

Solvent extraction separation of barium(II) from associated elements using 15-crown-5 from picrate medium

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Received 24 September 1999; revised 21 March 2000

A simple extraction method has been developed for separation of barium(II) from picric acid medium using 15-crown-5. Barium(II) has been quantitatively extracted from 0.001-0.05 *M* picric acid with 0.002-0.01 *M* 15-crown-5 in nitrobenzene as a diluent. From the organic phase barium(II) has been quantitatively stripped with 0.5-10 *M* HNO₃ and HCl and 10 *M* HClO₄. Separation of barium has been carried out from a number of elements in binary and multicomponent mixtures. Most of the elements from *s*-block, *p*-block and *d*-block show high tolerance limit. The method has been extended for the determination of barium (II) in various rock samples.

Barium has attracted considerable attention because it is one of the major constituent of fission products. It is also found in a number of rocks. Therefore the determination of barium(II) in environmental samples such as rocks, minerals, marine organisms and bone is important. However, the environmental samples often contain large amount of calcium(II) and other alkaline earth metals which may cause spectral interference. Therefore, for the precise determination, it is essential to separate barium(II) from other elements. The classical methods involve selective precipitation either as insoluble sulfate or chromate. These methods are applicable only when the concentration of barium(II) is in milligram concentrations. In solvent extraction methods benzoylacetone and dibenzoylmethane¹ have been used as extractants but the extraction of barium(II) is very poor. The extraction of barium(II) has been carried out in alkaline media using thenoyltrifluoroacetone^{2,3}, bis-2-ethylhexyl phosphoric acid^{4,5} hexafluoroacetylacetone⁶, however no attempts have been made to separate barium(II) from the associated elements. Crown ethers like 18-crown-6(ref. 7,8), dibenzo-18-crown-6(ref. 9), dibenzo-24-crown-8(ref. 10) have been used for the study of extraction constant and stability con-

stant. The separation of barium(II) from other alkaline earths was achieved with dibenzo-18-crown-6 and 18-crown-6 (ref. 11). The stability of strontium(II) and barium(II) has been reported with 18-crown-6 from picrate solution¹². The substoichiometric ion-pair extraction of barium with cryptand-2.2.2 and 18-crown-6 has also been carried out using picrate as a counter anion¹³. Spectrophotometric investigation of complex formation of barium(II) and calcium(II) with aza-15-crown-5 has also been carried out^{14,15}.

We report in this note the solvent extraction separation studies of barium(II) and its separation from the associated elements using 15-crown-5. The method developed is simple and allows extraction at trace levels. Barium(II) can be separated very effectively from associated elements in multicomponent mixtures. The developed method has also been extended for the determination of barium(II) in a number of certified rock samples.

Experimental

A Zeiss spectrophotometer, a digital pH meter, with glass and calomel electrodes, a digital flame photometer, a wrist action flask shaker and 125 ml separating funnels were used.

Stock solution of barium(II)(1.00 mg/ml) was prepared by dissolving 1.90 g of barium nitrate(AnalaR grade, BDH) in 1000 ml of distilled, deionised water and standardized gravimetrically¹⁶. A solution containing 100 µg/ml of barium(II) was prepared by appropriate dilution of the standard stock solution.

Solutions of crown ethers were prepared from 15-crown-5, benzo-15-crown-5, 18-crown-6, benzo-18-crown-6, dibenzo-18-crown-6, dicyclohexano-18-crown-6, dibenzo-24-crown-8 and dicyclohexano-24-crown-8 (Aldrich,USA.) without further purification.

Picric acid solution (0.05 *M*) was prepared by dissolving 2.846 g of picric acid in 250 ml of distilled, deionised water.