CARBOXYL

PRINCIPLE

The carboxyl containing starch is leached with hydrochloric acid to convert carboxyl salts to the acid form. Cations and excess acid are removed by washing with water. The washed sample is gelatinized in water and titrated with standard alkali.

SCOPE

The procedure is applicable to commercial oxidized starches and to many granular starch derivatives containing added carboxyl groups.

REAGENTS

1. Hydrochloric Acid Solution, 0.10 *N*: Standardization unnecessary

2. Sodium Hydroxide Solution. 0.10 *N*: Standard

3. Phenolphthalein Indicator, 1%

PROCEDURE

If necessary, grind sample completely through a laboratory cutting mill to 20 mesh or finer, taking precautions to prevent any significant change in moisture, and mix thoroughly.

Weigh accurately a sample containing not more than 0.25 milli-equivalents of carboxyl (Note 1), and transfer quantitatively to a 150 mL beaker. Add 25 mL of 0.10 *N* hydrochloric acid and stir occasionally for 30 minutes. Vacuum filter the slurry through a medium porosity fritted-glass crucible or small funnel, using a fine stream of water from a wash bottle to aid quantitative transfer of the sample. Wash the sample with purified water (300 mL is usually sufficient) until the filtrate is free of chloride determined by silver nitrate test (Note 2).

CARBOXYL — continued

Transfer the demineralized sample quantitatively to a 600 mL beaker with purified water, and slurry the sample in 300 mL of purified water. Heat slurry in a steam bath or boiling water bath (Note 3), stirring continuously until the starch gelatinizes. Continue heating for 15 minutes to insure complete gelatinization (Note 4).

Remove sample from bath and titrate while hot with standard 0.10 *N* sodium hydroxide solution to a phenolphthalein end point. The end point may be detected electrometrically at pH 8.3.

Perform a blank determination on the original sample to correct for native acidic substances (Note 5). Weigh an equivalent amount of starch as used for carboxyl titration, and slurry in 100 mL of purified water. Stir at 5-minute intervals for 30 minutes. Vacuum filter the slurry quantitatively through a medium porosity fritted-glass crucible or small funnel, and wash sample with 200 mL of purified water. Transfer, gelatinize and titrate the sample with standard 0.10 *N* sodium hydroxide in the same manner as the demineralized sample.

CALCULATION

$$\% \text{ Carboxyl} = \frac{(\text{Sample Titer, mL} - \text{Blank Titer, mL})\,(0.0045)\,(100)}{\text{Sample Wt., (g)}}$$

NOTES AND PRECAUTIONS

1. Sample size should not exceed 5.0 g for a mildly oxidized starch nor less than 0.15 g for a highly oxidized commercial starch.

2. Add 1 mL of 1% aqueous silver nitrate solution to 5 mL of filtrate. Turbidity or precipitation occurs within 1 minute if chloride is present.

3. Heating on a hot plate or over a Bunsen burner is not recommended. Overheating or scorching in amounts too small to be visible will cause sample decomposition and high carboxyl results.

4. Thorough gelatinization facilitates rapid titration and accurate end point detection.

CARBOXYL — continued

5. A blank titration is run on a water washed sample to correct for acidic components which are not introduced by oxidation or derivatization. Free fatty acids complexed with amylose are the principal contributors to the blank titer.

METHOD HISTORY

Corn Starch (Modified), Carboxyl (C-22), Date of Acceptance 5-16-1966, Revised 3-31-1992.