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Shelf Life Prediction of a Novel Liquid Fuel, 2-Dimethylaminoethyl Azide (DMAZ)

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Abstract: 2-Dimethylaminoethyl azide (DMAZ) is a good replacement for the hydrazine family in space industries. In this article, the accelerated ageing test method was applied for predicting the shelf life of DMAZ. The effective parameters on the storage of the fuel were temperature, the type of gas atmosphere with its pressure over the liquid fuel, and moisture. Appropriate conditions for DMAZ storage were N_2 at a pressure of 3 bar and a moisture content of 0.05 wt.%. The sigmoid form of the decomposition curves obtained revealed that the decomposition reaction is autocatalytic. Modelling of the decomposition rate showed that the shelf life of DMAZ was 7.73 years at 25 °C.

Keywords: DMAZ, shelf life, temperature, moisture, modelling

1 Introduction

Hydrazine and its derivatives (including anhydrous hydrazine, monomethylhydrazine (MMH) and unsymmetrical dimethylhydrazine (UDMH)) have been extensively used in satellite carrier rockets since 1959 [1-3]. All members of this group are carcinogenic [1, 4]. Scientists and engineers are searching for a liquid fuel as a good replacement. 2-Dimethylaminoethyl azide (DMAZ) was found to be a good replacement and was introduced by the US army in 2001 [5-10]. It is non-carcinogenic and has desirable characteristics such as good thermophysical, energetic, safety and environmental properties [5, 11, 12]. Also, some of the performance properties of DMAZ with various liquid oxidizers have been studied [13].

The liquid DMAZ produced, like other space fuels, should be stored in storage tanks near the launch station. For this purpose, it is poured into the tanks and pressurized with an inert gas. Therefore, it is important to know the ambient conditions (such as temperature, atmosphere above the liquid DMAZ and moisture) for storage of liquid fuel DMAZ. Since DMAZ is a novel liquid fuel, there are no published data for its storage. However, there is adequate information for other liquid fuels. Sun and Law studied the thermal decomposition of MMH [14]. Experimental work was reported by Widegren and Bruno on the thermal decomposition of the liquid fuels RP-1 and RP-2 [15, 16]. Also, Cordes derived a first order equation for the degradation of UDMH during the thermal decomposition reaction [17]. Moreover, Zabarnick studied jet fuel stability in the presence of oxygen [18]. Recently, Gorji and Mohammadi investigated the shelf life of the xylidine – trimethylamine mixture [19, 20]. The effect of moisture on the thermal stability of the fuels was studied by Guoa *et al*. [21]. Based on these studies, the effective parameters for storage of a liquid fuel are essentially: temperature, type and pressure of atmosphere over the liquid fuel, and moisture. The undesirable products of fuel decomposition or destruction reaction(s) may influence some of the fuel's properties (such as boiling and melting points, viscosity, surface tension, formation of a new phase in the storage tank *etc*.) and ultimately the performance of the fuel [22]. For the novel liquid fuel DMAZ, these storage parameters need to be studied.

A time-consuming method for estimating the shelf life or storage time of a fuel is surveillance of the storage tank under normal storage conditions for a prolonged period of time and measurement of the performance parameters regularly to ascertain its actual shelf life. However, the use of accelerated ageing for fuels at elevated temperatures has been advised. On the basis of the shelf life at these temperatures, the shelf life under normal storage conditions (usually 25 °C) is calculated by the Arrhenius equation [20, 23-26].

In the present work, the effective parameters for the storage of liquid fuel DMAZ have been studied at elevated temperatures. A kinetic model has been presented for the prediction of the shelf life of DMAZ at normal storage temperatures.

2 Material and Methods

2.1 Chemicals

DMAZ was synthesized by the reaction between 2-dimethylaminoethyl chloride and sodium azide and concentrated in a vacuum distillation column (at P=40 kPa)

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up to a purity of 99.93 wt.% [27]. It was colourless. Distilled water was used to study the effect of moisture.

2.2 Apparatus

The arrangement used for this work consisted of a Pyrex glass cylinder (20 cm^3) equipped with a cap and had an inverted U-tube in the tail. A stainless steel 316-L cylinder, with an internal diameter of 80 cm and height of 90 cm, was also used. The metallic cylinder was equipped with a pressure gauge (Fentinelli model, Solbiate Olona Co., Italy) and a metallic effluent valve (D3712G2Y model, Germany) for purge gases.

2.3 Test procedure

Five cubic centimeters of liquid fuel DMAZ was poured into the glass cylinder, which was then inserted in the metallic cylinder. The contents of the glass cylinder was flushed several times with an inert gas to remove oxygen. The purge number was calculated as 6 with a pressure of 4 bar [28]. Then, the closed metallic cylinder was inserted in a programmable oven (model UFB-500-L, Memmert Company, Germany). After certain time periods, liquid samples were removed from the cylinder for analysis.

2.4 Analysis

A four-digit analytical balance (Sartorius, AC121S model, Germany) was used to weigh the initial DMAZ samples.

The water content in the initial DMAZ sample was measured by a Karl-Fischer titration apparatus (KF 701 Titrino model, Swiss).

For the determination of the DMAZ concentration in the shelf life tests, samples were injected into a calibrated gas chromatograph (model 6890, Agilent Technology Co., USA) equipped with a flame ionization detector. The detector temperature was 270 °C. Helium was used as the carrier gas at a flow rate of 1.5 mL/min.

3 Results and Discussion

As was mentioned earlier, the studied parameters were temperature, moisture, type of atmosphere gas over the liquid DMAZ and its pressure. Pyrex glass was used to eliminate any effect of storage tank material. The parameter levels used in the experiments are tabulated in Table 1.

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Table 1. Parameters levels used in the shelf life study for liquid fuel DMAZ

Parameters	Levels
Temperature [°C]	60, 70, 80, 90
Atmosphere over liquid DMAZ	N_2, He, Ar, zero air
Absolute pressure of the atmosphere over liquid DMAZ [bar]	1, 2, 4, 5
Moisture [wt.%]	0.05, 0.35, 1.04, 2.53

Pure DMAZ is colourless. But in the accelerated tests, the colour of the samples changed over time. As may be observed in Figure 1, the samples' colour became gradually yellowish and finally black.

Figure 1. The change in colour of the samples during the accelerated ageing test

3.1 Effect of temperature on the shelf life of DMAZ

The effect of temperature on the DMAZ concentration is shown in Figure 2 at four temperatures, 60 °C, 70 °C, 80 °C and 90 °C. As is shown, the DMAZ concentration changes very slowly initially. Then, the slope changes significantly. Also, greater changes in the DMAZ concentration are observed at the higher temperatures. DMAZ decomposition may occur through N-N_2 bond cleavage, with nitrene formation and N_2 release [29-31]:

$$(CH_3)_2\text{-N-}CH_2\text{-}CH_2\text{-}N_3 \rightarrow (CH_3)_2\text{-N-}CH_2\text{-CH=NH} + N_2 \quad (1)$$

An increase in the vessel pressure was observed when the temperature was increased, confirming the thermal decomposition of DMAZ. Since a nitrene is a very reactive intermediate [32-34], it may react with DMAZ molecules leading to degradation of the DMAZ, so that the DMAZ degradation reaction

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proceeds autocatalytically. An increase in temperature also leads to more rapid decomposition of DMAZ.

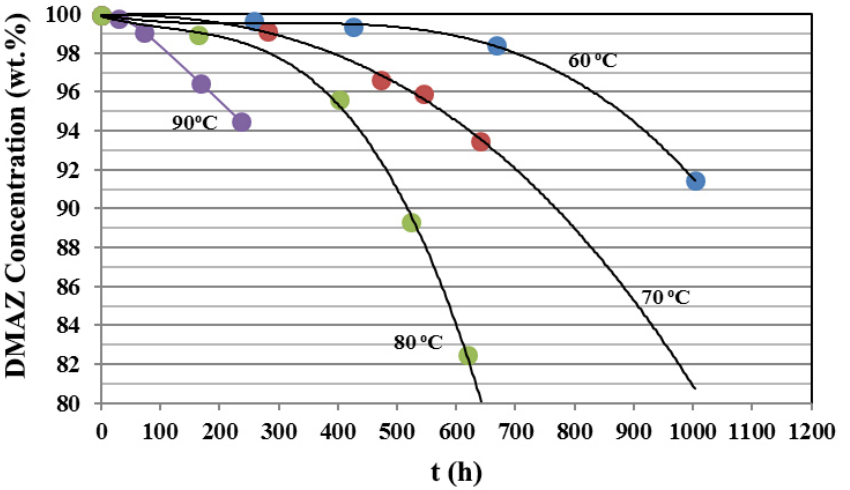

Figure 2. The destruction *vs*. time of liquid fuel DMAZ at four different temperatures (atmosphere: N_2, atmosphere pressure: 3 bar, moisture: 0.05 wt.%)

3.2 Effect of atmosphere type on the shelf life of DMAZ

N_2, Ar, He and zero air (or dry air) gases were used to evaluate the effect of the atmosphere type on the shelf life of liquid fuel DMAZ (Figure 3). The results showed that dry air had the most significant effect on the decomposition rate. This is due to the presence of oxygen in the zero air. In other words, DMAZ is oxidized in the presence of oxygen.

As was stated earlier, the temperature has a destructive effect on the shelf life of DMAZ. Therefore, the rate of heat input and its diffusivity within the liquid or gas phase should be lowered. Among the inert gases, N_2 exhibited the lowest decomposition rate. This is because of the low thermal diffusivity of N_2. Also, heat passes rapidly through He gas because helium conducts heat quickly relative to its volumetric heat capacity or thermal bulk. Table 2 shows the thermal diffusivity of N_2, Ar and He. As shown, N_2 has the lowest thermal diffusivity.

Table 2. Thermal diffusivity of the inert gases N_2, Ar, He [35]

Gas atmosphere	N_2	Ar	He
Thermal diffusivity [m^2/s]	1.6×10^{-5}	2.2×10^{-5}	1.9×10^{-4}

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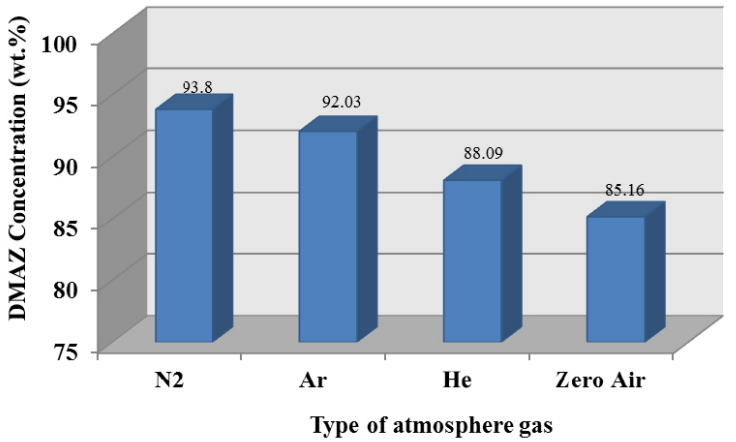

Figure 3. Effect of atmosphere type on the decrease in DMAZ concentration (vessel material: Pyrex, atmosphere pressure: 3 bar, moisture: 0.05 wt.%, T = 90 °C, t = 1000 h)

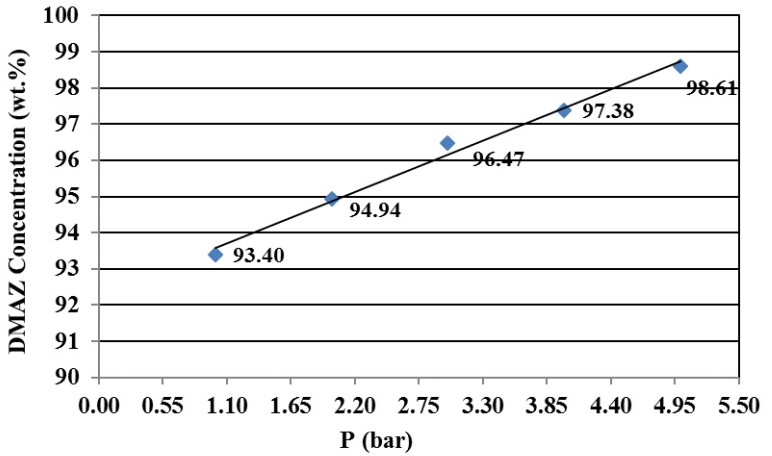

Figure 4. Effect of N_2 pressure on the decrease in DMAZ concentration (atmosphere: N_2, moisture: 0.05 wt.%, T = 90 °C, t = 1000 h)

3.3 Effect of atmosphere pressure on the shelf life of DMAZ

As was mentioned in Section 3.1, degradation of DMAZ occurs in the gas phase and subsequently the decomposition products return to the liquid phase and in turn accelerate autocatalytically the DMAZ decomposition. To prevent this, the vaporization rate of liquid DMAZ should be lowered. This rate depends on

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the atmospheric velocity distribution above the liquid, the partial pressure of DMAZ and its diffusion coefficient in the gas phase [36]. If pure liquid DMAZ is pressurized with an inert gas such as N_2 at an elevated temperature of 90 °C, the gas velocity above the liquid and the diffusivity of DMAZ in N_2 will be reduced. Also, since the vapour pressure of DMAZ at 90 °C is about 0.8 bar [37] (normal boiling point of DMAZ is 135 °C [37]), the partial pressure of DMAZ in the gas phase will be lowered. Figure 4 shows that an increase in pressure of an inert gas such as N_2 reduces the decomposition rate of DMAZ.

3.4 Effect of moisture on the shelf life of DMAZ

The presence of water in DMAZ generates an electrolyte. Water is a dielectric material which can change the electrostatic interaction between charges resulting from ions in solution. The viscosity of water is greater than that of DMAZ [38]. Thus the viscosity of the mixture is higher than that of pure DMAZ, so that the motion of electrostatically charged entities will be slower within the mixture and the energy distribution will not be proper, leading to the formation of molecules with higher energy than the activation energy barrier. Thus the entities with higher energy destroy DMAZ molecules faster. The products from decomposition of DMAZ may react with water and produce nitro acid or nitric acid. The above acids may again react with DMAZ molecules and accelerate the destruction of DMAZ. The effect of the moisture content is shown in Figure 5. As is illustrated, DMAZ storability will be longer at the lower moisture content.

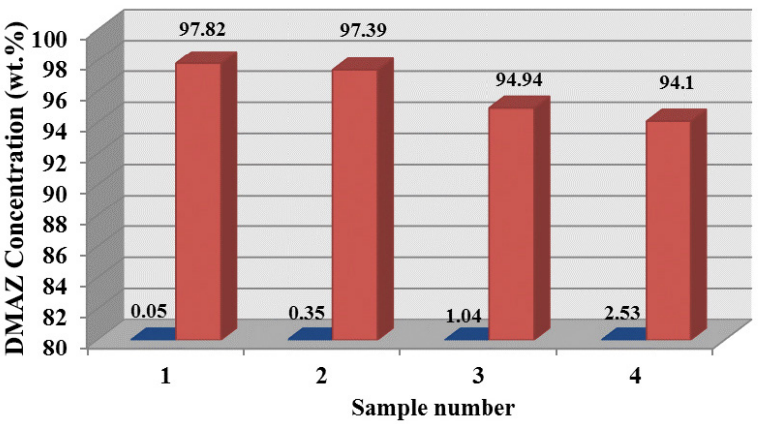

Figure 5. Effect of moisture on DMAZ concentration: moisture percentage (wt.%), final concentration of DMAZ (wt.%), (atmosphere above the DMAZ liquid: N_2, atmosphere pressure: 3 bar, T = 90 °C, t = 1000 h)

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3.5 Modelling of the decomposition rate

The sigmoid form of the decomposition curves (Figure 2) suggests that the decomposition reaction of DMAZ should be autocatalytic [39]. Generally, autocatalytic reactions are represented as:

$$A \xrightarrow{k_1} B + C \tag{2}$$

$$A + B \xrightarrow{k_2} B + C \tag{3}$$

where A is DMAZ, and B and C are decomposition products. B behaves as a decomposition catalyst. It is assumed that Equation 2 is a first order reaction. The autocatalytic reaction occurs in Equation 3 [40]. If the DMAZ concentration at t=0 and t=t were *a* and (*a*-*x*), respectively, the rate equation may be expressed as:

$$\frac{dx}{dt} = k_1(C_{o,DMAZ} - x) + k_2 x(C_{o,DMAZ} - x) \tag{4}$$

By integration and insertion of x=0 at t=0, the ordinary differential equation (ODE) may be solved as:

$$\ln\frac{(k_1 + k_2 x)C_{o,DMAZ}}{(C_{o,DMAZ} - x)k_1} = (k_1 + k_2 C_{o,DMAZ})t \tag{5}$$

where:

x reduction in DMAZ concentration or DMAZ conversion,
k_1 the rate constant of the first order reaction,
k_2 the rate constant of the autocatalytic reaction,
$C_{o,DMAZ}$ initial concentration of DMAZ,
t: time.

Given x at any time, the rate constants were calculated using MATHCAD software (Version 14) at temperatures of 60 °C, 70 °C, 80 °C and 90 °C. The results are tabulated in Table 3.

Table 3. Decomposition reaction rate constants for DMAZ decomposition at different temperatures

Reaction rate constant	Temperature [°C]			
	60	70	80	90
k_1 [s^{-1}]	5.07×10^{-6}	1.40×10^{-5}	3.12×10^{-5}	1.03×10^{-4}
k_2 [s^{-1}]	4.27×10^{-3}	5.21×10^{-3}	5.95×10^{-3}	6.25×10^{-3}

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Plots of Lnk_1 and Lnk_2 versus 1/T represent negative slopes $-E_1$/RT and $-E_2$/RT respectively, where T is in Kelvin (Figures 6 and 7). The activation energies for Equations 2 and 3 were determined as 98.7 kJ/mol and 15.8 kJ/mol, respectively. In other words, the activation energy for the autocatalytic path (Equation 3) is about one-sixth of the first order path (Equation 2).

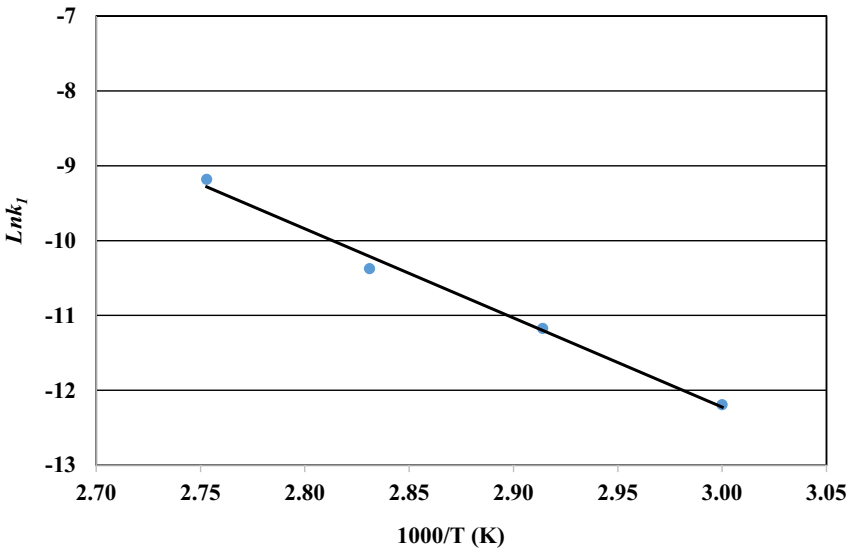

Figure 6. Temperature dependency of k_1

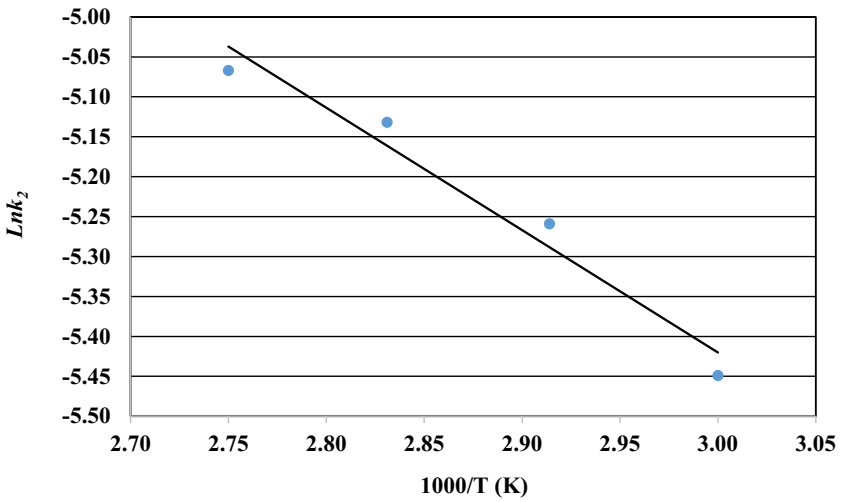

Figure 7. Temperature dependency of k_2

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3.6 Estimation of the shelf life of liquid fuel DMAZ

Under the appropriate conditions for DMAZ storage (vessel material: Pyrex, N_2 as the atmosphere over liquid fuel DMAZ at a pressure of 3 bar, moisture content of 0.05 wt.%), the shelf life is predictable. Since liquid fuel DMAZ is novel, no military standard data has as yet been reported for it. Therefore, based on the conventional standards for liquid fuels such as monomethylhydrazine (MMH) [41] or unsymmetrical dimethylhydrazine (UDMH) [42], if the acceptable DMAZ concentration were to be 98 wt.%, the time required to change the DMAZ concentration from 99.93 wt.% to 98 wt.% at 25 °C may be calculated using Equation 5. All of the parameters for Equation 5 are tabulated in Table 4, where x is the conversion percentage (99.93% − 98% = 1.93% = 0.0193). Thus, the shelf life was calculated as 67780 h or 7.73 years. Therefore, it seems that the novel liquid fuel DMAZ has a relatively good shelf life.

Table 4. Parameters used for the estimation of the shelf life of DMAZ at 25 °C (298 K)

Parameter	a	x	k_1	k_2
Value	99.93	1.93	7.71×10^{-8}	3.32×10^{-5}

4 Conclusions

To estimate the shelf life of liquid fuel 2-dimethylaminoethyl azide (DMAZ), the fuel was tested under accelerated conditions of temperature (60-90 °C), gas atmosphere over the liquid fuel (N_2, Ar, He and zero air) at pressures of 1-5 bar and moisture content (0.05-2.53 wt.%). The results showed that the appropriate conditions for storage of DMAZ are: N_2 as the atmosphere gas at a pressure of 3 bar and a moisture content of 0.05 wt.%. The sigmoid form of the decomposition curves showed that the decomposition reaction should be autocatalytic. The temperature dependencies of the decomposition reaction rate constants were determined. Under the appropriate conditions of storage, the calculations showed that the shelf life of this fuel was 7.73 years at 25 °C.

Abbreviations and symbols

$C_{o,DMAZ}$ Initial concentration of DMAZ
DMAZ 2-Dimethylaminoethyl azide ($(CH_3)_2$-N-CH_2-CH_2-N_3)
E_i Activation energy for step i [kJ/mol]
k_1 The rate constant of the first order reaction [s^{-1}]
k_2 The rate constant of the autocatalytic reaction [s^{-1}]

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MMH Monomethylhydrazine
P Pressure [bar]
R Universal gas constant [8.314 J/mol·K]
T Temperature [°C or K]
t Time [h]
UDMH Unsymmetrical dimethylhydrazine
x Reduction in DMAZ concentration or DMAZ conversion

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