International Standard



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Liquefied anhydrous ammonia for industrial use — Determination of water content — Karl Fischer method

Ammoniac anhydre liquéfié à usage industriel – Dosage de l'eau – Méthode de Karl Fischer

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Descriptors : industrial products, chemical compounds, liquid ammonia, chemical analysis, determination of content, water, test equipment, Karl Fischer method.

Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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WARNING – Liquefied anhydrous ammonia is a highly corrosive, toxic substance, which boils at -33,3 °C at standard atmospheric pressure. Its action on the skin is strongly corrosive, producing severe and painful burns. Contact with the eyes can cause permanent bindness.

Its vapour is strongly irritant to the mucous membrane and eyes, and produces a suffocating effect on the respiratory tract.

In concentrations of 16 to 25 % (V/V) aseous anhydrous ammonia forms explosive mixtures with air.

Personnel responsible for handling the product shall be fully informed as to its dangerous character and the precautions to be taken.

Operators shall wear thick rubber gloves, a rubber apron and full face and head protection, and shall be provided with a protective gas-mask fitted with a filter for amnonia.

The operations described shall be carried out only in a well-ventilated fume cupboard.

For further information, see the appropriate sections of 160 3165.

1 Scope and field of application

This International Standard specifies the Karl Fischer direct electrometric method for the determination of the water content of liquefied anhydrous ammonia for industrial use.

The method is applicable to products having water contents equal to or greater than 50 mg/kg.

NOTE — For water contents greater than 1 000 mg/kg, it is preferable to dilute the evaporation residue with anhydrous methanol in accordance with ISO 4276 and titrate an aliquot portion of the diluted solution.

2 References

ISO 760, Determination of water — Karl Fischer method (General method).

ISO 3165, Sampling of chemical products for industrial use – Safety in sampling.

ISO 4276, Anhydrous ammonia for industrial use – Evaluation of residue on evaporation – Gravimetric method.

ISO 7103, Liquefied anhydrous ammonia for industrial use – Sampling – Taking a laboratory sample.

Principle

Evaporation of a test portion in the presence of ethanediol and determination of the water content of the residue by the Karl Fische Wect electrometric method.

4 Reagens and materials

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

Reagents and materials given in clause 4 of ISO 760, and in addition:

4.1 Freezing mixture, consisting of a mixture of solid carbon dioxide and methanol (technical grade is suitable), capable of reaching a temperature of between -35 and -40 °C.

NOTE - Technical grade acetone may be used instead of methanol.

4.2 Sulfuric acid, (ρ approximately 1,84 g/ml), approximately 10 % (m/m) solution.

4.3 1,2-Ethanediol (ethyleneglycol) (CH₂OH-CH₂OH), having a water content not greater than 0,1 % (m/m).

NOTE — 1,2-Ethanediol is very hygroscopic; prevent absorption of atmospheric moisture.