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Plastics — Amine epoxide hardeners — Determination of primary, secondary and tertiary amine group nitrogen content

Plastiques — Durcisseurs pour résines époxy — Détermination de la teneur en azote des groupes amine primaire, secondaire, tertiaire

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Plastics — Amine epoxide hardeners — Determination of primary, secondary and tertiary amine group nitrogen content

1 Scope

This International Standard specifies a method for the determination of the primary, secondary and tertiary amine group nitrogen content of aliphatic or aromatic amine hardeners for epoxy resins.

2 Principle

2.1 Aliphatic amines

2.1.1 Determination of total amine group nitrogen content (X_T)

The total alkalinity is determined by potentiometric titration against hydrobromic or perchloric acid in glacial acetic acid, as described by the following reactions:

 $RNH_{2} + H^{+} \rightarrow RNH_{3}^{+}$ $(RR'NH + H^{+} \rightarrow (RR')NH_{2}^{+}$ $(RR'R'')N + H^{+} \rightarrow (RR'R'')NH^{+}$

The results are expressed as percentage nitrogen.

NOTE 1 Perchloric acid is not suitable for use with amine hardeners such as *N*-aminoethylpiperazine.

2.1.2 Determination of tertiary amine group nitrogen content (X_3)

The primary and secondary amine groups are converted into amide groups with acetic anhydride, as described by the following reactions: $RNH_2 + (CH_3CO)_2O \rightarrow$ $RNHCOCH_3 + CH_3COOH$

(RR')NH + (CH₃CO)₂O → (RR')NCOCH₃ + CH₃COOH

The tertiary amine group alkalinity is determined by potentiometric titration against hydrobromic or perchloric acid in glacial acetic acid/acetic anhydride, as described by the following reaction:

 $(RR'R'')NH + H^+ \rightarrow (RR'R'')NH^+$

The results are expressed as percentage nitrogen.

2.1.3 Determination of primary amine group nitrogen content (X_{L1})

The primary amine groups are reacted with a measured excess of 2,4-pentanedione (acetylacetone) in N,N-di-methylformamide to form imines, as described by the following reaction:

$$RNH_2 + CH_3COCH_2COCH_3 \rightarrow CH_3CNRCH_2COCH_3 + H_2O$$

The excess acetylacetone is determined by potentiometric titration against potassium hydroxide (the reaction products of acetylacetone and primary amine groups are neutral under these conditions).

The results are expressed as percentage nitrogen.