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Note

Redox flat-bed techniques

A study of papers loaded with amalgams

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Filter-paper has been known as a support for sensitive precipitation and colour reactions for a long time and has been used extensively in the field of spot tests.

The obvious advantages of filter-paper or flat beds for partition chromatography and electrophoresis have been pointed out repeatedly. Other chromatographic processes, such as adsorption with loaded papers and ion exchange, have also been adapted to flat-bed techniques. Oxidation–reduction reactions in a flat-bed arrangement have so far been limited to a few experiments with papers loaded with redox resins[1]. On the other hand, redox column techniques have been used extensively in analytical chemistry, for example the Jones reductor. We therefore felt that it would be interesting to study the possibilities of flat-bed redox reactions using papers loaded with metal amalgams. Such papers are readily prepared, if the amalgam has the consistency of a soft solid, by rubbing the amalgam on pieces of filter-paper.

EXPERIMENTAL

Amalgams of zinc, lead and cadmium were prepared by heating the required amounts of the metal and mercury in a crucible with occasional stirring. Silver amalgam was best prepared by grinding silver powder with mercury in a mortar. The amount of metal giving an amalgam that can be rubbed on paper was established by trial and error, and the proportions used are given below in each instance.

Whatman No. 1 strips (usually 4×20 cm) were washed with 1 *N* hydrochloric acid and water and dried, and then loaded by rubbing the amalgam of the right consistency over both sides (using rubber gloves). It takes little experience to prepare very uniformly loaded strips. The first 2 cm were usually left free from amalgam (where the paper dips into the solvent) and zones of various lengths were made along the strips.

The metal ions to be studied were solutions of *ca.* 0.01 *M* in 0.5 *N* hydrochloric acid, and 1–2 μl were applied just below the amalgam zone and developed at room temperature by the ascending technique in glass jars 25 cm high and 11 cm wide. The solvent was usually allowed to move 16–17 cm and the metal ions, which had moved out of the amalgam into the upper, uncovered part of the paper strip, were detected with the usual spot tests such as rubeanic acid for copper(II), ammoniacal silver nitrate solution for manganese, α-nitroso-β-naphthol for cobalt, potassium hexa-

cyanoferrate(II) and hexacyanoferrate(III) for iron(III) and iron(II), respectively, tin(II) chloride for palladium, and potassium iodide for bismuth(III).

RESULTS

Zinc amalgam

Amalgams containing 18.2% and 28% of zinc were prepared and zones from 0.5 to 8 cm in length loaded on to paper strips.

The following metals did not leave the amalgam zone and were presumably reduced: bismuth(III), copper(II), lead(II), cadmium(II) and antimony(III). Nickel(II) and cobalt(II) were still detected after passing the amalgam but the spots observed were much weaker, indicating partial reduction in the amalgam.

Iron(III) was completely reduced if the amalgam zone was 5 cm or longer, while 1 cm of amalgam allowed both iron(II) and iron(III) to leave the amalgam zone. In 0.5 cm of amalgam, only iron(III) was washed out, *i.e.*, the reduction had not even started.

Manganese(II) was not reduced at all and could therefore be separated readily from the completely reduced metals above.

Lead amalgam

An amalgam containing 43% of lead proved suitable, and the papers were loaded with an amalgam zone 8 cm long. Copper(II) and bismuth(III) were completely retained on such amalgam zones, while nickel(II), cobalt(II), cadmium(II) and manganese(II) travelled out of the zones unreduced.

Cadmium amalgam

The amalgam contained 14.8% of cadmium. Papers with zones of 7 and 8 cm of this amalgam were prepared and were found to reduce bismuth(III), copper(II) and lead(II) but not nickel(II), cobalt(II) and manganese(II). For the complete conversion of iron(III) into iron(II), the amalgam zone had to be 20 cm long, otherwise iron(III) could still be detected.

Silver amalgam

The amalgam contained 16% or 17.7% of silver. Zones 8 cm and 20 cm long did not reduce bismuth(III), but both palladium(II) and gold(III) were completely reduced. Copper(II) was only partially reduced while platinum(IV) was not completely reduced in 8-cm zones but was reduced in a 20-cm zone. Very dilute solutions of platinum(IV) could be reduced in shorter zones. Platinum(II) was also tried and was reduced analogously to platinum(IV).

DISCUSSION

The reduction observed on amalgam papers is that expected from the electromotive series, except for cobalt and nickel, which are reduced rather slowly, a phenomenon that is also observed in polarography as well as in static experiments[2].

Iron(III) can be reduced to iron metal on amalgam paper, but this seems not to be the case in a Jones reductor.

The use of amalgam papers may be of interest when one requires to remove a large number of metals from one other metal, and from these experiments it seems to be relatively simple to plan suitable conditions for most individual cases.

REFERENCES

1 B. Sansoni, *Naturwissenschaften*, 41 (1954) 212.
2 A. S. Russel and J. C. Carver, *Nature (London)*, 142 (1938) 210.