precipitated on boiling. The former may be dialyzed, while the latter diffuses readily through parchment membranes. Proteolytic or peptic activity does not seem to be a part of the true physiological characteristics of the milk-curdling enzyme. These conclusions are based on the facts that pepsin digests 25,000 times its own weight of freshly coagulated and disintegrated egg albumen in $2^1/_2$ hours at 52°, the assay method of the U. S. Pharmacopeia, and that rennin is capable of coagulating more than 2,000,000 times its weight of fresh milk in 10 minutes at 40°.

Both enzymes are present in the stomach of the suckling calf, while in that of the adult hog only pepsin was found.

CHICAGO, ILLINOIS

NOTE

A Method for the Simultaneous Determination of Sulfur and Halogen in Organic Compounds.—When, in 1886, Peter Klason described a simplification[1] of his then new method[2] for the determination of sulfur in organic compounds, he stated that it probably would not supercede the Carius method, but might serve as a supplement to the latter. Through years of usage, however, in the laboratories of Sweden the process has received further simplification until to-day it surpasses the Carius method and rivals that of fusion in ease, speed and accuracy.

American chemists seem to be totally unacquainted with either the old or the new technique, so a summary of the present modified method as used in the laboratory of Bror Holmberg[3] at the Tekniska Hogskola, Stockholm (and also at the laboratories of Upsala and Lund) may serve to call attention to this facile analysis, useful not only for sulfur alone but for simultaneous determination of sulfur and halogen.

Procedure.—The process is carried out in a glass combustion tube in the combustion furnace. One end of the tube is drawn out and bent down to dip below the surface of 100 cc. of distilled water in a receiver flask. At the opposite end there is attached by a fume-treated cork a bulb of sulfur-free, fuming nitric acid. A stream of dry air or oxygen may be passed through acid and tube.

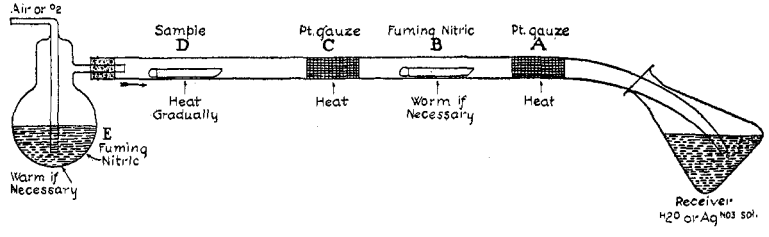
The latter contains, over the end of the furnace nearest the bent portion of the tube, a roll of freshly-ignited, fine-meshed platinum gauze (A) and then a porcelain boat (B) filled with fuming nitric acid. At the midpoint

[1] Klason, *Ber.*, **19**, 1910 (1886).

[2] Claësson, *Z. anal. Chem.*, **22**, 177 (1883).

[3] This method is used in the following work: Holmberg, Stereokemiska Studier, etc., *Z. anorg. Chem.*, **56**, 385 (1907); *J. Prakt. Chem.*, [2] **71**, 264 (1905); **75**, 170 (1907); **79**, 253 (1909); **81**, 451 (1910); **84**, 634 (1911); **87**, 456 (1913); **88**, 553 (1913); *Ber.*, **47**, 159, 167 (1914); *Ark. Kemi, Min. Geol.*, **6**, Nos. 1, 8, 17, 23 (1915–17); **8**, Nos. 2, 8 (1920–21). Leonard, Diethylrhodanine, *Medd. Vetenskapsakad. Nobelinst.*, **14**, No. 4 (1921).

another similar platinum gauze is placed (C). A second boat (D) containing the substance to be analyzed is pushed into the fore part of the tube after this has been prepared by heating the gauzes to dull redness and passing air to fill the tube with brown fume. A low flame is applied at or near the boat (B) of fuming acid in order to keep this portion of the tube filled at all times with brown nitrogen oxide fumes. After introduction of the boat containing the material to be analyzed, heat is applied as gradually as necessary to the tube near and finally to the region below the boat, as in ordinary combustions. However, some substances may at first oxidize without application of external heat; evidence of this is afforded by the production of white fumes. The speed of the combustion and the passage of air, together with the heat applied to the sources of fumes, must be so regulated that there is *never* an absence of brown fumes beyond A,[4] but white fumes may be permitted to appear just beyond Gauze C if this is immediately followed by application of a low flame to Bulb E (and perhaps by increased heat below Boat B) thus supplying more nitric fumes until the whole tube is again brown. Finally the material is driven down the tube by the application of heat, with the strict avoidance of heating to redness below the nitric acid boat, however. Combustion



must never become sufficiently rapid to cause flashes in the tube. There is most danger of this in burning with oxygen, as is sometimes desirable with refractory substances.

After the combustion the apparatus is cooled, the boats and platinum spirals are removed, placed in a clean evaporating dish, thoroughly rinsed and finally boiled with fresh water. The walls of the combustion tube are carefully rinsed with water into the receiver flask. All of the rinsings are then combined and, in the determination of sulfur alone, are heated on the steam-bath until all red fumes and the odor of nitric acid are gone; every precaution is taken to prevent loss of sulfuric acid. The sulfates are then precipitated with barium chloride, filtered and weighed as usual.

For halogen determinations alone, or for the simultaneous determination of halogen and sulfur, a blackened receiver-flask is used which con-

[4] It should be pointed out that in practice, no free halogen is evolved. The continual presence of an excess of the lower oxides of nitrogen, on which the whole process depends, assures the formation of NOCl, when chlorine for example, is determined.

tains a water solution of silver nitrate in a slight excess above that required for the chlorine content of the sample. The silver halide formed during the combustion and after the rinsing is filtered on a Gooch crucible, washed with warm 2% nitric acid and weighed as usual. For halogen alone, a Volhard titration can be made. In the combined determination, the filtrate and washing from the Gooch filtration are treated with an excess of hydrochloric acid to remove silver, and the filtered solution and wash waters then precipitated with barium chloride solution after the nitric acid has been removed, as described for the analysis for sulfur alone.

The amount of sample to be taken is from about 0.1 to 0.2 g. depending on the sulfur content; 0.1 g. suffices for 30–40% of sulfur. The combustion occupies $1^1/_2$ to 2 hours; the process is shorter but more difficult when oxygen is used. Platinum is the only expensive equipment. There is no sealing of tube, high temperature, or danger of explosion as with the Carius method. The simultaneous determination of halogen and sulfur makes possible the use of a very small sample for analysis.

WASHINGTON, D. C. CLIFFORD S. LEONARD[5]

Received October 21, 1922

[5] American Scandinavian Fellow, 1920–1921.

NEW BOOKS

Zirconium and Its Compounds. By FRANCIS P. VENABLE. American Chemical Society Monograph Series. The Chemical Catalog Company, Inc., 1 Madison Avenue, New York, U. S. A., 1922. 173 pp. 23.5 × 15.5 cm. Price $2.50.

Although zirconium is an element of perhaps minor importance from the standpoint both of its abundance and of its useful applications, it is a matter of very great consequence to have our knowledge of it concentrated in this available form. This monograph is, therefore, a most important one in this very important series of Scientific and Technologic Monographs which is being prepared under the auspices of the American Chemical Society, and Dr. Venable and the Publishers may well feel that they have made a distinct contribution to chemical science.

The purpose of the author is expressed in the following extract from the preface:

"I have not sought to record every observation or detail given in the literature, many of which are faulty or erroneous, but only such as seemed to have an essential bearing on the subject. My purpose has been to give a systematic, clear, and sufficiently full account of the chemistry of zirconium which should prove useful in connection with the increasing interest attaching to the element."

The personality of the author would be sufficient assurance that this purpose has been accomplished and it is perhaps needless for the reviewer even to state his own opinion to that effect.