

AZODICARBONAMIDE

Prepared at the 19th JECFA (1975), published in NMRS 55B (1976) and in FNP 52 (1992). Metals and arsenic specifications revised at the 63rd JECFA (2004)

An acceptable level of treatment of 0-45 mg/kg was established at the 9th JECFA (1965)

SYNONYMS

Azobisformamide; INS No. 927a

DEFINITION

Chemical names

Azodicarbonamide, azodicarboxylic acid diamide

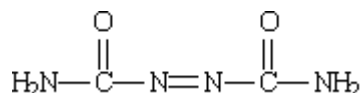
C.A.S. number

123-77-3

Chemical formula

C₂H₄N₄O₂

Structural formula



Formula weight

116.08

Assay

Not less than 98.6%; not less than 47.2% and not more than 48.7% of N on the dried basis

DESCRIPTION

Yellow to orange-red, odourless, crystalline powder

FUNCTIONAL USES Maturing agent for flour

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4)

Practically insoluble in water and in ethanol; slightly soluble in dimethyl sulfoxide

Melting range (Vol. 4)

Above 180° with decomposition

Test for oxidation

Liberates iodine from potassium iodide TS solution in the presence of 10% sulfuric acid

Test for carbon dioxide

Heat about 10 mg of the sample in a crucible. A drop of barium hydroxide solution held above the sample by means of a glass rod becomes turbid

PURITY

Loss on drying (Vol. 4)

Not more than 0.5% (50°, 2h in vacuo)

<u>pH</u> (Vol. 4)	Not less than 5.0 (1 in 50 suspension made by adding 2 g of sample to 100 ml of water and agitating the mixture with a power stirrer for 5 min).
<u>Sulfated ash</u> (Vol. 4)	Not more than 0.15% Test 1.5 g of the sample (Method I)
<u>Lead</u> (Vol. 4)	Not more than 2 mg/kg Determine using an atomic absorption technique appropriate to the specified level. The selection of sample size and method of sample preparation may be based on the principles of the method described in Volume 4, "Instrumental Methods."

METHOD OF ASSAY

Titrimetric method/azodicarbonamide:

Transfer about 225 mg of the accurately weighed sample, previously dried in a vacuum oven at 50° for 2 h, into a 250-ml glass-stoppered iodine flask. Add about 23 ml of dimethyl sulfoxide to the flask, washing any adhered sample down with the solvent, stopper the flask, and place about 2 ml of the solvent in the cup or lip of the flask. Swirl occasionally until complete solution of the sample is effected, and then loosen the stopper to drain the remainder of solvent into the flask and to rinse down any dissolved sample into the solution. Add 5 g of potassium iodide followed by 15 ml of water, immediately pipet 10 ml of 0.5 N hydrochloric acid into the flask, and stopper quickly. Swirl until the potassium iodide dissolves, and allow to stand for 20-25 min protected from light.

Titrate the liberated iodine with 0.1 N sodium thiosulfate to the disappearance of the yellow colour. Titrate with additional thiosulfate if any yellow colour appears within 15 min. Perform a blank determination on a solution consisting of 25 ml of dimethyl sulfoxide, 5 g of potassium iodide, 15 ml of water, and 5 ml of 0.5 N hydrochloric acid, and make any necessary correction. Each ml of 0.1 N sodium thiosulfate is equivalent to 5.804 mg of C₂H₄N₄O₂.

Kjeldahl method (semimicro)/nitrogen:

Transfer about 50 mg of the accurately weighed sample, previously dried in a vacuum oven at 50° for 2 h, into a 100-ml Kjeldahl flask, add 3 ml of hydriodic acid (min. 57%) and digest the mixture for 75 min adding sufficient water, when necessary, to maintain the original volume. Increase the heat at the end of the digestion period and continue heating until the volume is reduced by about one-half. Cool to room temperature, add 1.5 g of potassium sulfate and 3 ml of water. Carefully add 4.5 ml of concentrated sulfuric acid and heat until iodine fumes are no longer evolved. Allow the mixture to cool, wash down the sides of the flask with water, heat until charring occurs, and again cool to room temperature. To the charred material add 40 mg of mercuric oxide, heat until the colour of the solution is pale yellow, then cool, wash down the sides of the flask with a few ml of water and digest the mixture for 3 h. Cool the digest, add 20 ml ammonia-free water, 16 ml of a 50% sodium hydroxide solution and 5 ml of a 44% sodium thiosulfate solution. Connect the flask to a distillation apparatus and distil, collecting the distillate in 10 ml of a 4% boric acid solution. Add a few drops of methyl red-methylene blue TS to the distillate and titrate with 0.05 N sulfuric acid. Perform a blank determination. Each ml of 0.05 N sulfuric acid is equivalent to 0.7004 mg of N.

