Modern Environmental Science and Engineering (ISSN 2333-2581)
September 2015, Volume 1, No. 3, pp. 152-155
Doi: 10.15341/mese(2333-2581)/03.01.2015/007
Academic Star Publishing Company, 2015
www.academicstar.us

Substoichiometric Solvent Extraction and Radio-chemical Separation of Co(II) from other Elements with 2-mercaptobenzimidazole into Nitrobenzene

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Abstract: Ideal conditions for the substoichiometric solvent extraction and radiochemical separation of Co(II) from other elements with 2-mercaptobenzimidazole into nitrobenzene have been established. The effects of various parameters such as pH, time of equilibration, solvents, cation and anion interferences have been studied. The stoichiometry of metal to reagent has been evaluated.

Key words: substoichiometric solvent extraction, 2-mercaptobenzimidazole, Co(II), nitrobenzene

1. Introduction

Solvent extraction also known as liquid-liquid extraction is considered the most versatile and popular among various methods of separation, because of its ease, simplicity, speed and wide scope. Utilizing apparatus no more complicated than a separated funnel, requiring several minutes at the most to perform and applicable both to macro and trace levels of metals, this extraction technique offers much to the analyst.

^{60}Co is useful in industry, medicine and science. ^{60}Co is used in place of X-rays or radium in the inspection of material to reveal internal structure, flaws or foreign objects. The advantages being lower cost, more homogeneous gamma-radiation and softer beta-radiation, no radiation contamination from radon gas and ability to be shaped in any form before irradiation to fit special requirements.

^{60}Co is used in cancer therapy and extensively as radioactive tracer in biology and industry. Its half-life of 5.3 years eliminates the need to consider changes in source strength and provides tracer at economical rate.

The ever expanding interest and applications of cobalt has generated a great demand for more accurate and reliable techniques for separating it from a variety of complex matrices. Solvent extraction has been used extensively as a rapid and selective separation technique for the extraction of cobalt using several reagents into organic phase. Radiochemical studies of extraction of Co(II) with 4-methoxy-N-m-tolylbenzohydoxamic acid into chloroform have been carried out [1]. Liquid-liquid extraction of divalent Co and several other metal-ions using various reagents into organic solvent has been carried out [2-10].

The present investigation deals with the rapid and selective radiochemical separation of Co(II) from other elements by solvent extraction with 2-mercapto-benzimidazole into nitrobenzene.

2. Experimental

All the chemicals and reagents used were of A.R. grade. 2-mercapto-benzimidazole of Lobachemie make was used without further purification. The stock

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solution of Co(II) and other metal-ions was prepared by dissolving appropriate amount of salts in distilled water. Acid was added wherever necessary. The strength of the solution was determined by standard methods given in Vogel [11] and Scott [12]. The stock solution was diluted with distilled water to give the desired concentration. Radioisotopes used were supplied by the Board of Radiation and Isotope Technology, B.A.R.C., Trombay.

2.1 Instrumentation

Gamma emitters were counted on a gamma ray spectrometer in conjunction with a 3.5 cm x 3.5 cm flat type NaI(Tl) crystal detector provided with a single channel pulse-height analyser. Beta emitters were counted on an end-window type G.M. Counter in conjunction with a decade scaler, high voltage unit and a timer.

2.2 Extraction Procedure

1.0 mg of Co(II) labelled with ^{60}Co was taken in a separating funnel. 5.0 ml of 1.0% ethanolic solution of 2-mercapto-benzimidazole was added. The pH of the solution was adjusted to 10.0 with ammonia solution (vol = 20 ml). The aqueous phase was equilibrated with 20 ml of nitrobenzene for 5.0 minutes. The phases were allowed to separate and the volume of each phase was found to be equal. A 2-ml aliquot of each phase was taken for counting on a gamma-ray spectrometer at the channel corresponding to 1.332 MeV photopeak of ^{60}Co. The extraction coefficient (E) and percent extraction (%E) were calculated in the usual way.

The stoichiometric ratio of Co(II) to reagent was determined by the method of substoichiometric extraction which was carried out as follows: Increasing amounts of Co(II) labelled with ^{60}Co along with 11.94 mg ethanolic solution of 2-mercapto-benzimidazole was taken in a series of separatory funnels. The aqueous phase was made with distilled water. The pH of the solution was adjusted to 10.0 with ammonia solution. The mixture was then equilibrated for 5.0 minutes with 20 ml of nitrobenzene. A 2-ml aliquot of the organic phase was counted on a gamma ray spectrometer at the channel number corresponding to the 1.332 MeV photopeak of ^{60}Co.

3. Results and Discussion

The effect of pH on the extraction coefficient value of Co(II) with 2-mercapto-benzimidazole into nitrobenzene is as shown in Table 1. The extraction coefficient value indicated a maximum at a pH of 10.0. The value was found to decrease in the more acidic range. The extraction coefficient value reached a maximum value of 107 (mean) for an equilibration time of 5.0 minutes with a reproducibility of 107±5. Determination of time of equilibration revealed that the percentage extraction was better than 98% for an equilibration time of 1.0 to 6.0 minutes.

The effect of various solvents on the E value of Co(II) clearly indicated that nitrobenzene was the best solvent for the extraction. The E values in favour of different

Table 1 Effect of pH on the E Value of Co(II) with 2-mercaptobenzimidazole into Nitrobenzene

Aqueous phase: 1.0 mg of Co(II)+^{60}Co+ 5.0 ml 1.0% ethanolic solution of 2-mercapto-benzimidazole + ammonia + distilled water; Organic phase: 20.0 ml of nitrobenzene; time of equilibration: 5.0 minutes; temperature : 28+2°C.

pH	Extraction coefficient (E)	Percentage extraction (%E)
5.0	0.022	2.15
6.0	0.37	27.01
7.0	12.49	92.48
8.0	68.58	98.56
8.5	78.36	98.73
9.0	89.16	98.89
9.5	97.11	98.98
10.0	107.0	99.07
10.0	104.0	99.05
10.0	112.0	99.11
10.0	102.0	99.02
10.0	109.0	99.09
10.5	107.0	99.07

Note*: Reproducibility: 107.0±5.0

solvents decrease in the following order: Nitrobenzene (107.0) > Chloroform (95.9) > 1,2-dichlorobenzene (73.3) > Toluene (67.8) > Benzene (31.1) > Chlorobenzene (24.9) > Cyclohexane (13.7) > MIBK (10.1) > Carbon tetrachloride (8.7) > Heptane (6.1) > Ethyl acetate (2.3) > Isoamyl acetate (2.3) > Isoamyl alcohol (2.2) > Nitromethane (1.9) > Benzyl alcohol (1.1) > n-butanol (0.9). The solvents ether, ethyl methyl ketone, tertiary butyl alcohol, sec-butyl alcohol and pyridine were found to be miscible with the aqueous phase.

The stoichiometry of the extracted species was determined by the method of substoichiometric extraction and was found to be 1:2. A graph of 2-ml aliquot of the organic phase against the initial concentration of Co(II) showed an initial increase in activity till the ratio of Co(II) concentration to that of 2-mercapto-benzimidazole was 1:2, after which it remained constant indicating that the stoichiometry of the metal to reagent was in the ratio of 1:2 (Fig. 1).

Gamma emitters were counted on a gamma ray The effect of sodium, potassium or ammonium salts on the extraction coefficient value of Co(II) revealed that 100 mg each of thiocyanate, bromide, sulphate, chloride, sulphide and bromate; 50 mg each of thiosulphate, sulphite, borate, arsenate, nitrite, iodide, perchlorate,

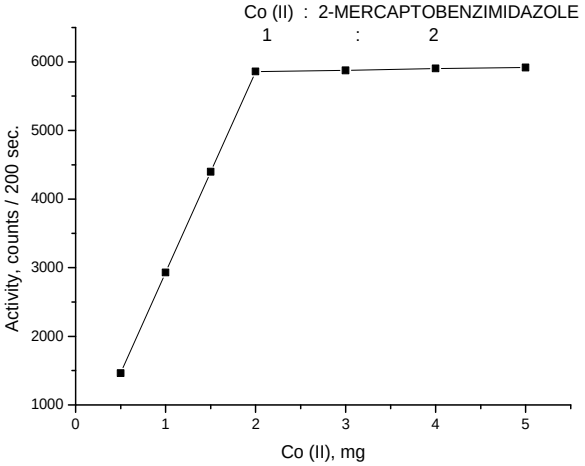

Fig. 1. Reproducibility of substoichiometric extraction of Co (II) with 2-mercaptobenzimidazole into nitrobenzene.

Fig. 1 Reproducibility of Substoichiometric Extraction of Co(II) with 2-Mercapto-Benzimidazole into Nitrobenzene

chromate, fluoride and nitrate and 10 mg each of cyanide and phosphate do not decrease the extraction coefficient value of Co(II). However thiourea, dichromate, tartrate, oxalate, EDTA and citrate were found to decrease the E value of Co(II).

The effect of various elements on the extraction coefficient value (E) of Co(II) was studied with or without carrier. The results are given in Table 2. It can be seen that Br(I), As(III), Ir(IV), As(V), Gd(III), Sr(II), Sn(IV), Sn(II), Sb(V), Eu(III), Cs(I) and Ba(II) were extracted to the extent of less than 1.0%. Sb(III), Re(VII), Mo(VI), Tc(VII), Na(I) and K(I) were extracted between 1.0 to 10.0%. Te(IV) was extracted to more than 31% but it was removed by precipitating it as tellurium metal with SO_2 water. Pd(II) was extracted to more than 85%, but it was removed by precipitation with iodide prior to the extraction of Co(II). Ce(III), Ce(IV) and La(III) were extracted to the extent of 90%, 94% and 96% respectively. The interference was suppressed by masking them with 50 mg of fluoride. Ag(I) was extracted to more than 95%, but it was removed by precipitating it as AgCl prior to the extraction of Co(II).

Table 2 Interference of Other Elements in the Extraction of Co(II) with 2-Mercaptobenzimidazole into Nitrobenzene

Aqueous phase: 1.0 mg of Co(II)+^{60}Co + 5.0 ml 1.0% ethanolic solution of 2-mercapto-benzimidazole + ammonia + distilled water; Organic phase: 20.0 ml of nitrobenzene; pH: 10.0; time of equilibration: 5.0 minutes; temperature: 28+2°C.

Extraction, %	With Carrier	Without Carrier
< 1	As(III), As(V), Ba(II), Cs(I), Eu(III), Gd(III), Ir(IV), Sb(V), Sn(II), Sn(IV), Sr(II), Ce(III)*, Ce(IV)*, La(III)*	As(III), As(V), Ba(II), Br(I), Cs(I), Eu(III), Gd(III), Ir(IV), Sb(V), Sn(II), Sn(IV), Sr(II), Ce(III)*, Ce(IV)*, La(III)*
1 to 10	K(I), Mo(VI), Na(I), Re(VII), Sb(III), Ag(I)*, Pd(II)*, Te(IV)*	K(I), Mo(VI), Na(I), Re(VII), Sb(III), Tc(VII), Ag(I)*, Pd(II)*, Te(IV)*
> 10	Ag(I), Ce(III), Ce(IV), La(III), Pd(II), Te(IV)	Ag(I), Ce(III), Ce(IV), La(III), Pd(II), Te(IV)

Note: * After appropriate treatment.

4. Conclusion

From the above results it can be concluded that the readiochemical separation method developed for the extraction of Co(II) from other elements is rapid and highly selective.

Acknowledgement

Sincere thanks are due to Prof. Dr. (Mrs.) Z. R. Turel for the valuable guidance and support given to me during the research investigation.

Sincere thanks are also due to Kishinchand Chellaram College, H.S.N.C. Board, Bombay, for all the help and support extended to me during the research investigation. I also thank all the staff members for their spontaneous help and cooperation.

Thanks are due to University of Bombay, Bombay for awarding a Research Project to me. I also thank the Scientists and Scientific Officers of Board of Radiation and Isotope Technology, B.A.R.C., Trombay, for the timely supply of radioisotopes required for research investigation.

Thanks are due to Wollo University, Dessie Ethiopia University for all the support and help given to me in publishing the research paper.

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