Electronic Supplementary Material (ESI) for Analytical Methods.

Liquid-liquid extraction and separation of lead(II) by using N-*n*–octylcyclohexylamine as an extractant : Analysis of real samples

Supplementary Information

Table 1. Extraction of lead(II) as a function of acid concentration

Acid,	HCl		HNO_3		H_2SO_4		$HClO_4$		CH_3COOH	
M	% E	D	% E	D	% E	D	% E	D	% E	D
0.1	00	00	00	00	00	00	00	00	00	00
0.5	30.6	1.10	14.2	0.14	14.1	0.41	18.8	0.57	17.0	0.51
1.0	64.9	17.66	36.6	2.56	23.6	0.77	32.6	1.20	29.0	1.13
1.5	81.8	21.08	52.6	2.77	26.4	0.88	31.7	1.16	34.2	1.19
2.0	91.2	36.56	56.4	3.23	27.9	1.24	27.4	0.94	36.0	1.23
2.5	96.2	48.52	61.3	3.95	29.7	1.05	26.0	0.87	37.8	1.51
3.0	99.6	622.5	63.9	4.55	33.9	1.28	26.2	0.98	43.0	1.88
3.5	99.0	247.5	66.0	4.85	35.3	1.35	23.9	0.78	44.1	1.97
4.0	99.4*	414.1	68.5	5.43	36.0	1.40	21.0	0.66	43.3	1.90
4.5	99.2	310.0	71.0	6.12	38.0	1.53	20.4	0.64	42.5	1.84
5.0	99.7	830.8	74.8	7.42	38.8	1.58	19.8	0.61	42.1	1.81
6.0	94.5	42.95	99.0	247.5	33.0	1.23	16.4	0.49	34.0	1.28
7.0	80.0	10.0	99.7	830.8	30.9	1.11	18.2	0.54	32.4	1.19
8.0	61.5	3.99	98.2	136.4	23.6	0.76	16.8	0.50	27.9	1.24
9.0	57.2	3.34	63.3	4.56	18.8	0.57	13.6	0.39	26.7	0.91
10	49.0	2.40	44.5	2.44	18.5	0.56	11.2	0.31	23.6	0.77

*****Acid concentration recommended for extraction procedure

Pb(II) = 100 µg, N-*n*-OCA = 0.04M in xylene, Equilibrium time= 3 min, Aqueous: organic volume ratio = 2.5:1, Strippant = 0.5M HNO_3 (3x10mL)

Table 2. Influence of amine concentration on extraction of lead(II)

N-*n*-octylcyclohexylamine, M	Percentage extraction, (%E)	Distribution Ratio, (D)
0.001	9.12	0.64
0.002	16.6	0.67
0.003	21.2	0.76
0.004	24.8	0.82
0.005	27.9	0.96
0.006	31.4	0.98
0.007	35.6	1.05
0.008	39.2	1.11
0.009	43.8	1.14
0.01	47.6	1.18
0.015	64.2	2.98
0.02	78.8	9.29
0.025	98.0	224.77
0.03	99.4	247.50
0.035	99.5	224.77
0.04*	99.4	310.00
0.045	99.6	622.50
0.05	99.1	164.16
0.055	99.0	176.07
0.06	93.4	21.77
0.07	81.3	12.20
0.08	68.5	4.985
0.09	59.4	3.65
0.1	54.5	2.99

*recommended for extraction procedure

Pb(II) = 100 µg, Hydrochloric acid = 4 M, Equilibrium time= 3 min, Aqueous: organic volume ratio = 2.5:1, Strippant = 0.5M HNO_3 (3x10mL), Solvent= xylene

Table 3. Effect of shaking time on N-*n*-OCA

Time, min	Percentage extraction, %E	Distribution ratio, D
0.25	6.3	0.16
0.5	12.7	0.36
0.75	27.3	0.93
1	53.9	2.92
2	82.4	11.70
3*	99.1	275.27
4	99.7	830.83
5	99.0	247.50
6	99.2	310.00
7	98.5	42.95
8	95.8	21.72
9	92.1	13.32
10	88.6	8.36
15	84.3	6.86
30	82.6	6.65

* recommended for extraction procedure

Pb(II) = 100 µg, Hydrochloric acid = 4 M, Aqueous: organic volume ratio = 2.5:1, Strippant = 0.5M HNO_3 (3x10mL), Solvent= xylene, N-*n*-OCA = 0.04M in xylene

Table 4. Extraction of lead(II) as an aqueous: organic volume ratio with N-*n*-OCA

Volume of aqueous phase, mL	Volume of organic phase, mL	Aqueous to organic volume ratio	Percentage extraction,(%E)	Distribution ratio, D
10	10	1:1	99.2	310.00
20	10	2:1	99.0	247.50
25*	10	2.5:1	99.5	497.5
50	10	5:1	99.3	354.64
60	10	6:1	99.1	275.27
75	10	7.5:1	82.1	11.46
100	10	10:1	74.8	7.42
125	10	12.5:1	69.7	5.75
150	10	15:1	53.9	2.92
200	10	20:1	36.3	1.42
250	10	25:1	31.4	1.14

***** recommended for extraction procedure.

Pb(II) = 100 µg, Hydrochloric acid = 4 M, N-*n*-OCA = 0.04M in xylene, Equilibrium time= 3 min, Strippant = 0.5 M HNO_3 (3x10mL)

Table 5. Loading capacity of lead(II) on N-*n*-octylcyclohexylamine

Pb(II) in µg	Percentage extraction , (% E)	Distribution ratio, (D)
50	99.4	414.16
100*	99.6	622.50
150	98.8	205.83
200	99.0	247.50
250	99.2	310.00
300	98.9	224.77
350	81.4	10.91
400	69.6	5.72
500	57.1	3.32
600	51.4	2.64
700	46.7	2.19
800	44.9	2.03
900	37.0	1.46
1000	35.6	1.38

*Pb(II) recommanded for extraction procedure

Hydrochloric acid = 4 M, N-*n*-OCA = 0.04M in xylene, Equilibrium time= 3 min, Strippant = 0.5M HNO_3 (3x10mL), Diluent = xylene, Aqueous: organic volume ratio = 2.5:1

Supplementary Information

Supplimentry informaction of figture captions

Fig 1. TLC

Fig 2. NMR Spectrum of N-*n*-octylcyclohexylamine

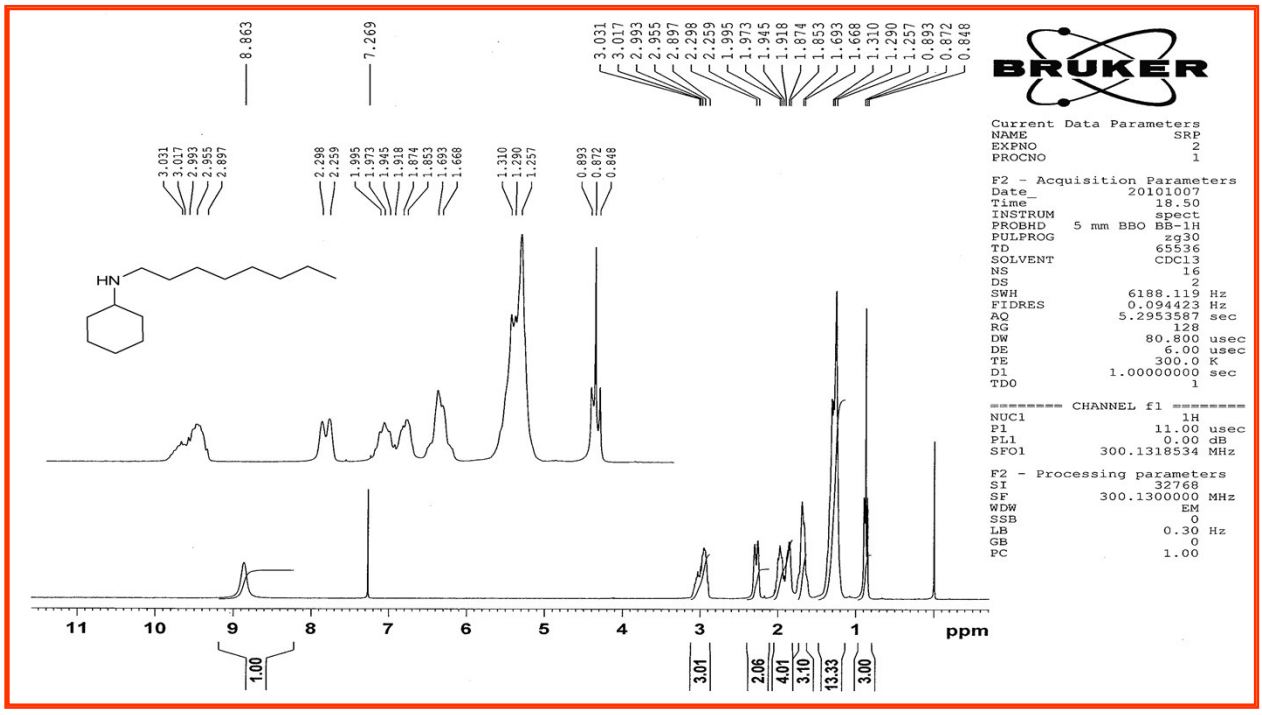
Fig 3. GC- MS data of the N-*n*-octylcyclohexylamine

Fig 4. UV-VIS Spetra

Fig 5. Effect of solvent

Figures:

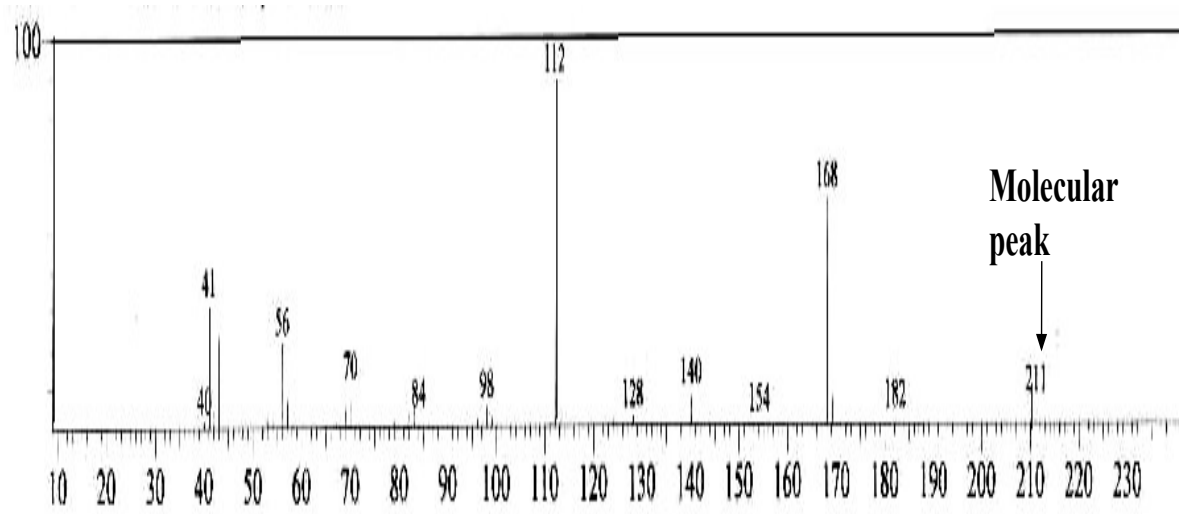
Fig. 1



NMR Spectrum of N-*n*-octylcyclohexylamine.

The ^{1}H NMR spectrum of N-*n*-octylcyclohexylamine exhibited a triplet at δ 0.893 due to methyl proton of n-octyl side chain. The singlet appeared at δ 8.86 confirmed the presence of -NH proton. The presence of twelve methylene group protons appeared as multiplet in the range of δ 1.25 - 3.03.

Fig. 2



GC- MS data of the N-*n*-octylcyclohexylamine

Finally the structure of the reagent was confirmed by GC-MS analysis which is in good agreement with the proposed structure. Molecular peak at 211 clearly shows that formation of desired product by condensation reaction at room temperature.

Fig. 3

Fig. 4

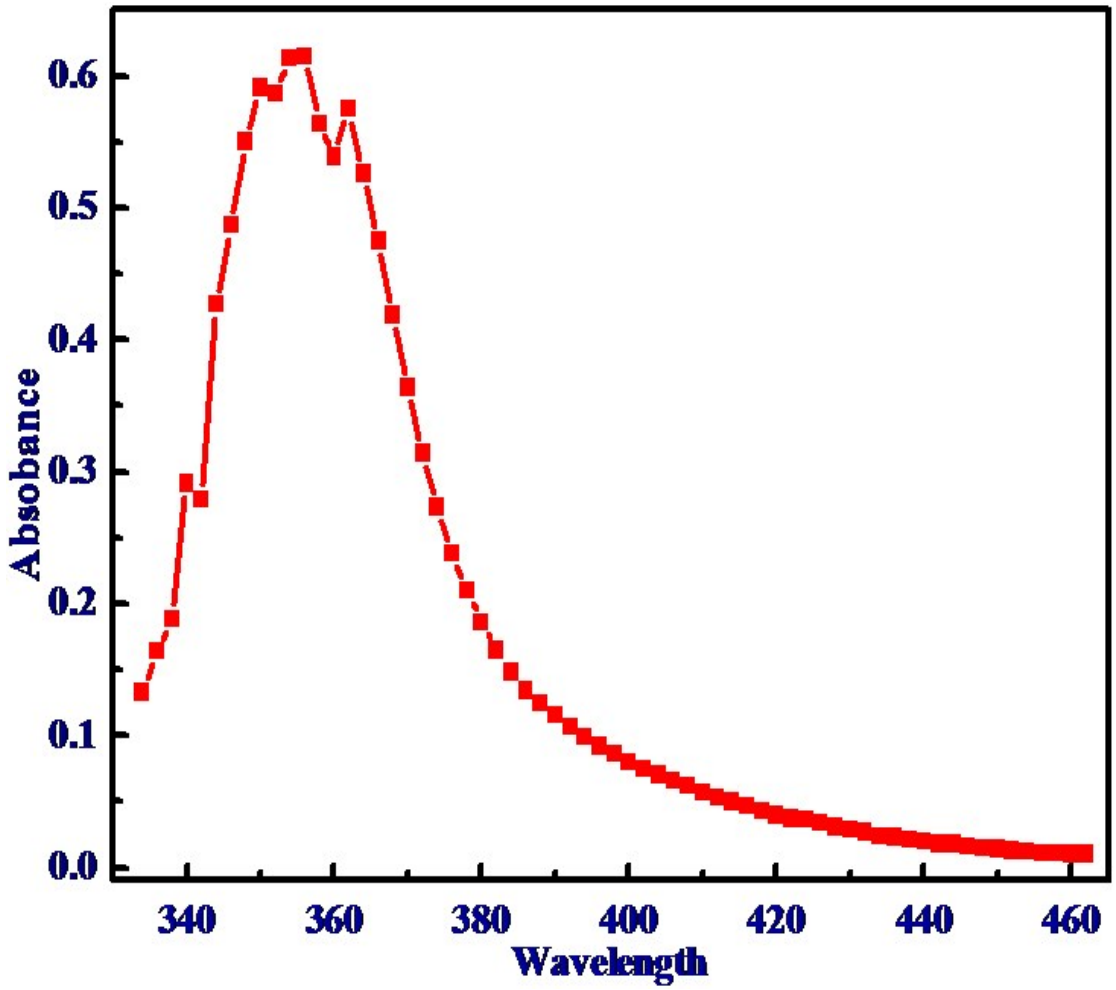
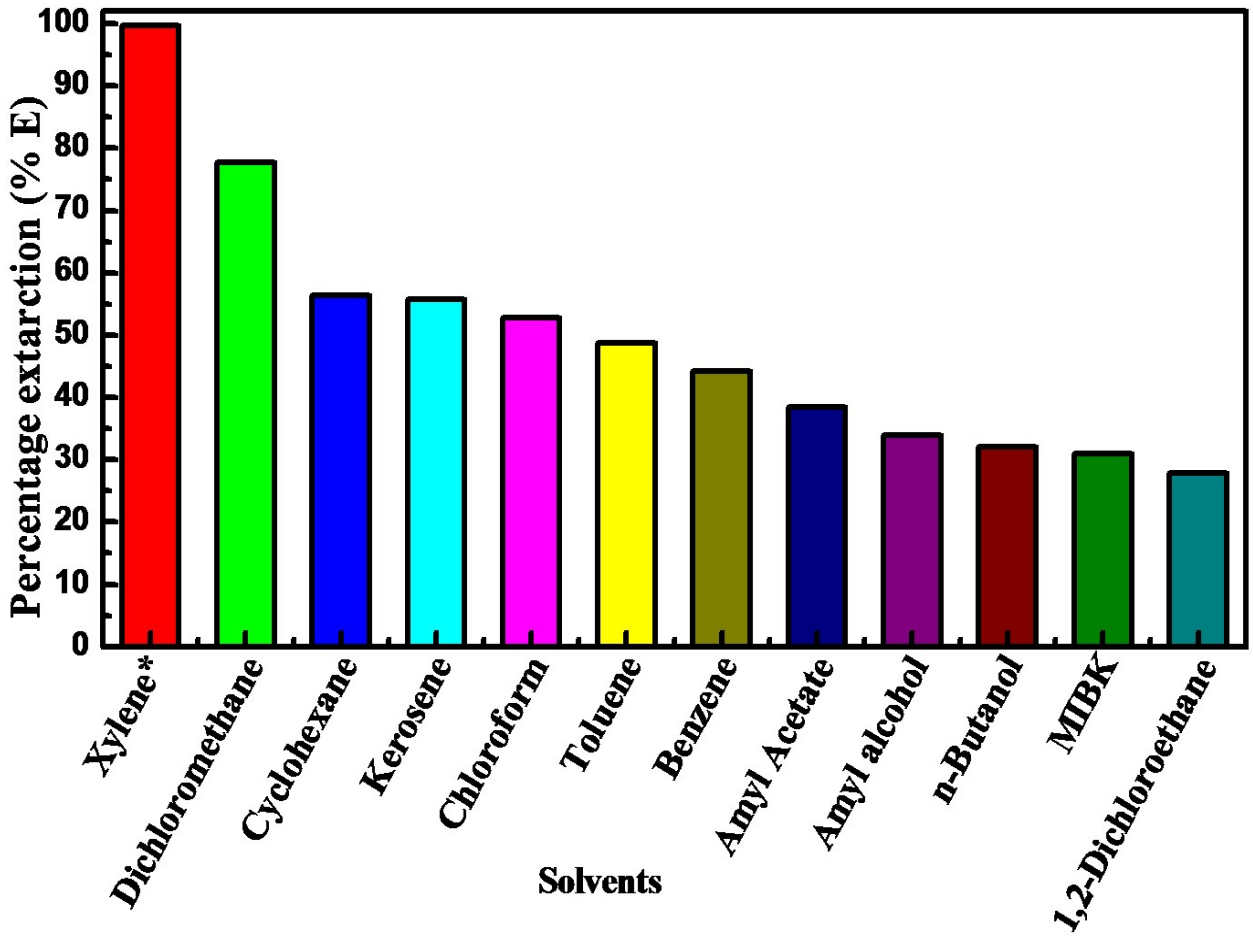

Absobance
0.0
0.1
0.2
0.3
0.4
0.5
0.6
340
360
380
400
420
440
460
Wavelength

Fig 5.


Percentage extarction (% E)
100
90
80
70
60
50
40
30
20
10
0
Xylene*
Dichloromethane
Cyclohexane
Kerosene
Chloroform
Toluene
Benzene
Amyl Acetate
Amyl alcohol
n-Butanol
MIBK
1,2-Dichloroethane
Solvents