# THE STANDARDIZATION OF NITROUS OXIDE.

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The tests given in the U. S. Pharmacopœia for laughing gas are inadequate as they do not enable the analyst to form a correct opinion as to the quality of the gas. It seems hardly possible to find on the market a cylinder of nitrous oxide which does not strictly fulfil all the requirements of the official tests, yet, at the same time, there is such a wide range in its chemical composition and, sometimes, in its effect upon the patients, that a more searching inquiry into the impurities of this useful gas becomes most urgent.

To begin with it is evident that the tests for "carbon dioxide, halogens, acids and bases, reducing substances" as officially given are not sufficient and delicate enough to detect the really poisonous other oxides of nitrogen, which are the cause of harmful effects in the majority of cases. As is well known, no reaction is 100% perfect, and in the manufacture of nitrous oxide, *e. g.*, the heating of ammonium nitrate until it decomposes according to the equation: $NH_4NO_3 = N_2O + 2H_2O$, there is the possibility, especially at the beginning and end of the reaction, that ammonia, nitrogen, nitrogen dioxide, nitrogen trioxide, nitrogen tetroxide, and nitrogen pentoxide have been formed. As a matter of fact, in slow heating the ammonium nitrate thermal dissociation takes place, traces of ammonia are evolved, and the liquid mass becomes more acid in character, while, especially toward the end, the other oxides of nitrogen are formed in small amounts.

The removal of these impurities must be the aim of every manufacturer—no matter what source of ammonium nitrate he is using. Because of the poisonous nature of nitric oxide it is of paramount importance to carefully analyze every lot of nitrous oxide for the amount of nitric oxide, which should not exceed ten parts per million by volume. This single analysis is a much better criterion for the quality of the gas, than the official tests for halogens, hardly ever present, or carbon dioxide, whose presence in traces might even be beneficial.

The following methods, which among others have become part of the routine work of the plant-chemist, are submitted in the hope that they may aid in raising the standards required for nitrous oxide gas and thereby give to the user the added confidence of a more reliable product.

## 1. OTHER OXIDES OF NITROGEN.

This method is adapted from the one used by the Bureau of Mines in analyzing mine gases.[1]

Two one-liter Erlenmeyer flasks are filled with nitrous oxide gas, while two other ones are used as blanks and contain only air. Into each of these four flasks is put a mixture of 5 cc. $H_2O_2$ and 5 cc. 10% NaOH. Shake repeatedly and let stand for about ten minutes, pour the liquid of each flask into a separate 50 cc. flat porcelain dish and evaporate to dryness on a water-bath (avoid temperatures higher than 100° C.). To the dry residue of each porcelain dish is added 5 cc. diphenylsulphonic acid, mix well by inclining the dish—add ten cc. water, pour the mixture into a 100-cc. Nessler tube, rinse porcelain dish with more water, and add 15 cc. of a mixture of equal parts concentrated ammonium hydroxide and water, fill up to 100 cc. with distilled water.

All four solutions should have the same colorless tint. Any slight pale yellowish green tint indicates oxides of nitrogen. As nitrates may have been present in

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[1] Bureau of Mines, *Tech. Paper* 249, 1921, "The Detrm. of Oxides of Nitrogen."

the reagents, there should be no perceptible difference in the shades of all four solutions.

The amount of nitric oxide can be determined colorimetrically by preparing a series of Nessler tubes with an added amount of nitrate equivalent to 8, 16, 24, 35, 45, 55, 75 and 100 parts per million by volume. This series is sufficient and enables one to determine the other oxides by comparison, *e. g.*, if the two blanks indicate 16 parts per million, the two samples should likewise be of the same shade. A detailed description of preparing the diphenylsulphonic acid, and the standard solutions for this test, are found in the before-mentioned paper of the Bureau of Mines.

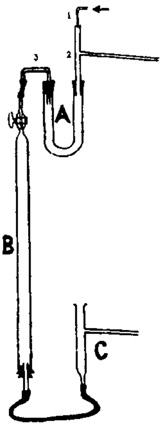
### 2. OTHER IMPURITIES.

Qualitative tests should be made for the following impurities which may, under certain circumstances, be present:

(*a*) Halogens and hydrochloric acid.
(*b*) Carbon dioxide.
(*c*) Sulphuric acid and sulphur dioxide.
(*d*) Ammonia.

These tests are readily executed by using a calibrated glass-tube or capillary tube attached to the valve and immersed in a test-tube with water to which one to three drops of reagents—(*a*) silver nitrate, (*b*) and (*c*) barium chloride, (*d*) litmus—had been added. By counting the bubbles passing through the test-tube per second, the total amount of gas passing through can be estimated. A convenient speed is about two bubbles per second for four to five minutes.

(*e*) Lubricating oil (from compressor). Invert a cylinder, let stand for about one minute and give a short blast of gas upon a piece of filter paper fastened to the wall. A quick wide opening of the valve and closing will produce a small cone of solidified nitrous oxide snow upon the paper which should be a pure white. The snow should evaporate without leaving the slightest trace of a residue. If there is a brown residue it may be either oil or iron rust or both. This is determined by adding a few drops of ether—in the case of oil a ring will be formed. Afterward add a drop of dilute hydrochloric acid and a few drops of potassium ferrocyanide, and observe the formation of Prussian blue if iron is present.



Absorption of Nitrous Oxide by Running Water. 1, water inlet; 2, overflow; A, u-tube; 3, capillary; B, burette; C, leveling tube.

### 3. NITROUS OXIDE, QUANTITATIVE.

To accurately determine the nitrous oxide content requires more elaborate apparatus in which the nitrous oxide is burned in a given quantity of hydrogen and the residual gases measured. The ordinary pharmaceutical equipment is not suited for this analysis, therefore the following rapid, approximate absorption-method was devised, which can be made in any laboratory and does not require any special apparatus. The results indicate approximately the dilution of air and manufactured nitrogen found in the nitrous oxide gas.

A 100 cc. burette, $B$, is inverted and attached to a large U-tube, $A$ as shown in the illustration, the other end is closed with a stopper with glass-tube connected to a bulb or tube $C$, by a 1 or 2 foot rubber tubing. To make an analysis proceed as follows:

Fill the burette entirely with water and connect with U-tube so that no air bubbles are inclosed. Let the water run for 10 minutes and note the air-space produced in the burette ($A$). Disconnect and fill burette again with water, close stopcock and attach to nitrous oxide cylinder—open valve slowly and fill burette with nitrous oxide to zero (or 100 cc.) mark. Attach to U-tube and let the water run for ten minutes. Note the amount of remaining air space ($B$). Fill burette again with water and let the water run through it for ten minutes ($A'$). Repeat the test by using nitrous oxide ($B'$) and finally repeat by filling again with water ($A''$). Adding $A$, $A'$ and $A''$ and dividing by three will give the correction, that is, the amount of air liberated in ten minutes from the tap water. Adding $B$ and $B'$, and dividing by two will give the amount of nitrous oxide absorbed by the water plus the air liberated from the water. Subtracting A from B will give the percentage of air and other non-absorbable gases of the nitrous oxide. No gas calculations are necessary as the temperature and barometric pressure remain relatively the same. Naturally each reading must be taken by leveling the bulb $C$.

A typical analysis of nitrous oxide is as follows:

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| 1. | Other oxides of nitrogen (colorimetric) | less than 10 pts. per million |
| 2. | Halogens and hydrochloric acid (2000 cc. passed through 25 cc. water) | none |
| 3. | Carbon dioxide (2000 cc. passed through 25 cc. water) | none |
| 4. | Sulphuric acid and sulphur dioxide (2000 cc. passed through 25 cc. water) | none |
| 5. | Ammonia (2000 cc. passed through 25 cc. water) | none |
| 6. | Lubricating oil | none |
| 7. | Iron and rust | none |
| 8. | Air dilution | 1.5% by volume |
| 9. | Nitrous oxide (by difference from absorption with water) | 98.5% by volume |

While this is not a complete quantitative analysis of nitrous oxide, it will be entirely sufficient for practical purposes and can be made in a pharmaceutical laboratory with simple apparatus. Especial emphasis must be given to the test for other oxides of nitrogen and to the approximate quantitative analysis which enables one to determine whether the air dilution is small or large within 0.5%. For more exact work the amount of oxygen and moisture must be determined, also the nitrous oxide by slow combustion—but these methods are too complicated for the general practice and restricted to the chemical laboratory.

A revision of the standards for nitrous oxide in the Pharmacopœia is essential, and if these lines should suggest and aid the pharmaceutical profession toward this end, they will have accomplished their purpose.

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