a written report of analysis of the experimental data will be submitted to FDA, together with a commitment not to continue the experiment beyond the proposed ending date without FDA approval;

(5) The geographic area or areas in which the experiment is to be conducted;

(6) The mechanism to measure the effectiveness of the experiment;

(7) The method for conveying to consumers the required nutrition and other labeling information that is exempted from the label during the experiment;

(8) The method that will be or has been used to determine the actual nutritional characteristics of foods for which a claim is made; and

(9) A statement of the sections of the regulations for which an exemption is sought.

(c) The written proposal should be sent to the Division of Dockets Management (HFA–305), Food and Drug Administration, 5630 Fishers Lane, rm. 1061, Rockville, MD 20852. The proposal should be clearly identified as a request for a temporary exemption for purposes of conducting authorized food labeling experiments and submitted as a citizen petition under §10.30 of this chapter.

(d) Approval for food labeling experiments will be given by FDA in writing. Foods labeled in violation of existing regulations will be subject to regulatory action unless an FDA-approved exemption to the specific regulation has been granted for that specific product.

(e) Reporting requirements contained in §101.108(b) have been approved by this Office of Management and Budget and assigned number 0910–0151.

[48 FR 15240, Apr. 8, 1983, as amended at 59 FR 14364, Mar. 28, 1994; 62 FR 15343, Mar. 31, 1997]

APPENDIX A TO PART 101—MONIER-WILLIAMS PROCEDURE (WITH MODIFICATIONS) FOR SULFITES IN FOOD, CENTER FOR FOOD SAFETY AND APPLIED NUTRITION, FOOD AND DRUG ADMINISTRATION (NOVEMBER 1985)

The AOAC official method for sulfites (*Official Methods of Analysis*, 14th Edition, 20.123–20.125, AOAC INTERNATIONAL) has been modified, in FDA laboratories, to facilitate the determination of sulfites at or near 10 ppm in food. Method instructions, including modifications, are described below.

Apparatus—The apparatus shown diagrammatically (Figure 1) is designed to accomplish the selective transfer of sulfur dioxide from the sample in boiling aqueous hydrochloric acid to a solution of 3% hydrogen peroxide. This apparatus is easier to assemble than the official apparatus and the back pressure inside the apparatus is limited to the unavoidable pressure due to the height of the 3% H_2O_2 solution above the tip of the bubbler (F). Keeping the backpressure as low as possible reduces the likelihood that sulfur dioxide will be lost through leaks.

The apparatus should be assembled as shown in Fig. 1 with a thin film of stopcock grease on the sealing surfaces of all the joints except the joint between the separatory funnel and the flask. Each joint should be clamped together to ensure a complete seal throughout the analysis. The separatory funnel, B, should have a capacity of 100 ml or greater. An inlet adapter, A, with a hose connector (Kontes K–183000 or equivalent) is required to provide a means of applying a head of pressure above the solution. (A pressure equalizing dropping funnel is not recommended because condensate, perhaps with sulfur dioxide, is deposited in the funnel and the side arm.) The round bottom flask, C, is a 1000 ml flask with three 24/40 tapered joints. The gas inlet tube, D, (Kontes K–179000 or equivalent) should be of sufficient length to permit introduction of the nitrogen within 2.5 cm of the bottom of the flask. The Allihn condenser, E, (Kontes K–431000–2430 or equivalent) has a jacket length of 300 mm. The bubbler, F, was fabricated from glass according to the dimensions given in Fig. 2. The 3% hydrogen peroxide solution can be contained in a vessel, G, with an i.d. of *ca*. 2.5 cm and a depth of 18 cm.

Buret—A 10 ml buret (Fisher Cat. No. 03–848–2A or equivalent) with overflow tube and hose connections for an Ascarite tube or equivalent air scrubbing apparatus. This will permit the maintenance of a carbon dioxide-free atmosphere over the standardized 0.01*N* sodium hydroxide.

Chilled Water Circulator—The condensor must be chilled with a coolant, such as 20% methanol-water, maintained at 5 °C. A circulating pump equivalent to the Neslab Coolflow 33 is suitable.

Reagents

(a) *Aqueous hydrochloric acid, 4N.*—For each analysis prepare 90 ml of hydrochloric acid by adding 30 ml of concentrated hydrochloric acid (12N) to 60 ml of distilled water.

(b) *Methyl red indicator*—Dissolve 250 mg of methyl red in 100 ml ethanol.

(c) *Hydrogen peroxide solution, 3%*—Dilute ACS reagent grade 30% hydrogen peroxide to

3% with distilled water. Just prior to use, add three drops of methyl red indicator and titrate to a yellow end-point using 0.01*N* sodium hydroxide. If the end-point is exceeded discard the solution and prepare another 3% H_2O_2 solution.

(d) *Standardized titrant, 0.01N NaOH*—Certified reagent may be used (Fisher SO–5–284). It should be standardized with reference standard potassium hydrogen phthalate.

(e) *Nitrogen*—A source of high purity nitrogen is required with a flow regulator that will maintain a flow of 200 cc per minute. To guard against the presence of oxygen in the nitrogen, an oxygen scrubbing solution such as an alkaline pyrogallol trap may be used. Prepare pyrogallol trap as follows:

1. Add 4.5 g pyrogallol to the trap.

2. Purge trap with nitrogen for 2 to 3 minutes.

3. Prepare a KOH solution prepared by adding 65g KOH to 85 ml distilled water (caution: heat).

4. Add the KOH solution to the trap while maintaining an atmosphere of nitrogen in the trap.

Determination

Assemble the apparatus as shown in Fig. 1. The flask C must be positioned in a heating mantle that is controlled by a power regulating device such as Variac or equivalent. Add 400 ml of distilled water to flask C. Close the stopcock of separatory funnel, B, and add 90 ml of 4*N* hydrochloric acid to the separatory funnel. Begin the flow of nitrogen at a rate of 200±10 cc/min. The condenser coolant flow must be initiated at this time. Add 30 ml of 3% hydrogen peroxide, which has been titrated to a yellow end-point with 0.01*N* NaOH, to container G. After fifteen minutes the apparatus and the distilled water will be thoroughly de-oxygenated and the apparatus is ready for sample introduction.

Sample preparation (solids)—Transfer 50 g of food, or a quantity of food with a convenient quantity of SO_2 (500 to 1500 mcg SO_2), to a food processor or blender. Add 100 ml of 5% ethanol in water and briefly grind the mixture. Grinding or blending should be continued only until the food is chopped into pieces small enough to pass through the 24/40 point of flask C.

Sample preparation (liquids)—Mix 50 g of the sample, or a quantity with a convenient quantity of SO_2 (500 to 1500 mcg SO_2), with 100 ml of 5% ethanol in water.

Sample introduction and distillation—Remove the separatory funnel B, and quantitatively transfer the food sample in aqueous ethanol to flask C. Wipe the tapered joint clean with a laboratory tissue, apply stopcock grease to the outer joint of the separatory funnel, and return the separatory funnel, B, to tapered joint flask C. The nitrogen flow through the 3% hydrogen peroxide solution should resume as soon as the funnel, B, is re-inserted into the appropriate joint in flask C. Examine each joint to ensure that it is sealed.

Apply a head pressure above the hydrochloric acid solution in B with a rubber bulb equipped with a valve. Open the stopcock in B and permit the hydrochloric acid solution to flow into flask C. Continue to maintain sufficient pressure above the acid solution to force the solution into the flask C. The stopcock may be closed, if necessary, to pump up the pressure above the acid and then opened again. Close the stopcock before the last few milliliters drain out of the separatory funnel, B, to guard against the escape of sulfur dioxide into the separatory funnel.

Apply the power to the heating mantle. Use a power setting which will cause 80 to 90 drops per minute of condensate to return to the flask from condenser, E. After 1.75 hours of boiling the contents of the 1000 ml flask and remove trap G.

Titration.—Titrate the contents with 0.01*N* sodium hydroxide. Titrate with 0.01*N* NaOH to a yellow end-point that persists for at least twenty seconds. Compute the sulfite content, expressed as micrograms sulfur dioxide per gram of food (ppm) as follows:

$$ppm=(32.03xV_B\ xNx1000)\div Wt$$

where 32.03=milliequivalent weight of sulfur dioxide; V_B=volume of sodium hydroxide titrant of normality, N, required to reach endpoint; the factor, 1000, converts milliequivalents to microequivalents and Wt=weight (g) of food sample introduced into the 1000 ml flask.

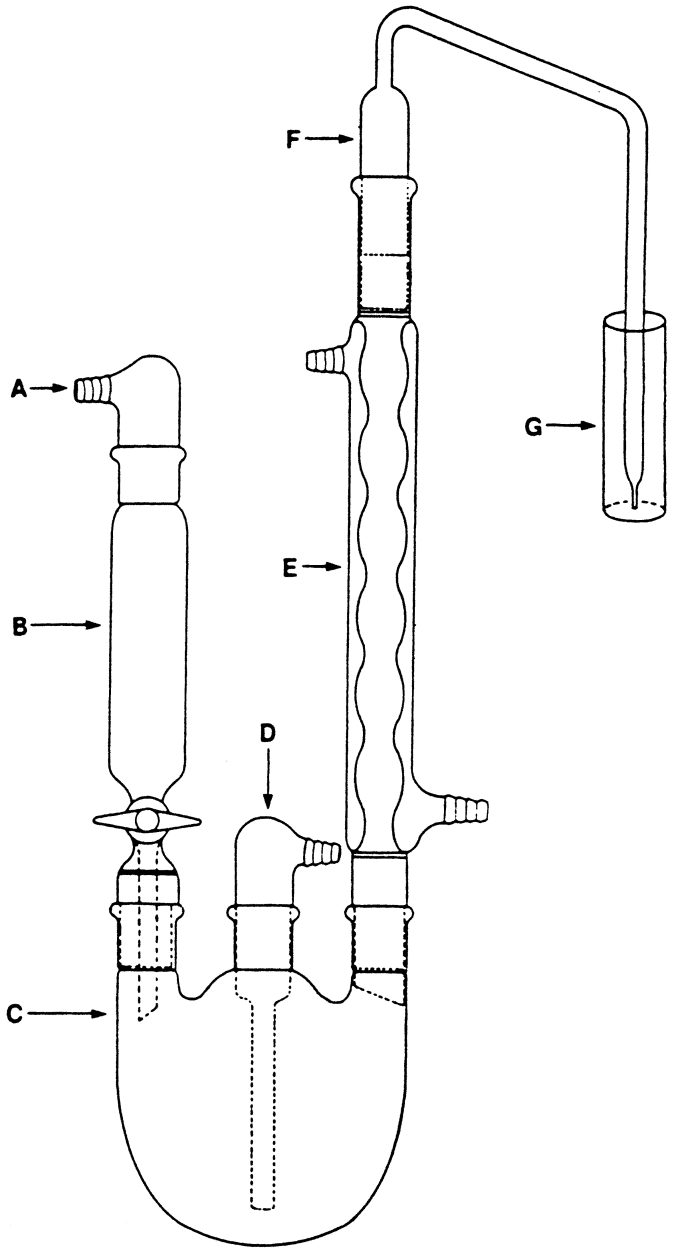

FIGURE 1. THE OPTIMIZED MONIER-WILLIAMS APPARATUS. COMPONENT IDENTIFICATION IS GIVEN IN TEXT.

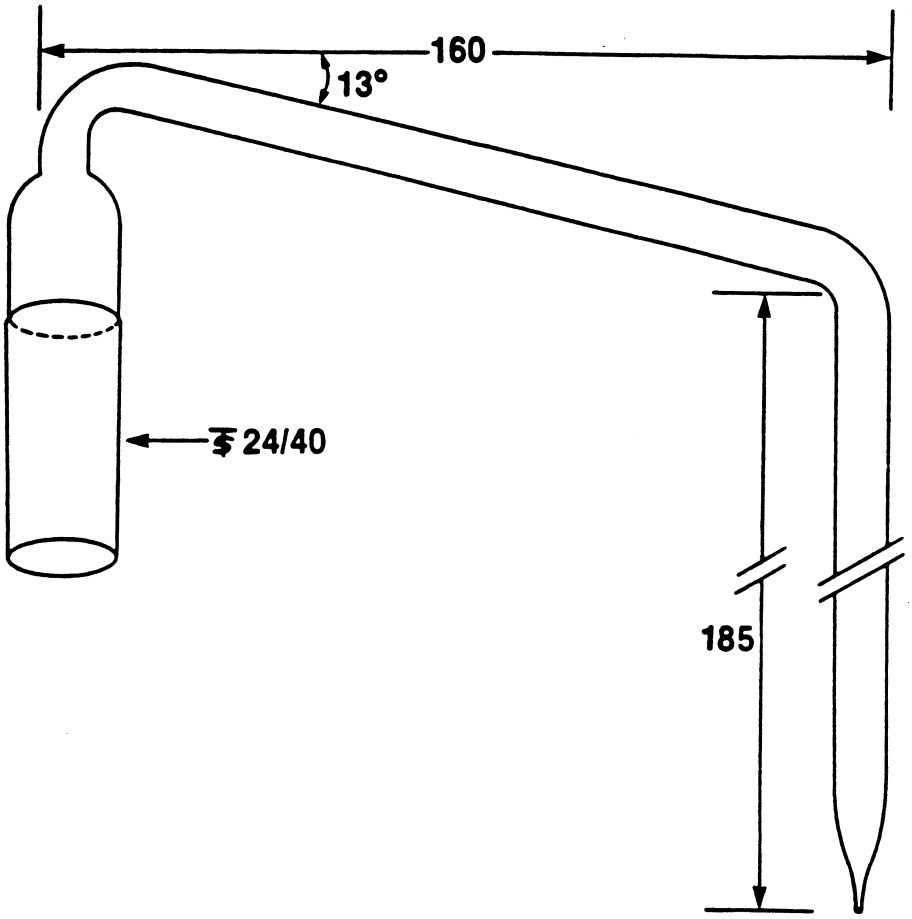

FIGURE 2. DIAGRAM OF BUBBLER (F IN FIGURE 1). LENGTHS ARE GIVEN IN MM.

[42 FR 14308, Mar. 15, 1977, as amended at 51 FR 25017, July 9, 1986]

APPENDIX B TO PART 101—GRAPHIC ENHANCEMENTS USED BY THE FDA