NITROSYL-SULFURIC ACID: AN EXPERIMENT ILLUSTRATING THE LEAD CHAMBER REACTION

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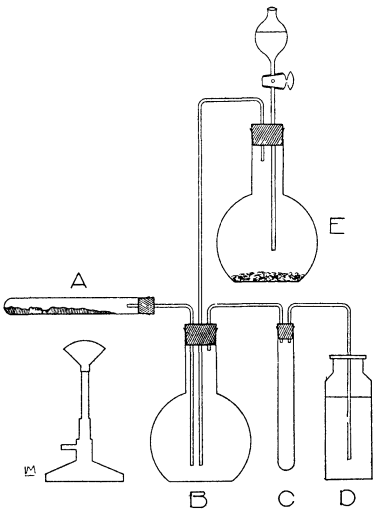
It is rather difficult in an elementary course in general chemistry to require students to prepare sulfuric acid by a process parallel to the Chamber process. This is usually shown to them in the course of lecture experiments. However, it is possible for advanced students to do a certain number of experiments involving considerable manipulation; and occasionally two beginning students working together will enjoy doing something beyond test tube reactions. For this purpose the following experiment has been developed to illustrate reactions in the Chamber process for making sulfuric acid.



A mixture of twelve grams of manganese dioxide and nine grams of flowers of sulfur is heated in an eight-inch hard glass test tube (*A*) and the sulfur dioxide formed is led into a 500-cc. flask (*B*). In the flask (*E*) nitrogen dioxide is generated by dropping concentrated nitric acid on copper clippings. This gas is led into the "chamber" (*B*) and the excess passes on through the safety tube (*C*) and the bottle (*D*) The latter contains concentrated sulfuric acid and is designed to function like the "Gay-Lussac tower" in the commercial plant.

The experiment is begun by carefully heating tube (*A*) until moisture is driven out; then on increasing the temperature sulfur dioxide will issue into (*B*). Next start the reaction in (*E*). Using a low rate of gas evolution, radiation is rapid enough to keep the temperature of the reaction vessel (*B*) below the melting point of the chamber crystals. If the flask (*B*) is dry, beautiful "chamber crystals" will soon coat the walls.

After the sulfur dioxide ceases to form, the bottle (*D*) is removed and the apparatus allowed to cool. Then the flask (*B*) is detached and 10 cc. of water added. Shake the flask to promote reaction between the crystals and water. Then test a small portion of the liquid by adding 5 cc. of a solution of barium chloride. The residue in the test tube (*A*) may be examined by adding a few drops of hydrochloric acid and testing the issuing gas with paper moistened with lead acetate solution.

The sulfur dioxide generator functions smoothly when the reactants are well mixed. If this is not done the sulfur may distil into the outlet tube and plug it.

Several alternative methods can be employed for generating the sulfur dioxide; *e. g.*, copper and hot concentrated sulfuric acid, or still more quickly by dropping an acid on sodium sulfite. However, the oxidation of sulfur is more nearly like the Chamber process. If desired the test tube (*A*) may be replaced by a tube open at both ends. Then the sulfur can be burned in a stream of air which is bubbled through the train.