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Note

An improved procedure for preparation of gas chromatographic salt-modified adsorbents

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Active solid adsorbents such as silica gel and alumina have been routinely used in liquid-solid chromatography. The utilization of these adsorbents in gas-solid chromatography (GSC) has been confined to the analysis of light hydrocarbons and permanent gases; however, if the adsorbent is coated with an inorganic salt, the active surface area of the adsorbent is reduced and becomes more homogeneous. Therefore, more symmetrical elution peaks with lower retention times can be observed for weakly interacting adsorbates. Sawyer and co-workers^{1,2} have greatly extended the usefulness of GSC by achieving selective analytical separations of complex mixtures with salt-modified aluminas, silica gels and porous silica beads. In particular, these workers have shown that the sorbate-salt-modified adsorbent interaction is a composite of non-specific and specific contributions with the latter effect dependent upon the π -electronic nature and geometrical configuration of the sorbate. Both the salt modifier and the adsorbent affect sorbate-modified adsorbent interaction.

In a recent investigation³ on transition metal chlorides (CrCl_3 , CoCl_2 , MnCl_2 , ZnCl_2) as modifiers of silica gel, we have found the conventional technique for the preparation of salt-modified adsorbents by rotary evaporation to be unacceptable. As the pre-weighed amount of salt modifier is being deposited on the adsorbent by rotary evaporation of the water, coated material collects on the wall of the 250-ml Pyrex roundbottomed flask. Analysis by atomic-absorption spectroscopy showed that the transition metal ion concentration in the electrostatically adhered material was consistently greater than that in the bulk material by an order of magnitude; thus, the desired percentage of a uniform salt coating on silica gel was not achieved. We have found that the following procedure yields the desired coating percentage and circumvents this difficulty. The desired amount of salt is dissolved in water and mixed with the appropriate amount of silica gel to yield a thick slurry in a 250-ml polyethylene beaker. The beaker is placed in a boiling water-bath positioned above a combination magnetic stirrer-hot plate unit. Continuous heating and stirring until the modified adsorbent is dry produces a uniform distribution of the salt modifier on silica gel. This technique is rapid, accurate, convenient and superior to the rotary evaporation procedure because meaningful thermodynamic and retention data associated with sorbate-adsorbent interactions can be determined.

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