
ERRATA

Reagent Chemicals, Ninth Edition

Official from January 1, 2000

The following corrections were inadvertently omitted from the print edition. The corrections include the name of the reagent chemical or solution; the page(s) on which the change appears; the nature of the change; and the text as it should now appear.

Check the Reagent Chemicals Web site regularly for changes and new reagents issued as supplements.

General Directions and Procedures

Sulfate

Page 33. Add a sentence after **Procedure**.

►**Procedure.** Unless otherwise stated, use 12% barium chloride reagent solution.

Reagent Solutions

Barium Chloride 12%

Page 84. Delete 12% from title.

Barium Chloride. Dissolve 120 g of barium chloride dihydrate, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in water, filter, and dilute with water to 1 L.

Dithizone Indicator Solution

Page 85. In the first line, change "25.6 mg" to "25 mg".

Dithizone Indicator Solution. Dissolve 25 mg of dithizone in 100 mL of alcohol. Store in a cold place, and use within 2 months.

Specifications for Reagent Chemicals

Ammonium Metavanadate

Page 151. **Solubility in Ammonium Hydroxide.** In the last line of the test, delete the words “and free from insoluble matter”.

SOLUBILITY IN AMMONIUM HYDROXIDE. Dissolve 5.0 g in a mixture of ammonium hydroxide and 250 mL of hot water, add ammonium hydroxide until the reagent dissolves. Heat the solution to boiling. The solution should be clear.

Boric Acid

Page 193. **Assay.** In the third line of the test, delete the first word, “warm”.

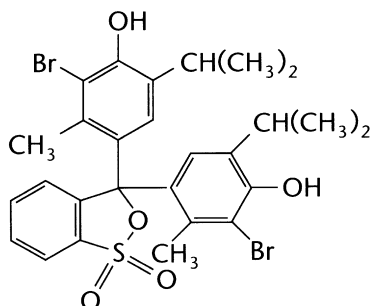
ASSAY. (By acid–base titrimetry). Weigh, to the nearest 0.1 mg, 2.5 g of sample and transfer to a 250-mL glass-stoppered flask. Dissolve in a mixture of 50 mL of water and 75 mL of glycerol, previously neutralized to a pH of 9.0 with 0.1 N sodium hydroxide. Titrate with 1 N sodium hydroxide to a pH of 9.0. One milliliter of 1 N sodium hydroxide corresponds to 0.06183 g of H_3BO_3 .

Nonvolatile with Methanol. In the last line of the test, replace “8” with the degree symbol.

NONVOLATILE WITH METHANOL. To 2.0 g of the sample in a platinum dish, add 25 mL of methanol and 0.5 mL of hydrochloric acid and evaporate to dryness. Repeat the evaporation twice, each with 15 mL of methanol and 0.3 mL of hydrochloric acid. Add to the residue 0.10 mL of sulfuric acid and ignite at 800 ± 25 °C for 15 min.

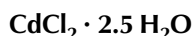
Bromthymol Blue

Page 198. Change the **structural formula** as shown by moving the right-hand “Br” one carbon down.



Cadmium Chloride, Crystals

Page 206. Correct the hydrate part of the **line formula** to appear as follows:



Formula Wt 228.35

Cupferon

Page 256. Correct the **line formula** to read as follows:



Cupric Oxide, Powdered

Page 263. **Calcium, Iron, Potassium, and Sodium.** In Sample Stock Solution B, line two, change “0.01 g” to “0.008 g”.

Sample Stock Solution B. Dilute 10.0 mL of sample stock solution A with water to 50 mL in a volumetric flask (1 mL = 0.008 g).

Ethyl Alcohol, Absolute

Page 294. **Water.** Change “20 mL” to “2 mL” and change “(15.8 g)” to “(1.58 g)”.

WATER. (Page 56, Method 2). Use 2 mL (1.58 g).

Hydrochloric Acid

Page 332. **Requirements.** For **Appearance**, substitute “Passes test” for “Free from suspended matter”. For **Extractable organic substances**, substitute “Passes test (about 5 ppm) for “5 ppm”.

Appearance.....	Passes test
Assay	36.5–38.0% HCl

MAXIMUM ALLOWABLE

Color (APHA)	10
Residue after ignition	5 ppm
Bromide (Br).....	0.005%
Sulfate (SO ₄).....	1 ppm
Sulfite (SO ₃)	1 ppm
Extractable organic substances	Passes test (about 5 ppm)
Free chlorine (Cl)	1 ppm
Ammonium (NH ₄)	3 ppm
Arsenic (As)	0.01 ppm
Heavy metals (as Pb).....	1 ppm
Iron (Fe)	0.2 ppm

Hydrogen Peroxide

Page 341. **Assay.** In the last line, delete one zero directly after the decimal to read “0.01700 g”.

ASSAY. (By titration of the reductive capacity). Weigh accurately about 1 mL in a tared 100-mL volumetric flask, dilute to volume with water, and mix thoroughly. To 20.0 mL of this solution add 20 mL of dilute sulfuric acid (1 + 15) and titrate with 0.1 N potassium permanganate. One milliliter of 0.1 N potassium permanganate corresponds to 0.01700 g of H₂O₂.

Isopropyl Alcohol

Page 354. **Requirements.** For **Carbonyl compounds**, change to appear as follows:

MAXIMUM ALLOWABLE	
Carbonyl Compounds.	0.002% propionaldehyde 0.002% acetone

Magnesium Chloride Hexahydrate

Page 390. **Calcium, Manganese, Potassium, Sodium, and Strontium.** In the AAS Table for the element Na, change the “Standard Added (mg)” from “0.20” to “0.02”.

<i>Element</i>	<i>Wavelength (nm)</i>	<i>Sample Wt (g)</i>	<i>Standard Added (mg)</i>	<i>Flame Type*</i>	<i>Background Correction</i>
Ca	422.7	0.20	0.02; 0.04	N/A	No
Mn	279.5	2.0	0.01; 0.02	A/A	Yes
K	766.5	1.0	0.05; 0.10	A/A	No
Na	589.0	0.20	0.01; 0.02	A/A	No
Sr	460.7	1.0	0.05; 0.10	N/A	No

*A/A is air/acetylene; N/A is nitrous oxide/acetylene.

Perchloric Acid

Page 455. **Requirements.** For **Assay**, add significant figures to make first and second sets of percentages conform to the third set, as follows:

Assay	48.0–50.0% HClO ₄ 60.0–62.0% HClO ₄ 69.0–72.0% HClO ₄
-----------------	--

Phenol

Page 463. **Assay.** In the third line, replace “glass-stoppered volumetric flask” with “glass-stoppered iodine flask”.

ASSAY. (By iodometry after bromination of hydroxyl groups). Weigh accurately about 1.5 g and dissolve in sufficient water to make exactly 1000 mL. Transfer 25 mL into a 500-mL glass-stoppered iodine flask, add 30 mL of 0.1 N bromine and 5 mL of hydrochloric acid, and immediately stopper the flask. Shake frequently during one-half hour and allow to stand for 15 min. Then quickly add 10 mL of 10% potassium iodide, being careful that no bromine escapes, and stopper immediately. Shake, add 1 mL of Chloroform, and titrate the liberated iodine (which represents the excess of bromine) with 0.1 N sodium thiosulfate, using 3 mL of starch indicator solution. One milliliter of 0.1 N bromine corresponds to 0.001568 g of C₆H₅OH.

Potassium Acetate

Page 479. **Calcium, Magnesium, and Sodium.** In the AAS Table for the element Ca, change the "Standard Added (mg)" from "0.005" to "0.05".

<i>Element</i>	<i>Wavelength (nm)</i>	<i>Sample Wt (g)</i>	<i>Standard Added (mg)</i>	<i>Flame Type*</i>	<i>Background Correction</i>
Ca	422.7	0.50	0.025; 0.05	N/A	No
Mg	285.2	0.50	0.01; 0.02	A/A	Yes
Na	589.0	0.03	0.01; 0.02	A/A	No

*A/A is air/acetylene; N/A is nitrous oxide/acetylene.

Potassium Dichromate

Page 499. **Calcium, Iron, and Sodium.** In the AAS Table for the element Fe, change the "Sample Wt (g)" from "2.50" to "2.0". Change the "Standard Added (mg)" from "0.05; 0.10" to "0.04; 0.08".

<i>Element</i>	<i>Wavelength (nm)</i>	<i>Sample Wt (g)</i>	<i>Standard Added (mg)</i>	<i>Flame Type*</i>	<i>Background Correction</i>
Ca	422.7	0.50	0.015; 0.03	N/A	No
Fe	248.3	2.0	0.04; 0.08	A/A	Yes
Na	589.0	0.05	0.01; 0.02	A/A	No

*A/A is air/acetylene; N/A is nitrous oxide/acetylene.

Potassium Hydrogen Sulfate, Fused

Page 509. **Ammonium Hydroxide Precipitate.** In the third and fourth lines, delete the clause ", saving the filtrate separate from the washings for the test for calcium and magnesium precipitate".

AMMONIUM HYDROXIDE PRECIPITATE. Dissolve 20 g in 200 mL of water, filter, add ammonium hydroxide until the solution is alkaline to methyl red, boil for 1 min, and digest in a covered beaker on a hot plate (≈ 100 °C) for 1 h. Filter through a tared filtering crucible. Wash thoroughly and dry at 105 °C.

Potassium Nitrate

Page 517. **Sulfate.** In the first line, change "3.0 g" to "1.7 g".

SULFATE. (Page 33, Method 2). Evaporate 1.7 g using 7 mL of dilute hydrochloric acid (1 + 1). Repeat the evaporation, and heat the last residue for 1 h at 120 °C.

Sodium Bromide

Page 566. **Assay.** In the fifth line, change "ferric ammonium nitrate" to "ferric ammonium sulfate."

ASSAY. (By argentimetric titration of bromide content). Weigh, to the nearest 0.1 mg, 0.4 g of sample. Transfer to a 250-mL titration flask and dissolve in 25 mL of water. Add slowly, while agitating, 50.0 mL of 0.1 N silver nitrate, then add 3 mL of nitric acid and 10 mL of benzyl alcohol, and shake vigorously. Add 2 mL of ferric ammonium sulfate and titrate the excess silver nitrate with 0.1 N ammonium thiocyanate.

Sodium Carbonate Monohydrate

Page 573. **Sulfur Compounds.** In the first line, change “2.0 g” to “0.50 g”.

SULFUR COMPOUNDS. Dissolve 0.50 g of sample in 20 mL of water, evaporate to 5 mL, add 1 mL of bromine water, and evaporate to dryness. Cool. Dissolve in 10 mL of 10% hydrochloric acid and evaporate to dryness. Cool. Add 5 mL of 10% hydrochloric acid and evaporate to dryness. Cool. Dissolve with 10 mL of water and evaporate to dryness. Cool. Dissolve in 10 mL of water and adjust the pH to approximately pH 2 with 10% hydrochloric acid or ammonium hydroxide (1 + 3). Filter through a washed, fine-porosity filter paper, wash with two 2-mL portions of water, and dilute with water to 20 mL.

To prepare 0.06 mg of sulfate ion (SO_4) standard, add 1 mL of (1 + 19) hydrochloric acid and dilute with water to 20 mL.

To sample and standard solutions, add 1 mL of barium chloride reagent solution and allow to stand 30 min. Any turbidity in the sample solution must not exceed that of the standard solution.

Ammonium Hydroxide Precipitate. In the fifth and sixth lines, delete the clause “, reserving the filtrate for the test for calcium and magnesium precipitate”.

AMMONIUM HYDROXIDE PRECIPITATE. Dissolve 10 g in 100 mL of water and filter if necessary. Cautiously add 30 mL of cooled dilute sulfuric acid (1 + 1). Evaporate until dense fumes of sulfur trioxide appear. Cool, dissolve the residue in 130 mL of hot water, and add ammonium hydroxide until the solution is just alkaline to methyl red. Heat to boiling, boil gently for 5 min, and filter. Wash the precipitate with hot water, rejecting the washings, and ignite. The residue includes some, but not all, of the silica in the sample.

Sodium Chloride

Pages 576 and 577. **Iodide.** In the fifth and seventh lines, replace “carbon tetrachloride” with “chloroform”.

IODIDE. Dissolve 11 g in 50 mL of water. Prepare a control by dissolving 1 g of the sample, 0.2 mg of iodide ion (I), and 1.0 mg of bromide ion (Br) in 50 mL of water. To each solution, in a separatory funnel, add 2 mL of hydrochloric acid and 5 mL of ferric chloride reagent solution. Allow to stand for 5 min. Add 10 mL of chloroform, shake for 1 min, allow the chloroform to settle, and draw it off. Reserve the water solution for the test for bromide. Any violet color in the chloroform extract from the solution of the sample should not exceed that in the extract from the control.

Bromide. In the seventh and eleventh lines, replace “carbon tetrachloride” with “chloroform”.

BROMIDE. Treat both the solution of the sample and the control, obtained in the test for iodide, as follows: wash twice by shaking with 10-mL portions of carbon tetrachloride. Each time allow to settle, then draw off and discard the carbon tetrachloride. To each of the water solutions add 10 mL of water, 65 mL of cool, dilute sulfuric acid (1 + 1), and 15 mL of a solution of chromic acid prepared by dissolving 10 g of chromium trioxide (CrO₃) in 100 mL of dilute sulfuric acid (1 + 3). Allow to stand for 5 min. Add 10 mL of chloroform, shake for 1 min, allow to settle, and draw off. (Half of a 7-cm piece of filter paper rolled and placed in the stem of the separatory funnel will absorb any of the aqueous solution that may pass the stopcock and thus assure a clear extract.) Any yellow-brown color in the chloroform extract from the solution of the sample should not exceed that in the extract from the control.

Sodium Fluoride

Page 588. **Assay.** In the formula at the top of the page, change “4.119” to “4.199”.

$$\% \text{ NaF} = (\text{mL NaOH} \times N \text{ NaOH} \times 4.199) / \text{Sample wt (g)}$$

Sodium Phosphate, Monobasic, Monohydrate

Page 614. **Calcium and Potassium.** In the AAS Table for the element K, change the “Flame Type” from “N/A” to “A/A”. In the note to the table, add “A/A is air/acetylene”.

<i>Element</i>	<i>Wavelength (nm)</i>	<i>Sample Wt (g)</i>	<i>Standard Added (mg)</i>	<i>Flame Type*</i>	<i>Background Correction</i>
Ca	422.7	0.40	0.02; 0.04	N/A	No
K	766.5	0.40	0.02; 0.04	A/A	No

*A/A is air/acetylene; N/A is nitrous oxide/acetylene.

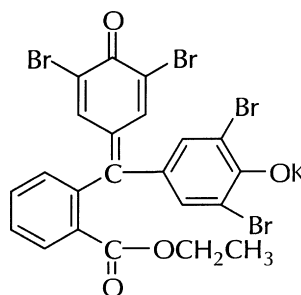
Strontium Chloride Hexahydrate

Page 634. **Assay.** In the last line, change “0.1 N” to “0.1 M”.

ASSAY. (By complexometric titration of strontium). Weigh accurately about 1.0 g of sample, transfer to a 250-mL beaker, and dissolve in 50 mL of water. Add 5 mL of diethylamine and 35 mg of methylthymol blue indicator mixture. Titrate immediately with 0.1 M EDTA until the blue color turns to colorless or gray. One milliliter of 0.1 M EDTA corresponds to 0.02666 g of SrCl₂ · 6H₂O.

Tetrabromophenolphthalein Ethyl Ester

Page 652. Correct the **structural formula** as shown by changing the "OH" functional group on the far right to "OK". Change the **CAS Number** from "1176-74-5" to "62637-91-6". Change the **Formula Wt** from "661.97" to "700.08".



C₂₂H₁₄Br₄O₄

Formula Wt 700.08

CAS Number 62637-91-6

Zinc

Page 692. Add the following **note** on a line between the CAS Number and "Requirements"

NOTE. For arsenic determination.

Zinc Oxide

Page 697. **Sulfur Compounds.** Add the formula that appears below:

$$\% \text{SO}_4 = \frac{\text{Residue wt (g)} \times 13.7}{5.0 \text{ g}}$$