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The authors are indebted to Messrs. Chester C. Hargreaves and William T. Winchester for their valuable assistance in making the assays in frogs.

# THE DETECTION OF CARBON MONOXIDE IN MEDICINAL OXYGEN.\*,1 BY FREDERICK K. BELL AND JOHN C. KRANTZ,<sup>2</sup> JR.

### INTRODUCTION.

The eleventh revision of the United States Pharmacopoeia includes a general test for the detection of carbon monoxide in the medicinal gases, oxygen and carbon dioxide. The procedure employed is a modification of the method set forth by Teague (1). In the Teague method thoroughly scrubbed, dried air is allowed to pass over heated, highly purified iodine pentoxide from which it quantitatively liberates iodine. The entire apparatus must be swept for hours and sometimes for days with carbon monoxide-free air in order to obtain a negative blank. To make this method applicable to medicinal oxygen we (2 and 3) inserted an additional  $I_2O_5$  tube through which the gas to be tested was previously washed to remove any carbon monoxide that might be present. Thus the gas to be tested could be employed to scrub the iodine pentoxide that was to be used in the test.

• Since this method became official it has become the subject of careful investigation on the part of manufacturers of medicinal gases. The principal criticism of the method is the difficulty and time involved in the obtaining of a negative blank and the complete failure to obtain a negative blank in the hands of certain experimentors. Success or failure in this endeavor depends first on the quality of the iodine pentoxide employed and secondly on the careful observance of all experimental details set forth in the method.

The experiments set forth in this communication describe a method for the detection of carbon monoxide in oxygen without the use of the troublesome iodine pentoxide.

#### EXPERIMENTAL.

The method developed depends upon the reduction of palladious chloride to palladium by the carbon monoxide (4). The flocculent metallic palladium in suspension reacts with a solution of ammonium molybdate producing molybdenum blue, the intensity of which is propor-

<sup>\*</sup> Scientific Section, A. PH. A., New York meeting, 1937.

<sup>&</sup>lt;sup>1</sup> The expense of this investigation has been defrayed by a grant from the Board of Trustees of the United States Pharmacopœial Convention.

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tional to the absolute amount of carbon monoxide in the sample tested. For these tests a standard mixture of  $CO-O_2$  was used. The carbon monoxide was prepared by the action of sulfuric acid on pure formic acid. The gas was liquefied and fractionated: the mixtures with oxygen were prepared for us by weight by Dr. Leo I. Dana of the Linde Air Products Company of Buffalo.

Procedure: Fill a 1000-cc. volumetric flask with distilled water and invert it over distilled water. Displace the water in the flask with the oxygen until less than 1 cc. of water remains in the inverted flask. Rapidly introduce 2 cc. of a saturated solution of palladious chloride, restopper and twirl the flask in order to bring the liquid and gas into intimate contact. The shaking is repeated intermittently for an hour and the mixture is then set aside for 12 hours in subdued light. Now add 2 cc. of ammonium molybdate solution (5 Gm. in 100 cc.), shake thoroughly and add 2 cc. of diluted hydrochloric acid. Allow the mixture to stand for one hour and then transfer to a suitable Nessler tube. Holding the tube over a white background and looking down through the liquid a bluish hue is observed in contradistinction to the yellowish green of a blank test.

#### DISCUSSION.

Using the foregoing procedure 10 p. p. m. of carbon monoxide in oxygen may be easily detected and in concentrations of 25 p. p. m. the deposit of metallic palladium prior to mixing with the ammonium molybdate solution is plainly visible. In applying this test many of the principal difficulties of the iodine pentoxide method are obviated. Nevertheless the non-specificity of the iodine pentoxide method obtains with the palladium procedure. Scrubbing with fuming concentrated sulfuric acid will easily remove one of the greatest offenders in this regard, namely, acetylene. As yet we have been unable to selectively absorb hydrogen, which if present in the oxygen tested in a concentration of 0.5 per cent gives a positive test.

Further experiments are being conducted to increase if possible the specificity of the test and to reduce the time required in which to perform it.

### SUMMARY.

1. A procedure has been set forth for the purpose of detecting carbon monoxide in oxygen based upon the reduction of palladious chloride by the former. The sensitivity is of the order of magnitude of 10 p. p. m.

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## A STUDY OF THE FACTORS INFLUENCING THE STABILITY OF LIQUOR MAGNESII CITRATIS, U. S. P. XI.\*,1

### BY GEORGE E. CROSSEN AND CHAS. H. ROGERS.

Osol and Tice, in a recently published paper (20), report that stable samples of Solution of Magnesium Citrate can be prepared from 15 Gm. of an official magnesium carbonate and 33 Gm. of citric acid if the potassium or sodium bicarbonate is withheld until the product is dispensed. Such solutions, of course, contain nei-

<sup>\*</sup> Scientific Section, A. PH. A., New York meeting, 1937.

<sup>&</sup>lt;sup>1</sup> Abstracted from a thesis presented to the Graduate Faculty of the University of Minnesota in partial fulfilment of the requirements for the degree of Master of Science.