(1:1), boil gently for 10 minutes under a reflux condenser, cool, and add 5 drops of phenolphthalein TS and 0.60 mL of 0.1 mol/L sodium hydroxide VS: a red color develops.

(2) Rancidity—No unpleasant odor of rancid oil is perceptible by warming Vitamin A Oil.

(3) Related substances—Vitamin A Oil meets the conditions determined as directed in Method 1 under the Vitamin A Assay, or its *f* value determined as directed in Method 2 under the Vitamin A Assay is not less than 0.85.

Assay Proceed as directed under the Vitamin A Assay.

Containers and storage Containers—Tight containers.

Storage—Light-resistant, and almost well-filled, or under nitrogen atmosphere.

Vitamin A Oil Capsules

Vitamin A Capsules

ビタミンA油カプセル

Vitamin A Oil Capsules contain not less than 90% and not more than 130% of the labeled Units of Vitamin A.

Method of preparation Prepare as directed under Capsules, using Vitamin A Oil.

Tests for Vitamin A Oil The oil obtained by the procedure directed in the Assay meets the requirements of the Description, Identification and Purity under Vitamin A Oil.

Assay Weigh accurately 20 Vitamin A Oil Capsules, cut open, transfer the oil contents, mix well, and proceed with the oil as directed under Vitamin A Assay. Wash the capsules with a small amount of diethyl ether, allow to stand at room temperature to evaporate the diethyl ether, and weigh accurately. Calculate the mass of Vitamin A Oil from the difference between the masses before and after the above-described procedure. Calculate the Vitamin A Units per 1 capsule from the mass and the Vitamin A Units of the oil.

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Compound Vitamin B Powder

複方ビタミンB散

Method of preparation

Thiamine Nitrate	10 g
Riboflavin	10 g
Pyridoxine Hydrochloride	10 g
Nicotinamide	100 g
Starch, Lactose or their mixture	a sufficient quantity
To make	1000 g

Prepare as directed under Powders, with the above ingredients.

Description Compound Vitamin B Powder is orange-yellow in color. It has a slighly bitter taste.

It is slowly affected by light.

Identification (1) Shake 2 g of Compound Vitamin B Powder with 100 mL of water, filter, and to 5 mL of the filtrate add 2.5 mL of sodium hydroxide TS and 0.5 mL of potassium hexacyanoferrate (III) TS. Then add 5 mL of 2-methyl-1-propanol, shake the mixture vigorously for 2 minutes, allow to stand, and observe under ultraviolet light: the 2-methyl-1-propanol layer shows a blue-purple fluorescence. This fluorescence disappears when the mixture is acidified, but reappears when it is again made alkaline (thiamine).

(2) Shake 0.1 g of Compound Vitamin B Powder with 100 mL of water, and filter. Perform the following tests with the filtrate (riboflavin).

(i) The filtrate is light yellow-green in color and has an intense yellow-green fluorescence. This color and fluorescence of the solution disappears upon the addition of 0.02 g of sodium hydrosulfite to 5 mL of the filtrate, and again appears by shaking the mixture in air. This fluorescence disappears upon the addition of dilute hydrochloric acid or sodium hydroxide TS.

(ii) To 10 mL of the filtrate placed in a glass-stoppered test tube add 1 mL of sodium hydroxide TS, after illuminating with a fluorescence lamp of 10 to 30 watts at 20-cm distance for 30 minutes between 20°C and 40°C, acidify with 0.5 mL of acetic acid (31), and shake thoroughly with 5 mL of chloroform: the chloroform layer shows yellow-green fluorescence.

(3) Shake 1 g of Compound Vitamin B Powder with 100 mL of diluted ethanol (7 in 10), filter, and to 5 mL of the filtrate add 2 mL of sodium hydroxide TS and 40 mg of manganese dioxide. Heat on a water bath for 30 minutes, cool, and filter. Add 5 mL of 2-propanol to 1 mL of the filtrate, and use the solution as the sample solution. To 3 mL of the sample solution add 2 mL of bartibal buffer solution, 4 mL of 2-propanol and 2 mL of a freshly prepared solution of 2,6-dibromo-N-chloro-1,4-benzoquinone monoimine in ethanol (95) (1 in 4000) prepared when required for use: a blue color develops. To 1 mL of the sample solution add 1 mL of a saturated boric acid solution, and proceed as directed in the same manner as above: no blue color develops (pyridoxine).

(4) Shake 0.5 g of Compound Vitamin B Powder with 10 mL of ethanol (95), filter, and evaporate 1 mL of the filtrate on a water bath to dryness. Add 0.01 g of 2,4-dinitrochlorobenzen to the residue, heat gently for 5–6 seconds to fuse, and after cooling, add 4 mL of potassium hydroxide-ethanol TS: a red color develops (nicotinamide).

(5) Shake 1 g of Compound Vitamin B Powder with 5 mL of diluted ethanol (7 in 10), filter, and use the filtrate as the sample solution. Separately, dissolve 0.01 g each of thiamine mononitrate, riboflavin, pyridoxine hydrochloride and nicotinamide in 1 mL, 50 mL, 1 mL and 1 mL of water, respectively, and use these solutions as standard solutions (1), (2), (3) and (4). Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 2 μL each of the sample solution and standard solutions (1), (2), (3) and (4) on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, ethanol (95) and acetic acid (100) (100:50:1) to a distance of about 10 cm, and air-dry the plate. Examine un-

der ultraviolet light (broad spectrum wavelength): four spots from the sample solution show the same color tone and the same *Rf* value as the corresponding spots from standard solutions (1), (2), (3) and (4).

Containers and storage Containers—Well-closed containers.

Storage—Light-resistant.

Water

常水

H_2O: 18.02

Water usually means tap water and well water.

Description Water occurs as a clear, colorless liquid.

pH 5.8 - 8.6

Purity **(1)** Color and turbidity—View downward 50 mL of Water placed in a Nessler tube against white and black backgrounds: it is clear and colorless.

(2) Odor and taste—Place 100 mL of Water in a 300-mL glass-stoppered Erlenmeyer flask, shake vigorously at ordinary temperature, and check the odor and taste. Then stopper the flask loosely, warm between 40°C and 50°C, and check the odor and taste again as soon as the flask is opened: it has no foreign odor (except a slight chlorine odor) and no foreign taste (except a slight chlorine taste).

(3) Chlorine ion—Pipet 50 mL of Water, and titrate with 0.01 mol/L silver nitrate VS against a white background until a pale red-brown color no longer disappears in the aqueous layer (indicator: 0.5 mL of silver chromate-saturated potassium chromate TS). The concentration of chlorine ion in Water, when calculated from the amount *a* (mL) of 0.01 mol/L silver nitrate VS consumed by using the following equation, is not more than 200 mg/L.

$$\text{The concentration (mg/L) of chlorine ion} = 0.35453 \times a \times \frac{1000}{50}$$

(4) Nitrogen from nitrates—Place 2.0 mL of Water in a 50-mL beaker, add 1 mL of sodium salicylate-sodium hydroxide TS, 1 mL of a solution of sodium chloride (1 in 500) and 1 mL of a solution of ammonium amidosulfate (1 in 1000), and evaporate to dryness on a water bath. After cooling, add 2 mL of sulfuric acid, allow to stand for 10 minutes with occasional shaking, add 10 mL of water, and transfer to a Nessler tube. After cooling, add slowly 10 mL of a solution of sodium hydroxide (2 in 5) and water to make 25 mL. Then view the Nessler tube downward or transversely: the solution has no more color than the following control solution.

Control solution: Pipet 2.0 mL of Standard Nitric Acid Solution, and proceed in the same manner as for the test solution (not more than 10 mg/L).

(5) Nitrogen from nitrites—Place 50 mL of Water in a Nessler tube, add 0.3 g of Griess-Romijin's nitrous acid reagent, dissolve with shaking, and allow to stand for 10 minutes: no light red color develops.

(6) Ammonium—Perform the test using 30 mL of Water as directed under the Ammonium Limit Test. Prepare the control solution with 0.15 mL of Standard Ammonium Solution, dilute with purified water for ammonium limit test to make 30 mL, and proceed in the same manner as for the test solution (not more than 0.05 mg/L).

(7) Cyanide—Place 20 mL of Water in a Nessler tube, add 5 mL of phosphate buffer solution, pH 6.8, and 1.0 mL of diluted sodium toluensulfonchloramide TS (1 in 5), stopper immediately, mix gently, allow to stand for 2 to 3 minutes, add 5 mL of pyridine-pyrazolone TS, mix well, and allow to stand between 20°C and 30°C for 50 minutes: the solution has no more color than the control solution.

Control solution: Pipet 1.0 mL of Standard Cyanide Solution, and dilute with water to make exactly 1000 mL. Place 20 mL of this solution in a Nessler tube, and proceed in the same manner as for the test solution (not more than 0.01 mg/L).

(8) Heavy metals—Proceed with 30 mL of Water, and perform the test according to Method 1. Prepare the control solution with 3.0 mL of Standard Lead Solution (not more than 1 mg/L).

(9) Iron—Prepare the test solution with 50.0 mL of Water according to Method 1, and perform the test according to Method B. Prepare the control solution with 1.5 mL of Standard Iron Solution (not more than 0.3 ppm).

(10) Zinc—Shake 50 mL of Water with 0.5 mL of nitric acid, allow to stand for 1 hour, and use this solution as the sample solution. Separately, dilute 2.0 mL of Standard Zinc Solution with water to make exactly 50 mL, add 0.5 mL of nitric acid, and use this solution as the standard solution. Perform the tests with these solutions as directed under the Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution (not more than 1 mg/L).

Gas: Combustible gas—Acetylene or hydrogen
Supporting gas—Air
Lamp: Zinc hollow-cathode lamp
Wavelength: 213.9 nm

(11) Cadmium—Shake 50 mL of Water with 0.5 mL of nitric acid, and allow to stand for 1 hour. To this solution add 10 mL of a solution of diammonium hydrogen citrate (1 in 4) and 2 drops of bromothymol blue TS, and add ammonia TS until the color of the solution changes from yellow to green. Then add 10 mL of a solution of ammonium sulfate (2 in 5) and 5 mL of a solution of sodium *N,N*-diethyldithiocarbamate trihydrate (1 in 20), mix, allow to stand for several minutes, add 10.0 mL of 4-methyl-2-pentanone, and shake vigorously. Allow to stand, separate the 4-methyl-2-pentanone layer, and use this solution as the sample solution. Separately, take 0.50 mL of Standard Cadmium Solution, add water to make exactly 50 mL, add 0.5 mL of nitric acid, 10 mL of a solution of diammonium hydrogen citrate (1 in 4) and 2 drops of bromothymol blue TS, proceed in the same manner as for the sample solution, and use this solution as the standard solution. Perform the tests with these solutions as directed under the Atomic Absorption Spectrophotometry according to the following conditions: the absorbance of the sample solution is not more than that of the standard solution (not more than 0.01 mg/L).

Gas: Combustible gas—Acetylene or hydrogen
Supporting gas—Air
Lamp: Cadmium hollow-cathode lamp