

JIS

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JAPANESE INDUSTRIAL STANDARD

N-Substituted Anilines
(N,N-Dimethylaniline·N,N-Diethylaniline·
N-Ethylaniline·N-Methylaniline·
N-Benzyl-N-Ethylaniline)



JIS K 4112-1981



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Translated

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JAPANESE STANDARDS ASSOCIATION



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J I S
K 4112-1981

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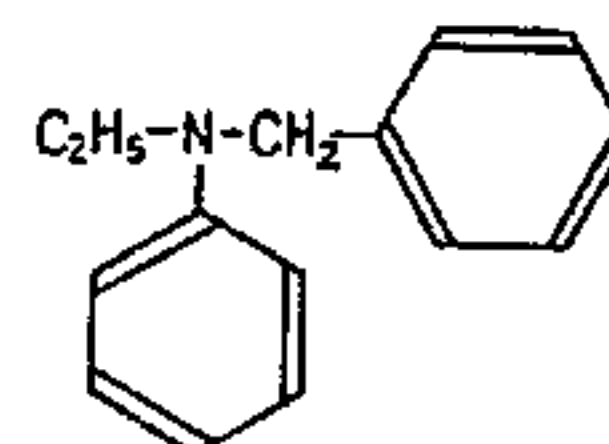
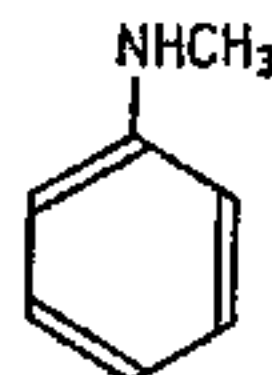
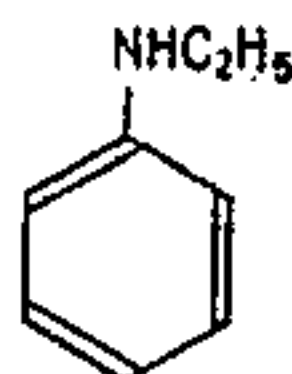
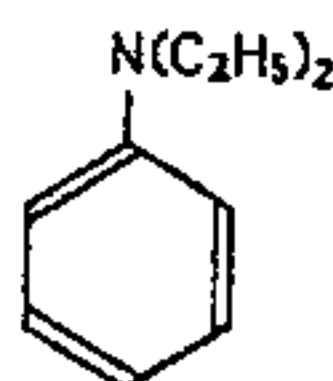
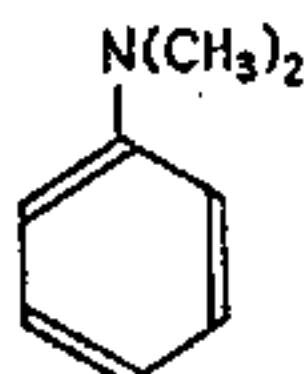
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1. Scope

This Japanese Industrial Standard specifies N,N-dimethylaniline, N,N-diethylaniline, N-ethylaniline, N-methylaniline and N-benzyl-N-ethylaniline, hereinafter referred to as the "N-substituted anilines", as organic intermediates.

2. Types

The types shall be as given in the following:

- (1) N,N-dimethylaniline
- (2) N,N-diethylaniline
- (3) N-ethylaniline
- (4) N-methylaniline
- (5) N-benzyl-N-ethylaniline

3. Qualities

The N-substituted anilines shall be tested in accordance with 5., and shall conform to the requirements of Table 1.

Applicable Standards:

JIS K 0113-General Rules for Methods of Potentiometric,
Amperometric, and Coulometric Titration

JIS K 0114-General Rules for Analytical Method in Gas Chromatography

JIS K 4101-General Testing Methods for Organic Intermediates

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Table 1

Item \ Type	N,N-dimethylaniline	N,N-diethylaniline	N-ethylaniline	N-methylaniline	N-benzyl-N-ethylaniline
Appearance	Liquid of light yellow to light brown	Liquid of light yellow to light brown	Liquid of light yellow to light brown	Liquid of light yellow to light brown	Liquid of light yellow to light brown
Specific gravity (15/4°C)	0.959 to 0.962	0.938 to 0.940	0.965 to 0.968	0.991 to 0.994	1.032 to 1.036
Purity (%)	99.0 min.	98.5 min.	98.5 min.	98.5 min.	98.5 min.
Impurity %	Aniline	0.03 max.	0.03 max.	0.3 max.	0.3 max.
	N-methylaniline	0.5 max.	-	-	-
	N-ethylaniline	-	0.8 max.	-	0.3 max.
	N,N-dimethylaniline	-	-	0.8 max.	-
	N,N-diethylaniline	-	-	0.8 max.	0.2 max.
	N-benzylaniline	-	-	-	0.7 max.
Water content %	0.1 max.	0.1 max.	0.1 max.	0.1 max.	0.1 max.
Nonvolatile matters %	0.1 max.	0.1 max.	0.1 max.	0.1 max.	0.1 max.

4. Sampling Method

The sampling method shall be in accordance with 3. of JIS K 4101.

5. Testing Methods

5.1 General Items The general items in common with the tests shall be in accordance with 2. of JIS K 4101.

5.2 Appearance The appearance shall be examined by visual inspection.

5.3 Specific Gravity The specific gravity shall be in accordance with 8. of JIS K 4101.

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5.4 Purity and Impurities The purity and impurities shall be in accordance with JIS K 0114, and others shall be in accordance with the following:

(1) N,N-Dimethylaniline

(1.1) Objective Sample, Analytical Items and Range of Concentration

The objective sample, analytical items and range of concentration shall be as follows:

(1.1.1) Objective Sample N,N-dimethylaniline

(1.1.2) Analytical Items Purity and impurities (aniline and N-methylaniline)

(1.1.3) Range of Concentration It shall conform to Table 2.

Table 2

Item	Range of concentration
N,N-dimethylaniline %	97 min.
Aniline %	0.5 max.
N-methylaniline %	1.5 max.

(1.2) Apparatus

(1.2.1) Column A hard glass tube or stainless steel tube of 3 to 4 mm in inside diameter and 2 to 3 m in length.

(1.2.2) Detector A thermal conductivity detector or hydrogen flame ionization detector.

(1.2.3) Sample Injector A microsyringe of 5 to 10 μ l in capacity.

(1.2.4) Column Packings

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(1.2.4.1) Liquid Phase Hydrocarbon system

Reference There is paraffin wax, in the hydrocarbon system.

(1.2.4.2) Carrier Diatomaceous earth of 177 to 250 μm (80 to 60 mesh) in grain size.

Reference There is diasolid L in the diatomaceous earth.

(1.2.4.3) Liquid Phase Concentration 8 to 10%

(1.3) Analytical Conditions Because analytical conditions are different according to instruments used, the most suitable conditions shall be selected on each instrument. An example is given in the following:

(1.3.1) Column A stainless steel tube of 3 mm in inside diameter and 3 m in length

(1.3.2) Detector A thermal conductivity detector

(1.3.3) Packings 10 g of the hydrocarbon system and 100 g of the diatomaceous earth.

Reference The paraffin wax and diasolid L are used in this example.

(1.3.4) <u>Temperatures</u>	Column tank	150°C
	Detector tank	240°C
	Injecting part of sample	280°C

(1.3.5) Carrier Gas Helium 50 ml/min

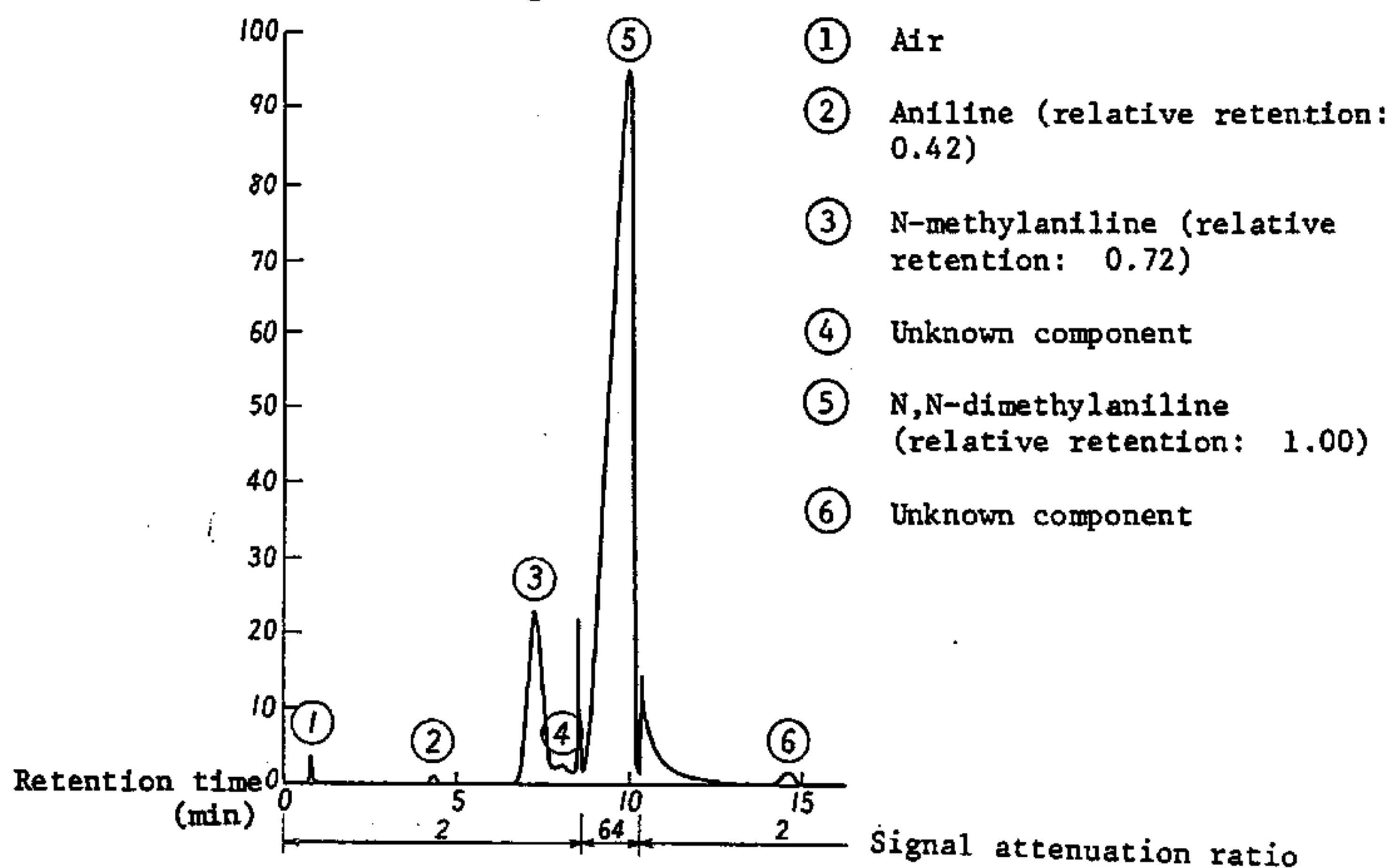
(1.3.6) Injecting Quantity of Sample 2 μl

(1.3.7) Feed Speed of Recording Paper 10 mm/min

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- (1.4) Representative Example of Chromatogram The representative example of chromatogram under the analytical conditions given in (1.3) is given in Fig. 1.

Fig. 1



(1.5) Quantitative Determination

- (1.5.1) Measuring Method of Peak Area The measuring method of peak area shall be in accordance with the peak width at half height method of 10.1.2(1) of JIS K 0114 or the integrator.

- (1.5.2) Type of Method for Determination The method for determination shall be in accordance with the corrected area percentage method of 10.2 (3) of JIS K 0114.

- (1.5.3) Calculation The purity and impurities (aniline and N-methylaniline) shall be obtained respectively from the following formula:

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$$P = \frac{rA}{rA + \frac{r_1 A_1}{f_1} + \frac{r_2 A_2}{f_2} + \sum_{i=3}^n r_i A_i} \times (100 - W - R)$$

$$X_1 = \frac{\frac{r_1 A_1}{f_1}}{rA + \frac{r_1 A_1}{f_1} + \frac{r_2 A_2}{f_2} + \sum_{i=3}^n r_i A_i} \times 100$$

$$X_2 = \frac{\frac{r_2 A_2}{f_2}}{rA + \frac{r_1 A_1}{f_1} + \frac{r_2 A_2}{f_2} + \sum_{i=3}^n r_i A_i} \times 100$$

where P : purity (%)

X_1 : aniline (%)

X_2 : N-methylaniline (%)

A : peak area of the N,N-dimethylaniline (mm^2)

A_1 : peak area of aniline (mm^2)

A_2 : peak area of N-methylaniline (mm^2)

A_i : peak area of component other than the N,N-dimethylaniline, aniline and N-methylaniline (mm^2)

r : signal attenuation ratio of the N,N-dimethylaniline

r_1 : signal attenuation ratio of the aniline

r_2 : signal attenuation ratio of the N-methylaniline

r_i : signal attenuation ratio of component other than the N,N-dimethylaniline, aniline and N-methylaniline

f_1 : relative sensibility of the aniline as compared with the N,N-dimethylaniline

f_2 : relative sensibility of the N-methylaniline as compared with the N,N-dimethylaniline

W : water content of the N,N-dimethylaniline obtained in accordance with 5.5 (%)

R : non-volatile content of the N,N-dimethylaniline obtained in accordance with 5.6 (%)

(2) N,N-Diethylaniline

(2.1) Objective Sample, Analytical Items and Range of Concentration

The objective sample, analytical items and range of concentration shall be as follows: