve 98.00 g of ferrous ammonium sulfate, 6-hydrate.
3. Carefully add 20 mL of concentrated sulfuric acid.
4. Cool flask under running water.
5. Dilute to volume with distilled water; mix thoroughly.

0.10 N Ferrous Ammonium Sulfate

1. Add about 300 mL of distilled water to a 1-litre volumetric flask; stir on a magnetic stirrer.
2. Add and dissolve 39.20 g of ferrous ammonium sulfate, 6-hydrate.
3. Carefully add 25 mL of 7.0 N sulfuric acid.
4. Dilute to volume with distilled water; mix thoroughly.

0.001 N Ferrous Ammonium Sulfate

1. Place a magnetic stir bar and about 300 mL of distilled water to a 500 mL volumetric flask. Place on a magnetic stirrer.
2. Measure out 100 mL of 2.5 N sulfuric acid in a 100 mL graduated cylinder and add to the flask, while stirring.
3. Weigh 0.2 g of ferrous ammonium sulfate and transfer to the volumetric flask.
4. Dilute to volume with distilled water; mix thoroughly.

Note: This reagent is stable for four weeks.

Assay of FAS

Procedure Using Potassium Dichromate

See Reagent Preparation for:

- *Potassium Dichromate*, K₂Cr₂O₇, **NIST Oxidimetric Primary Standard**, SRM-136e (or subsequent lot of SRM-136)
- *Ferriin Indicator*
- *Sulfuric Acid*, concentrated
- Ferrous Ammonium Sulfate, 6-Hydrate (FAS)
 1. Weigh 14 g of FAS to the nearest 0.1 mg and record.
 2. Quantitatively transfer to a 200 mL volumetric flask with distilled water and swirl the flask to dissolve the FAS.
 3. Dilute the solution to the mark with distilled water. Invert the flask, 6 to 10 times, to mix thoroughly.
 4. Dry the potassium dichromate (SRM-136e) for 2 hours at 110°C and cool to room temperature. Weigh 1 g to the nearest 0.1 mg and record.
 5. Quantitatively transfer to a 100 mL volumetric flask containing 50 mL of distilled water. Swirl the flask to dissolve the potassium dichromate.
 6. Dilute the solution to the mark with distilled water. Invert the flask, 6 to 10 times, to mix thoroughly.

- Fill a 50 mL buret with the FAS solution (Step 3, above).
- Add 20 mL concentrated sulfuric acid to a 150 mL beaker containing 25 mL of distilled water and a magnetic stirring bar.



Caution

Hot acid solution is formed.

- Pipet 15.0 mL of the potassium dichromate solution into the beaker.
- Add 2 drops of ferroin indicator to the beaker and place the beaker on a magnetic stirrer.
- Slowly add the FAS solution from the buret to the beaker. The solution will change colors as the titrant is added, going from a *green-blue* color to a *reddish-brown* color. The point that produced the first *reddish-brown* color change is the end point.
- Record the volume of FAS titrant consumed to the nearest 0.05 mL at the end point.
- Repeat Steps 7 to 12, two more times.

Calculations:

N of Potassium Dichromate

$$N \text{ K}_2\text{Cr}_2\text{O}_7 = \frac{(g, \text{K}_2\text{Cr}_2\text{O}_7)(1000)}{(100 \text{ mL})(294.2/6)} = 0.2039(g, \text{K}_2\text{Cr}_2\text{O}_7)$$

Where:

- $g, \text{K}_2\text{Cr}_2\text{O}_7$ = recorded weight from Step 4
- 1000 = factor to convert eq wt to meq wt
- 100 mL = volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution
- 294.2 = the molecular wt of $\text{K}_2\text{Cr}_2\text{O}_7$
- 6 = factor to convert g-mole wt to eq wt of $\text{K}_2\text{Cr}_2\text{O}_7$

N of Ferrous Ammonium Sulfate, 6-Hydrate (FAS)

$$N \text{ FAS} = \frac{(N \text{ K}_2\text{Cr}_2\text{O}_7)(\text{mL K}_2\text{Cr}_2\text{O}_7)}{(\text{mL FAS})} = \frac{15.0(N \text{ K}_2\text{Cr}_2\text{O}_7)}{(\text{mL FAS})}$$

Where:

- $N \text{ K}_2\text{Cr}_2\text{O}_7$ = normality (meq/mL) calculated, above
- mL $\text{K}_2\text{Cr}_2\text{O}_7$ = 15.0 mL volume of $\text{K}_2\text{Cr}_2\text{O}_7$ solution taken
- mL FAS = average* volume of the three titrations required to reach the end point color change.

* The average volume for the three titrations should not have a standard deviation (1s) greater than 0.050 mL.

g/L, Iron(II) in Ferrous Ammonium Sulfate

$$g/L, \text{Iron(II)}_{\text{actual}} = 55.85 (N \text{ FAS})$$

Where:

$$55.85 = \text{eq wt (g-eq) of Iron(II) in FAS}$$

Theoretical wt of Iron(II) in Solution

$$g/L, \text{Iron(II)}_{\text{theoretical}} = \frac{(g, \text{FAS})(55.85)(1000)}{(200 \text{ mL})(391.85)} = 0.7139(g, \text{FAS})$$

Where:

- g, FAS = g-weight $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ recorded in Step 1
- 55.85 = atomic weight of iron (g-mole)
- 1000 mL = factor to convert mL to L
- 200 mL = dilution vol of wt of $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$
- 391.85 = mole wt (g-mole) of ferrous ammonium sulfate, 6-hydrate.

Assay Percentage

$$\% \text{ Iron(II)} = \frac{[g/L, \text{Iron(II)}_{\text{actual}}](100)}{[g/L, \text{Iron(II)}_{\text{theoretical}}]}$$

ECN-0007/1

ECN-2-3275-2

ECP-0007/1

Alternate Procedure Using Potassium Permanganate

See Reagent Preparation for:

- Potassium Permanganate, KMnO_4 ,
- Reagent Water
- Sulfuric Acid, concentrated
- Ferrous Ammonium Sulfate, 6-Hydrate (FAS)

Note: This procedure is based on the method in Reagent Chemicals, 8th Edition, American Chemical Society, 1993.

Note: This procedure should be repeated in triplicate with the average of the three results used as the assay value.

- Accurately weigh 1.6 grams of sample to the nearest milligram and dissolve in a mixture of 100 mL reagent water and 3 mL of concentrated sulfuric acid contained in a 250 mL Erlenmeyer flask.



Caution

Always add acid to water and not water to acid.

- Titrate while stirring with standardized 0.1 N potassium permanganate (KMnO_4) from a 50 mL buret to a permanent faint pink endpoint that lasts for at least 15 seconds.
- Repeat steps 1 and 2 without any sample. This is the blank.

Calculations

$$\frac{\% \text{ (wt./wt.) ferrous ammonium sulfate, hexahydrate} = (\text{mL KMnO}_4 \text{ sample} - \text{mL KMnO}_4 \text{ blank}) \times \text{N KMnO}_4 \times 0.3921 \times 100}{\text{sample size in grams}}$$

ECN-0007/1

ECN-2-3275-2

ECP-0007/1

Ferrous Chloride

- Ferrous Chloride, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$

Reagent Grade, ACS Specifications



Warning

IRRITANT. Avoid eye and skin a eye protection and impervious gloves when handling.

Ferrous/Ferric Reagent

- *Ferrous Chloride*, $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$

Reagent Grade, ACS Specifications



Caution

IRRITANT. Avoid eye and skin a eye protection and impervious gloves when handling.

- *Ferric Chloride*, $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$

Reagent Grade, ACS Specifications



Warning

IRRITANT. Use with adequate ventilation. An exhaust hood is recommended. Avoid contact with eyes, skin and clothing. Wear impervious gloves and eye protection when handling.

- *Hydrochloric Acid*, HCl, Conc., approx. 12 N

Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Preparation

1. Place a 50 mL beaker containing 20 mL of distilled water on a magnetic stirrer.
2. Add and dissolve 0.75 g of ferrous chloride, and 0.75 g of ferric chloride, and 3 mL of concentrated hydrochloric acid.
3. Dilute to 30 mL with distilled water and mix.

ECN-0025-1

1122B

Florisil

- Florisil®, 100/200 mesh

Analytical Grade

Foamex

- Foamex®, anti-foaming compound

Analytical Grade

ECR-617B, ECR-1612

Formaldehyde

- Formaldehyde, HCHO, 37.5% Solution by weight

Also: Formalin

Reagent Grade, ACS Specifications



Warning

Causes severe eye burns. Harmful if inhaled. May be fatal or cause blindness if swallowed. Causes skin irritation and may cause allergic skin or respiratory reaction.

COMBUSTABLE. Do not get in eyes, on skin, or on clothing. Use only with adequate ventilation. Do not breathe vapor. Keep away from heat and flame.

Note: The Occupational Safety and Health Administration (OSHA) has determined that formaldehyde is a possible cancer hazard based on animal data. OSHA Standard 29 CFR 1910.1048 should be consulted for specific details regarding respiratory, skin and eye protection against formaldehyde. The Standard also provides for establishing regulated areas, respiratory protection programs, housekeeping, medical surveillance, record keeping, and employee information and training when necessary.

- 0.10 N *Sodium Hydroxide*
- 0.10 N *Sulfuric Acid*

Preparation

Formaldehyde, 37.5%, pH 3.9

1. Pour 1 litre of reagent-grade formaldehyde, 37.5%, into a 1500 mL beaker.
2. Place the beaker on a magnetic stirrer.
3. Adjust the pH to pH 3.9 with a small amount of either 0.10 N sulfuric acid or 0.10 N sodium hydroxide. Use a pH meter calibrated according to Method ULM-191-2, or subsequent revisions.

ECR-617B

Formaldehyde, 6%, pH 3.9

1. Add, using a graduated cylinder, 150 mL of reagent grade formaldehyde, 37.5%, to a 1500-mL beaker containing about 800 mL of reagent water.
2. Place the beaker on a magnetic stirrer.
3. Adjust to pH 3.9 with a small amount of either 0.1 N sulfuric acid or 0.1 N sodium hydroxide. Use a pH meter calibrated according to Method *ULM-191-2*, or subsequent revisions.
4. Transfer the adjusted solution to a 1-litre volumetric flask. Dilute to volume with reagent water and mix.

ECN-0002/1

ECP-0002/1

Formaldehyde, 6%, pH 4.3

1. Add, using a graduated cylinder, 150 mL of reagent grade formaldehyde, 37.5%, to a 1500-mL beaker containing about 800 mL of reagent water.
2. Place the beaker on a magnetic stirrer.
3. Adjust to pH 4.3 with a small amount of either 0.1 N sulfuric acid or 0.1 N sodium hydroxide. Use a pH meter calibrated according to Method *ULM-191-2*, or subsequent revisions.
4. Transfer the adjusted solution to a 1-litre volumetric flask. Dilute to volume with reagent water and mix.

Gelatin

(4 g/L)

- Gelatin

Reagent Grade, ACS Specifications

Preparation

1. Weigh 4 g of gelatin. Place in a 1-litre volumetric flask.
2. Add 20 mL of distilled water to the volumetric flask. Soak for 5 minutes. The solution may be warmed to aid in dissolving the gelatin.
3. Dilute to 1 litre with distilled water.

Note: This solution is stable for 5 days.

ECN-0023-01

1208E

Hydrochloric Acid

- Hydrochloric Acid, HCl, Conc., approx. 12 N
Reagent Grade, ACS Specifications

1122B

ECN-2-3263, ECN-2-ML-3260, ECN-0025-1



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

- 0.1 N Sodium Hydroxide

Solution Strengths

4.0 N **Calcium Chloride/Calcium Hydroxide Buffer**
Ferric Nitrate

3.0 N **Acid-Alcohol Cleaning Solution**
ECN-0019-01
ECP-0019-01
ECR-754A

Note: If possible, use a fresh unopened bottle of concentrated acid. The values given in the table below are for 12.0 N hydrochloric acid and are intended to give slight over concentration which can be adjusted easily by addition of distilled water.

Procedure

1. Add approximately 400 mL of distilled water to a 1-litre volumetric flask.
2. Add very cautiously, in an exhaust hood, the required amount of concentrated hydrochloric acid (see the below) from a graduated cylinder.

Normality HCl Desired	Add Concentrated 12 N HCl, mL/L
4.0	333
3.0	249

3. Allow the solution to reach room temperature. Dilute to volume with distilled water and mix.

Hydrogen Peroxide

- 30% Hydrogen Peroxide, H₂O₂

Reagent Grade, ACS Specifications



Warning

STRONG OXIDIZER. Contact with other materials may cause fire. Refrigeration required. Recommended refrigeration temperature: 0 to 5°C. Causes severe eye and skin burns. Avoid contact with eyes, skin, and clothing. Wear eye protection and impervious gloves.

ECP-0022/1

Hydroquinone

- Hydroquinone, $C_6H_4-1,4-(OH)_2$
Reagent or Photographic Grade



Warning

Causes eye and skin irritation. May cause allergic skin reaction. Avoid contact with eyes, skin, and clothing.

Hydroxylamine Sulfate

- Hydroxylamine Sulfate, $(NH_2OH)_2 \cdot H_2SO_4$, 98% Assay, minimum
Reagent Grade, ACS Specifications

Preparation

2.5 M Hydroxylamine Sulfate

- Add approximately 800 mL of distilled water to a 1-litre volumetric flask.
- Weigh 410 g of hydroxylamine sulfate and transfer to the 1-litre volumetric flask.
- Mix the solution until the hydroxylamine sulfate dissolves.
- Dilute the contents of the volumetric flask to volume with distilled water and mix.

ECR-1603E

Iodine

- Iodine, I_2
Reagent Grade, ACS Specifications



Warning

CAUSES BURNS. Avoid contact with eyes, skin, and clothing. In case of contact, flush with water. HARMFUL IF INHALED. Use with adequate ventilation.

- Potassium Iodide, KI
Reagent Grade, ACS Specifications

Preparation

0.1 N Iodine

- In a 1-litre flask, dissolve 40 g of potassium iodide in 25 mL distilled water.
- Add and dissolve 12.7 g of iodine.
- Dilute to 1 litre with distilled water.
- Store in a Pyrex stoppered bottle in the *dark*.

0.1 N Iodine (standardized to 4 decimal places)

The 0.10 N iodine prepared above may be standardized using Procedure A or B described below. As an alternative, manufacturer-standardized iodine may be purchased and prepared according to the manufacturer's instructions. Its

normality should then be checked using Procedure A or B described below. The standardized 0.1 N iodine should be stored in a Pyrex stoppered bottle in the *dark*.

ECN-0002/1

ECP-0002/1

Standardization

Procedure A—Using standardized 0.1 N Sodium Thiosulfate

See Reagent Preparation for:

- 0.1 N Sodium Thiosulfate
(standardized to 4 decimal places)
- Starch Indicator
 - Pipet (wipe the pipet before leveling) 20.0 mL of *freshly standardized* 0.1 N sodium thiosulfate into a 125 mL conical flask containing 50 mL of distilled water.
 - Add 2 mL of starch indicator from a tip-up pipet.
 - Using a 25 mL buret, titrate with the iodine being standardized. The end point is indicated by the appearance of the *first blue color*. Record the volume of iodine used for the titration.
 - Calculation:

$$N \text{ Iodine} = \frac{(N \text{ Na}_2\text{S}_2\text{O}_3)(\text{mL Na}_2\text{S}_2\text{O}_3)}{\text{mL iodine titrated}} = \frac{(N \text{ Na}_2\text{S}_2\text{O}_3)(20)}{\text{mL iodine}}$$

- Repeat the standardization two more times.
- Average the three standardizations.

Procedure B—Using Sodium Arsenite Reagent

See Reagent Preparation for:

- Sodium Bicarbonate
- Starch Indicator
- Sodium Arsenite Reagent
 - Add approximately 50 mL of distilled water to a 250 mL conical flask.
 - Add and dissolve 2.0 g of reagent-grade sodium bicarbonate.
 - Add 2 mL of starch indicator.
 - Pipet (wipe the pipet before leveling) 100.0 mL of sodium arsenite reagent into the flask.
 - Using a 25 mL buret, titrate with the iodine being standardized. The end point is indicated by the appearance of the *first blue color*. Record the volume of titrant to the nearest 0.01 mL.
 - Calculation:

$$N \text{ Iodine} = \frac{\left[\begin{array}{c} \text{aliquot} \\ \text{of} \\ \text{treated} \\ \text{sample} \end{array} \right] \left[\begin{array}{c} \text{g As}_2\text{O}_3 \\ \text{weighed for} \\ \text{sodium} \\ \text{arsenite} \\ \text{reagent} \end{array} \right] (1000)}{(\text{eq wt As}_2\text{O}_3)(\text{mL I}_2 \text{ titrated})} =$$

$$\frac{100(\text{g As}_2\text{O}_3)(1000)}{197.82(\text{mL I}_2)} = \frac{2.022 (\text{g As}_2\text{O}_3)}{\text{mL I}_2}$$

7. Repeat the standardization two more times.
8. Average the three standardizations.

Storage

Store the standardized 0.1 N iodine in a Pyrex stoppered bottle in the *dark*.

Iodine/Chloride Solution

- *Potassium Iodide*
Reagent Grade, ACS Specifications
- *Potassium Iodate*, KIO₃
Reagent Grade, ACS Specifications
- *Hydrochloric Acid*, HCl, Conc., approx. 12 N



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Preparation

1. Weigh 0.28 ± 0.01 g of Potassium Iodide.
2. Weigh 0.178 ± 0.001 g of Potassium Iodate.
3. Transfer both the Potassium Iodide and the Potassium Iodate to a beaker containing 250 mL of concentrated Hydrochloric Acid. Stir solution until dissolved.



Caution

AVOID CONTACT WITH ACID AND FUMES. Use an exhaust hood. Wear eye protection and impervious gloves.

4. Store in a bottle that will protect the solution from light.
5. Discard Iodine/Chloride solution after 24 hours

Iron

- Iron, wire, 0.5 mm diameter, 99.99 percent pure
Reagent Grade, ACS Specifications

ECN-0006/1

ECP-0006/1

- FerroVer® Iron Reagent Powder Pillow

ECN-2-ML-3280, ECN-2-UL-3280

Isopropyl Alcohol

- Isopropyl Alcohol, (CH₃)₂CHOH
Reagent Grade, ACS Specifications

ULM-0001/1



Warning

FLAMMABLE. IRRITANT. Avoid contact with eyes and skin. Wear impervious gloves and eye protection when using. Keep away from heat, sparks and flame.

Mercuric Iodide

- Mercuric Iodide, HgI₂
Reagent Grade, ACS Specifications



Warning

POISONOUS. Avoid inhalation. Use with adequate ventilation. Avoid contact with eyes and skin. Wear protective gloves when using. Photographic contaminant.

Methanol

- Methanol, CH₃OH
Also: Methyl Alcohol
Practical Grade
Spectro Grade, Reagent Grade

ECN-2-1570C

ECP-2-407, ECP-2-936A



Warning

POISON. May be fatal or cause blindness if swallowed. Can not be made non-poisonous. Harmful if inhaled. Harmful if absorbed through the skin. Causes skin and eye irritation. Avoid contact with eyes, skin and clothing. Wash thoroughly after handling. Use with adequate ventilation. FLAMMABLE. Keep away from heat, sparks, and flame. Store in original tightly closed container.

Methyl Alcohol Containing Thymol Blue

(formerly called Neutralized CH₃OH)

- Methyl Alcohol, CH₃OH
Also: *Methanol*
Practical Grade



Warning

POISON. Can be fatal or cause blindness if swallowed. Cannot be made non-poisonous. Harmful if inhaled. Use with adequate ventilation. **FLAMMABLE.** Keep away from heat, sparks, and flame.

- *Thymol Blue*
Reagent Grade
- 0.25 N *Sodium Hydroxide*
- 0.10 N *Sulfuric Acid*

Preparation

1. Add 25 mL of thymol blue indicator to 1 litre of practical grade methyl alcohol.
2.
 - a. If the solution is yellow, titrate with 0.25 N sodium hydroxide to the first *green* color.
 - b. If the solution is green or blue, titrate with 0.10 N sulfuric acid to the first *yellow* color.

ECR-1603E

4-Methyl-2-Pentanone

- 4-Methyl-2-pentanone, $\text{CH}_3\text{COCH}_2\text{CH}(\text{CH}_3)_2$
Reagent Grade, ACS Specifications

ECN-2-1570C



Warning

FLAMMABLE. Harmful if inhaled. Avoid breathing vapor. Use with adequate ventilation. Causes eye and skin irritation. Avoid contact with eyes, skin and clothing. Keep away from heat, sparks and flame. Do not allow to evaporate to near dryness. May form explosive peroxides. Keep container closed. Store in a cool place.

Methyl Red Indicator

Color change: Red to yellow pH4.4 to 6.2.

- Methyl Red, p-dimethylaminoazobenzene-o-carboxylic acid
Reagent Grade
- 0.10 N *Sodium Hydroxide*

Preparation

1. Grind 1.0 g of methyl red in a mortar with 37 mL of 0.10 N sodium hydroxide.
2. Dilute to 1 litre with distilled water and mix.

Molybdenum Reagent

- *Ammonium Molybdate*, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$
Reagent Grade, ACS Specifications



Caution

Use only with adequate ventilation. Use of a hood is preferable.

- *Phosphoric Acid*, H_3PO_4 , Conc., min. Assay 85%
Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid to water. Corrosive to skin, metals, and clothing. Avoid eye and skin contact with liquid and vapor. Use in an exhaust hood.

Preparation

1. Add 25.0 g of ammonium molybdate to a 1-litre volumetric flask.
2. Add about 150 mL of distilled water, and stir on a magnetic stirrer to produce a slurry.
3. Continue stirring and add 250 mL of concentrated phosphoric acid.
4. Dilute to volume with distilled water.
5. Allow the solution to cool to room temperature and again dilute to 1 litre with distilled water.

ECR-0001-1

Note: This reagent must be prepared fresh weekly.

Nitric Acid

- Nitric Acid, HNO_3 , Conc., 69 to 71%, approx. sp gr 1.42, approx. 16 N
Reagent Grade, ACS Specifications

ECN-0006/1

ECP-0006/1



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Solution Strengths

5.0 N	ECN-0006/1 ECP-0006/1
0.5 N	ECN-0006/1 ECP-0006/1
0.1 N	ECN-2-3263, ECN-2-ML-3260

Preparation

0.10 N Nitric Acid

1. Add approximately 700 mL of distilled water to a 1-litre volumetric flask. Add a magnetic stir bar and stir.
2. Using a 10 mL graduated cylinder, carefully add 6.6 mL of concentrated nitric acid. Stir to mix.
3. Cool the solution to room temperature. Remove the magnetic stir bar.
4. Dilute to volume with distilled water. Invert 6 to 10 times to mix.

0.5 N Nitric Acid

1. Add approximately 700 mL of distilled water to a 1-litre volumetric flask. Add a magnetic stir bar and stir.
2. Using a 10 mL graduated cylinder, carefully add 33 mL of concentrated nitric acid. Stir to mix.
3. Cool the solution to room temperature. Remove the magnetic stir bar.
4. Dilute to volume with distilled water. Invert 6 to 10 times to mix.

5.0 N Nitric Acid

1. Add approximately 500 mL of distilled water to a 1-litre volumetric flask. Add a magnetic stir bar and stir.
2. Using a 10 mL graduated cylinder, carefully add 333 mL of concentrated nitric acid. Stir to mix.
3. Cool the solution to room temperature. Remove the magnetic stir bar.
4. Dilute to volume with distilled water. Invert 6 to 10 times to mix.

Nitrogen Gas

- Nitrogen Gas, N₂

Reagent Grade, ACS Specifications

ECN-2-3275-2

ECR-1603E

pH 3.63 Tartrate Low pH Control Buffer

- pH 3.63 Tartrate Low pH Control Buffer

Reagent Grade, ACS Specifications

ECN-0019-01

ECP-0019-01, ECP-0020-01

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 4 Phthalate Calibrating Buffer

- pH 4 Phthalate Calibrating Buffer

Reagent Grade, ACS Specifications

ECN-0019-01

ECP-0019-01, ECP-0020-01

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 4 Phthalate Reference Buffer

- pH 4 Phthalate Reference Buffer

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 7 Equimolar Phosphate Calibrating Buffer

- pH 7 Equimolar Phosphate Calibrating Buffer

Reagent Grade, ACS Specifications

ECN-0019-01

ECP-0019-01, ECP-0020-01

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 7 Equimolar Phosphate Reference Buffer

- pH 7 Equimolar Phosphate Reference Buffer

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 9 Borate Reference Buffer

- pH 9 Borate Reference Buffer

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 10 Carbonate Calibrating Buffer

- pH 10 Carbonate Calibrating Buffer

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

pH 11.1 Sodium Carbonate Buffer

- Sodium Carbonate, Na₂CO₃

Reagent Grade, ACS Specifications

Preparation

1. Add 85.5 g of reagent quality sodium carbonate, Na₂CO₃, in distilled water and dilute to 1 litre.

ECP-2-2020

pH 11.43 Phosphate High pH Control Buffer

- pH 11.43 Phosphate High pH Control Buffer

ULM-191-2

For preparation instructions, see Method *ULM-191-2, pH Measurement of Photographic Processing Solutions*.

1,10-Phenanthroline/Sodium Acetate Reagent

- *Methanol*, CH₃OH

Spectro Grade, Reagent Grade



Warning

POISON. Can be fatal or cause blindness if swallowed. Cannot be made non-poisonous. Harmful if inhaled. Use with adequate ventilation. **FLAMMABLE.** Keep away from heat, sparks, and flame.

- 1,10-Phenanthroline 1-Hydrate
Reagent Grade
- *Sodium Acetate*, NaC₂H₃O₂, Anhydrous
Reagent Grade, ACS Specifications
- *Glacial Acetic Acid*
Reagent Grade, ACS Specifications



Warning

CAUSES SEVERE EYE AND SKIN BURNS. Do not get in eyes, on skin, on clothing. Wear eye protection and impervious gloves when handling. Vapor extremely irritating. Avoid breathing vapor. Use with adequate ventilation. An exhaust hood is recommended. **COMBUSTIBLE.** Keep away from heat and flame.

Preparation

1. Add, from a graduated cylinder, 500 mL of methanol to a 1-litre volumetric flask.
2. Add 5 g of 1,10-phenanthroline to the volumetric flask.
3. Wash down the sides of the flask with 100 mL of methanol.
4. Stir on a magnetic stirrer for 5 minutes.
5. Add and dissolve 11.5 g of sodium acetate.
6. Add, from a graduated cylinder, 3 to 4 mL of glacial acetic acid.
7. Dilute to volume with distilled water and mix thoroughly.

ECN-2-3275-2

Phenolphthalein Indicator

Color change: Colorless to pink pH 8.0 to pH 10.0

- Phenolphthalein

Reagent Grade, ACS Specifications

- *Methanol*

Practical or Reagent Grade



Warning

POISON: Can be fatal or cause blindness if swallowed. Can not be made non-poisonous. Harmful if inhaled. Use with adequate ventilation. **FLAMMABLE.** Keep away from heat, sparks, and flame.

Preparation

1. Dissolve 0.5 g phenolphthalein in 500 mL of methanol.
2. Dilute to 1 litre with distilled water and mix.

ECN-0002/1

ECP-0002/1

Phosphoric Acid

- Phosphoric Acid, H₃PO₄, Concentrated, Assay 85% minimum.

Reagent Grade, ACS Specifications



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid to water. Corrosive to skin, metals, and clothing. Avoid eye and skin contact with liquid and vapor. Use in an exhaust hood.

Polystyrene Sulfonate Solution

- Polystyrene Sulfonate, Na MW 70,000



Caution

IRRITANT. Avoid eye and skin contact.

Preparation

0.1% Polystyrene Sulfonate Solution

1. Place a 1-litre volumetric flask containing about 800 mL of distilled water on a magnetic stirrer.
2. Add and dissolve 1.0 g of polystyrene sulfonate, sodium salt.
3. Dilute to volume with distilled water and mix thoroughly.

ECN-0001/1

ECP-2-2010A, ECP-0001/1

ECR-125F

Potassium Biiodate

(2.40 g/L)

- Potassium Biiodate, $\text{KH}(\text{IO}_3)_2$

GFS Certified No. 76 (NIST Working Standard), GFS Chemicals or equivalent

Preparation

Note: For the sodium thiosulfate standardization, it is preferable to make three smaller weighings of dried potassium biiodate and titrate them directly, rather than making a single weighing to prepare the 2.40 g/L solution and taking three aliquots. However, for labs that do not have the capability of weighing to ± 0.1 mg, the 2.40 g/L solution preparation below is acceptable.

1. Weigh 2.4 g (to the nearest 0.1 mg) of GFS No. 76 potassium biiodate, that has been dried for 2 hours in an oven at 105°C and cooled to room temperature in a desiccator (the drying temperature should not exceed 120°C or degradation of the material may occur).
2. Quantitatively transfer the weighed material to a 1-litre volumetric flask containing about 500 mL of distilled water and swirl to dissolve. Dilute to volume with distilled water.

Note: The solution is stable for 3 months.

Potassium Bromide

- Potassium Bromide, KBr

Reagent Grade, ACS Specifications

Potassium Chloride

- Potassium Chloride, KCl

NIST Primary Standard SRM-999
(or subsequent lot of SRM-999)

Preparation

3.5 N or 3.5 M Potassium Chloride

1. Add 261 g of potassium chloride, to a 2-litre beaker containing 400 mL of distilled or demineralized water.
2. Dissolve the salt, heating if necessary.
3. Transfer the solution to a 1-litre volumetric flask, fill the flask to the mark with distilled or demineralized water and mix thoroughly.

Potassium Dichromate

- Potassium Dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$

NIST Oxidimetric Primary Standard SRM-136e
(or subsequent lot of SR-136)

ECN-2-3275-2, ECN-0007/1

ECP-0007/1

Technical Grade

ULM-0005/1



Warning

POISON. May be fatal if swallowed. Causes severe skin and eye burns. Suspected cancer hazard. May cause kidney damage. Harmful if inhaled or absorbed through the skin. Dust irritating to eyes and respiratory tract. May cause allergic skin reaction.

STRONG OXIDIZER. Contact with other material can cause fire. Do not breathe dust. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. Use with adequate ventilation. Keep from contact with clothing and combustible materials. Remove and wash contaminated clothing promptly. Before using, read Material Safety Data Sheet.

Potassium Dihydrogen Phosphate

- Potassium Dihydrogen Phosphate, KH_2PO_4

Also: Potassium Phosphate, Monobasic

Reagent Grade, ACS Specifications

Preparation

10% Solution

1. Weigh out 13.6 g potassium dihydrogen phosphate.
2. Transfer to 1-litre volumetric flask containing 800 mL of distilled water.
3. After dissolution, dilute to the mark and invert the stoppered flask 6 to 8 times for complete mixing.

Potassium Ferricyanide

- Potassium Ferricyanide, $\text{K}_3\text{Fe}(\text{CN})_6$

Reagent Grade, ACS Specifications



Caution

Contact with acid, under certain conditions, can liberate poisonous hydrogen cyanide gas. Use in a well ventilated area when mixing with an acid.

Note: All pipets and volumetric glassware should be Class A as defined by the National Institute of Standards and Technology (NIST).

Preparation

Standardized 0.3 N Potassium Ferricyanide

Standardize this solution in accordance with the standardization instructions.

1. Add 500 mL of distilled water to a 1-litre volumetric flask containing a Teflon stir bar.
2. While stirring, add 98.78 ± 0.001 g of potassium ferricyanide and dissolve completely.
3. Dilute to volume with distilled water and mix.

Note: This solution is stable for two weeks when stored in a stoppered bottle in a dark place.

Standardized 0.03 N Potassium Ferricyanide

1. Pipet 50.0 mL of 0.3 N potassium ferricyanide into a 500-mL volumetric flask containing approximately 300 mL distilled water and mix well.
2. Dilute to volume with distilled water and mix.
3. Calculation of exact normality:

$$N \text{ K}_3\text{Fe}(\text{CN})_6 = \frac{\text{Mean N of 0.3000 N K}_3\text{Fe}(\text{CN})_6}{10.0 \text{ mL}}$$

Note: This solution should be prepared fresh daily from the standardized 0.3 N solution.

0.1 M Potassium Ferricyanide

1. Add approximately 800 mL distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
2. Add 33 ± 0.01 g potassium ferricyanide to the 1-litre volumetric flask and stir to dissolve.
3. Dilute to volume with distilled water and mix well.

1209

0.2 M Potassium Ferricyanide

1. Add approximately 800 mL distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
2. Add 66 ± 0.01 g potassium ferricyanide to the 1-litre volumetric flask and stir to dissolve.
3. Dilute to volume with distilled water and mix well.

1209

40 g/L Potassium Ferricyanide

1. Add 800 mL distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
2. Add 40 ± 0.01 g potassium ferricyanide to the volumetric flask and stir to dissolve.
3. Dilute to volume with distilled water and mix well.

Standardization of 0.3 N Potassium Ferricyanide

Reagents

- Potassium Iodide, KI, 0.6 M
- Sulfuric Acid, H_2SO_4 , 7 N
- Zinc Sulfate, ZnSO_4 , 50 g/L
- Sodium Thiosulfate, $\text{Na}_2\text{S}_2\text{O}_3$, 0.1 N (standardized to four decimals)

Apparatus

- Metrohm E536 Potentiograph
- 50-mL buret for the Potentiograph

- Electrodes

Platinum inlay (Beckman 39273 or equivalent)

Silver/silver chloride double-junction reference with outer filling of 10% potassium nitrate (Orion 900200 or equivalent)

Procedure

1. Pipet 10.0 mL of the 0.3 N potassium ferricyanide to be standardized into a 250-mL beaker containing a Teflon stirring bar and 100 mL of distilled water.
2. While stirring, add 25 mL of 0.6 M potassium iodide, 25 mL of 7 N sulfuric acid, and 25 mL of 50 g/L zinc sulfate. Immediately titrate with standardized 0.1 N sodium thiosulfate using the following parameters with the E536 Potentiograph:

Rate:	15 min/100% vol
Auto control:	OFF
Mode:	mV/pH
Range:	500 mV
Buret size:	50 mL
Chart speed:	400 mm/100% vol

3. Determine the mL of 0.1 N sodium thiosulfate at the end point using concentric arcs (see Method *ULM-0003-01* or subsequent edition).
4. Run the standardization in triplicate
5. Calculation:

$$N \text{ Potassium Ferricyanide} = \frac{\text{mL Thiosulfate} \times N \text{ Thiosulfate}}{10.0 \text{ mL}}$$

Calculate the mean normality and standard deviation:

$$\text{Mean, } \bar{x} = \frac{\sum x}{n}$$

$$\text{Standard deviation, } s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

where: x = individual measurement
 n = the number of measurements
 \bar{x} = x - bar

From laboratory experience at Kodak, the standard deviation (1s) should be ≤ 0.0003 . This should be determined in each laboratory.

Potassium Hydrogen Phthalate

- Potassium Hydrogen Phthalate, $\text{C}_6\text{H}_4\text{COOHCOOK}$, Assay 99.95 to 100.05%
Also: Phthalic Acid Monopotassium Salt, Potassium Acid Phthalate

Reagent Grade, ACS Specifications

NIST **Ion Activity/pH Standard** SRM-185f
(or subsequent lot of SRM-185)

NIST **Acid/Base Primary Standard** SRM-84j
(or subsequent lot of SRM-84)

Note: Before using these standard materials, dry in an oven at 110°C for two hours, cool and store in a desiccator over Drierite.

Potassium Hydrogen Phthalate Buffer (0.05 M)

Please refer to ULM 191-2

for instructions on preparing buffers for pH Measurement of Photographic Processing Solutions.

Potassium Hydroxide

- Potassium Hydroxide, KOH, Pellets
Reagent Grade, ACS Specifications



Warning

CORROSIVE. Causes severe eye and skin burns. Avoid contact with eyes, skin or clothing. Wear eye protection and impervious gloves. Do not weigh in an aluminum dish.

Note: Potassium hydroxide pellets are deliquescent. The supply container should only be open long enough to weigh the required amount of reagent. Keep the container tightly closed at other times.

Potassium Iodate

- Potassium Iodate, KIO_3
Reagent Grade, ACS Specifications

Preparation

0.1 N Potassium Iodate (standardized to 4 decimal places)

- Weigh 3.567 g (to the nearest 0.1 mg) of reagent-grade potassium iodate.
- Quantitatively transfer the potassium iodate to a 1-litre volumetric flask containing about 500 mL of distilled water.
- Dissolve and dilute to volume with distilled water.
- Standardize the solution using the procedure below.

ECN-2-1305A, ECN-1315, ECN-2-1340, ECN-0002/1
ECP-2-1305C, ECP-0002/1, ECP-0025/01
ECR-1303, ECR-1305L, ECR-1340A, ECR-1803G
D94-0002/1

Standardization

See Reagent Preparation for:

- 7.0 N Sulfuric Acid
- 0.60 M Potassium Iodide, KI
- 0.1 N Sodium Thiosulfate
(standardized to 4 decimal places)
- Starch Indicator

Procedure

- Pipet (wipe the pipet before leveling) 20.0 mL of the 0.1 N potassium iodate solution to be standardized into a 125 mL conical flask.
- Add 10 mL of 7.0 N sulfuric acid from a tip-up pipet.
- Add 15 mL of 0.60 M potassium iodide from a tip-up pipet.
- Titrate the liberated iodine with standardized 0.1 N sodium thiosulfate, using a 25 mL buret.
- Titrate the solution to a *light yellow color*, add 5 mL of starch indicator from a tip-up pipet, and continue until the *blue color* is just discharged.
- Repeat Steps 1 through 5, on two more portions of the potassium iodate being standardized and apply the average volume of sodium thiosulfate in the calculation below.
- Calculation:

$$\text{N of KIO}_3 = \frac{(\text{N Na}_2\text{S}_2\text{O}_3)(\text{mL Na}_2\text{S}_2\text{O}_3)}{\text{mL KIO}_3}$$

Potassium Iodide

- Potassium Iodide, KI
Reagent Grade, ACS Specifications

ECN-2-3263, ECN-2-ML-3260
ECP-0022/1
ECR-1113D
900

Solution Strengths

0.60 M	ECN-2-1305B, ECN-2-1315, ECN-2-1340, ECN-0002/1, ECN-00021/1
	ECP-2-1305C, ECP-2-1315, ECP-0002/1, ECP-00021/1, ECP-0025/01
	ECR-1100G, ECR-1303, ECR-1305L, ECR-1340A, ECR-1803G
	D94-0002/1

Preparation

- Add and dissolve the amount of reagent-grade potassium iodide indicated below in 650 mL of distilled water contained in a 1-litre volumetric flask.

KI Solution	KI g/L
0.60 M	100
0.01 M	1.66
0.6 N	

- Dilute to volume with distilled water and mix.
- For standardized solutions, follow the procedure below.

Standardization

See Reagent Preparations for:

- Acetone, Reagent Grade
- 7.0 N Sulfuric Acid
- Ferriin Indicator
- 0.05 N Sulfato Cerate (standardized to 4 decimal places)

0.01 N Potassium Iodide (standardized to 4 decimal places)

- Pipet (wipe the pipet before leveling) 100.0 mL of potassium iodide being standardized into a 250 mL conical flask.
- Add approximately 50 mL of reagent-grade acetone.
- Add 25 mL of 7.0 N sulfuric acid from a tip-up pipet.
- Dilute the mixture to approximately 175 mL with distilled water.
- Add 1 drop of Ferriin indicator.
- Titrate with standardized 0.05 N sulfato cerate until the pink color changes to pale blue.
- Calculation:

$$N \text{ of KI} = \frac{(N \text{ cerate})(\text{mL cerate})}{(2^*)(\text{mL KI})} = \frac{(N \text{ cerate})(\text{mL cerate})}{2(100.0)}$$

* The reaction between sulfato cerate and potassium iodide in the presence of acetone requires 2 ceric ions for each iodide ion.

- Repeat the standardization on a second portion of the reagent.

0.00100 and 0.000100 N Potassium Iodide

- Pipet (wipe the pipet before leveling) the indicated amount of standardized 0.01 N potassium iodide into a 1 Litre volumetric flask.

Desired KI Solution	mL of Standardized 0.01 N KI
0.00100 N	100.0
0.000100 N	10.0

- Dilute to volume with distilled water and mix.

Note: If precision greater than 2 parts per thousand is required, standardize by potentiometric titration using silver nitrate as titrant.

Potassium Monohydrogen/Dihydrogen Phosphate Buffer

- Potassium Monohydrogen Phosphate, K_2HPO_4
Reagent Grade, ACS Specifications
- Potassium Dihydrogen Phosphate, KH_2PO_4
Reagent Grade, ACS Specifications

Preparation

Buffer pH 7.9 at 25°C

- Add approximately 300 mL of distilled water to a 4-litre beaker and heat it on a hot plate equipped magnetic stirrer.
- Add 740 g of reagent-grade potassium monohydrogen phosphate, stir to dissolve.
- Add and dissolve 250 g of reagent-grade potassium dihydrogen phosphate.
- Remove the solution from the hot plate and cool to room temperature.
- Transfer the solution to a 1-litre volumetric flask, dilute to volume with distilled water, and mix.
- Using a pH meter, determine the pH of the buffer at 25°C. It should be 7.90 ± 0.20 .

ECP-2-407

Note: If it is necessary to adjust the pH of the buffer, add potassium hydroxide, to raise, or phosphoric acid to lower the pH.

Potassium Permanganate

- Potassium Permanganate, $KMnO_4$, Crystals
Reagent Grade, ACS Specifications

Note: Potassium Permanganate is reduced to MnO_2 when exposed to light. This reduction is noted by the formation of brown precipitate in the reagent container. Storage in an amber glass container is recommended.

Preparation

Saturated Potassium Permanganate (formerly 0.41 M)

- Weigh 65.0 g of Potassium Permanganate crystals and transfer to a 1-litre volumetric flask.
- Add 900 mL of distilled water to the volumetric flask. Stir for two hours.
- Dilute to volume with distilled water.

ECR-1603E

Note: Some Potassium Permanganate crystals will remain undissolved.

0.1 N Potassium Permanganate

Purchase standardized (analyzed) material. This should be restandardized monthly for continued use. We do not recommend that you prepare 0.1 N Potassium Permanganate, because of the difficulty in doing so.

Standardization Procedure – 0.1 N Potassium Permanganate

Note: Repeat this procedure in triplicate, and use the average of the three results as the normality value.

1. Accurately weigh 0.24 - 0.28 grams of NIST primary standard reference material grade sodium oxalate in 250 mL of reagent water containing 7 mL of concentrated sulfuric acid contained in a Pyrex 500 mL Erlenmeyer flask.



Caution

Always add acid to water and not water to acid.

2. Heat to 70° C on a hot plate. Titrate immediately while stirring with the permanganate titrant solution to be standardized from a 50 mL buret to a permanent faint pink endpoint that lasts for at least 15 seconds. The temperature at the end of the titration should not be less than 60° C.

$$N \text{ KMnO}_4 = \frac{\text{grams Na}_2\text{C}_2\text{O}_4}{\text{mL KMnO}_4 \times 0.06700}$$

ECN-0007/1

ECN-2-3275-2

ECP-0007/1

Potassium Persulfate

- Potassium Persulfate, $\text{K}_2\text{S}_2\text{O}_8$

Also: Potassium Peroxydisulfate

Reagent Grade, ACS Specifications



Warning

STRONG OXIDANT. Keep separate from readily oxidizable materials. Contact with such materials may cause fire. May cause allergic skin reaction.

Preparation

Note: Potassium persulfate is stable for at least 8 weeks in unopened glass-stoppered bottles. However, one week's supply should be stored in a bottle of appropriate size. All reagent remaining in the opened bottle at the end of a week should be discarded.

Potassium Persulfate, 40 g/L

1. Add approximately 800 mL of distilled water to a 1-litre volumetric flask and stir on a magnetic stirrer.
2. Add and dissolve 40.0 g of potassium persulfate.
3. Dilute to volume with distilled water, stopper and invert the flask several times to mix.

ECN-2-3263, ECN-2-ML-3260, ECN-0006/1

ECP-0006/1

Silver Halide Developer

- *Ammonium Hydroxide*, NH_4OH , Conc., Approx. 15 N

Reagent Grade, ACS Specifications



Warning

CORROSIVE. Avoid contact with solution and breathing of vapor. Use an exhaust hood. Wear eye protection and impervious gloves. Store in a cool place to prevent pressure build-up in the container.

- *Hydroquinone*, $\text{C}_6\text{H}_4\text{-1,4-(OH)}_2$

Reagent or Photographic Grade



Warning

Causes eye and skin irritation. May cause allergic skin reaction. Avoid contact with eyes, skin, and clothing.

- *Sodium Sulfite*, Na_2SO_3

Reagent Grade, ACS Specifications

Preparation

Note: Use all possible care to keep air contact to a minimum once the hydroquinone is added to the flask.

1. Place 630 mL of distilled water in a 1-litre beaker.
2. Add 270 mL of reagent-grade concentrated ammonium hydroxide.
3. Add and dissolve 50 g of reagent-grade sodium sulfite in the ammonium hydroxide.
4. Add 50 g of hydroquinone to a dry, 1-litre volumetric flask using a dry powder funnel.
5. Add the sodium sulfite/ammonium hydroxide solution to the flask; dilute to volume with distilled water. Place a magnetic stirring bar in the flask, stopper the flask, and then stir the solution just long enough to dissolve the hydroquinone.
6. Bottle the developer in rubber-stoppered 60 mL bottles. The solution should be discarded if it turns brown.

ECN-926C

ECP-926C

ECR-929C, ECR-930E

Note: Store in a dark, cool place to maximize the stability of the reagent.

Silver Nitrate

- Silver Nitrate, AgNO₃

Reagent Grade, ACS Specifications



Warning

STRONG OXIDIZER. Causes eye and burns. Stains the skin. Avoid contact. Harmful if inhaled.

Solution Strengths

0.1 N D94-0003/1

Standardized

0.05 N ECN-2-926C, ECN-2-2100B, ECN-0004/1,
ECN-0005/1, ECN-0009/1, ECN-0022/1
ECP-2-926C, ECP-0004/1, ECP-0005/1,
ECP-0009/1, ECP-0027-01
ECR-930E, ECR-937, ECR-2100B, ECR-0004/1
D94-0001/1

0.005 N ECP-0022/1
ECR-925A

0.001 N ECR-929C

Preparation

0.05 N Silver Nitrate

- Add about 800 mL of chloride-free distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
- Weigh 8.495 ± 0.0001 g of reagent-grade silver nitrate and quantitatively transfer to the 1-litre volumetric flask. Stir until dissolved.
- Remove the stir bar, rinse with chloride-free distilled water and add all rinses to the 1-litre flask.
- Dilute to volume with chloride-free distilled water, stopper and invert six to ten times to mix.

0.1 N Silver Nitrate

- Add about 800 mL of chloride-free distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
- Weigh 16.990 ± 0.0001 g of reagent-grade silver nitrate and quantitatively transfer to the 1-litre volumetric flask. Stir until dissolved.
- Remove the stir bar, rinse with chloride-free distilled water and add all rinses to the 1-litre flask.
- Dilute to volume with chloride-free distilled water, stopper and invert six to ten times to mix.

Standardization

See Reagent Preparation for:

- Potassium Chloride, KCl

NIST Primary Standard SRM-999

- 7.0 N Sulfuric Acid, H₂SO₄

0.05 N Silver Nitrate

(standardized to 4 decimal places)

Preferred Method

- Weigh 55 mg to the nearest 0.1 mg of potassium chloride, NIST SRM-999, that has been previously dried in a 110°C oven for 2 hours and cooled in a desiccator over Drierite to room temperature.
- Quantitatively transfer the potassium chloride to a 150 mL beaker.
- Add approximately 50 mL of chloride-free distilled water.
- Carefully add 25 mL of 7.0 N sulfuric acid from a tip-up pipet.
- Titrate the acidified potassium chloride solution potentiometrically using the 0.05 N silver nitrate being standardized. Use the following settings for a Metrohm Potentiograph Model E536 or equivalent

E536 Potentiograph:	Control Settings
Stop %U:	OFF
Autocontrol:	OFF
Feeding Time:	15 min/100% volume
Selector Switch:	mV, pH
Measuring Span:	750 mV
Changeover Switch:	400 mm/100% volume
Buret Size:	20 mL
Reference Electrode:	Double Junction, Orion No. 900200
Indicator Electrode:	Silver Billet, Beckman No. 39261

- Record titration volume in litres.
- Repeat the standardization procedure two more times.

8. Calculation:

a.

$$N \text{ AgNO}_3 = \frac{(g, \text{KCl})}{(\text{eq wt of KCl})(L, \text{AgNO}_3)}$$

Where:

$$74.55 = \text{eq wt of KCl}$$

b. Calculate the mean normality (\bar{N}) and standard deviation (s):

$$\bar{N} = \frac{\sum N}{n}$$

Where:

N = the individual normality results

$\sum N$ = the sum of the n normality results

n = the number of replicate results

$$s = \sqrt{\frac{\sum (N - \bar{N})^2}{n-1}}$$

c. Accept your laboratory standardization value if (s) ≤ 0.0005 .

Alternate Method

1. Add approximately 50 mL of distilled water to a 100 mL volumetric flask. Stir on a magnetic stirrer.
2. Weigh 600 mg to the nearest 0.1 mg of potassium chloride, NIST SRM-999, that has been previously dried in a 110°C oven for 2 hours and cooled in a desiccator over Drierite to room temperature.
3. Quantitatively transfer the potassium chloride to the 100 mL volumetric flask. Stir until the potassium chloride is dissolved.
4. Remove the stir bar, rinse with distilled water and add all rinses to the 100 mL volumetric flask.
5. Dilute to volume with distilled water.
6. Add approximately 50 mL of distilled water to a 150 mL beaker, equipped with a magnetic stirrer.
7. Pipet a 5.00 mL aliquot from the 100 mL volumetric flask and transfer to the 150 mL beaker.
8. Carefully add 10 mL of 7.0 N sulfuric acid from a tip-up pipet.

9. Titrate the acidified potassium chloride solution potentiometrically using the 0.05 N silver nitrate being standardized. Use the following settings for a Metrohm Potentiograph Model 682 or equivalent.

682 Potentiograph:	Control Settings
Stop U:	300 mV
Stop V:	10.00 mL
Stop EP:	1
EP Crit.:	2
Anticip.:	50
Feeding Rate:	1.50 mL/min
Buret Size:	10 mL
Reference Electrode:	Double Junction, Orion No. 900200
Indicator Electrode:	Silver Billet, Beckman No. 39261

10. Record titration volume in litres.

11. Repeat the standardization procedure two more times.

12. Calculation:

a.

$$N \text{ AgNO}_3 = \frac{(\text{mg, KCl})(5.00)}{(\text{meq wt of KCl})(\text{mL, AgNO}_3)(100)}$$

Where:

$$74.55 = \text{meq wt of KCl}$$

$$100 = \text{dilution factor}$$

$$5.00 = \text{KCl transfer aliquot in mL}$$

b. Calculate the mean normality (\bar{N}) and standard deviation (s):

$$\bar{N} = \frac{\sum N}{n}$$

Where:

N = the individual normality results

$\sum N$ = the sum of the n normality results

n = the number of replicate results

$$s = \sqrt{\frac{\sum (N - \bar{N})^2}{n-1}}$$

c. Accept your laboratory standardization value if (s) ≤ 0.0005 .

Stability and Storage

Restandardize the 0.05 N silver nitrate solution every 3 months. Store in amber Pyrex glass bottles or equivalent. Protect from direct light and heat.

0.005 N and 0.001 N Silver Nitrate

1. Add 800 mL of chloride-free distilled water to a 1-litre volumetric flask containing a magnetic stir bar.
2. Pipet (wipe the pipet before leveling) the indicated volume of standardized 0.05 N silver nitrate into the 1-litre volumetric flask:

Desired Normality	Volume of Std. 0.05 N AgNO_3
0.005 N	100.0 mL
0.001 N	20.0 mL

3. Remove the stir bar, rinse with chloride-free distilled water and add all rinses to the 1-litre flask.
4. Dilute to volume with chloride-free distilled water, stopper and invert six to ten times to mix.
5. Calculation:

$$N \text{ AgNO}_3 = \frac{(N \text{ of Std AgNO}_3 \text{ being diluted})}{(DF)}$$

Where:

N = normality of standardized AgNO_3 being diluted (to 4 decimal places)

DF = dilution factor

$$= \frac{\text{Total Dilution Volume}}{\text{Aliquot of Std AgNO}_3}$$

Stability and Storage

Prepare the 0.005 N and 0.001 N silver nitrate solutions fresh weekly. Store in amber Pyrex (or equivalent) glass bottles. Protect from direct light and heat.

Sodium Acetate

- Sodium Acetate, CH_3COONa , Anhydrous

Reagent Grade, ACS Specifications

Sodium Arsenate Reagent

- Arsenic Trioxide, As_2O_3

Also: Arsenous Oxide, Arsenous Acid Anhydride, Arsenous Acid, Arsenic Sesquioxide or White Arsenic

NIST Oxidimetric Primary Standard SRM-83d (or subsequent lot of SAM-83)



Warning

POISON. HARMFUL IF INHALED. IRRITANT. Avoid contact with eyes and skin. Wear impervious gloves and eye protection when using. Do not inhale vapors. Suspected human carcinogen. Use only with adequate ventilation.

- 2.5 N Sodium Hydroxide, NaOH
- 1.0 N Sulfuric Acid, H_2SO_4

Preparation

1. Weigh 1 g of NIST SRM-83d arsenic trioxide to the nearest 0.1 mg and place it in a 125 mL Phillips beaker.



Caution

Arsenic Trioxide is poisonous.

2. Add, from a tip-up pipet, 10 mL of 2.5 N sodium hydroxide; swirl to dissolve. Rinse down the sides of beaker with distilled water from a wash bottle and swirl.
3. Add, from a tip-up pipet, 30 mL of 1.0 N sulfuric acid; cool to room temperature.
4. Quantitatively transfer the solution to a 1-litre volumetric flask.
5. Dilute to volume with distilled water; mix thoroughly.

Note:

- a. This reagent is used in the standardization procedure for iodine.
- b. This reagent is stable for eight weeks.

Sodium Bicarbonate

- Sodium Bicarbonate, NaHCO_3

Reagent Grade, ACS Specifications

Sodium Bromide

- Sodium Bromide, NaBr , minimum assay 99.0%

Reagent Grade, ACS Specifications

ECP-2-936A

Sodium Chloride

- Sodium Chloride, NaCl , Crystals

Reagent Grade, ACS Specifications

ECN-0003/1, ECN-0009/1

ECP-0003/1, ECP-0009/1

ECR-925A

Preparation

Saturated Sodium Chloride Solution

1. Place a 1500 mL beaker containing 1 litre of distilled water on a magnetic stirrer.
2. Add 400 g of reagent-grade sodium chloride; stir for 5 minutes.

ECR-1603E

Note: Always maintain an excess of undissolved sodium crystals in the reagent container.

0.10 N Sodium Chloride

1. Place a 1-litre volumetric flask containing 750 mL of distilled water on a magnetic stirrer.

2. Add and dissolve 5.85 ± 0.001 g of sodium chloride; dilute to volume with distilled water and mix.

ECN-0004/1
ECP-0004/1
ECR-0004/1

Sodium Citrate

- Sodium Citrate 2-Hydrate,
 $\text{NaOC}(\text{COOH})(\text{CH}_2\text{COONa})_2 \cdot 2\text{H}_2\text{O}$
Reagent Grade, ACS Specifications

Sodium Dichromate

- Sodium Dichromate, $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot \text{H}_2\text{O}$

ULM-0005/1



Warning

POISON. May be fatal if swallowed. Causes severe skin and eye burns. Suspected cancer hazard. May cause kidney damage. Harmful if inhaled or absorbed through the skin. Dust irritating to eyes and respiratory tract. May cause allergic skin reaction.

STRONG OXIDIZER. Contact with other material can cause fire. Do not breathe dust. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. Use with adequate ventilation. Keep from contact with clothing and combustible materials. Remove and wash contaminated clothing promptly. Before using, read Material Safety Data Sheet.

Sodium Diphenylamine Sulfonate Indicator

(SDS Indicator)

- Sodium Diphenylamine Sulfonate,
 $\text{C}_6\text{H}_5\text{NHC}_6\text{H}_4\text{-4-SO}_3\text{Na}$
Also: p-Diphenylaminesulfonic Acid Sodium Salt
Reagent Grade, ACS Specifications

Preparation

0.01 M Sodium Diphenylamine Sulfonate

1. Add 400 mL distilled water to a 500 mL volumetric flask; stir on a magnetic stirrer.
2. Add 1.4 g sodium diphenylamine sulfonate to the 500 mL volumetric flask; stir to dissolve.
3. Dilute to volume with distilled water and mix.

ECN-2-1101
ECP-2-1101
ECR-1101E

Sodium Hydroxide

- Sodium Hydroxide, NaOH, Pellets
Reagent Grade, ACS Specifications



Warning

CORROSIVE. Causes severe eye and skin burns. Avoid contact with eyes, skin, or clothing. Wear eye protection and impervious gloves. Do not weigh in an aluminum dish.

Note: Sodium hydroxide solutions should be stored in rubber-stoppered Pyrex bottles. If a solution is to be used in a dispensing system, there should be a soda-lime tube between the solution and the air.

Note: Sodium hydroxide pellets are deliquescent. The supply container should only be open long enough to weigh the required amount of reagent. Keep the container tightly closed at other times

Solution Strengths

10 N	ECN-2-2100B ECNP-2-2020, ECP-0027-01 ECR-925A, ECR-2100B
6 N	ECN-2-2010A ECP-0022/1
2.5 N	1122B ECN-0025-1 ECR-1803G
1.0 N	ECN-0002/1, ECN-0019-01, ECN-0023-01 ECP-0002/1, ECP-0019-01, ECP-0020-01
0.10 N	ECR-1611D
0.01 N	m-Cresol Purple Indicator

Standardized

1.0 N	ECN-2-755 ECR-754A, ECR-755A
0.1 N	ECR-617B, ECR-1603E, ECR-1611D, ECR-1612

Preparation

Non-standard Solutions

1. Add (with extreme caution), in an exhaust hood, the indicated amount of reagent-grade sodium hydroxide 800 mL of distilled water in a 2-litre Pyrex beaker. See table below:

Desired Concentration	Sodium Hydroxide g/L
10 N	410
6 N	246
2.5 N	102
1.0 N	41
0.10 N	4.1

Note:

- A slight excess of sodium hydroxide is added because of the deliquescent nature of the pellets.
 - 16 N and 10 N sodium hydroxide will contain white sodium carbonate precipitate which need not be removed.
- Stir to dissolve; cool to room temperature. (Use care when handling the beaker of solution. It is advisable place the beaker in a polyethylene pail.)
 - Transfer to a 1-litre volumetric flask and dilute to volume with distilled water.

0.01 N Sodium Hydroxide

- Pipet (wipe the pipet before leveling) 10.0 mL of 1.0 N sodium hydroxide or 4.00 mL of 2.5 N sodium hydroxide into a 1-litre volumetric flask.
- Dilute to volume with distilled water.

Note: No standardization is necessary for this reagent since it is used primarily as a dilution medium absorbance readings and not for titrations. Its absorbance should be no more than 0.095 ± 0.005 at 240 nm vs an air blank.

The solution should have a pH of 12.00 ± 0.30 at 70°F (21°C), 11.90 ± 0.30 at 75°F (24°C), or 11.80 ± 0.30 at 80°F (27°C).

Standardization

Prepare the approximate concentration according to the ion (non-standardized solutions) and perform standardization procedure.

See Reagent Preparation for:

- Potassium Hydrogen Phthalate*
NIST Acid/Base Primary Standard SRM-84j
(or subsequent lot of SRM-84)
- Phenolphthalein Indicator*

Preferred Method**0.01 N Sodium Hydroxide****(standardized to 4 decimal places)**

- Weigh the indicated amount of NIST potassium hydrogen phthalate (SRM-84j) to the nearest 0.1 mg. Record the weight and place the standard in a 125 mL Erlenmeyer flask.

Desired Sodium Hydroxide Normality, N	Weight of Potassium Hydrogen Phthalate SRM-84j
1	4 g
0.1	0.4 g

- Dissolve the sample in 50 to 75 mL of distilled water, stirring on a magnetic stirrer.
- Add 3 drops of phenolphthalein indicator.
- Using a 25 mL buret, titrate with the sodium hydroxide being standardized. The end point is indicated by a *pink color* persists for 30 seconds.
- Calculations:

$$N \text{ NaOH} = \frac{(g \text{ KHC}_8\text{H}_4\text{O}_4)(1000)}{(\text{mL NaOH})(\text{eq wt KHC}_8\text{H}_4\text{O}_4\text{K})} =$$

$$\frac{(g \text{ KHC}_8\text{H}_4\text{O}_4)(1000)}{(\text{mL NaOH})(204.2)} = \frac{4.897(g \text{ KHC}_8\text{H}_4\text{O}_4)}{(\text{mL NaOH})}$$

- Repeat the standardization on two more portions of NIST potassium hydrogen phthalate.
- To decide whether to accept the reagent or adjust it, see Method IA, Figure 1, "Sequential Analysis for Standardization of Reagents."
- If possible, use a potentiometric titration rather than visual. Use a pH indicator electrode and a calomel reference electrode such as:
 - CORNING 476024, glass, rugged bulb (US Standard Connector) or equivalent
 - CORNING 476002, reference, ceramic junction, calomel, or equivalent (Filled with 3.5 N potassium chloride solution)

Alternative Method**0.01 N Sodium Hydroxide****(standardized to 4 decimal places)**

- Pipet (wipe the pipet before leveling) 100.0 mL of standardized 1 N sodium hydroxide into a 1-litre volumetric flask.
- Dilute to volume with distilled water.

1 N Sodium Hydroxide/EDTA Reagent

- Sodium Hydroxide*, NaOH, Pellets
Reagent Grade, ACS Specifications

**Warning**

CORROSIVE. Causes severe eye and skin burns. Avoid contact with eyes, skin, or clothing. Wear eye protection and impervious gloves. Do not weigh in an aluminum dish.

Note: Sodium hydroxide pellets are deliquescent. The supply container should only be open long enough to weigh the required amount of reagent. Keep the container tightly closed at other times.

- Ethylenedinitrilotetraacetic Acid (EDTA)*
Reagent Grade, ACS Specifications

Preparation

- Add 800 mL distilled water to a 1-litre volumetric flask; stir on a magnetic stirrer.
- Cautiously add 40.0 g of sodium hydroxide to the volumetric flask; stir to dissolve.
- Add 2.0 g of (ethylenedinitrilo) tetraacetic acid to the volumetric flask; stir to dissolve.
- Dilute to volume with distilled water and mix.

1208E

6% Sodium Methoxide in Methanol

(59 g/L)



Warning

Sodium methoxide is a strong caustic; it burns the eyes and the skin; breathing the dust is harmful. It is flammable and reacts with water, solvents, and oxidizing agents. For these reasons, it is recommended that the sodium methoxide in methanol reagent be prepared from a 25% sodium methoxide solution if that alternative is available.

- 25% Sodium Methoxide in Methanol
Reagent Grade, ACS Specifications
- Methanol, CH₃OH
Spectro Grade, ACS Specifications



Warning

POISON: May be fatal or cause blindness if swallowed. Cannot be made non-poisonous. Harmful if inhaled. Harmful if absorbed through the skin. Causes skin and eye irritation. Avoid contact with eyes, skin, or clothing. Wash thoroughly after handling. Avoid breathing vapor. Use with adequate ventilation. **FLAMMABLE.** Keep away from heat, sparks, and flame. Store in original tightly closed container.

- Sodium Methoxide, CH₃NaO, powder
Also: Sodium Methylate
Reagent Grade, ACS Specifications



Warning

Flammable solid. Causes severe eye and skin burns. Dust irritating to eyes and respirator tract. Water reactive. May ignite if exposed air. Contact with water may cause flash fire. Keep away from heat, sparks, and flame. Do not get in eyes, on skin, or on clothing. Do not breathe dust. Do not store near combustible material. Store in original tightly closed container. Keep from any contact with water. Use with adequate ventilation. Wash thoroughly after handling.

Preparation

6% Sodium Methoxide in Methanol

1. Add from a graduated cylinder 250 mL of 25% sodium methoxide in methanol to a 1-litre volumetric flask.
2. Dilute to volume with methanol.
3. Stopper and invert the flask several times to thorough mix its contents.
4. Readjust to volume and again mix, if necessary.

ECN-2-1570C

Note: The 6% solution of sodium methoxide is the recommended reagent. The 59 g/L solution should be used only when the 6% solution alternative is unavailable.

59 g/L Sodium Methoxide in Methanol

1. In a dry 800 mL beaker, weigh 59 grams of sodium methoxide.
Note: Do not use more than 59 grams at a time or excessive heat may be formed in later steps.
2. Add slowly and without agitation 500 mL of methanol.



Caution

Heat is Evolved.

3. Let stand for 2 minutes.
4. Add a small stirring bar (about one inch long) and begin stirring slowly.
5. When the sodium methoxide is dissolved, transfer the solution to a 1-litre volumetric flask. Use additional methanol, as necessary, to complete the transfer.
6. Fill the volumetric flask to volume with methanol. Stir on a magnetic stirrer. Cool to room temperature and readjust the volume with additional methanol. Stir to mix again.
7. Transfer to a glass-stoppered conical flask or similar short-neck container with a tight lid or stopper. Allow to stand overnight.
8. Decant the clear solution from the precipitate that settles to the bottom. Do not save the bottom 200 mL of solution.
9. If the solution is still turbid, let stand another night and decant again.

Note: Both solid sodium methoxide and the reagent are attacked by moisture and carbon dioxide in air. Prolonged storage of solid methoxide exposed to air is a fire hazard. Excess solid sodium methoxide should be discarded or resealed under nitrogen. If resealing under nitrogen is not possible, ¼ lb. jars of sodium methoxide should be used to minimize the excess to be discarded.

Solid sodium methoxide or the reagent should be disposed of by incineration, including use of a licensed waste disposal company.

Stability and Storage

With brief exposure to air twice a week, the reagent is stable for about six months. The longer and more frequent the exposure to air, the sooner the reagent should be discarded.

Sodium Nitrite

- Sodium Nitrite, NaNO₂
Reagent Grade, ACS Specifications

Preparation

0.5 M Sodium Nitrite

1. Weigh 34.5 ± 0.01 g sodium nitrite.

2. Transfer, using distilled water, to a 1-litre volumetric flask. Add 800 mL distilled water.
3. Stir on a magnetic stirrer to dissolve.
4. Remove and rinse stirrer bar.
5. Dilute to volume with distilled water.

ECP-2-936A

Sodium Oxalate

- Sodium Oxalate, $\text{Na}_2\text{C}_2\text{O}_4$

NIST **Oxidimetric Working Standard** SRM-40h
(or subsequent lot of SRM-40)



Warning

Oxidizing material. Avoid contact with eyes, skin, or clothing.

Sodium Sulfate

- Sodium Sulfate, Na_2SO_4 , Granular

Reagent Grade, ACS Specifications

ECN-2-1570C

ECP-2-936A

Sodium Sulfide

- Sodium Sulfide, $\text{Na}_2\text{S} \cdot 9\text{H}_2\text{O}$

Reagent Grade, ACS Specifications



Warning

POISON. Wash thoroughly after handling. **STRONG IRRITANT.** Avoid contact with eyes and skin. Wear eye protection and impervious gloves when handling. Avoid inhalation of dust. Use with adequate ventilation.

DO NOT MIX WITH ACID. Sodium sulfide reacts with acid and forms highly toxic hydrogen sulfide gas.

Solution Strengths

0.06 N **ECN-0023-01**

0.8 M **ECP-0022/1**

Preparation

0.06 N Sodium Sulfide

1. Add about 800 mL of reagent water to a 1-litre volumetric flask; stir on a magnetic stirrer.
2. Add 7.5 g sodium sulfide, 9-hydrate, to the volumetric flask; stir to dissolve.
3. Dilute to volume with reagent water and mix.
4. Store the reagent in small, rubber-stoppered Pyrex bottles, filling the bottles to overflowing.
5. Avoid unnecessary air exposure of the reagent. Once a week, standardize using the following procedure.

0.8 M Sodium Sulfide

1. Add approximately 800 mL of reagent water to a 1-litre volumetric flask containing a magnetic stirrer.
2. While stirring, add 192 g of sodium sulfide, 9-hydrate to the volumetric flask and dissolve.
3. Dilute to volume with reagent water and mix.

Standardization

0.06 N Sodium Sulfide

See Reagent Preparation for:

- 0.10 N *Sodium Chloride*
- 0.05 N *Silver Nitrate* (standardized to 4 decimals)
- 1.0 N *Sodium Thiosulfate*
- 1 N *Sodium Hydroxide/EDTA Reagent*
- 4 g/L *Gelatin Solution*
 1. Add, from a tip-up pipet, 30 mL of 0.10 N sodium chloride to a 600 mL beaker.
 2. Pipet, wipe the pipet before leveling, 50.0 mL of standardized 0.05 N silver nitrate into the beaker.
 3. Add 300 mL of 1.0 N sodium thiosulfate from a graduated cylinder.
 4. Add 100 mL of 1 N sodium hydroxide/EDTA reagent from a tip-up pipet.
 5. Add 10 mL of 4 g/L gelatin solution from a tip-up pipet.
 6. Titrate the samples potentiometrically using the sodium sulfide to be standardized. Use the following electrodes:

Reference Electrode: Double Junction, Orion No. 900200 or equivalent

Indicator Electrode: Silver Billet, Beckman No. 39261 or equivalent

7. Calculations:

$$N \text{ Na}_2\text{S} = \frac{(N \text{ AgNO}_3)(\text{mL AgNO}_3)}{(\text{mL Na}_2\text{S})} = \frac{(N \text{ AgNO}_3)(50.0)}{(\text{mL Na}_2\text{S})}$$

8. Repeat the standardization. If the range of two results does not exceed 0.00035 N, average the results. If the range exceeds this value, repeat the standardization. Do not attempt to adjust the reagent to its nominal value 0.06 N, but use the calculated normality.

Sodium Sulfite

- Sodium Sulfite, Na_2SO_3 , Anhydrous

Reagent Grade, ACS Specifications

Sodium Tetraborate

- Sodium Tetraborate, $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$

Also: Borax or Sodium Borate 10-Hydrate

NIST **Ion Activity/pH Standard** SRM-187c
(or subsequent lot of SRM-187)

Sodium Thiosulfate

- Sodium Thiosulfate, 5-Hydrate, $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$

Reagent Grade, ACS Specifications

Solution Strengths

1.0 M ECN-0023-01

Standardized

0.1 N ECN-2-1305B, ECN-1315, ECN-2-1340,
ECN-2-ML-3260, ECN-2-3263, ECN-0002/1,
ECN-00021/1

ECP-2-1305C, ECP-1315, ECP-0002/1,
ECP-00021/1, ECP-0025/01

ECR-1100G, ECR-1113D, ECR-1303,
ECR-1305L, ECR1340A, ECR-1803G

D94-0002/1

Preparations

1.0 M Sodium Thiosulfate

- Add 800 mL of distilled water to a 1-litre volumetric flask containing a Teflon stir bar.
- Add and dissolve 248.0 ± 0.1 g of sodium thiosulfate 5-hydrate. Remove the Teflon stir bar.
- Add 1 mg of mercuric iodide as a preservative and dissolve.
- Dilute to volume with distilled water and mix.

0.1 N Sodium Thiosulfate

(standardized to 4 decimal places)

- Add 800 mL of freshly boiled and cooled distilled water to a 1-litre volumetric flask containing a Teflon stir bar.
- Add and dissolve 24.82 ± 0.001 g of sodium thiosulfate 5-hydrate. Remove the Teflon stir bar.
- Add 1 mg of mercuric iodide as a preservative and dissolve.
- Dilute to volume with freshly boiled and cooled distilled water and mix.
- Standardize as described below.

Standardization

See Reagent Preparation for:

- Potassium Biiodate
GFS Certified No. 76, GFS Chemicals
- Potassium Iodide
- 7.0 N Sulfuric Acid
- Starch Indicator (for manual titration)
- Mercuric Iodide



Warning

POISONOUS. Avoid inhalation. Use with adequate ventilation. Avoid contact with eyes and skin. Wear protective gloves when using. Photographic contaminant.

Note: It is preferable to make three separate smaller weighings of dried potassium biiodate rather than making a 2.40 g/L solution and taking three subsequent aliquots. However, for labs that do not have the capability of weighing to ± 0.1 mg, using the 2.40 g/L solution is acceptable. See the potassium biiodate reagent preparation section if you need to make the solution.

- Dry enough GFS Certified No. 76 potassium biiodate for the weighings, in a 105°C oven for two hours. Do not exceed 120°C or degradation of the material may occur.
- Cool the material to room temperature in a desiccator.

Potentiometric Titration

Equipment

- Metrohm E-536 Potentiograph
 - 50 or 20 mL buret for the Potentiograph (depending on size of the buret used for the sample)
 - Reference Electrode, Double Junction, Orion No. 900200 or equivalent
 - Indicator Electrode, Platinum Inlay/Disc, Beckman No. 39273 or equivalent
- According to the buret size used, weigh the amount of potassium biiodate indicated in the table below (or pipet the volume of 2.40 g/L potassium biiodate solution) and quantitatively transfer into a 250 mL beaker containing a Teflon stirring bar and 100 mL of distilled water.

Buret Size	g Biiodate	mL Biiodate
50 mL	0.12*	50.0
20 mL	0.05*	20.0

* Weighed to the nearest 0.0001 g.

- While stirring, add 10 mL of 7.0 N sulfuric acid and 2.00 ± 0.01 g of reagent-grade potassium iodide.
- After the potassium iodide has dissolved, immediately titrate with the 0.1 N sodium thiosulfate to be standardized. If using a Metrohm E536 Potentiograph autotitrator, use the following settings:

E536 Potentiograph:	Control Settings
Cut-Off:	OFF
Autocontrol:	OFF
Feeding Time:	15 min/100% volume
Selector Switch:	mV, pH
Measuring Span:	500 mV
Changeover Switch:	400 mm/100% volume
Buret Size:	50 or 20 mL as required
Reference Electrode:	Double Junction, Orion No. 900200
Indicator Electrode:	Platinum Inlay/Disc. Beckman No. 39261

$$\bar{N} = \frac{\sum N}{n}$$

Where:

N = the individual normality results

$\sum N$ = the sum of the n normality results

n = the number of replicate results

$$s = \sqrt{\frac{\sum (N - \bar{N})^2}{n-1}}$$

4. Determine the volume (mL) of 0.1 N sodium thiosulfate at the end point using concentric arcs.
5. Run the standardization in triplicate.
6. Calculations:
 - a. For direct weighing of Potassium Biiodate

$$N \text{ Na}_2\text{S}_2\text{O}_3 = \frac{[\text{g KH}(\text{IO}_3)_2]}{[\text{mL titrated}][0.03250]} = \frac{30.769[\text{g KH}(\text{IO}_3)_2]}{[\text{mL titrated}]}$$

- b. For 2.4 g/L solution used:

- For 50 mL Buret

$$N \text{ Na}_2\text{S}_2\text{O}_3 = \frac{[\text{g KH}(\text{IO}_3)_2 \text{ weighed}][50.0 \text{ mL}]}{[\text{mL titrated}][0.03250][1000]} =$$

$$\frac{1.538[\text{g KH}(\text{IO}_3)_2 \text{ weighed}]}{[\text{mL titrated}]}$$

- For 20 mL Buret

$$N \text{ Na}_2\text{S}_2\text{O}_3 = \frac{[\text{g KH}(\text{IO}_3)_2 \text{ weighed}][20.0 \text{ mL}]}{[\text{mL titrated}][0.03250][1000]} =$$

$$\frac{0.615[\text{g KH}(\text{IO}_3)_2 \text{ weighed}]}{[\text{mL titrated}]}$$

0.03250 = milliequivalent wt. of $\text{KH}(\text{IO}_3)_2$

1000 = factor for converting mL to L

- c. Calculate the mean normality (\bar{N}) and standard deviation (s):
- d. Laboratory experience at Kodak shows the standard deviation (s) should be ≤ 0.0003 . Determine the standard deviation (s) for your laboratory.

Manual Titration

1. Weigh 0.12 ± 0.0001 g of potassium biiodate and quantitatively transfer (or pipet 50.0 mL of 2.40 g/L potassium biiodate solution) into a 250 mL Erlenmeyer flask containing a Teflon stirring bar and 100 mL of distilled water.
2. While stirring, add 10 mL of 7.0 N sulfuric acid and 2.00 ± 0.01 g of reagent-grade potassium iodide.
3. After the potassium iodide has dissolved, immediately titrate (while stirring) with the 0.1 N sodium thiosulfate to be standardized from a 50 mL buret to a *light yellow color*. Add 5 mL of starch indicator and titrate drop by drop until the disappearance of the *blue color*.
4. Calculations:
 - a. For direct weighing of Potassium Biiodate

$$N \text{ Na}_2\text{S}_2\text{O}_3 = \frac{[\text{g KH}(\text{IO}_3)_2]}{[\text{mL titrated}][0.03250]} = \frac{30.769[\text{g KH}(\text{IO}_3)_2]}{[\text{mL titrated}]}$$

- b. For 2.4 g/L solution used:

For 50 mL Buret

$$N \text{ Na}_2\text{S}_2\text{O}_3 = \frac{[\text{g KH}(\text{IO}_3)_2 \text{ weighed}][50.0 \text{ mL}]}{[\text{mL titrated}][0.03250][1000]} = \frac{1.538[\text{g KH}(\text{IO}_3)_2 \text{ weighed}]}{[\text{mL titrated}]}$$

0.03250 = milliequivalent wt. of $\text{KH}(\text{IO}_3)_2$

1000 = factor for converting mL to L

4. Continue boiling and stirring the solution for 20 minutes.
5. Let the solution cool to room temperature, and then transfer the solution to a 1-litre reagent bottle.
6. Store the starch solution at a cool (40°F) temperature to retard mold growth.
7. Discard the starch solution when either excessive mold growth appears or after 3 months.

ECN-2-1305B, ECN-2-1315, ECN-2-1340, ECN-2-3263, ECN-2-ML-3260, ECN-0002/1, ECN-00021/1

ECP-2-1305C, ECP-2-1315, ECP-0002/1, ECP-00021/1, ECP-0025/01

ECR-1100G, ECR-1113D, ECR-1303, ECR-1305L, ECR-1340A, ECR-1803G

Starch Indicator

- Starch, soluble, powdered
Reagent Grade, ACS Specifications
- Mercuric Iodide, HgI_2
Reagent Grade, ACS Specifications



Warning

POISONOUS. Avoid inhalation. Use with adequate ventilation. Avoid contact with eyes and skin. Wear protective gloves when using.

Procedure

1. Add just enough cold distilled water to 2.0 grams of reagent-grade soluble starch to make a thin paste.
2. Slowly, while stirring, add the paste to 1 litre of rapidly boiling distilled water, contained in a 1500 mL beaker.
3. Add 1 mg/L of mercuric iodide as a preservative to the boiling solution.

Sulfato Cerate

- *Ceric Ammonium Nitrate*, $(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6$
GFS Certified No. 15 (NIST Working Standard), GFS Chemicals or equivalent
- *Sulfuric Acid*, H_2SO_4 , Conc., 95 to 98%, sp gr 1.84, approx. 36 N
Reagent Grade, ACS Specifications



Warning

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor.

- 7.0 N *Sulfuric Acid*

Preparation

0.0500 N Sulfato Cerate (standardized to 4 decimal places)

1. Weigh 27.413 (to nearest 0.001 g) of GFS No. 15 ceric ammonium nitrate in a 150 mL beaker, and record the weight.

Note: The ceric ammonium nitrate is weighed in a glass beaker to avoid contamination by a metal dish.

2. Transfer the ceric ammonium nitrate to a 1-litre beaker.
3. Add 100 mL of distilled water.
4. Add cautiously (in an exhaust hood, wearing safety goggles and rubber gloves) 28 mL of concentrated sulfuric acid to the 1-litre beaker. Stir for two minutes.



Caution

ACID. Avoid contact with skin and eyes. In case of contact, flush with water.

5. Using distilled water, rinse into the 1-litre beaker any ceric ammonium nitrate that has adhered to the weighing container, transfer funnel, etc.
6. Add consecutive 100 mL portions of water (no more than a total of 800 mL of distilled water), while stirring, until all the ceric ammonium nitrate has dissolved.
7. Place the beaker in a cooling bath and cool the solution to room temperature.
8. Transfer the solution quantitatively to a 1-litre volumetric flask and dilute to volume with distilled water.
9. Standardize using the procedure below.

ECN-2-1101, ECN-0003/1, ECN-0020-01, ECN-0024/1

ECP-2-407, ECP-2-1101, ECP-2-2010A, ECP-2-2020, ECP-0003/1, ECP-0023/01, ECP-0026/1

ECR-125F, ECR-1101E, ECR-1102C, ECR-1113D, ECR-1125B

Standardization

See reagent preparations for:

- *Sodium Oxalate*

NIST **Oxidimetric Working Standard** SRM-40h
(or subsequent lot of SRM-40)

- *Iodine/Chloride Solution*
- *Ferriin Indicator*
- *Hydrochloric Acid*, concentrated



Caution

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor. Use an exhaust hood.

Equipment

- Thermometer, 0 to 110°C
- Analytical Balance
- Hot Plate
- Reference Electrode, Double Junction, Orion No. 900200 or equivalent
- Indicator Electrode, Platinum Inlay/Disc, Beckman No. 39273 or equivalent
- Metrohm 536 Automatic Titrator
- 25 mL Buret

Procedure A—Standardization by Autotitrator

1. Dry approximately 0.5 g of sodium oxalate in oven at 105°C for 2 hours. Place in desiccator to equilibrate to room temperature.
2. To a 250 mL beaker, add 100 mL of distilled water.
3. Weigh 0.05 g (to nearest 0.1 mg) of dried sodium oxalate. Quantitatively transfer to the 250 mL beaker and stir to dissolve.
4. Add 20 mL of concentrated hydrochloric acid and stir the solution.
5. Heat the solution to $70 \pm 5^\circ\text{C}$.
6. Place the solution on a Metrohm 536 titrator equipped with the above electrodes. Allow the electrodes to equilibrate for 5 minutes.

Note: Do not allow solution to stand for longer than 5 minutes before titrating.

7. Titrate the solution with the sulfato cerate being standardized. If using a Metrohm E536 Potentiograph autotitrator, use the following settings:

E536 Potentiograph:	Control Settings
Autocontrol:	OFF
Temperature:	25°C
Feeding Time:	15 min/100°/% volume
Selector Switch:	mV, pH
Measuring Span:	1 V
Buret Size:	25 mL
Reference Electrode:	Double Junction, Orion No. 900200
Indicator Electrode:	Platinum Inlay/Disc. Beckman No. 39273

8. Determine the end point using the concentric arcs technique.
9. Run the standardization in triplicate.

Procedure B—Standardization by Manual Titration

- To a 250 mL beaker, add 100 mL of distilled water.
- Weigh 0.05 g (to nearest 0.1 mg) sodium oxalate that has been previously dried in an oven at 105°C for 2 hours. Quantitatively transfer to the 250 mL beaker and stir to dissolve.
- Add 20 mL of concentrated hydrochloric acid and stir solution for 1 minute.
- Add 5 mL of Iodine/Chloride catalyst solution and stir solution for 1 minute.
- Heat solution to $50 \pm 5^\circ\text{C}$.
- Add 5 drops of Ferroin indicator.
- Place on a magnetic stirrer and titrate solution with the sulfato cerate to be standardized, to a permanent blue end point using a 25 mL buret.
- Run the standardization in triplicate.
- Calculations:
 -

$$\text{N Sulfato Cerate} = \frac{\text{wt of Sodium Oxalate}}{(\text{mL Cerate titrated} \times 67.0)}$$

Where:

67.0 = the equivalent weight of Sodium Oxalate

- Calculate the mean normality (\bar{N}) and standard deviation (s):

$$\bar{N} = \frac{\sum N}{n}$$

Where:

N = the individual normality results

$\sum N$ = the sum of the n normality results

n = the number of replicate results

$$s = \sqrt{\frac{\sum (N - \bar{N})^2}{n-1}}$$

- Laboratory experience at Kodak shows the standardization (s) should be ≤ 0.0002 . Determine the standard deviation (s) for your laboratory.

Storage and Stability

This reagent is stable for 1 month in a glass-stoppered bottle. Never use rubber or similar materials for containers or stoppers.

Sulfuric Acid

- Sulfuric Acid, H_2SO_4 , Conc., 95 to 98%, sp gr 1.84, approx. 36 N

Reagent Grade, ACS Specifications

ECN-2-UL-3275, ECN-0007/1

ECP-0007/1

Sulfato Cerate

Technical Grade

Cleaning Solutions



Warning

Observe safety precautions for handling concentrated acids. Wear eye protection and impervious gloves. Use caution and always add acid slowly to water. Corrosive to skin, metals, and clothing. Avoid contact with liquid and vapor.

Solution Strengths

18 N

ECN-926C

ECP-926C

ECR-929C, ECR-930E, ECR-1603E

7.0 N	Potassium Iodate, Potassium Iodide, Silver Nitrate, Sodium Thiosulfate, Sulfato Cerate ECN-2-1101, ECN-2-1305B, ECN-2-1315, ECN-2-1340, ECN-0005/1, ECN-0020-01, ECN-0022/1, ECN 0024/1 ECP-2-407, ECP-2-1101, ECP-2-1305C, ECP-2-1315, ECP-0005/1, ECP-0023/01, ECP-0025/01, ECP-0026/1 ECR-937, ECR-1101E, ECR-1102C, ECR-1113D, ECR-1125B, ECR-1303, ECR-1305L, ECR-1340A, ECR-1611D, ECR-1612, ECR-1803G D94-0001/1, D94-0002/1
2.5 N	ECN-2-3263, ECN-2-ML-3260
1.0 N	Calcium Chloride/Calcium Hydroxide Buffer ECN-2-755, ECN-0002/1, ECN-0003/1 ECP-2-2010A, ECP-2-2020, ECP-0002/1, ECP-0020-01, ECP-0003/1 ECR-125F, ECR-440B, ECR-617B, ECR-755A, ECR-0001-1
0.25 N	ECP-2-890
0.10 N	Formaldehyde, Methyl Alcohol Containing Thymol Blue ECR-1611D

Standardized

0.1 N	ECN-0001/1 ECP-0001/1 ECR-702J, ECR-1612
0.01 N	Potassium Dichromate

Preparation

Small Volumes

1. To prepare 27 N and 18 N reagent, begin by adding 200 mL and 400 mL, respectively, of distilled water to a 1-litre Pyrex bottle. For the other solutions, begin by adding 500 mL of distilled water.
2. While stirring the water with a magnetic stirrer, add slowly and cautiously the amount of reagent-grade concentrated (36 N) sulfuric acid to the water indicated in the table below.
3. Cool to room temperature and dilute to 1 litre with distilled water.

Desired Sulfuric Acid Solution	=	mL Distilled Water	+	mL Concentrated Sulfuric Acid	+	More Water, mL to Total Volume of
18 N		400		500		1000
7.0 N		500		194		1000
2.5 N		500		69		1000
1.0 N		500		28		1000

0.25 N Sulfuric Acid

1. Pipet (wipe the pipet before leveling) 250.0 mL of standardized 0.1 N sulfuric acid into a 1-litre volumetric flask.
2. Cool to room temperature and dilute to 1 litre with distilled water.

0.01 N Sulfuric Acid

1. Pipet (wipe the pipet before leveling) 100.0 mL of standardized 0.1 N sulfuric acid into a 1-litre volumetric flask.
2. Cool to room temperature and dilute to 1 litre with distilled water.

Large Volumes

1. When preparing larger volumes of 7.0 N or stronger acid, place the Pyrex bottle in a strong rubber or plastic pail. The bottles should not exceed 4 litres for 18 N or 9 litres for 7.0 N acid.
2. Set a magnetic stirrer on the floor. The stirrer must have a platform and base as large as the bottle.
3. Add the volume of distilled water, indicated in the table above, to the bottle.
4. Place a graduated cylinder on the floor, and measure the appropriate volume of acid.
5. Add the acid to the bottle slowly and cautiously while stirring the water.
6. When the solution is thoroughly mixed, stopper the bottle and place the pail in a sink. Remove the stopper and cover the neck of the bottle with a beaker.
7. Cool to room temperature by first running warm water on the side of the bottle, then reduce the temperature of water.
8. Dilute to volume with distilled water.

Standardization

See Reagent Preparation for:

- *Tris(hydroxymethyl)aminomethane*, (Tris)
NIST SRM-723a
- *Methyl Red Indicator*

0.01 N Sulfuric Acid (standardized to 4 decimal places)

1. Weigh 0.1 g (to nearest 0.1 mg) of Tris, NIST SRM-723a, which has been previously dried for 24 hours in a desiccator over anhydrous calcium chloride or equivalent
2. Quantitatively transfer the weighed material to a 150 mL beaker.
3. Add 50 to 75 mL of distilled water. Stir until the standard material is dissolved.
4. Titrate the solution potentiometrically. Use the followings parameters with a Metrohm Potentiograph Model E536 (or equivalent).

E536 Potentiograph:	Control Settings
Autocontrol:	OFF
Feeding Time:	15 min/100% volume
Selector Switch:	mV pH
Measuring Span:	750 mV
Buret Size:	20 mL
Reference Electrode:	Ceramic Junction Calomel Corning No. 476002 or Beckman No. 38423
Indicator Electrode:	Glass pH Rugged Bulb Corning No. 476024

5. Repeat the above procedure two more times.
6. Average the three results and calculate the normality of the sulfuric acid.

Note: Use 5 drops of Methyl red indicator in the standardization procedure if an automatic titrator and electrodes are not used.

7. Calculations

$$N \text{ H}_2\text{SO}_4 = \frac{(\text{Tris g})(1000)}{(\text{mL H}_2\text{SO}_4)(\text{eq wt Tris})}$$

Where:

$$121.1 = \text{eq wt Tris}$$

1 N and 2.5 N Sulfuric Acid (standardized to 4 decimal places)

Standardize these solutions following the above procedure.

1. For 1 N sulfuric acid, weigh 1 g of Tris (to nearest 0.1 mg).
2. For 2.5 N sulfuric acid, weigh 2.5 g of Tris (to nearest 0.1 mg).

Note: Use 5 drops of Methyl red indicator in the standardization procedure if an automatic titrator and electrodes are not used.

Stability and Storage

1. Re-standardize the solution every two months.
2. Store the solution in Pyrex glass bottles.

Thioacetamide

- Thioacetamide, C₂H₅NS

Reagent Grade, ACS Specifications

Preparation

0.01 N Thioacetamide

Reagents

- *Thymol Blue*
 1. Dissolve 0.70 g or reagent quality thioacetamide in approximately 2000 mL of distilled water.
 2. Add approximately 0.5 g of thymol.

Note: The thymol will not dissolve completely. This reagent is stable for two months.

0.00926 N Thioacetamide

Reagents

- *Silver Nitrate*
- *Sodium Chloride*
- *Sodium Thiosulfate*
- *Accelerator Reagent*
 1. Pipet (wipe the pipet before leveling) 5.00 mL of 0.5000 (1/20) normal silver nitrate into each of two 50-mL glass-stoppered graduated cylinders containing 3 mL* of 0.100 N sodium chloride.
 2. Swirl to mix the contents thoroughly.
 3. Add to each, 10 mL of 1.0 M sodium thiosulfate. Swirl to dissolve the precipitate.
 4. Add, stopper, and mix 10 mL of 6 N Accelerator Reagent.
 5. Add, from a buret, 26.70 mL of the 0.01 N thioacetamide to be standardized to one and 27.30 mL to the other.
 6. Stopper and shake vigorously for five seconds.
 7. Filter approximately half the contents of each cylinder through a Whatman No. 3 (12.5 cm diameter) filter paper and collect each filtrate in a beaker containing approximately 5 mL of 0.01 N thioacetamide.
 8. The contents of the beaker from the cylinder containing 26.70 mL of 0.01 N thioacetamide should turn color, and the contents of the beaker from the cylinder containing 27.30 mL of 0.01 N thioacetamide should remain clear. If both conditions are met, the thioacetamide solution is 0.00926 ± 0.0001 N and should be used without modification.
 9. If the filtrate was dark-colored when 27.30 mL of 0.01 N thioacetamide were used (Step 7), the thioacetamide solution is too weak. Discard it and prepare a new one using 0.73 g of thioacetamide.

* Unless otherwise indicated, use the markings on the cylinder for volume measurements.

10. If the filtrate was clear when 26.70 mL of 0.01 N thioacetamide were used, the thioacetamide solution is too strong and must be diluted with water. Add 40 mL of water to what remains of the 2 litres, mix, and retest. Add 40-mL increments of water until the 0.01 N thioacetamide tests within tolerances of 26.70 and 27.30 mL.*

1209

Thymol Blue

- Thymol Blue
Reagent Grade

Tris(hydroxymethyl)aminomethane

- Tris(hydroxymethyl)aminomethane, $\text{NH}_2(\text{CH}_2\text{OH})_3$
NIST Acid/Base Working Standard SRM-723a
(or subsequent standard)

Water

- Water, H_2O
Reagent Grade, ACS Specifications

Note: The methods described in *Processing KODAK Motion Picture Films, Module 3, Analytical Procedures* were developed, and the resulting statistical data were obtained using reagent water equivalent to or purer than Type I Grade, as defined in ASTM Standard D 1193. Other grades of water, e.g., reverse osmosis (RO), demineralized, or distilled water, may give equivalent results, but the effects of water quality on method performance have not been studied.

D94-0001/1, D94-0002/1

**ECN-2-1305B, ECN-2-3263-2, ECN-2-3275-2,
ECN-0001/1, ECN-0002/1, ECN-0004/1, ECN-0005/1,
ECN-0006/1, ECN-0007/1, ECN-0009/1, ECN-0019-01,
ECN-0020-01, ECN-0021/1, ECN-0022/1,
ECN-0023-01, ECN-0024/1, ECN-0025-01**

**ECP-2-1305C, ECP-0001/1, ECP-0002/1, ECP-0004/1,
ECP-0005/1, ECP-0006/1, ECP-0007/1, ECP-0009/1,
ECP-0019-01, ECP-0020-01, ECP-00021/1,
ECP-0023/01, ECP-0025/01, ECP-0026/1, ECP-0027-1**

ECR-1305L, ECR-0004/1

* The nominal titration value for 0.00926 N thioacetamide is 27.00 mL. By accepting as limits 26.70 and 27.30 mL, an error of about $\pm 1\%$ is permitted. The sensitivity of the method is such that an increase in precision can be realized by decreasing these tolerances.

ULM-0001/1, ULM-0004/1, ULM-191-2

Distilled

ECN-2-ML-3260

ECP-2-806

Zinc

- Zinc, Zn, 20-mesh
Reagent Grade, ACS Specifications

ECP-2-936A

Zinc Sulfate

- Zinc Sulfate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$
Reagent Grade, ACS Specifications

Preparation

190 g/L Zinc Sulfate

1. Dissolve 338 grams of zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) in 800 mL of distilled water.
2. Adjust pH to 4 using 1 N sulfuric acid.
3. Dilute to 1 litre, pH = 4.

ECN-0004/1

ECP-0004/1

50 g/L Zinc Sulfate

1. Add 800 mL of distilled water to a 1-litre volumetric flask; stir on a magnetic stirrer.
2. Add 89 g of zinc sulfate heptahydrate ($\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$) to the volumetric flask; stir to dissolve.
3. Dilute to volume with distilled water.

ECR-1113D

Zinc Sulfate/7.0 N Sulfuric Acid Reagent

- 7.0 N Sulfuric Acid, H_2SO_4
Reagent Grade, ACS Specifications
- Zinc Sulfate, $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$
Reagent Grade, ACS Specifications

Preparation

1. Add 500 mL of 7.0 N sulfuric acid to a 1-litre volumetric flask.
2. Add and dissolve 250 g of zinc sulfate.
3. Dilute to volume with 7.0 N sulfuric acid.

ECN-2-1100, ECN-00021/1

ECP-2-1100, ECP-00021/1

ECR-1100G

CHEMICAL SUPPLY SOURCES

The following list of suppliers is not intended to be exclusive, nor are the various suppliers listed in any order of preference. Mention of a supplier is not intended as a recommendation by Eastman Kodak Company. Many of the listed chemicals may be available from local chemical supply houses. For additional suppliers, refer to *Chemical Week 's Guide*, *OPD Chemical Buyer's Directory*, *CPI Purchasing Directory* or *Chemyclopedia*, available in many public libraries.

Supplier Information

Aldrich Chemical Company
PO Box 2060
Milwaukee, WI 53201
414-273-3850 / 800-558-9160

J.T. Baker Chemical Company
222 Red School Lane
Phillipsburg, NJ 08865
201-859-2151 / 800-JTBAKER

Carus Chemical Company
1001 Boyce Memorial Drive
Ottawa, IL 61350
815-433-9070 / 800-435-6856

E.I. DuPont de Nemours & Co., Inc.
1007 Market Street
Wilmington, DE 19898
800-441-9422 / 800-441-7515

Eastman Kodak Company
343 State Street
Rochester, NY 14650
716-458-4014 / 800-225-5352

Fisher Scientific Company
50 Fadem Road
Springfield, NJ 07081-3193
800-766-7000

Floridin Company
5380 Capital Circle, NW
Tallahassee, FL 32303
904-562-5005 / 800-258-2600

FMC Corporation
Chemical Products group
2000 Market Street
Philadelphia, PA 19103
215-299-6000 / 800-526-3649

GFS Chemicals, Inc.
PO Box 245
Powell, OH 43065
614-881-5501 / 800-858-9682

ICI America, Inc., Chemicals Div.
Concord Pike & New Murphy Road
Wilmington, DE 19897
302-886-3000 / 800-456-3669

ICI Biomedicals, Inc.
ICN Biomedicals Div.
4911 Commerce Pkwy.
Cleveland, OH 44128
216-831-3000 / 800-321-6842

Lonza Inc.
17-17 Route 208
Fairlawn, NJ 07410
201794-2400 / 800-777-1875

Mallinckrodt Specialty Chems., Inc.
PO Box 22684
St. Louis, MO 63147
314-539-1241 / 800-325-7155

National Institute of Standards and
Technology, SRM Office
Room B311, Chemistry Building
Gaithersburg, MD 20899
301-975-6775

Peridot Chemicals, Inc.
1333 Broad Street
Clifton, NJ 07015
201-614-3500 / 800-222-0121

Spectrum Chemical Mfg. Corp.
14422 S. San Pedro St.
Gardena, CA 90248-9985
213-516-8000 / 800-772-8786

Union Carbide Corp.
39 Old Ridebury Rd.
Danbury, CT 06817-0001
203-794-5300 / 800-336-7384

U.S. Borax & Chemical Corp.
3075 Wilshire Blvd.
Los Angeles, CA 90010
213-251-5400 / 800-USBORAX

Whittaker Corporation
Heico Division
Route 611
Delaware Water Gap, PA 18327
717-476-0353 / 800-34HEICO

W.R. Grace & Company
55 Hayden Avenue
Lexington, MA 02173
617-861-6600 / 800-GRACENP

General Information

This section lists the chemicals required in the processing solution formulas and analytical procedures used in this process. The grades, specifications, catalog numbers, etc. are those used in Kodak laboratories. Other grades, specifications, catalog numbers may be suitable, but Eastman Kodak Company has had no processing experience with them.

MORE INFORMATION

For more information on motion picture products, call or write to the Professional Motion Imaging office nearest you.

Or access our home page on the Internet, web site address—

<http://www.kodak.com/go/motion/>

You may want to bookmark our location so you can find us more easily.

Processing KODAK Motion Picture Films, Module 4

Reagent Preparation Procedures



**Entertainment
Imaging**