4219 Determination of Volatile Sulfide for Rubber Closures

Rubber materials often use sulfur or sulfocompound as crosslinking agents, which is difficult to react completely in the vulcanization process, resulting free sulfur produced from excess sulfur or sulfocompound. If such vulcanized rubber material is placed in the medium extracted from the aqueous solution, volatile sulfides will be formed under certain acidity conditions. Such released sulfides be can be reacted with the lead acetate test paper to form sulfur spots and thus can be measured visually by comparison of the sulfur spots left on the test paper.

This method applies to the determination of volatile sulfide in rubber closure as a part of pharmaceutical packaging system.

Preparation of standard sodium sulfide solutions Freshly prepared immediately before use. Take 1.0 g of sodium sulfide, add water to dissolve and dilute to 200 ml, shake well. Accurately measure 50 ml of obtained solution into an iodine flask, accurately add 25 ml of iodine titration solution (0.05 mol/L) and 2 ml of hydrochloric acid, shake well, and titrate with sodium thiosulfate titration solution (0.1 mol/L). Near the end point, add 1 ml of starch indicator solution, continue with the titration until the blue color fades away, and apply blank correction to the titration result. Each 1 ml of iodine titration solution (0.05 mol/L) corresponds to 1.603 mg of S. According to the above determination results, take a proper amount of the remaining stock solution and accurately dilute with water to produce a solution containing 20μg of S per ml.

Method I

Preparation of standard sulfur spots Accurately measure 1 ml of standard sodium sulfide solution into a conical flask (recommended caliber: 19/26), add 50 ml of 2% citric acid solution, place a piece of lead acetate test paper on the mouth of the conical flask, fasten it with a reversed beaker, place them in the autoclave, keep them at 121 ℃ ±2 ℃ for 30 minutes, and then take out the lead acetate test paper for test.

Procedure Take an appropriate amount of test samples with a total surface area of 20 cm^2±2cm^2 (cut if necessary), place in a conical flask (recommended caliber: 19/26), add 1 ml of water, add 50 ml of 2% citric acid solution, place a piece of lead acetate test paper on the mouth of the conical flask, fasten with a reversed beaker, place in the autoclave, keep at 121 ℃ ±2 ℃ for 30 minutes, then take out the lead acetate test paper and compare the sulfur spots formed with the above standard sulfur spots. The color shall not be more significant.

Method II

Apparatus and device In accordance with the apparatus and device for the Method I, Determination of Arsenic (General Chapter 0822) (the Gutzeit method), among which A is a conical flask with grinding edge (recommended caliber: 19/26), connect the airway C (which is not loaded with lead acetate cotton) to the top for test, and place a piece of lead acetate test paper on the top plane of the stopcock D.

Preparation of standard sulfur spots Accurately measure a proper volume of standard sodium sulfide solution, precisely dilute with water to produce a solution containing 20μg of S per ml. Accurately measure 1 ml of the solution into a conical flask A, add 50 ml of 2% citric acid solution. Insert the airway C with a lead acetate test paper fixed on it into the flask A, place in the autoclave, keep at 121 ℃ ±2 ℃ for 30 minutes, and then take out the lead acetate test paper.

Determination Take an appropriate amount of test samples with a total surface area of 10 cm^2±1 cm^2 (cut if necessary) into a conical flask A, add 1 ml of water, add 50 ml of 2% citric acid solution. Insert the airway C with a lead acetate test paper fixed on it into the flask A, place them in the autoclave, keep them at 121 ℃ ±2 ℃ for 30 minutes, then take out the lead acetate test paper and compare the sulfur spots formed with the above standard sulfur spots. The color shall not be more significant.

[Notes]

(1) If the standard sodium sulfide solution is to be prepared by sodium sulfide reference material, accurately measure a proper volume of sodium sulfide reference material, add water to dissolve and quantitatively dilute to produce a solution containing 20μg of S per ml.

(2) When weighing sodium sulfide, deliquescent reagents should be avoided as far as possible.

Drafted by: Sichuan Institute for Drug Control (Sichuan Medical Device Testing Center), Jiangxi Institute for Drug Control and Detection Contact number: 028-64020264

Participants: National Institutes for Food and Drug Control, Anhui Institute for Food and Drug Inspection, Shanghai Food and Drug Packaging Materials Testing Institute, Jiangsu Best New Medical Material Co, Ltd., Shandong Institute of Medical Device and Drug Packaging Inspection, China National Pharmaceutical Packaging Association, Zhengzhou Aoshine Pharmaceutical Technology Co., Ltd., Hubei Huarun Technology Co., Ltd.