

- Benzoinoxime

Mo

Selective Dissolution and Property of Molybdenum Precipitate Formed by -Benzoinoxime

, , , , , , *

150, * 220

- benzoinoxime Mo

Mo ,

- benzoinoxime-Mo Mo 가 - benzoinoxime

. Mo - benzoinoxime - benzoinoxime

MoO₂⁺² - benzoinoxime-Mo ,

- benzoinoxime-Mo . 0.4 N NaOH 5

Mo 97.5 % 가 , 가

- benzoinoxime 가

Zr Ru 1.3, 7.7% Mo

- benzoinoxime-Mo

가 50%

Abstract

This study investigated the property of molybdenum precipitate formed with -benzoinoxime and performed experiments to dissolve selectively precipitate without hydrogen peroxide, which is compound to affect interference in a following purification process. The precipitate was composed of -benzoinoxime-Mo precipitate and re-precipitate of -benzoinoxime added excessively for increasing precipitation efficiency of molybdenum. It was confirmed that -benzoinoxime-Mo precipitate was formed by reaction two -benzoinoxime molecules and one MoO₂⁺². The form of -benzoinoxime-Mo precipitate was amorphous. Dissolving in 0.4 N NaOH solution without hydrogen peroxide dissolved molybdenum to 97.5 % from precipitate within 5 minutes. This result was similar to the case adding hydrogen peroxide. Hydrogen peroxide serve only as dissolving rapidly re-precipitate of -benzoinoxime. And also, this dissolution method was favorable in the purification aspect as zirconium and ruthenium were contained to 1.3 and 7.7 % respectively in dissolving solution. Organic quantity of the dissolving solution to be fed in a following silver coated activated carbon adsorption process could be decreased to 50 % more or as dissolving only -benzoinoxime-Mo precipitate without dissolving a part of precipitate, re-precipitate of -benzoinoxime.

1.

^{123}I , $^{99\text{m}}\text{Tc}$, ^{131}I , ^{89}Sr , $^{99\text{m}}\text{Tc}$, ^{201}Tl , ^{111}In , [1].
 (6 hour) gamma energy (140 KeV) 80%
 $^{99\text{m}}\text{Tc}$ 66 ^{99}Mo 가 decay
 ^{99}Tc Ru ^{99}Mo ^{98}Mo
 (neutron capture) ^{235}U (fission)
 (specific activity) generator 가
 ^{235}U 가 generator 가 가
 [2, 3]. ^{235}U ^{99}Mo
 ^{99}Mo / ^{99}Mo 가
 ^{99}Mo / ^{99}Mo Mo /
 [4, 5], Chelex - 100
 [6, 7], DEPHA [2, 4, 8, 9], - benzoinoxime [2,
 10, 11, 12], (thermal sublimation) [7]
 2가
 - benzoinoxime Mo U
 1 Mo / - benzoinoxime Mo
 Mo (steel) (pig
 iron) Mo [13]. - benzoinoxime
 Mo
 - benzoinoxime Mo / Mo
 SEM, FTIR, TG/DTA XRD Mo
 - benzoinoxime - benzoinoxime-Mo
 Mo
 NaOH 가
 Mo 가 가 [12].

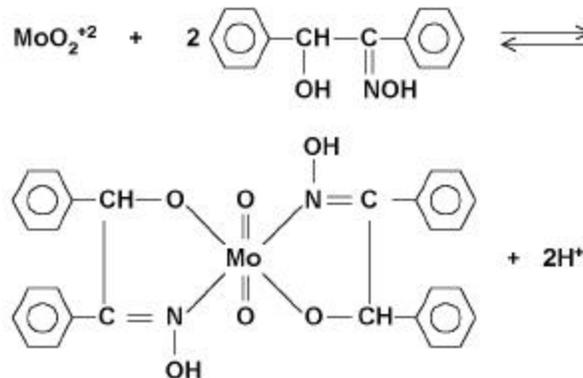
2.

13 (Rb, Cs, Sr, Ba, Mo, Zr, Te, Ru, Y, Ce, Pr, Nd, Sm),
 Table 1 ^{99}Mo 1 batch 93% U 4.39g 5
 1 300ml 가 ORIGEN 2 code
 U 5 30 가
 가 Mo
 $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ $\text{Te}(\text{OH})_6$ (RbNO_3 , CsNO_3 , $\text{Sr}(\text{NO}_3)_2$,

$Ba(NO_3)_2$, $ZrO(NO_3)_2 \cdot H_2O$, $Ru(NO)(NO_3)_3$, $Y(NO_3)_3 \cdot 4H_2O$, $Ce(NO_3)_3 \cdot 6H_2O$, $Pr(NO_3)_3 \cdot 6H_2O$,
 $Nd(NO_3)_3 \cdot 6H_2O$, $Sm(NO_3)_3 \cdot 6H_2O$, Ru ,
 - benzoinoxime 0.4 N NaOH 2 wt% . /
 - benzoinoxime-Mo Mo 1
 - benzoinoxime-Mo 50 ml - benzoinoxime
 (batch) 가 .
 0.45 μm cellulose nitrate membrane filter(Whatman)
 (desiccator) 1
 ^{99}Mo NaOH 1 wt%
 가 , 0.1 N, 0.4 N NaOH
 - benzoinoxime-Mo SEM (scanning electron microscope,
 Model Akashi DS 130S), FTIR (fourier transform infrared spectrometer, Model Nicolet 800),
 TG/DTA (thermogravimetry-differential thermal analysis, Model Setaram TG-DTA 92), XRD
 (X-ray diffractor, Model Siemens D5000) . Sr, Ba, Mo, Zr,
 Te, Y, Ce, Pr, Nd, Sm Mo ICP (inductively coupled plasma
 spectrometer, model ISA Jobin-Yvon JY 50P) , Ru monochromator ICP
 (inductively coupled plasma spectrometer, model ISA Jobin-Yvon JY 38 plus)
 , Rb Cs AA (atomic absorption spectrophotometer, model GBC 906A)
 TOC (total organic carbon analyzer, model Shimadzu TOC
 5000A) - benzoinoxime-Mo SEM
 (scanning electron microscope, Model Akashi DS 130S) .

3.

가. - benzoinoxime Mo
 - benzoinoxime Mo
 [14, 15], 가 . Fig.1 Mo



- benzoinoxime 가 (BzO/Mo) 20
 stoichiometry (BzO/Mo = 4.7) 가 - benzoinoxime-Mo SEM
 - benzoinoxime Mo Mo
 Mo - benzoinoxime 가 0.5 - 4.0 N ,

Mo 가 10ppm , Mo -benzoinoxime 가 가 20 97%
 Mo가 [15]. Mo
 -benzoinoxime 가 . BzO/Mo가 20
 1 μ m aggregation 가 50 μ m
 , BzO/Mo가 4.7 1 μ m
 . 가 가 20
 -benzoinoxime 가 , Fig.2
 -benzoinoxime 0.4 N NaOH 2 wt% 1 N 가
 . -benzoinoxime-Mo 1 μ m
 가 가 4.7
 . Fig.3 -benzoinoxime () -benzoinoxime-Mo FTIR .
 benzene C-H 3100 - 3000 cm^{-1} band , (monosubstitution)
 benzene 1500, 1450, 750, 697 cm^{-1} peak , 3300 - 3150 cm^{-1}
 OH peak가 . oxime(C=N-OH) OH 3300 - 3150
 cm^{-1} , C=N 1690 - 1620 cm^{-1} , N-O 930 cm^{-1} peak가
 , C-H 2900 cm^{-1} , C-O 1075 - 1000 cm^{-1} -benzoinoxime
 [16]. -benzoinoxime-Mo
 -benzoinoxime OH 900 cm^{-1}
 peak . M=O, V=O, Nb=O, Ta=O, W=O, Re=O, Os=O
 M=O group (M: metal, O: oxide) 1050 - 800 cm^{-1} peak가 [17]
 , 900 cm^{-1} peak가 Mo=O .
 -benzoinoxime-Mo FTIR 2 -benzoinoxime
 H⁺ 가 가 MoO₂⁺² . Fig.4
 0.4 N NaOH -benzoinoxime 1N
 FTIR -benzoinoxime peak
 -benzoinoxime 가 가
 . Fig.5 -benzoinoxime-Mo , MoO₃, -benzoinoxime() TG-DTA
 -benzoinoxime 550 , MoO₃ 785
 . TG-DTA
 MoO₂⁺² MoO₃ . -benzoinoxime-Mo 550
 -benzoinoxime MoO₂⁺² 785
 . TG -benzoinoxime Mo
 25% mole 1 : 1.9 FTIR
 가 . Fig.6 -benzoinoxime-Mo
 -benzoinoxime () 0.4 N NaOH -benzoinoxime 1N
 XRD pattern , XRD CuK 2.4 $^\circ$ /min
 (scanning speed) 2 = 5.5 - 35 $^\circ$. XRD pattern ,
 -benzoinoxime-Mo SEM
 -benzoinoxime -benzoinoxime() peak

-benzoinoxime peak가 FTIR 가

. Mo
Mo -benzoinoxime NaOH
가 20 가 5
Mo
. Fig.7 Mo 5 Mo
0.1 N NaOH
97 % 0.1 N NaOH 5, 88.8%
-benzoinoxime-Mo 가
60 가 20
가 -benzoinoxime Mo
가

Table 2 . Mo
Sr, Ba Nd

Zr Ru
Zr Ru
5 % Mo , 0.1 N NaOH
Zr Ru
-benzoinoxime-Mo NaOH
5
Fig.8 -benzoinoxime-Mo
-benzoinoxime
가
가 , Mo
가 20 TOC Fig.9
84.2 %가 0.4 N NaOH -benzoinoxime 가
-benzoinoxime 39.7 %
가

4.
-Benzoinoxime Mo -benzoinoxime-Mo

- 1) -benzoinoxime -benzoinoxime-Mo Mo
가 -benzoinoxime .
- 2) FTIR TG-DTA Mo -benzoinoxime -benzoinoxime
 MoO_2^{+2} -benzoinoxime-Mo .
, -benzoinoxime-Mo .
- 3) 0.4 N NaOH 5 Mo 97.5 % 가
가 , 가 -benzoinoxime
가 Zr Ru 1.3 ,
7.7 % Mo .
- 3) -benzoinoxime
-benzoinoxime-Mo 가
50 % .

1. M.A. Langton, *Trans. Am. Nucl. Soc.*, **72**, 134 (1995).
2. IAEA, Fission Molybdenum for Medical Use, IAEA-TECDOC-515 (1989).
3. A. Ali. Sameh and Ache. Hans. J., *Radiochimica Acta*, **41**, 65 (1987).
4. W. L. Cheng, C. S. Lee, C. C. Chen, and G. Ting, *Radiochim. Acta.*, **47**, 69 (1989).
5. J. L. Iturbe, *Appl. Radiat. Isot.*, **41**, 7, 693 (1990).
6. A. K. Gupta, E. S. Williams, and A. A. Aguwa, *Adsorption and Ion Exchange*, **78**, 103 (1979).
7. A. A. Sameh and J. A. Hans, *Radiochim. Acta.*, **42**, 65 (1987).
8. E. Ejaz, A. M. Mamoon, and M. A. Qureshi, *Appl. Radiat. Isot.*, **39**, 1 (1988).
9. T. Wei, W. L. Cheng, and G. Ting, *Solvent Extract. And Ion Exchange*, **2**, 3, 435 (1984).
10. C. K. Sivaramakrihan, Rep. BARC-84 (1976).
11. H. Arino, F. J. Cosolito, K. D. George, and A. K. Thrnton, USP 3940318.
12. W. L. Cheng, C. S. Lee, C. C. Chen, Y. M. Wang, and G. Ting, *Appl. Radiat. Isot.*, **40**, 4, 315 (1989).
13. H. Knowles, *Bur. Stds. J. Research*, **9**, 1 (1932).
14. E. Jdid, P. Blazy, *Ind. Miner., Mines Carrier Tech.*, **2**, 83 (1989).
15. D. Wu, S. Landsberger, G. F. Vandegrift, *J. Radioanal. & Nucl. Chem.*, **216**, 1, 101 (1997).
16. N. B. Colthup, L. H. Daly, S. E. Wiberley, *Introduction to Infrared and Raman Spectroscopy*, 3rd ed., Academic Press, San Diego (1990).
17. K. Nakamoto, *Infrared and Raman Spectra of Inorganic and Coordination Compounds*, 3rd ed., John Wiley & Sons, New York (1978).

Table 1. Chemical composition of the estimated UO₂ target and the simulated solution

| Element | Concentration (ppm) | | Element | Concentration (ppm) | |
|---------|---------------------|-----------|---------|---------------------|-----------|
| | Estimated | Simulated | | Estimated | Simulated |
| Ag | 0.15 | - | Pd | 0.26 | - |
| As | 0.01 | - | Pm | 2.91 | - |
| Ba | 15.6 | 15.6 | Pr | 13.8 | 13.8 |
| Br | 0.37 | - | Pu | 0.63 | - |
| Cd | 0.21 | - | Rb | 6.72 | 6.72 |
| Ce | 39.3 | 39.3 | Rh | 0.91 | - |
| Cs | 6.68 | 6.68 | Ru | 32.4 | 32.4 |
| Eu | 0.54 | - | Sb | 0.42 | - |
| Gd | 0.05 | - | Se | 0.95 | - |
| Ge | 0.01 | - | Sm | 4.73 | 4.73 |
| I | 10.5 | - | Sn | 0.59 | - |
| In | 0.02 | - | Sr | 27.3 | 27.3 |
| Kr | 7.12 | - | Tc | 7.16 | - |
| La | 1.58 | - | Te | 12.0 | 12.0 |
| Mo | 50.0 | 50.0 | U | 14123.0 | - |
| Nb | 0.53 | - | Xe | 21.0 | - |
| Nd | 36.9 | 36.9 | Y | 11.9 | 11.9 |
| Np | 0.42 | - | Zr | 64.5 | 64.5 |

Table 2. Fraction of the other elements remained in the dissolving solution with dissolution method

| Element | 0.4 N NaOH, % | 0.1 N NaOH, % | 0.4 N NaOH + H ₂ O ₂ , % | H ₂ O, % |
|---------|---------------|---------------|--|---------------------|
| Rb | 0 | 0 | 0 | 0 |
| Cs | 0 | 0 | 0 | 0 |
| Sr | 0 | 0 | 0 | 0 |
| Ba | 0 | 0 | 0 | 0 |
| Zr | 1.3 | 0.9 | 1.3 | 0 |
| Te | 0 | 0 | 0 | 0 |
| Ru | 7.3 | 5.3 | 7.7 | 0 |
| Y | 0 | 0 | 0 | 0 |
| Ce | 0 | 0 | 0 | 0 |
| Pr | 0 | 0 | 0 | 0 |
| Nd | 0 | 0 | 0 | 0 |
| Sm | 0 | 0 | 0 | 0 |

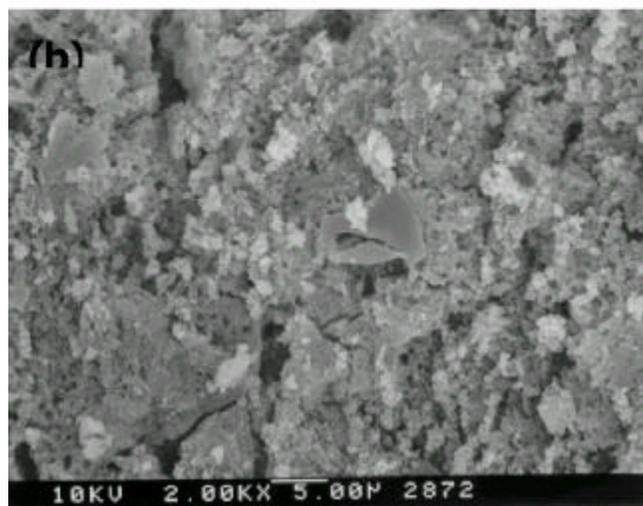
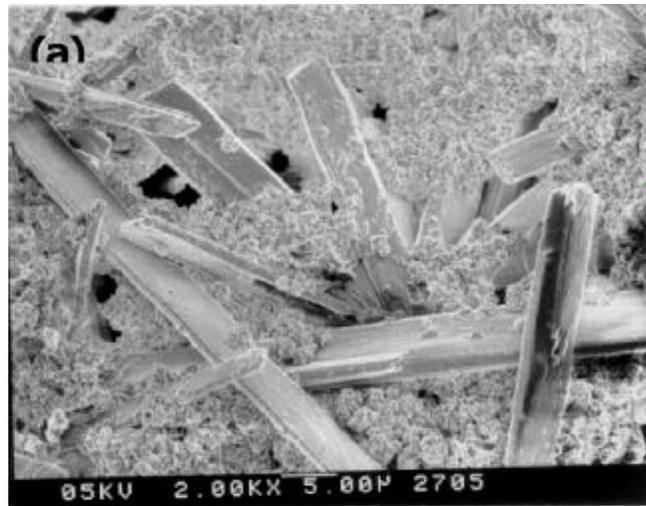


Fig. 1. SEM photographs of molybdenum precipitate
(a) BzO/Mo = 20 (b) BzO/Mo = 4.7



Fig. 2. SEM photograph of re-precipitated
-benzoinoxime

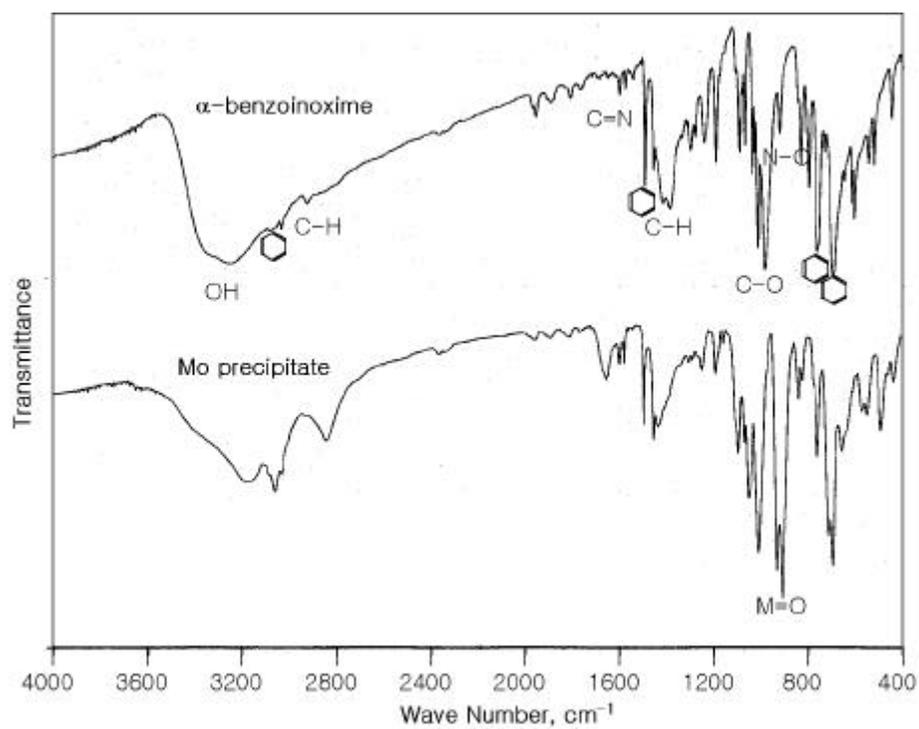


Fig. 3. FTIR spectrums of α -benzoinoxime and α -benzoinoxime-Mo precipitate

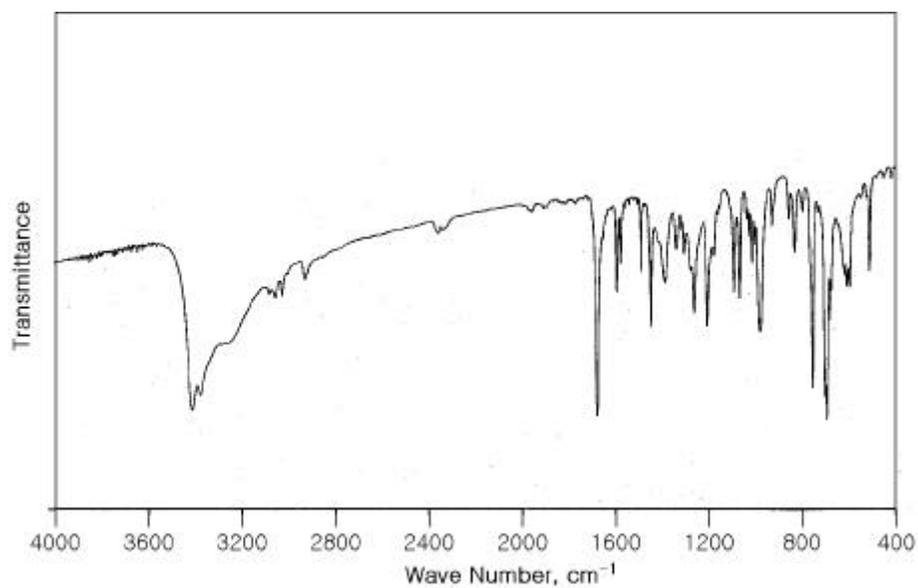


Fig. 4. FTIR spectrum of re-precipitated α -benzoinoxime

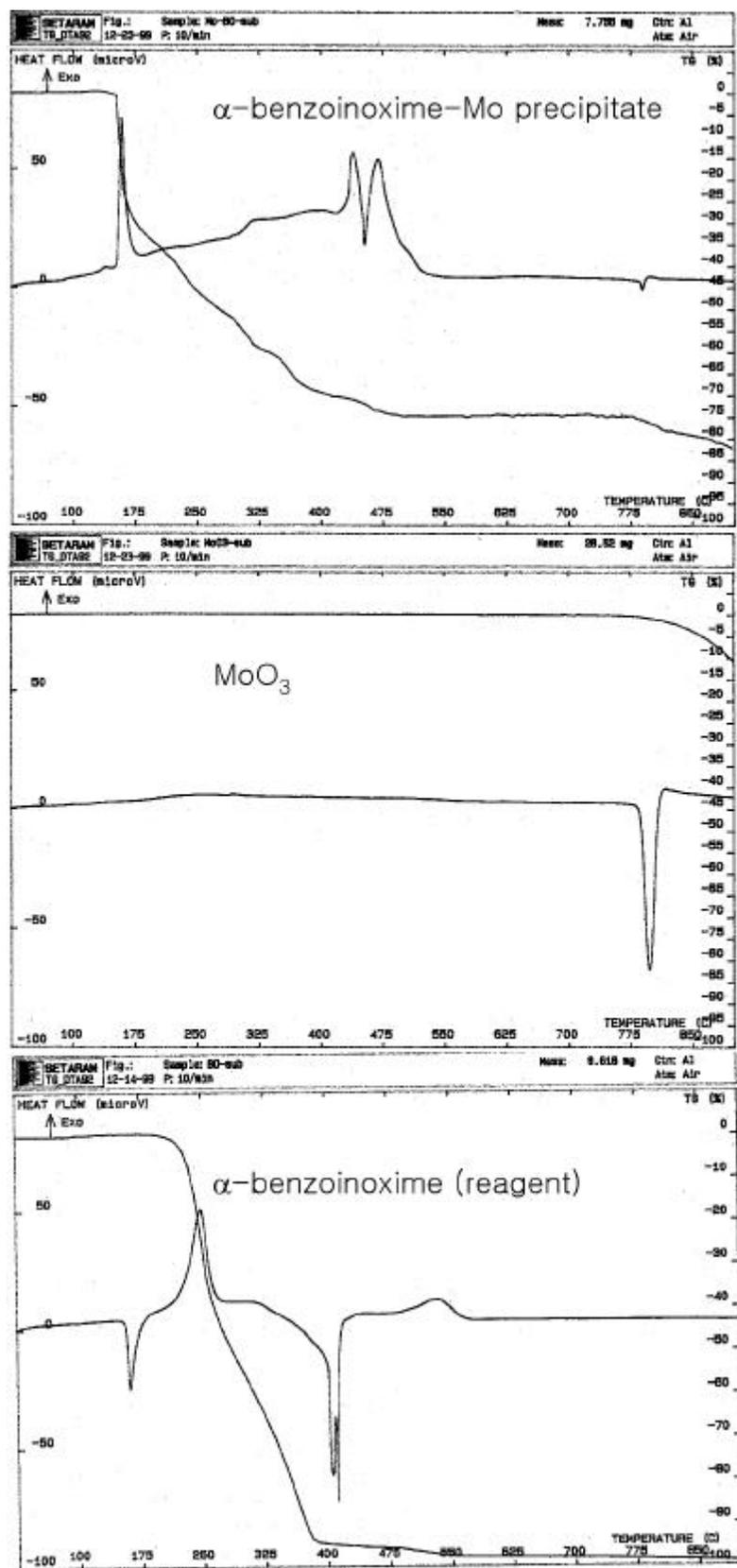


Fig. 5. TG-DTA of α -benzoinoxime precipitate, MoO_3 , and α -benzoinoxime

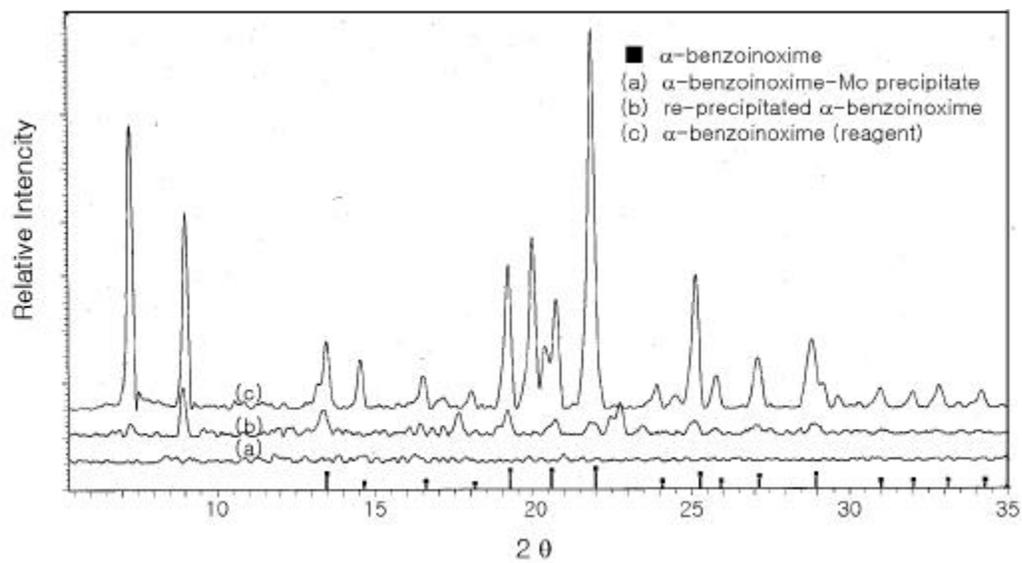


Fig. 6. XRD patterns of α -benzoinoxime-Mo precipitate, re-precipitated α -benzoinoxime, and α -benzoinoxime

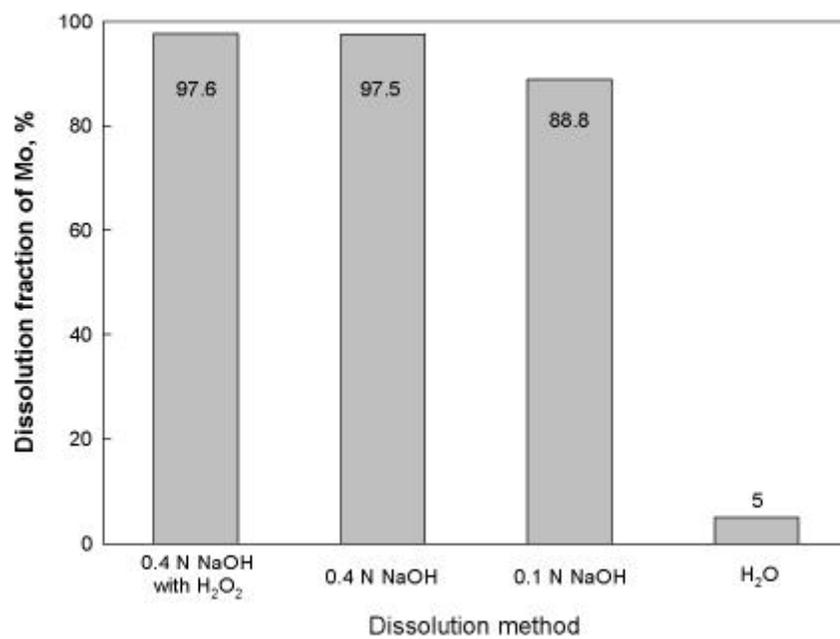


Fig. 7. Dissolution fraction of Mo with dissolution method



Fig. 8. SEM photographs of undissolved precipitate after dissolving during 5 minutes
(a: dissolution by 0.4N NaOH
b: dissolution by 0.4N NaOH+1wt% H₂O₂)

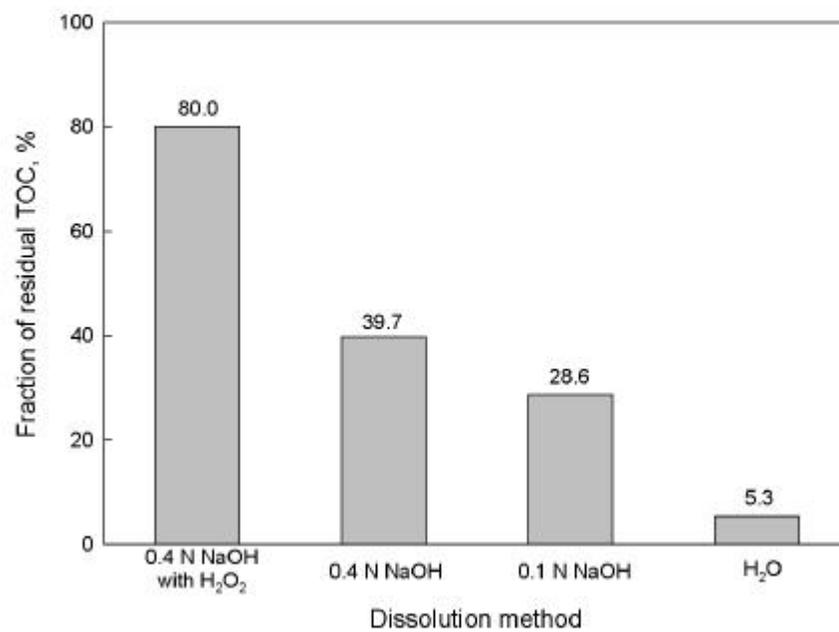


Fig. 9. Residual TOC fraction in the dissolution solution