CAPSULES
preliminary notes and applications from Bioanalytical Systems, Inc.

Determination of Total Iodine in Thyroid Tablets Using Differential Pulse Polarography

Purpose

The analysis of thyroid tablets for total iodine content requires two separate methods, which have been determined by the U.S. Pharmacopoeia in the 1995 edition of the USP23-NF18 [1]. The initial assay consists of the determination of iodine by titrimetry. The determination of content uniformity was, in years past, accomplished by oxygen flask combustion followed by spectrophotometry [2]. This determination is currently performed using a more efficient method, differential pulse polarography (DPP).

This Capsule is intended to cover the general procedure for the determination of content uniformity. For the exact procedures, preparations, and solutions necessary for this analysis, please consult the 1995 edition of USP23-NF18 and current supplement [3].

Method

Differential pulse polarography is one of many electrochemical techniques being used for concentration measurements in the pharmaceutical industry. Electrochemical techniques can be used for the analysis of pharmaceuticals and multivitamin supplements for a variety of different compounds, metals, and halides. For example, classical d.c. polarography has been used for the determination of iodine, as well as other active ingredients of thyroid tablets, such as thyroxine and liothyronine [4-5].

However, the use of d.c. polarography is not applicable to the determination of these compounds in thyroid tablets, due to its inherent lack of sensitivity attributed to its characteristic large charging current. DPP's main attribute is its unique potential waveform, which provides for excellent discrimination against the charging current, thus lowering detection limits and enhancing sensitivity. The sensitivity is such that the DPP method can be used for the determination of iodine in a single tablet. The titrimetric method lacks adequate sensitivity for single tablet analysis [6].

Conditions

Electrochemical Workstation: BAS 100B/W
Electrode: Controlled Growth Mercury Electrode (CGME)
Technique: DPP
Initial E (mV): -800
Final E (mV): -1500
Sensitivity (μA/V): 10
Scan Rate (mV/s): 5
Pulse Amplitude (mV): 50
Drop Time (ms): 1000
Quiet Time (ms): 0

General Procedure

The determination of total iodine in thyroid tablets consists of three steps. The first is the ashing of the tablet with potassium carbonate to release the iodide ion. The second step consists of the chemical oxidation of iodide to iodate by the addition of bromine. Finally, the peak at approximately -1180 mV *vs.* SCE, corresponding to the reduction of iodate to iodine, is recorded.

One thyroid tablet was crushed in a porcelain crucible using a glass rod. Potassium carbonate (8 grams) was mixed into the tablet with a glass rod. The crucible was placed in a pre-heated muffle furnace and the contents were ashed at 700°C for 25 minutes. The mixture was allowed to cool to room temperature. Water (30 mL) was added to the crucible and the solution was heated on a hot plate until all of the residue dissolved. The solution was filtered through a plug of glass wool into a 100 mL volumetric flask. The heating and filtration were repeated two more times, both aliquots were added to the original 30 mL filtrate in the 100 mL volumetric. Bromine test solution (1 mL) was added to the flask with approximately 20 mg of sodium sulfite. (The sodium sulfite

scavenges any excess bromine in the solution). The solution was mixed thoroughly and diluted to volume with water.

Standard Preparation

An iodine stock solution of approximately 1 mg per mL was prepared by accurately weighing out 1.69 g of potassium iodate, transferring to a 1 mL volumetric flask, and diluting to volume with deionized water. Next, an aliquot (8 mL) of the stock solution was transferred by pipette to a 250 ml volumetric flask and diluted to volume. Depending on the dosage of the tablet being analyzed, an aliquot of the diluted stock solution (1 mL of diluted stock solution per 1/4 grain thyroid tablet) was transferred to a 100 mL volumetric flask containing 8 g of potassium carbonate dissolved in approximately 10 mL of deionized water. Bromine test solution (1 mL) was added to the flask and mixed. Next, sodium sulfite (approximately 20 mg) was added to the flask until the solution became colorless and then mixed. The solution was diluted to volume with water and then mixed.

Polarography

A 10 mL aliquot of the above solution was transferred to a glass polarographic cell and deaerated by nitrogen stream for 5 minutes. A blanket of nitrogen was applied on the surface of the solution after deaeration was completed. The BAS Controlled Growth Mercury Electrode (CGME) was used in the Stationary Mercury Drop Electrode (SMDE) mode with the BAS 100B/W as the potentiostat. The reference and auxiliary electrodes were a saturated calomel (SCE) and platinum wire, respectively.

Results

The polarograms were recorded for test preparation, blank, and standard solutions, which were prepared according to USP procedures. The polarogram for the tablet solution is shown in F1. The amount of iodine (µg) per thyroid tablet is determined using the following formula:

$$I = (0.593)(54.08V)(PH_U / PH_S)$$

where V=volume of sample solution, in mL; PH_S and PH_U are the ip (peak height) values obtained from the polarograms for the standard and thyroid tablet solutions, respectively. The constant 0.593 corresponds to the ratio of atomic weight of I to the molecular weight of KIO_3. The constant 54.08 is a conversion factor resulting from the dilution of tablets of varying dosage.

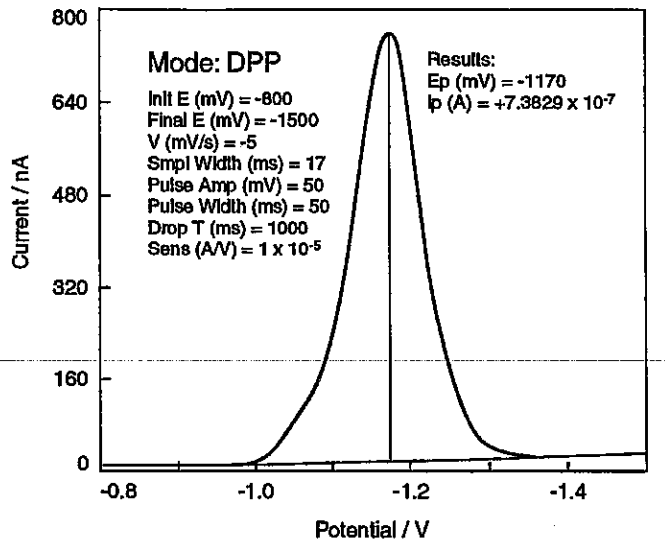

Figure 1. Polarogram of the thyroid tablet solution.

References

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