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SELENIUM AS A CATALYST IN THE DETERMINATION OF NITROGEN
IN COTTON SEED MEAL BY THE KJELDAHL METHOD

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The method almost universally employed for the determination of nitrogen in agricultural and food products is the well known one devised by Kjeldahl (1) in 1883. Briefly, it consists in boiling the sample in concentrated sulfuric acid which oxidizes the carbonaceous matter and converts the nitrogen into ammonia. The ammonia at once reacts with the excess acid forming ammonium sulfate. The acid solution is then diluted, an excess of caustic soda added, and the mixture distilled. The ammonia set free is received in a measured volume of standard acid, the excess of which is titrated with standard alkali.

Numerous modifications of the original method have been proposed and used, most of them designed to shorten, by means of some catalyst, the period of digestion with the sulfuric acid. Quite a number of substances seem to have the desired catalytic effect, compounds of copper and mercury being most effective.

In 1931, Lauro (2) showed that a small amount of selenium, or certain selenium compounds, if used as catalyst, would shorten the time of digestion about one half. To date, since Lauro's work, Chemical Abstracts lists over forty papers having to do with selenium as a catalyst in the determination of nitrogen by the Kjeldahl method. While his work has been tested, verified, and accepted by a large number of investigators, a few, mostly in the field of cereal chemistry, complain of low results with selenium, and it has not replaced the older catalysts in the official methods. For example, the National Cottonseed Products Association still use mercury or mercuric oxide as the catalyst in their procedure (3) which is essentially as follows.

"Digest 1.7034 grams of sample with approximately 0.5 gram metallic mercury or 0.7 gram mercuric oxide, 10 grams of sodium or potassium sulfate, and 25 cc. of sulfuric acid (Sp. G. 1.84). Place the flask in an inclined position and heat below the boiling point of the acid for from five to fifteen minutes, or until frothing has ceased. Increase the temperature and continue digestion until the liquid has become colorless or until complete digestion has been obtained." (It is common practice to digest 70 minutes.)

The above procedure not only requires over an hour for digestion but requires the addition of sodium sulfide to precipitate the mercury prior to the distillation of the sample. The present work was undertaken to obtain information on the role of selenium as a catalyst in the determination of nitrogen specifically, in cotton seed meal, for obviously, if selenium could be used as a catalyst instead of mercury, not only would the more or less troublesome use of sodium sulfide be avoided, but a great saving in time of digestion would be effected, and time is an important item in the commercial testing laboratories during the height of the cotton ginning season.

In this investigation, nitrogen was determined in three different samples of cotton seed meal, first by the official method with mercury as catalyst, then by exactly the same procedure but substituting selenium for mercury, and omitting

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the sodium sulfide treatment. The amount of selenium and the time of digestion was purposely varied somewhat. The following results were obtained.

Amount selenium as catalyst	Time of Digestion	Nitrogen Se catalyst		Nitrogen Off. Method
0.75 g.	35 min.	6.48		
0.75 g.	35 min.	6.50		
0.75 g.	35 min.	6.48		
0.75 g.	45 min.	6.48		
0.75 g.	45 min.	6.52	Average	6.56
0.75 g.	45 min.	6.48	6.49	
1.00 g.	45 min.	6.48		
1.00 g.	45 min.	6.48		
1.00 g.	45 min.	6.48		
0.50 g.	35 min.	8.10		
0.50 g.	35 min.	8.18		
0.50 g.	35 min.	8.08	8.10	8.23
0.50 g.	35 min.	8.08		
0.50 g.	35 min.	8.07		
0.50 g.	35 min.	7.85		
0.50 g.	35 min.	7.86		
0.50 g.	35 min.	7.86	7.86	7.88
0.50 g.	35 min.	7.89		
0.20 g.	35 min.	7.86		
0.20 g.	35 min.	7.83		

These results seem to warrant the conclusion that selenium as a catalyst in the determination of nitrogen in cotton seed meal by the Kjeldahl method, with a shorter digestion period, gives values in fairly close agreement with those obtained by the official method but definitely lower. This supports the claim of others that selenium is an effective catalyst but in some cases gives low results.

Literature Cited

- (1) Kjeldahl, "A New Method of Determining Nitrogen in Organic Materials" *Z. anal. Chem.*, 22, 366.
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- (3) U. S. Dept. Agriculture. 1939. "Methods of Chemical Analysis and Grade Calculations for Cottonseed", p. 4.