hydroxybenzoate in the mobile phase (1 in 12,500).

Operating conditions—

Detector: An ultraviolet absorption photometer (wavelength: 257 nm).

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

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

Mobile phase: Dissolve 2.0 g of sodium lauryl sulfate in 1000 mL of diluted phosphoric acid (1 in 1000), adjust the pH to 3.0 with sodium hydroxide TS, and to 550 mL of this solution add 450 mL of acetonitrile.

Flow rate: Adjust the flow rate so that the retention time of pethidine is about 7 minutes.

System suitability—

System performance: When the procedure is run with 20 μL of the standard solution under the above operating conditions, pethidine and the internal standard are eluted in this order with the resolution between these peaks being not less than 2.0.

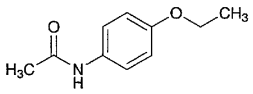
System repeatability: When the test is repeated 6 times with 20 μL of the standard solution under the above operating conditions, the relative standard deviation of the ratios of the peak area of pethidine to that of the internal standard is not more than 1.0%.

Containers and storage Containers—Hermetic containers, and colored containers may be used.

Storage—Light-resistant.

Phenacetin

フェナセチン



$C_{10}H_{13}NO_2$: 179.22

N-(4-Ethoxyphenyl)acetamide [*62-44-2*]

Phenacetin, when dried, contains not less than 98.0% of $C_{10}H_{13}NO_2$.

Description Phenacetin occurs as white crystals or crystalline powder.

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

Its saturated solution is neutral.

Identification Boil 0.1 g of Phenacetin with 1 mL of hydrochloric acid for 1 minute, dilute with 10 mL of water, cool, and filter. Add 1 drop of potassium dichromate TS to the filtrate: a red color develops gradually.

Melting point 134 - 137°C

Purity **(1)** Acetanilide—Boil 0.5 g of Phenacetin with 10 mL of water for 1 minute, cool, filter, and add bromine TS dropwise to the filtrate, agitating after each addition until the color of the solution remains permanently: no turbidity is produced.

(2) *p*-Chloroacetanilide—To 1.5 g of Phenacetin add 0.05 g of Raney nickel catalyst, 2 mL of sodium hydroxide TS, 5 mL of ethanol (95) and 10 mL of water, and boil for 10 minutes under a reflux condenser. Cool, filter, and wash the residue with a small quantity of water. Combine the washings with the filtrate, add 10 mL f dilute nitric acid and water to make 50 mL, and use this solution as the sample solution. Take 0.05 g of Raney nickel catalyst, 2 mL of sodium hydroxide TS, 5 mL of ethanol (95) and 10 mL of water, and boil for 10 minutes under a reflux condenser. Cool, filter, and wash the residue with a small quantity of water. Combine the washings with the filtrate, add 10 mL of dilute nitric acid and 1.0 mL of 0.01 mol/L hydrochloric acid VS and water to make 50 mL, and use this solution as the control solution. To each solution add 1 mL of silver nitrate TS, mix, and allow to stand for 5 minutes: the sample solution has no more turbidity than the control solution.

(3) *p*-Phenetidine—Boil 0.30 of Phenacetin with 1 mL of ethanol (95), 1 drop of iodine TS and 3 mL of water: no red color develops or, if any color develops, the solution has no more color than the following control solution.

Control solution: Weigh accurately 0.2613 g of phenacetin, add 30 mL of dilute hydrochloric acid, and boil for 1 hour under a reflux condenser. Cool, and add 25 mL of a solution of sodium hydroxide (1 in 5), transfer to a separator, and extract with three 30-mL portions of chloroform. Filter the combined chloroform extract, wash the filter paper with five 2-mL portions of chloroform, combine the washings with the filtrate, and add chloroform to make exactly 100 mL. Pipet 3 mL of the solution, and add ethanol (95) to make exactly 100 mL. Pipet 1 mL of the solution, add 1 drop of iodine TS and 3 mL of water, and boil.

(4) Readily carbonizable substances—Take 0.5 g of Phenacetin, and perform the test: the solution has no more color than Matching Fluid T.

Loss on drying Not more than 0.5% (1 g, 105°C, 2 hours).

Residue on ignition Not more than 0.05% (1 g).

Assay Weigh accurately about 0.3 g of Phenacetin, previously dried, add 30 mL of dilute hydrochloric acid, and boil for 1 hour under a reflux condenser. Cool, add 25 mL of a solution of sodium hydroxide (1 in 5), and transfer to a separator. Extract with three 30-mL portions of chloroform, filter each extract through the same pledget of cotton successively, and wash the cotton with five 2-mL portions of chloroform. Combine the washings with the filtrate, and titrate with 0.1 mol/L perchloric acid VS (indicator: 2 drops of crystal violet TS). To 30 mL of dilute hydrochloric acid add 25 mL of a solution of sodium hydroxide (1 in 5). Proceed with the solution as directed for the sample, add 15 mL of acetic acid (100) to a solution prepared by combining the chloroform extract with the washings, perform a blank determination, and make any necessary correction.

Each mL of 0.1 mol/L perchloric acid VS
= 17.922 mg of $C_{10}H_{13}NO_2$

Containers and storage Containers—Well-closed containers.