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The quantitative determination of phenols by an anodic oxidation wave using a mercury pool electrode during chromatography

The authors reported earlier[1–4] that inorganic substances could be determined conductimetrically by passing them between electrodes on the filter-paper during paper chromatography.

In this report, for the purpose of the electrolytic determination of organic substances, the authors have made a "multi-linear sweeping applied voltage" device§ on filter paper.

A schematic diagram of the apparatus is shown in Fig. 1. The filter paper is clamped between the electrodes (A, B) which supply a right-angled triangular wave input D.C. potential to the paper, from 0 V to +1 V (or 0 to +2 V, 0 to +3 V) for about 30 s.

When an organic material passes between the electrodes, the change in the electric current due to electrolysis is recorded by the recorder (Toa, EPR 2T type).

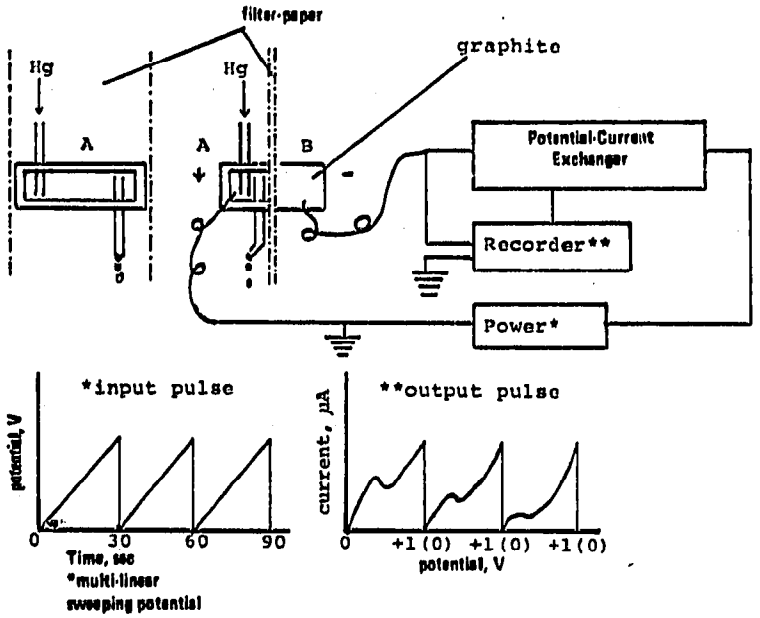

Fig. 1. Schematic diagram of apparatus.

Fig. 2 shows the current–potential curve obtained with the above apparatus, when as an example, 50 μg of resorcinol were developed by the solvent mixture methanol–0.1 N lithium chloride solution (80:20). In this case, the wave form is recognized as being similar to the current–potential curve of oxygen found in polarographic analysis, the maximum current (82 μA) is obtained at 0.30 V. The height of this maximum current (h) changes with time, and disappears when the resorcinol has passed between the electrodes.

The R_F value is given by the ratio between the distance from the electrodes to

§ Patent applied for.

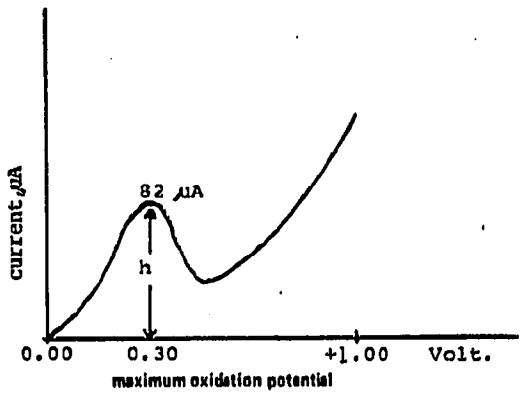

Fig. 2. Current–potential curve for one range of applied potential. Sample: 50 μg of resorcinol. Developer: methanol–0.1 *N* lithium chloride solution (80:20 v/v).

the starting point and the distance from the starting point to the solvent front when the current reaches its maximum. Table I shows the R_F values of different phenols and their maximum oxidation potentials (M.O.P.).

In the case of resorcinol and hydroquinone, the M.O.P. values obtained by the neutral developer do not differ much, but with an alkaline developer (methanol–0.4 *N* lithium hydroxide solution, 80:20 v/v) they differ a great deal. When the latter developer is used, it can be seen from the M.O.P. values that hydroquinone is oxidized much more easily than resorcinol because the M.O.P. of hydroquinone (0.23 V, first wave) is lower than that of resorcinol (0.65 V).

TABLE I

R_F VALUES AND MAXIMUM OXIDATION POTENTIALS (M.O.P.) OF PHENOLS (50 μg)

Neutral developer: methanol–0.1 *N* lithium chloride solution (80:20 v/v). Alkaline developer: methanol–0.4 *N* lithium hydroxide solution (80:20 v/v).

Phenol	*Neutral developer*		*Alkaline developer*	
	R_F	*M.O.P. (V)*	R_F	*M.O.P. (V)*
Phenol	0.81	0.32	0.50	0.75
o-Cresol	0.70	0.33	0.35	(I) 0.27 (II) 0.84[a]
m-Cresol	0.53	0.23	0.73	(I) 0.34 (II) 0.98[a]
Resorcinol	0.80	0.30	0.79	0.65
Hydroquinone	0.73	0.24	0.52	(I) 0.23 (II) 0.67[a]
Pyrogallol	0.58	0.21	0.60	0.72
Phloroglucinol	0.84	0.30	0.81	0.35
α-Naphthol	0.91	0.23	0.90	0.91
β-Naphthol	0.85	0.18	0.83	0.85

[a] (I) = 1st M.O.P. at maximum peak current; (II) = 2nd M.O.P. at maximum peak current.

The calibration curves for resorcinol and hydroquinone are given in Fig. 3. In the same way, calibration curves for other phenols can be obtained easily.

A quantitative determination of phenols by polarography has been reported only by VLČEK *et al.*[5–7] for the half-wave potentials of +0.15 V(pH 7.4) and +0.10 V (pH 6.5) for catechol and pyrogallic acid, respectively, but nothing has been reported on the oxidation wave for resorcinol and phloroglucinol.

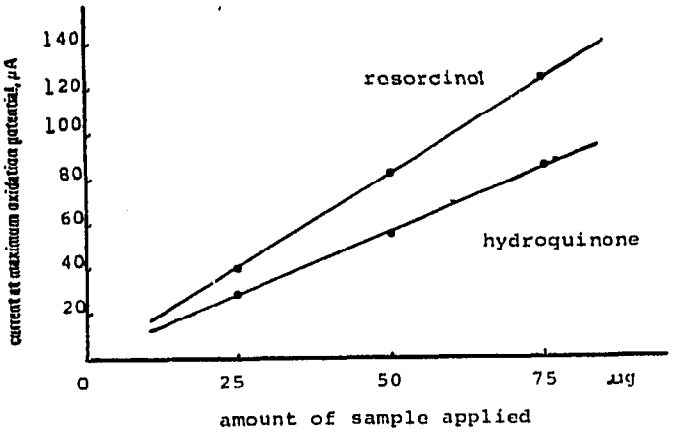

Fig. 3. Calculation curves for resorcinol and hydroquinone. Developer: methanol–0.1 *N* lithium chloride solution (80:20 v/v).

The reason why oxidation waves for phenols can be easily obtained with our apparatus is probably that an easier anodic oxidation is secured through the larger anode of the mercury-pool electrode, with a surface area about 500 times greater than a dropping mercury electrode in polarography.

The phenols that have passed through the electrodes show colors (brown or green) which indicate that they have been oxidized.

Kobe Women's College of Pharmacy, ITSUHIKO MORI
Higashinada-ku, Kobe (Japan)

Kobe Municipal Technical College, SHUMPEI CHIKUI
Tarumi-ku, Kobe (Japan)

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