

differing from that noted here because of a different method of release of the spheres.

When seven or more spheres were present in the cluster a different régime was entered. The spheres, if released as a compact cluster, no longer assumed a regular arrangement; though if seven or eight spheres were arranged at the vertices of a regular heptagon or octagon respectively and allowed to fall, the regularity of the orientations was soon destroyed. With compact clusters a vortex, the axis of which was vortical, was set up, the circulation being inwards at the top of the cluster. Simultaneously the individual spheres each acquired a rotational motion about a horizontal axis perpendicular to the line joining the centre of the sphere to the axis of the vortex. The subsequent behaviour of these clusters was complex and varied with both the number and size of the spheres.

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Rejuvenation of Gas Liquid Chromatography Columns *in situ*

Atkinson and Tuey¹ mention the *in situ* reconditioning of chromatographic columns. An *in situ* treatment of columns in use in this laboratory has been regularly utilized for some years. Our experience of this technique may be of interest to others.

A column made up of 20 per cent phenanthrene² on 100–120 mesh brickdust in a copper tube (8 ft. long, 0.25 in. outer diam.), at a working temperature of 110° C, is in regular use for the analysis of works samples of aromatic hydrocarbons. A hydrogen flame thermocouple detector is used with a carrier gas of 75 per cent hydrogen and 25 per cent nitrogen. Samples are injected with a hypodermic syringe through a rubber serum cap. The range of samples analysed covers crudes and fractions associated with the refining of gas works crude benzole. The column is in use for an average period of 24 h a week.

Although the separation of *meta*- and *para*-xylenes is not complete, the degree of separation is used as the criterion for the resolving power of the column. It is found that, after two months' use, the resolution between the *meta*- and *para*-xylenes decreases to such an extent that it is desirable to remake the column. However, it has been found that the column can be regenerated *in situ* in the following manner.

A small amount of stationary phase (approximately 10 per cent of original weight) is introduced at the inlet of the column. In this instance, as phenanthrene is a solid, the best method of introduction is found to be by stopping the flow of carrier gas, removing the serum cap, adding the stationary phase, replacing the serum cap, and restarting the flow of carrier gas. This procedure is carried out at the normal working temperature of the column. The phenanthrene is then flushed through with enough benzene to obtain the required resolution. The benzene is introduced by use of the syringe. It is advisable as the benzene is passing through the column to remove the thermocouple to avoid sooting up and also to allow enough oxygen to the column exit to permit combustion of the benzene without altering the existing flow setting on the air line.

Alternatively, the phenanthrene may be introduced directly as a 20 per cent solution in benzene, in 1-ml. portions followed by two or three 1-ml. portions of pure

benzene. This is repeated until the required original retention time is obtained.

The column as described here has now been in use for 30 months and has been rejuvenated many times. After the first rejuvenation the original resolving power was not quite attained, but at successive rejuvenations there was no further loss of resolution and the column for practical purposes is still satisfactory.

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Hydrogen Bonding in Gaseous Mixtures: Amine-Alcohol Systems

Millen and Zabiicky¹ have recently reported the infra-red detection of hydrogen bonding in gaseous mixtures of methanol and methylamines. The strongest complex appears to be formed with trimethylamine, and the OH stretching band, displaced to 3,350 cm⁻¹, shows pronounced shoulders at 3,495 cm⁻¹ and 3,200 cm⁻¹. These have been interpreted by Millen and Zabiicky as the sum and difference combinations of 3,350 cm⁻¹ with the stretching of the H-bond itself, implying a frequency for the latter of about 140 cm⁻¹. An alternative explanation of the shoulder on the high-frequency side is that it is due to a weak band associated with the trimethylamine part of the complex, intensified by Fermi resonance with the 3,350 cm⁻¹ band. At Dr. Millen's suggestion we

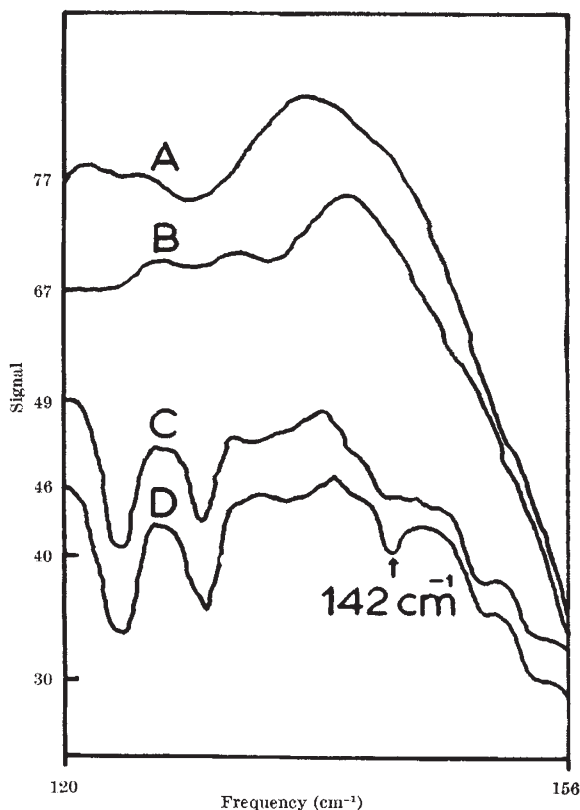


Fig. 1. A, vacuum background; B, 30 cm Me₃N; C, 9 cm MeOH; D, 30 cm Me₃N + 9 cm MeOH. Identical operating conditions were used throughout. The signal scale is the same for all four spectra and is shown for curve D. The relative positions of the curves are indicated by the signal to the left of each curve