

F (cf. Table I). The results obtained are shown in Table II.

TABLE I

Comparison of the Results Obtained in the Analysis of Soap for Silica Content by the Gravimetric and Volumetric Methods

| Sample | % SiO ₂ | |
|--------|--------------------|------------|
| | Gravimetric | Volumetric |
| A..... | 4.80 | 4.76; 4.84 |
| B..... | 9.86 | 9.94 |
| C..... | 6.58 | 6.71 |
| D..... | 10.71 | 10.58 |
| E..... | 11.93 | 12.03 |
| F..... | 10.08 | 10.07 |
| G..... | 7.46 | 7.37 |
| H..... | 14.48 | 14.48 |

TABLE II

Volumetric Estimation of SiO₂ vs. Gravimetric Determination Using Sample F (Table I)

| Determination No. | % SiO ₂ Volumetric Method | % SiO ₂ Gravimetric Method | Error |
|-------------------|--------------------------------------|---------------------------------------|--------|
| 1..... | 10.14 | 10.08 | +0.06% |
| 2..... | 10.10 | | +0.02% |
| 3..... | 10.25 | | +0.17% |
| 4..... | 10.08 | | 0.00% |
| 5..... | 10.12 | | +0.04% |
| 6..... | 10.07 | | -0.01% |

Discussion

The values given in Table I indicate that the method gives results agreeing satisfactorily with

those obtained by applying the more lengthy but exact gravimetric method. The utility of the volumetric method is due to the rapidity with which a determination may be run.

It has been found that if the paraffin is applied to the flask in a thin coating, it is quite adherent. Such coated flasks may be used for several determinations before the wax cracks away from the glass. A thin coating of wax is desirable, also, to enable better examination of the visual endpoint. It is unfortunate that owing to the nature of the reagents employed it is impractical to follow the course of this reaction electrometrically.

Acknowledgment

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REFERENCES

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Report of the American Representative on the Fat and Oil Commission of the International Union of Chemistry

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The following matters were considered by the commission and action taken as noted:

Samples. For large-size units, such as copra and olives, a minimum is 1 kilo; for all seeds except very small ones, 500 grams; for such as linseed, sesame, etc., 100 grams.

Moisture. For large samples the units are to be crushed but for very small seeds they may be used whole. A sample of 5 ± 0.5 gram weighed to 1 mg. is to be dried in a dish, preferably of aluminum, 7 cm. in diameter and 3-4 cm. high at $103 \pm 2^\circ$. Weighing is done after 3 hours then reweighed to constant weight, or until deviations between 2 weighings are no more than 5 mg.

Fatty Matter. The extraction shall be with petroleum ether distilling at $40-60^\circ\text{C}$., having a bromine index below 1. Use of pentane or normal hexane is permissible. The sample shall be 10 ± 1 gram accurate to 1 mg. The proposal of the Dutch commission to dry for 45 minutes at $103-5^\circ$ to inactivate enzymes was opposed by the British and French Commissions and set aside for further consideration. Wet seeds are to be dried at about 10% relative humidity. The first extraction is carried out with finely ground material for 4 hours, the second for 2 hours. The solvent is removed by distillation and final traces of solvent are removed by warming to 100° for not more than 20 minutes, at the same time passing a current of air.

Free Alkali in Soap. The barium chloride method was in general considered unsatisfactory. Absolute

alcohol shall be used with a 10-gram sample in amount such that the water in the soap will not dilute it below 95%. Dissolved gas in the alcohol shall be eliminated by refluxing for 10 minutes. The alcohol shall be neutralized to phenolphthalein at 70° with 0.1 N alcoholic potassium hydroxide. Then dissolve the soap by refluxing and titrate at 70°C . with 0.1 N alcoholic sulfuric acid, up to 0.1% alkali or 0.2 N for high concentrations.

Free Alkali Carbonate. For small amounts determine the carbon dioxide. Alternatively, after titration of free alkali, add an amount of carbon-dioxide-free water equal to the volume of alcohol and titrate to bicarbonate with 0.1 N sulfuric acid. Where the precipitate in determination of free alkali is appreciable, filter and subject to mineral analysis.

Rosin in Soap. The method of Vizern and Guillot was adopted in preference to the MacNickol method.

The Halphen test was preferred to the Liebermann-Storch for detection of small amounts of rosin.

Soluble and Insoluble Volatile Acids. The Reichert-Meissl value and Polenske number are to be expressed as mg. of potassium hydroxide per gram of sample. Distillation is to take 19-21 minutes. Cooling for 15 minutes at 15° is retained.

Discussion of details of the digitonine method for sterols, thiocyanate index, and peroxide index or Lea index was indecisive.

It is proposed to publish the methods of the Commission in 1947 and to reconvene in Paris in July, 1948.