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Determination of Impurities in Resorcinol by Thin-Layer Chromatography

Rezorsinol İçindeki Kirliliklerin İnce Tabaka Kromatografisi ile Teşhisi

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A rapid and sensitive method is described to detect phenol and catechol contamination in resorcinol by a single test which simplifies the USP(1) technique involving two separate procedure. The proposed TLC method is not only faster, but also more sensitive than the official one.

Resorcinol, one of the most popular constituents in lotions for external uses may contain impurities which increases its toxicity. According to USP XVII, phenol and catechol should be nearly absent.

There is no useful published method for the detection of the possible impurities in resorcinol. The author's objective is to develop a simple, rapid and sensitive technique for the detection of the two impurities. In attempting to develop an improved procedure for detecting the impurities in resorcinol, there are basically two problem to be solved: selection the most suitable technique and finding the optimal condition for both the separation and identification of theirs. For this purpose, TLC method is proposed.

EXPERIMENTAL

Apparatus. Desaga TLC equipment; tanks lined with filter paper saturated with solvent; 10 × 20 cm glass plates coated with a 0.5 mm of Aluminum Oxide, Type DO and $UV_{254-366}$ mμ lamps.

Reagents. Reagent grade bromine and ferric chloride.

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Solvents (given in Table II). Purified for chromatography.

Material. Commercial sample of resorcinol is used without further purification.

Procedure. One per cent solution of each substance prepared in methanol, 10 μl are applied, than developed with appropriate solvent systems. The solvent's front is allowed to run on the plates about 15 - 18 cm. The plates developed are sprayed with bromine, then immediately exposed to ammonia vapors. The colors obtained are shown in Table I.

Table I. Colors obtained with spray reagent

	Color of spots	
Substances	With bromine	After exposure to ammonia vapors
Catechol	pink	darkgray-brown
Phenol	dirty yellow	light brown
Resorcinol	light yellow	dark yellow

RESULTS

In selecting the optimal solvent system, it is obvious that the major difficulty is the separation of the phenol and resorcinol, while the solubility of these compounds are very similar. Polar solvents, such as water, metanol and ethanol, move all the compounds too fast and give practically no separation. Nonpolar solvents do not give good results with spray reagents. Acetone mixed with a small volume (5 to 10 per cent) of nonpolar solvent yield a safe and good separation for this three compounds.

Table II. Developing solvents and Rf values.

Solvent systems (Developing solvents)	Resorcinol		Phenol		Catechol	
	Single	in mixt.	Single	in mixt.	Single	in mixt.
S_1 : Water	0.72	0.69	0.72	0.75	0.14	0.07
S_2 : Acetone-Water (1 : 1)	0.83	0.87	0.91	0.93	0.08	0.04
S_3 : Acetone	0.72	0.69	0.80	0.82	0.07	0.03
S_4 : Acetone-Ether (1 : 1)	0.93	0.93	0.96	0.96	0.08	0.08
S_5 : AcOH-Acetone-Water (1 : 4 : 5)	0.75	0.82	0.82	0.85	0.53	0.53
S_6 : 3 % AcOH	0.70	0.66	0.60	0.66	0.20	0.20
S_7 : Ether (*)	0.55	0.48	0.96	0.93	0.04	0.02
S_8 : Acetone-Ether (1 : 3)	0.85	0.85	0.96	0.92	0.03	0.03
S_9 : Acetone-Ether (1 : 19) (*)	1.00	1.00	0.61	0.61	no migr.	no mig.
S_{10} : Acetone-Ether (1 : 9.5) (*)	0.64	0.64	0.85	0.85	no migr.	no mig.
S_{11} : Acetone-Ether (1 : 9) (*)	0.33	0.33	0.79	0.79	no migr.	no mig.

* The best results are obtained with these solvent systems.

We would like to report the best reagent for identification of them. In both cases (after spraying with bromine or exposing to ammonia vapors), the examination of plates under white fluorescent light is found to be useful for detection of phenol and catechol, while for detection of resorcinol, UV 366 mμ light is necessary. The minimum detectable amounts are 1 mcg for phenol, 10 mcg for catechol and 40 mcg for resorcinol. A list of solvents and solvent mixtures and Rf values are given in Table II.

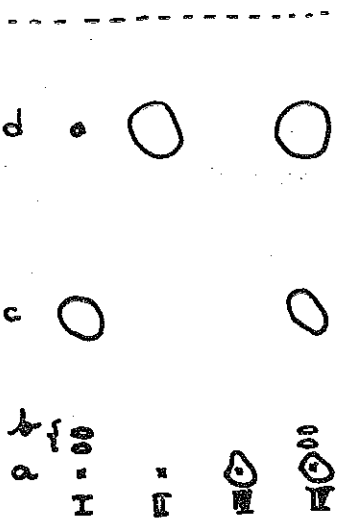

Fig. 1. I - Resorcinol, Rf=0.33 (b: its oxydation products, c: resorcinol, d: phenol); II - Phenol, Rf=0.79; III - Catechol, (no migration); IV - Mixture, (a: no migr., b: oxydation products of resorcinol, c: Rf=0.33, d: Rf=0.79).

SUMMARY

A thin-Layer chromatographic method is described for the separation of resorcinol USP XVII from catechol and phenol, and for its identification. It is possible to determine about 1 mcg of phenol, 10 mcg of catechol and 40 mcg of resorcinol with this method which may be preferable to the USP XVII method.

ÖZET

Bu yazıda, USP XVII de kayıtlı rezorsinolü ve ihtiva edebileceği kirlilikleri (katekol ve fenol) biribirinden ayırarak teşhislerine imkân

verebilen bir metod tarif edilmiştir. İnce tabaka kromatografisi diğer metotlardan daha kolay, süratli, randımanlı ve maddelerin çok küçük miktarlarının teşhisine imkân verecek hassaslıktadır. Bu yolla 1 mcg fenol, 10 mcg katekol ve 40 mcg rezorsinolün teşhisi mümkündür. Bu usul farmakope metoduna kolaylığı ve hassaslığı bakımından tercih edilebilir.

REFERENCE

1. The United States Pharmacopeia XVII, p. 564.

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