CEPHALEXIN SULFOXIDE OSCILLOPOLAROGRAPHIC QUANTITATIVE DETERMINATION USING POTASSIUM HYDROGENPEROXOMONOSULFATE

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Cephalexin monohydrate is a derivate of amynodesacetoxycephalosporane acid, belongs to β-lactamic antibiotics of the I generation with a wide range of pharmacological activity. Cephalexin has a great Gram-negative and Gram-positive antimicrobial properties. It is produced in gelatin capsules and in the form of suspension. The aim of this work research is the development of a simple, rapid, and costeffective method for the determination of cephalexin in neat substances and powders by the preliminary oxidation of cephalexin in weakly acidic media to respective S-oxide, followed by its quantification by oscillographic polarography.

We used a cephalexin substance meeting the requirements of GFU with the concentration of the titular material 100.9% The oxidant was Oxone®, i.e., a triple potassium salt of Caro's acid, $2KHSO_5 \cdot KHSO_4 \cdot K_2SO_4$ (Acros Organics). Its active ingredient was the potassium hydrogen salt of peroxomonosulfuric acid, $KHSO_5$. The choice of the reagent was determined by its rather high oxidative capacity, $E^0 = 1.84$ V, easy availlability, and satisfactory solubility in water, and also by sufficiently high stability in use and storage.

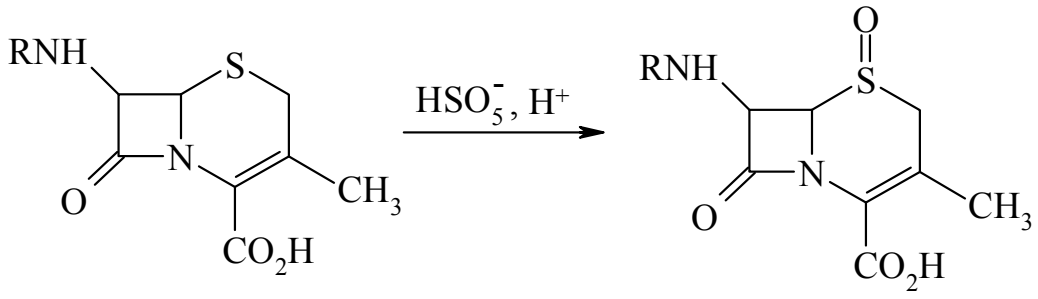
Working solutions of cephalexin and potassium peroxomonosulfate were prepared by diluting stock solutions with twice-distilled water.

Polarograms were recorded in a 0.03 M supporting acetate buffer solution with pH 4.0. To prepare it, 20.4 g of $CH_3COONa \cdot 3H_2O$ of chemically pure grade were dissolved in 50 mL of twice-distilled water, and pH 4.0 was set using 0.1 M HCl; the mixture was diluted to 500 mL with twice-distilled water and stirred carefully

The calibration plot was obtained. The relation of current I, μA, at –0.800 V to the concentration of cephalexin c, M, was approximated by the equation $I = (1.55 \pm 0.1) \cdot 10^4 c + (0.04 \pm 0.01)$, $r = 0.996$. The plot is linear in the range $(1–10) \cdot 10^{–5}$ M. This made possible the further determination by a refference method.

Cephalexin S-oxide in the reaction under study forms through an electrophilic attack of the β-oxygen atom in the peroxide group of the peroxoacid to sulfur within 1 min, i.e., the time of observation. The scheme of cephalexin oxidation by potassium peroxomonosulfate to give the respective S-oxide is presented below.

As was found, the redox reaction proceeds commpletely and stoichiometrically, 1 mol of $KHSO_5$ is consumed per 1 mol of the substance. Three peaks were recorded in the cathodic branch of the voltammograms of solutions: at –0.485 V (average sharp), at –0.800 V (average smooth), and –1.120 V (small smooth, Fig. 1). Considerable changes in peak height at –0.800 V was observed with changes in cephalexin concentration. It was chosen for analytical purposes.



Scheme. Cephalexin oxidation using potassium hydrogenperoxomosulfate

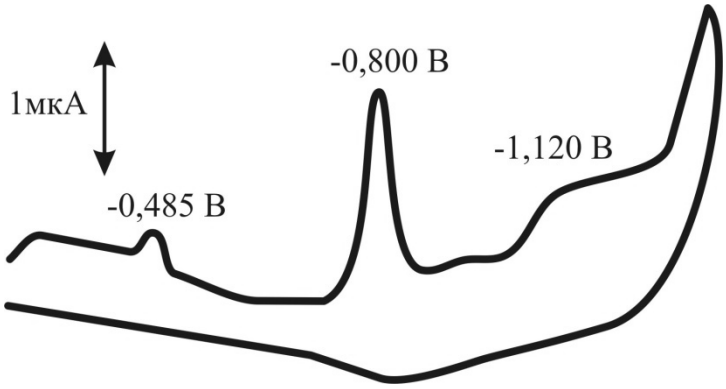


Fig. 1. An oscillographic polarogram of cephalexin S-oxide prepared by the reaction of cephalexin with potasssium peroxomonosulfate.

The data of determining cephalexin in the neat substance are given in the table.

Table. Results of cephalexin quantitative determination in the form of its S-oxide using potassium hydrogenperoxomonosulfate as analytical reagent

Added, neat substance, g	Cephalexin found, $C_{16}H_{17}N_3O_4S$		Metrological characteristics ($P = 0.95$; $n = 5$)
	g	%	
0.3654 ($100.9^{+2.0}_{-3.0}$ %)*	0.3551	102.22	$\overline{X}$ = 0.3428 (98.70%)*
	0.3474	100.00	S = $\pm 1.07 \cdot 10^{-2}$
	0.3358	96.66	$S_{\overline{x}} = \pm 4.79 \cdot 10^{-3}$
	0.3474	100.00	$\Delta\overline{X} = \pm 1.33 \cdot 10^{-2}$
	0.3281	94.44	RSD = 3.12%
			ε = ±3.88 %; δ = –0.46%

As is shown, in the determination of 5 10^{-5} M cephalexin in the substance, RSD was 3% at the accuracy errors δ = -0.46 %.