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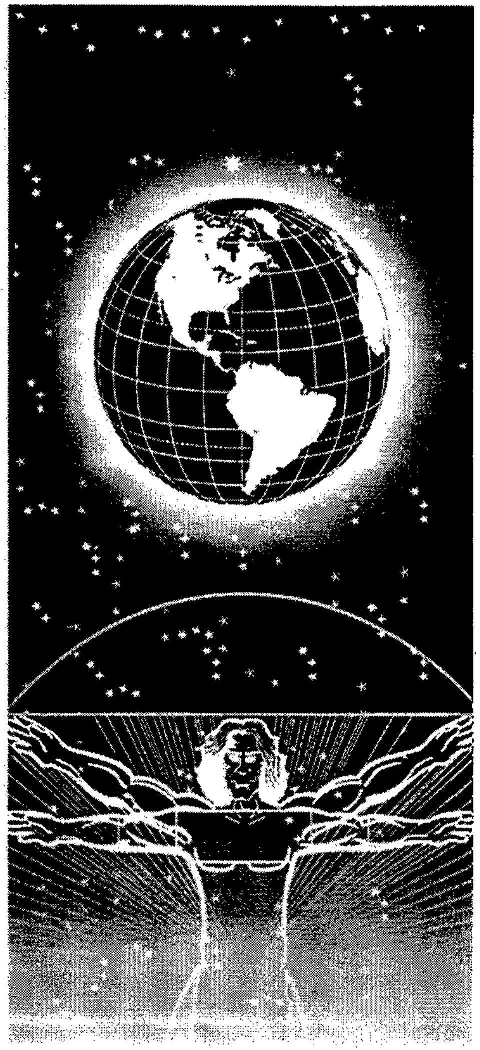

UNITED STATES AIR FORCE RESEARCH LABORATORY

RISK ASSESSMENT FOR PHOSPHORUS TRIBROMIDE

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The animal use described in this study was conducted in accordance with the principles stated in the "Guide for the Care and Use of Laboratory Animals", National Research Council, 1996, and the Animal Welfare Act of 1966, as amended.

This report has been reviewed by the Office of Public Affairs (PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

FOR THE DIRECTOR

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13. ABSTRACT *(Maximum 200 words)*

Phosphorus tribromide (PBr3) has effective fire fighting properties which make it a possible replacement for Halon 1301. Results of genotoxicity testing using bacterial Salmonella strains indicted PBr3 is not a mutagen for either frame shift or base-pair substitution tester strains in both buffered and unbuffered solutions. Acute aquatic toxicity testing determined a 96-hour LC50 value for fathead minnows at 71 mg/L (25 mg/L NOEC) and a 48-hour EC50 value for *Daphnia magna* at 22.6 mg/L (6.25 mg/L NOEC). Application of 10 ul neat PBr3 to intact skin of an anesthetized NZW rabbit caused edema and necrosis of the treated skin within 10 minutes of dosing. Toxicity was not observed in rats exposed for 4 hr to 0.4 mg/L in an inhalation exposure. Male rats (5/group) were exposed to PBr3 vapor, 4 hr/d for 5d, at 0, 0.06, 0.16 and 0.51 mg/L PBr3; 0.06 mg/L was the NOAEL. Rats (10/sex/group) were exposed to PBr3 vapor, 4 hr/d, 5 d/wk, for 4 wk at 0, 0.03, 0.01 and 0.3 mg/L; the NOAEL was 0.1 mg/L. Phosphorus tribromide reacts with moisture in the air to produce phosphonic acid and hydrogen bromide gas (HBr). A 10 cc cartridge of PBr3, tested as fire suppressant, would react to form an estimated 25 g of HBr. If uniformly distributed in a room 10x10x5 m in size, the maximum HBr concentration would be 0.05 mg/L (50 mg/m3); if in a 61.5x46.2x14.8 m hanger, it would be 5.9x10-4 mg/L (0.59 mg/m3). Since the exposure limit for HBr is 10 mg/m3, the maximal hanger concentration would be below the action level, indicating that an acceptable exposure level during usage on aircraft is possible based on the physical characteristics of PBr3 and the small amount necessary for fire suppression. Dermal and eye contact with PBr3 should be avoided due to its corrosive behavior.

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PREFACE

This is one of a series of technical reports describing a preliminary risk assessment for phosphorus tribromide tested at the Operational Toxicology Branch under the ManTech Geo-Centers Joint Venture contract. This document serves as a final report for the project conducted for phosphorus tribromide as a potential Halon replacement. The research described in this report began in October 1996 and was completed in March 1997 under Department of Defense Contract No. F41624-96-C-9010. Lt. Col Terry A. Childress, served as Contract Technical Monitor for the U.S. Air Force, Armstrong Laboratory. This study was sponsored by WL/MLBT.

The animal use described in these studies was conducted in accordance with the principles stated in the "Guide for the Care and Use of Laboratory Animals", National Research Council, 1996, and the Animal Welfare Act of 1966, as amended.

TABLE OF CONTENTS

LIST OF FIGURES

FIGURES **PAGE**

ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
BTU/hr	British thermal units per hour
°C	Degrees centigrade
$CaCO_3$	Calcium carbonate
cc	Cubic centimeter
cm^2	Square centimeter
cm^2/s	Square centimeter per second
cm^3	Cubic centimeter
DoD	Department of Defense
EC_{50}	Median effective concentration
F-344	Fischer 344 (rats)
ft	Feet
g	Grams
g/cm^3	Grams per cubic centimeter
H_20	Water
HBr	Hydrogen bromide
H_3PO_3	Phosphonic acid
LC_{50}	Median lethal concentration
m^3	Cubic meter
mg/m^3	Milligrams per cubic meter
mg/L	milligrams per liter
mL	Milliliter
NIOSH	National Institute for Occupational Safety and Health
NOAEL	No-obserable-adverse-effect-level
NOEC	No Effect Concentration
NZW	New Zealand White (rabbits)
OSHA	Occupational Safety and Health Administration
PBr_3	Phosphorus tribromide
PEL	Permissable exposure limit
ppb	Parts per billion
ppm	Parts per million
RH	Relative humidity
TLV	Threshold limit value
TWA	Time-weighted average
ug/cm^2s	Microgram per square centimeter seconds
ug/L	Microgram per liter
uL	Microliter
U.S.	United States

SECTION I

HAZARD ASSESSMENT

Phosphorus tribromide (PBr_3) has effective fire fighting properties which make it a possible replacement for Halon 1301. The DoD requires the development of a toxicity profile for replacement candidates of Halons, which includes the results from acute and subchronic toxicity testing. Many potential replacements, including phosphorus tribromide, have not been thoroughly investigated to determine their toxicological properties. The purpose of this report is to summarize the toxicity studies conducted for phosphorus tribromide and to conduct a preliminary risk assessment for this compound. The risk assessment will help determine if phosphorus tribromide (PBr_3) can be used as a Halon replacement compound.

Tests in a 500,000 BTU/hr test burner demonstrated that PBr_3 is an effective fire extinguishant. The test burner was quenched by only 0.2 mL PBr_3. This volume is several thousand times less than the amounts required of other Halon replacements to suppress fires. New fire extinguishant systems utilizing PBr_3 would occupy less volume, weigh less and require less mechanics than current systems. Also, PBr_3 has no ozone depleting potential since it is rapidly hydrolyzed in the troposphere.

Phosphorus tribromide reacts with moisture in the air and on wet surfaces to produce phosphonic acid and hydrogen bromide gas (HBr) according to the following reaction:

$$PBr_3 + 3H_2O \rightarrow 3HBr + H_3PO_3$$

There is limited toxicity information available in the literature for phosphorus tribromide and hydrogen bromide. No acute toxicity information is available for phosphonic acid or hydrobromic acid, the aqueous form of HBr. The combined interaction of these compounds to cause potential health hazards is not known.

In a study of rats exposed to 1300 ppm HBr for 30 minutes, nose-breathing effects were compared to pseudo-mouth-breathing effects. Tissue injury in the nasal region of the respiratory tract was observed, including epithelial and submucosal necrosis. Pseudo-mouth-breathing exposure to HBr caused higher mortality rates and major tissue disruption in the trachea. Observations of the trachea included necrosis of the epithelium, submucosa, glandular tissue and cartilage (Stavert *et al.*, 1991). In a separate study, the rat LC_{50} for one hour was 2858 ppm while the mouse LC_{50} for one hour was 814 ppm (RTECS, 1995a).

Human inhalation of 1300 to 2000 ppm HBr over a period of minutes was reported to be lethal (HSDB, 1995a). A short exposure to 35 ppm caused throat irritation (HSDB, 1995b), while exposure to 5 and 6 ppm for several minutes by 6 human volunteers also resulted in nose (6 out of 6) and throat irritation (1 out of 6) without eye discomfort (6 out of 6) (ACGIH, 1991a; HSDB, 1995c).

An accidental human exposure to PBr_3 and HBr was reported by Kraut and Lilis (1988). While mixing PBr_3, a female laboratory assistant was exposed to PBr_3 and HBr via splashing on the face, chest and hair, and by inhalation of resulting vapors. She remained in the area of the exposure for five to ten minutes. Immediate effects noted were complaint of dry cough, light-headedness and slight congestion of the throat. Over the next two weeks, the subject experienced increasing shortness of breath. Chest x-rays revealed bilateral lobe infiltrates resulting in a diagnosis of chemical pneumonitis. She was allowed to return to work a few months later, though dyspnea on exertion persisted and chest x-ray findings had not yet completely resolved. Recovery was slowed by a number of relapses, apparently due to exposure to other respiratory irritants (Kraut and Lilis, 1988). No exposure assessment was conducted and attempts to get exposure information were not successful.

Results of genotoxicity testing of bacterial Salmonella strains indicated PBr_3 is not a mutagen. These results were consistent for frame shift and base-pair substitution tester strains in both buffered and unbuffered solutions (ManTech, 1996).

No ecological dose-response levels were reported for HBr. Acute aquatic toxicity tests on fathead minnows (*Pimephales promelas*) and *Daphnia magna* were recently conducted with Pbr_3. Fathead minnows (10 per group) were exposed to 0.0, 6.25, 12.5, 25.0, 50.0 or 100.0 mg/L PBr_3 in fresh water (88 mg/L hardness as $CaCO_3$) at 22 ±2 °C. The static tests were performed in replicate and the results were pooled. The 96-hour LC_{50} value for fathead minnow was 71 mg/L (50 - 100 mg/L confidence limit); the no observed effect concentration (NOEC) was 25 mg/L (Aqua Survey, 1996a). These results indicate that an isolated or intermittent exposure to a concentration of PBr_3 equal to 71 mg/L, is likely to cause death to 50 percent of fathead minnows, *Pimephales promelas*. A concentration equal to or less than 25 mg/L is not likely to have an adverse effect. Daphnia (10 per group) were exposed to 0.0, 6.25, 12.5, 25.0, 50.0 or 100.0 mg/L PBr_3 in fresh water (88 mg/L hardness as $CaCO_3$) at 20 ±2 °C. The static tests were performed in replicate and the results were pooled. The 48-hour EC_{50} value for *Daphnia magna* was 22.6 mg/L (18.2 - 27.9 mg/L confidence limit); the NOEC was 6.25 mg/L (Aqua Survey, 1996b). These results indicate that an isolated or intermittent exposure to a concentration of PBr_3 equal to 22.6 mg/L is likely to cause mortality/immobilization to 50 percent of the Cladoceran *Daphnia magna*, while a concentration equal to or less than 6.25 mg/L is unlikely to have an adverse effect.

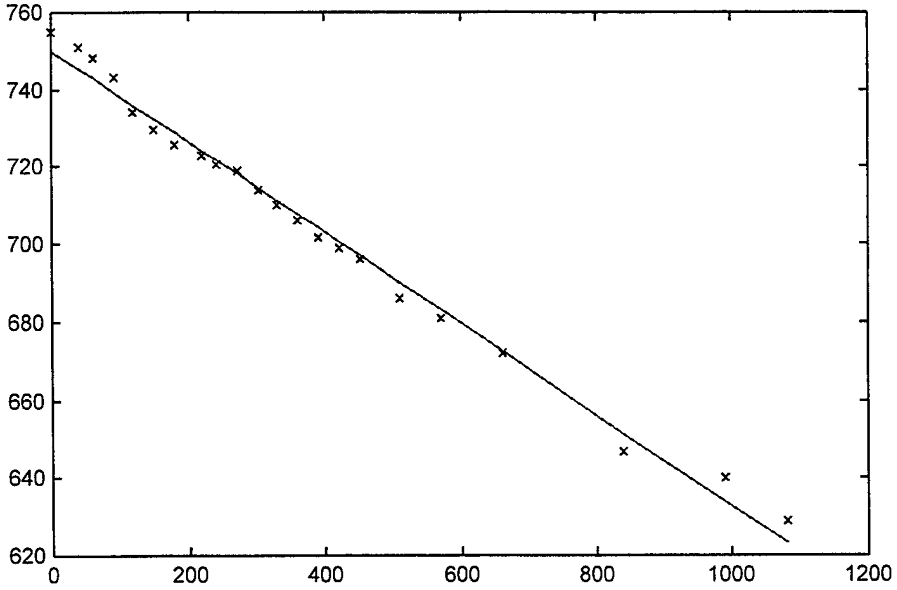
Acute and subchronic PBr_3 studies were designed to determine the effects following single, high-concentration exposures which could occur in accidents, as well as repeated, low-concentration exposures which could occur on flight lines or during maintenance. Application of 10 or 50 μL neat PBr_3 to intact skin of an anesthetized NZW rabbit caused edema and necrosis of the treated skin within 10 minutes of dosing. Microscopic examination confirmed necrosis of the skin and underlying areas, including skeletal muscle of the subcutis. Application of 10 or 50 μL neat PBr_3 to intact skin of an anesthetized NZW rabbit for 30 seconds followed by a one minute water wash resulted in necrosis of the entire skin, but not the underlying skeletal muscle. An acute 4-hour nose-only exposure of Fischer 344 rats to PBr_3 vapor resulted in mortality at 4.1 mg/L. At 1.5 mg/L, labored breathing, body weight loss, ulceration of anterior nares and rhinitis of the nasal passage were observed. Adverse effects were not observed in rats exposed for 4-hour to 0.4 mg/L. Male rats (5 per group) were exposed to PBr_3 vapor, 4 hours/day for 5 days, at 0, 0.06, 0.16 and 0.51 mg/L PBr_3. There were no signs of adverse effects at the low and mid-exposure levels. Rats in the 0.51 mg/L group had decreased body weights, gross lesions (reddened nares) and microscopic lesions (inflammation of mucosa and ulceration of epithelium in the nares). Rats (10 per sex per group) were exposed to PBr_3 vapor, 4 hours per day, 5 days per week, for 4 weeks at 0, 0.03, 0.1 and 0.3 mg/L. There were no signs of toxic stress, alterations in body weights or changes in organ weights in PBr_3 exposed animals. Minor serum chemistry and hematology effects were observed in the treated animals. Microscopic tissue findings were limited to rats of the 0.3 mg/L group and consisted of mild inflammation of the nasal passages. A concentration of 0.1 mg/L is the no-observable-adverse-effect level (NOAEL) in the 28-day inhalation study (Wolfe *et al.*, 1997).

SECTION II

EXPOSURE ASSESSMENT

In order to calculate an inhalation exposure for PBr_3, the following information is needed: vapor pressure (2.25 Torr at 20 °C), molecular weight (270.7), density (2.8 g/cm^3) and evaporation rate of the material as a function of temperature (7.6 μg/cm^{2}s). The evaporation rate was based on an experiment conducted with a puddle of PBr_3 (4.755 g or 1.66 cc) with a surface area was 15.5 cm^2. The puddle was placed in a moderately ventilated room with a low relative humidity and the mass change was monitored over time at room temperature. Figure 1. shows the evaporation results, which fit a mass transfer (evaporation) rate of 7.6 micrograms per square centimeter per second. While this value may increase a few fold with more vigorous ventilation, it is still four orders of magnitude slower than evaporation into a vacuum. This material (PBr_3) is so dense and its vapor pressure so low that mass transfer becomes dominated by diffusion.

Figure 1. Milligrams of PBr_3 versus time in an open weigh boat with moderate ventilation at room temperature.



These numbers can be used to estimate the rate at which PBr_3 is released following a spill or accidental discharge. The density of PBr_3 is greater than aluminum; a spray, stream or aerosol will settle rapidly on the floor in the event of an accidental release.

After evaporation into the air, the material would react rapidly with atmospheric moisture to form HBr and H_3PO_3, according to the reaction:

$$PBr_3 + 3H_2O \rightarrow 3HBr + H_3PO_3.$$

The hydrolysis rate for this reaction is $4 \times 10^{-17} cm^2/s$. This hydrolysis rate is based on the amount of water in air in molecules per cubic centimeter. At room temperature this quantity is expressed as the product of the relative humidity and the saturated vapor pressure of water (17.5 torr or 6.18 molecules per cubic centimeter). At 50% relative humidity (RH), the hydrolysis lifetime is 81 milliseconds. At 5% RH the hydrolysis lifetime increases to 810 milliseconds, while at 100% RH it decreases to 41 milliseconds.

The diffusion of PBr_3 vapor is very slow. The slow diffusion of the agent implies that its mixing will be primarily convective and subject to ventilation rates in the work area. Given the high hydrolosis rate even at low RH, PBr_3 would be expected to travel less than 1 cm from the liquid without reacting, under normal work ventilation conditions.

Based on the rapid reaction of liquid PBr_3 with water vapor, it is reasonable to assume that all of the released agent will be quickly converted to HBr and H_3PO_3. H_3PO_3 melts at 74 °C and is very soluble in water (300 grams per 100 cc H_2O), so this material will not be airborne except as an aerosol. HBr, although also very soluble in water (221 grams per 100 cc water), is a gas at room temperature (20 °C). A conservative assumption for accidental release is, therefore, that all of the PBr_3 is rapidly converted to gaseous HBr and aerosol H_3PO_3, either on contact or by reaction in a zone less than a centimeter from the liquid.

Using this conservative model, the 10 cc PBr_3 cartridge (28.5 grams) that was tested in full scale engine nacelle tests should generate approximately 25.5 grams (0.315 moles or 7.05 liter atmospheres) of HBr. If all of the PBr_3 is converted to HBr and is uniformly distributed in a room 10x10x5 meters in size, the maximum concentration of HBr would be 14.1 ppm. The concentration is inversely proportional to the volume of the room; in a storage closet 2x2x2 meters in size, it would be 881 ppm. These concentrations are upper bounds since the gas will be immobilized as it freely dissolves in the moisture present on surfaces in the room. The rate of surface adsorption of HBr could be easily quantified by experiment if it is important for modeling smaller rooms. For direct comparison with the inhalation toxicology data, these concentrations can be expressed in mg/liter; 0.05mg/liter (51 mg/m^3) for the small room and 2.96 mg/liter (2,961 mg/m^3) for the closet release.

Although there is no typical hangar size, dimensions of the hangars at Wright-Patterson Air Force Base are approximately 200x50x8 ft or 61.5x46.2x14.8 meters. The total volume of such a hangar is 42,051 m^3. If 25.5

grams of HBr is uniformly distributed in one of these hangars when it is empty, the maximum concentration would be 0.00061 mg/L (6.1×10^{-4}mg/L) or 0.61 μg/L. This would be equal to 0.61 mg/m^3 or 0.18 ppm or 180 ppb. This still is an upper bound because it assumes that all of the agent is sprayed over moist surfaces, through moist air and reacts 100% with the surface water and water vapor without any of it becoming dissolved in water.

SECTION III

RISK CHARACTERIZATION

For HBr exposure limits, National Institute for Occupational Safety and Health (NIOSH) set a ceiling limit of 10 mg/m^3, Occupational Safety and Health Administration (OSHA) set a permissible exposure limit (PEL) of 10 mg/m^3 as an 8-hr time weighted average (TWA) and American Conference of Governmental Industrial Hygienists (ACGIH) set a 10 mg/m^3 threshold limit value (TLV) as a ceiling. The maximal hangar concentration of 0.61 mg/m^3 would be an order of magnitude below the action level for these standards.

The NOAEL for the 28-day inhalation study was 0.1 mg/L for PBr_3. Since PBr_3 reacts to form HBr, the maximum possible concentration of HBr present at the NOAEL was 9.0×10^{-2} mg/L or 90 mg/m^3 (Wolfe *et al.*, 1997). Therefore, the maximal concentration of HBr in the hangar after an accidental discharge of 10 cc PBr_3 would be two orders of magnitude lower than the concentration of HBr at the NOAEL for PBr_3 in the 28-day study.

The issue then becomes one of using either the standards for HBr or the NOAEL for PBr_3 as exposure limits. Until a standard for PBr_3 is developed and accepted, the standard for HBr will most likely be used by industrial hygientists. Even after a standard is developed for PBr_3, exposure assessment will depend on the measurement of HBr since PBr_3 reacts so quickly with water vapor in the air. PBr_3 would not likely be used in anydrous or dessicated areas in real world situations.

Due to the density of the PBr_3 and slow diffusion rate, it will not travel far after accidental discharge, allowing time for personnel to exit the area. Due to its reactivity, it will convert to HBr which should achieve high concentrations only in small rooms. In the 10x10x5 meter room, the maximum concentration of HBr possible (51 mg/m^3) is still lower (by almost one half) than the concentration of HBr present at the NOAEL for the 28-day PBr_3 study. There is still the issue of potential exposure to an individual at or very close to the point of release. Acid concentrations should to be measured or estimated for an accidental release scenario. The acid concentration could be determined for various heights and distances near the source in order to address this issue.

The primary hazard arises from direct contact with the skin. The use of this agent in occupied spaces is possible but may not be advisable in all cases. How PBr_3 is supplied, its placement in a system and industrial hygiene procedures can minimize the risks of its use.

SECTION IV

CONCLUSIONS

The reactivity of the agent with moisture and the solubility of the acid products in water make elevated airborne concentrations of PBr_3 and HBr extremely unlikely. The concentrations of HBr should be below the action level for exposure standards.

SECTION V

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