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**INDUSTRY STANDARD OF THE
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Chemical analysis of nickel laterite ore - Part 6: Determination of nickel, calcium, titanium, magnesium, copper, cobalt, chromium, zinc and phosphorus content - Inductively coupled plasma atomic emission spectrometric method

红土镍矿化学分析方法 第6部分：镍、钙、钛、锰、铜、铬、锌、磷含量的测定
电感耦合等离子体原子发射光谱法

(English Translation)

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1 Scope

This standard specifies a gas chromatographic method for determining the free glycerol content in crude glycerol.

This standard applies to crude glycerol, which are byproducts of biodiesel production with a mass fraction of less than 95%, industrial-grade saponified glycerol, and synthetic glycerol.

2 Method summary

After acetic anhydride is added, the sample is heated, and a derivatization reaction is carried out to generate glycerin triacetate, which is then determined by using a gas chromatograph and quantified using the internal standard method.

3 Reagents

Unless otherwise specified, only reagents confirmed to be of analytical grade shall be used in the analysis.

3.1

Glycerol standard ($C_3H_8O_3$). with a content of greater than 99%.

3.2

Acetic anhydride ($C_4H_6O_3$). with a content of greater than 95%.

3.3

Internal standard. ethyl benzoate (C₉H₁₀O₂), with a content of greater than 99%.

4 Apparatus

4.1

Gas chromatograph. It is equipped with a flame ionization detector (FID).

4.2

Microsyringe. 10 µL.

4.3

Pipettes. 1 mL, 25 mL.

4.4

Dropping bottle. 25 mL.

4.5

Stoppered conical flask. 125 mL.

4.6

Analytical balance. with a sensitivity of 0.1 mg.

5 Sample

The crude glycerol sample is thoroughly mixed and placed in a dropping bottle for later use.

6.1 Sample preparation

Weigh 0.4 g~0.5 g of the above crude glycerol sample into a stoppered conical flask using the differential weighing method, accurate to 0.2 mg.

6.2 Derivatization

Add 25 mL of acetic anhydride (3.2) to a stoppered conical flask containing the glycerol sample, and place it in a 120 °C oven to react for 2 h. Remove and cool to room temperature.

6.3 Chromatographic conditions

See Appendix A.

6.4 Preparation of standard solutions

Weigh 0.1 g, 0.2 g, 0.3 g, 0.4 g, and 0.5 g of glycerol standard (3.1) into stoppered conical flasks using the differential weighing method, accurate to 0.2 mg, and derivatize them together with the sample according to 6.2.

6.5 Measurement

Take the derivatized sample solution (6.2) and standard solution (6.4), accurately transfer 1 mL of ethyl benzoate (3.3) into an Erlenmeyer flask, shake well, take the supernatant, and proceed with the conditions as described in 6.3. See Appendix B for the gas chromatogram of the glycerol derivative.

6.6 Blank test

Perform a blank test on acetic anhydride according to the above determination steps.

7.1 Plotting the standard curve

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