# The Indirect Gas Chromatographic Determination of Palladium in an Aqueous Solution

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In conventional gas chromatographic determinations of non-volatile substances, the constituents have always been converted into their volatile forms; therefore, metal ions have been converted into their $\beta$-diketone chelates.[1] Practical analyses of metal ions, however, require operations of several steps and involve serious problems, such as the interference of an excess chelating agent or solvent and decompositions of the metal chelates.

The present paper will offer a simple and indirect method for the determination of palladium ion in an aqueous solution. In this method a mixture of propylene and propane in a definite volume ratio is placed in contact with a perchloric acid solution containing palladium ions, and the resultant gas is analyzed by a gas chromatograph, using propane as an internal standard. Since the reaction between propylene and palladium ions proceeds quantitatively and very fast to form metallic palladium, a linear relationship is held in an acidic solution between the amounts of propylene and the palladium ions.

A mixture of propylene and propane in a 1 : 1 vol. ratio is stored in a balloon, and an aliquot of it is used for each experiment. In order to determine the amount of palladium, 1 ml. of the mixed gas is placed in the gas sampler A, and then A is connected, as is shown in Fig. 1, with the tube, B, in which a palladium solution has previously been charged. After the different ingredients have been in contact with one another at room temperature for some time, the gas is collected in A. The tube is removed and the gas is analyzed under the following conditions: 30 ml./min. of $H_2$ as the carrier gas; activated alumina as the stationary phase; a $0.4\times225$ cm. column (70°C), and a thermal coductivity cell as the detector. The relative retention volumes of propane and propylene are 162 and 300 ml. respectively. Up to 480 p. p. m. of palladium gives a straight line against the ratio of the peak area of propylene/propane (Fig. 2).

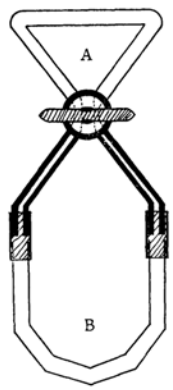


Fig. 1. Gas sampler and tube.
A: Gas sampler B: Tube

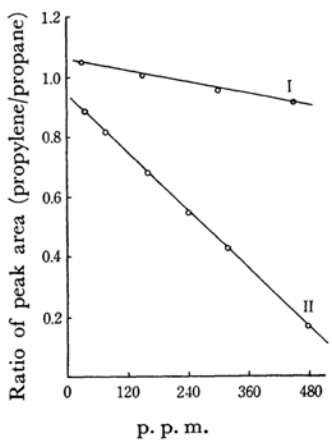


Fig. 2. Calibration curves.
I: $Ag^+$, II: $Pd^{2+}$.

The present method is not interfered with by platinum(II) copper(I) mercury(II), or nickel(II) ions which have $d^8$ or $d^{10}$ electrons and which more or less show affinities for olefins, but it is slightly interfered with by a large amount of silver ions.

An analysis can be made in about 15 min.

1) For example, R. E. Sievers, B. W. Ponder, M. L. Morris and R. W. Moshier, *Inorg. Chem.*, **2**, 693 (1963); K. J. Eisentraut and R. E. Sievers, *J. Am. Chem. Soc.*, **87**, 5254 (1965).