

# A Note on the Determination of Salt in Self-Rising Flour

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The importance of controlling the amount of salt in self-rising flour is recognized by millers and cereal chemists. However, no standard method for salt in flour has been available. In private communications several millers and chemists have indicated that in their practice the presence of approximately the desired amount of salt was determined by taste alone; they were concerned that a precise method was not available.

Recently Byer<sup>1</sup> reported an electrometric method that is very efficient for routine analyses of large numbers of samples. The method reported here is expected to be of value in analyzing occasional samples. It can be used when a pH meter with alternate millivolt scale is not available.

## Equipment

1. Vacuum filtering flask, 500-ml. capacity.
2. Erlenmeyer flask, 500-ml. capacity.
3. Büchner funnel, 75-mm.
4. Filter paper, Whatman No. 1, 7.0-cm.
5. Filter tablets, Whatman, 2.4 g.
6. Buret, 50-ml.
7. Graduated cylinder, 100-ml. capacity.
8. Water aspirator, to provide source of vacuum for filtration.
9. Balance, capable of weighing to the nearest 0.05 g.
10. Magnetic stirrer and Teflon-covered stirring bars.
11. pH test paper, pHDrion or equivalent.

## Reagents

1. Standard 0.1N Silver Nitrate Solution (Bio-Rad Laboratories, 32nd and Griffin Ave., Richmond, Calif.).
2. Neutral chromate indicator. Dissolve 4.2 g. potassium chromate and 0.7 g. potassium dichromate in 100 ml. water.

## Analysis of Sample

1. Weigh out  $3.0 \pm 0.05$  g. of flour into the 500-ml. Erlenmeyer flask.
2. Add 100 ml. distilled water, a filter tablet, and a magnetic stirring bar.
3. Swirl flask and contents vigorously by hand to disperse flour. Make sure none sticks to bottom of flask.
4. Set Erlenmeyer flask on magnetic stirrer and stir vigorously for 2 min.
5. While sample is being stirred, place a 7.0-cm. filter paper (Whatman No. 1) into the Büchner funnel. Mix a filter tablet with a little water, then pour resulting slurry onto filter paper; this will keep filter from clogging while sample is being filtered.
6. After 2-min., filter contents by vacuum through 75-mm. Büchner funnel into 500-ml. vacuum filter flask.
7. Rinse Erlenmeyer flask twice with about 20-ml. portions of distilled water and filter these through Büchner funnel.
8. Check pH of combined filtrate with a wide-range pH test paper. If pH is not within 5.5-10.0, adjust as needed, using dilute nitric acid or sodium bicarbonate.
9. Place a magnetic stirring bar into filtration flask containing combined filtrates. Add approx. 1 ml. of neutral chromate indicator.

<sup>1</sup> Byer, M. J. Rapid analysis of salt, soda, and shortening stability. Symposium on Specialized Testing, Central States Section AACC, St. Louis, Mo., Feb. 19, 1966.

10. Set flask on magnetic stirrer and stir at a rapid rate.
11. Titrate with 0.1*N* silver nitrate. Near end point, titrate dropwise until first pink color persists for 15 sec.
12. Calculate concentration of salt as follows:

$$\text{Percent NaCl} = \frac{\text{ml. titration} \times N \text{ AgNO}_3 \times \text{g.-meq. NaCl} \times 100}{\text{sample weight}}$$

Since a fixed normality is used:

$$\text{Percent NaCl} = \frac{\text{ml. titration} \times 0.585}{\text{sample weight}}$$

$$\text{Percent salt (corrected)} = \text{Percent NaCl of sample} - \text{Percent NaCl of blank.}$$

### Results and Discussion

The method was evaluated by analyzing standard self-rising flour samples prepared in the laboratory. Recovery data are tabulated below:

<i>NaCl Added</i> %	<i>NaCl Found</i> %	<i>Deviation</i> %
1.90	1.91	+0.01
1.90	1.88	-0.02
1.90	1.86	-0.04
1.90	1.90	0
0.66	0.66	0
1.33	1.35	+0.02

Because flour contains substances that interfere with the test—e.g., protein, minerals, etc.—a blank must be determined by performing the same analysis on the flour. A soft wheat patent flour that was highly bleached with chlorine (pH 4.8) gave a blank equivalent to 0.4% salt. The blank for a semi-hard patent flour with a low to medium bleach (pH 5.6) was equivalent to 0.3% salt. Omitting the phosphate leavening acid from the blank reduced the equivalent amount of salt from 0.3 to 0.2%. Also, gross variations in the amount of sodium bicarbonate present could slightly affect the value for the blank. Thus, all ingredients except salt should be present in the blank.

The procedure is intended for use with self-rising flours containing soda, salt, and orthophosphate; a pyrophosphate leavening system would introduce major errors. Several indicators were investigated, including some absorption indicators, to determine which gave the most visible end point. The neutral chromate indicator gave the best results; it also acts as a buffer and will automatically adjust solutions of initial pH of 5.5 to 10.0. The filtration step was required because the flour gave an opaque slurry that obscured the end point.

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