# Reprimied from Atomic Aboar tion Newsletter Yol. 10, Yo. 3 DETERNINATION OF MOLYOOSNUM IN GEOLOGICA MATEMALS 

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## INFRODUCTION

The determination of molvhdenum ly atomic alsoption has rectived emisiderable attention Several iny wirstors have evaluated the eflets of interfering clement and the process of overeoming the effects by, the atidition of alumi: num or ammonium salts il, 2, 3, 1. 5. 6. Smivent extrattion systems used ammonium pyrmoldine dithicinathanatis IAPVC. (7. 1 ), 8 hy droxyquinoline 1.9. 10. 111 . Whimel (11), 2-octanol i12), $n$-amy acetate 113. These sytems. are quite efficient although the extrations requils close control of pll. Iem, an interfering clement that situily depresses molybienum absorption, is oflen coeviracted into the orranic phase.

Liquid ion exchangers, fie. quaternary ammonium halides have been used nith ulvantage in many anty tical applications. The selected ion exchanger should not lie soluble in water, but soluble in an oremil solvent. The halide ion is exchanged with the amion of interest in the aqueous layer and thus concentration of the wimon is achieved, while eliminating interfering element. l. extraction into an organic solvent. Accurate comli.il of pll is mut necessary to achieve complete extraction.

This paper will describe a method for the deternination of molybdenum in seological materials.

It is known that molybuenum as molybdare or phosphomolybate ion ean be extracted using lhe liquil ion exchanger. Sliquat 3.36 methyl tricaprll ammomimm chloride, available from General Mills, Ine. Kanlakee. III. ) Aliquat 336 has been used for analitical eparation of gold 114), tungsten (15), and metinidelamhanide ble: ments ( 16,17 ).

## procedure

Mix 0.2 s of finely pround cellogical sample with $1 \%$ of lithium metaborate and fuse for 15 minutes in a pruphte
crachle (18), Cool the melt and drop the plobule with foreeps into a 30re test tule contuinine 0 mi of a mixure at 10: phosphoric acid and 2 : 4 . 0 . Phosphoric actid an erts the moly bdenum to phosphomity bilute. My drogen permide is added to ensure conversion of all of the motyb. Ilemum the hexavalent stite. Any lower valence molyh. lenum is not extractalle with this sy term. (With this sy tem no interference due to iton occilis. Mdition of 0.2 g of Fe to the aqueous phase did not calise any itppression of molublenum signal. Place the test tibes on a magnetic stirrer hot plate and cominue stiring and heating until the sample is all in solution. Coml the test tuhe and add to ml of MIBK containine 3. Mquat 3sh. When greater concentration ratios are desired. \% mil MBK can be used. Shake for one minute and measure the moly liferum content of the organic laver by atomic absorption after the phases separate.
Standards: Fuse 0.3751 s of MoO, with 2 g of lithum metaborate in a platimm crucible: Diswhe the fusion in dilute phomphori aid las deerribed ahoyel and make up to 250 ml . The resulting solution will he a 1000 ks ml aqueous moly blenum standard This is limether diluted and extracted into UIBK containing. Aliquat 336 lo give $0.5,1$, 2.5. 5. 7.5 and 10 ug mil standards.

## Analysis of Geological Samples for Molybdenum

Five samples containing moly bdenum were analized by the proposed method, using: Perkin Elmer Model 303 atumic uhs option spectrophotometer with a nitrous oxideacetylene flame. The samples were also sent to three commercial laboratories. The results are compared in Table I. The precision obtained lor the lirst four samples is quite good. The poor precision for sample Do. LSob is apparently due to sampling errors particl- sparsity effect. The arree ment between result oltatied by the proposed method and ly the commercial lahoratories is quite good.

TABEE:
Anclysis of Qeological Materials for Molybdenum

| Sample No | Atomic Absorption, ppm | Average | Coefl of Varialion (\%) | Commercial laboratories |  |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  | A |  |  |  | $B^{b}$ | C |
|  |  |  |  | 1 | 2 | 3 | Av. |  |  |
| 1292 | $\begin{aligned} & 355,355,355,333,368, \\ & 368,388,353,353,367 \end{aligned}$ | 360 | 3.8 | 530. | 340 | 380 | 417 | 380 | 338 |
| 1302 | $\begin{aligned} & 190,195,212,201,210 \\ & 208,202,202,203,203 \end{aligned}$ | 203 | 2.9 | 175 | 258 | 202 | 215 | 200 | 182 |
| 1346 | $\begin{aligned} & 183,168,160,158,182 \\ & 155,155,175,160,161 \end{aligned}$ | 166 | 5.4 | 152 | 142 | 150 | 148 | 160 | 141 |
| 1357 | $\begin{aligned} & 100,100,100,105,95 \\ & 100,105,105,100,105 \end{aligned}$ | 102 | 2.2 | 108 | 96 | 96 | 99 | 83 | 87 |
| 1566 | $\begin{aligned} & 32,25,24,29,25,29, \\ & 35,25,33,36 \end{aligned}$ | 29 | 14.7 | 20 | 68 | 23 | 37 | 14 | 25 |

[^0]b. Acid diestion followed by atomie absorption inalysis.

Table 11 shows the results of an analysis of U.S.G.S. rock standards for molybdenum by this method compared with the reported average value (19). The analytical procedure is the same as described earlier except that only 5 ml of MIBK was used. A scale expansion of $10 x$ and a noise suppression setting of 2 were used. Considering the low level of molybdenum the agreement is quite good. The sensitivity obtained was 0.1 ppm/ $1 \%$ absorption.

TABLE IT
Molylodenum in Standard Reck Samples

| U.G.G.S. <br> Rock Siondards | A.A. ppm Mo | Average <br> Flanagon (19) |
| :---: | :---: | :---: |
| BCR-1 | 5.0 | 3.9 |
| PCC-1 | 4.1 | 5.5 |
| DTS-1 | 4.2 | 6.6 |
| AGU-1 | 6.5 | 3.7 |

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[^0]:    4. Colorimetrie analyi, 1 and 2 , carse sample, 3, resround samples.
