

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*
- *Ferroin Indicator*
- *Hydrochloric Acid*, concentrated



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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 $70 \pm 5^\circ\text{C}$.

- 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.

- 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

- Determine the end point using the concentric arcs technique.
- 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 ± 5°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:

a.

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

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.