CAPSULES
notes and applications from Bioanalytical Systems, Inc.

Determination of Phenylmercuric Nitrate by Differential Pulse Polarography

Purpose

Phenylmercuric nitrate's antimicrobial quality has been employed as a fungicide in tree wound applications as well as an antiseptic and germicide in topical ointments used by veterinarians [1]. The most common use of this compound has been as a preservative in pharmaceutical preparations. The United States Pharmacopoeia (USP) describes a polarographic method for the determination of phenylmercuric nitrate as well as another common mercurial antimicrobial agent, thimerosal, in the manual USP 23-NF18, 1995 edition.

According to the USP, the addition of an antimicrobial agent to a formulation must be noted and the concentration of the compound must be given. Realizing that the concentration of such preservatives often decreases over the given shelf-life of the product, the concentration stated on the label is usually lower than the actual concentration, especially at the time of manufacture. The procedures outlined by the USP have been established "to demonstrate that the declared agent is present, but does not exceed the labeled amount by more than 20% of the labeled amount" [2].

Method

It should be noted that a modification has been made to the USP method for this Capsule. Differential Pulse Polarography (DPP) using the Controlled Growth Mercury Electrode (CGME) in the Stationary Mercury Drop Electrode (SMDE) mode has replaced the classical d.c. polarography experiment. The use of DPP affords the experimenter a more efficient way to obtain the polarograph of the target analyte. DPP's main attribute is its unique potential waveform, which provides excellent discrimination against the charging current, thus lowering detection limits and enhancing sensitivity. Also, the use of a gelatin solution (for peak suppression) is not necessary for the DPP procedure. For comparison, F1 illustrates the typical response for classical d.c. polarography of the phenylmercuric standard solution.

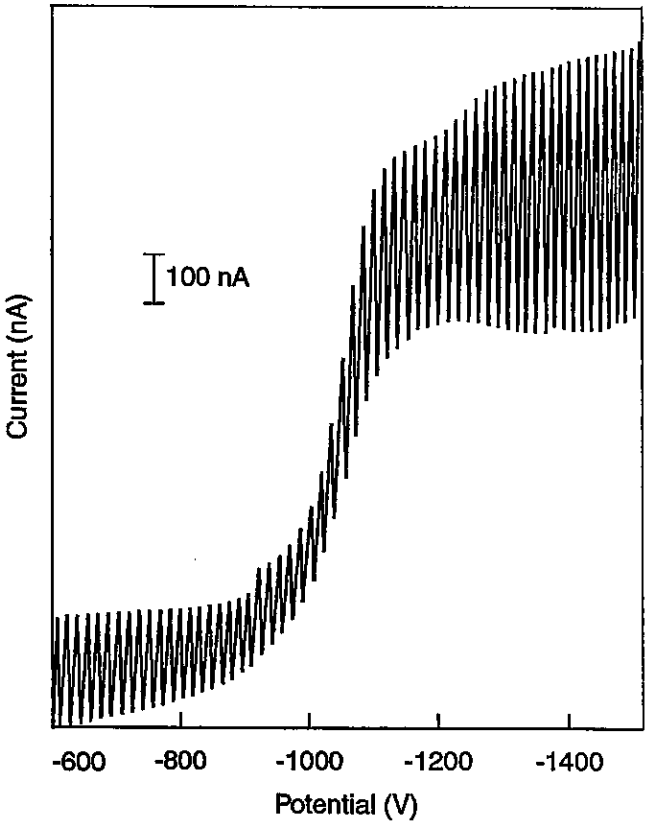

Figure 1. Classical d.c. polarogram of phenylmercuric nitrate.

General Procedure

Standard Preparation: Approximately 100 mg of phenylmercuric nitrate was weighed out and added to 250 mL of 0.1 M NaOH in a 1000 mL volumetric flask. The solution was stirred by a stir bar and heated on a hot plate for about one hour to aid dissolution. After complete dissolution, 0.1 M NaOH was added to volume.

Boric Acid / KCl Solution: In a 1000 mL volumetric flask, 12.37 g of boric acid and 14.91 g of KCl were dissolved in 200 mL of deionized water. After thorough mixing, deionized water was added to volume.

pH 9.2 Alkaline Borate Buffer: A 26.4 mL aliquot of 0.2 M NaOH solution was placed in a 200 mL volumetric flask. The boric acid / potassium chloride solution (50 mL) described above was added to the flask. The solution was diluted to volume with deionized water.

Test Preparation: A 10 mL aliquot of the solution to be analyzed (in this case, the standard solution described above) was transferred into a 25 mL volumetric flask. Two milliliters of 0.1 M KNO_3 solution was added to the flask. The solution was brought to volume with pH 9.2 alkaline borate buffer.

Conditions	DPP	LSV
EC Workstation:	BAS 100B/W	BAS 100B/W
Electrode:	SMDE mode	DME mode
Initial E (mV):	-600	-600
Final E (mV):	-1500	-1500
Sensitivity (µA/V):	1	1
Scan Rate (mV/s):	5	5
Pulse Amplitude (mV):	50	NA
Drop Time (s):	1000	NA
CGME Drop Size:	8	NA

Experimental Procedure

A 10 mL aliquot of the test preparation was transferred to a low-volume CGME cell and deaerated with a nitrogen stream for 5 minutes. A blanket of nitrogen was applied to the surface of the solution after deaeration was completed to prevent oxygen from dissolving into the solution during the experiment. The BAS CGME was used with the BAS 100B/W Electrochemical Workstation. The reference and auxiliary electrodes were a saturated calomel (SCE) and platinum wire, respectively. The polarogram (F2) was recorded from an initial potential of -600 mV to a final potential of -1500 mV.

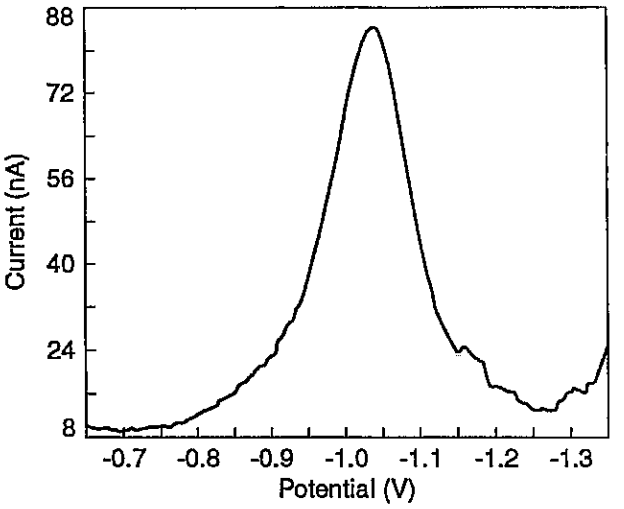

Figure 2. Polarogram of phenylmercuric nitrate.

Results

The following formula was used to calculate the quantity, in µg, of phenylmercuric nitrate per mL of specimen:

$$2.5C[(i_p)U/(i_p)S]$$

where C is the concentration of phenylmercuric nitrate in µg/mL of the standard preparation, $(i_p)U$ is the peak current of the test solution, $(i_p)S$ is the peak current of the standard preparation, and 2.5 is a factor needed to calculate the amount of phenylmercuric nitrate in 25mL of specimen.

References

1. *The Merck Index, An Encyclopedia of Chemicals, Drugs, and Biologicals, 11th edition, Merck & Co., Inc. Rahway, NJ, (1989) 1159.*
2. *The United States Pharmacopoeia, the National Formulary (USP 23-NF18), The United States Pharmacopoeial Convention, Inc. Rockville, MD (1995) 1733-1735.*