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Research Paper :

Separation and identification of p-methoxy phenacylidene-p-dimethyl amino aniline-Cd(II), Fe(III), Mn(II), Cu(II) and Co(II) complexes by Thin Layer Chromatography

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ABSTRACT

Thin Layer Chromatographic study on separation and identification of metal complexes of p-methoxy phencyclidine-p-dimethyl amino aniline (Schiff Base) with Cd(II), Fe(III), Mn(II), Cu(II) and Co(II) has been carried out. For this, Silica Gel is used as adsorbent and the mixture of complexes was run on thin layer of Silica Gel; the R_f values of the complexes were determined in Methanol, Benzene-Acetone, Nitrobenzene-Methanol and Dioxane. The complexes were separated and were identified by comparing their R_f values and developing time. It was found that the extent of separation of complexes varied considerably with the nature of solvent systems employed and comes in Methanol > Benzene-Acetone > Nitrobenzene-Methanol > Dioxane order.

KEY WORDS : Thin layer chromatography, Amino aniline, Schiff base, Complexes

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Thin layer chromatography (TLC) is one of the most handy and rapid chromatographic techniques often used in the analysis of complex mixture of substances. Some workers have described TLC as open chromatography, spread chromatography or surface chromatography. Although a large number of workers have done a significant quantum of research employing TLC[1-6]. It is used for the separation and characterization of complex mixture. In this techniques, a thin layer of adsorbents are coated on glass plates. This technique was introduced by Izmailov and Shraiber in 1938[7]. After that this technique is developed by other workers[8-12]. It is extremely surprising to note that till 1960, TLC was not utilized for the separation and identification of mixture of inorganic compounds as well as metal ion complexes[13].

Some of the salient features of TLC may be given as follows[13]:

– This technique is rapid and makes possible the separation of even micro quantities of substances in a complex mixture in very short time.

– It is very efficient and the fractionations are sharper and cleaner than those of column or paper chromatography.

– The inorganic layers are free from background substances which would interfere with spectroscopic analysis and cause intrinsic fluorescence in paper chromatography.

– The chromatoplates may be heated to higher temperature for the detection of compound.

– A very high degree of reproducibility can be achieved and moreover many reactions may be monitored.

Literature survey reveals that, TLC is frequently employed for the separation and identification of metal ion complexes of Schiff Bases[8-13].

In view of the rapidity, efficiency, sensitivity and reproducibility afforded by TLC for the separation and identification of organic compounds, it was considered worthwhile to separate and characterized metal ion complexes.

The present study concerns with the separation and identification of the complexes of Cd(II), Fe(III), Mn(II), Cu(II) and Co(II) with p-methoxy-phenacylidene-p-dimethyl amino aniline.

EXPERIMENTAL METHODOLOGY

Complexes of Cd(II), Fe(III), Mn(II), Cu(II) and Co(II) with p-methoxy-phenacylidene-p-dimethyl amino aniline have been prepared by mixing two solutions in

each case. First solution was of p-methoxy-phenacylidene-p-dimethyl amino aniline in methanol free acetone and second one was the solution of corresponding metal salt in acetone.

Silica gel G was mixed with starch as binder (19:1 w/w). It was use for coating the plates. Aqueous slurry was used to prepare the layers of 0.10 cm thickness on 30 x 8 cm glass plates. The coated plates were dried and activated at 100^0C for about three hours in an electric oven. Acetonitrile solutions of complexes were spotted on the plates with help of micropipette. The chromatographic plates were kept in a chromatographic chamber equilibrated with different solvents and then developed by ascending technique until the solvent front had traveled about 20 cm. The time required for each run was also noted. The solvent front was marked and the solvent front then allowed to evaporate. The R_f valve of the different metal complexes in different solvent systems such as methanol, dioxane, nitrobenzene-methanol, benzene-acetone, were determined[13] and are tabulated in table and the mixture of all the complexes was also be resolved in different solvent systems. Their R_f values are tabulated.

Observation:

Observed data such as colour of spots, R_f values and developing time of the complexes has been tabulated in Table 1 and R_f values of complexes in their mixture in Table 2. The R_f is tabulated as R_f x 100 and developing time is taken in minutes. Colours of the spots have been observed by naked eyes.

EXPERIMENTAL FINDINGS AND ANALYSIS

The R_f values of the mixture of complexes in different solvent systems are in close agreement with those obtained with the individual complex. The colour of the spots in different systems remains unchanged. The analysis may be made quantitative by scrapping off the components of the various chromatograms, dissolving them in suitable solvents and measuring their colour at absorption maxima.

It was found that the extent of separation of complexes varied considerably with the nature of solvent systems employed and comes in following order:

Methanol>Benzene-Acetone > Nitrobenzene-Methanol > Dioxane
(1:4 v/v) (1:4 v/v)

It is concluded that the extent of separation of complexes varied considerably with the nature of solvent systems and this result is satisfactory resembles with the previously obtained results[4,9-13].

Table 1: Spot colour and R_f values of the complexes

System	R_f x 100 Mixture of complexes				
	L-Cd	L_2-Fe	L-Mn	L-Cu	L-Co
A	52.6	72.6	70.0	50.8	61.2
B	49.6	46.2	60.7	44.8	51.6
C	58.5	60.1	64.6	52.8	54.6
D	62.6	77.9	74.2	53.6	62.8

Table 2 : R_f values of complexes in their mixture

Ligand / Chelate	Colour of spot	R_f x 100			
		System A	System B	System C	System D
Developing time (min.)	-	35	70	85	40
L	Yellow	78.4	60.8	70.6	58.1
L-Cd	Light blue	52.6	49.6	58.5	62.6
L_2-Fe	Green	72.6	46.2	61.1	77.9
L-Mn	Light red	70.0	60.7	64.6	74.2
L-Cu	Blue	50.8	44.8	52.8	53.6
L-Co	Brown	61.2	51.6	54.6	62.8

where:
L = Ligand (p-methoxy-phenacylidene-p-dimethyl amino aniline)
System A = Methanol
System B = Dioxane
System C = Nitrobenzene-Methanol (1:4 v/v)
System D = Benzene-Acetone (1:4 v/v)
Room temperature = 27 ± 1^0 C

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REFERENCES

1. **Seethala, R.**, Peterson, T., Dong, J., Chu, C. H., Chen, L., Golla, R., Ma, Z., Panemangalore, R., Lawrence, R. M. and Cheng, D. (2008). *Analytical. Biochem.,* **383**(2) : 144-150.
2. **Cossuta, D.,** Simandi, B., Vagi, E., Hohmann, J., Prechl, A., Lemberkovics, E., Kery, A. and Keve, T. *The J. Supercritical Fluids,* **47**(2): 188-194.
3. **Talansier, E.,** Braga, M. E. M., Rosa, P. T. V., Paolucci-Jeanjean, D. and Meriles, M.A.A. (2008). *The J. Supercritical Fluids,* **47**(2): 200-208.
4. **Fair, J. D.** and Kormos, C.M. (2008). *J. Chromatography,* **1211**(1-2) : 49-54.
5. **Gu, L.,** Wu, T. and Wang, Z. (2009). *LWT-Food Sci. & Technol.,* **42**(1): 131-136.
6. **Paramasivam, M.,** Poi, R., Banerjee, H. and Bandopadhyay, A. (2009). *Food Chem.,* **113**(2): 640-644.
7. **Ismailov, N.A.** and Shrabier, M.S. (1938). *Formatzija* (Moscow), **3**(1).
8. **Seiler, H.** and Seiler, M. (1965). *Helv. Chem. Acta.,* **48**: 117.
9. **Stahl, E.** (1966). *Z. Analyt, Chemie.,* **221**: 3.
10. **Uppadhyay, R.K.,** Singh, V.P. and Vajpai, U. (1974). *J. Chromatogr.,* **93**: 494.
11. **Gupta, D.R.,** Singh, Y. and Tayal, H.D. (1969). *J. Ind. Chem. Soc.,* **46**: 164.
12. **Tayal, H.D.** and Gupta, A.K. (1979). *Chromatographia,* **12**: 740.
13. **Gupta, A.K.D.** (1980). Phil. thesis, submitted to Garhwal University, Srinagar Garhwal (India).

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