ADN Propellant Development

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Abstract

Ammonium dinitramide (ADN), is a powerful oxidizer salt of interest for both missile and space propulsion applications. It has the potential not only to improve the performance, but also to make future propellants more environmental benign. It is mainly intended as oxidizer in solid propellants, but it can also be used in liquid monopropellants dissolved in a fuel/water blend. The physical and toxicological properties of ADN have thoroughly been studied during the last years. The properties of ADN and an overview of the development of solid and liquid ADN-based propellants are presented in this paper.

1. Introduction

Useful oxidizers for rocket propellant applications are rare. Thus the relatively newly discovered inorganic solid salt, ammonium dinitramide (ADN), $NH_4N(NO_2)_2$, has gained substantial attention. ADN was first synthesized in 1971 at the Zelinsky Institute of Organic Chemistry in Moscow. It is claimed that ADN-based solid propellants are in operational use in Russian Topol intercontinental ballistic missiles [1] and that ADN previously was produced in ton-size quantities in the former USSR [2]. The USSR's dinitramide technology was strictly classified and unknown to the rest of the world until 1988 when it was "re-invented" at SRI in the USA [3]. In the beginning of the 1990s, the Swedish Defence Research Agency, FOI, started its research on ADN in order to develop minimum smoke propellants for tactical missile applications.

ADN has the potential not only to improve the performance, but also to make future propellants more environmental benign. It is today of interest for both missile and space propulsion applications. Currently ADN is considered in;

- Solid propellants
    - to replace ammonium perchlorate in composite propellants
    - to replace double-base in minimum smoke propellants
- Liquid monopropellants
    - to replace monopropellant hydrazine

This paper presents an overview of the solid and liquid ADN propellant development at FOI, basic properties of ADN, current projects and future perspectives.

2 Properties of ADN

ADN (CAS no. 140456-78-6) is a solid white salt of the ammonia cation (NH_4^+) and the dinitramide anion ($N(NO_2)_2^-$), Figure 1. It has a high oxygen balance, +25.79 %, melts at 93 °C and starts to decompose at approximately 150 °C at a heating rate of 10 K per minute, as seen in Figure 2. Similarly to ammonium nitrate, ADN is hygroscopic and readily soluble in water and other polar solvents but scarcely soluble in non-polar solvents. The solubility of ADN in different solvents is shown in Table 1, and the phase diagram for the system ADN-water is shown Figure 3. The critical relative humidity for ADN is 55.2 % at 25.0 °C [5]. This means that the relative humidity must be below 55.2 % to prevent ADN from absorbing moisture from the atmosphere. The density of ADN in the solid state is 1.81 g/cm^3 [6]. Its molar volume and corresponding density in the liquid state at 25.0 °C is 74.08 g/mol and 1.675 g/cm^3 respectively [5].

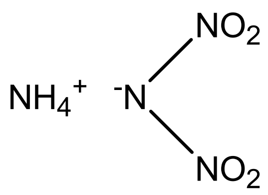

Figure 1: The structure of ADN.

Table 1: Solubility of ADN at 20.0°C [4].

Solvent	Solubility in 100 g solvent	
	(g)	(%)
Water	357	78.1
Methanol	86.9	46.5
Butyl acetate	0.18	1.8e-3
N-heptane	0.005	0
Dichloromethane	0.003	0

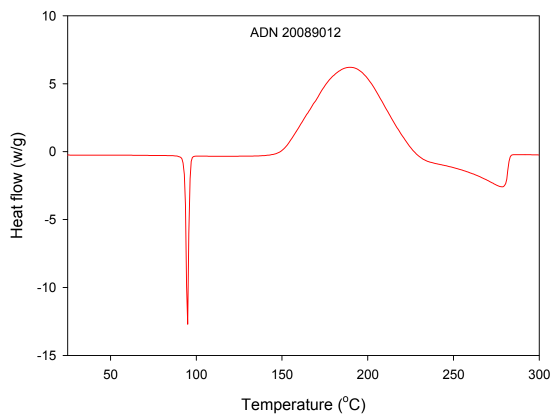

Figure 2: Differential Scanning Calorimetry (DSC) thermogram of ADN. Heating rate 10 K/min. [7].

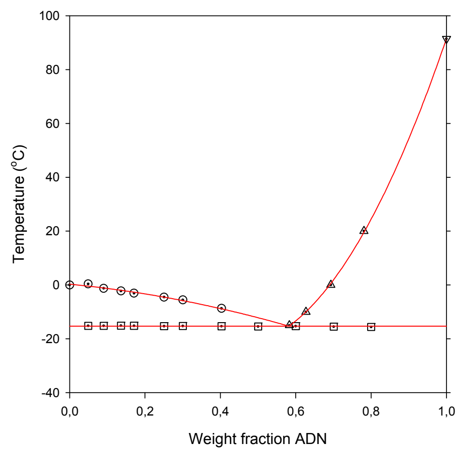

Figure 3: Solid-liquid phase diagram for the system ADN-water [5].

In order to calculate the specific impulse and to accurately evaluate the combustion efficiency, the heat of formation, ΔH_f, for respective propellant ingredient must be known. In the literature the heat of formation for ADN varies substantially from -162.8 kJ/mol [8] to -125.3 kJ/mol [9]. In one of the most thorough studies published the heat of formation for ADN was determined by two independent methods, using combustion and solution calorimetry, to be -134.6±0.46 kJ/mol [10].

It seems to exist a linear relationship between the heats of formation of nitrates and dinitramide salts having the same cation, as seen in Figure 4. Thus, if the heat of formation for a nitrate salt (XN) is known, the heat of formation of its corresponding dinitramide salt (XDN) can be estimated according to equation 1 [11],

$$\Delta H_f(XDN) = \Delta H_f(XN) + 230n \quad \text{(kJ/mol)} \qquad (1)$$

where n is the number of anions per cation. Using the established data for the heat of formation for AN (-365.6 kJ/mol [12, 13]), the heat of formation for ADN was estimated to -135.6 kJ/mol, which is in good agreement with the experimental value (-134.6 kJ/mol). Thus this experimental value seems reasonable and is recommended for use. Table 2 show the heat of formation for AN and ADN and their respective ions, as well as the heat of solution, ΔH_{sol}, for AN and ADN. The heat of formations and the heat of solution for respective salt are related to each other according to equation 2.

Table 2: Enthalpies for AN and ADN and their respective ions.

Salt, AY	**$\Delta H_f\ (AY)_s$ (kJ/mol)**	**$\Delta H_{sol}\ (AY)$ (kJ/mol)**	**$\Delta H_f\ (Y)_{aq}$ (kJ/mol)**	**$\Delta H_f\ (NH_4)_{aq}$ (kJ/mol)**
AN	-365.6 [12, 13]	+25.5 [14]	-206.8 [12]	-133.3 [12]
ADN	-134.6 [10]	+36.4 [10]	+35.1 [10]	-133.3 [12]

$$\Delta H_f(AY)_s + \Delta H_{sol}(AY) = \Delta H_f(Y^-)_{aq} + \Delta H_f(NH_4^+)_{aq} \tag{2}$$

ADN is today produced by EURENCO Bofors in Sweden [15]. The purity of the material is above 99% pure and it has a high thermal stability. Thus no stabilizer is required to be added. Some of the physical properties of ADN are summarized in Table 3, and information concerning its toxicological properties is shown in Table 4. According to the toxicological tests, ADN is considered as non-carcinogenic and non-allergenic. It may cause irritation if inhaled and it is harmful if swallowed but is not irritating in contact with eyes or skin [16].

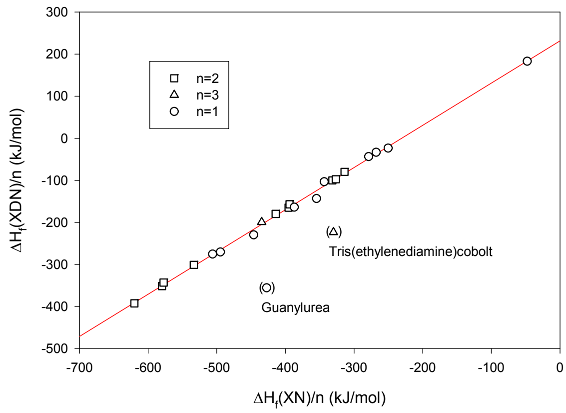

Figure 4: ΔH_f of XDN/XN pares divided by the number, of anions per cation. Salts in parenthesis were excluded in the linear regression analysis [11].

Table 3: Properties of ADN at 25.0°C.

Property	**Value**
Molecular weight	124.07 g/mol
Oxygen balance	+25.79 %
Melting point	93.2°C [17]
Heat of formation	-134.6 kJ/mol [10]
Heat of solution	+36.4 kJ/mol [10]
Heat of combustion	437.0 kJ/mol
Density (solid)	1.81 g/cm^3 [6]
Density (liquid)	1.675 g/cm^3 [5]
Molar volume (liquid)	74.08 cm^3/mol [5]
Critical relative humidity	55.2 % [5]

Table 4: ADN toxicity data.[a]

Acute toxicity	LD_{50} (oral rat) 823 mg/kg [18] LD_{50} (dermal rabbit) >2000 mg/kg [18] LC_{50} Not applicable[b]
Toxicity to aquatic organisms	EC_{50} >10000 mg/l (15 min)
Acute inhalation	May cause irritation
Sensitizing	Not sensitizing
Effect on skin	Not irritating
Effect on eyes	Not irritating
Lipo-philicity	Log Po/w < -2.8
Mutagenicity	Mammalian cell, negative

a) Data from reference [16] unless otherwise stated.
b) ADN is a salt and is thus not present in the gas phase.

3. ADN-based solid propellants

Current solid composite propellants are based on the oxidizer ammonium perchlorate (AP). AP is in many ways an excellent oxidizer due to its relative low hazardness and the possibility to tailor its ballistic properties. Unfortunately, AP has negative impacts on the environment and on personal health [19, 20]. Similarly, there is a need for more environmentally benign minimum smoke propellants to replace current lead containing double-base propellants [21].

Though the development of ADN-based solid propellants has faced a number of challenges, the research has continued motivated by the high potential. Through the years, many technical problems have been solved, such as synthesis, stability, compatibility etc. One of the most critical problems has been the production of ADN particles with acceptable morphology. The particle shape of ADN received from EURENCO is needle shaped and thus not suitable for formulation.

At FOI a method to produce spherical ADN particles, prills, have been developed [22]. The prills are produced by spraying molten ADN through a nozzle. In the nozzle the molten ADN is atomized to form droplets which then solidify to the desired prills seen in Figure 5. The particle size can be controlled to some extent by varying spray nozzle size and pressure. Typical particle sizes (d_{50}) for the fine and coarse prills produced are 60 and 200 μm respectively [23]. The prills are produced using up to 250 g ADN per batch. Currently work is ongoing to scale up the batch size and to improve the control of the particle size and distribution.

As binder material several different polymers have been studied, including HTPB and polyglycidylazide (GAP). In our initial trials formulations with HTPB/ADN seem to provide high burn rate exponent (~0.9) [24]. However, in the work performed no burn rate modifiers or ballistic additives were used. Further work should thus look into possible burn rate modifiers and to study the influence of the ADN-particle size on the ballistic properties.

At this stage the focus has been on using an energetic binder based on GAP. GAP was chosen because it:

- is compatible with ADN
- improves performance
- provides good ballistic properties in combination with ADN.

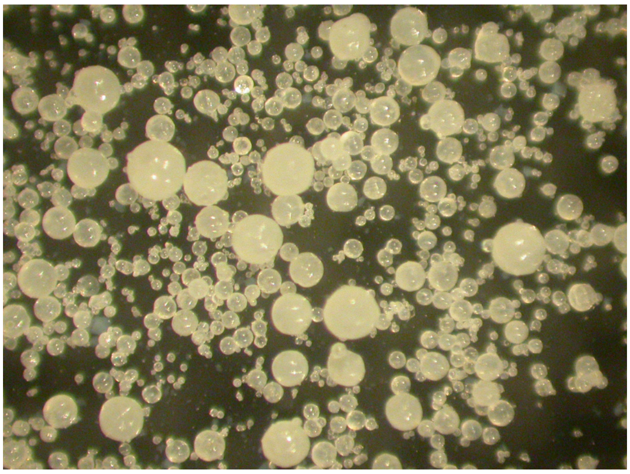

Figure 5: Spray-prilled ADN [23].

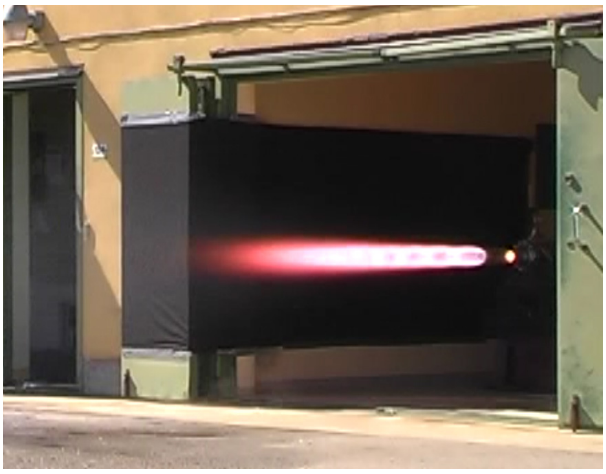

Figure 6: Test firing of a 3 kg ADN/GAP rocket motor [23].

An ADN propellant with a GAP-based binder has been formulated containing 70% bimodal prilled ADN [23]. The propellant had low viscosity and higher solid loading can be used. Batches up to 3.75 kg propellant have been mixed and casted. The propellant had excellent thermal stability, high burning rate and a pressure exponent below 0.5. 3 kg case bonded grains were casted and test fired, Figure 6 [23]. The results from the test firing confirmed the minimum smoke characteristics and the high specific impulse. The ADN/GAP propellant did not contain any plasticizer or bonding agent and its mechanical properties needs to be improved to obtain the desired mechanical properties.

The recently started European project *HISP* aims to develop ADN-based solid propellants for space crafts. The project is funded by the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement no. 262099. The *HISP* project will advance the chemical propulsion technology by linking together some of the key players in Europe to develop an advanced high performance solid propellant by using ADN, GAP and high energy density fuels such as aluminium hydride (AlH_3), nano-aluminium or activated aluminium. Apart from developing a high performance ADN-based propellant, the aim of the project is also to promote the industrial production of ADN by improving the synthesis and prilling methods and to estimate the future cost of ADN when produced in large quantities. More information about *HISP* can be found on: www.hisp-fp7.eu.

4. Liquid ADN-based monopropellants

State of the art monopropellant hydrazine is highly toxic, volatile and carcinogenic, and has limited performance. This has prompted the search for new superior propellants to replace hydrazine. One of the most promising alternatives to monopropellant hydrazine is blends based on an oxidizer salt dissolved in a fuel/water mixture. Similarly as hydroxylammonium nitrates (HAN) [25-29], ADN can be used for this purpose due to its high solubility in polar solvents. The development of ADN-based monopropellants started at FOI in 1997 on a contract from SSC.

The first fuels considered in the development of ADN/fuel/water mixtures were acetone, ethanol and methanol respectively. Low volatile fuels such as 1,4-butanediol, glycerol, ethylene glycol and trimethylol propane where studied to minimize the amount of ignitable and/or toxic fumes. First glycerol was chosen due to the superior thermal ignition properties of the ADN/glycerol/water-blend. This monopropellant formulation was called LMP-101 [30]. However, it was discovered that LMP-101 suffered from poor thermal stability, and as a consequence it was rejected from further development. During the years, several different ADN-based monopropellants have been developed [31, 32]. Two formulations, LMP-103S and FLP-106 have received particular attention. LMP-103S has been selected by SSC and FLP-106 has been selected by FOI as the main monopropellant candidate for further development.

FLP-106 is a low-viscous yellowish liquid, as seen in Figure 7, with high performance, low vapour pressure and low sensitivity. It is based on a low volatile fuel, water and 64.6 % ADN. The development, characterization and selection of FLP-106 are reported elsewhere [31, 33-35]. Some of the properties of FLP-106 are shown in Table 5.

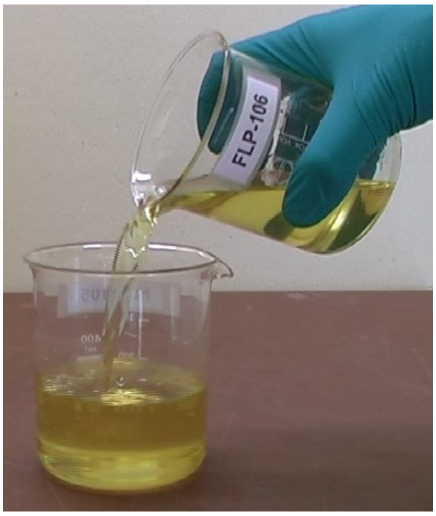

Figure 7: Monopropellant FLP-106.

Table 5. Properties of hydrazine and FLP-106[a].

Property	Hydrazine	FLP-106
Specific impulse[b] (s)	230 [36]	259
Density (g/cm^3)	1.0037	1.357
Temp. in chamber (°C)	1120	1880
T_{min} [c] (°C)	2.01	0.0
Viscosity (cP, mPas)	0.913	3.7
Therm. exp. coeff. (1/K)	$9.538 \cdot 10^{-4}$	$6.04 \cdot 10^{-4}$
Heat capacity (J/gK)	3.0778	2.41

a) All properties at 25 °C. Hydrazine data from Schmidt [37] and FLP-106 data from Wingborg et al. [31, 33-35].
b) Calculated Isp. Pc = 2.0 MPa, Pa = 0.0 MPa, ε = 50.
c) Minimum storage temperature determined by freezing (hydrazine) or precipitation (FLP-106).

One important aspect in the development of a new monopropellant is the ignition. State of the art hydrazine thrusters use catalytic ignition, which is simple and reliable. To replace hydrazine, ADN-based monopropellants must be as easy to ignite. However, a disadvantage of the ADN-based monopropellants is the high combustion temperature, which is approximately 800°C higher than hydrazine, as seen in Table 5. The combustion temperature is in the same range as for HAN-based monopropellants, and it has been reported that the current state of the art hydrazine catalyst (Shell 405) cannot withstand such high temperatures [29, 38]. This and the fact that hydrazine and ADN-based liquid propellants are very different, both physically and chemically, require development of new ignition methods, or new catalysts. When dripping FLP-106 on a hot plate, with a temperature in the range of 200 to 250°C, it ignites and burn fast. This clearly shows that thermal ignition is possible and thermal ignition might be a feasible ignition method. Three different methods of heating the propellant to the ignition temperature have been identified:

- Pyrotechnic (by forming hot gases using a solid energetic material which in turn will heat the propellant)
- Thermal conduction (by spraying the propellant on a hot object which in turn is heated by electric means)
- Resistive (ADN is a salt and the propellants thereby possess a relatively high electric conductivity. This means that an ADN-based monopropellant can be resistively heated)

Development of catalytic [39], thermal [34], and resistive [35] ignition methods is ongoing.

Both FLP-106 and LMP-103S are compatible with materials currently used in propulsion systems. They both also have similar *oral* toxicity and should be considered as harmful, but not toxic. However, FLP-106 has a substantial lower vapour pressure and requires no respiratory protection during handling. This is reflected by the amounts, boiling points and hazard labels of the fuel/water-blend in LMP-103S and FLP-106 respectively, as shown in Table **6**.

Table 6. Properties of the fuel/water-blend in LMP-103S and FLP-106 respectively.

Propellant	**Component**	**Amount (%)**	**Boiling point (°C)**	**EU CLP hazard labels**
LMP-103S	Methanol	~18	65	
	Ammonia (aq 25%)	~20	38	
FLP-106	Fuel “F-6”	11.5	>180	
	Water	23.9	100	

LMP-103S and FLP-106 are not sensitive to shock initiation and should, from this point of view, not be considered as hazard class 1.1 materials [33, 40]. The advantage using FLP-106, apart from its lower volatility, is its higher performance and higher density as shown in Table 7. The specific impulse for FLP-106 is 7 s higher compared to LMP-103S, and the density-impulse ($\rho \cdot I_{sp}$) is 13 % higher.

Table 7. Properties of ADN-based monopropellants.

Propellant	FLP-106	LMP-103S
I_{sp} (s) [a]	259	252 [41]
ρ (g/cm^3) [b]	1.362	1.240 [40]
$\rho \cdot I_{sp}$ (gs/cm^3)	353	312

a) at a nozzle area expansion ratio of 50.
b) at 20 °C.

FLP-106 is currently used and evaluated in the European project *GRASP*. The project is funded by the European Community's Seventh Framework Programme (FP7/2007-2013) under grant agreement no. 218819. The aim of GRASP is to develop less toxic or “green” propellants to replace the currently used highly toxic and carcinogenic propellants. More information about *GRASP* can be found on: www.grasp-fp7.eu. LMP-103 on the other hand has already been flight proven on the Prisma satellites[i].

[i] www.prismasatellites.se

5. Conclusions

The development of liquid and solid ADN-based propellants is well under way. A solid rocket motor with 3 kg ADN/GAP propellant has successfully been test fired and promising liquid ADN-based monopropellants have been developed and tested. It thus seems that ADN has come to a technological level where it seriously can be considered as propellants in future missiles and space crafts.

Acknowledgments

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