Tested Demonstrations

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Catalytic Oxidation of Ammonia: A Sparkling Experiment

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The usual lecture demonstrations of the catalytic oxidation of ammonia involve the use of preheated platinum or copper metal coils (*1*, *2*). Several transition metal oxides can be used as catalysts for oxidizing ammonia, but chromium(III) oxide, Cr_2O_3, allows this reaction to be demonstrated in a really spectacular way, generating sparks in a flask of ammonia gas. Several lecture demonstrations on Cr_2O_3 catalysis have previously appeared in this *Journal* (*3*), including that on the oxidation of ammonia, but we have now expanded it with a truly vivid effect. The experiment described below is relatively simple, but surprisingly, we could not find any mention of it in the published literature.

Experimental Procedure

A suitable form of chromium(III) oxide is best prepared by thermal decomposition of ammonium dichromate, by the well-known "volcano" experiment (*4*, *5*). This yields an active catalyst with light particles. Commercially available Cr_2O_3 reagent is generally not suitable for this particular experiment. Once prepared from the dichromate, the catalyst can be stored for a long time without loss of activity.

CAUTION: Chromium(VI) compounds and chromium(III) oxide are irritants for the skin and eyes and especially the respiratory tract if inhaled (*6*). In addition, some chemical suppliers note that chromium(VI) compounds, including $(NH_4)Cr_2O_7$, are suspected carcinogens, the risk being related to the length of exposure. Gloves and safety goggles should be worn when handling $(NH_4)_2Cr_2O_7$ and Cr_2O_3. The "volcano" experiment should be performed in a well-ventilated area. If performed on an open bench, this reaction can be carried out in large beaker covered with several layers of fine wire gauze as suggested in ref *4*.

The demonstration of the catalytic oxidation of NH_3 is best performed in a darkened room. The effect is clearly visible even in a large room, but a video projector (if available) can be used. The capacity and shape of the vessel in which the reaction is demonstrated does not matter much, for the experiment works well in a 1-L conical flask or in a 20-L bottle. For our demonstrations we used a 10-L round-bottom flask. Approximately 15–20 mL of concentrated ammonia solution (35% [0.880 g cm^{-3}] or higher) is placed in the flask, which is then stoppered and swirled to mix the ammonia with the air in the flask. Chromium oxide is heated in a deflagrating spoon in a Bunsen flame until it begins to glow red, for ca. 5–10 s. The stopper is then removed and the catalyst is gently tipped from the spoon into the flask. A fountain of red sparks is immediately produced, momentarily coming out of the neck of the flask (CAUTION!). It is wise at this time to replace the stopper to trap the hot particles and the smell of ammonia. Particles of the catalyst remain suspended in the flask, glowing red and yellow, some staying hot for up to a minute. The addition of the catalyst and the reaction can be repeated several times. At the end of several additions the flask is seen filled with a white cloud of water vapor. Ideally, the whole demonstration ought to be performed in a hood or next to a source of down-draft ventilation. However, if the chromium(III) oxide is prepared in advance, then there is very little dust from Cr_2O_3 particles in the demonstration itself and the main source of irritation is just the smell of ammonia.

The mechanism of this reaction is quite complicated. Most of the ammonia is oxidized to nitrogen:

$$4NH_3 + 2O_2 = 2N_2 + 6H_2O \qquad (1)$$

In principle some ammonia may oxidize further, forming nitrogen(II) oxide, ultimately producing ammonium nitrate:

$$4NH_3 + 5O_2 = 4NO + 6H_2O \qquad (2)$$

$$4NO + 4NH_3 + 3O_2 + 2H_2O = 4NH_4NO_3 \qquad (3)$$

However, we tested solutions after several experiments and were unable to detect the presence of nitrate ions. Therefore, if any nitrate is formed its concentration is very small. The absence of nitrate may be due to the reaction between NH_3 and NO to form N_2, N_2O, and H_2O, catalyzed by chromium(III) oxide (*7*, *8*). A detailed investigation of this apparently simple reaction can thus be a topic for a student project.

We have used this experiment successfully in several public chemical "magic shows". It works especially well when a sequence of reactions is undertaken. First, the catalyst is prepared by the "volcano" experiment, and then it is used to oxidize ammonia ("sparks in the flask"). Finally, the rest of the prepared chromium(III) oxide can be used to demonstrate the thermite reaction (*5*).

Literature Cited

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