International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 07 Issue: 08 | Aug 2020 www.irjet.net p-ISSN: 2395-0072

# Separation of Molybdenum (VI) with Crown Ethers using Solvent Extraction and Adsorption Chromatographic Techniques

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**Abstract -** *DB-18-C6 was used for the extractive separation analysis of molybdenum (VI) from a range of other elements. Molybdenum (VI) was quantitatively extracted from 8M hydrochloric acid with 0.01M DB-18-C6 in nitrobenzene. It was stripped from the organic phase with 2M nitric acid and determined spectrophotometrically with Tiron at 390 nm. Molybdenum was separated from a large number of elements in binary mixtures, the tolerance limit for most elements being very high. Selective extraction of molybdenum permits its separation from barium, thorium, cesium, rubidium, strontium, lanthanum, chromium (III) and cerium (III). The method was extended for the analysis of molybdenum in a soil sample.*

*A very simple column chromatographic separation method has been developed for molybdenum (VI) using poly-(dibenzo-18-crown-6). The separations are carried out from hydrochloric acid medium. The adsorption of molybdenum (VI) on a poly-(DB-18-C-6) was quantitative from 2.5 to 10.0*M *HCl. Amongst the various eluents tested, 0.5*M *ammonium hydroxide was found to be an efficient eluent. Molybdenum (VI) was separated from a large number of elements in binary form, as well as from multicomponent mixtures. The method was applied for the analysis of molybdenum from various alloy samples. The method is very simple, rapid, selective and reproducible. The reproducibility of the procedure is ±2%.*

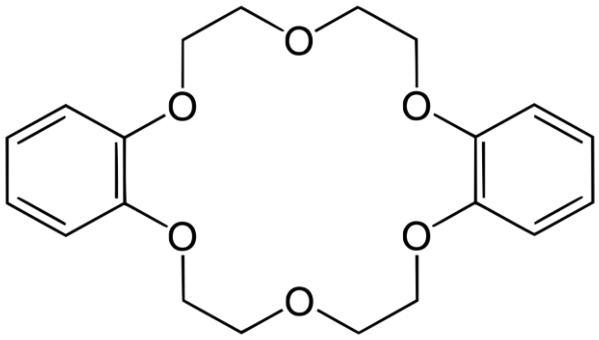
***Key Words: Molybdenum, crown ethers, solvent extraction, adsorption chromatography***

## 1. INTRODUCTION

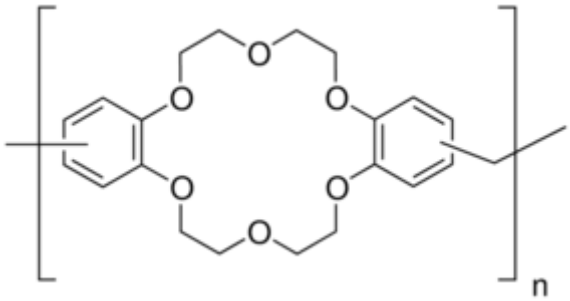
Molybdenum has a large number of applications in various fields. It is used in radios, wireless sets, X-ray tubes and in the production of special steels. Molybdenum occurs in water, soils, plants and animals at trace level. Molybdenum is one of the seven micronutrients required for the growth and development of plants.

Crown ethers have been extensively used in extraction analysis studies of various metal ions and those methods have been reviewed. But the use of crown ethers for the extractive separation analysis of Mo (VI) is limited. DB-18-crown-6 was used for the solvent extraction of Mo (VI) showed very poor extraction. This paper presents a systematic investigation of column chromatography separation studies of Mo (VI) from associated elements in hydrochloric acid medium using poly-dibenzo-18-crown-6. The method has been extended to the separation of Mo (VI) from large number of other elements and also from a number of various alloy samples.

Solvent extraction separation of Mo (VI) was achieved by using DB-18-crown-6. This paper presents a systematic investigation of the solvent extraction separation of Mo (VI) from other elements in hydrochloric acid medium DB-18-crown-6 with nitrobenzene as diluent. The method has been extended to separation of Mo (VI) from a large number of other elements in binary mixtures as well as from multi component mixtures.



Dibenzo-18-crown-6



Poly dibenzo-18-crown-6

**General Procedure for solvent extraction separation of Mo (VI):**

To an aliquot of solution containing Mo (VI) (25 microgram) hydrochloric acid was added to a concentration of 8M in 10

ml total volume. The solution was then transferred a separating funnel and was equilibrated with 10 ml 0.01 M crown ether in nitrobenzene for 10 minutes on a wrist action flask shaker. The 2 phases were allowed to settle and separate. From the organic phase Mo (VI) was stripped with 10 ml 2M nitric acid. It was then determined spectrophotometrically with Tiron at 390 nm. The concentration of Mo (VI) was calculated from the calibration curve.

**Results and discussions:**

In order to ascertain the optimum concentration of hydrochloric acid for the quantitative extraction of molybdenum, the extraction studies were performed with a range of crown ethers of 0.01M concentration in nitrobenzene. The hydrochloric acid concentration was varied from 1 to 10M. The results show that DB-18-C-6 was the best extractant for the quantitative extraction of molybdenum from 7 to 10M hydrochloric acid. To study the effect of crown ether concentration, the concentration of DB-18-C-6 was varied from 0.0001 to 0.1M. It was found that the extraction of Molybdenum was quantitative at 0.008 to 0.1M. In further studies 10 ml 0.01M DB-18-C-6 in nitrobenzene was therefore used.

To study the effect of various diluents on the extraction of molybdenum, extractions were performed using various diluents. The results showed that the extraction of molybdenum is quantitative only with nitrobenzene as diluent. To determine the optimum extraction period, the equilibrations carried out over various time periods (1 to 60 minutes). It was found that quantitative extraction of molybdenum by 1 minute. Various amounts of molybdenum (5 to 100 microgram) were extracted from 8M hydrochloric acid with 10 ml 0.01M DB-18-C-6 in nitrobenzene. It was found that extraction of molybdenum was quantitative up to 50 microgram per 10ml solution. The separation of molybdenum was carried out from binary mixtures and multi component mixtures.

**Analysis of molybdenum in a soil sample:**

About 5.0 gram of a finely powdered soil sample was digested and dissolved as described elsewhere. From an aliquot of solution, Molybdenum was extracted from 8M hydrochloric acid with 0.01M Db-18-C-6 in nitrobenzene, iron (III) interference being masked by addition of 2 mg EDTA. Other micronutrients remained in the aqueous phase. Molybdenum from the organic phase was stripped with 2M nitric acid. In triplicate analysis, the molybdenum concentration was found to be 2.45, 2.38 and 2.40 microgram per gram. The reported value was 2.42 microgram per gram.

**CONCLUSION:**

The important feature of this method is that it permits the separation of molybdenum from barium, cesium, thorium, strontium, rubidium, chromium and lanthanum which are usually associated with nuclear fission products. The method is applicable to the analysis of molybdenum in real samples. The proposed method is very simple, rapid, selective and reproducible. The total time required for separation and determination is 2 hours.

**General Procedure for separation of Mo (VI) with POLY-(DIBENZO-18-CROWN-6):**

An aliquot of solution containing 500 microgram of Mo (VI) was mixed with hydrochloric acid in the concentration range of 0.5-10M in a total volume of 10 ml. The solution was then passed through the column, preconditioned with hydrochloric acid of the same acidity as that of sample solution at a flow rate of 0.5 ml per minute. The column was subsequently washed with hydrochloric acid of the same acidity and then with water. The adsorbed molybdenum was eluted with different eluting agents at a flow rate of 0.5 ml/ minute. 5 ml fractions were collected and after evaporating the acid, it was extracted with water and the Mo (VI) content was determine spectrophotometrically Tiron with at 390 nm. The concentration of Mo (VI) was calculated from the calibration curve.

**Results and discussions:**

In order to ascertain the optimum concentration of hydrochloric acid for the quantitative adsorption of Mo (VI) on poly-(DB-18-C-6) various studies were conducted by varying the concentration of hydrochloric acid from 5 to 10M. It was found that the quantitative adsorption was at 5M hydrochloric acid concentration. In order to ascertain quantitative adsorption of Mo (VI) on 1 gm. of poly-(DB-18-C-6) various adsorption studies were carried out. From the results it is clear that, there was quantitative adsorption of Mo (VI) up to 70 mg per liter. The separation of Mo (VI) from binary mixture and multi component mixture was achieved.

***Application to analysis of molybdenum from alloys and steel:***

The method was applied for the analysis of molybdenum from nickel base alloy (BCS-CRM No. 345), alloy steel (BCS-CRM 401/1), low alloy steel (SS-407/1) and high speed tool steel (SS-486/1). The results obtained by our method are similar to the results obtained by standard methods.

**CONCLUSION:**

The important feature of this method is that it permits the separation of molybdenum from led, strontium, barium, lanthanum, chromium, vanadium, iron, cesium and cobalt. The method was applied to the analysis of molybdenum

from various alloy samples. The proposed method is very simple, rapid, selective and reproducible. The recovery of molybdenum in all instances from duplicate determinations is 100±2%.

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