Method 17.0

Cinnamic Aldehyde in Cassia Oil

Purpose: To determine the amount of cinnamic aldehyde in steam volatile oil from cassia or cinnamon by indirect titration.

A. Apparatus:

1. Erlenmeyer flask, 500 mL with ground glass stopper.
2. Beaker, tall, 500 mL.
3. pH meter.
4. Buret, 50 mL (graduated 0.1 mL).
5. Magnetic stirrer and teflon covered stirring bar.
6. Graduated cylinder, 250 mL.
7. Pipette 50 mL, TD.

B. Reagents:

1. Sulfuric acid, standard solution, 1.00 N.
2. Sodium hydroxide, standard solution, 1.00 N.
3. Sodium sulfite, 1.0 M solution adjusted to pH 9.6.
4. Sodium sulfate (Na_2SO_4) - Anhydrous, ACS grade.

C. Preparation of Sample:

1. The steam volatile oil obtained by method 16.0 or alternatively, Methods 5.0, 5.1 or 5.2, contains xylene and some moisture (Note 1). Dry the oil by adding anhydrous Na_2SO_4 and allow this mixture to stand for one hour. Pipette a 2.00 mL sample into the flask and weigh. Calculate the weight (See E. Calculations).

D. Procedure:

1. Weigh accurately ca. 1 g of cassia oil into a 500 mL Erlenmeyer flask.

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2. Add 250 mL of 1.0 M sodium sulfite solution, from a graduated cylinder.

3. Pipette 50.00 mL of the standard sulfuric acid solution into the flask with continuous agitation. Stopper the flask and shake vigorously for 5 minutes.

4. Quantitatively transfer the contents of the flask to a 500 mL beaker.

5. Insert the electrodes from a pH meter and titrate to pH 9.6 with a standardized sodium hydroxide solution. Stir the solution continuously during the titration, using a magnetic stirrer.

E. Calculations:

Wt. of the oil = wt. of sample - wt. of xylene

$$\text{Wt. of xylene} = \text{mL xylene} \times \frac{0.860 \text{ g xylene}}{\text{mL xylene}} \times \frac{2.00 \text{ mL sample}}{(\text{mL oil} + \text{mL xylene})\ (\text{Note 2})}$$

$$\text{Cinnamic aldehyde, \%} = \frac{(A-B) \times N \times 0.1322}{2 \times \text{weight of oil(g)}} \times 100$$

Where A = mL of standard base required to neutralize 50 mL of the standardized acid solution.
B = mL of standard base required for the sample.
N = norality of the sodium hydroxide solution.

F. Statistics:

TBD

G. Notes:

1. The xylene volume left in trap is determined by running blanks. See Method 16.0.
2. Total volume obtained in volatile oil determination.

H. Reference:

N/A