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ACUTE TOXICITY OF A MIXED AMINE FUEL

by

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November 1959

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Foreword

FOREWORD

This report of range-finding studies of the toxicity of a mixed amine fuel known as MAF-1 was requested by the Naval Air Rocket Test Station. The information is being issued as a Technical Memorandum, well in advance of the formal report, with limited distribution to those most directly concerned.

At a later date this information will be published in permanent form as a CWL technical report and will be given wide distribution; on issuance of the CWLR this technical memorandum should be considered obsolete. Comments on the present publication are invited; all remarks received prior to 15 December will be considered for incorporation into the CWLR.

Previous releases of summaries of the information contained herein requested that the formulation of the fuel be considered a proprietary secret of the developing company. This restriction no longer exists.

I. INTRODUCTION

A mixed amine fuel, MAF-1, has recently been proposed as a candidate liquid rocket propellant. It is composed of three compounds in the following proportions by weight: 1,1-dimethyl hydrazine (UDMH) 50.5%, diethylene triamine (DETA) 40.5%, and acetonitrile 9.0%. Range-finding inhalation and cutaneous toxicity studies of MAF-1 were initiated to determine and compare the toxicity of the fuel with those of the separate components. Data from these experiments might indicate whether present safety precautions based on the toxicity of the individual components are adequate for the safe handling of MAF-1.

Previous studies have shown UDMH to be a central nervous system stimulant irrespective of how administered (1,2). A study of the acute vapor toxicity of this compound has been reported by Jacobson et al (3). They described the material as a respiratory irritant and convulsant. The LC50 for single 4-hour exposure of rats to UDMH vapor was 0.620 mg/l (252 ppm)*.

DETA has an irritant and a sensitizing effect upon contact with the skin (4). Inhalation of this compound appears to produce a considerable degree of sensitization which may be manifested by asthmatic attacks (5). Rats breathing saturated vapor generated at room temperature were not killed by an 8-hour exposure (6). The range-finding LD50 for the penetration of guinea pig skin by this compound has been reported to be 0.17 ml/kg (5).

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* For UDMH - 1 ppm = 0.0024 mg/l

Acetonitrile (methyl cyanide) is highly toxic and produces severe systemic disturbances from acute exposure. Single 4-hour exposure of rats to 8000 ppm of acetonitrile vapor killed one of six animals (6). The range-finding LD50 for rabbits by the percutaneous route has been reported as 5.0 ml/kg (7).

II. EXPERIMENTAL

The liquid MAF-1 used in these experiments was obtained from the Naval Rocket Test Station, Lake Denmark, N. J. It was brown in color, had a pungent odor, and a vapor pressure of 105 mm Hg at 25°C. UDMH was obtained from the Westvaco Chemical Company. The liquid is colorless, soluble in water, alcohol, and ether. It has a vapor pressure of 156.8 mm Hg at 25°C, a boiling point of 62.5°C at atmospheric pressure and a molecular weight of 60.1.

DETA was purchased from the Carbide and Carbon Chemicals Co., New York, and is a straw colored liquid, soluble in water and alcohol. It has a vapor pressure of 0.22 mm Hg at 20°C, a boiling point of 207.1°C at atmospheric pressure; its density is 0.9586 and molecular weight 103.17.

Acetonitrile (Eastman Organic Chemicals, Rochester, N. Y.) is a colorless liquid, soluble in water, alcohol, and ether. It has a melting point of -41°C and a boiling point of 82°C at atmospheric pressure. The vapor pressure is 100 mm Hg at 27°C, density 0.7828 and molecular weight is 41.05.

Acute inhalation range-finding toxicities were determined by single exposures of rats to air saturated with either MAF-1, UDMH, DETA, or acetonitrile and to air containing lower vapor concentrations of MAF-1 and UDMH. The cutaneous toxicity of MAF-1 was estimated from the effects produced after its application to intact and abraded rabbit skin.

A. Vapor Inhalation

White male, CWL (Wistar) rats about 150 gm in weight, in groups of six were exposed in a glass dynamic gassing chamber having a capacity of 8 liters. The length of each exposure ranged from 5 minutes to 6 hours in the saturated vapor studies but was of 4 hours' duration in the unsaturated vapor concentration studies. All rodents surviving exposure were placed in observation cages for a period of 7 days before being killed for studies of pathological changes. Animals presented for necropsy immediately after exposure, moribund or very recently dead, were examined at once. Those that survived exposure and the 7-day observation period were anesthetized with ether, bled by cutting axillary vessels, and examined. The following tissues were selected during gross examination, fixed in neutral 4% formaldehyde, and stained with hematoxylin and eosin: trachea, lungs, heart, stomach, spleen, pancreas, kidney, adrenal, urinary bladder, testis, liver, spinal cord, vertebral marrow.

The compounds were vaporized in the saturated vapor studies by placing 50 ml of the material being tested in a fritted disc gas washing bottle and drawing air through the liquid and into the exposure chamber at a

rate of 1.8 l/min. In the unsaturated vapor concentration studies, dispersion of UDMH and MAF-1 was effected by dropping a small amount of material continuously onto the fritted disc of a gas washing bottle. Air drawn up through the disc at the rate of 1.9 l/min carried the vapors into the exposure chamber. The amount of material delivered onto the fritted disc was dependent upon the size of the syringe containing the material and on the speed of a Phipps-Bird infusion pump.

The concentration of vapor to which animals were exposed was estimated nominally in all experiments and by chemical analyses of chamber air samples for UDMH in the unsaturated vapor concentration studies. Nominal concentrations (mg/l) were calculated from the weight of the material used, the length of exposure, and the total volume of air that passed through the system during the exposure. Analyses for UDMH were performed by an iodate titration using a visual end-point (8). It was necessary in the MAF-1 unsaturated vapor concentration studies to determine if acetonitrile or DETA interfered in the UDMH analysis. Control recovery studies with standard UDMH solutions containing acetonitrile or DETA showed that acetonitrile did not interfere in the UDMH analysis whereas DETA lowered the recovery of UDMH forty per cent. However, since DETA has such a low vapor pressure (0.22 mm Hg at 20°C), it is expected that under the conditions of dispersion negligible amount of its vapor would pass into the exposure chamber.

B. Cutaneous Application

Range-finding percutaneous toxicity experiments were carried out

on the clipped skins of white albino rabbits weighing 2 - 2.5 kg. The animals were tied to restraining boards and placed in fume hoods with their heads protruding into the room. The linear airflow over the backs of the animals was adjusted to 30 m/min. The animals were restrained for several hours in this manner until the applied material evaporated or was absorbed into the skin. They were then released and placed into holding cages for observation of local or systemic signs of poisoning.

In the first group applications of MAF-1 ranging from 100-3000 mg/kg were made to clipped unabraded skin. In the second group, 631 mg/kg of the material was applied to the clipped backs of rabbits, whose skins were uniformly abraded by needle scratches according to the method of Draize (9).

III. RESULTS

A. Vapor Inhalation

1. Mortality

The mortality resulting from exposure of rats for varied lengths of time to air saturated with vapors of MAF-1, UDMH, acetonitrile and DMFA are summarized in Table 1. Mortalities in rats exposed four hours to the vapors from the dispersion of lesser amounts of MAF-1 and pure UDMH are shown in Table II.

2. Clinical Observations

a. UDMH Saturated Vapors

Rats exposed for a period of 5 minutes showed signs of

eye and nose irritation. After exposure for 15 minutes they exhibited head shaking, gasping, eye and nose irritation, and rasping lung sounds. In these two groups no deaths occurred during the exposures for the seven day observation period. The groups exposed for 30 minutes and 60 minutes showed signs similar to those previously noted, in addition to frothing at the mouth and convulsions. All deaths occurred within 3 hours after exposure.

b. HAF-1 Saturated Vapors

Rats exposed for a period of 5 minutes showed no toxic signs. After 15 minutes per exposure, they exhibited head shaking and gasping. In the 30 minute exposure group, gasping, eye and nose irritation, and dyspnea were noted. All deaths in this group occurred within 24 hours after exposure. Those exposed 60-min. showed all the above toxic signs plus frothing, and convulsions. All deaths in this group occurred within 1 hour after exposure.

c. Acetonitrile Saturated Vapors

Three saturated vapor exposures, each of varying length, were made using acetonitrile. The toxic signs produced in the 30-minute, 2-hour, and three-hour exposures were rapid, shallow breathing, weakness, stupor progressing to coma, and nasal and oral discharge. In the 3-hour exposure all became unconscious after 90 minutes of exposure and 5 out of 6 died 45 minutes after termination of exposure. The last rodent survived 48 hours but never regained consciousness. No rats died during the 2-hour exposure, but all were in a coma after 90 minutes of exposure. This coma

lasted 72 hours, after which three died and three regained consciousness and survived the 7-day observation period. In the 30-minute exposure, rapid shallow breathing and weakness were noted during exposure. Rats did not become unconscious and all survived the observation period.

d. DETA Saturated Vapors

No toxic signs were observed during 6 hours exposure.

e. UDMH Unsaturated Vapor Exposures

Rats exposed to the lowest nominal concentration (1.24 mg/l) showed signs of eye and nose irritation and some dyspnea. At intermediate nominal concentrations (1.76 mg/l) rodents showed the same signs as noted in the lower concentration plus gasping, tremors, and convulsant spasms. However, the onset of toxic signs were slightly more rapid. At the highest nominal concentration (2.24 mg/l), the signs as previously seen occurred more rapidly with frothing, spasms, cyanosis, and terminal convulsions. All rats expired during exposure to the highest concentration.

f. MAF-1 Unsaturated Controlled Vapor Exposures

Rats exposed to this mixture at the lowest nominal concentration showed signs of head shaking, rapid breathing, and gasping. Slight eye and nose irritations were observed. At a nominal concentration of 0.84 mg/l, dyspnea was noted after 45 minutes. Eye, nose irritation and weakness occurred after 90 minutes of exposure. Spasms occurred after 2 hours and rodents became stuporous after 3 hours exposure. All rats survived the 7-day observation period. In those exposed to 1.5 mg/l the toxic signs were similar but more severe with two rats dying, one after

3 hours of exposure and the other, 2 days post exposure. At a concentration of 2.8 mg/l, the onset of previously mentioned signs was more rapid, with the addition of frothing and terminal convulsions. Mortality from exposure at this concentration was 5 out of 6 dying within 24 hours.

3. Pathologic Observations

a. Gross examination

Animals that were dead or moribund following exposure to MAF-1 or to UDMH showed similar gross changes: externally, muzzles were wet, and clear foam and bubbles extruded from the nostrils; the abdomens were tightly bloated, bulging more on the left side. Internally, a white mucoid foam in the trachea was extruded by pressure from the emphysematous lungs. The lungs were reddish to dark maroon in color, and on some, red spots 2-3 mm in diameter were visible on the surface. The stomachs were grossly distended with gas crowding the thoracid and abdominal viscera. A white mucoid foam, also in the stomach, extended along with the gas, into the bloated small intestine. The livers were distended and dark, and their cut surfaces oozed dark blood.

Rats, dead or moribund, from acetonitrile exposure differed grossly in the following respects: abdominal distention was slight or absent, and though muzzels were wet, there was no foam in the air tracts, and the lungs collapsed normally. Stomach distention was slight to moderate, more from a mucoid foam than from gas collection, and did not involve the duodenum.

Seven days after exposure there was no gross difference noted in survivors of MAF-1 or of its components.

b. Histopathologic examination

With the exceptions noted later, the only histopathologic changes seen were in the pulmonary system. Seven days after 4-hour exposures to MAF-1 concentrations up to 0.85 mg/l, no microscopic changes were noted except that one animal had a slight neutrophil-containing exudate in some air passages. In animals dead or moribund after 4-hour exposures to MAF-1 (1.5 - 2.8 mg/l) or after shorter exposures to saturated vapors, the following changes were noted: erosion of pulmonary epithelium; subepithelial, peribronchial, peribronchiolar and perivascular edema; parenchymal congestion; small focal hemorrhages with variable intra-alveolar edema.

A week after exposure to high MAF-1 concentrations, the following changes were noted: bronchial and bronchiolar epithelial metaplasia, or erosion and neutrophil infiltration in and around the air passages. Small focal hemorrhages were noted in some lungs, with small numbers of red blood cells in air passages. The most severe residual changes involved entire lung lobes consisting of massive, generalized neutrophil infiltration, with many foamy macrophages in the alveoli; confluent, granuloma-like areas with necrotic centers; areas of fibrous tissue formation, and metaplasia of some bronchial and bronchiolar epithelium.

Changes noted in rats exposed to UDMH were essentially similar to those produced in rats exposed to MAF-1 under comparable conditions. In one animal that did not survive a 30-minute saturated vapor exposure, a very small focus of degenerative myocarditis was

present, but this condition is not uncommon in unexposed laboratory rats, and is not considered related to the test compound.

In animals that were dead or moribund after exposure to saturated acetonitrile vapor, congestion was noted in the liver, stomach, and duodenum, a condition not noted in animals exposed to UDMH or MAF-1. The bronchial epithelium was eroded, with loosening and separation of the epitholial cells in some alveoli and air passages. Scattered red blood cells were also present in alveoli and air passages. Seven days after 30 minutes exposure to acetonitrile saturated vapors, one of two animals examined histologically appeared essentially normal. The lungs of the other had changes similar to, but milder than those that were noted in two hour exposures: namely, massive, generalized neutrophil infiltration, with many foamy macrophages in the alveoli; confluent granuloma-like areas with necrotic centers; areas of fibrous tissue formation, and metaplasia of some bronchial epithelium.

c. Summary of morphologic changes

Morphologic alterations noted in rats immediately after, and a week after exposure to low and high air concentrations of MAF-1 were essentially similar to those found in rats exposed to its major component, UDMH. Tracheo-bronchial and salivary secretions increased, during exposure, and hair on muzzles and chests were wet. There was pulmonary edema, with much frothy fluid in the air passages, as well as edema of the peribronchial connective tissue, and slight intraalveolar hemorrhage. There was severe air bloating incident to the

partial blocking of the air passages just described.

A week after exposure to low concentrations, a slight neutrophilic exudate was noted in lumina of bronchi of some animals. After more severe insults, erosion of pulmonary epithelium and edema around air passages and vascular channels were still in effect, with metaplasia of epithelium in some. Acute peribronchitis, peribronchiolitis and pneumonia with fibrous tissue formation were present, varying from a slight amount of consolidation and organization of entire lobes.

In contrast, rats exposed to fatal concentrations of acetonitrile, although salivating freely, exhibited only slight peribronchial edema, and showed only slight gassy distension of the stomach. A week after exposure, however, survivors exhibited residual pulmonary changes similar to those seen in animals exposed to high MAF-1 exposures.

B. Cutaneous Toxicity

1. Systemic effects of MAF-1

The 24 hr LD50 on unabraded skin was approximately 2.5 g/kg and the 48 hr LD50 approximately 2.0 g/kg. These values place the percutaneous toxicity of MAF-1 in the slightly toxic class (10).

The application of 631 mg/kg of MAF-1 to the abraded skin group produced no fatalities one week after dosing. This would indicate that the LD50 of MAF-1 to abraded skin is greater than 631 mg/kg.

2. Local skin effects of MAF-1

Skin areas upon which MAF-1 was applied showed erythema and edema within 2 hours. Assay, by means of a micrometer syringe showed that 0.54 cu mm was the smallest amount of MAF-1 which produced visible edema.

IV. DISCUSSION

MAF-1 is composed of compounds that individually show rather specific degrees of toxicity by inhalation. DETA showed no toxicity in a saturated vapor study and can be presumed to contribute little to the vapor hazard in the handling of MAF-1. Acetonitrile exhibited some toxicity in the saturated vapor studies but was slow-acting as compared to UDMH. The most striking toxic sign with acetonitrile was the coma produced in the rats, some of which recovered without further signs of intoxication. The danger with this compound would seem to be with the stupor produced and if added in larger quantities to other newly formulated mixed amine fuels would constitute a real hazard to unprotected handlers. The toxicity of UDMH is well known and in these studies produced toxic signs quickly and in a manner which has been characterized in other acute studies (3). MAF-1 in the saturated vapor studies was slightly more toxic than any of its constituents based on 60 minute exposure to saturated vapor where 121 mg/l of MAF-1 caused death in all the exposed rats.

Smyth (5) has devised a method for classification of compounds according to the hazard from inhalation. Using his rating system for saturated vapor inhalation, MAF-1 and its constituents fall into the activity grades as seen below.

Saturated Vapor	Activity Grade
DETA	S-1
Acetonitrile	S-4
UDMH	> S-6 < S-7
MAF-1	S-6

Grade 0-1 is classified as the least hazardous from which the classification number increases as the hazard increases. These grades are classifications of hazard rather than quantitative toxicity, since the amounts inhaled are dependent upon vapor pressure. Classification of the compounds by this method show that MAF-1 is about as hazardous as UDMH but much more hazardous than either DETA or acetonitrile.

In the controlled concentration inhalation studies the signs of intoxication from UDMH or MAF-1 were about the same. Classification of the toxicity of these two compounds based on the amount delivered (nominal concentration) or on the analytical concentration of UDMH (the amount of UDMH in the air) that the solution of UDMH is somewhat more toxic than MAF-1.

These data indicate that by the route of inhalation MAF-1 is probably no more hazardous that UDMH. Percutaneous toxicity studies show little hazard from this compound. The precautions and safety practices now in use for handling UDMH should be adequate for MAF-1. The literature on DETA shows that this compound may present an added hazard by causing skin sensitization that probably does not occur with UDMH. Therefore, added precautions above that for UDMH would probably be desirable to eliminate this possible hazard.

V. SUMMARY

Range-finding toxicity tests on MAF-1 indicate that MAF-1 vapor is similar in toxicity to 1,1-dimethylhydrazine (UDMH). It produced effects similar to UDMH in exposed animals and produced death at about

the same concentration levels. MAF-1 is considered to be slightly less hazardous than UDMH, because of the lower volatility of MAF-1. Cutaneous application of MAF-1 showed that it would require large amounts to cause death. However, very low doses caused irritation of exposed skin areas. The precautions and safety practices now in use for handling UDMH should be adequate for MAF-1.

TABLE 1

MORTALITY FROM SATURATED VAPOR EXPOSURES

Compound	Exposure time	Nominal concn	Mortality in rats
	min	mg/l	
MAF-1	60	121	0/6
	30	150	4/6
	15	152	0/6
	5	133	0/6
UDMH	60	307	3/6
	30	307	6/6
	15	301	0/6
	5	290	0/6
Acetonitrile	180	88	6/6
	120	101	3/6
	30	114	0/6
DETA	360	0	0/6

TABLE 2

Mortality From Single 4-Hour Exposure of Rats to MAF-1 and UDMH Vapors

MAF-1			UDMH		
Nominal conen	Analytical conen	Mortality in Rats	Nominal conen	Analytical conen	Mortality in Rats
mg/l	mg/l		mg/l	mg/l	
2.8	1.02	5/6	2.24	0.50	0/6
1.5	0.53	2/6	1.76	0.17	0/6
0.84	0.34	0/6	1.24	0.25	0/6
0.50	0.26	0/6			

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13. ABSTRACT

(U) A mixed amine fuel, MAF-1, was proposed as a liquid rocket propellant. It is composed of three compounds in the following weight proportions: 1,1-dimethylhydrazine (UDMH), 50.5%; diethylenetriamine (DETA), 40.5%; and acetonitrile, 9.0%. UDMH is a central-nervous-system stimulant; DETA has an irritant effect upon the skin. Acetonitrile (methyl cyanide) produces severe systemic effects from acute exposure. All compounds are soluble in water, alcohol, and ether. Their vapor pressures, boiling points, and molecular weights are given. In vapor-inhalation studies, exposures ranged from 5 min to 6 hr in saturated-vapor studies, but was of 4-hr duration in unsaturated-vapor studies. Control recovery studies with standard UDMH solutions containing acetonitrile showed that acetonitrile did not interfere in the UDMH analysis, whereas DETA lowered the recovery of UDMH by 40%. MAF-1 vapor is similar in toxicity to UDMH. Death in animals is at about the same concentration level. MAF-1 is slightly less hazardous because of its lower volatility. Very low doses of MAF-1 caused irritation to skin areas. Safety precautions in force for handling UDMH should be adequate for MAF-1.

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14. KEYWORDS

Toxicity
Amine
Fuel, amine
Fuel-MAF-1-amine
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Irritant
Convulsant
Sensitization
Trachea
Lungs
Heart
Stomach
Spleen
Pancreas
Kidneys
Adrenal glands
Urinary bladder
Testis
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Nostrils
Alveoli
Tissue
Myocardium
Amine, diethylenetri-