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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 28193-2011

Determination of chloroacetic acid and chloroacetate content in surfactants

表面活性剂中氯乙酸（盐）残留量的测定

Issued on: December 30, 2011 **Implemented on: September 1, 2012**

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Standardization Administration of the PRC

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Foreword

This standard was drafted in accordance with GB/T 1.1-2009 given rules. The standard proposed by China National Light Industry Council. This standard by the National Standardization Technical Committee and the detergent surfactant (SAC/TC272) centralized. This standard was drafted. China Daily Chemical Industry Research Institute, Guangzhou Star Technology Development Co., Ltd., Zhejiang Chan Yu Technology Co. Shares The company, surfactants and detergent industry Productivity Promotion Center. The main drafters of this standard. Feng Yu, Yip, Huang Yaru, Yao Chen's, into Xiaojing, Xu Linshou, Xiaxiong Yan, Guan Jing Cai, Lei Xiaoying. Determination of surfactants chloroacetate (salt) Residues

1 Scope

China's national method for determining the residual chloroacetic acid and chloroacetate content of surfactants. It specifies a liquid chromatographic method for the determination of monochloroacetic acid and its salts and of dichloroacetic acid and its salts, and it applies to the surfactants manufactured using chloroacetic acid or sodium chloroacetate as a raw material - the alcohol and alkylphenol ether carboxylates, the undecyl imidazoline carboxylates, the fatty alkyl dimethyl betaines and the fatty acid amidopropyl dimethyl betaines among them. Chloroacetic acid is the reagent that puts the carboxylate group onto a surfactant. It is cheap, it works, and it is used at scale in the manufacture of the amphoteric and anionic surfactants that go into shampoos, body washes and baby products. It is also corrosive, acutely toxic and readily absorbed through the skin, and what remains unreacted in the finished surfactant goes into the product. Cocamidopropyl betaine, the most widely used amphoteric surfactant in personal care, is made this way, which is why a residue method for it is not an academic exercise: the material ends up on skin, daily, on people including infants. So the determination has to be sensitive at low levels and specific enough to separate monochloroacetate from dichloroacetate, which is the more toxic of the two and is present as a by-product rather than as an unreacted reagent. The standard specifies the liquid chromatographic conditions, the sample preparation, the calibration and the handling of samples whose content falls outside the calibration range -

by concentrating the standards or by diluting or reducing the test portion. Issued on 30 December 2011 and in force since 1 September 2012.

This standard specifies the use of liquid chromatography in a surfactant acid (salt) and dichloroacetic acid (salt) method for the determination of residual amount. This standard applies to a acid or sodium chloroacetate as raw materials to produce a surfactant, such as. alcohol, phenol ether carboxylates, undecyl microphone Oxazoline carboxylates, fatty alkyl dimethyl betaines, fatty acid amide propyl dimethyl betaine and other residual products of monochloroacetic acid (salt) and dichloroacetyl Determination of acid (salt) content. When a sample is acid (salt) and dichloroacetic acid (salt) content exceeds the range of the standard curve, the need to increase Concentration of the standard or sample dilution or reduction assay.

2 Normative references

The following documents for the application of this document is essential. For dated references, only the dated version suitable for use herein Member. For undated references, the latest edition (including any amendments) applies to this document. Laboratory use specifications and test methods GB/T 6682 Analysis

GB/T 13659 strongly acidic styrene cation exchange resin

3 Liquid chromatography

3.1 Principle Using reverse phase C8 column with a 10% aqueous acetonitrile (2.0mL per 1000mL was added phosphoric acid) as the mobile phase, with UV detection Or diode array detector at a wavelength of 214nm at the detection of a residual product acid (salt) and dichloroacetic acid (salt).

3.2 Reagents

3.2.1 water, in line with GB/T 6682 requested a water.

3.2.2 a TCA, analytical content of not less than 99%.

3.2.3 dichloroacetate, analytical content of not less than 99%.

3.2.4 acetonitrile, liquid chromatography special.

3.2.5 orthophosphate, AR.

3.2.6 hydrochloric acid solution, 11 (volume fraction), whichever is analytically pure hydrochloric acid 10mL and water (3.2.1) 10mL shake mix.

3.3 Instrument Common laboratory equipment and the following.

3.3.1 liquid chromatograph. with a data processing system, equipped with an ultraviolet

detector or diode array detector, the detector baseline noise in 254nm at not more than 2 x 10-5AU/s (empty tank); baseline drift at 254nm at not more than 1 x 10-3AU/h (empty pool, stable 60min).

3.3.2 Liquid Column. C8 bonded silica HPLC column, 250mm x 4.6mm (diameter), stationary phase particle size 5μm, or rather who, Applicable pH range from 1 to 8.

3.3.3 micro syringe, 25μL.

3.3.4 Analysis of balance, a sense of the amount of 0.1mg.