

(2) Calibrations due to differences in thermal conductivities of the separated components are unnecessary; however, the carbon number effect must be considered.

(3) The method is applicable to other detectors where hydrogen is used as a carrier gas, such as the hydrogen flame detector³.

(4) It offers a convenient way of analysing aqueous solutions of aldehydes, alcohols and ketones, since these are easily converted to methane and water over the catalyst. The water may be removed by a suitable drying column containing, say, calcium sulphate.

Compounds susceptible of complete 'hydrocracking' to methane have been analysed using hydrogen as the carrier gas. The methane conversion procedure has been used for mixtures containing (a) C_1 - C_{12} paraffins and olefins, (b) C_5 - C_8 naphthenes, and (c) C_2 - C_6 aldehydes, alcohols and ketones. The catalyst remained active for more than 100 samples.

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¹ Martin, A. E., and Smart, J., *Nature*, **175**, 422 (1955).

² Green, G. E., *Nature*, **180**, 295 (1957).

³ Scott, R. P. W., "Vapour Phase Chromatography", 131 (Academic Press, New York, 1957).

Contamination of Surfaces by Conductivity Water from Ion-exchange Resins

IN several recent papers research workers in surface chemistry have reported using ion-exchange resins as a convenient means of removing electrolytes—in place of dialysis in the treatment of inorganic sols, for example, and to obtain 'conductivity water' for studies of dilute detergent solutions or monolayers on a Langmuir trough. For most purposes where 'pure' water is needed, even in colloid chemistry, the water obtained by passing ordinary distilled water through a 'mixed-bed' ion-exchange resin column is perfectly satisfactory, but for any critical work in surface chemistry it is not. The dangers are shown by the following experience.

In a study of the surface charge on finely divided particles by the micro-electrophoresis method, water from a mixed-bed resin column was used throughout for washing the cell and preparing the solutions. It had a specific conductivity of 0.3 micromho cm^{-1} and would normally be considered of good quality compared with ordinary distilled water, which gives about 3.5 and contains traces of copper, etc. A series of determinations with different dilute salt solutions gave what seemed to be satisfactory results until similar measurements were made with dilute hydrochloric acid. As the concentration of hydrochloric acid was increased from 10^{-6} to 10^{-4} N, the electro-osmotic flow along the glass walls of the cell fell to zero and then reversed in direction; that is to say, the glass became positively charged. (The suspended particles on the contrary remained negatively charged.) The anomalous charge on the glass was eventually ascribed to contamination by traces of a weakly basic substance from the resin column—

almost certainly soluble polymer fragments released from the nitrogenous anion-exchange resin.

The presence of traces of organic impurities in water from resin columns is very difficult to check by direct analysis, but it is well known that water which has stood for some hours in a mixed-bed shows a relatively high conductivity. (Several 'bed-volumes' must first be run off to waste.) Clearly, this cannot be due to micromolecular electrolytes since these are effectively removed by the resins. It must be caused by colloidal polyelectrolytes, released by the resins and unable to penetrate into the resin particles of opposite charge because of size.

The purity of water from a mixed-bed column is therefore dependent on the time of contact with the resin. Rapid flow through a short column may allow electrolytes present in the feed water to 'break through', but slow flow through a long column may introduce organic polyelectrolytes.

Conductivities as low as 0.065 micromho cm^{-1} at 25° C. have been reported¹ for water from a mixed resin bed in favourable circumstances, and this is remarkably close to the theoretical value of 0.055 at 25° C. for perfectly pure water; but it is common experience to find values in the range 0.1–0.5 micromho cm^{-1} . This conductivity value is a dangerous index. It can easily be dismissed as due to an 'insignificant trace' of electrolyte (for example, 10^{-6} N potassium chloride), but in fact may indicate a considerably higher (though quite unknown) amount of surface-active polyelectrolytes which may accumulate by adsorption on suitable surfaces.

With improvements in the stability of ion-exchange resins and careful attention to flow conditions, it may become possible to reduce the danger to almost negligible levels. But at the present time it is clearly advisable to avoid the use of resins entirely in all critical work in surface chemistry where small surface areas are concerned. Repetition of the experiments mentioned above, but using triple-distilled water finally condensed in silica, gave significantly different results for the suspended particles as well as for the glass surfaces.

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¹ Akeroyd, E. I., and Kressman, R., *Chem. and Indust.*, 189 (1950).

Void Nucleation Sites in Creep of Metals

FOR metals undergoing slow deformation at elevated temperatures, it has been shown that nucleation of voids by condensation of lattice vacancies requires either a very high concentration of excess vacancies (approximately 100 times the equilibrium value¹), or very high concentration of stress². It appears that such concentrations of vacancies are never approached even at high rates of deformation³, so that voids must form at points where sufficiently large stresses develop. It has been suggested that ledges on a sliding grain boundary may provide these sites for nucleation.

The nature of ledges in the boundary is uncertain. Gifkins⁴ suggested that they result from slip in one grain on planes almost perpendicular to the boundary being accommodated by slip in the adjoining grain. He believed that this mechanism required a pile-up