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OHV Analysis of Fatty Alcohol C18 Acetic Anhydride-Pyridine Method as Quality Control at PT X

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Abstract
Fatty alcohol C18 is an essential raw material in the surfactant, cosmetic, and lubricant industries whose quality is measured based on hydroxyl value (OHV) parameters. This study aimed to evaluate the OHV value of C18 fatty alcohol samples using the acetylation method of acetic anhydride-pyridine reagents, as well as verify its conformity with PT X's internal quality specifications. The results of the analysis showed that the average OHV value of the sample was 210.5 mg KOH/g, which consistently met the company's internal standard range of 205–215 mg KOH/g. The achievement of these values indicates a high level of hydroxyl group purity in the sample without significant contamination of short-chain alcohol fractions or residual water. Thus, this acetylation titrimetry method has proven to be accurate, reproducible, and very reliable to be applied as a routine testing procedure in PT X quality control system
Keywords: *acetic anhydride-pyridine, acetylation, fatty alcohol C18, hydroxyl value, quality Control.*

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INTRODUCTION

Fatty alcohol is one of the major oleochemical products widely used as a raw material in the manufacture of surfactants, detergents, personal care products, and various industrial chemicals. Among these, C18 fatty alcohol is commonly utilized because it contains a hydroxyl (-OH) functional group attached to its carbon chain, which plays an important role in determining the compound's physical properties and chemical reactivity. Therefore, the characterization of hydroxyl groups is essential to ensure that the product quality complies with the established specifications (Hartman Leopold & C.A. Lago Regina, n.d.).

One of the key parameters used to evaluate this characteristic is the Hydroxyl Value (OHV). The hydroxyl value represents the amount of hydroxyl groups present in one gram of sample and is expressed as the equivalent milligrams of potassium hydroxide (KOH). This parameter serves as an important quality control indicator because it reflects the purity of the material, the consistency of the production process, and the conformity of the product with applicable quality standards. A hydroxyl value outside the specified range may indicate changes in product quality or inconsistencies during manufacturing (AOCS ORGANIZATION, n.d.; ASTM International Organization, 2017)

Several analytical methods are available for determining the Hydroxyl Value, one of which is the acetic anhydride–pyridine method. This method is based on an acetylation reaction, in which the hydroxyl groups in the sample react with acetic anhydride in the presence of pyridine to form ester compounds. The excess reagent is subsequently hydrolyzed, and the remaining acetic acid is quantified by titration with a standardized alkaline solution. Owing to its high accuracy and precision, this method is widely employed in analytical laboratories and the oleochemical industry for hydroxyl value determination(ASTM International Organization, 2017; Lubrizol, 2007).

In quality control activities, hydroxyl value analysis plays a crucial role in assessing whether the produced material meets the company's quality specifications. This analysis also helps ensure batch-to-batch consistency, allowing the final product to maintain uniform characteristics that satisfy customer requirements. Therefore, the determination of the Hydroxyl Value of C18 fatty alcohol using the acetic anhydride–pyridine method was conducted as part of the quality evaluation process to verify compliance with the quality standards established by PT. X.

RESEARCH METHODS

The research used is a Laboratory Experiment study to determine the value of Hyroxyl Value (OHV) Fatty Alcohol C18 from PT X using the Titration method using Acetic Anhydride-Pyridine referring to ASTM D1957-86. The sample was reacted with a mixture of Acetic Anhydride and Pyridine, refluxed for 1 hour, hydrolyzed and then titrated using a 0.5 N KOH-Ethanol solution with a Phenolphthalein Indicator. The analysis was carried out seven times, the OHV value was calculated based on the difference in the titration volume of blanks and samples, The results were expressed as the average of SD ± and compared with the Specifications of PT X.

Materials and Tools

The materials used include Mineral Water, Pyridine Reagent, Anhydrous Acetate Reagent, Fresh Pyridine-Acetate Anhydrous 3:1 as much as 5mL, Phenolphthalein Indicator, Ethanol Ethanol Sodium Hydroxide Solution 0.5 N, Distillate Water, Neutral Ethanol. The tools used include Sartorius Analytical Balance, KEM EBU-610-20B dosimate titrator with Magnetic Stirrer, Erlenmeyer TS29/32 Ground joint 250 mL, Reflux Condenser with 300 nm Length, Oil bath with temperature of 100 ± 2ºC, Pipette Volume 5 mL, Measuring Pipette 10 mL.

Methods

The method using Acetic Anhydride-Pyridine referring to ASTM D1957-86.

Procedure

The working procedure is based on AOCS Cd 13-60 / EP Method A 2.5.3. A total of 2.0 ± 0.1 g samples were weighed into seven separate Erlenmeyer flasks containing 5 mL of Acetic Anhydride-Pyridine Reagent (3:1). Each sample flask was added 5 mL of Acetic Anhydride-Pyridine (3:1), then homogenized slowly. Next, the sample flask and blank are heated in a steam bath at 100 ± 2ºC for 1 hour using a reflux condenser. After heating, as much as 10 mL of aquadest is added through a fixed condenser. The condenser is then removed and rinsed along with the pumpkin using 25 mL of Neutral Ethanol. Next, 1 mL of Phenolphthalein (PP) Indicator is added, then the Titrated solution uses 0.5 N Ethanolic NaOH until a stable pink color is formed as the Titration End Point.

$$AOCS\ OHV = \frac{(Vb - Vs)x\ N\ x\ BE\ KOH}{\mathrm{W}}$$

Description :
Vb = Blank Titration Volume (mL)
Vs = Sample Titration Volume
N = Normality of Titran (molek/L)
W = Sample Weight (grams)

RESULTS AND DISCUSSION

Table 1. Determination of hydroxyl values by acetic anhydride pyridine method

Pengulangan	Hasil (mg KOH/g)
I	206,821
II	206,064
III	207,724
IV	207,307

V	205,509
VI	208,271
VII	208,386
Rata-rata	207,154

Formulation

OHV Calculation of Acetic Anhydride-Pyridine Method based on AOCS Cd 13-60, Hydroxyl Value.

$$OHV = \frac{(Vb - Vs)x\ N\ x\ BE\ KOH}{\text{W}}$$

Description: Vb = KOH titrant volume for blanks (mL); Vs = KOH titrant volume for sample (mL); N = normality of the KOH solution; BE KOH = KOH equivalent weight (g/mol); W = sample weight (g).

The hydroxyl value (OHV) analysis of fatty alcohol C18 using the acetic anhydride-pyridine method produced values ranging from 205.509 to 203.86 mg KOH/g, with an average OHV of 207,15 mg KOH/g. These results indicate good consistency among the measurements and remain within the company's quality specification of 205-208 mg KOH/g. The findings suggest that the fatty alcohol C18 sample contains an appropriate amount of hydroxyl groups and satisfies the required quality standards for oleochemical raw materials.

Since hydroxyl value reflects the concentration of hydroxyl (-OH) groups present in a compound, it is commonly used as an important indicator for evaluating fatty alcohol quality. The OHV values obtained in this study also imply that the hydrogenation and purification processes were successfully carried out, resulting in a product with the expected hydroxyl group content. Hydroxyl value is an important parameter in the characterization of oleochemical-based materials because it is directly related to the number of active hydroxyl groups that determine the reactivity of materials in advanced processes, such as the composition of surfactants, esters, and polyols (Li et al., 2024).

The acetic anhydride-pyridine method applied in this study is based on the acetylation of hydroxyl (-OH) groups using acetic anhydride, while pyridine functions as the reaction medium and catalyst. After the reaction is completed, the remaining excess reagent is hydrolyzed and quantified by titration, allowing the hydroxyl value to be determined indirectly. Owing to its good accuracy and reproducibillity, this method has been widely adopted as a standard procedure for determining hydroxyl values in hydroxyl-containing compounds (Petersen,Hedberg, & Christensen, 1943).

Altough slight differences were observed among replicate measurements, the variations were relatively small and did not influence the conclusion that the samples complied with the specified quality requirements. Minor differences in the obtained hydroxyl values may arise from several factors, including inaccuracies during sample weighing, fluctuations in the normality of the titrant, variations in sample homogeneity, and uncertainty in determining the titration endpoint. Nevertheless, when experimental conditions are properly controlled, the acetylation method is capable of providing hydroxyl value measurements with high precision (Fernandes et al.,2014). Therefore, the results of this study show that the acetic anhydride-pyridine method can be used as a reliable method for quality control fatty aalcohol C18 in PT X.

CONCLUSIONS

Determination of hydroxyl value in C18 fatty alcohol samples using the acetic anhydride-pyridin acetylation method resulted in an average OHV value of 207.154 mg KOH/g. These results meet the specifications of PT X's internal quality standards which require a range of 205-208 mg KOH/g. acetic anhydride-pyridine method has been proven to be used as a routine analysis method in the company's quality control system.

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