

Fig. 1. ABSORPTION BANDS DUE TO WATER VAPOUR AND OXYGEN IN THE EARTH'S ATMOSPHERE

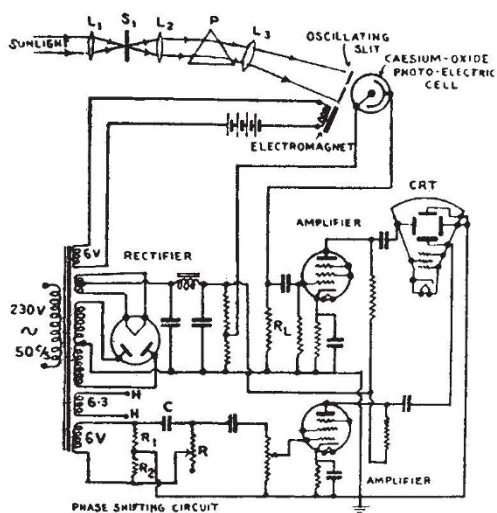


Fig. 2. CIRCUIT DIAGRAM OF CATHODE RAY TUBE SPECTROGRAPH

region between 0.75μ and 1.3μ in the near infra-red region of the solar spectrum was being scanned on a clear afternoon in January 1947. The absorption bands which can be seen in the photograph are, from left to right: oxygen band at 0.76μ , ρ band (H_2O) at 0.93μ and ϕ band (H_2O) at 1.13μ . Fig. 2 is a schematic circuit diagram of the apparatus.

This apparatus was developed for the rapid estimation of the total precipitable water vapour in the atmosphere. Its spectral range is limited to the visible and some portions of the near ultra-violet and near infra-red regions of the solar spectrum; the limiting factors being the transmission characteristics of the spectroscopy optics, which are of glass, and the sensitivity of the photo-cells, which is restricted to the region between 0.34μ and 1.5μ . A detailed account of the apparatus will appear elsewhere.

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¹ Daly, E. F., and Sutherland, G. B. B. M., *Nature*, 157, 547 (1946); King, J., Temple, R. B., Thompson, H. W., *Nature*, 158, 197 (1946). See also *Nature*, 153, 591 (1946).

Interaction of Styrene with Magnesium Perchlorate and Other Salts

IN connexion with an investigation into the mechanism of Friedel-Crafts reactions involving styrene, we required a supply of this olefin of maximum possible dryness¹. Before proceeding to drying by evaporation in an all-glass high-vacuum apparatus of the Stock type, a preliminary treatment with magnesium perchlorate ('Anhydrone') was given. We found, however, that polymerization was induced by this drying agent: in three weeks a highly viscous polymer was obtained.

This effect can be attributed to several mechanisms. It may be due to oxygen derived from the perchlorate, to perchloric acid generated hydrolytically, or to a co-ordinating action of the magnesium atom². Organic complexes of magnesium perchlorate are known, as with dioxan, while styrene forms complexes with, for example, platinum chloride³.

To test these views in a preliminary way styrene (a) dried over sodium sulphate and (b) saturated with water, was treated with magnesium perchlorate and magnesium chloride hexahydrate. The 'dry' perchlorate system showed 13 per cent polymerization in 48 hours: the 'wet' system 6 per cent; both as judged by estimation of residual monomer using a method based on the work of Williams⁴ and Mulliken and Wakeman⁵.

With magnesium chloride the effect is much slower, but the 'dry' olefin is converted to a viscous polymer in two months. 'Wet' styrene is largely unaffected in the same time. This last observation may be connected with the preferential formation of phenyl ethyl chloride, much as the reduced polymerization in the 'wet' perchlorate system may be due to hydroxylation—comparable with the acetylations induced by the Bertram-Walbaum and similar reagents⁶.

We have further found that anhydrous calcium sulphate, which has been recommended for drying styrene, promotes polymerization after prolonged induction periods.

The above preliminary notes do not allow us to distinguish the mechanisms of polymerization involved, which we propose to elucidate by kinetic studies using a wide range of inorganic salts. In this way we hope to obtain information about the mode of action of free radical or electrophilic catalysts, and possibly uncover new examples of the latter type.

In the meantime, we wish to indicate that it is not safe to use magnesium perchlorate for the drying of styrene, and hence probably for drying other olefins. Further, it is probably not safe to leave this olefin in contact for more or less prolonged periods with salts which can exhibit co-ordination, or which can yield strong hydrogen acids by hydrolysis.

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¹ See, for example, Polanyi *et al.*, *J. Chem. Soc.*, 252 (1947).

² Lewis, G. N., *J. Franklin Inst.*, 226, 293.

³ Anderson, J. S., *J. Chem. Soc.*, 971 (1934); 1042 (1936).

⁴ Williams, G., *J. Chem. Soc.*, 1046 (1938).

⁵ Mulliken, S., and Wakeman, H., *Ind. Eng. Chem., Anal. Ed.*, 7, 59.

⁶ See, for example, Lennartz, *Ber.*, 76, 831. Farmer and Pitkethly, *J. Chem. Soc.*, 11 (1938).