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MEMORANDUM REPORT BRL-MR-3860

BRL

REMOVAL OF NO AND NO₂
FROM CONTAMINATED ATMOSPHERES

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SEPTEMBER 1990

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U.S. ARMY LABORATORY COMMAND

BALLISTIC RESEARCH LABORATORY
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PREFACE

In the summer of 1989, the Ballistic Research Laboratory (BRL) participated in the George Washington University Science and Engineering Apprentice Program (SEAP). This program brings high school students into the laboratory so that students who have an inclination toward technical careers may be given some exposure to this type of work. As part of the SEAP, three high school students, Matthew Schroeder, Lisa Copland, and Tanya Swiderski, were assigned to two BRL professionals.

Plans for the summer work were formulated in the spring of 1989 so that an effective program could be conducted by the students under supervision by the professionals. It was extremely advantageous that all the students had previous laboratory experience. This allowed them to quickly pick up the various aspects of the program, even though the analytical techniques were new to them.

The authors wish to express their appreciation to the George Washington University SEAP for providing the mechanism which allowed the students to gain exposure to a scientific laboratory.

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ACKNOWLEDGMENT

The authors wish to express their gratitude to Mr. Rick Losey, an employee of Dynamic Science Incorporated (DSI), assigned to the Combat Systems Test Activity at Aberdeen Proving Ground. He very generously volunteered to calibrate our main analytical instrument, the BINOS IR-VIS/UV analyzer. His extensive experience with over 50 of these instruments ensured his strong qualifications.

Mr. Richard Schwanke of the BRL Safety and Health Physics Office checked over the procedures involved in handling the toxic gases to ensure safety of laboratory personnel. His guidance was appreciated and followed.

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1. INTRODUCTION

The United States Army has a strong commitment to the survivability of personnel in vehicles which have been hit in combat situations. There is a continuing emphasis on measuring any toxic gases which may be formed inside the vehicles. Two of the gases of interest are nitrogen dioxide (NO_2) and nitric oxide (NO). These gases are produced when gun propellants burn. It is to be expected that if ammunition which is present in a vehicle's crew compartment is struck when the armor is defeated by a penetrator, the propellant will burn.

It is not obvious how much NO_2 and NO will be generated in a crew compartment. The amount of propellant available in each round is important. The number of rounds involved is important. The size of the crew compartment, as well as the amount of ventilation, will also affect the concentration of these gases. NO_2 and NO are of particular importance, since the currently fielded personal protective system used in vehicles such as the M1 tank are not fully effective in removing these gases (Ripple 1988).

Our approach has been to investigate materials which could possibly be injected into the air of the crew compartment to remove the NO_2 and NO . It is obviously important that the materials injected into the air be non-toxic, especially in the case where injured personnel, who would be unable to don protective gear, are present. The work documented in this report concerned only non-toxic chemicals which could be used to remove NO_2 and NO , not methods of injecting these materials into the crew compartment.

2. EXPERIMENTAL

2.1 Liquid Materials. Since NO_2 has a definite solubility in water, a water mist was an obvious starting point for removal of NO_2 from air. Water and a one-molar solution of sodium bicarbonate in water were the only liquids employed in this work.

2.2 Powder Materials. The other materials tested were all powders. Some were available as fine powders; others were ground into fine powders. The solid materials are listed on the following page.

- Monnex[®], a carbamate-based, fire-extinguishing agent, $\text{KC}_2\text{N}_2\text{H}_3\text{O}_3$, made from urea and potassium bicarbonate.
- Purple K, a potassium bicarbonate-based, fire-extinguishing agent, KHCO_3 .
- Baking Soda, a sodium bicarbonate-based material sometimes used as a fire-extinguishing agent, NaHCO_3 .
- MAP, a monoammonium phosphate, fire-extinguishing agent, NH_6PO_4 .
- Diammonium Phosphate, a commonly used fire-extinguishing agent, $\text{N}_2\text{H}_9\text{PO}_4$.
- Boric Acid, a white crystalline powder, H_3BO_3 .
- Urea, a white crystalline powder, $\text{N}_2\text{H}_6\text{CO}$.
- Alum, a white crystalline powder, $\text{KAl}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$.
- Cab-O-Sil[®], an amorphous, fumed silica material added, where required, to make the powders free flowing.

2.3 Additional Materials. In addition to the powders, the following materials were also used in the laboratory procedures:

- Commercial Grade Argon, Ar.
- Commercial Grade Nitrogen, N_2 .
- Commercial Grade Nitrogen Dioxide, NO_2 .
- Commercial Grade Nitric Oxide, NO.
- Certified Calibration Gas Mixture of NO_2 in Air.
- Certified Calibration Gas Mixture of NO in N_2 .

3. INSTRUMENTATION

The analytical instrument used in these experiments was a BINOS dual-channel IR-VIS/UV gas analyzer. The first channel utilized non-dispersive infrared to analyze nitric oxide. This channel was equipped with an optical filter, which reduced water vapor interference so that a gas saturated with water vapor at 20° C would give a NO reading of less than 10 parts per million (ppm). Carbon monoxide (CO) was not present in the test gases used. However, if it had been, the optical filter was also capable of reducing the CO interference. The NO channel would give a reading of less than 10 ppm in the presence of a full atmosphere pressure of CO.

[®] Trademark of ICI Incorporated

[®] Trademark of Cabot Corporation

The second channel utilized non-dispersive ultraviolet to analyze nitrogen dioxide. The two sections of the instrument were internally connected in series so that the gases flowed first through the NO₂ analysis cell, then through the NO analysis cell. Direct reading analog dials indicated the concentration of NO₂ as 0-800 ppm and of NO as 0-2000 ppm. Analog voltage outputs from each channel were recorded using a strip chart recorder. A vacuum pump was used on the exhaust side of the instrument. The gas flow rate through the instrument was set to the manufacturer's specification using a flowmeter. The vacuum pump and flowmeter were situated in the laboratory fume hood to prevent exposure of personnel to the NO₂ fumes. The BINOS gas analyzer is shown in Figure 1.

4. REACTION CHAMBERS

The reactions of interest were carried out in two types of gas sampling bags. The first type of bag used was a Teflon[®] bag of 50-liter capacity, with a nickel-plated brass inlet valve. The second was a 4.7-liter Teflon[®] bag, also with a nickel-plated brass valve.

Polyethylene tubing was used to connect the gas bag to the BINOS instrument since Teflon[®] tubing was not available.

5. MIST GENERATOR

A commercial airless paint sprayer was used to generate the water mist and the 1 mol sodium bicarbonate solution mist in the 50-liter Teflon[®] gas bag. The amount of mist generated was controlled by nozzle opening and the time of spraying. The spray gun was inclosed inside a protective bag, except for an opening at the nozzle. This bag was to prevent contact of nitrogen dioxide and nitric acid with the spray gun. The bag was taped around the sprayer. The spray gun in its protective bag is shown in Figure 2.

The 50-liter Teflon[®] bag was slit open at one corner. The protected sprayer was placed inside the gas bag, and the opening taped shut on the base of the sprayer. The seal prevented gas leakage from the bag while allowing the electrical line and the plastic dip tube, which connected the sprayer to the reservoir, to extend from the bag. The sprayer in the gas bag is shown in Figure 3.

[®] *Registered Trademark of DuPont Company*

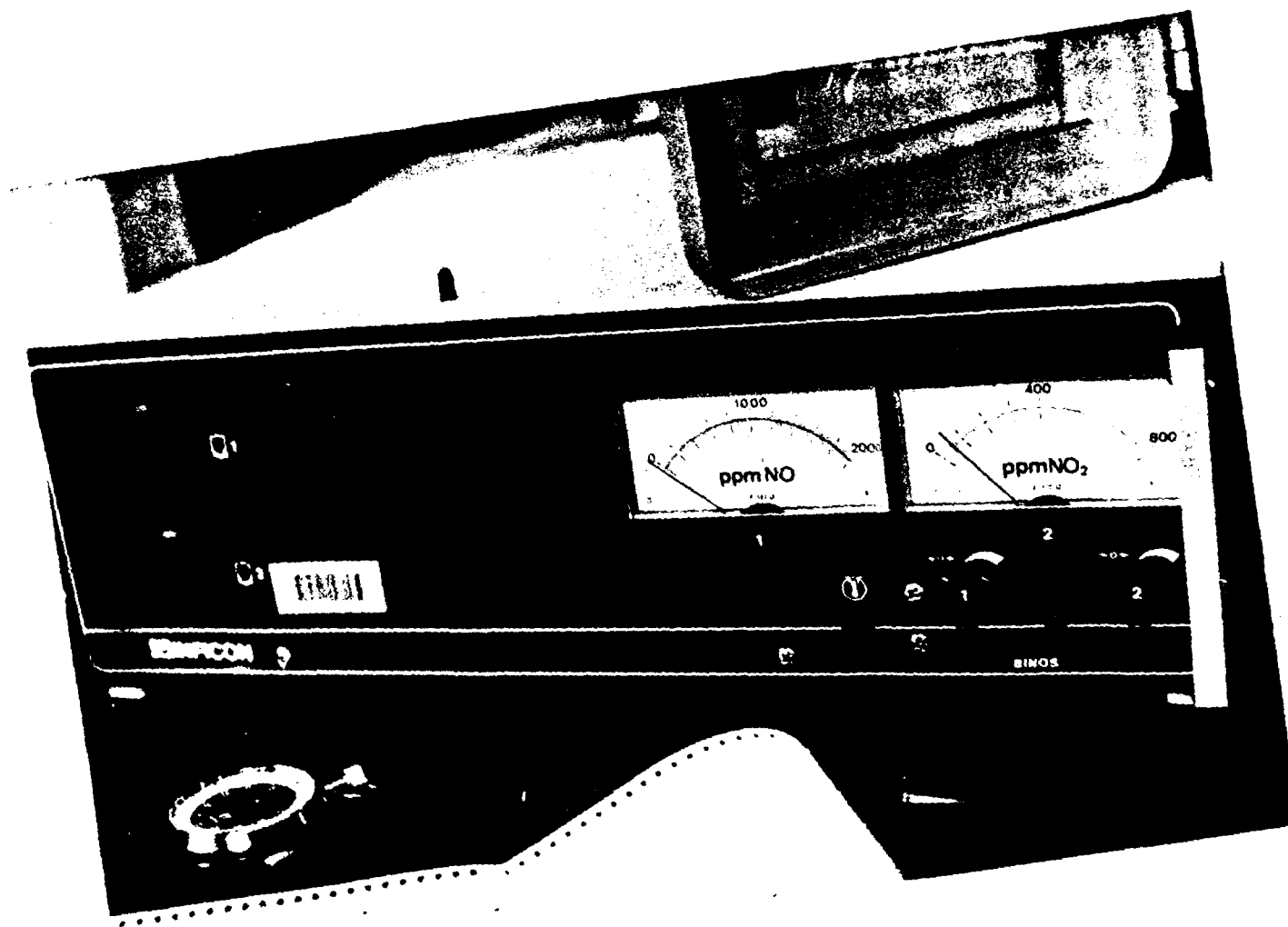


Figure 1. BINOS Gas Analyzer.

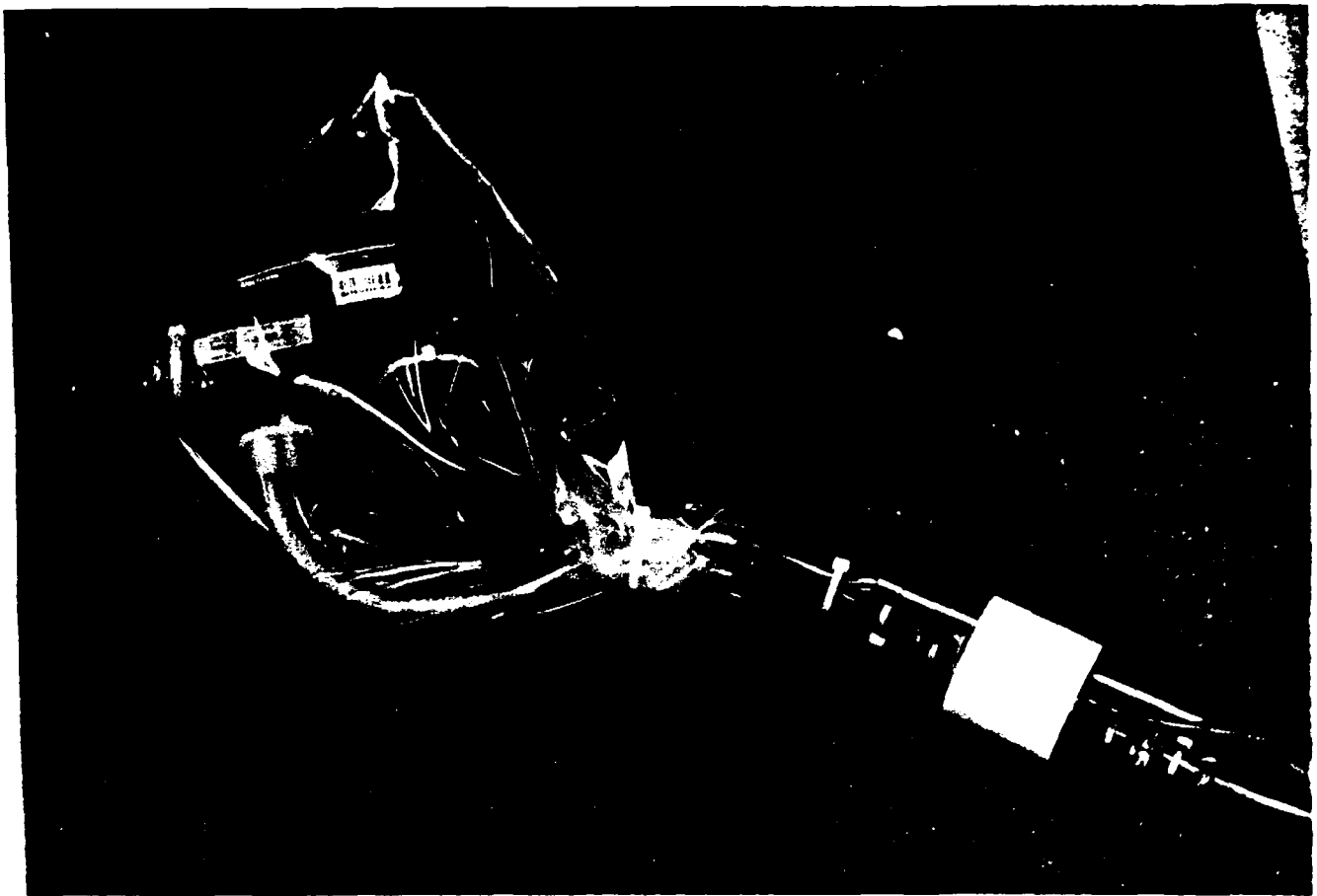


Figure 2. Spray Gun in Protective Bag.



Figure 3. Sprayer Sealed in Teflon[®] Gas Bag.

Calibration tests showed that a 6-sec burst at full nozzle opening converted 36 ml of water into mist. At the smallest nozzle opening, 432 ml of water were consumed in 360 sec (6 min).

6. POWDER DISPENSER

The powders used in these experiments were dispensed from small plastic containers sealed inside the gas bags. Some containers were made from plastic 35-mm film canisters, cut down to one-half height. A second type of plastic container was a small polyethylene bag.

The powder to be tested was weighed into the dispenser, which was then sealed in the gas bag (see Figure 4). The powder was manually dispensed into the NO_2 or NO gas after the proper initial concentration of gas had been established.

7. PROCEDURE

The safety of the personnel performing these experiments was of paramount importance and a safe procedure was set up before using the toxic gases. Adherence to the procedure was mandatory. The personnel were informed of the reasons for each step in the procedure. Checks were made to ensure continued compliance with safe handling of the gases.

Important points of the procedure were:

- a. All toxic gases, both neat and low concentration calibration mixtures, were stored in a fume hood. The fan blower was kept in operation 24 hours a day.
- b. The toxic gas cylinders never left the fume hood. Tubing connections were made to the cylinder regulators inside the hood.
- c. When toxic gases were to be added to the Teflon[®] gas bags, the bags were put into the hood. Connections were made from the gas regulators to the bags, and gases were transferred into the bags. All operations were done inside the fume hood.

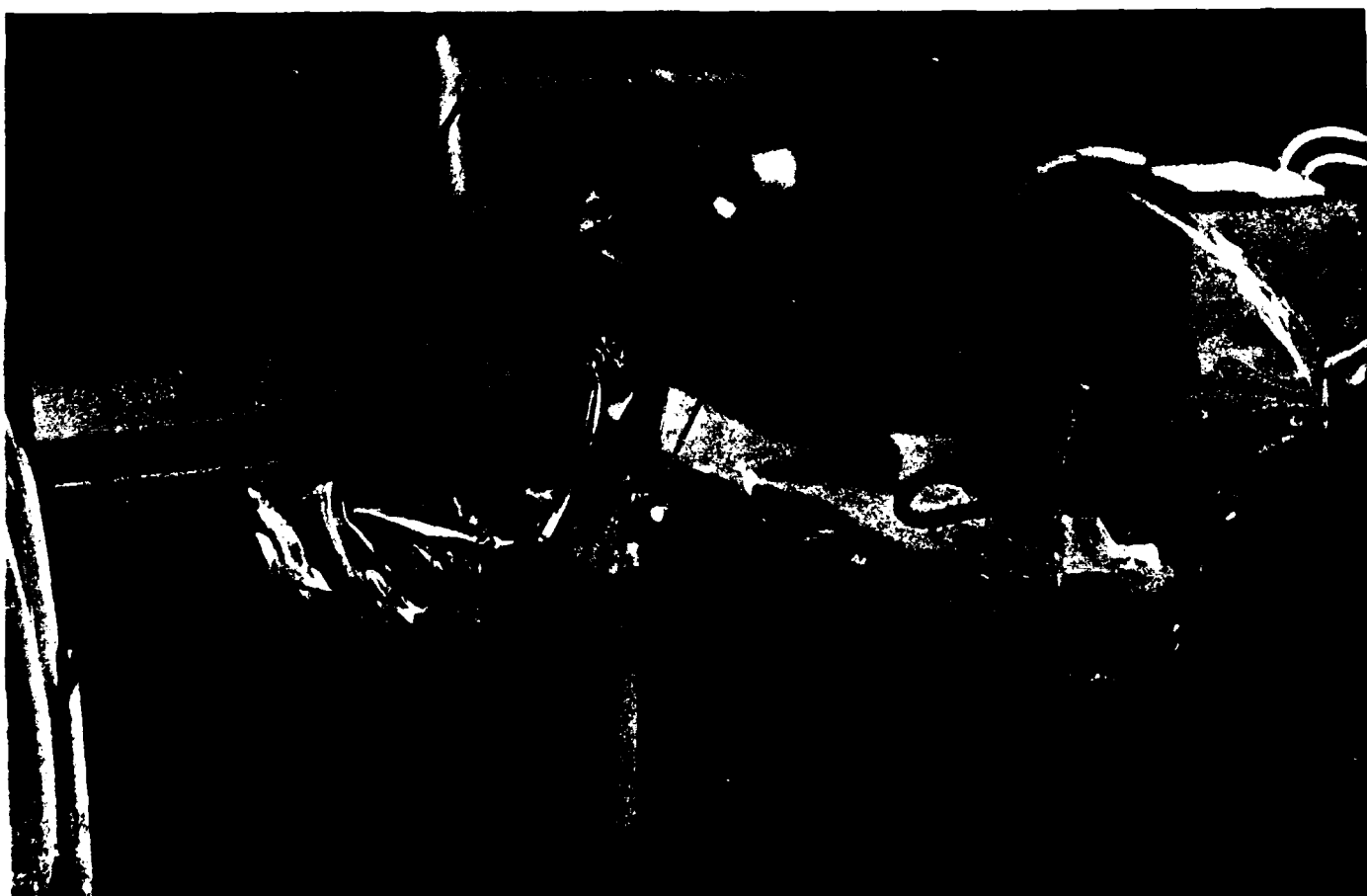


Figure 4. Gas Bags Used for Powder Tests.

d. When gas was to be stored inside a Teflon[®] gas bag for any length of time, the bag with gas was kept inside the hood.

e. The exhaust from the BINOS gas analyzer was routed through tubing into the fume hood. The gases were not allowed into the air in the laboratory.

For a typical powder test, if the material to be tested was finely divided and free-flowing, it was used as received. If not, the material was ground with 2% by weight Cab-O-Sil[®] until a finely divided, free-flowing powder was obtained. One-half gram of powder was normally used. This weight was chosen, since, when fluffed up into the 4.7-liter volume of the Teflon[®] gas bag, it approximated 100 mg/l concentration of powder. This is the concentration of fire extinguishing powder normally considered sufficient to render a volume non-flammable. Many of the powders are fire extinguishing agents, hence are often used at this concentration or higher.

A corner was slit open in a Teflon[®] gas bag. The container, with powder, was placed in the bag. The cut corner was rolled up and sealed to prevent leakage. Air was removed from the gas bag through its metal valve using a vacuum pump. When all air was removed, the valve was closed. The valve was then attached to a gas cylinder and a small quantity of either NO₂ or NO was introduced into the bag. Argon or nitrogen gas was then added to essentially fill the bag. The valve was closed. The bag was connected to the BINOS gas analyzer. The valve was opened and gas flow adjusted by the flowmeter and vacuum pump inside the fume hood. When the BINOS instrument settled down (about 10 sec), a reading was taken. If the concentration of toxic gas was too low, more of the gas was added to the bag. If the concentration was too high, some gas was allowed to pump out, and more argon or nitrogen was added. In this manner, the initial concentration of the gas could be adjusted to an acceptable value.

At this point, the container of powder was opened, and the powder was fluffed up into the gas bag. Continuous readings were taken using the BINOS. A filter in the gas line from the bag to the BINOS protected the BINOS from the powder. Since the Teflon[®] gas bag had only one valve, a continuous gas loop was not possible. The gas was exhausted into the fume hood after analysis. Since the gas bag had no strength, as gas was removed, the volume simply decreased. The pressure in the bag was maintained at one atmosphere at all times.

For a typical mist test, the airless spray gun was sealed into the bag. The dip tube of the sprayer was outside the bag. The bag was evacuated using a vacuum pump. NO_2 was added to the bag through the metal valve. Argon or nitrogen was then added. The bag was connected to the BINOS analyzer, and the concentration of the gas determined. The gas concentration could be adjusted to an acceptable value by the addition of more NO_2 , more nitrogen, or more argon. The dip tube of the sprayer was set in a reservoir of water or a water-based solution. The trigger of the spray gun was depressed for the desired time (the nozzle had been previously adjusted). Continuous analysis was given by the BINOS instrument. The toxic gas concentration was followed for 6 min (typically). By this time, the mist had settled out of the gas. The BINOS was protected from the mist by a filter.

8. RESULTS

8.1 Powders With Nitrogen Dioxide. The effectiveness of the several powders tested in removing NO_2 from the test gas mixture is indicated by the change in concentration data presented in Table 1 and Figure 5. It is clear that certain powders were capable of removing NO_2 . The best of the powders were diammonium phosphate and Monnex[®]. Both of these powders are recognized fire-extinguishing agents and are believed to be non-toxic. When a test was made using a 50-50 mixture of each to make the 1/2-g sample, no improvement was detected. This indicates that there is no synergistic effect when using both materials. A slight improvement was found when 1/2 g of each (1 g total sample size) was used. This indicates that 100 mg/l concentration may be less than optimum for removing NO_2 from the gas phase.

8.2 Powders With Nitric Oxide. In general, the results of the experiments aimed at removing NO from a test gas were not encouraging. In some tests, a small increase in NO concentration was recorded when the powder was added. This may have been due to perturbations in the gas flow through the analyzer when the powder was shaken into the test gas. The changes in concentration of NO when the powders were added are given in Table 2 and Figure 6.

There were only two tests in which substantial reductions in NO concentrations were recorded. These tests were with monoammonium phosphate and with alum. Yet, in each case, these results were not duplicated in similar tests with the same material. There is no obvious explanation for the failure to duplicate results.

Table 1. Effect of Powders on NO₂ Concentration.

Name of Powder	Weight of Powder, grams	Initial Concentration of NO ₂ , ppm	Final Concentration NO ₂ after 5 min, ppm	NO ₂ Change %
Purple K	.5038	640	573*	-16
Purple K	.5282	365	253	-31
Purple K	.5246	345	210	-39
Monnex [®]	.4996	650	512	-21
Monnex [®]	.5238	385	163	-58
Monnex [®]	.5272	445	227	-38
Urea	.5134	295	240	-19
Urea	.5173	340	270	-21
Urea	.5255	370	200	-46
Boric Acid	.5170	300	289	- 4
Boric Acid	.5217	375	308	-18
Diammonium Phosphate	.5229	355	191	-46
Diammonium Phosphate	.5201	380	167	-56
Sodium Bicarbonate	.5195	375	300	-20
			275**	-27
Sodium Bicarbonate	.5196	375	346	- 8
Sodium Bicarbonate	.5284	355	304	-14
Sodium Bicarbonate	.5256	475	480	+ 1
Sodium Bicarbonate	.5198	415	408	- 2
MAP	.5165	290	271	- 7
MAP	.5196	340	312	- 8
Diammonium Phosphate plus Monnex [®]	.2467	530	311	-41
	.2644			
Diammonium Phosphate plus Monnex [®]	.5083	263	112	-57
	.5037			
Diammonium Phosphate plus Monnex [®]	.5075	680	504	-26
	.5081		400***	-41
Alum	.5162	630	748	+19
Alum	.5147	650	766	+18

* Experiment terminated at 4 min

** Value at 6 min

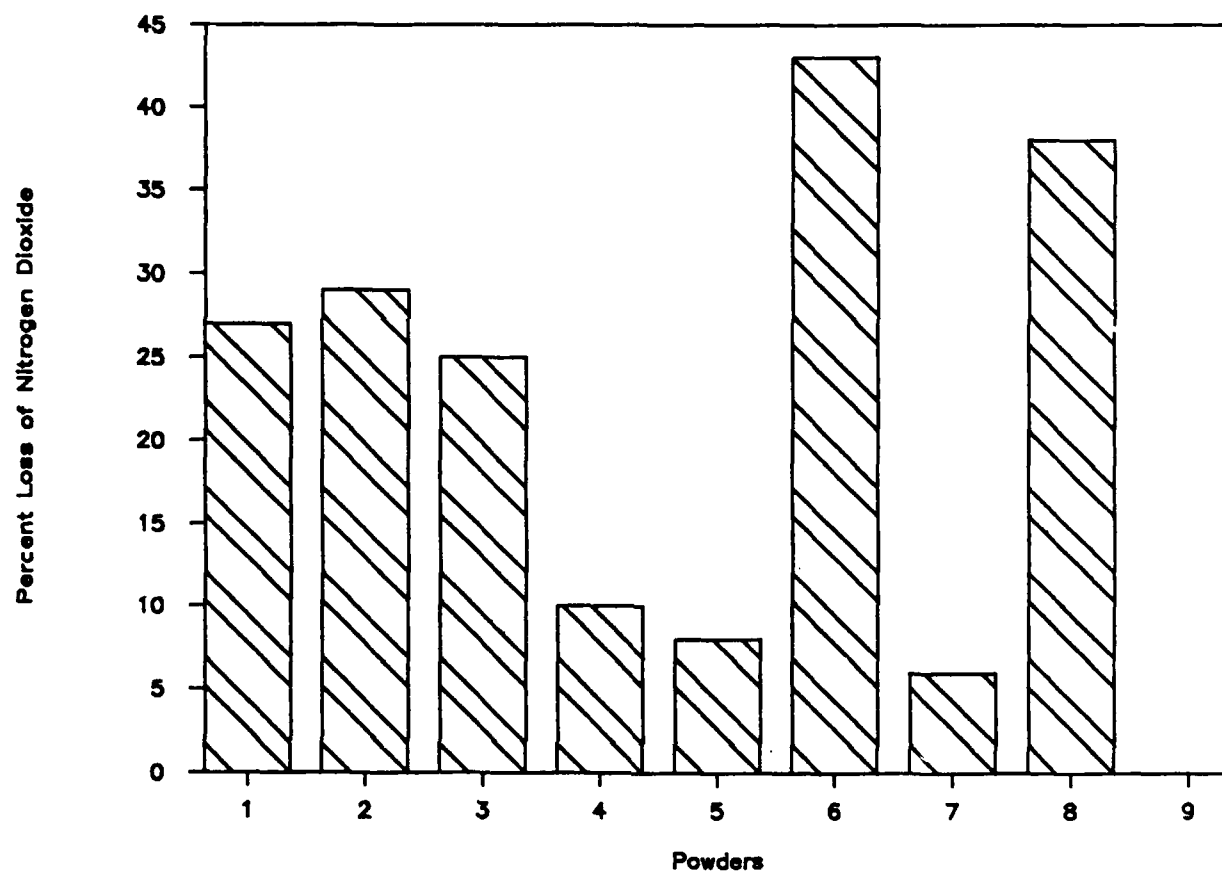
*** Value at 7 min

Table 2. Effects of Powders on NO Concentration.

Name of Powder	Amount, grams	Initial Concentration of NO, ppm	Final Concentration NO after 4 min, ppm	NO Change %
Monnex®	.5030	1100	1120	+ 2
Monnex®	.4995	1500	1560	+ 4
Urea	.5347	1800	1720*	- 4
Urea	.4842	1060	1180	+11
Boric Acid	.5057	1220	1180	- 3
Boric Acid	.4853	1000	980	- 2
MAP	.5051	1880	1800	- 4
MAP	.4982	1080	960	-11
			630**	-42
Purple K	.5118	710	740	+ 4
Purple K	.4833	1580	1600	+ 1
Sodium Bicarbonate	.5640	720	670	- 7
Sodium Bicarbonate	.5126	1410	1280	- 9
Diammonium Phosphate	.4760	1980	1970	0
Diammonium Phosphate	.5234	1980	1900	- 4
Alum	.4929	1750	1700**	- 3
Alum	.5004	750	490	-35

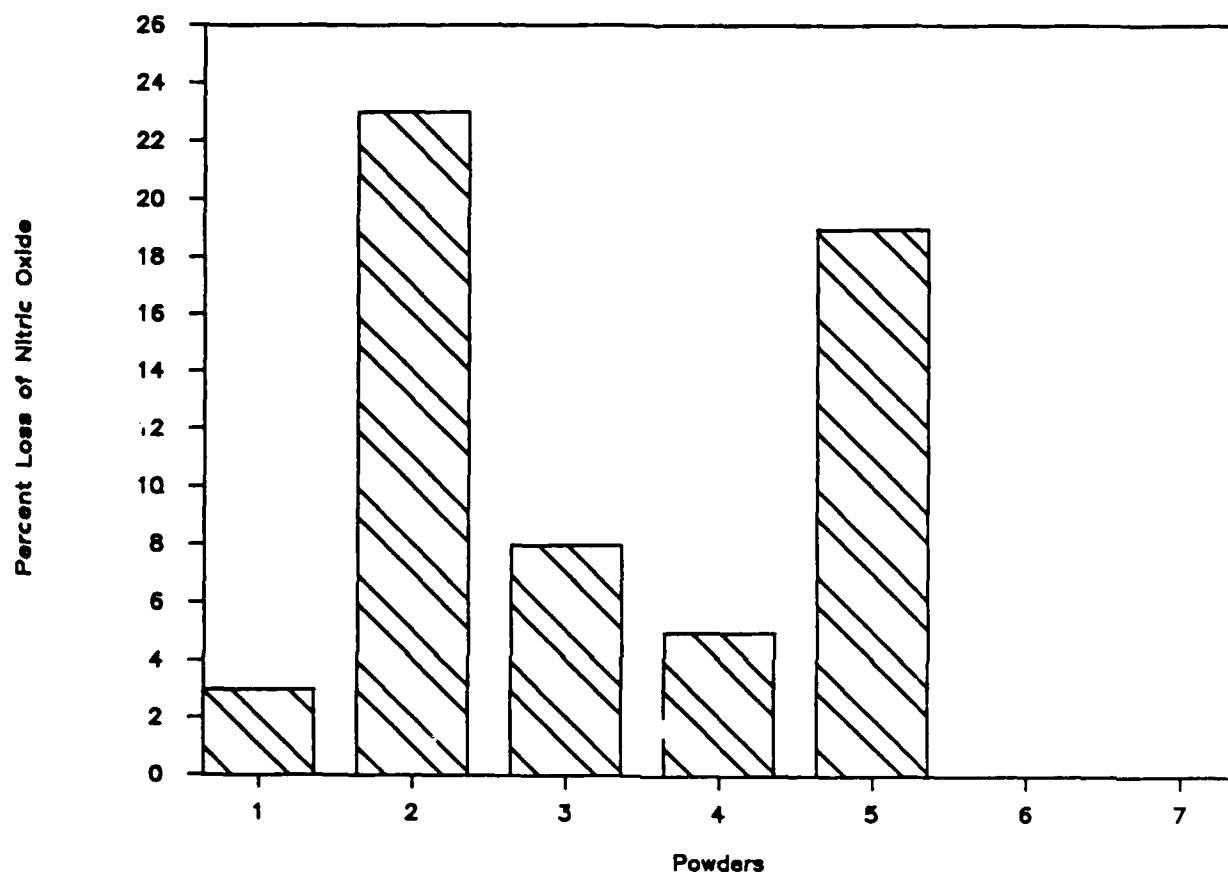
* Experiment terminated at 2 min

** Value at 5-min mark



<u>Procedures</u>	<u>Average % Loss of NO₂</u>
1. Potassium Bicarbonate	27
2. Monnex [®]	29
3. Urea	25
4. Boric Acid	10
5. Sodium Bicarbonate	8
6. Diammonium Phosphate	43
7. Monoammonium Phosphate	6
8. Diammonium and Monnex [®]	38
9. Alum	0

Figure 5. Effect of Powders on Concentration of NO₂.



<u>Procedures</u>	<u>Average % Loss of NO</u>
1. Boric Acid	3
2. Monoammonium Phosphate	23
3. Sodium Bicarbonate	8
4. Diammonium Phosphate	5
5. Alum	19
6. Urea	0
7. Purple K	0

Figure 6. Effect of Powders on Concentration of NO.

8.3 Water Mist With Nitrogen Dioxide. Water mist proved to be the most effective way of removing NO₂ from the test gas. Six of the 12 tests involved 6-sec sprays of 37.2 ml of water in the 20-liter gas bag. These were effective in reducing the concentration of NO₂. The removal of the nitrogen dioxide, however, was not instantaneous. In 1 min, the concentration of NO₂ had fallen by an average of 29% for the six tests. The average loss of NO₂ at the end of 6 min was 44%. In the three tests which involved high NO₂ concentrations (620 to 765 ppm), the NO₂ concentration was still decreasing at the 6-min mark; whereas, in the three low NO₂ concentration tests (210 to 285 ppm), there was no further removal of NO₂ after one or two minutes.

When two sprays of 6-sec duration each (with 2 min between sprays) were used, there was no improvement in the 1-min NO₂ reduction, as should be expected. However, there was some improvement noted in regard to the 6-min loss. It averaged 54% for NO₂ reduction. The increased amount of water spray did remove more of the NO₂.

Continuous, low intensity water sprays (432 ml over six min) were slightly less effective than the short, high intensity sprays at the 1-min mark (only an average loss of NO₂ of 25%), but showed some improvement at the 6-min mark (average 69% loss of NO₂). The increased time and amount of water spray did remove more of the NO₂.

The two tests involving an aqueous 1 M sodium bicarbonate solution in place of water showed no improvement in removing the acidic nitrogen dioxide. In fact, the 6-sec high intensity sprays showed only an average loss of NO₂ at 1 minute of 16%, compared to 29% for water. The results at the end of 6 min were virtually identical (average of 43% for the 1 M sodium bicarbonate versus 44% for water). We have no explanation for the slow initial loss of NO₂ when the basic solution was used. The data for the loss of NO₂ for the 12 spray tests are given in Table 3 and Figures 7 and 8. A comparison of the average NO₂ loss for 1 min and 6 min is given in Figure 9 where the data are divided into five general groups.

It was observed that the NO channel of the BINOS analyzer responded with an increase in NO concentration as NO₂ was removed by the water sprays. This is easily explained by the chemistry involved:

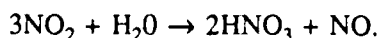
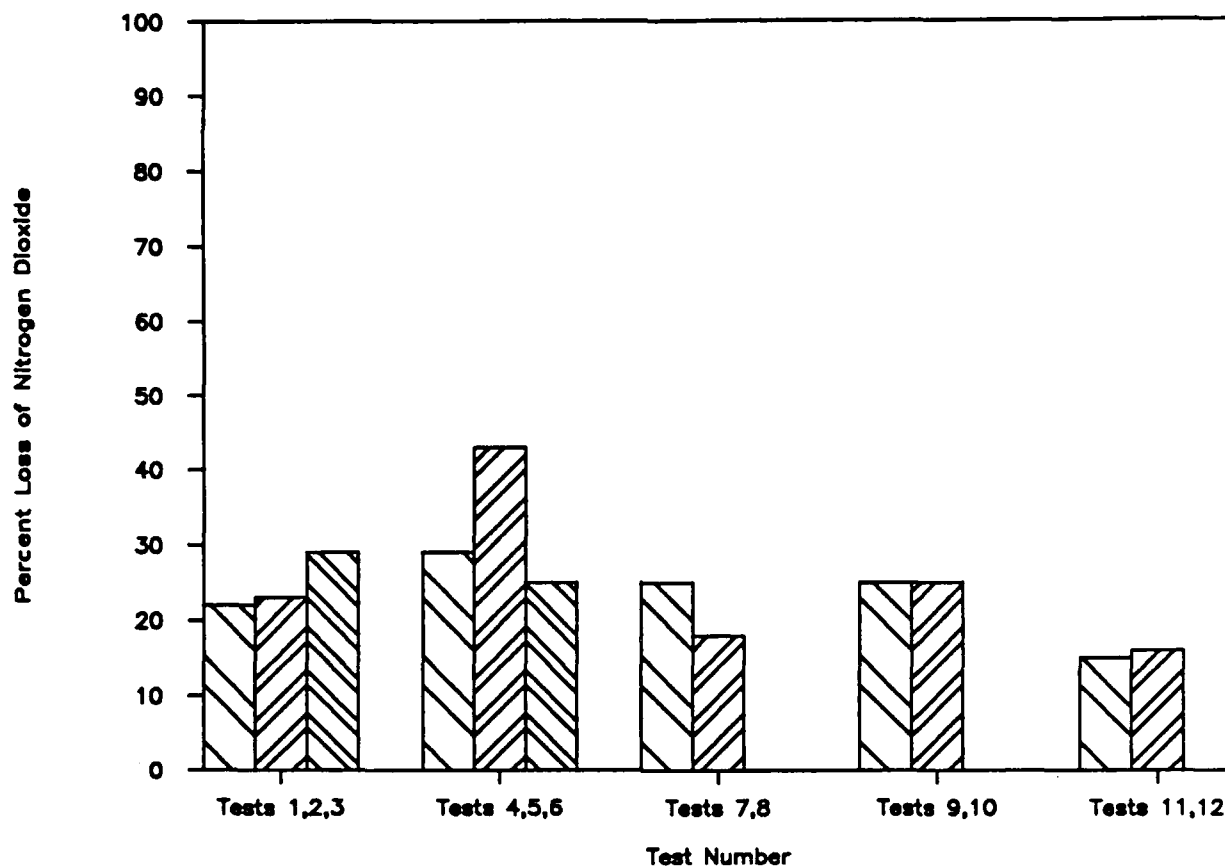


Table 3. Effects of Water Mist Sprays on NO₂ Concentration.

Test	Mist	Spray Time On	Amount of Mist, ml	Initial Concentration of NO ₂ , ppm	% Loss of NO ₂ at 1 min	% Loss of NO ₂ at 6 min	Comments
1.	Water	6 sec	37.2	765	22	46	Further loss of NO ₂ beyond 6 min
2.	Water	6 sec	37.2	735	23	46	Further loss of NO ₂ beyond 6 min
3.	Water	6 sec	37.2	620	29	48	Further loss of NO ₂ beyond 6 min
4.	Water	6 sec	37.2	285	29	48	No further loss of NO ₂ beyond 6 min
5.	Water	6 sec	37.2	210	43	43	No further loss of NO ₂ beyond 1 min
6.	Water	6 sec	37.2	210	25	31	No further loss of NO ₂ beyond 2 min
7.	Water	6 sec, 2 min off, 6 sec	74.4*	750	25	57	Further loss of NO ₂ beyond 6 min
8.	Water	6 sec, 2 min off, 6 sec	74.4*	690	18	51	Further loss of NO ₂ beyond 6 min
9.	Water	6 min	43.2	740	25	66	Further loss of NO ₂ beyond 6 min
10.	Water	6 min	43.2	790	25	71	Further loss of NO ₂ beyond 6 min
11.	Water	6 sec	37.2	790	15	42	Further loss of NO ₂ beyond 6 min
	1 M Sodium Bicarbonate in Water						
12.	1 M Sodium Bicarbonate in Water	6 sec	37.2	790	16	44	Further loss of NO ₂ beyond 6 min

* Total of 2 sprays



Tests 1, 2 and 3 - 6-sec high intensity water spray, high NO_2 concentration

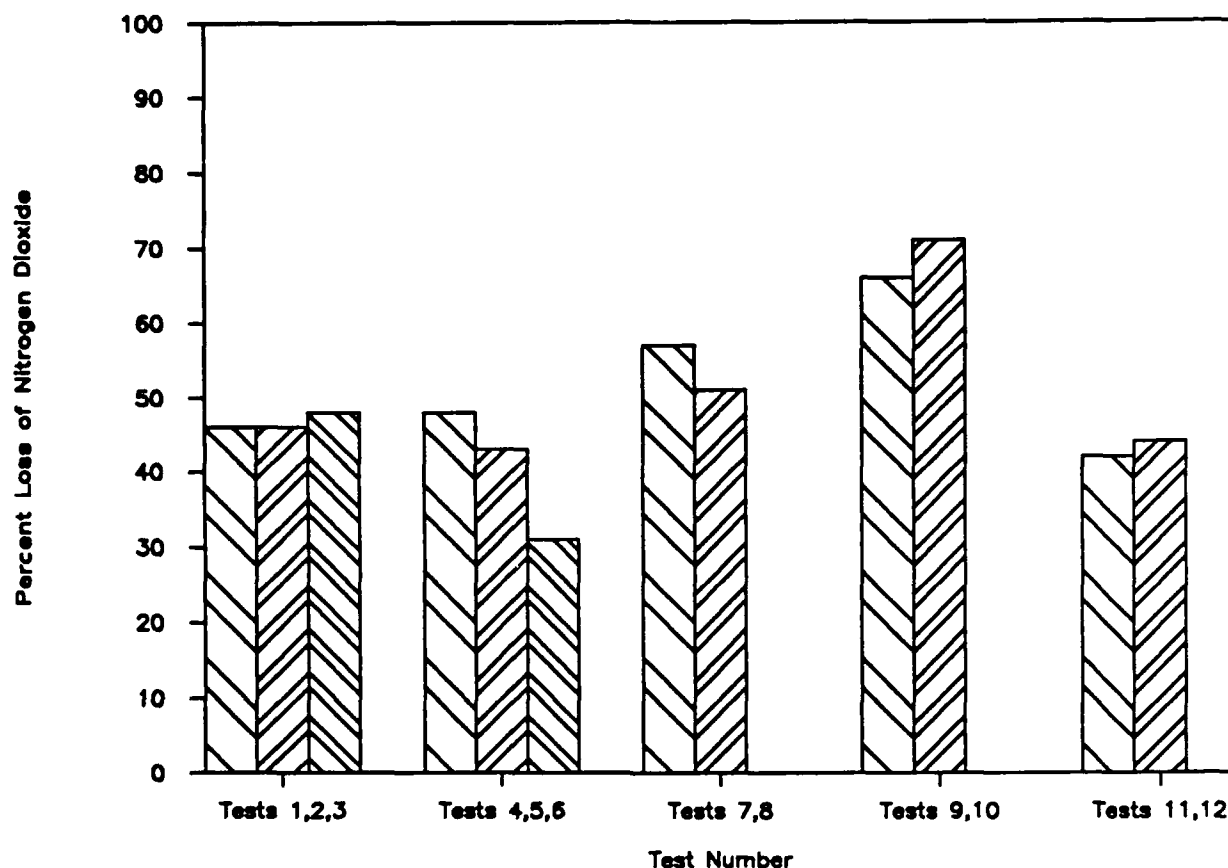
Tests 4, 5 and 6 - 6-sec high intensity water spray, low NO_2 concentration

Tests 7 and 8 - Two 6-sec high intensity water sprays, high NO_2 concentration

Tests 9 and 10 - 6-min low intensity water sprays, high NO_2 concentration

Tests 11 and 12 - 6-sec high intensity spray of 1 mol sodium bicarbonate solution, high NO_2 concentration

Figure 7. Loss of NO_2 in One Minute Due to Water Mist Sprays.



Tests 1, 2 and 3 - 6-sec high intensity
water spray, high NO_2
concentration

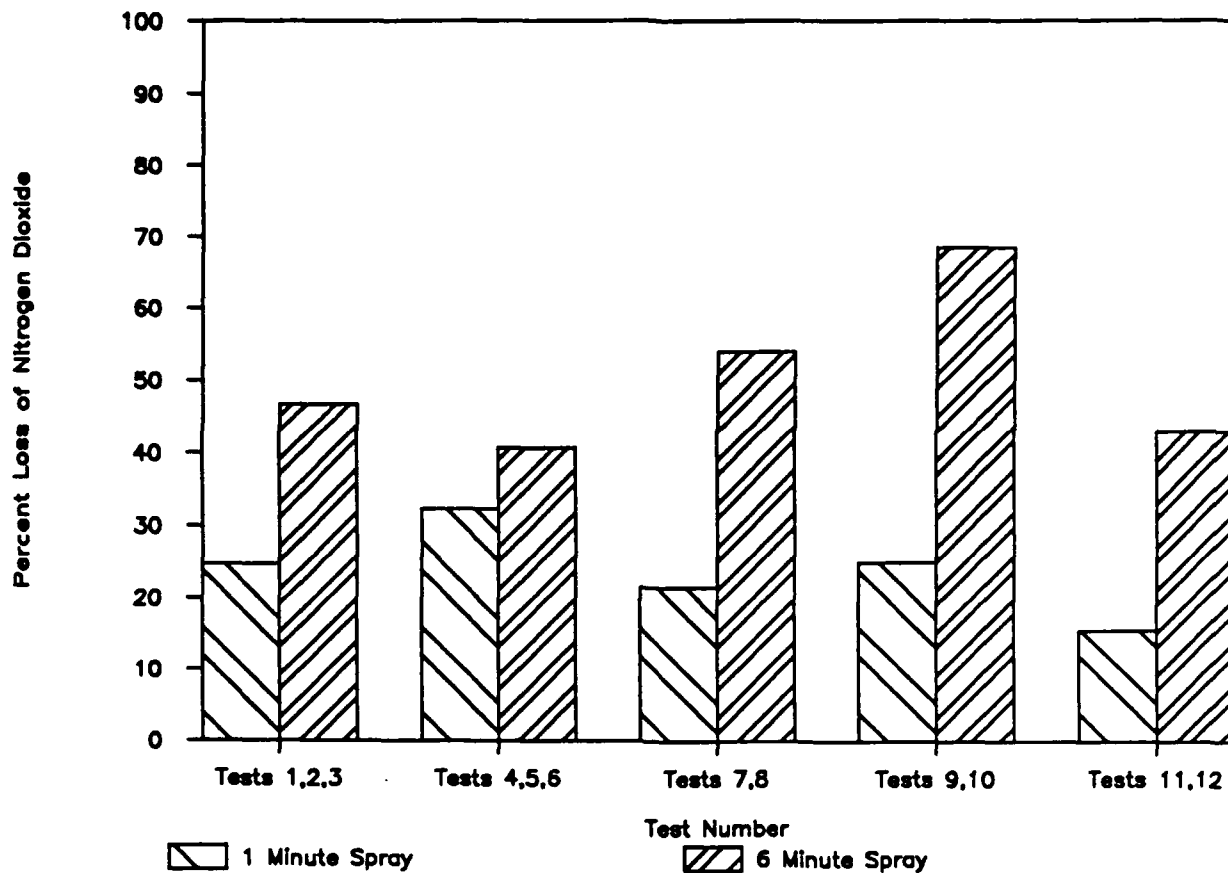
Tests 4, 5 and 6 - 6-sec high intensity
water spray, low NO_2
concentration

Tests 7 and 8 - Two 6-sec high intensity
water sprays, high NO_2
concentration

Tests 9 and 10 - 6-min low intensity
water sprays, high NO_2
concentration

Tests 11 and 12 - 6-sec high intensity spray
of 1 mol sodium bicarbonate
solution, high NO_2
concentration

Figure 8. Loss of NO_2 in Six Minutes Due to Water Mist Sprays.



Tests 1, 2 and 3 - 6-sec high intensity water spray, high NO_2 concentration

Tests 4, 5 and 6 - 6-sec high intensity water spray, low NO_2 concentration

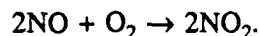
Tests 7 and 8 - Two 6-sec high intensity water sprays, high NO_2 concentration

Tests 9 and 10 - 6-min low intensity water sprays, high NO_2 concentration

Tests 11 and 12 - 6-sec high intensity spray of 1 mol sodium bicarbonate solution, high NO_2 concentration

Figure 9. Comparisons of One Minute and Six Minute Average Losses of NO_2 .

For every three nitrogen dioxide molecules that are removed due to reaction with water, one nitric oxide molecule is formed. Since the test gas did not contain oxygen, the NO was not oxidized to NO₂, as would be expected if air had been present,



9. CONCLUSIONS

The following conclusions may be drawn from this work:

- a. Certain powders were capable of removing NO₂ from the test gas mixtures. Diammonium phosphate and Monnex[®] were the most efficient powders.
- b. There is no synergistic effect when diammonium phosphate is added to Monnex[®].
- c. NO is much less reactive than NO₂. Only monoammonium phosphate and alum showed any ability to remove NO from the test gas.
- d. No single powder showed promise of being effective in removing both oxides of nitrogen.
- e. A water mist was effective in removing NO₂. The presence of a base in the water did not enhance the removal of NO₂.

10. RECOMMENDATIONS

The following recommendations are made for follow-on work on removal of toxic gases from air.

- a. Test a combination of powder and water spray to remove NO₂.
- b. Test additional additives to water to try to increase the rate of NO₂ removal.

- c. Test other powders on NO and NO₂.
- d. Test a combination of powders to remove NO and NO₂ simultaneously from a test gas.
- e. Test powders to remove other toxic gases, such as CO.
- f. Measure amounts of CO, NO and NO₂ generated by different types of propellants (single base, double base, LOVA).
- g. Test agents, shown to remove toxic gases, from test gases on burning propellant to reduce the amount of toxic gases formed.
- h. Design and test a prototype toxic gases removal system on a vehicle subjected to attack on its ammunition.

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11. REFERENCES

Imperial Chemical Industries Limited. "Toxicity of Powders." Technical Data Sheet No. 19, June 1971. Imperial Chemical Industries Limited, Monde Division, Research and Development Department, Health Laboratory, Runcom, Cheshire, United Kingdom, June 1971.

Ripple, MAJ Gary R. "Nonfragment Injury Assessment Criteria for M1 Live Fire Test." Walter Reed Army Institute of Research, Walter Reed Army Medical Center, Washington, D.C., May 1988.

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**APPENDIX:
TOXICITY OF POWDER**

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It was found that certain powders were capable of removing NO₂ from the test gas mixture. The best of the powders that were tested were diammonium phosphate and Monnex[®]. Both of these powders are recognized fire extinguishing agents believed to be non-toxic. However, we were unable to find any toxicity inhalation data at the concentration levels used in our work (100 mg/l). We did find results of tests conducted on rats by ICI Corporation using Monnex[®] at levels of 15 mg/l of air. The rats were exposed to this level of powder in air for 15 daily doses of 6 hours each. The results were very encouraging as to the lack of toxicity (Imperial Chemical Industries Limited 1971). However, no tests were conducted at 100 mg/l of air.

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