Description Ethyl Aminobenzoate occurs as white crystals or crystalline powder. It is odorless. It has a slightly bitter taste, numbing the tongue.

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

It dissolves in dilute hydrochloric acid.

Identification (1) Dissolve 0.01 g of Ethyl Aminobenzoate in 1 mL of dilute hydrochloric acid and 4 mL of water. This solution responds to the Qualitative Tests for primary aromatic amines.

(2) Dissolve 0.1 g of Ethyl Aminobenzoate in 5 mL of water with the aid of dilute hydrochloric acid added dropwise, and add iodine TS dropwise: a brown precipitate is produced.

(3) Warm 0.05 g of Ethyl Aminobenzoate with 2 drops of acetic acid (31) and 5 drops of sulfuric acid: the odor of ethyl acetate is perceptible.

Melting point 89 - 91°C

Purity (1) Acid—Dissolve 1.0 g of Ethyl Aminobenzoate in 10 mL of neutralized ethanol, and add 10 mL of water, 2 drops of phenolphthalein TS and 0.50 mL of 0.01 mol/L sodium hydroxide VS: a red color is produced.

(2) Chloride—Dissolve 0.20 g of Ethyl Aminobenzoate in 5 mL of ethanol (95), add 2 to 3 drops each of dilute nitric acid and of silver nitrate TS: no change occurs immediately.

(3) Heavy metals—Dissolve 2.0 g of Ethyl Aminobenzoate in 20 mL of ethanol (95), add 2 mL of dilute acetic acid and ethanol (95) to make 50 mL, and perform the test using this solution as the test solution. Prepare the control solution as follows: to 2.0 mL of Standard Lead Solution add 2 mL of dilute acetic acid and sufficient ethanol (95) to make 50 mL (not more than 10 ppm).

(4) Readily carbonizable substances—Perform the test with 0.5 g of Ethyl Aminobenzoate: the solution has no more color than Matching Fluid A.

Loss on drying Not more than 1.0% (1 g, silica gel, 3 hours).

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

Assay Weigh accurately about 0.25 g of Ethyl Aminobenzoate, previously dried, dissolve in 10 mL of hydrochloric acid and 70 mL of water, add 10 mL of a solution of potassium bromide (3 in 10), and cool to a temperature below 15°C. Then titrate with 0.1 mol/L sodium nitrite VS as directed in the potentiometric titration or the amperometric titration under the Electrometric Titration.

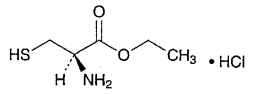
Each mL of 0.1 mol/L sodium nitrite VS
= 16.519 mg of $C_9H_{11}NO_2$

Containers and storage Containers—Well-closed containers.

Ethyl L-Cysteine Hydrochloride

Ethyl Cysteine Hydrochloride

塩酸 L-エチルシステイン



$C_5H_{11}NO_2S.HCl$: 185.67
Ethyl (2*R*)-2-amino-3-sulfanylpropanoate monohydrochloride
[*868-59-7*]

Ethyl L-Cysteine Hydrochloride, when dried, contains not less than 98.5% of $C_5H_{11}NO_2S.HCl$.

Description Ethyl L-Cysteine Hydrochloride occurs as white crystals or crystalline powder. It has a characteristic odor, and has a bitter taste at first with a burning aftertaste.

It is very soluble in water, and freely soluble in ethanol (95).

Melting point: about 126°C (with decomposition).

Identification (1) Determine the infrared absorption spectrum of Ethyl L-Cysteine Hydrochloride 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.

(2) A solution of Ethyl L-Cysteine Hydrochloride (1 in 20) responds to the Qualitative Tests (1) for chloride.

Optical rotation $[\alpha]_D^{20}$: −10.0 - −13.0° (after drying, 2.0 g, 1 mol/L hydrochloric acid TS, 25 mL, 100 mm).

Purity (1) Sulfate—Perform the test with 0.6 g of Ethyl L-Cysteine Hydrochloride. Prepare the the control solution with 0.35 mL of 0.005 mol/L sulfuric acid (not more than 0.028%).

(2) Heavy metals—Proceed with 1.0 g of Ethyl L-Cysteine Hydrochloride according to Method 1, and perform the test. Prepare the control solution with 1.0 mL of Standard Lead Solution (not more than 10 ppm).

(3) Related substances—Conduct this procedure rapidly. Dissolve 0.05 g each of Ethyl L-Cysteine Hydrochloride and *N*-ethylmaleimide in 5 mL of mobile phase, allow to stand for 30 minutes, and use this solution as the sample solution. Pipet 3 mL of the sample solution, add the mobile phase to make exactly 200 mL, and use this solution as the standard solution. Perform the test with 2 μL each of the sample solution and the standard solution as directed under the Liquid Chromatography according to the following conditions. Determine each peak area of these solutions by the automatic integration method: a peak area from the sample solution with the ratio of the retention time to ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex from the standard solution being about 0.7 is not larger than the peak area of ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex from the standard solution. Each area of all peaks other than the peaks of ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex and *N*-ethylmaleimide from the sample solution is not larger than 1/3 of the peak area of

ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex from the standard solution.

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 250 nm).

Column: A stainless steel column about 6 mm in inside diameter and about 15 cm in length, packed with octadecylsilanized silica gel for liquid chromatography (5 μm in particle diameter).

Column temperature: A constant temperature of about 25°C.

Mobile phase: A mixture of 0.02 mol/L monobasic potassium phosphate TS and acetonitrile (2:1).

Flow rate: Adjust the flow rate so that the retention time of ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex is about 4 minutes.

Selection of column: Dissolve 0.05 g of Ethyl L-Cysteine Hydrochloride, 0.01 g of L-cysteine hydrochloride and 0.05 g of *N*-ethylmaleimide in 25 mL of the mobile phase, and allow to stand for 30 minutes. Proceed with 2 μL of this solution under the above conditions, and calculate the resolution. Use a column giving elution of L-cysteine hydrochloride-*N*-ethylmaleimide complex, ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex and *N*-ethylmaleimide in this order, complete resolution of each component, and the resolution of the peaks of L-cysteine hydrochloride-*N*-ethylmaleimide complex and ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex being not less than 3.

Detection sensitivity: Adjust the detection sensitivity so that the peak height of ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex obtained from 2 μL of the standard solution is between 10 mm and 20 mm.

Time span of measurement: About 3 times as long as the retention time of ethyl L-cysteine hydrochloride-*N*-ethylmaleimide complex.

Loss on drying Not more than 0.5% (1 g, in vacuum, phosphorus oxide (V), 5 hours).

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

Assay Weigh accurately about 0.25 g of Ethyl L-Cysteine Hydrochloride, previously dried, transfer into a glass-stoppered flask, and dissolve in 10 mL of water previously freshly boiled and cooled to a temperature not exceeding 5°C in a stream of nitrogen. Add exactly 20 mL of 0.05 mol/L iodine VS, previously cooled to a temperature not exceeding 5°C, and allow to stand for 30 seconds, then titrate with 0.1 mol/L sodium thiosulfate VS, on cooling below 5°C (indicator: 1 mL of starch TS). Perform a blank determination, and make any necessary correction.

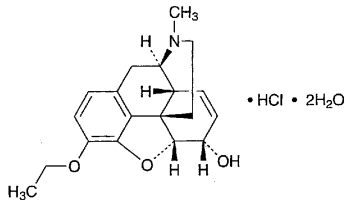
Each mL of 0.05 mol/L iodine VS
= 18.567 mg of $C_5H_{11}NO_2S.HCl$

Containers and storage Containers—Tight containers.

Ethylmorphine Hydrochloride

Dionin

塩酸エチルモルヒネ



$C_{19}H_{23}NO_3.HCl.2H_2O$: 385.88

(5*R*,6*S*)-7,8-Didehydro-4,5-epoxy-3-ethoxy-17-methylmorphinan-6-ol monohydrochloride dihydrate [*125-30-4*, anhydride]

Ethylmorphine Hydrochloride contains not less than 98.0% of $C_{19}H_{23}NO_3.HCl$ (mol.wt.: 349.86), calculated on the anhydrous basis.

Description Ethylmorphine Hydrochloride occurs as white to pale yellow crystals or crystalline powder.

It is very soluble in methanol and in acetic acid (100), freely soluble in water, soluble in ethanol (95), sparingly soluble in acetic anhydride, and practically insoluble in diethyl ether.

It is affected by light.

Melting point: about 123°C (with decomposition).

Identification **(1)** Determine the absorption spectrum of a solution of Ethylmorphine Hydrochloride (1 in 10,000) 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 wavelengths.

(2) Determine the infrared absorption spectrum of Ethylmorphine Hydrochloride 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.

(3) A solution of Ethylmorphine Hydrochloride (1 in 50) responds to the Qualitative Tests (2) for chloride.

Optical rotation $[\alpha]_D^{20}$: −103 – −106° (0.4 g calculated on the anhydrous basis, water, 20 mL, 100 mm).

pH Dissolve 0.10 g of Ethylmorphine Hydrochloride in 10 mL of water: the pH of this solution is between 4.0 and 6.0.

Purity Related substances—Dissolve 0.20 g of Ethylmorphine Hydrochloride in 10 mL of diluted ethanol (95) (1 in 2), and use this solution as the sample solution. Pipet 0.5 mL of the sample solution, add diluted ethanol (95) (1 in 2) 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 ethanol (99.5), toluene, acetone and ammonia solution (28)