

The mono- and di-carboxylic acids which could not be detected by the silver nitrate and amine sprays were located by first spraying the papers with a 50 per cent aqueous alcoholic solution containing 5 per cent of an amine (for example, aniline or *p*-anisidine) and 5 per cent sodium nitrite, and then spraying soon afterwards with an alkaline solution of either a phenol (for example, β -naphthol) or an aromatic amine (β -naphthylamine, 6,8-disulphonic acid). The acids are located by the appearance of colours due to azo-dye formation. When these sprayed chromatograms are dry, they exhibit a strong background fluorescence upon which the dark non-fluorescing azo-dye spots could readily be detected; this is particularly noticeable when an amine is used as the coupling agent.

The acids could also be detected by making use of a browning reaction catalysed by acids. When the chromatograms are sprayed with a 50 per cent aqueous alcoholic solution containing 5 per cent of an aromatic amine (for example, aniline or *p*-anisidine) and 5 per cent of a reducing sugar (D-xylose is preferred) and heated for a short time at 125–130°C., brown spots appear wherever acids are present.

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¹ Lugg, J. W. H., and Overell, B. T., *Aust. J. Sci. Res., Phys. Sci.*, **1**, 98 (1948). Buchanan, J. G., Decker, C. A., and Long, A. G., *J. Chem. Soc.*, 3162 (1950). Howard, G., and Martin, A. J. P., *Biochem. J.*, **48**, 532 (1950). Partridge, S. M., *Biochem. J.*, **42**, 238 (1948). Buch, M. L., Montgomery, R., and Porter, W. L., *Anal. Chem.*, **24**, 489 (1952).

² Hamilton, J. K., Smith, F., and Spriestersbach, D. (unpublished work).

Estimation of Impurities in Liquid Chlorine by Infra-red Absorption Spectrophotometry

THE method of E. R. Wright and R. A. Smith¹ for the determination of moisture in liquid chlorine by infra-red absorption spectrophotometry has been extended to include impurities that give absorption bands as far as 1,180 cm.⁻¹ (8.5 μ). In the range 4,000–1,180 cm.⁻¹, liquid chlorine is, for practical purposes, completely transparent in the infra-red. This enables the use of path-lengths a thousand times greater than normal. Certain impurities can thus be estimated to a few parts per million by this method.

A 5.25-cm. tantalum-clad steel pressure cell (200 lb./sq. in.) was fitted with calcium fluoride windows and 'Teflon' gaskets. This cell was designed and made by the Perkin Elmer Corporation (Norwalk, Connecticut). It was fitted with stainless-steel needle valves and a stainless-steel reservoir which enabled the cell to be filled under pressure directly from an inverted chlorine cylinder, or placed in a Dewar flask and surrounded by solid carbon dioxide for filling at atmospheric pressure. Spectra were recorded on a Perkin Elmer Model 12C single-beam instrument fitted with a calcium fluoride prism to give adequate resolution.

The concentration of impurity is given by the Beer's law relationship which (adopting the usual symbols) is as follows:

$$c = \frac{1}{E \cdot l} \log \left(\frac{I_0}{I} \right) \text{ max.}$$

Using standardized conditions, the concentration in parts per million can be expressed as a constant times the optical density, which is read directly from the absorption curve.

The method has been evaluated by adding weighed amounts of the following compounds to liquid chlorine (wave numbers of absorption bands in cm.⁻¹ given in parentheses): chloroform (1,213), methylene chloride (1,259), hexachlorobenzene (1,348). For these compounds, and at the path-length used, this method is sensitive to approximately 5 p.p.m. with an accuracy at 50 p.p.m. of ± 10 per cent.

The method has been used for the estimation of moisture in liquid chlorine using the absorption band at 1,640 cm.⁻¹ which is free from interference by carbon dioxide.

This work is being submitted for publication more fully elsewhere. So far as we know, this is the first account of infra-red absorption spectrophotometry of liquid chlorine from 2,860 cm.⁻¹ to 1,180 cm.⁻¹ (3.5–8.5 μ).

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¹ Wright, E. R., and Smith, R. A., American Chemical Society Meeting, Buffalo, N.Y. (March 1952).

Production of Individual, Sized, Droplets by High-Voltage 'Firing' from a Micropipette

THERE are several satisfactory methods for the continuous production of uniform droplets. The range below 10 microns may be covered in the La Mer vapour condensation apparatus, 10 μ –1,000 μ , by the spinning disk principle, 100 μ to several millimetres by the vibrating tip, interrupted jet and microburette principles and 800 μ to 6–7 mm. by liquid falling by gravity from the tip of a vertical tube.

In experiments on the evaporation-rates of various airborne droplets, we wished to produce individual droplets and place them with accuracy in the field of the microscope, for example, on a 1- μ diameter wire. Of the above methods, only the last is suitable for producing individual drops; but these are much too large for our purpose. The size of drops falling by gravity from a fine tip is given with reasonable accuracy by equating the upward force of surface tension acting round the circumference of the tip to the gravitational pull at the moment of breakaway: $\pi d T = \pi D^3 \rho g / 6$, where d and D are the diameters of the tip and droplet respectively. For a 20- μ tip, the diameter of the drop which falls is still as large as 0.95 mm. The cubic relationship between d and D prohibits any further worth-while reduction in D as d is reduced to the smallest practical size.

We have found that droplets of any desired size down to about 10 μ may be 'fired' off from the tip of a micropipette by applying a momentary high voltage to the liquid. Suitable micropipettes in thin-walled glass tubing may be drawn by hand; but much greater precision, particularly in the finer sizes, is given by a 'microforge' such as Powell's microscope attachment¹. The micropipette needle is fitted to a