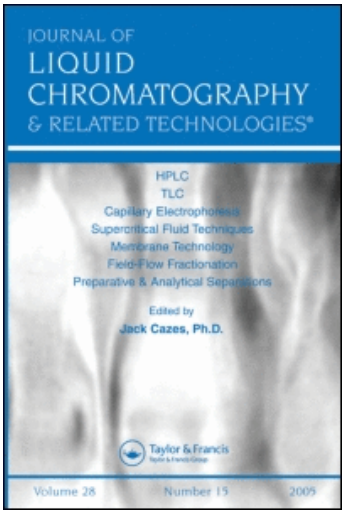

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TLC Separation of some closely Related Synthetic dyes Impregnated Silica Gel Layers

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TLC SEPARATION OF SOME CLOSELY RELATED SYNTHETIC DYES IMPREGNATED SILICA GEL LAYERS

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ABSTRACT

Ammonium molybdate and Copper sulphate were found as good impregnants for improving the separation of some closely related thirty synthetic dyes on silica gel 'G' layers using the solvent system BuOH–AcOH–H_2O (25:5:10). Tables are presented to illustrate the comparison in resolution of dyes on plain and impregnated silica gel layers.

INTRODUCTION

A survey of literature reveals that very little work has been done on the separation of synthetic dyes using impregnated silica layers[1,2]. However Srivastava and coworkers[3] separated certain dyes on cadmium acetate impregnated layers. The present paper presents an efficient separation of 30 dyes on silica gel plates impregnated with Ammonium molybdate and Copper sulphate using BuOH–AcOH–H_2O (25:5:10) solvent system.

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EXPERIMENTAL

The TLC plates of 0.5 mm thickness were prepared by spreading a slurry of a mixture of silica gel and 1.0% aq. solution of ammonium molybdate or 0.25% aq. solution of copper sulphate in the ratio 1:2 . The plates were dried for 24 hr at a constant temperature of 60°C. Solution of dyes were prepared in distilled water or water–ethanol mixture. Two solvent systems: BuOH–AcOH–H_2O (25:5:10) and BuOH–50% NH_3–Dioxan (25:5:10) were tried. The chromatograms were developed at a temperature of 35 ± 1°C. Spots were self visualised.

RESULT AND DISCUSSION

A close examination of data in Table I shows that some dyes show tailing on plain silica gel plates in both the solvent systems tried while on 0.25% copper sulphate impregnated plates tailing was observed for some dyes in the solvent system: BuOH–50% NH_3–Dioxan (25:5:10). However, no tailing of spots was observed on ammonium molybdate and copper sulphate impregnated layers using the solvent system BuOH–AcOH–H_2O (25:5:10). Tables II and III show the improved separation of dyes on impregnated layers. All dyes having hR_f values differing by 4 or more units were considered as resolved and this was exhibited by putting 'R' against them in the table. The '0' symbol indicates that resolution was not possible due to overlapping of spots either because of less than 4 units of difference in hR_f values or because of tailing of spots. As it is not possible to separate all the 30 dyes in a single run, the dyes were divided into the following groups for satisfactory separation on ammonium molybdate and copper sulphate impregnated layers in solvent system: BuOH–AcOH–H_2O (25:5:10)

On Ammonium Molybdate Impregnated Layer

	Dye No.	27	28	30	24	26	3	25	11	12	18
	hR_f	(00)	(11)	(25)	(29)	(40)	(49)	(55)	(59)	(67)	(72)
A		13	17	21	15						
		(82)	(87)	(95)	(99)						

TABLE I

Dye	BuOH–AcOH–H_2O (20:5:10) hR_f			BuOH–50% NH_3–Dioxan (25:5:10) hR_f		
	Plain	Imp.*	Imp.**	Plain	Imp*	Imp.**
1. Rosaniline HCL	84	57	85	84	62	77
2. Chrysoidine	83	69	84	93	86	87
3. Malachite green	65	49	65	99	97	95
4. Methyl red	88	78	80	56	46	45
5. Crystal violet	72	61	73	97	95	97
6. Fuchsine basic	83	62	70	94	86	86
7. Auramine O	73	52	82	86	71	77ST
8. Bromophenol blue	90	90	92	39	32	28
9. Eosine bluish	98	98	98	42	35	34
10. Bromocresol purple	84LT	85	87	32	23	22
11. Congo red	60	59	72	33	22	21
12. Titan Yellow	66	67	65	40	31	34
13. Aluminon	75LT	82	66	03	00	03
14. Alizarin	45ST	33	60	05LT	00	00
15. Magneson	99	99	99	66	65	63
16. Orange G	53	43	54	20	15	09
17. Bromocresol green	88	87	90	40	34	29
18. Phenol red	73	72	75	27	29	18
19. Thymol blue	85	84	86	73	74	63
20. Gentian Violet	73	52	75	67	47	71
21. Navilline Brilliant pink	97	95	96	97	92	93
22. Aniline blue	88	67	80	95	95	95
23. Dichlorofluroscein	98	98	97	21	16	13
24. Xylidine Ponceau	30	29	32	00	00	00
25. Benzopurpurine	62	55	60	38	28	24MT
26. Methylene blue	42	40	43	38LT	22	33
27. Nigrosin	00	00	00	00	00	00
28. Fuchsine acid	11	11	09	00	00	00
29. Light green	43	28	47	03	03	00
30. Alizarin blue	24LT	25	26	00	00	00

* Ammonium molybdate impregnation
**Copper sulphate impregnation
ST–Slight tailing, MT–Medium tailing, LT–Larg tailing.
hR_f Values are in 10cm. development.

TABLE II

RESOLUTION OF DYES ON PLAIN SILICA GEL

Dye No.	30	29	28	27	26	25	24	23	22	21	20	19	18	17	16	15	14	13	12	11	10	9	8	7	6	5	4	3	2	
1	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	O	R	R	R	O	
2	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	O	R	R	R		R	29
3	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	O	O	R	O	R	R	R	R	R	R		R	R	28
4	R	R	R	R	R	R	R	R	O	R	R	O	R	O	R	R	R	R	R	R	R	R	O	R	R	R		R	R	R	27
5	R	R	R	R	R	R	R	R	R	R	O	R	O	R	R	R	R	O	R	R	O	R	R	O	R		R	R	R	R	26
6	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R		R	R	R	R	R	25
7	R	R	R	R	R	R	R	R	R	R	O	R	O	R	R	R	R	O	R	R	O	R	R		R	R	R	R	O	R	24
8	R	R	R	R	R	R	R	R	O	R	R	R	R	O	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	23
9	R	R	R	R	R	R	R	O	R	O	R	R	R	R	R	O	R	R	R	R	R		R	R	R	R	R	R	R	R	22
10	R	R	R	R	R	R	R	O	O	O	R	R	R	O	R	O	R	R	R	R		R	O	R	R	R	R	R	R	R	21
11	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	O	R		R	R	R	R	O	R	R	R	R	R	20
12	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	O		R	R	R	R	R	R	R	R	R	R	R	19
13	O	O	O	O	O	O	O	R	R	R	O	R	O	R	O	R	O		R	R	R	R	R	R	R	R	R	R	R	R	18
14	R	O	R	R	O	R	R	R	R	R	R	R	R	R	R	R		R	O	R	R	R	R	R	R	R	R	R	R	R	17

15	R	R	R	R	R	R	R	O	R	O	R	R	R	R	R		R	R	R	R	R	R	R	R	R	O	R	R	R	R	16
16	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	15
17	R	R	R	R	R	R	R	R	O	R	R	O	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	14
18	R	R	R	R	R	R	R	R	R	R	O	R		R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	13
19	R	R	R	R	R	R	R	R	O	R	R		R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	12
20	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	11
21	R	R	R	R	R	R	R	O	R		R	R	O	R	R	R	O	R	O	R	R	R	R	R	R	R	R	R	R	R	10
22	R	R	R	R	R	R	R	R		R	R	R	R	R	O	R	R	R	R	R	O	R	O	R	R	R	R	R	R	R	9
23	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	8
24	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	O	R	R	R	R	R	7
25	R	R	R	R	R		R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	6
26	R	O	R	R		O	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	5
27	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	4
28	O	R		R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	3
29	R		R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	O	R	R	R	O	R	R	R	R	R	R	R	R	2
		R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	1
	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	Dye No.	

RESOLUTION OF DYES ON AMMONIUM MOLYBDATE IMPREGNATED LAYER

TABLE III

RESOLUTION OF DYES ON PLAIN SILICA GEL

Dye No.	30	29	28	27	26	25	24	23	22	21	20	19	18	17	16	15	14	13	12	11	10	9	8	7	6	5	4	3	2		
1	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	O	R	R	R	O		
2	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	O	R	R	R		R	29
3	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	O	O	R	O	R	R	R	R	R	R		R	R	28
4	R	R	R	R	R	R	R	R	O	R	R	O	R	O	R	R	R	R	R	R	R	R	O	R	R	R		R	R	R	27
5	R	R	R	R	R	R	R	R	R	R	O	R	O	R	R	R	R	O	R	R	O	R	R	O	R		R	R	R	R	26
6	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R		R	R	R	R	R	25
7	R	R	R	R	R	R	R	R	R	R	O	R	O	R	R	R	R	O	R	R	O	R	R		R	R	R	R	R	R	24
8	R	R	R	R	R	R	R	R	O	R	R	R	R	O	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	23
9	R	R	R	R	R	R	R	O	R	O	R	R	R	R	R	O	R	R	R	R	R		R	R	R	R	R	R	R	R	22
10	O	O	O	O	O	O	O	R	R	R	O	O	O	R	O	R	O	O	O	O		R	O	R	R	R	R	R	R	R	21
11	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	O	R		R	R	R	R	R	R	R	R	R	R	20
12	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	O		R	R	R	R	R	R	R	R	R	R	R	19
13	O	O	O	O	O	O	O	R	R	R	O	R	O	R	O	R	O		R	O	R	R	R	R	R	R	R	R	R	R	18
14	R	O	R	R	O	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	17

15	R	R	R	R	R	R	R	O	R	O	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	16
16	R	R	R	R	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	O	R	O	R	R	R	R	R	R	15
17	R	R	R	R	R	R	R	R	O	R	R	O	R		R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	14
18	R	R	R	R	R	R	R	R	R	R	O	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	13
19	R	R	R	R	R	R	R	R	O	R	R		O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	12
20	R	R	R	R	R	R	R	R	R	R		R	R	R	R	R	R	O	R	O	R	R	R	R	R	R	R	R	R	11
21	R	R	R	R	R	R	R	O	R		R	R	R	R	R	R	O	R	O	R	R	R	R	R	R	R	R	R	R	10
22	R	R	R	R	R	R	R	R		R	R	R	R	R	O	R	R	R	R	R	O	R	O	R	R	R	R	R	R	9
23	R	R	R	R	R	R	R		R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	8
24	R	R	R	R	R	R		R	R	R	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	7
25	R	R	R	R	R		R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	6
26	R	O	R	R		O	R	R	R	R	O	R	R	R	R	R	R	O	R	O	R	R	R	R	R	R	R	R	R	5
27	R	R	R		R	R	O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	4
28	O	R		R	R	R	R	R	R	R	R	O	O	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	R	3
29	R		R	R	R	R	O	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	2
		O	R	R	R	R	O	R	R	O	R	R	R	R	R	R	R	R	O	R	R	R	R	R	R	R	R	R	R	1
	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	Dye No.

RESOLUTION OF DYES ON COPPER SULPHATE IMPREGNATED LAYER

B	Dye No.	28	29	14	16	20	1	5	22	4	19
	hR_f	(11)	(28)	(33)	(43)	(52)	(57)	(61)	(67)	(78)	(84)
		8	23								
		(90)	(98)								
C	Dye No.	28	30	14	16	7	1	6	2	4	10
	hR_f	(11)	(25)	(33)	(43)	(52)	(57)	(62)	(69)	(78)	(85)
		11									
		(98)									

<u>On Copper Sulphate Impregnated Layer</u>

A	Dye No.	27	28	30	24	26	16	25	3	6	18
	hR_f	(00)	(09)	(26)	(32)	(43)	(54)	(60)	(65)	(70)	(75)
		4	1	17	21						
		(80)	(85)	(90)	(96)						
B	Dye No.	28	30	26	29	16	14	13	5	22	19
	hR_f	(09)	(26)	(43)	(47)	(54)	(60)	(66)	(73)	(80)	(86)
		8	9								
		(92)	(98)								
C	Dye No.	27	30	24	29	16	12	11	7	10	8
	hR_f	(00)	(26)	(32)	(47)	(54)	(65)	(72)	(82)	(87)	(92)
		15									
		(99)									
D	Dye No.	28	30	24	26	29	16	14	13	20	2
	hR_f	(09)	(26)	(32)	(43)	(47)	(54)	(60)	(66)	(75)	(84)
		17	23								
		(90)	(97)								

It is worthwhile to point out that the hR_f value is not altered when mixtures of these dyes are run. The behaviour of the dyes on impregnated plate depends on the following factors:

1. Formation of a metal-dye interaction product.
2. Differential solubility of the dye and the interaction product in the solvent system employed.
3. Adsorption behaviour of the dye on the impregnated silica gel plate.

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