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THE RADIOLYTIC DECOMPOSITION OF HYDRAZINE, RP-1, AND HYDYNE ROCKET FUELS

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ABSTRACT

One-hundred-milliliter samples of the storable liquid rocket fuels hydrazine, RP-1, and Hydyne generate, respectively, 89.1, 50.3 and 149.4 ml of radiolytic off-gas (measured at 25°C and 1 atm) when irradiated to 8.5×10^6 rads with gamma rays. When approximately 5 wt-% of an efficient, olefinic, free-radical scavenger is added to the samples, the off-gas volume produced by RP-1 fuel is reduced by 18.7 % while those of hydrazine and Hydyne fuels are not reduced.

These scavenging effects show that RP-1 fuel decomposes radiolytically by both free-radical (18.7 %) and molecular mechanisms, and that hydrazine and Hydyne fuels decompose entirely by a molecular or ionic mechanism.

SUMMARY

Problem

The problem was to determine whether it is possible to suppress the generation of non-condensable gases when the storable liquid rocket fuels hydrazine, RP-1, and Hydyne are subjected to cobalt-60 gamma radiation. The greater the suppression of such off-gas generation, the greater the storability of the fuels with respect to ionizing radiations in space and from nuclear rocket engines. This information is of importance to the Navy MOL program because of the possibility of severe solar flares affecting the performance of the liquid fuel rockets on board.

Findings

Hydrazine, RP-1, and Hydyne rocket fuels produce copious quantities of non-condensable off-gases when irradiated to nearly 10^7 rads with gamma rays. Since it was conceivable that this gas generation proceeds via reactive chemical intermediates known as free-radicals, gas suppression was attempted with chemical additives that could render the free-radicals impotent by reacting with them. Addition of an efficient, olefinic, free radical scavenger reduced the quantity of radiolytic off-gas from RP-1 fuel by 18.7 %, but it was ineffective with hydrazine and Hydyne fuels.

INTRODUCTION

Storable liquid, rocket fuels will generate appreciable quantities of non-condensable off-gases when they are subjected to large doses of Van Allen belt, solar cosmic and nuclear rocket radiations. Appreciable gas formation was shown by Shelberg,[1] Robinson,[2] and Plank[3] through exposure of monomethylhydrazine rocket fuel to gamma and reactor radiations and by Shelberg[4] through gamma-irradiation of 1,1-dimethylhydrazine and diethylenetriamine rocket fuels. These off-gases and the fuel can mix non-homogeneously and assume various juxtapositions in a fuel tank at zero gravity in space.[5] This could result in uneven combustion and require venting to relieve excessive tank pressure. Venting would be wasteful of liquid fuel and would entail undesirable engineering complexities.

This laboratory has been engaged in a series of studies to determine whether the formation of radiolytic off-gases in rocket fuels can be suppressed by the addition of free-radical scavengers to the fuels. An efficient free-radical scavenger will suppress that off-gas which is produced by a free-radical mechanism, but not that produced by molecular or ionic mechanisms. The production of a molecule of gas, such as methane, via free-radicals generally involves the radiation-engendered separation of a methyl radical from a solvent molecule, its diffusion, and its abstraction of a hydrogen atom from another solvent molecule. The scavenger will react avidly with the methyl radical, breaking the reaction sequence to the formation of methane.

Shelberg[1] has demonstrated that the free-radical scavengers methyl methacrylate and pentadiene-1,3 fail to suppress the generation of radiolytic off-gases in monomethylhydrazine. This was taken as evidence that monomethylhydrazine decomposes via a molecular or ionic reaction, rather than a diffusion-controlled free-radical reaction. It was also revealed that the erstwhile scavenger carbon tetrachloride sets up a chain reaction in irradiated monomethylhydrazine, which increases the total quantity of off-gas by a factor of approximately 5.

Shelberg[4] has demonstrated also that the free-radical scavenger methyl methacrylate does suppress the generation of radiolytic off-gas with 1,1-dimethylhydrazine and diethylenetriamine by 18.2 and 11.0 %, respectively, when the fuels are gamma-irradiated to 8.5×10^6 rads. These values indicate the extent to which the radiolytic decompositions proceed via a diffusion-controlled free-radical process; accordingly, simultaneous molecular or ionic processes predominate. Since the free-radical scavenger reduces somewhat the off-gas from these fuels due to laboratory ionizing radiation, it may be expected to improve their storability somewhat with respect to large doses of ionizing space and nuclear rocket radiations.

The investigation reported herein determined the ability of free-radical scavengers to suppress the generation of radiolytic off-gases in other important rocket fuels: hydrazine, RP-1, and Hydyne. RP-1 fuel is a kerosine-cut hydrocarbon fuel. Hydyne is a mixture of 60 wt-% of 1,1-dimethylhydrazine and 40 wt-% of diethylenetriamine.

Little information is reported in the literature on the radiolysis of hydrazine and Hydyne, and none on RP-1 fuel. Lucien[6] irradiated hydrazine vapor at 15 mm-Hg with X rays up to 6×10^5 r and observed the production of ammonia, nitrogen and possibly hydrogen; the initial hydrazine was decreased about 3 %. Shelberg[4] estimated the extent of radiolytic gas production with Hydyne fuel from the gas production of its components. This calculated Hydyne result is compared with an experimental Hydyne result in this report.

EXPERIMENTAL

The experimental work consisted of preparing degassed specimens of hydrazine, RP-1 and Hydyne fuels with and without a free-radical scavenger, gamma-irradiating them with cobalt-60 to 8.5×10^6 rads at 22-25°C, and analyzing the non-condensable products by gas chromatography.

The hydrazine was Eastman White Label Grade, 95+ % pure (Distillation Products Industries, Rochester, N.Y.). The Hydyne was prepared from 1,1-dimethylhydrazine (60 wt-%) and diethylenetriamine (40 wt-%), which were Eastman Practical and Technical Grade, respectively. The RP-1 fuel was provided by the Tidewater Oil Company, Western Division.

Methyl methacrylate scavenger was used with Hydyne and RP-1 fuels at a concentration of 5 vol-%, and was a polymerization grade inhibited with 60 ppm of hydroquinone (Rohm and Haas Co.). Acrylamide scavenger was used with hydrazine since methyl methacrylate scavenger is insoluble in hydrazine. Four and one-half grams of acrylamide (American Cyanamide Co.) was dissolved in hydrazine (ca 50 ml) in a 100-ml volumetric flask, and the solution was diluted to volume with hydrazine.

Dosimetry was done with the ferrous sulfate dosimeter, which duplicated the specimen in volume and geometry.[7]

The unfilled radiolysis cell[1] consisted of a 50-ml, round-bottom, long-neck, Pyrex flask fitted with a horizontal break-seal on the lower neck, an upward slanted side-arm of 10 mm diameter opposite the break-seal, and a seal-off constriction on the upper neck. Above the seal-off constriction, the cell was sealed to the stopcock of a vacuum manifold having outlets to the air and to a high vacuum.

Before being filled, the cell was flushed with nitrogen, which entered via the side-arm and vented to the air via the manifold. The nitrogen supply line was disconnected briefly from the side-arm, while 20 ml of the specimen was introduced into the cell with a hypodermic syringe. Care was taken not to wet the side-arm. The specimen was frozen with liquid nitrogen (stopcock closed, nitrogen valve closed). Then the side-arm was warmed with an air-heater (not with hydrazine - it could detonate), while the frozen cell was flushed with nitrogen to remove any trace of specimen possibly contaminating the side-arm. The side-arm was sealed off at atmospheric pressure (stopcock open to the air, nitrogen valve closed). The specimen was degassed by the conventional technique of repeated freezing and pumping, and the frozen cell was sealed off at 10^{-6} mm Hg.

After irradiation, the specimen was frozen with liquid nitrogen, and the non-condensable products (hydrogen, nitrogen and methane) were transferred via the break-seal to a calibrated, 1-liter, gas-storage bulb by means of a Toepler pump. A 10-ml aliquot of the contents of the gas-storage bulb was analyzed quantitatively with a Perkin-Elmer gas chromatograph (Model 154) equipped with a thermistor detector, a 50-ft Linde molecular sieve column at 30°C, and helium carrier gas. The high-vacuum gas-inlet apparatus for gas chromatography previously developed at this laboratory was used.[8]

RESULTS AND DISCUSSION

Table 1 gives product yields for hydrazine, RP-1 fuel, Hydyne fuel, and their scavenged solutions, irradiated to 8.5×10^6 rads. Product yields for monomethylhydrazine, 1,1-dimethylhydrazine and diethylenetriamine, determined previously under the same conditions,[1,4] are included for comparison.

TABLE 1

Radiation Yields at 8.5 x 10^6 Rads

	OFF-GAS (Ml of Gas, at 25°C and 1 Atm Generated per 100 ml of Sample)				AVERAGE G (Molecules/100 ev)			
Sample	H_2	N_2	CH_4	Total	H_2	N_2	CH_4	Total
HYDRAZINE								
No Scavenger								
1	43.2	46.3	0.0	89.5				
2	42.0	46.7	0.0	88.7				
Average	42.5	46.5	0.0	89.1	2.0	2.1	0.0	4.1
With Acrylamide Scavenger								
1	39.1	59.8	0.0	98.9				
2	39.6	60.5	0.0	100.1				
Average	39.4	60.2	0.0	99.5	1.8	2.8	0.0	4.6
RP-1 FUEL								
No Scavenger								
1	49.1	0.1	1.4	50.6				
2	48.2	0.3	1.5	50.0				
Average	48.7	0.2	1.5	50.3	2.9	0.01	0.09	3.0
With Methyl Methacrylate Scavenger								
1	37.7	-	1.1	-				
2	40.8	0.5	1.1	42.4				
Average	39.3	0.5	1.1	40.9	2.3	0.03	0.06	2.4

Continued

TABLE 1 (Contd)

Radiation Yields at 8.5×10^6 Rads

OFF-GAS (Ml of Gas, at 25°C and 1 Atm, Generated per 100 ml of Sample)					AVERAGE G (Molecules/100 ev)			
Sample	H_2	N_2	CH_4	Total	H_2	N_2	CH_4	Total
HYDYNE								
No Scavenger								
1	74.3	64.7	5.9	144.9				
2	79.8	68.3	5.8	153.9				
Average	77.1	66.5	5.9	149.4	4.3	3.6	0.3	8.2
With Methyl Methacrylate Scavenger								
1	68.6	81.5	7.7	157.8				
2	66.4	75.4	6.5	148.3				
Average	67.5	78.5	7.1	153.1	3.7	4.3	0.4	8.4
MONOMETHYLHYDRAZINE[a]								
No Scavenger								
1	81.4	71.8	58.9	212.1				
2	99.7	83.0	69.4	252.1				
3	93.7	74.3	61.6	229.6				
4	77.4	75.2	63.3	215.9				
Average	88.1	76.1	63.3	227.4	4.7	4.0	3.4	12.1
With Methyl Methacrylate Scavenger								
1	80.9	85.9	75.7	242.5				
2	84.5	88.3	78.4	251.2				
Average	82.7	87.1	77.1	246.9	4.4	4.6	4.1	13.1

Continued

TABLE 1 (Contd)

Radiation Yields at 8.5 x 10^6 Rads

OFF-GAS (Ml of Gas, at 25°C and 1 Atm, Generated per 100 ml of Sample)					AVERAGE G (Molecules/100 ev)			
Sample	H_2	N_2	CH_4	Total	H_2	N_2	CH_4	Total
				1,1-DIMETHYLHYDRAZINE[b]				
				No Scavenger				
1	98.5	90.3	12.7	201.5				
2	95.0	88.7	12.7	196.4				
Average	96.8	89.5	12.7	199.0	5.7	5.3	0.75	11.8
				With Methyl Methacrylate Scavenger				
1	60.6	90.5	9.3	160.4				
2	66.1	88.7	10.1	164.9				
Average	63.4	89.6	9.7	162.7	3.7	5.3	0.57	9.6
				DIETHYLENETRIAMINE[b]				
				No Scavenger				
1	100.2	1.2	0.0	101.4				
2	100.1	0.8	0.0	100.9				
Average	100.2	1.0	0.0	101.2	4.9	0.05	0.0	5.0
				With Methyl Methacrylate Scavenger				
1	78.5	2.2	6.0	86.7				
2	74.8	1.3	15.5	91.6				
3	77.7	1.3	13.0	92.0				
Average	77.0	1.6	11.5	90.1	3.8	0.08	0.56	4.4

a. Reference 1.
b. Reference 4.

It is not meaningful to speculate on specific reaction routes to the non-condensable gaseous products from Hydrazine, RP-1 fuel, and Hydyne fuel. Complexities are possible in their radiolytic decompositions, and radiation yields for important condensable products are not known.

HYDRAZINE

Hydrazine produces a large volume of radiolytic off-gas. A 100-ml sample generates an average of 89.1 ml of total off-gas (25°C, 1 atm) having a molecular composition of 47.8 % hydrogen and 52.2 % nitrogen.

Acrylamide scavenger not only fails to reduce the volume of total off-gas but actually increases it by 11.7 %. The increase is due to an increased production of nitrogen gas, possibly resulting from the radiolytic decomposition of the scavenger. A 100-ml sample generates an average of 99.5 ml of total off-gas (25°C, 1 atm), with a molecular composition of 39.6 % hydrogen and 60.4 % nitrogen.

RP-1 FUEL

RP-1 fuel generates a substantial volume of radiolytic off-gas. A 100-ml sample produces an average of 50.3 ml of total off-gas (25°C, 1 atm) having a molecular composition of 96.8 % hydrogen, 0.4 % nitrogen, and 2.8 % methane. Irradiation changes the color, originally red, to colorless.

The irradiation of RP-1 fuel containing methyl methacrylate scavenger results in a white slush or emulsion. The slush is probably not suitable as a rocket fuel. Methyl methacrylate scavenger reduces the volume of total off-gas by 18.7 %, and this results from decreased generation of hydrogen gas. However, a significant amount of off-gas is still produced, a 100-ml scavenged sample generating an average of 40.9 ml of total off-gas (25°C, 1 atm), with a molecular composition of 96.1 % hydrogen, 1.2 % nitrogen and 2.7 % methane.

HYDYNE FUEL

Hydyne fuel evolves a large volume of radiolytic off-gas. A 100-ml sample evolves an average of 149.4 ml of total off-gas (25°C, 1 atm) having a molecular composition of 51.6 % hydrogen, 44.5 % nitrogen, and 3.9 % methane.

Methyl methacrylate scavenger fails to reduce the volume of radiolytic off-gas. A 100-ml scavenged sample evolves an average of 153.1 ml of total off-gas (25°C, 1 atm) having a molecular composition of 44.1 % hydrogen, 51.3 % nitrogen and 4.6 % methane.

In a previous report,[4] the radiolytic yields for Hydyne were calculated approximately from the experimental yields for its components, Hydyne being a mixture of 60 wt-% 1,1-dimethylhydrazine and 40 wt-% diethylenetriamine. The calculated and experimental volumes for total off-gas from a 100-ml sample are respectively as follows (25°C, 1 atm): (non-scavenged) 164.1 and 149.4 ml; (scavenged) 136.7 and 153.1 ml. These results are in fair agreement, and are not in better agreement possibly because unpredictable synergistic effects occur when a mixture of 1,1-dimethylhydrazine and diethylenetriamine is irradiated. While the previously calculated, approximate results indicate that methyl methacrylate scavenger reduces the volume of total off-gas by 16.7 %, the experimental results of the present report show that there is no reduction.

MONOMETHYLHYDRAZINE, 1,1-DIMETHYLHYDRAZINE AND DIETHYLENETRIAMINE

The results of the radiolysis of these fuels have been considered previously in detail.[1,4]

INTERCOMPARISONS OF FUELS

The averaged volumetric yield data of Table 1 allow comparisons of the fuels.

Total Off-Gas

Considering the non-scavenged fuels, RP-1 fuel produces the smallest quantity of radiolytic total off-gas, with hydrazine, diethylenetriamine, Hydyne fuel, 1,1-dimethylhydrazine and monomethylhydrazine producing more by the respective factors of 1.8, 2.0, 3.0, 4.0 and 4.5. Considering scavenged fuels, the same order exists, and the respective factors are 2.4, 2.2, 3.7, 4.0, and 6.0.

Hydrogen

Hydrogen gas is an important radiolytic product for all the fuels, non-scavenged or scavenged. Considering the non-scavenged fuels, hydrazine generates the smallest quantity of hydrogen gas, with RP-1 fuel, Hydyne fuel, monomethylhydrazine, 1,1-dimethylhydrazine and diethylenetriamine generating more by the respective factors of 1.1, 1.8, 2.1, 2.3, and 2.4. Considering scavenged fuels, hydrazine and RP-1 fuel generate the smallest quantity (the same) of hydrogen gas, with 1,1-dimethylhydrazine, Hydyne fuel, diethylenetriamine and monomethylhydrazine generating more by the respective factors of 1.6, 1.7, 2.0, and 2.1.

Nitrogen

Nitrogen is an important volumetric product for those fuels having two nitrogen atoms mutually bonded: hydrazine. Hydyne fuel, monomethylhydrazine and 1,1-dimethylhydrazine. When these fuels are non-scavenged, hydrazine evolves the smallest quantity of radiolytic nitrogen gas, with Hydyne, monomethylhydrazine and 1,1-dimethylhydrazine evolving more by the respective factors of 1.4, 1.6 and 1.9. When these fuels are scavenged, hydrazine still produces the smallest quantity of nitrogen gas, with Hydyne, monomethylhydrazine and 1,1-dimethylhydrazine evolving more by the respective factors of 1.3, 1.4 and 1.5. Nitrogen gas is an insignificant product for diethylenetriamine, which contains three nitrogen atoms not mutually bonded.

Methane

Monomethylhydrazine produces by far the largest volume of radiolytic methane. Without scavenger, diethylenetriamine produces none, RP-1 fuel produces 1.5 ml, and Hydyne fuel, 1,1-dimethylhydrazine and monomethylhydrazine out-produce RP-1 fuel by the respective factors of 3.9, 8.5 and 42. With scavenger, RP-1 fuel produces 1.1 ml of methane, and Hydyne fuel, 1,1-dimethylhydrazine, diethylenetriamine and monomethylhydrazine out-produce RP-1 fuel by the respective factors of 6.5, 8.8, 10.5 and 70

FINAL RESULT

This report terminates the problem on the stability of storable liquid rocket fuels in ionizing space and reactor radiations. Free-radical scavengers did not significantly reduce the radiolytic off-gas with any of the fuels studied: hydrazine, RP-1, Hydyne, monomethylhydrazine, 1,1-dimethylhydrazine, and diethylenetriamine. This result could be determined only by experimentation, and was due to the fact that all fuels decomposed wholly or almost wholly via a molecular and/or ionic process rather than by a free-radical process.

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