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Rancidity in Lipsticks and Hair Oils

Practical 18 - Analytical practical

Aim

To compare appropriate rancidity indicators in lipstick and hair-oil preparations.

Principle

Acid value measures neutralisable acidity, saponification value measures alkali consumption under the specified saponification process, iodine value describes halogen uptake under its method, and peroxide value assesses primary oxidation products. These indices answer different quality questions.

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

- Representative oil and the selected adopted methods
- Standardised alkali, acid and thiosulphate reagents as required
- Appropriate blanks and controlled reaction 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. Choose the individual test and record the oil mass, reagent identity, concentration and method conditions.
4. Perform acid-value titration with the prescribed solvent and endpoint.
5. For saponification value, treat test and blank under the same defined saponification and back-titration conditions.
6. For iodine and peroxide determinations, use their separate reagent and endpoint procedures and preserve the required light and timing controls.
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
Index	Record	Record	Calculate
Oil mass	Record	Record	Calculate

Preparation / attribute	Method condition	Raw measurement	Calculated result
Blank and test volumes	Record	Record	Calculate
Calculated index	Record	Record	Calculate

Calculations

- Acid value = 56.1 x N x (Vtest - Vblank) / m, in mg KOH/g
- Saponification value = 56.1 x N x (Vblank - Vtest) / m, in mg KOH/g
- Iodine value = 12.69 x N x (Vblank - Vtest) / m, in g iodine/100 g
- Peroxide value = 1000 x N x (Vtest - Vblank) / m, in milliequivalents/kg

Worked Example

Illustrative acid value: m = 2.00 g, N = 0.100 and corrected volume 1.00 mL give 2.805 mg KOH/g.
Illustrative saponification value: m = 2.00 g, N = 0.500 and blank-minus-test difference 14.00 mL give 196.35 mg KOH/g.
Illustrative iodine value: m = 0.500 g, N = 0.100 and difference 10.00 mL give 25.38 g iodine/100 g.
Illustrative peroxide value: m = 2.00 g, N = 0.0100 and corrected volume 0.200 mL give 1.00 milliequivalent/kg. Each example belongs to its own test.

Cosmetic matrix preparation

- Use separate validated extraction or dissolution procedures for the wax-rich lipstick and the liquid hair-oil matrix.
- Assess relevant peroxide and acid values with blanks and matrix recovery. Odour and colour changes are supporting observations, not substitutes for chemical evidence.

Result and Interpretation

- Report each index with its units and separate method conditions. Apply the relevant oil monograph or material specification.
- Report replicate variability and the evidence that the method can measure the intended attribute in this matrix.

Precautions

- Record normality consistently with the formula.
- Check the direction of each blank correction.
- Control oxidation during storage and handling.
- Use the designated precautions for corrosive and halogen-containing reagents.
- Use the adopted laboratory procedure, suitable controls and the designated waste arrangements.

Viva Questions and Answers

Are acid value and peroxide value interchangeable?

No; they assess different chemical changes.

Why use a saponification blank?

It establishes the reagent consumption basis for the back-titration.

Does iodine value directly measure rancidity?

It primarily characterises unsaturation under its method.

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