Amount (mg) of hyoscyamine ($C_{17}H_{23}NO_3$)
= amount (mg) of Atropine Sulfate Reference Standard, calculated on the dried basis
$\times \frac{Q_T}{Q_S} \times \frac{1}{5} \times 0.855$

Internal standard solution—A solution of brucine dihydrate in the mobile phase (1 in 2500).

Operating conditions—

Detector: An ultraviolet absorption spectrometer (wavelength: 210 nm).

Column: A stainless steel column about 4 mm in inside diameter and about 15 cm in length, packed with octadesilcylanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 20°C.

Mobile phase: Dissolve 6.8 g of potassium dihydrogenphosphate in 900 mL of water, add 10 mL of triethylamine, adjust with phosphoric acid to a pH of 3.5, and add water to make 1000 mL, and mix this solution with acetonitrile (9:1).

Flow rate: Adjust the flow rate so that the retention time of atropine is about 14 minutes.

Selection of column: Proceed with 10 μL of the standard solution under the above operating conditions, and determine the resolution. Use a column giving elution of atropine and the internal standard in this order with the resolution between these peaks being not less than 4.

Bentonite

ベントナイト

Bentonite is a natural, colloidal, hydrated aluminum silicate.

Description Bentonite occurs as a very fine, white to light yellow-brown powder. It is odorless. It has a slightly earthy taste.

It is practically insoluble in water and in diethyl ether.

It swells in water.

Identification **(1)** Add 0.5 g of Bentonite to 3 mL of diluted sulfuric acid (1 in 3), and heat until white fumes are evolved. Cool, add 20 mL of water, and filter. To 5 mL of the filtrate add 3 mL of ammonia TS: a white, gelatinous precipitate is produced, which turns red on the addition of 5 drops of alizarin red S TS.

(2) Wash the residue obtained in (1) with water, add 2 mL of a solution of methylene blue trihydrate (1 in 10,000), and wash again with water: the residue is blue in color.

pH To 1.0 g of Bentonite add 50 mL of water, and shake: the pH of the suspension is between 9.0 and 10.5.

Purity **(1)** Heavy metals—To 1.5 g of Bentonite add 80 mL of water and 5 mL of hydrochloric acid, and boil gently for 20 minutes with thorough stirring. Cool, centrifuge, collect the supernatant liquid, wash the residue with two 10-mL portions of water, and centrifuge each. Combine the supernatant liquid and the washings, and add dropwise ammonia solution (28). When a precipitate is produced, add dropwise dilute hydrochloric acid with vigorous stirring, and dissolve. To the solution add 0.45 g of hydroxylammonium chloride, and heat. Cool, and add 0.45 g of sodium acetate trihydrate, 6 mL of dilute acetic acid and water to make 150 mL. Pipet 50 mL of the solution, and perform the test using this solution as the test solution. Prepare the control solution as follows: mix 2.5 mL of Standard Lead Solution, 0.15 g of hydroxylammonium chloride, 0.15 g of sodium acetate trihydrate, and 2 mL of dilute acetic acid, and add water to make 50 mL (not more than 50 ppm).

(2) Arsenic—To 1.0 g of Bentonite add 5 mL of dilute hydrochloric acid, and gently heat to boil while stirring well. Cool immediately, and centrifuge. To the residue add 5 mL of dilute hydrochloric acid, shake well, and centrifuge. To the residue add 10 mL of water, and perform the same operations. Combine all the extracts, and heat on a water bath to concentrate to 5 mL. Perform the test with this solution as the test solution using Apparatus B (not more than 2 ppm).

(3) Foreign matter—Place 2.0 g of Bentonite in a mortar, add 20 mL of water to swell, disperse evenly with a pestle, and dilute with water to 100 mL. Pour the suspension through a No. 200 (74 μm) sieve, and wash the sieve thoroughly with water. No grit is felt when the fingers are rubbed over the wire mesh of the sieve.

Loss on drying 5.0 - 10.0% (2 g, 105°C, 2 hours).

Gel formation Mix 6.0 g of Bentonite with 0.30 g of magnesium oxide. Add the mixture, in several portions, to 200 mL of water contained in a glass-stoppered 500-mL cylinder. Agitate for 1 hour, transfer 100 mL of the suspension to a 100-mL graduated cylinder, and allow to stand for 24 hours: not more than 2 mL of supernatant appears on the surface.

Swelling power To 100 mL of water in a glass-stoppered 100-mL cylinder add 2.0 g of Bentonite in ten portions, allowing each portion to settle before adding the next, and allow to stand for 24 hours: the apparent volume of the sediment at the bottom is not less than 20 mL.

Containers and storage Containers—Well-closed containers.

Benzoin

Benzoinum

アンソッコウ

Benzoin is the resin obtained from *Styrax benzoin* Dryander or other species of the same genus (*Styracaceae*).

Description Benzoin occurs as grayish brown to dark red-brown blocks varying in size; the fractured surface exhibiting whitish to light yellow-red grains in the matrix; hard and brittle at ordinary temperature but softened by heat. Odor, characteristic and aromatic; taste, slightly pungent and acrid.

Identification **(1)** Heat a fragment of Benzoin in a test tube: it evolves an irritating vapor, and a crystalline sublimate is produced.

(2) Digest 0.5 g of Benzoin with 10 mL of diethyl ether, decant 1 mL of the diethyl ether into a porcelain dish, and

add 2 to 3 drops of sulfuric acid: a deep red-brown to deep red-purple color develops.

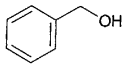
Purity Ethanol-insoluble substances—Boil gently 1.0 g of Benzoin with 30 mL of ethanol (95) on a water bath for 15 minutes under a reflux condenser. After cooling, collect the insoluble substances through a tared glass filter (G3), and wash with three 5-mL portions of ethanol (95). Dry the residue at 105°C for 4 hours: the mass of the residue does not exceed 0.30 g.

Total ash Not more than 2.0%.

Acid-insoluble ash Not more than 1.0%.

Benzyl Alcohol

ベンジルアルコール



C_7H_8O: 108.14
Benzyl alcohol [*100-51-6*]

Benzyl Alcohol contains not less than 98.0% of C_7H_8O.

Description Benzyl Alcohol is a clear, colorless liquid. It is odorless, or has a faint, aromatic odor. It has an irritative, burning taste.

It is miscible with ethanol (95) and with diethyl ether.

It is soluble in water.

It is gradually affected by air and by light with decreasing solubility in water.

Identification Add 1 mL of Benzyl Alcohol to 5 mL of a solution of potassium permanganate (1 in 20), add 2 mL of dilute sulfuric acid, and shake for 2 minutes. Add 20 mL of chloroform, and shake well. Evaporate the chloroform layer on a water bath: the residue has the odor of benzaldehyde. Dissolve the residue in 5 mL of aldehyde-free ethanol, and add 1 mL of 2,4-dinitrophenylhydrazine TS: an orange precipitate is produced.

Refractive index n_D^{20}: 1.538 - 1.541

Specific gravity d_{20}^{20}: 1.043 - 1.053

Purity **(1)** Clarity and color of solution—Dissolve 1.0 mL of Benzyl Alcohol in 40 mL of water: the solution is clear and colorless.

(2) Acid—To 10 mL of Benzyl Alcohol add 10 mL of neutralized ethanol, 0.20 mL of 0.1 mol/L sodium hydroxide VS and 2 drops of phenolphthalein TS: a red color develops.

(3) Benzaldehyde—Dissolve 1.0 mL of Benzyl Alcohol in water to make 100 mL. Transfer 10.0 mL of the solution to a Nessler tube, add water to make 25 mL, add 1 mL of 2,4-dinitrophenyhydrazine TS, mix, and allow to stand for 5 minutes: no turbidity nor floating matters forms.

(4) Chlorinated compounds—Perform the test with 2 drops of Benzyl Alcohol according to the Flame Coloration Test (2): no green color appears.

Residue on ignition Not more than 0.005% (20 g, after evaporation).

Distilling range 202.5 - 206.5°C, not less than 96.0 v/v%.

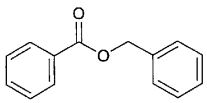
Assay Weigh accurately about 0.5 g of Benzyl Alcohol, add exactly 10 mL of a mixture of pyridine and acetic anhydride (17:3), and heat on a water bath under a reflux condenser for 30 minutes. Cool, add 25 mL of water, and titrate the excess acetic acid with 1 mol/L sodium hydroxide VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination.

Each mL of 1 mol/L sodium hydroxide VS
= 108.14 mg of C_7H_8O

Containers and storage Containers—Tight containers.
Storage—Light-resistant.

Benzyl Benzoate

安息香酸ベンジル



$C_{14}H_{12}O_2$: 212.24
Benzyl benzoate [*120-51-4*]

Benzyl Benzoate contains not less than 99.0% of $C_{14}H_{12}O_2$.

Description Benzyl Benzoate is a colorless, clear, viscous liquid. It has a faint, aromatic odor and a pungent, burning taste.

It is miscible with ethanol (95) and with diethyl ether.

It is practically insoluble in water.

Congealing point: about 17°C

Specific gravity d_{20}^{20}: about 1.123

Boiling point: about 323°C

Identification **(1)** Heat gently 1 mL of Benzyl Benzoate with 5 mL of sodium carbonate TS and 2 mL of potassium permanganate TS: the odor of benzaldehyde is perceptible.

(2) Warm the titrated mixture obtained in the Assay on a water bath to remove ethanol, and add 0.5 mL of iron (III) chloride TS: a light yellow-red precipitate is produced, which turns white on the addition of dilute hydrochloric acid.

Refractive index n_D^{20}: 1.568 - 1.570

Purity Acid—Dissolve 5.0 mL of Benzyl Benzoate in 25 mL of neutralized ethanol, and add 0.50 mL of 0.1 mol/L sodium hydroxide VS: a red color develops.

Residue on ignition Not more than 0.05% (2 g).

Assay Weigh accurately about 2 g of Benzyl Benzoate, add exactly 50 mL of 0.5 mol/L potassium hydroxide-ethanol VS, and boil gently for 1 hour under a reflux condenser with a carbon dioxide-absorbing tube (soda lime). Cool, and titrate the excess potassium hydroxide with 0.5 mol/L hydrochloric acid VS (indicator: 2 drops of phenolphthalein TS). Perform a blank determination.