



Separation of Au(III) from other precious and base metals using 1-methoxy-2-octoxybenzene in acidic chloride media



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ABSTRACT

Ethereal compounds act as good extractants for Au(III) from hydrochloric-acid media. We developed a novel aromatic ethereal compound 1-methoxy-2-octoxybenzene (*o*-MOB) as an extractant for Au(III). Au(III) extraction using *o*-MOB in hydrochloric-acid media reached equilibrium rapidly within 10 min. Of a series of ethereal extractants, including positional isomers, *o*-MOB showed the highest extractability for Au(III). Additionally, *o*-MOB showed a higher extractability for Au(III) than a commercially available extractant, dibutyl carbitol, under diluted conditions. Au(III) was extracted selectively using *o*-MOB over other precious- and base-metal ions in 0.1–1.0 M HCl. Au(III) that was extracted by using *o*-MOB was stripped quantitatively using hydrochloric-acid solution that contained thiourea.

1. Introduction

In addition to its use in jewelry and investment, gold is used in electronic devices such as mobile phones, personal computers, and laptops. An annual excess of 300 t of gold is used in electronic components (Hagelüken and Corti, 2010). Therefore, gold recovery from waste electrical and electronic equipment (WEEE) has attracted significant attention (Kang and Schoenung, 2005; Park and Fray, 2009). Precious-metal recycling is important from an economic and environmental viewpoint.

Solvent extraction is a promising method for the separation and recovery of precious metals from WEEE. Because many element types are contained in WEEE, the extractant extractability and selectivity are key factors that are required in the development of the separation process. Various extractants, such as sulfur-containing extractants and amine extractants, have been developed for the extraction of Au(III) (El Aamrani et al., 1998; Iwakuma et al., 2008; Argiropoulos et al., 1998; Fu et al., 1994; Martínez et al., 1999; Kejun et al., 2004). Additionally, oxygen-containing extractants have been developed for the extraction of Au(III) in hydrochloric-acid media (Narita et al., 2006). 4-Methyl-2-pentanone (methyl isobutyl ketone, MIBK) is a commercially available extractant for Au(III) (Cox, 1992; Kargari and Kaghazchi, 2004). MIBK shows a high extractability and loading capacity for Au(III) in hydrochloric-acid media.

Ethereal compounds are effective for Au(III) extraction from hydrochloric-acid media (Koshima and Onishi, 1986). In particular, a glyme compound, dibutyl carbitol (bis(2-butoxyethyl) ether, DBC), is

used commercially for the selective extraction of Au(III) from other metals (Mironov, 2012, 2013; Jung et al., 2009). Au(III) is extracted selectively from the platinum-group metals by using DBC, and the extracted Au(III) is stripped reductively as metallic gold. Au(III) extraction using DBC from acidic aqueous solutions is applied commercially, and is used in INCO's operations (Edward and te Riele, 1983; Javanshir et al., 2011). Recently, an ethereal compound, cyclopentyl methyl ether (CPME), was found to be useful for the extraction of Au(III) from hydrochloric-acid media (Oshima et al., 2017).

Although DBC is powerful for the extraction of gold, it has a few problems, such as low levels of contamination of the aqueous phase and an elevated viscosity. Other ethereal compounds act as powerful extractants for Au(III) with fewer problems. However, limited reports exist on the solvent extraction of gold using aromatic ethereal extractants. In this study, 1-methoxy-2-octoxybenzene (*o*-MOB, Fig. 1) was prepared as a novel extractant for Au(III). This is the first report on Au(III) extraction using *o*-MOB. The extractant has two ethereal oxygen atoms, which are placed in the vicinity of a structurally rigid aromatic skeleton. The ethereal oxygen atoms act as electron donors and protonate under strong acidic conditions to interact with Au(III) that is present as an anionic chloride complex under higher hydrochloric-acid concentrations. Various aromatic ethereal compounds, including the positional isomers of *o*-MOB, were synthesized to compare their extractability with *o*-MOB. The extractability using the most popular ethereal extractant, DBC, was compared with that of *o*-MOB under dilute conditions. The extraction selectivity for various metal ions using *o*-MOB was also investigated. Finally, conditions for stripping Au(III)

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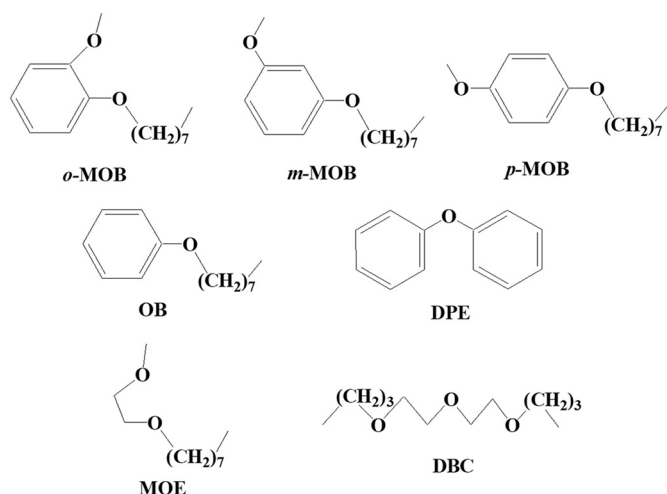


Fig. 1. Molecular structures and abbreviations for ethereal extractants used.

extracted using *o*-MOB were studied.

2. Experimental section

2.1. Materials

Analytical-grade gold(III), palladium(II), platinum(IV), rhodium(III), iron(III), aluminum(III), gallium(III), indium(III), cobalt(II), nickel(II), copper(II) and zinc(II) chlorides (Wako Pure Chemical Ind. Ltd., Japan) were used to prepare test solutions of the metal ions. The molecular structures of ethereal extractants that were used in this study are shown in Fig. 1. Analytical-grade DBC and diphenylether (abbreviated as DPE) (Wako Pure Chemical Ind. Ltd., Japan) that were used in the extraction tests were used without further purification. *o*-MOB, 1-methoxy-3-octoxybenzene (*m*-MOB), 1-methoxy-4-octoxybenzene (*p*-MOB), 1-octoxybenzene (OB) and 1-methoxy-2-octoxyethane (MOE) were synthesized as described in Section 2.2. The viscosity of *o*-MOB was determined by using a Cannon–Fenske viscometer. The *o*-MOB density was determined by using a pycnometer. The logarithm of the partitioning coefficient between *n*-octanol and water ($\log P$) is widely accepted as a quantitative indicator of the hydrophilic–lipophilic balance. The $\log P$ values of the ethereal extractants were estimated by using MarvinSketch 6.2.1 (ChemAxon Ltd., Budapest, Hungary) and the KLOP method (Klopman et al., 1994). All other reagents and solvents were of analytical grade and were used as received.

2.2. Synthesis of ethereal extractants

2.2.1. 1-Methoxy-2-octoxybenzene (*o*-MOB)

The synthesis scheme for 1-methoxy-2-octoxybenzene (*o*-MOB) is shown in Fig. 2. To 50 cm³ DMF, 2-methoxyphenol (guaiacol, 6.0 g, 48 mmol) and potassium carbonate (7.8 g, 56 mmol) were added. To the solution, a DMF solution that contained 15.6 g (80.8 mmol) 1-bromooctane was added dropwise and the mixture was stirred for 24 h at 60 °C. After DMF evaporation *in vacuo*, a chloroform solution of the residue was washed three times with 1 mol/dm³ hydrochloric acid, 1 mol/dm³ aqueous sodium-hydroxide solution and with distilled water. After drying over anhydrous sodium sulfate and filtration, the solution was evaporated *in vacuo*. The resulting viscous-liquid *o*-MOB

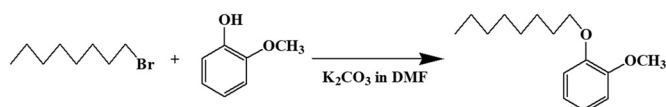


Fig. 2. Synthetic scheme for *o*-MOB.

product had the following properties: ¹H NMR (400 MHz, CDCl₃, 25 °C) 0.88 (3H, t, CCH₃), 1.28 (8H, m, C–CH₂–C), 1.45 (2H, m, C–CH₂–C–O), 1.83 (2H, m, C–CH₂–C–O), 3.82 (3H, s, CH₃–O–Ar), 3.98 (2H, t, C–CH₂–O–Ar), 6.86 (4H, d, Ar–H).

2.2.2. 1-Methoxy-3-octoxybenzene (*m*-MOB)

m-MOB was prepared by using 3-methoxyphenol and 1-bromooctane, according to a manner similar to that of *o*-MOB. The resulting *m*-MOB product had the following properties: ¹H NMR (400 MHz, CDCl₃, 25 °C) 0.88 (3H, t, CCH₃), 1.29 (8H, m, C–CH₂–C), 1.43 (2H, m, C–CH₂–C–O), 1.74 (2H, m, C–CH₂–C–O), 3.74 (3H, s, CH₃–O–Ar), 3.90 (2H, t, C–CH₂–O–Ar), 6.47 (3H, m, Ar–H), 7.14 (1H, m, –O–C–ArH–C–O).

2.2.3. 1-Methoxy-4-octoxybenzene (*p*-MOB)

p-MOB was prepared by using 4-methoxyphenol and 1-bromooctane, in a manner similar to that of *o*-MOB. The resulting *p*-MOB product had the following properties: ¹H NMR (400 MHz, CDCl₃, 25 °C) 0.88 (3H, t, CCH₃), 1.29 (8H, m, C–CH₂–C), 1.43 (2H, m, C–CH₂–C–O), 1.73 (2H, m, C–CH₂–C–O), 3.72 (3H, s, CH₃–O–Ar), 3.86 (2H, t, C–CH₂–O–Ar), 6.80 (4H, d, Ar–H).

2.2.4. 1-Octoxybenzene (OB)

OB was prepared using phenol and 1-bromooctane, in a manner similar to that of *o*-MOB. The resulting OB product had the following properties: ¹H NMR (400 MHz, CDCl₃, 25 °C) 0.89 (3H, t, CCH₃), 1.30 (8H, m, C–CH₂–C), 1.44 (2H, m, C–CH₂–C–O), 1.75 (2H, m, C–CH₂–C–O), 3.90 (2H, t, C–CH₂–O–Ar), 6.87 (3H, m, Ar–H), 7.23 (2H, m, ArH–C–O).

2.2.5. 1-Methoxy-2-octoxyethane (MOE)

The synthesis scheme for 1-methoxy-2-octoxyethane (MOE) is shown in Fig. 3. To 50 cm³ DMF, 2-methoxyethanol (9.13 g, 120 mmol) and sodium hydride (5.76 g, 240 mmol) were added. To the solution, a DMF solution that contained 19.3 g (100 mmol) 1-bromooctane was added dropwise and the mixture was stirred for 72 h at 0 °C. After inactivation of the sodium hydride using methanol and water, the mixture was evaporated *in vacuo*. Subsequently, a chloroform solution of the residue was washed three times with 1 mol/dm³ hydrochloric acid and distilled water. After drying over anhydrous sodium sulfate and being filtered, the solution was evaporated *in vacuo*. The product was purified by using a single-channel automated flash-chromatography system (Yamazen AI-580, Osaka, Japan) on a silica-gel column (SI-40D, D50 × 300 mm, Yamazen Corp.) under a linear gradient of 1-hexane and ethyl acetate at 45 cm³/min at room temperature and using an ultraviolet detector at 254 nm. The resulting MOE product as a viscous liquid had the following properties: ¹H NMR (400 MHz, CDCl₃, 25 °C) 0.88 (3H, t, CCH₃), 1.27 (10H, m, C–CH₂–C), 1.60 (2H, m, C–CH₂–C–O–C), 3.39 (3H, s, CH₃–O), 3.45 (2H, t, O–C–CH₂–C–), 3.57 (4H, m, O–CH₂–CH₂–O).

2.3. Liquid–liquid extraction tests

Liquid–liquid extraction tests were conducted batchwise with a typical procedure as follows. An aqueous solution was prepared by dissolving each metal ion (Au(III), Pd(II), Pt(IV), Rh(III), Fe(III), Al(III), Ga(III), In(III), Co(II), Ni(II), Cu(II) or Zn(II)) to form 0.1-mM solutions in HCl. The initial concentration of hydrochloric acid was adjusted to 0.1–8.0 M. An extracting solution was prepared by dissolving 200 mM of an extractant (*o*-MOB, *m*-MOB, *p*-MOB, OB, MOE, DPE or DBC) in

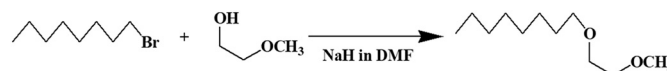


Fig. 3. Synthetic scheme for MOE.

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