Determination of Traces of Oxygen in Sodium Metal by Infra-red Spectrophotometry

THE principal methods for the determination of traces of oxygen in sodium metal are based on the titration of sodium monoxide after the metal has been amalgamated and the oxide filtered off^{1,2}; distilled in vacuum^{3,4}; or allowed to react with an alkyl halide⁵. A 2 gm. sample of sodium metal containing 50 p.p.m. oxygen contains 1.25×10^{-5} equivalents of sodium monoxide. In the alkyl halide method, the presence of a 7×10^3 fold quantity of sodium bromide introduces difficulties in the determination of sodium monoxide by conventional volumetric methods. Elsewhere this difficulty has been discussed and a solution suggested⁶. The amalgam and distillation methods also suffer from some disadvantages associated with quantitative separation and subsequent estimation of the oxide.

Fig. 1. Oxygen content of sodium metal by infra-red spectrophotometry

The use of an infra-red pressed-disk technique⁷ with the alkyl halide method obviates difficulties associated with determinations of sodium monoxide in the range 5–100 p.p.m. in sodium metal. Following the reaction of the sodium metal with the alkyl bromide⁵, the reaction mixture is dissolved in water and the unchanged oxide (now hydroxide) is converted into sodium bicarbonate by passing in excess carbon dioxide. The solution is then freeze-dried and the resultant powder heated at 400° C. for 2 hr. 1,500 mgm. of the sodium bromide–sodium carbonate mixture is then compressed to a clear disk by the usual technique⁷. Infra-red spectrophotometric analysis, using a Perkin Elmer model 21 infra-red spectrophotometer, shows absorption bands at 4.00, 6.93, 11.38 and 14.19μ. The absorbance at 11.38μ due to sodium carbonate is not subject to any interference, and although Lambert's law is not obeyed, from a standard curve (Fig. 1) the oxygen concentration in the original metal can be estimated with an accuracy of approximately 10 p.p.m. by weight.

The infra-red procedure also opens the way for the simultaneous determination of other impurities in

sodium metal provided that they may be converted quantitatively to compounds with absorption bands in the infra-red region. Full details of the above method will be reported elsewhere.

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A New Configuration of Deoxyribonucleic Acid

STRUCTURES for deoxyribonucleic acid in the A and B configurations have been described¹. They are compatible with X-ray diffraction data and embody the base-pairing hypothesis suggested by Watson and Crick². The results we describe here strengthen our belief that no alternative stereochemical hypotheses could be used to build models in agreement with the data.

We have observed a reversible transition between the B X-ray diffraction pattern of the lithium salt of deoxyribonucleic acid and a similar but distinct pattern we call C (Fig. 1). The transition from B to C is induced by lowering the relative humidity from 66 to 44 per cent. By slight alteration of the B model we have arrived at a model for C and accounted for the main differences between B and C diffraction patterns. The base pairs were moved by about 2 Å. away from the helix axis, and tilted by about 5°; the pitch of the helix was altered so that there were 9.3 instead of 10 base pairs per turn.

Fig. 1. Diffraction pattern of lithium salt of deoxyribonucleic acid in the B (left) and C (right) configuration