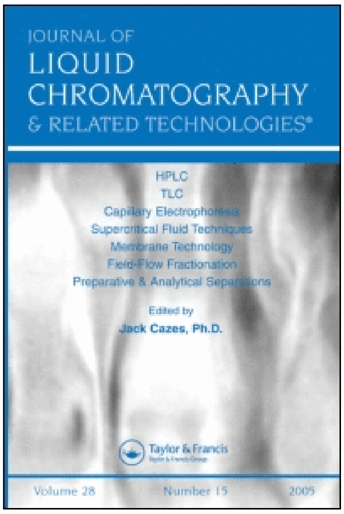

Journal of Liquid Chromatography & Related Technologies
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713597273

Thin Layer Chromatography of Metal Ions Complexed with Anils Part - VI

R. K. Upadhyay[a][b]; Usha Bajpai[a]; Arun K. Bajpai[a]
[a] Department of Chemistry, N.R.E.C. College, Khurja [b] Dr. V.P. Singh Sarai Nasuralla, KHURJA

To cite this Article Upadhyay, R. K. , Bajpai, Usha and Bajpai, Arun K.(1980) 'Thin Layer Chromatography of Metal Ions Complexed with Anils Part - VI', Journal of Liquid Chromatography & Related Technologies, 3: 12, 1913 — 1919
To link to this Article: DOI: 10.1080/01483918008064779
URL: http://dx.doi.org/10.1080/01483918008064779

JOURNAL OF LIQUID CHROMATOGRAPHY, 3(12), 1913-1919 (1980)

THIN LAYER CHROMATOGRAPHY OF METAL IONS COMPLEXED WITH ANILS PART - VI.

R.K. Upadhyay*, Usha Bajpai and Arun K. Bajpai
Department of Chemistry, N.R.E.C. College, Khurja-203131.

SUMMARY

Thin layer chromatographic detection, separation and determination of Ti(IV), UO_2(II), Au(III) and Hg(II) complexes with α - and β - naphthylanils of methylenediglyoxal have been made on silica gel layers. No locating agent was used as the complex spots were self evident in day light.

INTRODUCTION

In continuation to our previous work[1-5] on thin layer chromatography (TLC) of metal ions complexed with keto anils as ligands, present communication reports the TLC detection, separation and determination of Ti(IV), UO_2(II), Au(III) and Hg(II) complexes using α- and β- naphthylanils of methylenediglyoxal (abbreviated as A and B respectively) as ligands.

---

[*] Postal Address : Dr. V.P. Singh
Sarai Nasuralla, Behind Thana Dehat
KHURJA-203131.

EXPERIMENTAL

Preparation and Development of Plates

Glass plates (18x3cm) were coated with silica gel (free from chloride and iron ions) mixed with starch as binder (19:1, w/w) to prepare layers of 0.10 cm thickness with home-built apparatus[6] and dried at ∽ 100°C in an oven. Complex solutions prepared in alcohol were applied with fine capillaries. Dry plates were developed in rectangular glass chambers with ground-in-lids by ascending technique. The time of development given in Table -2 is for the distance 6-8 cms travelled by the solvent front. No locating agent was used: spots were self discernible in day light.

Synthesis of Ligands and Complexes

Synthesis and characterization of both the ligands (A & B) have been reported[7] earlier.

Ligand and metal chloride solutions of equimolar concentrations prepared in acetone were mixed in appropriate (stoichiometric) proportions taking slight excess of ligand, and mixture solutions were concentrated on water bath and left for crystallization. Dark products were air dried and finely powdered and washed with ether and chloroform successively, several times till un-reacted ligand, if any, was completely removed. Complexes were purified by recrystallization from methylcyanide or acetone and dried over anhydrous calcium-chloride under reduced pressure.

Analysis and Physical Measurements

Elemental analysis of the complexes was performed by C.D.R.I., Lucknow. Molar conductance measurements and conductometric titrations were made with Toshniwal's conductivity

TABLE - 1

ANALYSIS MOLAR CONDUCTANCE AND SPECTRAL CHARACTERISTICS OF COMPLEXES

Compound	Elemental Analysis				Λ_M	I.R. Frequencies cm^{-1}		λmax visible (nm)
	Nitrogen (%)		Metal(%)		(Mhos)			
	Calcd.	Found	Calcd.	Found		$\nu C=N$	$\nu C=O$	
A	-	-	-	-	-	1688	1744	-
$TlACl_4$	4.92	4.78	8.45	8.19	183.6	1652	1728	490
UO_2ACl_2	3.89	3.78	33.10	32.92	199.2	1648	1712	480
$AuACl_3$	4.10	4.00	28.90	28.81	252.2	1600	1680	480
$HgACl_2$	4.31	4.14	30.88	30.72	135.4	1620	1696	460
B	-	-	-	-	-	1632	1716	-
$TlBCl_4$	4.92	4.72	8.45	8.27	192.3	1628	1712	490
UO_2BCl_2	3.89	3.84	33.10	32.89	210.5	1597	1684	470
$AuBCl_3$	4.10	3.96	28.90	28.79	259.0	1565	1656	470
$HgBCl_2$	4.31	4.22	30.88	30.79	147.6	1597	1660	450

TABLE - 2

SPOT COLOUR AND R_F OF THE COMPLEXES

Complex	Spot Colour	MeOH	EtOH	BuOH	AcOH	BuOH--AcOH 2:1	BuOH--AcOH 1:1	BuOH--AcOH 1:2	BuOH. C_6H_6 2:1	BuOH. C_6H_6 1:1	BuOH. C_6H_6 1:2	Aq.BuOH--AcOH-$CHCl_3$ 5:5:1	Aq.BuOH--AcOH--$CHCl_3$ 5:5:2	Aq.BuOH--AcOH--$CHCl_3$ 5:5:3	BuOH--Hexane--$CHCl_3$ 1:2:1	BuOH--Hexane--$CHCl_3$ 2:1:1
$TiACl_4$	Brown	0.97	0.95	0.99	0.99	0.99	0.98	0.97	0.98	0.98	0.99	0.99	0.99	0.98	0.00	0.99
UO_2ACl_2	Canary Yellow	0.98	0.98	0.99	0.99	0.98	0.98	0.96	0.99	0.99	0.98	0.94	0.92	0.97	0.96	0.99
$AuACl_3$	Canary Yellow	0.98	0.97	0.94	0.99	0.95	0.94	0.94	0.99	0.99	0.99	0.90	0.96	0.91	0.96	0.99
$HgACl_2$	Canary Yellow	0.93	0.95	0.99	0.99	0.99	0.99	0.90	0.98	0.99	0.99	0.92	0.99	0.89	0.06	0.08
$TiBCl_4$	Canary Yellow	0.97	0.98	0.99	0.99	0.99	0.98	0.96	0.98	0.98	0.98	0.99	0.99	0.91	0.00	0.99
UO_2BCl_2	Yellow	0.97	0.95	0.99	0.94	0.99	0.96	0.89	0.98	0.98	0.99	0.99	0.90	0.98	0.00	0.99
$AuBCl_3$	Canary Yellow	0.98	0.99	0.99	0.99	0.99	0.98	0.96	0.99	0.99	0.99	0.98	0.95	0.99	0.99	0.99
$HgBCl_2$	Yellow	0.93	0.98	0.99	0.99	0.99	0.99	0.85	0.98	0.99	0.99	0.94	0.99	0.90	0.99	0.99
Developing time (min)		15	25	30	30	40	40	40	25	20	15	60	45	25	20	16

Room Temperature 35°C

bridge. Optical density measurements were made with 'spectronic-20' Bausch & Lomb spectrophotometer. Infrared spectra of the ligands and their complexes were recorded with Perkin Elmer-621 spectrophotometer in Nujol as mulling agent using CsF optics in frequency range 250 cm^{-1} to 4000 cm^{-1}.

RESULTS AND DISCUSSION

Conductometric titrations and elemental analysis of solid complexes revealed 1:1 (metal:ligand) stoichiometry in them. Each ligand acted as tetradentate ligand with two azomethine and two carbonyl donor centres. This fact is well exhibited by the considerable shift in azomethine and carbonyl groups infrared frequencies of the ligands on complexation. Molar conductance (ΛM) values (Table - 1) indicated 1:2 and 1:3 electrolytic nature of Ti(IV), UO_2(II), Hg(II) and Au(III) complexes respectively with each ligand. Molecular formulae deduced from these results are noted in Table - 1.

A perusal of R_F data reveals that mixtures of almost three complexes of ligand 'A' with metal ions Ti(IV), UO_2(II) or Au(III), Hg(II) can be resolved in several solvents but separation of the ternary mixtures can only be achieved in BuOH-Hexane-$CHCl_3$ (1:2:1, v/v) mixture solvent. The R_F of the complexes were found unchanged when their mixture was chromatographed. An increase in R_F of a particular complex with increasing number of substituted chlorine atoms in the hydrocarbon indicated the effect of solvent polarity on R_F; low R_F values could be accounted for by the high polarity of the solvents. The decrease in R_F of every complex from BuOH to MeOH supported the conclusion deduced earlier. In many instances abnormally high or low R_F values were observed in benzene-butanol and acetic-acid-butanol mixtures as compared with either of the mixture solvent components. This abnormality may be attributed[8] to the azeotropic properties of the solvent mixtures.

Quantitative Estimation

Chromatogram components were scrapped from the plates, eluted with ethanol and optical density of each elute was determined at the λ max of respective complex. Concentrations of chromatogram components were deduced from standard calibration curves obtained under similar conditions, of medium and temperature. Quantitative analyses of both the ternary mixtures of complexes were made.

ACKNOWLEDGEMENTS

We are thankful to Dr. S.S. Gupta, Principal, D.S. College, Aligarh, for providing the necessary facilities in this work, and to U.G.C., New Delhi, for the financial assistance to U. Bajpai.

REFERENCES

1. R.K. Upadhyay and R.R. Bansal, J. Indian Chem. Soc., 1976, 53, 15.
2. R.K. Upadhyay and V.P. Singh, J. Indian Chem. Soc., 1975, 52, 1164.
3. R.K. Upadhyay and V.P. Singh, J. Indian Chem. Soc., 1976 Accepted.
4. R.K. Upadhyay and R.R. Bansal, J. Indian Chem. Soc., 1977 Accepted.

5. R.K. Upadhyay and A.P. Tiwari, J. Indian Chem. Soc., 1977 Communicated.

6. E. Stahl, "Thin Layer Chromatography", Springer-Verlag, Berlin, Second Ed. 1969.

7. R.K. Upadhyay, S.C. Sharma, Usha Bajpai and Arun K. Bajpai Monatschefte fur Chemie, 1977, Communicated.

8. R.K. Upadhyay, U. Bajpai and R.R. Bansal, Monatschate fur Chemie, 1976, 107, 1221.