

ing to 2 grams of soil is filtered off into a porcelain dish and evaporated to semi-dryness on a water-bath, finishing by heating cautiously over a gentle flame, being careful that loss occurs neither from creeping nor decrepitation. When dry, ignite gently to decompose oxalates and expel ammonium salts. Take up with 10 to 15 cc. of hot distilled water, acidify with 3 or 4 drops of hydrochloric acid, and filter. Determine the potash in the filtrate by precipitation with chloroplatinic acid, etc., as directed in the regular Lindo-Gladding method.

[CONTRIBUTION FROM THE UNIVERSITY OF ILLINOIS, AGRICULTURAL
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IMPROVED METHOD FOR SEPARATION AND DETERMINATION OF TOTAL ALKALIES IN SOILS.

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WHILE working upon soils in this laboratory, considerable difficulty was found in the determination of the total alkalies. The method used was that outlined in the "Methods of Analysis of the Association of Official Agricultural Chemists." By this method the iron, aluminum, phosphorus, and magnesium are removed from the hydrochloric acid solution by precipitating with barium hydroxide. From the filtrate, barium and calcium are precipitated by ammonium hydroxide and ammonium carbonate. This filtrate is evaporated to dryness, the ammonium salts driven off, and the residue taken up with a little hot water. Ammonium carbonate is again added to remove the last traces of barium, the solution filtered, the filtrate evaporated to dryness in platinum dishes, and the chlorides of the alkalies ignited.

It was found, however, that considerable barium carbonate remained with the ignited alkalies. This appeared as a thin white layer in the bottom of the platinum dishes, when hot water was added. Also, by the addition of ammonium carbonate to this more concentrated solution, more barium was precipitated. This process of precipitation, filtration, and evaporation was necessarily repeated five or six times before a constant weight of chlorides was obtained. Further, it was found that in this solution, in which ammonium carbonate finally gave no precipitate, the addition of a solution of ammonium sulphate gave a precipitate of

barium sulphate. Ten determinations of the barium in such solutions gave the following weights of barium sulphate:

	Gram.		Gram.
1.....	0.0065	6.....	0.0080
2.....	0.0083	7.....	0.0113
3.....	0.0088	8.....	0.0107
4.....	0.0075	9.....	0.0099
5.....	0.0061	10.....	0.0111

As the weight of the total alkalies is often under 20 mg. the presence of such amounts of barium would give rise to considerable error in the determination. Accordingly, the usual method was modified as follows: Just previous to evaporating the solution of the chlorides, in platinum dishes, a few cubic centimeters of an ammonium sulphate solution—75 grams to the liter—are added. The precipitate is filtered out and the filtrate evaporated as usual, in platinum dishes, and ignited. In this way, the barium is completely removed at one operation, and the alkalies are changed into sulphates, which can be ignited over the full heat of the Bunsen burner without danger of loss through volatilization. The evaporation, however, must be to complete dryness, otherwise there is danger of loss, through the decrepitation of the sulphates upon ignition. After igniting to a red heat, about 1 gram of dry, powdered ammonium carbonate is added to the dish and volatilized, thus breaking up any acid sulphates which may have formed.

As a check upon the possibility of loss through volatilization, during the ignition, four determinations of the sodium, the more volatile of the two alkalies, as sulphate in a solution of chemically pure sodium chloride, gave the following weights of sodium sulphate: 0.0291, 0.0292, 0.0291 and 0.0290 gram. Repeated ignition over the Bunsen burner did not alter these weights. The weight of sodium sulphate, calculated from the weight of sodium chloride used, was 0.02916 gram.

Below are some duplicate determinations of total alkalies obtained by this method, in our ordinary work:

Total alkalies.		Total alkalies.	
Gram.	Gram.	Gram.	Gram.
1.....	0.0191	9.....	0.0632
2.....	0.0383	10.....	0.0331
3.....	0.0288	11.....	0.0325
4.....	0.0285	12.....	0.0410
5.....	0.0332	13.....	0.0224
6.....	0.0128	14.....	0.0215
7.....	0.0324	15.....	0.0197
8.....	0.0124	16.....	0.0277
	0.0187		0.0636
	0.0378		0.0331
	0.0283		0.0328
	0.0286		0.0411
	0.0326		0.0221
	0.0130		0.0219
	0.0320		0.0196
	0.0123		0.0283

The advantages of this modified method are that the alkalies are entirely freed from barium in one operation and are transformed into sulphates, which do not present the difficulties of ignition found with the chlorides.

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DOES CHOLESTEROL OCCUR IN OLIVE OIL ?

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UPON this question there has been much uncertainty. Beneke¹ stated in 1862 that he believed he had obtained cholesterol from olive oil though the amount was too small to admit of proof. Salkowski² in 1887 found the amount obtainable from 50 grams too small for a determination of the melting-point, though the color reactions of phytosterol were given, and he expressed the opinion that olive oil contained neither phytosterol nor cholesterol but that the small amount present in the sample examined by him was due to the presence of olive kernel oil. Gerard³ reported in 1892 that he had obtained phytosterol from olive oil and had identified it by the melting-point. Bömer⁴ declared in 1898 that from two samples of Provence oil he had obtained an alcohol whose melting-point and crystal form left its identity with phytosterol beyond question. In the same year, however, Lewkowitsch⁵ stated definitely that "The unsaponifiable matter occurring in olive oil is cholesterol, whereas all other vegetable oils contain phytosterol," and in 1898, Allen⁶ also stated that "it (cholesterol) occurs in olive oil" and again that "Phytosterol is found in most vegetable oils with the notable exception of olive and palm." That Allen was not unfamiliar with the work of Bömer seems evident from the fact that he refers to the article in which the latter's work is described. Soltsien in 1901 reported the detection of phytosterol in olives from Bari pressed by himself.⁷

In view of the fact that the matter was of interest not only from

¹ *Ann. Chem. Pharm.*, **122**, 249-255.

² *Ztschr. anal. Chem.*, **26**, 581.

³ *Compt. rend.*, **114**, 1544-1546.

⁴ *Ztschr. Unter. Nahr. u. Genuss.* (1898), pp. 81-96.

⁵ "Chemical Analysis of Oils, Fats and Waxes," second edition, p. 452.

⁶ "Commercial Organic Analysis," third edition, Vol. II, Part I, pp. 347 and 351.

⁷ *Chem. Centrbl.*, **72**, II, 243 (1901).