

### 3. Standard Solutions

#### Aluminum Standard Stock Solution

Weigh 1.0 g of aluminum, add 60 ml of diluted hydrochloric acid (1 : 2), and heat to dissolve. Cool, and add water to make 1,000 ml. Measure exactly 10 ml of this solution, add 30 ml of water and 5 ml of ammonium acetate buffer solution (pH 3.0), adjust pH to about 3 adding ammonia TS dropwise. Add 0.5 ml of Cu-PAN TS, and titrate with 0.01 mol/l EDTA in boiling until the red color of the solution changes to yellow and maintain more than one minute. Perform a blank test in the same manner, and make any necessary correction.

1 ml of 0.01 mol/l EDTA = 0.26982 mg of Al

#### Arsenic Standard Solution

Measure exactly 10 ml of Arsenic Standard Stock Solution, add 10 ml of diluted sulfuric acid (1 : 20), and add freshly boiled and cooled water to make exactly 1,000 ml. One ml of this solution contains 1 µg of arsenic trioxide (As<sub>2</sub>O<sub>3</sub>). Prepare freshly before use, and store in a bottle with a ground-glass stopper.

#### Arsenic Standard Stock Solution

Weigh exactly 0.10 g of arsenic trioxide, previously very finely powdered and dried at 105 °C for 4 hours, and dissolve it in 5 ml of sodium hydroxide solution (1 : 5). Neutralize with diluted sulfuric acid (1 : 20), and add 10 ml of diluted sulfuric acid (1 : 20) and freshly boiled and cooled water to make exactly 1,000 ml. One ml of this solution contains 0.1 mg of arsenic trioxide (As<sub>2</sub>O<sub>3</sub>).

#### Barium Chloride Standard Solution

Weigh exactly 4.30 g of barium chloride, and dissolve in water to make exactly 1,000 ml. Perform the gravimetric analysis for barium on this solution, and calculate the amount of sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) equivalent to 1 ml of this solution. One ml of this solution corresponds to about 2.5 mg of sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>).

#### Barium Standard Solution

Weigh exactly 1.779 g of barium chloride, and dissolve in water to make exactly 1,000 ml. One ml of this solution contains 1 mg of barium (Ba).

#### Bromide Ion Standard Stock Solution

Weigh exactly 0.129 g of Sodium Bromide, previously dried at 110 °C for 2 hours, and dissolve it in water to make exactly 1,000 ml. One ml of this solution contains 100 µg of Bromide ion (Br<sup>-</sup>).

#### Chloride Ion Standard Stock Solution

Weigh exactly 0.165 g of sodium chloride (standard reagent), previously dried at 500 - 600 °C for 1 hour, and dissolve it in water to make exactly 1,000 ml. One ml of this solution contains 100 µg of chloride ion (Cl<sup>-</sup>).

#### Chromium Standard Solution

Weigh exactly 0.934 g of potassium chromate, and dissolve in 1 drop of sodium hydroxide solution (1 : 10) and water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add 1 drop of sodium hydroxide solution (1 : 10) and water to make exactly 1,000 ml. One ml of this solution contains 2.5 µg of chromium (Cr).

#### Cyanide Standard Solution

Measure exactly the amount of Cyanide Standard Stock Solution equivalent

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to 10 mg of cyanide ion ( $\text{CN}^-$ ), and add 100 ml of sodium hydroxide solution (1 25) and water to make exactly 1,000 ml. Prepare freshly before use. One ml of this solution contains 0.01 mg of cyanide ion ( $\text{CN}^-$ ).

#### **Cyanide Standard Stock Solution**

Weigh 2.5 g of potassium cyanide, and dissolve in water to make exactly 1,000 ml. Standardize before use. Stopper tightly, and store in a cold, dark place.

*Standardization:* Measure exactly 100 ml of cyanide standard stock solution, and titrate with 0.1 mol/l silver nitrate (indicator: 0.5 ml of *p*-dimethylamino benzylidenerhodanine TS) until a red color develops.

1 ml of 0.1 mol/l silver nitrate = 5.204 mg of CN

#### **Dilute Formaldehyde Standard Solution**

See Formaldehyde Standard Solution, Dilute.

#### **Dimethylamine Hydrochloride Standard Solution**

Weigh exactly 1.116 g of dimethylamine hydrochloride, and dissolve in water to make exactly 1,000 ml. Measure exactly 1 ml of the solution, and add water to make exactly 1,000 ml. One ml of this solution contains dimethylamine hydrochloride equivalent to 1  $\mu\text{g}$  of dimethylformamide ( $\text{C}_3\text{H}_7\text{NO}$ ).

#### **Formaldehyde Standard Solution, Dilute**

Weigh exactly 0.54 g of formalin (equivalent to 37% of HCHO), and add water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add water to make exactly 1,000 ml. One ml of this solution contains 2  $\mu\text{g}$  of formaldehyde (HCHO). Prepare freshly before use.

#### **Iodide Ion Standard Stock Solution**

Weigh exactly 0.118 g of Sodium Iodide, previously dried at 110 for 2 hours, and dissolve it in water to make exactly 1,000 ml. One ml of this solution contains 100  $\mu\text{g}$  of Iodide ion ( $\text{I}^-$ ).

#### **Iron Standard Solution**

Weigh exactly 8.63 g of ferric ammonium sulfate, and dissolve in 20 ml of diluted nitric acid (1 10) and water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add 20 ml of diluted nitric acid (1 10) and water to make exactly 1,000 ml. One ml of this solution contains 0.01 mg of iron (Fe). Store, protecting from light.

#### **Lead Standard Solution**

Measure exactly 10 ml of Lead Standard Stock Solution, and add water to make exactly 100 ml. One ml of this solution contains 10  $\mu\text{g}$  of lead (Pb). Prepare freshly before use.

#### **Lead Standard Stock Solution**

Weigh exactly 0.1599 g of lead nitrate, dissolve in 10 ml of diluted nitric acid (1 10), and add water to make exactly 1,000 ml. One ml of this solution contains 0.1 mg of lead (Pb). In preparation and storage, use glass instruments not containing soluble lead salts.

#### **Lithium Lactate Standard Solution**

Weigh exactly 0.1066 g of lithium lactate, previously dried at 105 for 4 hours, and dissolve it in water to make exactly 1,000 ml. One ml of this solution contains 0.1 mg of lactic acid ( $\text{C}_3\text{H}_6\text{O}_3$ ). Prepare freshly before use.

#### **Manganese Standard Solution**

Weigh exactly 0.2877 g of potassium permanganate, dissolve in 100 ml of

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water and 1 ml of sulfuric acid, add 0.5 g of sodium hydrogen sulfite, and boil. Cool, and add water to make exactly 200 ml. Measure exactly 20 ml of the solution, and add water to make exactly 1,000 ml. One ml of this solution contains 0.01 mg of manganese (Mn).

#### Matching Fluid

According to the table below, transfer the prescribed volumes of Colorimetric Standard Stock Solutions prepared as directed under Colorimetric Standard Stock Solution (CSSS) and water into a test tube, using burets or pipets with 0.1-ml or more precise graduations, and mix.

Symbol for Matching Fluid	Volume (ml) of Cobaltous Chloride CSSS	Volume (ml) of Ferric Chloride CSSS	Volume (ml) of Cupric Sulfate CSSS	Volume (ml) of Water
A	0.1	0.4	0.1	4.4
B	0.3	0.9	0.3	3.5
C	0.1	0.6	0.1	4.2
D	0.3	0.6	0.4	3.7
E	0.4	1.2	0.3	3.1
F	0.3	1.2	0.0	3.5
G	0.5	1.2	0.2	3.1
H	0.2	1.5	0.0	3.3
I	0.4	2.2	0.1	2.3
J	0.4	3.5	0.1	1.0
K	0.5	4.5	0.0	0.0
L	0.8	3.8	0.1	0.3
M	0.1	2.0	0.1	2.8
N	0.0	4.9	0.1	0.0
O	0.1	4.8	0.1	0.0
P	0.2	0.4	0.1	4.3
Q	0.2	0.3	0.1	4.4
R	0.3	0.4	0.2	4.1
S	0.2	0.1	0.0	4.7
T	0.5	0.5	0.4	3.6

#### Colorimetric Standard Stock Solution (CSSS)

Prepare by the following manners, and store in a bottle with a ground-glass stopper.

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#### Cobaltous Chloride CSSS

Weigh about 65 g of cobaltous chloride, and dissolve in diluted hydrochloric acid (1 : 40) to make 1,000 ml. Measure exactly 5 ml of the solution, transfer into a 250-ml flask with a ground-glass stopper, add 5 ml of hydrogen peroxide TS and 15 ml of sodium hydroxide solution (1 : 5), and boil for 10 minutes. Cool, and add 2 g of potassium iodide and 20 ml of diluted sulfuric acid (1 : 4). After the precipitate is dissolved, titrate the solution with 0.1 mol/l sodium thiosulfate (indicator: starch TS). One ml of 0.1 mol/l sodium thiosulfate corresponds to 23.79 mg of cobaltous chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ , molecular weight: 237.93). To the remaining portion of the cobaltous chloride solution, add diluted hydrochloric acid (1 : 40) to make the solution containing 59.5 mg of cobaltous chloride ( $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ ) per ml.

#### Cupric Sulfate CSSS

Weigh about 65 g of cupric sulfate, and dissolve in diluted hydrochloric acid (1 : 40) to make 1,000 ml. Measure exactly 10 ml of the solution, transfer into a 250-ml flask with a ground-glass stopper, and add 40 ml of water. Add 4 ml of diluted acetic acid (1 : 4) and 3 g of potassium iodide, and titrate with 0.1 mol/l sodium thiosulfate (indicator: starch TS). One ml of 0.1 mol/l sodium thiosulfate equivalent to 24.97 mg of cupric sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , molecular weight: 249.69). To the remaining portion of the cupric sulfate solution, add diluted hydrochloric acid (1 : 40) to make the solution containing 62.4 mg of cupric sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ) per ml.

#### Ferric Chloride CSSS

Weigh about 55 g of ferric chloride, and dissolve in diluted hydrochloric acid (1 : 40) to make 1,000 ml. Measure exactly 10 ml of the solution, transfer into a 250-ml flask with a ground-glass stopper, and add 15 ml of water and 3 g of potassium iodide. Stopper tightly, and allow to stand in a dark place for 15 minutes. Add 100 ml of water, and titrate with 0.1 mol/l sodium thiosulfate (indicator: starch TS). One ml of 0.1 mol/l sodium thiosulfate is equivalent to 27.03 mg of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ , molecular weight: 270.30). To the remaining portion of the ferric chloride solution, add diluted hydrochloric acid (1 : 40) to make the solution containing 45.0 mg of ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) per ml.

#### **Mercury Standard Solution**

Weigh exactly 0.135 g of mercuric chloride, and dissolve in 10 ml of diluted nitric acid (1 : 10) and water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add 10 ml of diluted nitric acid (1 : 10) and water to make exactly 1,000 ml. Measure exactly 10 ml of the resulting solution, and add 10 ml of diluted nitric acid (1 : 10) and water to make exactly 100 ml. One ml of this solution contains 0.1  $\mu\text{g}$  of mercury (Hg). Prepare freshly before use.

#### **Monopotassium Phosphate Standard Solution**

Weigh exactly 4.394 g of monopotassium phosphate, and dissolve in water to make exactly 1,000 ml. One ml of this solution contains 1 mg of phosphorus (P).

#### **Nickel Standard Solution**

Weigh exactly 6.73 g of ammonium nickel sulfate, dissolve in water to make exactly 1,000 ml. Measure exactly 5 ml of the solution, and add water to make

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exactly 1,000 ml. One ml of this solution contains 0.005 mg of nickel (Ni).

#### Nitrate Ion Standard Stock Solution

See Nitrate Standard Solution.

#### Nitrate Standard Solution

Weigh exactly 1.631 g of potassium nitrate and dissolve in water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add water to make exactly 100 ml. One ml of this solution contains 0.1 mg of nitrate ion ( $\text{NO}_3^-$ ).

#### Phosphate Standard Solution

Weigh exactly 0.1433 g of monopotassium phosphate, and dissolve in water to make exactly 100 ml. Measure exactly 10 ml of the solution, and add water to make exactly 1,000 ml. One ml of this solution contains 0.01 mg of phosphate ion ( $\text{PO}_4^{3-}$ ).

#### Sulfate Ion Standard Stock Solution

Weigh exactly 0.148 g of Sodium Sulfate, previously dried at 110 °C for 2 hours, and dissolve it in water to make exactly 1,000 ml. One ml of this solution contains 100 µg of sulfate ion ( $\text{SO}_4^{2-}$ ).

#### Tyrosine Standard Solution

Dry Tyrosine Reference Standard at 105 °C for 3 hours, weigh exactly 0.050 g of the dried Tyrosine Reference Standard, and dissolve in 0.1 mol/l hydrochloric acid to make exactly 50 ml. Measure exactly 5 ml of this solution, add 0.1 mol/l hydrochloric acid to make exactly 100 ml.

#### Water - Methanol Standard Solution

Measure 500 ml of methanol for Water Determination, transfer into a dry 1,000-ml volumetric flask, and add 2 ml of water and methanol for Water Determination to make 1,000 ml. The standardization of this solution is done immediately after water determination TS is standardized.

Store in a cold place, protecting from light and moisture.

*Standardization:* According to the procedure directed under Water Determination, transfer 25 ml of methanol for Water Determination into a dry titration flask, and add carefully TS for Water Determination to the end point. Add exactly 10 ml of water determination TS and titrate with Water-Methanol Standard Solution to the end point. Calculate the number of mg of water ( $\text{H}_2\text{O}$ ),  $f'$ , contained in 1 ml of Water-Methanol Standard Solution by the following formula:

$$f' = \frac{f \times 10}{\text{Volume (ml) of Water-Methanol Standard Solution titrated}}$$

Where  $f$  = The number of mg of water ( $\text{H}_2\text{O}$ ) equivalent to 1 ml of water determination TS.

#### Zinc Standard Solution

Weigh exactly 4.40 g of zinc sulfate, and dissolve in water to make exactly 1,000 ml. Measure exactly 10 ml of the solution, and add water to make exactly 1,000 ml. One ml of this solution contains 0.01 mg of zinc (Zn).