Sulfonanilide.—White crystals (89%) from isoöctane, m. p., 133.5-134.2°.

Anal. Calcd. for  $C_{16}H_{18}O_4N_2S$ : C, 58.0; H, 5.3. Found: C, 57.8; H, 5.1.

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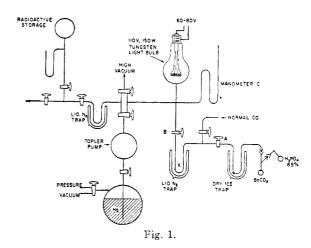
## Production of Radioactive Carbon Monoxide from Barium Carbonate

By Joseph T. Kummer<sup>1</sup>

It has been found possible to prepare conveniently a supply of radioactive carbon monoxide of a specific activity and sufficient in quantity for use in the study of many catalytic reactions. This is done by exchanging, over a hot tungsten filament, the C14 in a small amount of carbon dioxide obtained from barium carbonate of high specific activity (about 0.5% of the carbon in the barium carbonate was C14) with the carbon in a large amount of normal carbon monoxide. Since the apparatus and procedure are extremely simple, and since such exchange experiments have not previously been described for a tungsten surface,<sup>2</sup> they are described below in conjunction with Fig. 1. It is hoped that the method will be useful to those wishing to employ carbon monoxide for tracer studies.

The system is completely evacuated, before the exchange run, to  $10^{-6}$  mm. or better. Then, with stopcock A closed, the bulb containing 85%H<sub>3</sub>PO<sub>4</sub> is rotated and the acid allowed to react with a few mg. of the radioactive barium carbonate. When stopcock A is opened, the carbon dioxide is allowed to pass through the Dry Ice trap into trap X, cooled in liquid nitrogen. The acid-carbonate mixture is evacuated and warmed to drive all of the carbon dioxide out of the solution and into trap X. This carbon dioxide is next allowed to evaporate into the electric light bulb and is diluted with the required amount of carbon monoxide, manometer C being used for estimating approximately the amount of carbon monoxide added. If the light bulb is run at 60-80 volts overnight (sixteen hours) the exchange will be complete. No experiments have been made as to the rate of exchange or length of time it would take if the bulb were run at 110 volts. Carbon filament bulbs were originally tried but were found to be unsatisfactory because their filaments burned out in an atmosphere of carbon monoxide within a few hours. After a year of use, the tungsten filament showed no deterioration. When the exchange is complete, the radioactive carbon monoxide is pumped into the storage reservoir by means of a Töpler pump through

- (1) Guif Research & Development Company Fellowship, Mellon Institute of Industrial Research, Pittsburgh, Pa.
- (2) Brandner and Urey, J. Chem. Phys., 13, 351 (1945), have studied the kinetics of  $C^{13}$  exchange between CO and  $CO_2$  over quartz. Au, and Ag.



a liquid nitrogen trap, to remove the carbon dioxide.

Below are data for a particular test run using a new, 150-watt, 110-volt Westinghouse tungsten filament light bulb.

A sample of 2.6 mg. of BaC\*O<sub>3</sub>³ was taken; it was capable of producing approximately 10<sup>7</sup> disintegrations per minute. The C\*O<sub>2</sub> in trap X was flushed into the light bulb by 200 cc. of normal carbon monoxide (to a total pressure of about 300 mm.) after the liquid nitrogen was removed from trap X. After the C\*O<sub>2</sub> and carbon monoxide had been mixed by a few strokes of the Töpler pump, a 1-cc. sample was removed for analysis (without interposing a liquid nitrogen trap); this sample showed 42,000 disintegrations per minute per cc. of gas.

A similar sample taken through a liquid nitrogen trap for removing carbon dioxide showed 190 disintegrations per minute per cc. of gas.

After the filament in the bulb had been operated at 70 volts for sixteen hours, a 1-cc. sample removed through a liquid nitrogen trap showed a count of 42,100 disintegrations per minute per cc. of gas. Apparently, therefore, the exchange over the tungsten filament was complete in this period of time.

(3) An asterisk is used to designate C14.

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## Thiophene-Containing Antihistaminic Agents

By L. P. Kyrides, F. C. Meyer and F. B. Zienty

The high order of antihistaminic activity recently reported for the thiophene analog  $(I)^{1,2,3}$  of Pyribenzamine was observed on (I) prepared in this Laboratory and tested prior to Dr. Weston's disclosure. In addition, several other

- (1) Weston, This Journal, 69, 980 (1947).
- (2) Clapp, Clark, Vaughan, English and Anderson, ibid., 69, 1549 (1947).
- (3) Roth, Richards and Shepperd, Federation Proc., 6, 366 (1947).
  (4) Lee, Dinwiddie and Chen, J. Pharmacol. Exptl. Therap., 90, 83 (1947).