DETERMINATION OF SMALL AMOUNTS OF NITROGEN IN GASES

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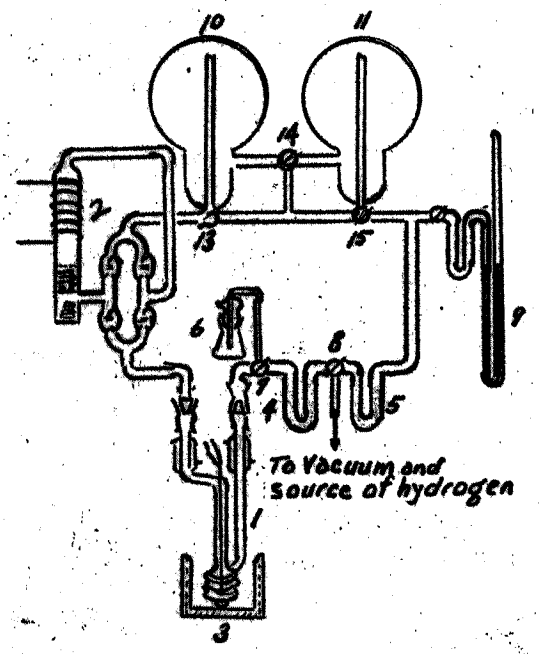
Hydrogen and other gases often contain small amounts of nitrogen. By means of the method described below it is possible to determine nitrogen directly in gaseous mixtures. In its simplest form the method can be applied to the determination of traces of nitrogen in hydrogen.

The hydrogen which contains nitrogen is circulated several times through a closed system containing a catalyst for converting the nitrogen into ammonia and a trap cooled with liquid air until all the nitrogen has been removed as ammonia. The amount of ammonia formed is then determined by the usual methods.



The apparatus is shown schematically in the diagram. The total volume of the apparatus as used in our experiments could be varied from 1-10 liters (the volume is changed by including or excluding supplementary flasks 10 and 11 by means of the taps 13, 14 and 15). The gas is circulated by means of a glass electromagnetic pump 2 [1]. Flask 1 made of pyrex contains 30 g of the catalyst for ammonia synthesis in the form of grains 2-3 mm in diameter. The catalyst is reduced at the

begining of an experiment. The vessel is provided with a coil for preliminary heating of the gas. The gas is heated by means of an electric furnace 3. The temperature of the catalyst is measured with a thermocouple. Experiments are conducted at 475°. Circulation of the gas mixture is continued until the ammonia which is frozen in the tube 4 filled with broken glass ceases to be formed. For a content of 1% of nitrogen in the test gas a few hours is necessary for complete conversion into ammonia at a circulating rate of 300-500 liters per hour. In order to check that all the nitrogen has been converted into ammonia, U tube 5 which precedes tube 4 is periodically cooled with liquid air. The absence of any deposit of solid ammonia in this tube indicates the end of the reaction. The trap 4 is then allowed to warm up to room temperature and the ammonia blown by a stream of hydrogen through tap 8 into flask 6 containing standard sulfuric acid. Excess acid is then backtitrated with NaOH using a microburet. Before intake of a new sample the apparatus is evacuated[1]. By means of this method nitrogen can be determined in hydrogen at a concentration of $1 \cdot 10^{-3}$% vol. with a possible error of $\pm 5 \cdot 10^{-5}$.

The method can also be used for the determination of small amounts of nitrogen in various gases such as argon or other inert gases[2]. A small amount of nitrogen-free hydrogen must be added to other gases containing nitrogen before carrying out the procedure described above.

The method can also be used for freeing hydrogen from contamination with nitrogen. Hydrogen cylinders usually contain up to 1% nitrogen. It could also be used for removing nitrogen from inert gases. E.g. if it is required to determine nitrogen in argon using hydrogen from a cylinder, the hydrogen is allowed into the system up to a pressure of 1/2 atmosphere. The nitrogen in the hydrogen is converted into ammonia which is frozen in trap 4. After disconnecting tap 4 from the system by means of taps 7 and 8 the ammonia is removed by pumping out in a frozen state under high vaccuum. The gas to be analyzed is then led into the system, the amount introduced being found by the increase in pressure measured by manometer 9 (the volume of the system should be known). The determination is then carried out as described above. The hydrogen used for combining with the nitrogen can be removed by heated cupric oxide.

LITERATURE CITED

[1] I.G.Katsnelson, B.P.Bruns and D.Yu.Gamburg, Factory Labs.12, 379 (1946).

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1) Air must be excluded from the apparatus since this will lead to deterioration of the catalyst

2) I.P.Krichevsky pointed this out.