

Short Communications

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SEPARATION OF MAGNESIUM, CALCIUM, STRONTIUM AND BARIUM BY CIRCULAR THIN LAYER CHROMATOGRAPHY

MANZOOR AHMAD CHAUDHRI, N. A. CHUGHTAI*
and A. U. AFZAL

PCSIR Laboratories, Lahore

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There has always been need for separation and identification of alkaline earth metals from a mixture of metal ions, hence attempts were made for the identification of Be, Mg and Ba in alkaline earths on TLC¹.

A method has been worked out for the separation and identification of alkaline earth metals such as Mg, Ca, Sr and Ba using circular TLC technique.^{2,3}

Experimental

Reagents

All reagents were of analytical grade or of comparable purity. Microgranular cellulose power (CC 41 Whatman) was used for the preparation of TLC plates. Rhodizonic acid, sodium salt, 0.2% aqueous and 8-hydroxyquinoline, 0.5% in 60% ethanol were applied as spray reagents. 6M HCl-methanol (1:5) and 8M HCl were used as solvents.

The solutions of Mg, Ca, Sr and Ba were prepared by dissolving their nitrates in water. The solution of each ore was prepared separately by fusion method.⁴

Apparatus

Circular thin layer chromatograph was used for the development of chromatoplates. Thin layers of uniform thickness (0.25 mm) were prepared by making slurry of cellulose powder using the Camag applicator. Shandon micropipette was used for spotting the sample on the chromatoplate.

Procedure

The chromatoplates were developed by circular TLC technique described earlier.^{2,3} After drying, the chromatoplate is sprayed with 8-hydroxyquinoline^{5,6} or sodium rhodizonate.^{7,8}

Results and Discussion

Mg and Ca are visible under UV light when sprayed with 8-hydroxyquinoline. For Sr and Ba, the chromatoplate is sprayed with sodium

rhodizonate followed by exposure to NH₃ vapours. The results are given in Table 1.

The best results were obtained with 6M HCl-methanol (1:5). A fine separation of Mg and Ca has also been made on cellulose powder impregnated with tributyl phosphate in CCl₄ using 8M HCl as eluent (Cr Table 1).

Mg and Ca rings are only visible under UV light when exposed to NH₃-vapours. By this procedure presence of Mg, Ca, Sr and Ba has been confirmed in some ore samples. R_f values may vary slightly,⁹ but the sequence of separation is the same.

A solvent extraction scheme³ is applied when other interfering cations are present. Lithium, though extracted with alkaline earth metals, gives dim coloured ring with 8-hydroxyquinoline, but has different R_f value. Similarly Th and Ce form dim coloured rings with sodium rhodizonate having different R_f values; hence do not interfere with the identification of alkaline earth metals.

TABLE 1. SEPARATION OF MAGNESIUM, CALCIUM, STRONTIUM AND BARIUM BY CIRCULAR THIN LAYER CHROMATOGRAPHY.

Cation	Colour	R _f value	Sensitivity
Mg	Bluish green (bright)	0.86	5 µg
	Yellowish green	0.93 *	
Ca	Bluish green (dim)	0.76	15 µg
	Yellowish green	0.83 *	
Sr	Reddish brown	0.65	15 µg
Ba	Reddish	0.43	2.5 µg

*Chromatoplates coated with cellulose powder impregnated with TBP in CCl₄ were used.

References

1. E. Gagliardi and W. Likussar, *Mikrochim. Acta* (Wien), 1053 (1965).
2. M. H. Hashmi, M. A. Shahid and A. A. Ayaz, *Talanta*, 12, 713 (1965).
3. M. H. Hashmi, M. A. Shahid, A. A. Ayaz, F. R. Chughtai, N. Hassan and A. S. Adil, *Anal. Chem.*, 38, 1554 (1965).
4. M. H. Hashmi, G. A. Minhas, M. I. Durrani and R. Rehman, *Mikrochim. Acta* (Wien), 286 (1969).
5. F. H. Pollard and J. P. W. Mcomic, *Chromatographic Methods of Inorganic Analysis*, (London, 1953).
6. L. S. Bark, G. Duncan and R. J. T. Graham, *Analyst* (London), 92, 347 (1967).
7. N. S. Poonia and N. S. Sahni, *Indian J. Chem.*, 5, 203 (1967).
8. Warner Bock-Werthmann, *Z. anal. Chem.*, 198, 403 (1963).
9. M. H. Hashmi, M. A. Chaudhri, N. A. Chughtai and R. Rehman, *Mikrochim. Acta* (Wein), 200 (1970).

*Present Address : PINSTECH, Nilore, Rawalpindi.