

DETERMINATION OF NCO CONTENT IN ISOCYANATES

Application

This method is used to determine the NCO content in isocyanates used in the manufacture of polyurethane foam, by reaction of the NCO groups with dibutylamine or di-isobutylamine and titration with hydrochloric acid.

Apparatus and Reagents Required

Erlenmeyer Flask - 250 mL

Graduated Flask - 1000 mL

Burette - 50 mL

Hydrochloric Acid - 0.1 N

Methanol - (A.R.)

Bromophenol Blue - 0.04 % in ethanol

Dibutylamine or Di-isobutylamine (A.R.)

Dibutylamine or di-isobutylamine is refluxed over barium oxide as drying agent for 3 h and then distilled. Use a guard tube filled with sodium hydroxide and calcium oxide to avoid contact of the distillate with water vapour or carbon dioxide.

Monochlorobenzene

Monochlorobenzene must be redistilled. Discard the first azeotropic runnings. Distilled monochlorobenzene is water-free.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of 23 +/- 2 °C and 50 +/- 5 % relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Date	Action	Revisions
2001 01 30	Revised	Editorial – no technical change A. Cockman
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Page 1 of 2

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Procedure

1. Prepare a dibutylamine solution (0.1 N) by weighing 12.93 g freshly distilled dibutylamine or diisobutylamine into a clean, dry beaker and making up to 1000 mL with monochlorobenzene.
2. Depending on the isocyanate content weigh 150 +/- 0.1 mg to 250 +/- 0.1 mg of material under test into the Erlenmeyer flask, add 20 mL of the 0.1 N dibutylamine solution. If the isocyanate is not dissolved completely add a few mL of chlorobenzene. Allow to stand for 10 minutes.

Note: Any precipitate is ignored.

3. Dilute with 100 mL methanol and add the 0.04 % bromophenol blue solution until a faint blue colour develops in the test solution.
4. Titrate the test solution with the 0.1 N hydrochloric acid. The colour changes from blue to colourless or very slight yellow.
5. Run a blank test using the relevant solutions. (20 mL dibutylamine solution 0.1 N)

Evaluation

Calculate and report the NCO content using the following formula:

$$\text{NCO (\%)} = \frac{(B-A) \times f \times 4.2}{W} \times 100$$

Where: A = HCl (0.1 N) used for the test solution (in mL)
 B = HCl (0.1 N) used for the blank test (in mL)
 f = Factor of the HCl
 W = Sample weight in mg

Chemicals, materials, parts, and equipment referenced in this document must be used and handled properly. Each party is responsible for determining proper use and handling in its facilities.