

Total fume samples were obtained with the sampler described by Silverman and Ege.⁶ Oxides of nitrogen were collected simultaneously in a pair of evacuated gas sample flasks connected in series. One flask was analyzed for nitrogen dioxide and the other for total oxides.

In order to determine nitric oxide (NO), the gas sample flasks were followed (experiments 4, 5 and 6) by two freezing traps similar to those described by Tebbens and Drinker,⁷ placed in a liquid nitrogen bath. These traps remove higher oxides of nitrogen and leave only nitric oxide. Low temperature vapor pressure properties of the oxides, indicated in table 2, show that at least 77.5 p. p. m. of NO can escape this trap, whereas all others should be retained. Gases were drawn through the traps continuously at a rate of 1 L. per minute. It was necessary between samples to warm the traps to allow condensed oxygen and carbon dioxide to volatilize. By this means it was possible to determine NO in addition to NO₂ and total oxides. In run 6 two grab samples were collected after the freezing trap, one of which was analyzed for NO as total oxides; the other was analyzed for NO as NO₂. This gave an indication of the rate at which NO is oxidized to NO₂ in the short period of passing into the flask containing an absorbing reagent.

The analytic method employed for nitrogen dioxide was that using β -naphthylamine 6-8 disulfonic acid reagent with the α -naphthylamine method as outlined in Snell and Snell.⁸ This method detects nitrogen dioxide (NO₂), nitrogen trioxide (N₂O₃) and nitrogen tetroxide (N₂O₄). Harrold and associates⁹ indicated the desirability of using this method. We found it was necessary to prepare fresh standards daily to get uniform and accurate results.

TABLE 2.—Low Temperature Properties of Oxides of Nitrogen*

Gas	Temperature Range, C.	Vapor Pressure at -196 C.,† Mm.Hg.	Gas Escaping Trap at Atmospheric Pressure, P.p.M.
NO	-200 to -161 solid	0.059	77.5
NO ₂	-100 to -40 solid	1×10^{-24}	0.000
N ₂ O ₅	-30 to +30 solid	1×10^{-26}	0.000
N ₂ O ₃	-25 to 0 liquid	3.98×10^{-17}	0.000
N ₂ O	-144 to -90 solid	1.2×10^{-6}	0.001

* Based on International Critical Table data.

† Temperature of liquid nitrogen bath.

For total nitrates, the United States Bureau of Mines' method¹⁰ using phenoldisulfonic acid was employed. All samples were analyzed on a photoelectric colorimeter (Klett or Coleman).

Although it has been indicated that nitrous oxide may be present, it is very difficult to determine this gas, since there are no direct procedures. The methods available are too complicated to use in a situation such as resulted in this experiment, and therefore attempts to determine N₂O directly were unsuccessful. Attempts to oxidize N₂O to total nitrates or to reduce it to ammonia failed, since only 3 per cent conversion was attained in one case and less than 1 per cent in the other. This is in agreement with other studies which indicate the chemical stability

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