similar intensities of absorption at the same wavelengths.

(4) To 1 mL of a solution of Benzalkonium Chloride Concentrated Solution 50 (1 in 50) add 2 mL of ethanol (95), 0.5 mL of dilute nitric acid and 1 mL of silver nitrate TS: a white precipitate is produced. This precipitate does not dissolve on the addition of dilute nitric acid, but dissolves on the addition of ammonia TS.

Purity **(1)** Clarity and color of solution—Dissolve 2.0 g of Benzalkonium Chloride Concentrated Solution 50 in 10 mL of water: the solution is clear and colorless to light yellow.

(2) Petroleum ether-soluble substances—To 6.0 g of Benzalkonium Chloride Concentrated Solution 50 add water to make 50 mL, then add 50 mL of ethanol (99.5) and 5 mL of 0.5 mol/L sodium hydroxide TS, and extract with three 50-mL portions of petroleum ether. Combine the petroleum ether extracts, and wash with three 50-mL portions of dilute ethanol. After shaking well with 10 g of anhydrous sodium sulfate, filter through a dry filter paper, and wash the filter paper with two 10-mL portions of petroleum ether. Evaporate the petroleum ether on a water bath by heating, and dry the residue at 105°C for 1 hour: the residue is not more than 1.0%.

Residue on ignition Not more than 0.20% (1 g).

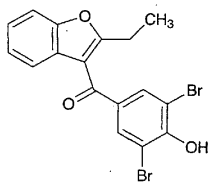
Assay Weigh accurately about 0.30 g of Benzalkonium Chloride Concentrated Solution 50, and dissolve in 75 mL of water. Adjust the pH to between 2.6 and 3.4 by adding dropwise diluted dilute hydrochloric acid (1 in 2), add 1 drop of methyl orange TS, and titrate with 0.02 mol/L sodium tetraphenylboron VS until the color of the solution becomes red.

Each mL of 0.02 mol/L sodium tetraphenylborate VS
= 7.080 mg of $C_{22}H_{40}ClN$

Containers and storage Containers—Tight containers.

Benzbromarone

ベンズブロマロン



$C_{17}H_{12}Br_2O_3$: 424.08
3,5-Dibromo-4-hydroxyphenyl 2-ethylbenzofuran-3-yl ketone [*3562-84-3*]

Benzbromarone, when dried, contains not less than 98.5% of $C_{17}H_{12}Br_2O_3$.

Description Benzbromarone occurs as a white to light yellow, crystalline powder. It is odorless and tasteless.

It is very soluble in *N,N*-dimethylformamide, freely soluble in acetone and in chloroform, soluble in diethyl ether, sparingly soluble in ethanol (95), and practically insoluble in water.

Identification **(1)** Dissolve 0.02 g of Benzbromarone in 1 mL of chloroform, and add 4 drops of dehydrated iron (III) chloride-pyridine TS: a blue-purple color develops.

(2) Dissolve 0.1 g of Benzbromarone in 10 mL of ethanol (95), add 3 mL of 2,4-dinitrophenylhydrazine TS, and heat in a water bath for 20 minutes: a red precipitate is formed.

(3) Determine the infrared absorption spectrum of Benzbromarone, previously dried, as directed in the potassium bromide disk method under the Infrared Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wave numbers.

(4) To 0.1 g of Benzbromarone in a porcelain crucible add 1 g of anhydrous sodium carbonate, and fuse and incinerate at 700°C. After cooling, dissolve the residue in 50 mL of water by warming, and neutralize with dilute nitric acid: the solution responds to the Qualitative Tests for bromide.

Melting point 149 - 153°C

Purity **(1)** Clarity and color of solution—Dissolve 1.0 g of Benzbromarone in 10 mL of chloroform: the solution is clear and colorless to light yellow.

(2) Sulfate—Dissolve 1.0 g of Benzbromarone in 40 mL of acetone, and add 1 mL of dilute hydrochloric acid and water to make 50 mL. Perform the test using this solution as the test solution. Prepare the control solution as follows: to 0.40 mL of 0.005 mol/L sulfuric acid VS add 40 mL of acetone, 1 mL of dilute hydrochloric acid and water to make 50 mL (not more than 0.019%).

(3) Soluble halides—Dissolve 0.5 g of Benzbromarone in 40 mL of acetone, and add 6 mL of dilute nitric acid and water to make 50 mL. Proceed with this solution as directed under the Chloride Limit Test. Prepare the control solution as follows: to 0.25 mL of 0.01 mol/L hydrochloric acid VS add 40 mL of acetone, 6 mL of dilute nitric acid and water to make 50 mL.

(4) Heavy metals—Proceed with 2.0 g of Benzbromarone according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 10 ppm).

(5) Iron—Prepare the test solution with 1.0 g of Benzbromarone according to Method 3, and perform the test according to Method A. Prepare the control solution with 2.0 mL of Standard Iron Solution (not more than 20 ppm).

(6) Arsenic—Prepare the test solution with 2.0 g of Benzbromarone according to Method 3, and perform the test using Apparatus B (not more than 1 ppm).

(7) Related substances—Dissolve 0.10 g of Benzbromarone in 10 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of the sample solution, add chloroform to make exactly 100 mL, and use this solution as the standard solution. Perform the test with these solutions as directed under the Thin-layer Chromatography. Spot 10 μL each of the sample solution and the standard solution on a plate of silica gel with fluorescent indicator for thin-layer chromatography. Develop the plate with a mixture of chloroform, acetone and acetic acid (100) (100:2:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultraviolet light (main wavelength: 254 nm): the spots other than the principal spot from the sample solution are not more intense than the spot from the standard solution.

Loss on drying Not more than 0.5% (1 g, in vacuum at a pressure not exceeding 0.67 kPa, phosphorus (V) oxide, 50°C, 4 hours).

Residue on ignition Not more than 0.10% (1 g).

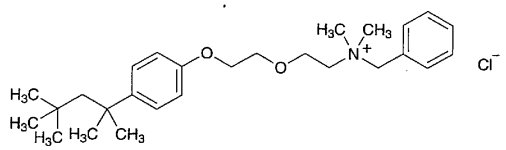
Assay Weigh accurately about 0.6 g of Benzbromarone, previously dried, dissolve in 30 mL of *N,N*-dimethylformamide, and titrate with 0.1 mol/L tetramethylammonium hydroxide VS (indicator: 5 drops of thymol blue-dimethylformamide TS). Perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L tetramethylammonium hydroxide VS
= 42.41 mg of $C_{17}H_{12}Br_2O_3$

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

Benzethonium Chloride

塩化ベンゼトニウム



$C_{27}H_{42}ClNO_2$: 448.08
N-Benzyl-*N*,*N*-dimethyl-*N*-(2-{2-[4-(1,1,3,3-tetramethylbutyl)phenoxy]-ethoxy}ethyl)ammonium chloride
[121-54-0]

Benzethonium Chloride, when dried, contains not less than 97.0% of $C_{27}H_{42}ClNO_2$.

Description Benzethonium Chloride occurs as colorless or white crystals. It is odorless.

It is very soluble in ethanol (95), freely soluble in water, and practically insoluble in diethyl ether.

A solution of Benzethonium Chloride foams strongly when shaken.

Identification **(1)** Dissolve 0.2 g of Benzethonium Chloride in 1 mL of sulfuric acid, add 0.1 g of sodium nitrate, and heat for 5 minutes on a water bath. After cooling, add 10 mL of water and 0.5 g of zinc powder, heat for 5 minutes, cool, and filter: the filtrate responds to the Qualitative Tests for primary aromatic amines, developing a red color.

(2) To 2 mL of a solution of Benzethonium Chloride (1 in 1000) add a mixture of 0.2 mL of a solution of bromophenol blue (1 in 2000) and 0.5 mL of sodium hydroxide TS: a blue color develops. Add 4 mL of chloroform to this solution, and shake vigorously: the blue color shifts to the chloroform layer. Collect the chloroform layer, and add dropwise a solution sodium lauryl sulfate (1 in 1000) with stirring: the chloroform layer turns colorless.

(3) Determine the absorption spectrum of a solution of Benzethonium Chloride in 0.1 mol/L hydrochloric acid TS (1 in 5000) as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum: both spectra exhibit similar intensities of absorption at the same wavelengths.

(4) To 1 mL of a solution of Benzethonium Chloride (1 in 100) add 2 mL of ethanol (95), 0.5 mL of dilute nitric acid and 1 mL of silver nitrate TS: a white precipitate is produced. This precipitate does not dissolve on addition of dilute nitric acid, but dissolves on addition of ammonia TS.

Melting point 158 - 164°C (after drying).

Purity Ammonium—Proceed as directed in the Purity under Benzalkonium Chloride.

Loss on drying Not more than 5.0% (1 g, 105°C, 4 hours).

Residue on ignition Not more than 0.10% (1 g).

Assay Weigh accurately about 0.2 g of Benzethonium Chloride, previously dried, dissolve in 75 mL of water, add diluted dilute hydrochloric acid (1 in 2) dropwise to adjust the pH to 2.6–3.4, then add 1 drop of methyl orange TS, and titrate with 0.02 mol/L tetraphenylboron VS until the solution develops a red.

Each mL of 0.02 mol/L sodium tetraphenylboron VS
= 8.962 mg of $C_{27}H_{42}ClNO_2$

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

Benzethonium Chloride Solution

塩化ベンゼトニウム液

Benzethonium Chloride Solution contains not less than 93% and not more than 107% of the labeled amount of benzethonium chloride ($C_{27}H_{42}ClNO_2$: 448.08).

Method of preparation Dissolve Benzethonium Chloride in Water or Purified Water.

Description Benzethonium Chloride Solution is a clear, colorless liquid. It is odorless.

It foams strongly when shaken.

Identification **(1)** Evaporate a volume of Benzethonium Chloride Solution, equivalent to 0.2 g of Benzethonium Chloride according to the labeled amount, on a water bath to dryness, and proceed with the residue as directed in the Identification (1) under Benzalkonium Chloride.

(2) To a volume of Benzethonium Chloride Solution, equivalent to 0.01 g of Benzethonium Chloride according to the labeled amount, add water to make 10 mL, proceed with 2 mL of this solution as directed in the Identification (2) under Benzalkonium Chloride.

(3) To a volume of Benzethonium Chloride Solution, equivalent to 1 g of Benzethonium Chloride according to the labeled amount, and add water or concentrate on a water bath to make 10 mL. To 1 mL of this solution add 0.1 mol/L hydrochloric acid TS to make 500 mL, and determine the abosprition spectrum as directed under the Ultraviolet-visible Spectrophotometry: it exhibits maxima between 262 nm and 264 nm, between 268 nm and 270 nm, and between 274 nm and 276 nm.

(4) To a volume of Benzethonium Chloride Solution,