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Quantitative Determination of Hydroxyl Groups

Practical 9 - Analytical practical

Aim

To estimate hydroxyl-group content through an adopted derivatisation and back-titration method.

Principle

In a suitable hydroxyl-number method, hydroxyl groups react with a defined acylating reagent. The difference between blank and test back-titration relates to hydroxyl equivalents. Water, acidity and other reactive groups may require specific corrections.

The test preparation, blank, calibration or titrant standardisation and independent controls are part of the measurement. State the actual concentration units, test conditions and adopted method before interpreting the result.

Materials and Requirements

- Adopted hydroxyl-number method and representative material
- Instructor-prepared method reagents and standardised titrant
- Controlled reaction and endpoint equipment
- Qualified balance and volumetric equipment
- Controlled method and raw-data worksheet

Procedure

1. Identify the material, intended attribute, method version and acceptance criteria. Record preparation and instrument or reagent identities.
2. Prepare the prescribed blank and reference or standardised reagent. Verify the measurement with an independent check where applicable.
3. Record the method scope and the reactions of the selected material, including acidity and water corrections.
4. Treat weighed test and blank preparations under the same prescribed derivatisation conditions.
5. Complete the specified reaction work-up and back-titrate to the defined endpoint.
6. Calculate hydroxyl number with the method-required correction rather than assuming every volume difference represents hydroxyl groups.
7. Record individual readings for independently prepared test portions. Keep all raw observations, including unsuccessful measurements and their investigation.
8. Calculate the result on the stated mass, volume or area basis, including dilution, blank and recovery corrections as justified.
9. Compare the evidence with the applicable criteria and write a conclusion limited to the tested preparation and conditions.

Observations and Record

Enter actual measurements or the supplied teaching data. Record the method, material identity, conditions and units.

Preparation / attribute	Method condition	Raw measurement	Calculated result
Blank consumption	Record	Record	Calculate
Test consumption	Record	Record	Calculate

Preparation / attribute	Method condition	Raw measurement	Calculated result
Mass and corrections	Record	Record	Calculate
Hydroxyl number	Record	Record	Calculate

Calculations

• For the stated equivalent basis: hydroxyl number (mg KOH/g) = 56.1 x N x (Vblank - Vtest) / mass (g), with method-specific corrections

Worked Example

Illustrative blank and test consumptions are 12.00 and 8.00 mL of 0.500 N titrant for 2.00 g material.

Uncorrected hydroxyl number = 56.1 x 0.500 x 4.00 / 2.00 = 56.1 mg KOH/g.

Apply the adopted acidity and other corrections before reporting a formal result.

Result and Interpretation

• Interpret the value as an equivalent-based group determination under the selected method, not a complete structural identification.

• Report replicate variability and the evidence that the method can measure the intended attribute in this matrix.

Precautions

• Handle reactive derivatisation reagents through the approved hood procedure.

• Control moisture and blank treatment.

• Confirm the direction of the back-titration difference.

• Use the adopted laboratory procedure, suitable controls and the designated waste arrangements.

Viva Questions and Answers

What does hydroxyl number express?

A hydroxyl-equivalent amount on a potassium-hydroxide mass basis per gram.

Why control water?

Water can consume derivatisation reagent and bias the result.

Why is the blank important?

It establishes reagent consumption without the test material.

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