cording 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) Arsenic—Prepare the test solution with 1.0 g of Nicomol according to Method 3, and perform the test using Apparatus B (not more than 2 ppm).

(6) Related substances—Dissolve 0.20 g of Nicomol in 20 mL of chloroform, and use this solution as the sample solution. Pipet 1 mL of the sample solution, and add chloroform to make exactly 20 mL. Pipet 2 mL of this solution, add chloroform to make exactly 20 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 dichloromethane, ethanol (95), acetonitrile and ethyl acetate (5:3:1:1) 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 1.0% (1 g, 105°C, 4 hours).

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

Assay Weigh accurately about 1.5 g of Nicomol, previously dried, add exactly 40 mL of 0.5 mol/L sodium hydroxide VS, and boil gently under a reflux condenser connected to a carbon dioxide absorption tube (soda lime) for 10 minutes. After cooling, titrate immediately the excess sodium hydroxide with 0.25 mol/L sulfuric acid VS (indicator: 3 drops of phenolphthalein TS). Perform a blank determination.

Each mL of 0.5 mol/L sodium hydroxide VS
= 80.08 mg of $C_{34}H_{32}N_4O_9$

Containers and storage Containers—Tight containers.

Nicomol Tablets

ニコモール錠

Nicomol Tablets contain not less than 95% and not more than 105% of the labeled amount of nicomol ($C_{34}H_{32}N_4O_9$: 640.64).

Method of preparation Prepare as directed under Tablets, with Nicomol.

Identification To a portion of powdered Nicomol Tablets, equivalent to 0.5 g of Nicomol according to the labeled amount, add 20 mL of chloroform, shake, and filter. Evaporate the filtrate on a water bath to dryness. Proceed with the residue as directed in the Identification (1) and (2) under Nicomol.

Dissolution test Perform the test with 1 tablet of Nicomol Tablets at 75 revolutions per minute according to Method 2 under the Dissolution Test, using 900 mL of the 1st fluid under the Disintegration Test. Take 20 mL or more of the dissolved solution 60 minutes after starting the test, and filter through a membrane filter with pore size of not more than 0.8 μm. Discard the first 10 mL of the filtrate, pipet 2 mL of the subsequent, add the 1st fluid to make exactly 25 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.1 g of nicomol for assay, previously dried at 105°C for 4 hours, dissolve in the 1st fluid to make exactly 100 mL, then pipet 2 mL of this solution, add the 1st fluid to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 262 nm as directed under the Ultraviolet-visible Spectrophotometry.

The dissolution rate of Nicomol Tablets in 60 minutes is not less than 75%.

Dissolution rate (%) with respect to the labeled amount of nicomol ($C_{34}H_{32}N_4O_9$)

$$= W_S \times \frac{A_T}{A_S} \times \frac{1}{C} \times 225$$

W_S: Amount (mg) of nicomol for assay.
C: Labeled amount (mg) of nicomol ($C_{34}H_{32}N_4O_9$) in 1 tablet.

Assay Weigh accurately not less than 20 Nicomol Tablets and powder. Weigh accurately a portion of the powder, equivalent to about 1 g of nicomol ($C_{34}H_{32}N_4O_9$), add 100 mL of 1 mol/L hydrochloric acid TS, shake well, add water to make exactly 500 mL, and filter. Discard the first 50 mL of the filtrate, pipet 2 mL of the subsequent filtrate, add 50 mL of 1 mol/L hydrochloric acid TS and water to make exactly 250 mL, and use this solution as the sample solution. Separately, weigh accurately about 0.08 g of nicomol for assay, previously dried at 105°C for 4 hours, dissolve in 50 mL of 1 mol/L hydrochloric acid TS, and add water to make exactly 100 mL. Pipet 2 mL of this solution, add 20 mL of 1 mol/L hydrochloric acid TS and water to make exactly 100 mL, and use this solution as the standard solution. Determine the absorbances, A_T and A_S, of the sample solution and the standard solution at 262 nm as directed under the Ultraviolet-visible Spectrophotometry.

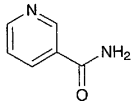
Amount (mg) of nicomol ($C_{34}H_{32}N_4O_9$)
= amount (mg) of nicomol for assay

$$\times \frac{A_T}{A_S} \times \frac{25}{2}$$

Containers and storage Containers—Tight containers.

Nicotinamide

ニコチン酸アミド



$C_6H_6N_2O$: 122.12
Pyridine-3-carboxamide [*98-92-0*]

Nicotinamide, when dried, contains not less than 98.5% of $C_6H_6N_2O$.

Description Nicotinamide occurs as white crystals or crystalline powder. It is odorless, and has a bitter taste.

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

Identification (1) Mix 5 mg of Nicotinamide with 0.01 g of 1-chloro-2,4-dinitrobenzene, heat gently for 5 to 6 seconds, and fuse the mixture. Cool, and add 4 mL of potassium hydroxide-ethanol TS: a red color is produced.

(2) To 0.02 g of Nicotinamide add 5 mL of sodium hydroxide TS, and boil carefully: the gas evolved turns moistened red litmus paper blue.

(3) Dissolve 0.02 g of Nicotinamide in water to make 1000 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Nicotinamide Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

pH Dissolve 1.0 g of Nicotinamide in 20 mL of water: the pH of this solution is between 6.0 and 7.5.

Melting point 128 - 131°C

Purity (1) Clarity and color of solution—Dissolve 1.0 g of Nicotinamide in 20 mL of water: the solution is clear and colorless.

(2) Chloride—Take 0.5 g of Nicotinamide, and perform the test. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021%).

(3) Sulfate—Take 1.0 g of Nicotinamide, and perform the test. Prepare the control solution with 0.40 mL of 0.005 mol/L sulfuric acid VS (not more than 0.019%).

(4) Heavy metals—Proceed with 1.0 g of Nicotinamide according to Method 1, and perform the test. Prepare the control solution with 3.0 mL of Standard Lead Solution (not more than 30 ppm).

(5) Readily carbonizable substances—Take 0.20 g of Nicotinamide, and perform the test. The solution has no more color than Matching Fluid A.

Loss on drying Not more than 0.5% (1 g, in vacuum, silica gel, 4 hours).

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

Assay Weigh accurately 2.405 g × f to 2.478 g × f of Nicotinamide, previously dried (f is the factor of 1 mol/L hydrochloric acid VS used in the following procedure), dissolve in exactly 20 mL of 1 mol/L hydrochloric acid VS, and add 1 mL of dilute thymol blue TS and water to make exactly 50 mL. Determine the absorbances, A_1 and A_2, of this solution at 435 nm and 544 nm, respectively, as directed under the Absorbance Ratio Method, using water as the blank. Calculate r by the equation $r = A_2/(A_1 + A_2)$. Calculate the value of x, using r thus obtained and the $x - r$ curve obtained from the following table.

Amount (g) of nicotinamide ($C_6H_6N_2O$) = 2.4426 × f × x

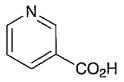
The table showing the relationship between x and r.

x	0.960	0.965	0.970	0.975	0.980	0.985	0.990	0.995	1.000
r	0.504	0.489	0.475	0.462	0.450	0.437	0.424	0.411	0.398
x	1.005	1.010	1.015	1.020	1.025	1.030	1.035	1.040	
r	0.384	0.372	0.360	0.348	0.335	0.322	0.311	0.302	

Containers and storage Containers—Tight containers.

Nicotinic Acid

ニコチン酸



$C_6H_5NO_2$: 123.11
Pyridine-3-carboxylic acid [*59-67-6*]

Nicotinic Acid, when dried, contains not less than 99.5% of $C_6H_5NO_2$.

Description Nicotinic Acid occurs as white crystals or crystalline powder. It is odorless, and has a slightly acid taste.

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

It dissolves in sodium hydroxide TS and in sodium carbonate TS.

Identification (1) Triturate 5 mg of Nicotinic Acid with 0.01 g of 1-chloro-2,4-dinitrobenzene, and fuse the mixture by gentle heating for 5 to 6 seconds. Cool, and add 4 mL of potassium hydroxide-ethanol TS: a dark red color is produced.

(2) Dissolve 0.02 g of Nicotinic Acid in water to make 1000 mL. Determine the absorption spectrum of the solution as directed under the Ultraviolet-visible Spectrophotometry, and compare the spectrum with the Reference Spectrum or the spectrum of a solution of Nicotinic Acid Reference Standard prepared in the same manner as the sample solution: both spectra exhibit similar intensities of absorption at the same wavelengths.

pH Dissolve 0.20 g of Nicotinic Acid in 20 mL of water: the pH of this solution is between 3.0 and 4.0.

Melting point 234 - 238°C

Purity (1) Clarity and color of solution—Dissolve 0.20 g of Nicotinic Acid in 20 mL of water: the solution is clear and colorless.

(2) Chloride—Perform the test with 0.5 g of Nicotinic Acid. Prepare the control solution with 0.30 mL of 0.01 mol/L hydrochloric acid VS (not more than 0.021%).

(3) Sulfate—Dissolve 1.0 g of Nicotinic Acid in 3 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 with 0.40 mL of 0.005 mol/L sulfuric acid VS and 3 mL of dilute hydrochloric acid, and dilute with water to make 50 mL (not more than 0.019%).

(4) Nitro compounds—Dissolve 1.0 g of Nicotinic Acid in 8 mL of sodium hydroxide TS, and add water to make 20 mL: the solution has no more color than Matching Fluid A.

(5) Heavy metals—Proceed with 1.0 g of Nicotinic Acid 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).

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

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

Assay Weigh accurately about 0.3 g of Nicotinic Acid, previously dried, dissolve in 50 mL of water, and titrate