



SORPTION OF SOME CATIONS FROM AQUEOUS AMMONIUM MALEATE SOLUTIONS BY AMBERLITE IRA-400

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ABSTRACT

The anion exchange equilibrium distribution coefficients (K_d) with anion exchanger, the Amberlite IRA-400 Cl⁻ form, has been determined for 10 metal ions in media consisting varying concentrations (0.8, 0.9, 1.1, 1.3, 1.5, 1.6, 1.8, 1.9, 2.1, 2.2, 2.4, 2.6, 2.8, 3.0 M) of ammonium maleate by the batch equilibration technique. The capacity and the moisture content were 2.75 meq/g and 42-48% respectively. The deviation in sorption behaviour of cations has been discussed on the basis of complexation with ammonium maleate. The relative separation factors of binary pairs of metal ion mixtures have been calculated as a function of ammonium maleate concentrations on the basis of their distribution coefficients. The separation possibilities for a number of binary, ternary and quaternary mixtures are worked out.

KEYWORDS: Ion exchange, Amberlite IRA-400, metal ions, ammonium maleate, distribution coefficient, separation.

INTRODUCTION

Maleic acid is widely used in medicine, in the preparation of drugs, in agricultural field (as a standard natural organic matter, i.e., biodegradable) as the plant growth regulators, in food industry, and in plastic production (Bychkova *et al.*, 2008). The potential applications of the maleic acid are mostly as its metal-complex species in several natural sources. A number of metal-maleate complexation activity behavioural characteristics were described in literature (Bychkova *et al.*, 2008, Helfferich, 1962, Topp & Davis, 1940, Martell, 1977). Apart the acids, a number of their salts especially the ammonium salts have been used to work out the eluting tendencies for the separation of metal ion species on ion-exchange resins (Chakravorty, *et al.*, 1977). Paul J. Killos (1976) studied and investigated the process of purification of DL-malic acid from its mixture containing maleic and fumaric acids (the cis- and trans-isomers of 2-butenedioic acid) using a strongly basic anion exchange resin (Amberlite IRA-400, IRA-401, IRA-402, IRA-410, and IRA-411). Maleic acid, like a number of low molecular weight dicarboxylic acids (oxalic, malonic, succinic, fumaric (its trans-isomer), and methylmalonic, etc.), secreted by plant roots plays an important role in a variety of processes at the soil-root interface. However, its concentration in root exudates are normally lower than the level detectable by aqueous based extractions. But with ion exchange resin, Szmigielska *et al.* (1995) successfully extracted and able to obtain its concentrates from the rhizosphere in hydroponic growth solution the eluent used was 0.5 M HCl (Szmigielska *et al.*, 1995, Qian, 2002). The determination of maleic acid in groundwater and the soil has been reported by Dietz and Singley (1994). Maleic acid is highly toxic and hazardous to animals and plants but the ammonium salts appears relatively moderate (Mallinckrodt, 2009). The ion-exclusion chromatographic

method has been developed to estimate maleic acid in the groundwater and soil. The method has utilized the anion

exchange cartridge (Dietz, 1994). Our laboratories have reported the cation exchange characteristic studies of several metal ions in HNO₃-CH₃COONH₄ media on Amberlite IR-120 (Mahan *et al.*, 1971). A comprehensive anion-exchange characteristic of some metal ions were carried out and presented considering the separation potentialities and the complexation aspects as distribution gradient in the ammonium maleate media.

MATERIALS and METHODS

The studies include the effect of 9 cations in mixtures 14 different concentrations of ammonium maleate and 0.1 M hydrochloric acid.

Ion exchange resin

Amberlite IRA-400 (20-50 mesh, 8-10% cross-linking in chloride-form (Rohm and Hass, Company, Analytical Grade) was pretreated in a column with 0.02 M EDTA solution (pH=8-10), and then with 2 M HCl. The final washing was done with distilled water until neutral to litmus. The resin, air-dried by suction on a sintered bed, was stored, and the capacity and the moisture content were determined by usual methods. These were 2.75 milliequivalents (per g of air-dried resin) and 48% of resin, respectively. The swelling factor () of the resin has also been determined by the method developed by Dey *et al* and was 0.990 (Mahan, 1973).

Metal solution

Stock solutions (0.05 — 0.10 M) of the various metal chlorides were prepared in 0.1 M HCl from freshly precipitated hydroxides or chlorides. The samples were either of Glaxo Lab (Fine Chemical/AR or Loba Chemie and other make and the prepared solutions were subsequently standardized (West, 1960).

Ammonium maleate solution

An aqueous solution of 4.0 M ammonium maleate, [(NH₄)₂Male.] was prepared by neutralizing the maleic acid (LOBA Chemie) with liquor ammonia Qualigens and then it was standardized by the indirect determination of

ammonia (Vogel, 2002).

Buffer solution

For EDTA titrations the buffer solutions of pH = 10 (NH₄Cl/ammonia) and pH = 5 (NaOAc/HCl) were used. Other desired pH ranges were obtained by maintaining the pH of the solution by the addition of suitable quantities of acid or alkali and with the help of a century CP. 901 pH meter. pH indicator papers (B.D.H.) were also employed in some cases during titrations, as when necessary.

Equilibration of the systems and determination of distribution coefficients (K_d)

$$K_d = \frac{\text{meq of the metal ion / g of resin (air - dried)}}{\text{meq of the metal ion / mL of solution (equilibrated)}}$$

The experiments were performed at a metal concentration ratio of total amount of cation (as anionic complex species) to total resin capacity ≈ 0.4 , using 0.1 M HCl with varying ammonium maleate concentrations (i.e., 0.8, 0.9, 1.1, 1.3, 1.5, 1.6, 1.8, 1.9, 2.1, 2.2, 2.4, 2.6, 2.8 and 3.0

A wrist-motion Microid flask shaker (Griffin & George) was used for shaking. Solutions were shaken in glass stoppered 100 mL flasks. The batch equilibration method was employed. The weighed amounts of air-dried resin (1.000 g each) were added to the mixture solution containing the metal ion, the acid (HCl), and ammonium maleate, and shaken for 12 hr (it was ascertained previously that this time was adequate to attain the equilibrium). The resin was filtered and the metal ion was determined were given by using the relation.

M), and at a room temperature of $30 \pm 2^\circ\text{C}$. The total volume of aqueous phase was 25 mL. The results obtained are given in Table 1 & 2. All experiments were carried in duplicate and average values taken.

TABLE 1: Metal species distribution coefficients (K_d) values in aqueous (NH₄)₂Male. IRA-400 systems

Cation	[Ammonium Maleate] M						
	0.8	0.9	1.1	1.3	1.5	1.6	1.8
Mg(II)	NS	NS	NS	NS	NS	NS	NS
Ca(II)	20.5	22.2	23.1	25.0	27.1	28.2	30.6
Cr(III)	9.1	11.5	15.4	19.4	24.6	28.8	37.4
Fe(II)	10.7	12.3	13.5	13.5	13.5	16.7	25.0
Fe(III)	7.0	9.7	13.8	16.7	18.9	26.3	30.6
Co(II)	30.6	32.5	34.5	35.2	37.5	39.1	42.6
Ni(II)	4.4	NS	NS	NS	NS	NS	NS
ZrO(II)	13.5	15.3	18.1	23.1	24.0	27.1	30.6
Ba(II)	6.3	8.3	10.7	11.8	13.5	16.7	18.9
Ce(III)	4.8	6.4	6.4	8.3	10.5	11.6	13.8

NS: Shows no sorption

TABLE 2: Metal species distribution coefficients (K_d) values in aqueous (NH₄)₂Male. IRA-400 system

Cation	[Ammonium maleate] M						
	1.9	2.1	2.2	2.4	2.6	2.8	3.0
Mg(II)	NS	NS	NS	NS	NS	NS	NS
Ca(II)	31.8	33.1	34.5	37.5	37.5	37.5	39.1
Cr(III)	51.8	60.5	77.2	100.3	126.5	166.5	216.3
Fe(II)	30.6	34.5	37.5	44.4	58.3	88.6	122.1
Fe(III)	34.5	49.1	58.3	73.0	86.1	94.0	101.3
Co(II)	48.5	54.4	61.2	67.6	75.0	88.6	94.0
Ni(II)	NS	NS	NS	NS	NS	NS	NS
ZrO(II)	36.0	39.1	42.6	48.5	58.3	100.0	106.6
Ba(II)	23.1	24.0	25.0	27.1	NS	NS	NS
Ce(III)	15.6	17.7	20.0	32.5	52.5	73.0	183.3

NS: Shows no sorption

RESULTS & DISCUSSION

Experimental errors in Table I & II are worked out to be less than about 2-5% of the results for the investigated K_d values. The sources of errors are inhomogeneity of the resin, water content of the resin (the air-dried resin is a strong desiccant), and analytical errors. The K_d values are determined at relatively higher resin loadings and,

therefore, more practical results are obtained compare to at lower concentrations, because the errors due to secondary effects such as simple adsorption by the walls of the vessels are not effective. Fig. 1 illustrates the sorption characteristics in which the distribution coefficient (K_d) values are plotted against the concentrations of ammonium maleate.

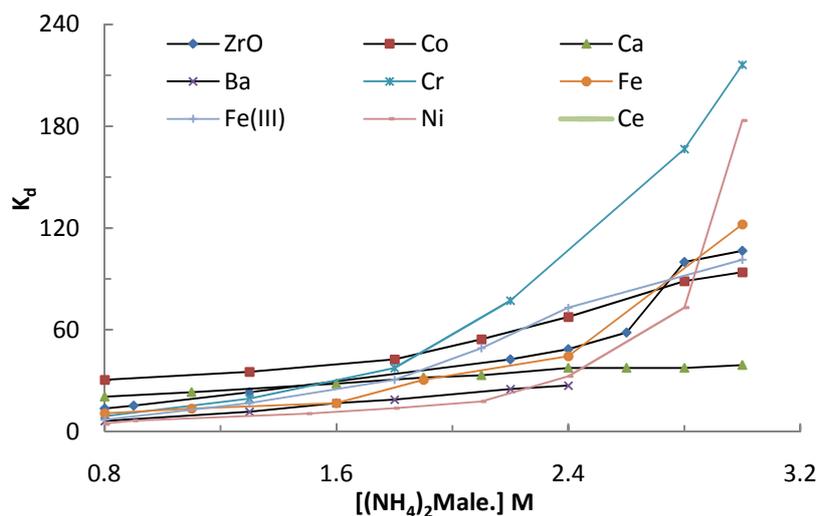
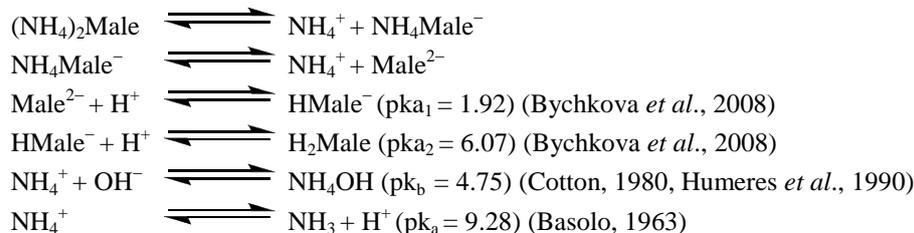


FIGURE 1- K_d vs $[(NH_4)_2Malea]$ M

Ammonium maleate is a salt of weak acid and weak base, therefore, its dissociation is dependent on the pH of the aqueous phase and the buffering capacity generated by interactions of H^+ , NH_4^+ , Cl^- , and maleate, resulting in the

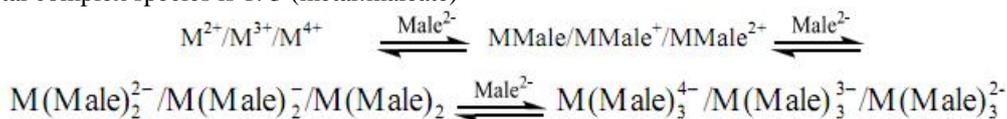


This causes increases in the pH of the aqueous phase. Consequently the formation of higher metal-maleate complex species, *i.e.*, the concentration of anionic complexes gradually increases in comparison to lower complex species (cationic or neutral complexes) at higher ammonium maleate medium. Therefore, in most of the cases sorption of the metal ion increases with increasing ammonium maleate concentration. Mg(II) shows no sorption at all the investigated range of ammonium maleate. Only at 0.8 M $(NH_4)_2Male$ concentration Ni(II) shows a very-very little sorption ($K_d = 4.4$) while in rest of the concentration range no sorption was observed by the nickel. No sorption was also observed by barium(II) between 2.6-3.0 M $(NH_4)_2Male$ concentration ranges. The media investigated here are worked out as buffer solutions, so the availability of maleate or protonated maleate ions depends on the pH of the solution or on the dissociation of the resulting maleic acid. Along with this, the sorption equilibria are also governed by the exchange affinities of Cl^- ion, maleate and protonated maleate anions and the metal-maleate anionic species existing in the systems. The competition of various anionic species ($HOOC.C=CH.COO^-$, $NH_4OOC.CH=CH.COO^-$, $^-OOC.CH=CH.COO^-$) and different negatively charged metal-maleate complexes (including protonated species) with chloride of the resin plays the key role for the sorption of metals. It is also explained by taking into consideration that the high charge and high molecular weight species have high exchange affinities because of their preference by the exchanger (Helfferich, 1962). It is

formation of maleic acid the protonated ammonium maleate ($NH_4OOC.CH=CH.COOH$), and ammonium chloride, as well as ammonium hydroxide and ammonia.

also proposed that the exchange mechanism is occurring in two steps. First, the protonated maleate or ammonium maleate anion is exchanging by replacing the chloride from the resin, and then the metal ion, which has strong affinity for maleate, associates by replacing proton or ammonium of the maleate site of the resin. The interaction of chloride along the maleate with the metal ion may also be possible, *i.e.*, mixed ligand complex formation and the negative charge on metal complex species increases and thereby facilitating the metal sorption. The hydrochloric acid was used to suppress the hydrolysis of certain metal ions in the solution. Dimethylsulphoxide (DMSO) (ϵ , dielectric constant, 46.6) was used to observe the effect of dielectric behaviours on these investigations. Because it slightly decreases the polarization or dielectric nature of solution phase as comparing the Zn(II) and Cu(II) studies in pure aqueous media with the DMSO mixed media. The general trend of decreased K_d value in presence of DMSO adversely affects the formation of anionic metal-maleate complex species due to the decrease in dielectric constant of the solution phase by DMSO. The resin swelling plays an important role for the exchanging mechanism and subsequent sorption or loading of the respective species on to the resin. The overall availability of maleate ligand anion for the maleate complex (anionic/negatively charged) species formation decreases due to suppressed dissociation and buffering action on the formed maleic acid. Various equilibria existing in solution are coparticipating with the ion exchange partition equilibrium, and may be given as when considering that

the highest metal complex species is 1: 3 (metal:maleate)



These metal complex species dominatedly contributing to the overall solution and two-phase ion exchange equilibria which are in simultaneous existence, other maleate co-anions, i.e., the protonated and the ammoniated maleate, may possibly, are also taking part in complexation processes, and consequently affecting the K_d values of the metal ion species. The overall general observed trend of K_d values at 3.0 M $[(\text{NH}_4)_2\text{Male}]$ is, $\text{Cr} > \text{Ce} > \text{Fe(II)} > \text{ZrO(II)} \geq \text{Fe(III)} > \text{Co} > \text{Ca}$. A large number of binary-separation possibilities are worked out, and are reported in

Table 3 and 4 and the practically feasible cation pairs in the particular ammonium maleate concentration are given in chart I. Some of the Clearcut binary separations are presented in the chart II. The chart III and Chart IV have listed the ternary separation combination of cations, and the Clearcut schematic workable procedure, respectively. Similarly, the charts V and VI have included the quarternary mixtures, and their possible Clearcut procedures, respectively.

TABLE 3: Separation Factors () $\left[\frac{\text{Metal}}{\text{Othermetal}} / \frac{\text{Othermetal}}{\text{Metal}} \right]$

Separated cation pairs	[(NH ₄) ₂ Male] M						
	0.8	0.9	1.1	1.3	1.5	1.6	1.8
Cu-Al	5.4	5.3	5.8	6.2	7.4	7.6	7.5
La-Al	—	—	—	—	—	—	5.5
Pb-Al	—	—	—	5.0	5.0	5.0	5.2
Cu-Mn	—	—	—	—	5.7	5.9	5.6
Cu-Fe(II)	8.8	8.1	8.5	9.8	12.4	11.0	7.7
Zn-Fe(II)	5.4	5.0	—	—	5.8	5.0	—
Cd-Fe(II)	7.0	6.3	8.8	6.6	8.0	6.2	—
La-Fe(II)	7.0	7.2	9.9	7.4	7.9	7.3	5.4
Pb-Fe(II)	7.8	7.2	7.0	7.9	10.3	7.0	5.3
Cu-Fe(III)	13.4	10.3	8.3	7.9	8.9	7.0	6.3
Zn-Fe(III)	8.3	6.3	4.6	—	—	—	—
Cd-Fe(III)	10.7	7.9	5.9	5.3	5.0	—	—
La-Fe(III)	10.7	8.5	6.2	5.4	—	—	—
Pb-Fe(III)	12.0	9.1	6.8	6.3	6.0	—	—
Cu-Ni	21.5	—	—	—	—	—	—
Zn-Ni	13.3	—	—	—	—	—	—
Cd-Ni	17.0	—	—	—	—	—	—
La-Ni	17.2	—	—	—	—	—	—
Hg-Ni	5.4	—	—	—	—	—	—
Pb-Ni	19.0	—	—	—	—	—	—
Cu-ZrO	7.0	6.5	6.3	5.7	7.0	6.8	6.3
Cu-Ba	15.0	12.0	10.6	11.2	12.4	11.2	10.2
Cu-Hg	—	—	—	—	5.4	5.6	5.5
Cu-Th	43.3	32.3	21.5	18.5	15.6	11.9	9.0
Cd-ZrO	5.6	5.0	—	—	—	—	—
La-ZrO	5.6	5.4	—	—	—	—	—
Pb-ZrO	6.2	5.8	5.2	—	—	—	—
Zn-Ba	9.0	7.3	6.0	5.6	5.2	—	—
La-Ba	12.0	9.9	8.0	7.6	6.8	6.0	5.4
Pb-Ba	13.3	10.6	8.7	9.0	8.5	7.0	6.9
Cd-Th	34.6	24.8	15.4	12.5	8.8	6.8	5.5
La-Th	34.9	26.6	16.2	12.7	8.6	6.5	4.8
Pb-Th	38.5	28.6	17.7	15.0	10.7	7.7	6.2

TABLE 4: Separation Factors () $\left[\frac{\text{Metal}}{\text{Othermetal}} / \frac{\text{Othermetal}}{\text{Metal}} \right]$

Separated cation pairs	[(NH ₄) ₂ Male] M						
	1.9	2.1	2.2	2.4	2.6	2.8	3.0
Cu-Al	7.7	9.3	9.4	10.5	14.2	12.9	15.8
Cd-Al	5.0	5.2	5.3	6.1	7.1	6.9	7.4
La-Al	6.3	5.0	5.6	5.8	7.5	10.6	16.9
Pb-Al	6.1	7.5	7.4	9.1	9.9	10.7	12.5
Th-Al	—	—	—	—	11.7	18.2	22.7
La-Ca	—	—	5.0	—	6.7	10.4	16.4
Pb-Ca	5.0	6.1	6.5	7.7	8.9	10.4	12.7
Th-Ca	—	—	—	—	10.4	17.9	22.1
Cu-Mn	5.4	6.4	7.3	8.4	12.1	12.1	15.3
La-Mn	—	—	—	—	6.4	10.0	16.4
Pb-Mn	—	5.2	5.8	7.4	8.5	10.0	12.1
Th-Mn	—	—	—	—	10.0	17.1	22.2
Cu-Fe(II)	6.6	7.3	7.7	7.5	8.1	5.4	5.0
La-Fe(II)	—	—	—	—	—	—	5.3
Pb-Fe(II)	5.3	5.3	5.0	—	5.7	—	—
Th-Fe(II)	—	—	—	—	6.7	7.6	7.1
Cu-Fe(III)	5.8	5.1	—	—	5.0	5.0	5.9
La-Fe(III)	—	—	—	—	—	—	6.3
Th-Fe(III)	—	—	—	—	—	7.1	8.5
Cu-Co	—	—	—	—	6.3	5.4	6.4
Cu-Zn	—	—	—	—	6.3	6.3	8.0
Cu-ZrO	5.6	6.4	6.8	6.8	8.1	—	5.6
Cu-Ba	8.8	10.5	11.5	12.3	—	—	—
Cu-Hg	5.6	6.4	6.7	7.3	9.3	8.5	10.2
Cu-Th	5.9	6.2	—	—	—	—	—
La-Zn	—	—	—	—	—	5.2	8.6
Pb-Zn	—	—	—	—	—	5.2	6.3
Th-Zn	—	—	—	—	5.2	8.9	11.6
La-ZrO	—	—	—	—	—	—	6.0
Pb-ZrO	—	5.2	5.3	5.9	5.7	—	5.6
Th-ZrO	—	—	—	—	6.7	6.7	8.1
La-Ba	5.2	5.6	6.9	6.8	—	—	—
Ce(III)-Ba	—	—	—	5.0	—	—	—
Pb-Ba	6.9	8.4	9.0	10.6	—	—	—

Chart I- Binary Separations

1. Th(2.2)/Ni(4.4)/Ce(III)(4.8)/Ba(6.3)/Fe(III)(7.0)—Cu(94.0)/Pb(83.7)/La(75.8)/Zn(58.3) in 0.8 M ammonium maleate.
2. Th(2.2)/Ce(III)(6.4)/Ba(8.3)—Cu(100.0)/Pb(88.6)/La(82.5)/Cd(77.0) in 0.9 M ammonium maleate.
3. Ce(III)(6.4)/Th(5.3)—Cu(113.9)/Pb(94.0)/La(86.1)/Cd(81.4) in 1.1 M ammonium maleate.
4. Th(7.1)/Ce(III)(8.3)—Cu(131.6)/Pb(106.6)/La(90.3)/Cd(88.6) in 1.3 M ammonium maleate.
5. Ce(III)(10.5)/Th(10.7)—Cu(167.3)/Pb(113.9)/Cd(94.0)/La(91.3) in 1.5 M ammonium maleate.
6. Ce(III)(11.6)—Cu(183.9)/Pb(117.9)/Cd(104.0)/La(99.4) in 1.6 M ammonium maleate.
7. Ce(III)(13.8)/Ba(18.9)—Cu(192.3)/Pb(131.3) in 1.8 M ammonium maleate.
8. Ce(III)(15.6)—Cu(202.3)/Pb(161.3)/Cd(131.3) in 1.9 M ammonium maleate.
9. Ce(III)(17.7)/Ba(24.0)—Cu(252.0)/Pb(202.3)/Cd(141.7) in 2.1 ammonium maleate.
10. Ba(25.0)/Ce(III)(20.0)—Cu(287.5)/Pb(225.0) in 2.2 M ammonium maleate.
11. Al(31.5)/Ce(32.5)—Cu(332.1)/Pb(287.5) in 2.4 M ammonium maleate.
12. Al(33.5)/Ca(37.5)—Cu(475.0)/Th(391.6)/Pb(332.1) in 2.6 M ammonium maleate.
13. Al(36.7)/Ca(37.5)/Mn(39.1)—Th(669.4)/Cu(475.0)/La(391.6)/Pb(391.7) in 2.8 M ammonium maleate.
14. Al(37.9)/Mn(391.1)/Ca(39.1)—Th(867.9)/La(641.6)/Cu(600.0)/Cd(281.5) in 3.0 M ammonium maleate.

Chart II- Clearcut Binary Separations

Separated Cations (NH₄)₂Male. Medium
Cu—Th in 0.8 M

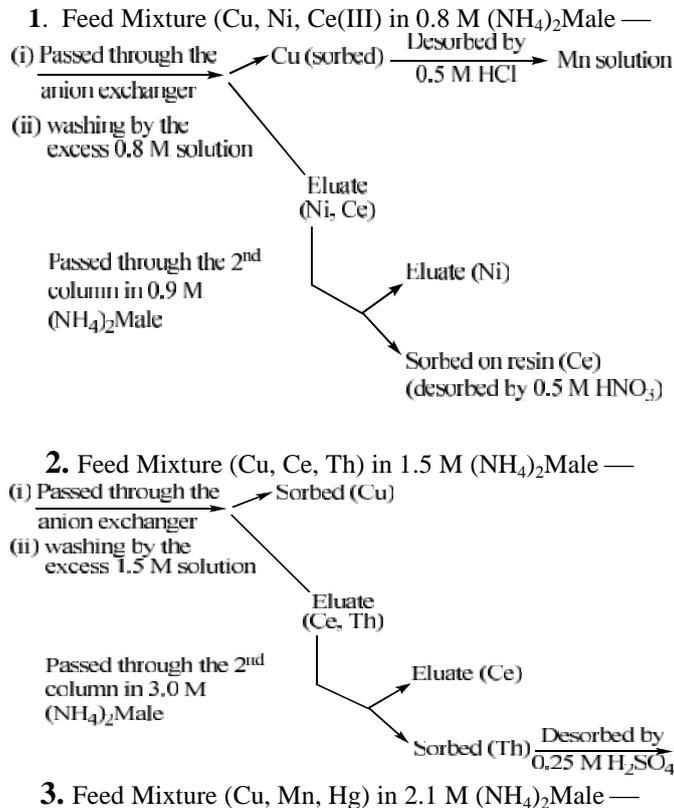
Sorption of some cations from aqueous ammonium maleate solutions

Cu—Th	in 0.9 M
Cu—Th	in 1.1 M
Cu—Th	in 1.3 M
Cu—Ce(III) or Th	in 1.5 M
Cu—Ce(III)	in 1.6 M
Cu—Ce(III)	in 1.8 M
Cu—Ce(III)	in 2.1 M
Cu—Ce(III)	in 2.2 M
Cu—Ce(III)	in 2.4 M
Cu—Ce(III)	in 2.6 M
Cu—Al or Ca	in 2.8 M
Th—Al or Ca	in 3.0 M

Chart III- Ternary Separations

1. Th(2.2)—Hg(24.0)—Cu(94.0) in 0.8 M ammonium maleate.
2. Th(3.1)—Al(18.9)/ZrO(15.3)—Cu(100.0) in 0.9 M ammonium maleate.
3. Ce(6.4)/Th(5.3)—Ca(23.1)/Mn(26.0)—Cu(113.9)/Pb(94.0) in 1.1 M ammonium maleate.
4. Th(7.1)/Ce(III)(8.3)—Hg(28.6)/Mn(28.6)—Cu(131.3) in 1.3 M ammonium maleate.
5. Th(10.7)/Ce(III)(10.5)/Fe(13.5)—Cr(III)(24.6)/Al(22.6)—Cu(167.3)/Pb(113.9) in 1.5 M ammonium maleate.
6. Ce(III)(11.6)/Ba(16.6)/Fe(16.6)—Co(39.1)—Cu(183.3) in 1.6 M ammonium maleate.
7. Ce(III)(13.8)—Hg(34.5)/Mn(34.5)—Cu(192.3)/Pb(131.3) in 1.8 ammonium maleate.
8. Ce(III)(15.6)—Cr(III)(51.8)—Cu(202.3) in 1.9 M ammonium maleate.
9. Al(27.6)/Ce(III)(17.7)—Co(54.4)/Fe(III)(49.1)—Cu(252.0)/Pb(202.3) in 2.1 M ammonium maleate.
10. Ce(III)(20.0)—Co(61.2)/Zn(75.0)—Cu(287.5) in 2.2 M ammonium maleate.
11. Al(31.5)/Ce(III)(32.5)—Cr(III)(100.3) in 2.4 M ammonium maleate.
12. Al(33.5)/Ca(37.5)/Mn(39.1)—Cr(III)(126.5)—Cu(475.0) in 2.6 M ammonium maleate.
13. Ca(37.5)/Al(36.7)—Cr(III)(166.5)—Cu(475.0)/Pb(391.0)/La(391.6) in 2.8 M ammonium maleate.
14. Al(37.9)/Ca(39.1)/Mn(39.1)—ZrO(106.6)—Th(867.9)/La(641.6) in 3.0 M ammonium maleate.

Chart IV- Clearcut Ternary Separations



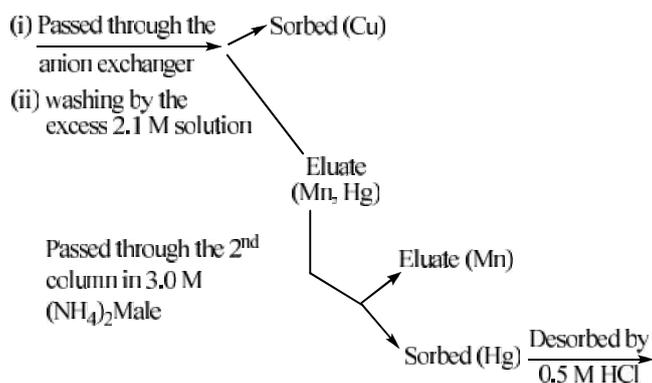


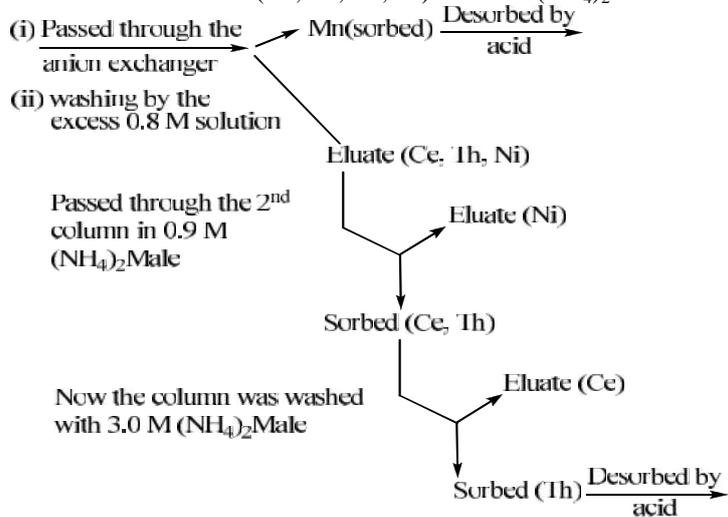
Chart V- Quarternary Separations

Al(36.7)/Ca(37.5)—ZrO(100.0)—Th(669.4)—Cu(475.0) in 2.8 M ammonium maleate.

Ca(39.1)/Al(37.9)—Co(94.0)/ZrO(106.6)—Cd(281.5)—Th(867.9) in 3.0 M ammonium maleate

Chart VI- Clearcut Quarternary Separations

1. Feed Mixture (Cu, Ce, Th, Ni) in 0.8 M (NH₄)₂Male —



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