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NOTE

Standardization of TLC Method for Identification of Carcino- genic Aryl Amines in Coloured Textile and Leather Products

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Many attempts have been made to detect carcinogenic aryl amines in coloured textile and leather products by TLC. But better resolution was the problem among mixtures of such amines. A chemical approach is shown to solve the resolution problems. For the good resoluton, a mixture of ethyl acetate and *n*-hexane was used as a mobile phase. The whole procedure was standardized with standard of carcinogenic aryl amines and tested with samples of coloured textile and leather products. Resolution of amines was also observed under HPTLC scanner.

According to the German Goods Ordinance, a German regulation,[1] dyes must be tested to ensure absence of carcinogenic aryl amines derived from coloured textile and leather products as a degradation product. For the last five years, the carcinogenic aryl amines literature has contained a great deal of information on the identification of these aryl amines in consumer coloured goods, like textile and leather products. We have discussed the analytical method for identification of these aryl amines by thin layer chromatography. The manufacturers and users of dyes require a rapid, economical and reliable test method. A number of thin layer chromatography methods have been developed in recent years for identification of these aryl amines.[2, 3] The following thin layer chromatography method gives very good resolution in the case of mixtures.

Apparatus and Instruments: TLC developing chamber, TLC plate $60F_{254}$, UV-lamp, microlitre pipette and HPTLC scanner

Reagents: Methanol, chloroform, acetic acid, *n*-hexane, ethyl acetate, 1-naphthol and potassium hydroxide

Standard Chemicals: Standard carcinogenic aryl amines (20 aryl amines have been declared carcinogenic)

Procedure: 30 mg/L solutions of all these aryl amines were made. 5 μL of standard solution was taken to make spot on TLC plate. (Spot should be 2.5 cm above the bottom of plate). TLC plate was developed in two types of mobile phase one in ethyl acetate and *n*-hexane (9 : 1) and another in chloroform and acetic acid (9 : 1) until solvent travelled up ¾th of plate. Allowed the plate to dry and kept in a nitration chamber for nitration and spraying reagent sprayed over the plate and observed the spot travelled on TLC plate under UV lamp and HPTLC scanner. Response factor (R_f) was calculated for individual amines

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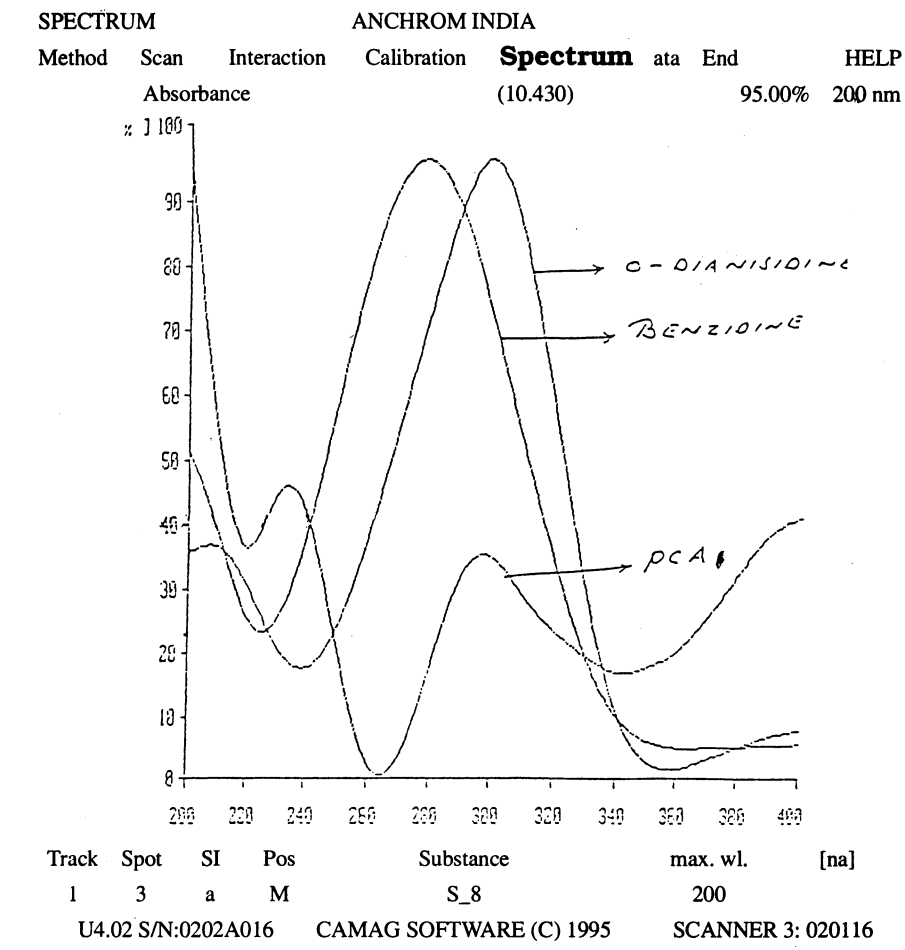
(Tables 1 and 2). Resolution in ethyl acetate and *n*-hexane mobile phase was observed in HPTLC for three standard aryl amines.

TABLE-1
(Chloroform-acetic Acid (9 : 1) was taken for mobile phase)

Name of the standard carcinogenic aryl amines	R_f Value
p-choraniline	0.70
o-toluidine	0.53
4,4-diaminodiphenylmethane	0.37
2-amino-4-nitrotoluene	0.78
3,3-dichlorobenzidine	0.74
4-aminobiphenyl	0.69
benzidine	0.47
2-naphthylamine	0.74
p-cresidine	0.70
4,4-oxydianiline	0.33

TABLE-2
(Ethylacetate-n-hexane (9 : 1) was taken for mobile phase)

Name of carcinogenic aryl amines	R_f-Value	Colour	Wevelength Maximum in nm
o-toluidine	0.90	grey	280/205
p-chloraniline	0.85	violet	235/290
p-cresidine	0.87	violet	280
4-chloro-*o*-toluidine	0.81	yellow	230/280
2,4,5-trimethylaniline	0.89	orange	225/225
2,4-tolyendiamine	0.43	brown-lilac	210/290/365
2-naphthylamine	0.80	yellow	220/295
2,4-diaminoanisole	0.41	violet-brown	205/295
4-aminobiphenyl	0.82	violet	270/360
2-amino-4-nitrotoluene	0.93	orange-voilet	245/290
benzidine	0.62	smoke-blue	280
4,4-oxydianiline	0.48	violet	240/290
4,4-diaminodiphenylmethane	0.52	violet	240/285/390
o-aminoazotoluene	0.96	violet-brown	245/365
3,3-dimethylbenzidine	0.94	grey	205/280
4,4-thiodianiline	0.65	violet	260
3,3-dichlorobenzidine	0.89	brown-lilac	210/280
3,3-dimethoxybenzidine	0.75	smoke-blue	210/300
4,4-diamino-3,3-dimethyldiphenylmethane	0.60	reddish-brown	240/285
4.4-methylene-bis-(2-chloraniline)	0.85	reddish-brown	205/340/295



Sample Preparation Method: It has already been published in Federal Health Gazette 2/96[4] 5 µl of extracted sample was spotted on TLC plate and developed in the prescribed mobile phase. R_f was calculated and matched with the R_f of standard aryl amines.

After carrying out TLC experiment, it has been found that the mixture of chloroform and acetic acid (mobile phase) is giving resolution of eleven standard aryl amines out of fourteen (Table-1), although ethyl acetate and *n*-hexane are giving resolution for all twenty aryl amines. (Table-2). This method is applicable to identification of carcinogenic aryl amines in the coloured textile and leather product.

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