**Identification** (1) A solution of Acrinol (1 in 40,000) shows a green fluorescence.

(2) To 5 mL of a solution of Acrinol (1 in 100) add 2 drops each of sodium nitrite TS and dilute hydrochloric acid: a dark red color is produced.

(3) To 5 mL of a solution of Acrinol (1 in 1000) add 3 drops of iodine TS: a deep, blue-green precipitate is formed, which dissolves on the addition of ethanol (95).

(4) To 5 mL of a solution of Acrinol (1 in 100) add 5 mL of dilute sulfuric acid, shake well, allow to stand for about 10 minutes at room temperature, and filter: the filtrate responds to the Qualitative Tests for lactate.

**Purity** (1) Chloride—Dissolve 1.0 g of Acrinol in 80 mL of water by warming on a water bath, cool, and add 10 mL of sodium hydroxide TS and water to make 100 mL. Shake well, allow to stand for 30 minutes, filter, to 40 mL of the filtrate add 7 mL of dilute nitric acid and water to make 50 mL, and perform the test using this solution as the test solution. Prepare 50 mL of the control solution with 4 mL of sodium hydroxide TS, 7 mL of dilute nitric acid, 0.30 mL of 0.01 mol/L hydrochloric acid VS and water (not more than 0.026%).

(2) Sulfate—Dissolve 0.5 g of Acrinol in 20 mL of water by heating, cool, add 2 mL of dilute hydrochloric acid, shake well, allow to stand for 30 minutes, filter, and to the filtrate add 3 drops of barium chloride TS: no turbidity is produced.

(3) Ammonium—Dissolve 0.5 g of Acrinol in 20 mL of water by heating, after cooling, add 0.5 mL of sodium hydroxide TS, filter, and boil the filtrate: the gas evolved does not change moistened red litmus paper to blue.

(4) Heavy metals—Proceed with 1.0 g of Acrinol according to Method 2, and perform the test. Prepare the control solution with 2.0 mL of Standard Lead Solution (not more than 20 ppm).

(5) Volatile fatty acids—Dissolve 0.5 g of Acrinol in a mixture of 20 mL of water and 5 mL of dilute sulfuric acid, shake well, filter, and heat the filtrate: no odor of volatile fatty acids is perceptible.

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

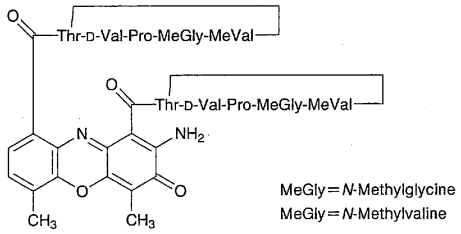
**Assay** Weigh accurately about 0.3 g of Acrinol, place in a 100-mL volumetric flask, add 25 mL of water, 20 mL of sodium acetate TS and 1.25 mL of dilute hydrochloric acid, and dissolve. Add exactly 50 mL of 1/60 mol/L potassium dichromate VS and water to make exactly 100 mL. Allow to stand for 1 hour with frequent shaking, and filter. Discard the first 20 mL of the filtrate, pipet the next 50 mL into an iodine-flask, and add 30 mL of dilute sulfuric acid and 6 mL of potassium iodide TS. Immediately stopper closely, and allow to stand for 5 minutes in a dark place. Add 50 mL of water, and titrate the liberated iodine with 0.1 mol/L sodium thiosulfate VS (indicator: 3 mL of starch TS). Perform a blank determination.

Each mL of 1/60 mol/L potassium dichromate VS
= 12.047 mg of $C_{15}H_{15}N_3O.C_3H_6O_3.H_2O$

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

# Actinomycin D

アクチノマイシン D




$C_{62}H_{86}N_{12}O_{16}$: 1255.42

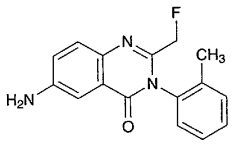
Actinomycin D conforms to the requirements of Actinomycin D in the Requirements for Antibiotic Products of Japan.

**Description** Actinomycin D occurs as an orange-red to red, crystalline powder.

It is freely soluble in methanol, in ethanol (95) and in acetone, and very slightly soluble in water and in diethyl ether.

# Afloqualone

アフロクアロン




$C_{16}H_{14}FN_3O$: 283.30
6-Amino-2-fluoromethyl-3-(2-tolyl)-3*H*-quinazolin-4-one
[*56287-74-2*]

Afloqualone, when dried, contains not less than 98.5% of $C_{16}H_{14}FN_3O$.

**Description** Afloqualone occurs as white to light yellow crystals or crystalline powder.

It is soluble in acetonitrile, sparingly soluble in ethanol (99.5), and practically insoluble in water.

It is gradually colored by light.

Melting point: about 197°C (with decomposition).

**Identification** (1) Conduct this procedure without exposure to light, using light-resistant containers. Determine the absorption spectrum of a solution of Afloqualone in ethanol (99.5) (1 in 150,000) 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 wavelength.

(2) Determine the infrared absorption spectrum of Afloqualone, 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.

**Purity** (1) Acid or alkali—Take 1.0 g of Afloqualone in a light-resistant vessel, add 20 mL of freshly boiled and cooled water, shack well, and filter. To 10 mL of the filtrate add 2 drops of bromothymol blue TS: a yellow color develops. The color changes to blue by adding 0.20 mL of 0.01 mol/L sodium hydroxide TS.

(2) Heavy metals—Proceed with 2.0 g of Afloqualone in a platinum crucible 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).

(3) Related substances—Conduct this procedure without exposure to light, using light-resistant vessels. Dissolve 0.010 g of Afloqualone in 25 mL of the mobile phase, and use this solution as the sample solution. Pipet 3 mL of the sample solution, add the mobile phase to make exactly 100 mL. Pipet 2 mL of this solution, add the mobile phase to make exactly 20 mL, and use this solution as the standard solution. Perform the test with 20 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions, and calculate the areas of each peak by the automatic integration method: the total of the peak areas other than the peak area of afloqualone from the sample solution is not more than the peak area of afloqualone from the standard solution.

*Operating conditions*—

Detector: An ultraviolet absorption photpmeter (wavelength: 254 nm).

Column: A stainless steel column 4.6 mm in inside diameter and 15 cm in length, packed with octylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 40°C.

Mobile phase: Dissolve 7.2 g of disodium hydrogenphosphate 12-water in 1000 mL of water, adjust to pH 5.5 with diluted phosphoric acid (1 in 10). To 600 mL of this solution add 400 mL of acetonitrile.

Flow rate: Adjust the flow rate so that the retention time of afloqualone is about 5.5 minutes.

Time span of measurement: About 4 times as long as the retention time of afloqualone after the solvent peak.

*System suitability*—

Test for required detection: Pipet 5 mL of the standard solution, add the mobile phase to make exactly 25 mL, and confirm that the peak area of afloqualone obtained from 20 μL of this solution is equivalent to 15 to 25% of that of afloqualone obtained from 20 μL of the standard solution.

System performance: Dissolve 0.01 g of Afloqualone in a suitable amount of the mobile phase, add 5 mL of a solution of propyl parahydroxybenzoate in the mobile phase (1 in 2000) and the mobile phase to make 100 mL. When the procedure is run with 20 μL of this solution under the above operating conditions, afloqualone and propyl parahydroxybenzoate are eluted in this order with the resolution between these peaks being not less than 4.

System repeatability: When the test is repeated 6 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the peak areas of afloqualone is not more than 5%.

**Loss on drying** Not more than 0.5% (1 g, in vacuum, 60°C, 2 hours).

**Residue on ignition** Not more than 0.10% (1.0 g, platinum crucible).

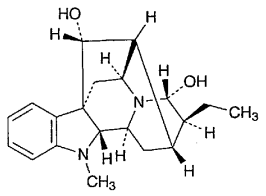
**Assay** Weigh accurately about 0.4 g of Afloqualone, previously dried, dissolve in 10 mL of hydrochloric acid and 40 mL of water, and add 10 mL of a solution of potassium bromide (3 in 10). After cooling at 15°C or below, titrate with 0.1 mol/L sodium nitrite VS according to the potentiometric titration or amperometric titration under the Electrometric Titration method.

Each mL of 0.1 mol/L sodium nitrite
= 28.331 mg of $C_{16}H_{14}FN_3O$

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

# Ajmaline

アジマリン




$C_{20}H_{26}N_2O_2$: 326.43
(17*R*,21*R*)-Ajmalan-17,21-diol [*4360-12-7*]

Ajmaline, when dried, contains not less than 96.0% of $C_{20}H_{26}N_2O_2$.

**Description** Ajmaline occurs as a white to pale yellow, crystalline powder. It is odorless, and has a bitter taste.

It is freely soluble in acetic anhydride and in chloroform, sparingly soluble in methanol, in ethanol (95), in acetone and in diethyl ether, and very slightly soluble in water.

It dissolves in dilute hydrochloric acid.

Melting point: about 195°C (with decomposition).

**Identification** (1) Dissolve 0.05 g of Ajmaline in 5 mL of methanol, and use this solution as the sample solution. Add 3 mL of nitric acid to 1 mL of the sample solution: a deep red color develops.

(2) Spot the sample solution of (1) on filter paper, and spray Dragendorff's TS: an orange color develops.

**Absorbance** $E^{1\%}_{1\,cm}$ (249 nm): 257 - 271 (after drying, 2 mg, ethanol (95), 100 mL). $E^{1\%}_{1\,cm}$ (292 nm): 85 - 95 (after drying, 2 mg, ethanol (95), 100 mL).

**Optical rotation** $[\alpha]_D^{20}$: +136 - +151° (after drying, 0.5 g, chloroform, 50 mL, 100 mm).

**Purity** Other alkaloids—Dissolve 0.10 g of Ajmaline in 10 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of this 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 diethylamine (5:4:1) to a distance of about 10 cm, and air-dry the plate. Examine under ultravio-