

Determination of Sulphur in Organic Products

THE methods in general use for the determination of sulphur in animal and vegetable products for sulphur-balance experiments with cattle and sheep depend upon the oxidation of not more than a gram of material with copper nitrate (S. R. Benedict, 1909) or fusion with sodium peroxide (R. E. Evans, 1931). From a material like hay with a small percentage of sulphur, the amount of barium sulphate finally obtained from 1 gm. does not exceed 2.5 mgm., so that liability to error is considerable.

To overcome this disadvantage, F. J. Warth and T. S. Krishnan have devised a method in which much larger amounts of the substance can be treated, consisting in a nitric acid oxidation followed by alkali fusion, the latter step being essential for complete oxidation of the sulphur (*Ind. J. Veterin. Sci. and Animal Husbandry*, 5, Pt. 3, September 1935, p. 210). For urine, 100 c.c. may be treated with 10–15 c.c. concentrated nitric acid and slowly heated on a sand bath until gentle effervescence commences. The beaker is then removed, and the reaction allowed to proceed in the cold until complete. After re-heating, and cooling overnight, nitrophenols and hippuric acid are filtered off, the filtrate is concentrated to a small volume, rediluted and again concentrated, this treatment being repeated until dilution causes no turbidity. The yellow solution is then made up to 100 c.c., and an aliquot taken for fusion, for which 5 c.c. of 50 per cent sodium hydroxide are added, mixed, and the whole is transferred to a silver basin. The water is evaporated off, and the dry residue is heated on the sand bath and finally fused. The fusion is completed in a few minutes, resulting in a pure white melt which is dissolved in water, treated with

hydrochloric acid to remove nitric acid, filtered to remove small amounts of silver chloride and silica, and is then ready for precipitation of barium sulphate in the usual way.

For foodstuffs, such as hay and cake, and faeces, 5–10 gm. of substance is oxidised with boiling nitric acid for six hours. The contents of the flask are transferred to a beaker, diluted and allowed to stand overnight. The liquid is filtered, the filtrate concentrated in a beaker to a volume of 25 c.c., covered with a watch glass, and gently boiled and concentrated, which causes further oxidation with production of brown fumes. More acid is added, if necessary, until oxidation is complete. The liquid is then cooled, diluted and, if necessary, filtered. It is then evaporated on the water-bath, 10 c.c. of 50 per cent sodium hydroxide and 1–2 gm. of potassium nitrate are added, the whole is transferred to a silver dish, dried and fused.

For some materials, for example, grass, it may be desirable to deal with 100 gm. of material. For this, 100 gm. or thereabouts is weighed into a litre beaker, 500 c.c. of dilute (1 : 3) nitric acid is added, and the whole is heated in order to start a gentle reaction. The source of heat is then removed, and the reaction is allowed to proceed to completion, with stirring. The beaker is then again heated, covered and the contents boiled vigorously for twelve hours, with addition of water to maintain the volume. The mixture is allowed to stand overnight, filtered on a Büchner funnel and thoroughly washed with nitric-acid-water. The filtrate and washings are finally made up to 2,000 c.c., and an aliquot of 100 c.c. used for the determination of sulphur by fusion.

Pleistocene Chronology

VARIOUS lines of scientific investigation are now converging on the chronological problems of geology, prehistoric archaeology and palaeontology. The investigation of sedimentary deposits in Scandinavia by De Geer, and A. E. Douglass's tree-ring chronology in the south-western United States are instances in point. The application of the results of the study of solar radiation to the Pleistocene chronology of Central Europe has been reviewed by Dr. Friedrich E. Zeuner in relation to the evidence of geology, prehistoric archaeology and palaeontology (*Geol. Mag.*, 72, 19, 350–376; 1935).

During the last twenty years, the Pleistocene of Central Europe has been investigated by a great number of geologists, with the result that a detailed stratigraphy has been established, which is generally applicable. When the 'radiation curve' of Milankovitch is applied to this stratigraphy, it becomes possible to date exactly fossils, skeletal remains and prehistoric implements from certain localities in thousands of years.

On the evidence of the north German area of glaciation, the Alpine area of glaciation and the 'periglacial zone' (middle and south Germany), it is possible to formulate a correlation for which, as a matter of convenience, the Alpine terminology of Penck and Brückner may be used. This system of stratigraphy extends over a wide area. The Silesian Pleistocene links up the north-west and mid-German areas with Poland and Russia, while the Ukrainian loesses admit of a detailed subdivision with the same accuracy as the loess of Central Europe. In north Russia the same conditions prevail as in Germany, and the Caucasian mountains exhibit the same Pleistocene divisions. The later glacial phases (Riss and after) are thus represented in the whole of Central and Eastern Europe.

An outstanding point to emerge is that every glaciation of the old north German and Alpine divisions now appears to consist of two cold phases, which belong closely together. The Würm glaciation, however, includes a third and smaller phase. Two