

## 溶媒抽出의 一般的 原理<sup>†</sup>

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## Liquid-Liquid Extraction General Principles - A Review<sup>†</sup>

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### 요 약

본 총설은 용매추출(또는 액-액 추출)에 대한 일반적인 원리와 추출제별 용매추출 공정의 기초 원리를 소개하고 있다. 용매추출은 서로 섞이지 않는 두 상(phase)간에 화합물이 한 상(phase)에서 다른 상으로 이동하는 현상을 이용하는 공정이다. 초기에는 분석 화학 분야에서 용매추출의 간편성, 신속성 및 넓은 적용성 때문에 많이 사용되었으며, 분액깔테기와 같은 간단한 도구로 수분내에 추출실험을 완료할 수 있다는 장점이 있다.

주요어 : 용매추출, 액-액 추출, 일반적 원리, 총설

### Abstract

The present review paper deals the liquid-liquid extraction (LLE) general principles and the basic fundamentals, general process of LLE followed by the importance of LLE reagents. LLE is a process of transferring a chemical compound from one liquid phase to a second liquid phase, immiscible with the first. In analytical chemistry, this method enjoys a favored position among separation techniques because of its simplicity, speed and wide scope. By utilizing apparatus no more complicated than a separatory funnel and requiring several minutes at most to perform, extraction procedures offer much to the analytical chemist.

**Key words :** solvent extraction, liquid-liquid extraction, General principles, review

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## 1. Introduction

### 1.1. Liquid - liquid extraction: General

Liquid-liquid extraction (LLE) highlights the usefulness of phase distribution and is based upon the principle that a solute can distribute itself in a certain ratio between two immiscible solvents. For the separation and purification of metal ions, this method is known since 1872, when Peligot<sup>1)</sup> found the removal of uranium as uranyl nitrate with ethyl ether. Among the separation methods, LLE enjoys advantage position because of its speed, convenience, appli-

cability to both trace and macro concentration levels and, in a large number of cases, because the extracted constituent of interest is transformed into a readily measured state.

The process of LLE as applied<sup>2)</sup> in metallurgical processing is an equilibrium process, which can be described simply by an equation:



In the first step the metal, M, is transferred from an aqueous phase to an organic phase as some ME, in which case the process requires that the equilibrium position in this equation be shifted to the right. The second step, the metal is transferred from the organic

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phase to an aqueous phase, in which the equilibrium position be shifted to the left.

In LLE, a solute distributed itself between two immiscible liquids. According to Gibbs phase rule:

$$P+V \rightleftharpoons C+2 \quad (2)$$

Where P is the number of phases, V, the variance or degrees of freedom and C, the number of components. That is if we choose the concentration of the solute in one phase, its concentration in the other phase is fixed.

The distribution law, stated by Berthelot and Jungfleisch<sup>3)</sup> and thermodynamically elaborated by Nernst<sup>4)</sup> in 1891, states that at a particular temperature for a solute 'A' distributing between two immiscible solvents 1 and 2, at equilibrium, the ratio of concentrations in the two phases is a constant, provided the nature of the species is the same in both the phases.

$$D = [A]_1 / [A]_2 \quad (3)$$

The constant, D, is known as the distribution, partition or extraction coefficient. The chemical potential ( $\mu$ ) of the solute is the same as in each phase at equilibrium provided temperature and pressure are constant, i.e.,

$$\mu_1 = \mu_2 \quad (4)$$

Where the subscripts 1 and 2 refer to the respective solvent phases. Substituting suitable expressions of  $\mu$ ,

$$\mu_1^\circ + RT \ln m_1 + RT \ln \gamma_1 = \mu_2^\circ + RT \ln m_2 + RT \ln \gamma_2 \quad (5)$$

Where  $\mu$  represents the chemical potential of the solute in a hypothetical ideal 1 molal solution, m, the solute concentration in molality and  $\gamma$ , the molal activity coefficient.

The molal distribution coefficient,

$$D = \frac{m_2}{m_1} = \frac{\gamma_1}{\gamma_2} e^{-(\mu_2^\circ - \mu_1^\circ)/RT} \quad (6)$$

for a system in which the two solvents are completely immiscible under all circumstances the exponential term is a constant, K, so that

$$D = \frac{m_2}{m_1} = \frac{\gamma_1}{\gamma_2} K \quad (7)$$

Thus, D depends on the activity coefficients of the solute in each of the phases. When the activity coefficients approach unity, i.e., at low concentrations, D becomes constant. The distribution coefficient is related to the percentage extraction, E by the equation

$$E = \frac{100D}{D + \frac{V_{aq}}{V_{org}}} \quad (8)$$

Where  $V_{aq}$  and  $V_{org}$  are the volumes of the aqueous and organic phases, respectively. Separation factor,  $\beta$  which describes the effectiveness of separation of two solutes, is given by

$$\beta = D_1 / D_2 \quad (9)$$

Where  $D_1$  and  $D_2$  are the extraction coefficients for solutes 1 and 2 respectively.

The general LLE process flow sheet is given below as Scheme 1. It involves extraction, scrubbing, stripping and solvent equilibration stages.

**Extraction stage:** The metal-bearing aqueous feed solution and solvent is feed usually in a counter current manner, into a contactor in which the two phases are mixed. In this stage the metal of interest is transferred from the aqueous to organic phase. After settling, the aqueous phase or raffinate is separated and treated for the recovery of other metals, recycled to some point upstream or downstream from the solvent extraction.

**Scrubbing stage:** From the extraction stage, the loaded solvent may go to another contactor where it is scrubbed with suitable aqueous solutions to remove small amounts of metals or impurities co extracted in the extraction stage. The scrub raffinate (aqueous) may then be recycled to either leaching stage or to the aqueous solvent extraction feed tank.

**Stripping stage:** After scrubbing, the loaded solvent phases transferred to a third stage in which the metal is stripped from the organic phase by some suitable aqueous solution producing a fairly concentrated solution of the metal salt which then goes to further processing for metal production. The stripped solvent

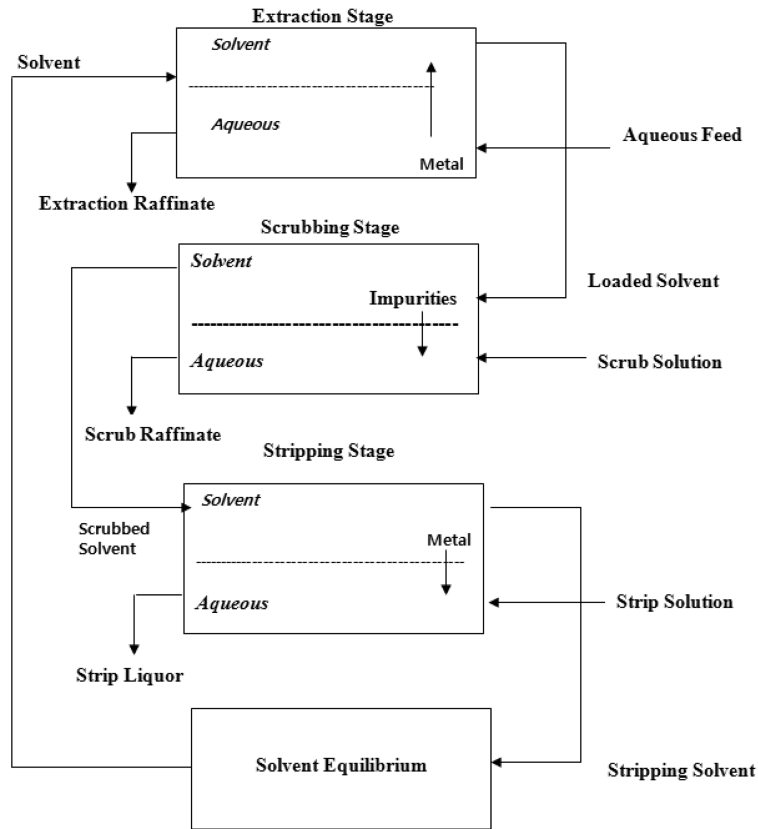


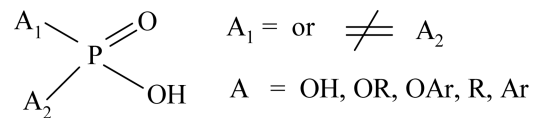
Fig. 1. The general process of liquid-liquid extraction.<sup>2)</sup>

is recycled back to the extraction stage. If necessary it is treated (equilibrated) prior to entering the extraction stage.

## 2. Importance of liquid-liquid extraction reagents

### 2.1. Organophosphorus extractants

Organophosphorus extractants have been used commercially for the extraction of many transition metals.<sup>5-22)</sup> Some particular advantages of the use of organophosphorus extractants in LLE processing are its chemical stability, good kinetics of extraction, good loading and stripping characteristics. Recently there has been increased interest in the potential applications of phosphonic and phosphinic acids in the separation of cobalt-nickel. Organophosphorus acid derivatives can be represented structurally as follows:



The active group = PO(OH) is responsible for their extractive properties.

Thioorganophosphorus extractants such as Cyanex 302 (bis(2,4,4-trimethylpentyl) monothiophosphinic acid) and Cyanex 301 (bis(2,4,4-trimethylpentyl) dithiophosphinic acid) were originally developed for use in the selective recovery of zinc from solutions containing magnesium and particularly, calcium.<sup>23)</sup> Since their introduction in 1985 and 1988, respectively, several more potential applications for these reagents have been examined. Two of these are (i) the recovery of cadmium from wet process phosphoric acid and (ii) the separation of cobalt from nickeliferous solutions

that also contain manganese. All are derived from the reaction of phosphine with 2,4,4-trimethylpentene-1 to produce intermediate bis(2,4,4-trimethylpentyl) phosphine, which is subsequently reacted with hydrogen peroxide to produce the phosphinic (Cyanex 272) and/or sulfur to produce monothiophosphinic (Cyanex 302) or dithiophosphinic (Cyanex 301) acids.

## 2.2. The oximes

The oximes as extraction reagents offer immense possibilities for chemical separations in view of the numerous combinations of long chain oximes, the wide array of diluents available and many anionic aqueous systems that can be readily produced. Oximes are designed specifically for the selective extraction of copper from dilute copper dump leach liquors by solvent extraction. These extractants being investigated mainly for the extraction of copper, however other metals are also extracted by the oximes depending on the aqueous pH and metal oxidation state. Several types of oximes have been used for metal extraction to study their extraction abilities, loading capacities and kinetics of extraction.

## 2.3. Isoxazolone

Isoxazolone group based extractants, have low acid dissociation constant due to electron delocalisation, and this property makes the isoxazolones an interesting class of -diketones with potential application as reagents for extraction of metal ions from acid media.

## 3. Liquid - liquid extraction systems

The nature of extractable metal species is of fundamental importance in metal extraction systems. Based on this fact, Ritcey and Ashbrook<sup>2)</sup> classified the extractants into three classes as indicated below.

- (1) Those, which involve compound formation
- (2) Those, which involve ion association
- (3) Those, which involve solvation of the metal ion

### 3.1. Extractions involving compound formation

These class of extractants can be further divided into two sub classes, namely acidic extractants and chelating extractants. The former are those having reactive groups such as COOH, = P(O)OH, -SO<sub>3</sub>H while the later is

those, which chelate with metal ions.

#### 3.1.1. Acid extractants

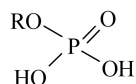
Acid extractants are cationic liquid ion exchangers, extract metal ions by a cation exchange mechanism, in which hydrogens of the extractant are exchanged for metal ions. Basically, the general mechanism is as shown in equation (10)



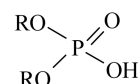
The metal extracts of acidic type used in commercial operations are organic derivatives of phosphorus acids and monocarboxylic acids.

#### 3.1.1.1. Acidic organophosphorus extractants

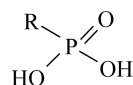
This type of extractions includes the following:



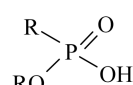
Mono alkyl phosphoric acid



Dialkylphosphoric acid



Mono alkyl phosphonic acid

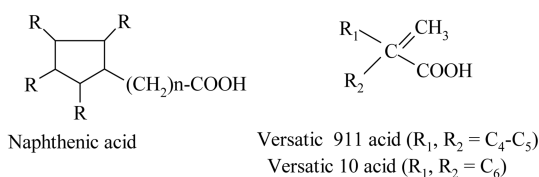


Dialkyl phosphonic acid

Where R can be alkyl or aryl substituent. Of these, alkyl phosphoric acids have proved to be most versatile, especially di-(2-ethylhexyl)phosphoric acid (D2EHPA). It has been used commercially for the extraction of many metals including uranium,<sup>24)</sup> cobalt and nickel,<sup>25)</sup> rare earths<sup>26)</sup> and vanadium.<sup>27)</sup> Among the esters of phosphonic acid, 2-ethylhexylphosphonic acid mono-2-ethylhexyl ester (EHEHPA) has been widely used for the extraction and separation of cobalt and nickel<sup>28)</sup> and rare earths.<sup>29)</sup> Various dialkyl phosphinic acid extractants such as bis(2,4,4 trimethyl pentyl)phosphinic acid (Cyanex 272) has been proved to be an important extractant for cobalt and nickel<sup>30)</sup> and rare earth separations.<sup>31)</sup>

#### 3.1.1.2. Carboxylic acids

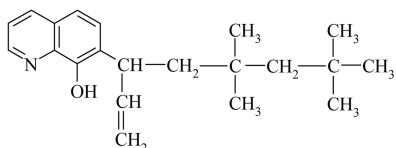
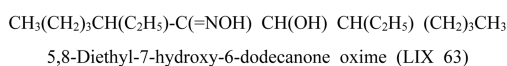
Carboxylic acids such as naphthenic acid and versatic acids have been well studied for the extraction and separation of metal ions.



Commercial LLE processes using carboxylic acids include the extraction of yttrium<sup>32)</sup> and separation of cobalt and nickel.<sup>33)</sup> Reviews on the use of carboxylic acids in LLE processing of metal ions and their applicability in this field are available in the literature.<sup>34-35)</sup>

### 3.1.2. Chelating extractants

Chelating extractants contain donor atoms such as nitrogen, sulphur or oxygen as the basic atom capable of forming bidentate complexes with metal ions. Examples of some chelating reagents are dithizone, cupferron, acetylacetone, thenoyltrifluoroacetone, 4-acetyl-5-pyrazolones and derivatives of 5-isoxazolones. Commercial useful chelating extractants are of two types—a series of substituted 2-hydroxybenzophenone oximes produced by Henkel Corporation under the name LIX reagents and the substituted 8-hydroxyquinolines produced by Sherex (Ashland Chemical Company) and marketed under the name Kelex. Most of these extractants were developed for the specific extraction of copper from acidic leach liquors and from alkaline solutions.<sup>36-37)</sup>

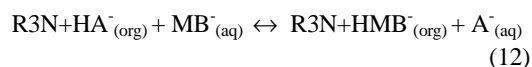


7-[3-(5,5,7,7-Tetramethyl-1-octenyl)]-8-hydroxyquinoline (Kelex 100)

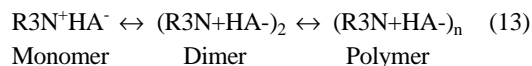
### 3.2. Extractants involving ion association

Quaternary ammonium compounds with high molecular weights (Aliquat 336) behave chemically as strong base anion exchangers and require lower concentrations of salting out reagents. Commercial processes have been developed which employ primary ( $RNH_2$ ),<sup>38)</sup> secondary ( $R_2NH$ ),<sup>39)</sup> tertiary ( $R_3N$ )<sup>40)</sup> amines and quaternary ammonium salts ( $R_4N^+$ ).<sup>41)</sup> The

usefulness of amines as extractants depends essentially on the ability of ions to form anionic species in the aqueous phase, which are extracted by amines in an anion exchange process. In order to achieve this exchange, the amine is first converted to the appropriate amine salt to provide an anion to exchange with the metal species.



One important factor influencing the extraction of metals by amine is the aggregation of the amine in the organic phase, which is determined by the nature of the diluent.



Another factor influencing metal extraction is the nature of carbon chain and also the number of carbon atoms in the chain. Some of the metal species, which have been reported to be extracted by quaternary ammonium halides (Aliquat 336) are studied by Miller and Fuerstenau.<sup>42)</sup>

### 3.3. Extractants involving solvation

Oxygenated organic solvents such as alcohols, ketones, ethers and esters show some basicity because of the lone pair of electrons on the oxygen atom and can, therefore, directly solvate protons and metal ions and bring about their extraction. Alcohols solvate better than ketones or ethers. Among ketones, methylisobutyl ketone (MIBK) has found commercial application in the separation of zirconium / hafnium<sup>43)</sup> and niobium/tantalum.<sup>44)</sup>

Esters of organophosphorus acids and a number of related compounds constitute a class of organic solvents which show remarkable solvating properties, whose essential functional group is the semi polar P=O bond with the oxygen atom having good steric availability. Tributylphosphate (TBP) has been used in LLE and may be taken as a typical example of compounds containing P=O bond. The difference in

the degree of extraction of elements is utilized for their mutual separation using TBP.<sup>45)</sup> The basicity and hence extractability of organophosphorous compounds is in the order: phosphates < phosphonates < phosphinates < phosphine oxides. Among trialkylphosphine oxides, tri-n-octylphosphine oxide (TOPO) has been used for the recovery of vanadium from phosphate liquors.<sup>46-47)</sup> Cyanex 923 and Cyanex 925 belong to the new class of extractants consisting of mixture of trialkylphosphine oxides.

Sulphoxides are pyramidal molecules with a lone pair of electrons on both the sulphur and oxygen atoms, capable of donation. A correlation of basicity with extracting power clearly establishes the position of sulphoxides between ketones and phosphine oxides. Thus the sulphoxides find their place as promising extractants for a number of metal ions.<sup>48-49)</sup>

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