

is hoped to publish a more detailed discussion of these and further results in due course.

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- <sup>1</sup> See, for example, Burnett, G. M., *Quart. Rev. Chem.*, **4**, 292 (1950).  
<sup>2</sup> Bawn, C. E. H., "The Chemistry of High Polymers" (London: Butterworths Scientific Publications, Ltd., 1948).  
<sup>3</sup> Williams, G., *J. Chem. Soc.*, 775 (1940).  
<sup>4</sup> Price, C. C., *Ann. N.Y. Acad. Sci.*, **44**, 351 (1943).  
<sup>5</sup> Price, C. C., "Mechanisms of Reactions at Carbon-Carbon Double Bonds", 112-115 (New York: Interscience Publishers, Inc., 1946).  
<sup>6</sup> Kemp and Peters, *Indust. Eng. Chem.*, **34**, 1097 (1942).

### Quantitative Separation of Small Amounts of Zinc from other Metals by Chromatography on Cellulose Columns

THE great usefulness and versatility of chromatography for qualitative separation of metals have been shown recently in a number of reports. So far, much less work appears to have been published on the quantitative possibilities of the method. Lacourt *et al.*<sup>1</sup> have described the determination of aluminium, iron and titanium separated on paper strips using colorimetric procedures for the final estimation. Burstall and his colleagues<sup>2</sup> have employed cellulose columns for the analysis of alloy steels; in the same paper the separation of gold from the metals of the platinum group is mentioned, and the possibility of other quantitative separations is foreshadowed. Using thick strips of filter paper, Anderson and Lederer<sup>3</sup> have recently separated thallium quantitatively from other metals.

As a step in an investigation on the effect of trace impurities on the performance characteristics of tin/lead solders, we have examined the quantitative separation of zinc from other metals likely to be encountered. Exploratory experiments had shown that it was possible to remove the bulk of the two major constituents by preliminary chemical separation, leaving these two metals with the impurities in quantities of about equal order of magnitude. Using paper strips according to the method described by Burstall *et al.*<sup>4</sup>, with methyl propyl ketone as eluent, it was possible to obtain complete separation of the following metals applied to a paper strip in approximately equal quantities ( $10^{-5}$  gm.): tin, aluminium, nickel, lead, manganese, bismuth, cobalt, zinc, copper and iron.

With solutions containing the same metals and using butanol hydrochloric acid as eluent, zinc moved in front of the other metals, followed by iron. The same sequence of separation was found when the analyses were carried out on cellulose columns. These conditions, therefore, are favourable for the quantitative separation of this metal. Solutions containing approximately 500  $\mu$ gm. of zinc and similar quantities of the other nine metals were applied to the top of the column and were eluted with a mixture of water-saturated butanol and concentrated hydrochloric acid in the volume ratio of 92 : 8. The visible iron band acted as a convenient marker, and the eluate was collected until this band had reached the bottom of the column. The zinc in the resulting solution was determined polarographically.

The following results were obtained :

Zn added (gm. $\times 10^4$ )	Zn recovered (gm $\times 10^4$ )
5.00	5.14
5.00	5.04
5.00	4.72
6.00	4.82
	6.07
	5.90

When zinc was added to 2 gm. of solder, similar recoveries were achieved.

Zn added (gm. $\times 10^4$ )	Zn recovered (gm. $\times 10^4$ )
1.00	1.05
2.00	1.96

We now hope to determine further the sensitivity and accuracy of this method for the estimation of zinc and to extend it to other trace impurities in solders.

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<sup>1</sup> Lacourt, A., *et al.*, *Nature*, **163**, 999 (1949).

<sup>2</sup> Burstall, F. H., *et al.*, *Trans. Farad. Soc., Discussions*, **7**, 179 (1949).

<sup>3</sup> Anderson, T. R. A., and Lederer, M., *Anal. Chim. Acta*, **4**, 513 (1950).

<sup>4</sup> Burstall, F. H., *et al.*, *J. Chem. Soc.*, 516 (1950).

### Bound Water in the System Cellulose-Aqueous Salt Solution

It has been concluded by Hermans<sup>1</sup> and others, from an examination of the apparent specific volume of cellulose in aqueous solutions of various solutes, that a certain amount of water is 'bound' to the cellulose and is not available as solvent.

It is true that, in general, the apparent specific volume of cellulose in water is increased by the addition of a solute. Although alkali halides have this effect, we have ascertained, by separation and complete analysis of the two phases, that the preferential absorption of the solute is not in favour of the aqueous phase. As the solute has a higher density than the solvent, the rise in apparent specific volume of the cellulose must be ascribed to some secondary effect, perhaps some action of the solute in reducing the adsorption compression or interpenetration between water and cellulose.

The use of specific-volume measurements for the determination of 'bound water' is therefore invalid.

Determinations based upon the take-up of water and of solute from solutions of known concentration are not subject to this complication. Sucrose<sup>2</sup>, sodium thiosulphate<sup>3</sup> and other substances have been used, leading to positive and fairly definite values for the 'bound' water, or, in other words, preferential absorption of the solvent by cellulose. Sodium sulphate also appears to behave in this way; but the use of alkali halides, which are preferentially absorbed from water, would clearly lead to negative values for the 'bound water'.

Measurements of this supposed quantity made with a particular solute are, therefore, devoid of general significance.

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<sup>1</sup> Hermans, "Contribution to the Physics of Cellulose Fibres" (Elsevier, 1946).

<sup>2</sup> Barkas, *Nature*, **130**, 699 (1932).

<sup>3</sup> Champetier, *C.R. Acad. Sci., Paris*, **195**, 280 (1932).

<sup>4</sup> Tankard, *J. Text. Inst.*, **23**, T263 (1937).