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DETERMINATION OF TRACES OF OXYGEN IN SODIUM  
METAL BY INFRARED SPECTROPHOTOMETRY

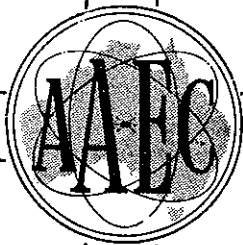
PART 1

by

H. J. de BRUIN

L. E. SMYTHE

Sydney, June 1958.



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DETERMINATION OF TRACES OF OXYGEN IN SODIUM

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PART 1

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*H. J. de Bruin*

*and*

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## EXPERIMENTAL DETAILS

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:—

- (a) amalgamated and the oxide filtered off 1, 2,
- (b) distilled in vacuum 3, 4; or
- (c) reacted with an alkyl halide 5.

A 2g. sample of sodium metal containing 50 p.p.m. oxygen contains  $1.25 \times 10^{-5}$  equivalents of sodium monoxide. In the alkyl halide method, the presence of a 7,000 times excess of sodium bromide over the amount of sodium monoxide, introduces difficulties in the determination of the sodium monoxide by conventional volumetric methods. Elsewhere this difficulty has been discussed and a solution suggested 6. The amalgam and distillation methods also suffer from some disadvantages associated with quantitative separation and subsequent estimation of the oxide.

The use of an infrared pressed disc technique 7 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 halide 5, 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 two hours. 1500 mg. of the sodium bromide – sodium carbonate mixture is then compressed to a clear disc by the usual technique 7. Infrared spectrophotometric analysis, using a Perkin Elmer model 21 I.R. spectrophotometer, shows absorption bands at 4.00, 6.93, 11.38, and 14.19 microns. The absorbance at 11.38 microns due to sodium carbonate, is not subject to any interference and although Lambert's law is not obeyed, from a standard curve (Figure 1) the oxygen concentration in the original metal can be estimated with an accuracy of approximately 10 p.p.m. by weight.

The infrared 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 infrared region. Full details of the above method will be reported elsewhere.

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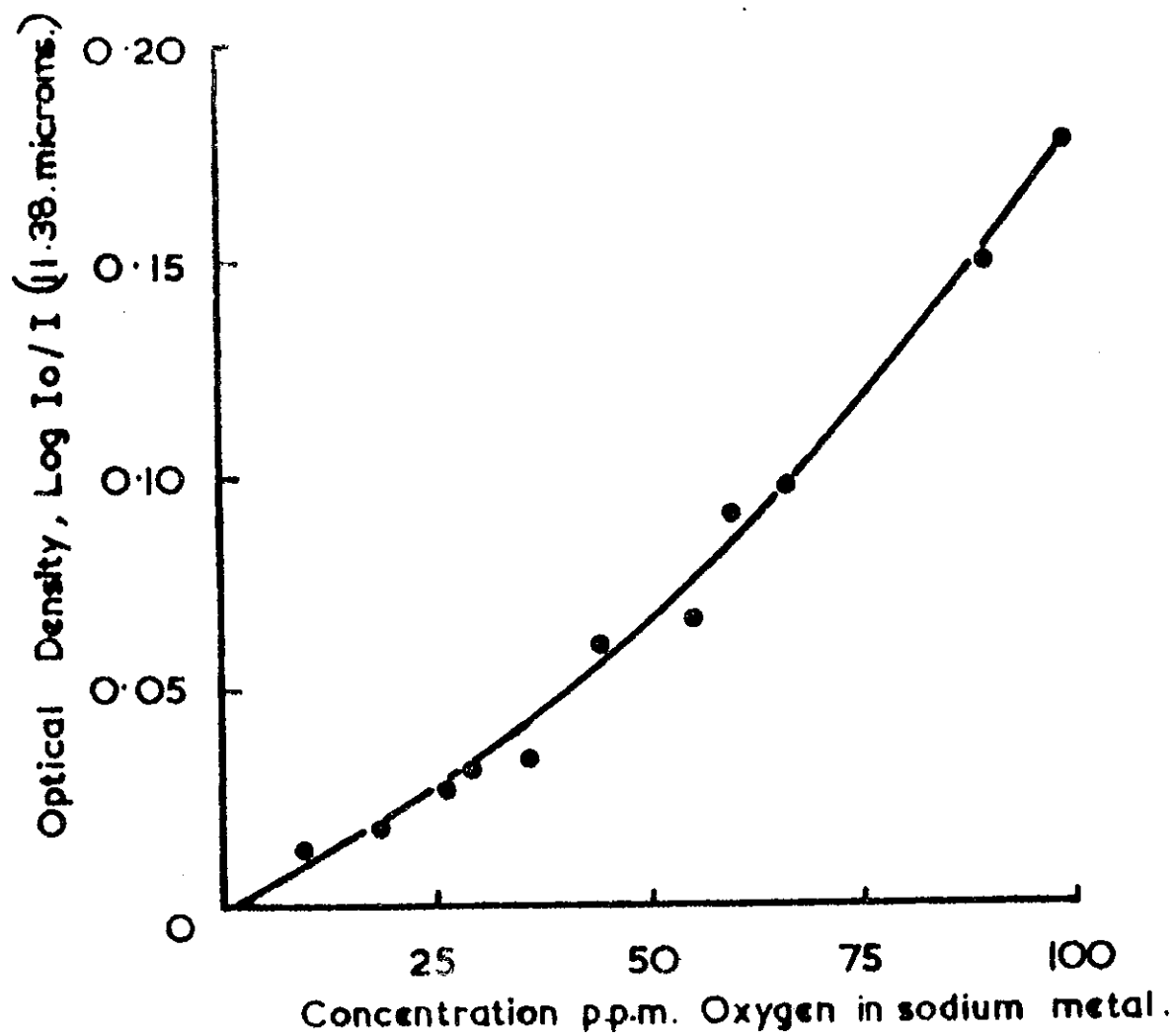


Fig. 1. Oxygen content of sodium metal by Infrared spectrophotometry.