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Research paper

Preparation and Characterization of Cationic Energetic Salts of 5-Amino-3-[(2,4,6-trinitrophenyl)amino]-1*H*-1,2,4-triazole (APATO)

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Abstract: The synthesis and properties of some 5-amino-3-[(2,4,6-trinitrophenyl)amino]-1*H*-1,2,4-triazole (APATO) cationic salts are described. A detailed structure of one of the salts obtained, 5-amino-3-[(2,4,6-trinitrophenyl)amino]-1*H*-1,2,4-triazol-4-ium perchlorate (APATO·HClO₄) was characterized using low temperature (173 K) single crystal X-ray diffraction: orthorhombic yellow block, space group ‘Pbc21’, crystal density at 173 K (calculated) and at 293 K (pycnom.) were 1.863 and 1.840 g/cm³, respectively. The APATO salts were characterized by spectroscopic methods and thermal analysis. Preliminary computer calculated energetic characteristics showed that the synthesized salts possess increased energetic characteristics, superior in comparison to the TNT. The APATO perchlorate salt may deserve special attention as a thermostable HEDM.

Keywords: 1,2,4-triazole-3,5-diamine derivatives, nitrocompounds, X-ray diffraction, 2,4,6-trinitrophenyl derivatives, energetic salts

1 Introduction

The general requirements for modern secondary explosives include high detonation performance, insensitivity to impact and thermostability [1-5]. Energetic salts are a separate group of modern high-energy materials (HEMs), possessing various properties, including:

- propellants and oxidizers,
- primaries,
- high-density energetic materials (HEDM),
- insensitive munition components, and
- thermostable HEMs.

Some representatives of energetic salts are shown in Figure 1.

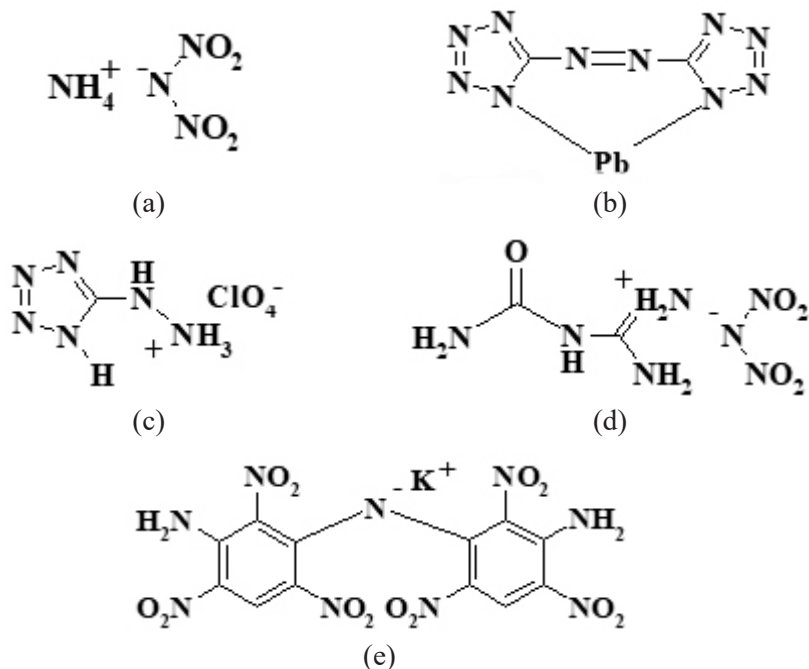


Figure 1. Representatives of different kinds of energetic salts:
 a) ammonium dinitramide (ADN), in propellants as an oxidizer [6],
 b) 5,5'-azotetrazole lead salt, as primary explosive [7],
 c) 5-hydrazinotetrazole perchlorate, in HEDM (density 2.068 g/cm³) [8],
 d) guanylurea dinitramide (GuDN, FOX-12), as an insensitive energetic material [9],
 e) 3,3'-diamino-2,2',4,4',6,6'-hexanitrodiphenylamine potassium salt, as a thermostable HEM (m. p. 334 °C) [10]

The investigations of various energetic ionic salts based on azoles were reviewed in some previous work [11-13]. The most recent requirements for new HEMs also included specific regulations for decreased environmental hazards [14]. For this reason, some azole-derived salts have deserved interest as green energetic materials [15]. Our laboratory's interest in the triazole energetic materials group started 15 years ago, when these compounds had been found to possess relatively low toxicity [16]. In previous fruitful collaboration with the Military Academy, we expanded our research in the area of thermostable HEMs and their structure-property relationships.

The thermostability of energetic compounds is also known to increase on the formation of salts [10]. The azole-based energetic salts have recently become a popular topic in the field of new energetic materials, especially structures containing imidazole, 1,2,3-triazole, 1,2,4-triazole and tetrazole [17-20]. Some triazole salts were also mentioned in a little known work by the Ukrainian researchers Panasiuk and Schestozub [21]. 5-Amino-3-[(2,4,6-trinitrophenyl)amino]-*1H*-1,2,4-triazole (APATO) is a typical amphoteric heterocyclic compound [22]. The study of four anionic energetic salts of 3-picrylamino-5-amino-*1H*-1,2,4-triazole has recently been reported by Chiato *et. al* from the Klapötke laboratory [23].

Currently our research is focused on the synthesis and structural study of different kinds of salts, specifically cationic salts of APATO. The present work expands on our previous studies on various picryl (2,4,6-trinitrophenyl) derivatives of triazole, in a search for a convenient synthesis route for energetic cation-based salts of APATO and preliminary exploration of their structures and properties as potential energetic materials.

2 Experimental

2.1 Test methods

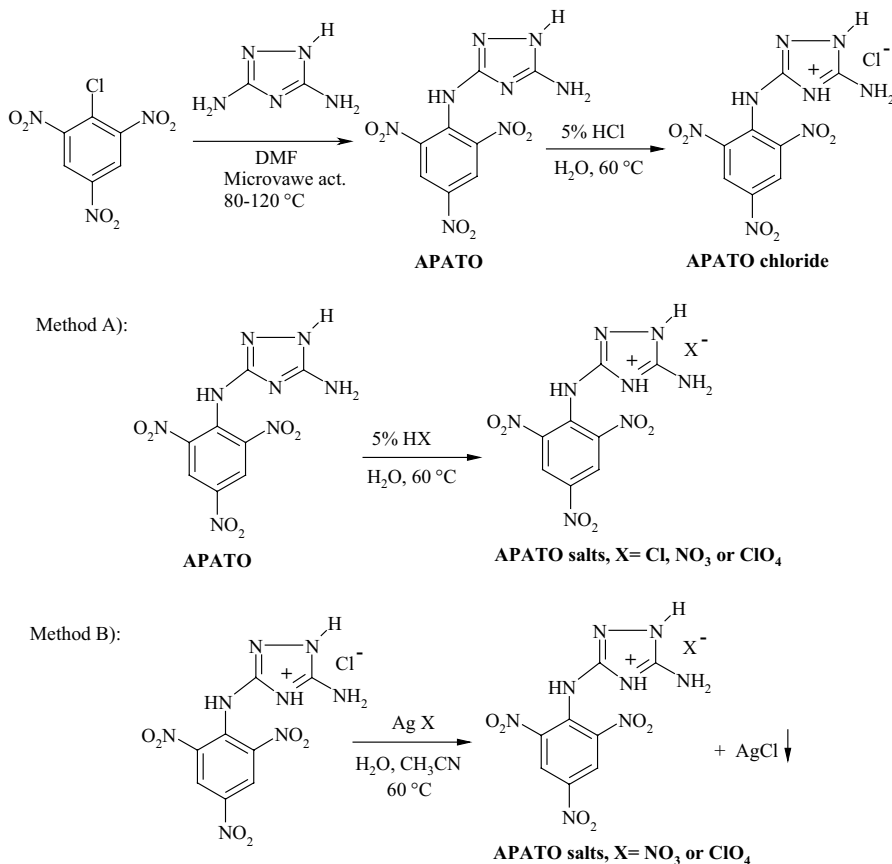
The melting points of the compounds synthesized were determined in open capillaries. The purity of the compounds was monitored by TLC using silica gel 60 F254 aluminium plates (Merck) and a LC-MS analyser Shimadzu 2020. UV-VIS spectra of the compounds were recorded using a Perkin-Elmer Lambda 25 UV-VIS spectrophotometer. IR spectra were recorded as KBr discs on a Perkin-Elmer spectrophotometer (FT-IR Spectrum BX II). NMR spectra were recorded using a Varian Unity Inova (400 MHz for ^1H NMR). For X-ray analysis a Bruker-Nonius' diffractometer with a KappaCCD detector was used. Diffraction radiation source: fine focus sealed tube; radiation monochromator: graphite. In the crystal structures hydrogen atoms were located and refined. Thermal analysis was investigated using

a differential scanning calorimeter Perkin Elmer DSC 8000. Density measurements were performed at 293 K using an AccuPyc 1330 Pycnometer (Micromeritics).

Caution! These compounds are energetic materials and tend to explode under certain conditions. Appropriate safety precautions (safety glasses, face shields, leather coat and ear plugs) should be worn, especially when the compounds are prepared on a large scale.

2.2 Synthesis

APATO was synthesized from picryl chloride and 3,5-diamino-1,2,4-triazole in DMF by using the microwave activation method, as described in our previous paper presented at a NTREM seminar [24]. The salts of APATO were prepared by two different methods (A and B, Scheme 1). The general scheme for the synthesis of APATO and its salts is shown in Scheme 1.



Scheme 1. General scheme for the synthesis of APATO salts

2.2.1 APATO salts preparation by direct reaction with aqueous acid (method A)

APATO chloride was prepared in dilute aqueous HCl. Pulverised APATO base (1 mmol) was dispersed in distilled water (10 mL), containing excess HCl (1.5 mmol), stirred on a magnetic stirrer and heated for 3 h at 70 °C. Some residual supernatant base was filtered off and the clear filtrate was concentrated *in vacuo*, until the first crystal crop appeared. The yellow solution with crystals obtained was further allowed to stand in the freezer for 12 h at +4 °C, the crystalline solid was then filtered off, was washed with a minimal volume of ice-cold water and dried in a vacuum desiccator over CaCl₂.

APATO chloride: yield: 86%, m.p. 168 °C (decomp. but not sharp, some degradation started even above 100 °C); yellow plates, C₈H₆ClN₈O₆:

– **anal.:**

(i) **found:** C(27.67) H(1.69) Cl(10.32) N(31.94),

(ii) **calc.:** C(27.80) H(1.75) Cl(10.26) N(32.42) O(27.77),

– **¹H NMR** (400 MHz, D₆-DMSO, 25 °C): δ = 8.96 (s, 2H, CH), 5.10 (br s, 5H, 3NH, NH₂) ppm.

– **¹³C NMR** (100 MHz, D₆-DMSO, 25 °C): δ = 153.3 (1C, CNH₂), 150.4 (1C, CN), 140.7 (2C, CNO₂), 138.0 (1C, CNO₂), 135.8 (1C, CN), 126.3 (2C, CH) ppm.

A similar synthesis method was further applied for the preparation of the nitrate and perchlorate salts (yields obtained by this method (A): 73% and 68%, respectively).

2.2.2 Alternative method (B) for the preparation of salts by a metathesis reaction

APATO salts (nitrate and perchlorate) by this method were prepared by applying metathesis reactions of APATO chloride with the appropriate silver salt. APATO chloride (1 mmol) was dissolved in distilled water (20 mL), and an aqueous solution of silver nitrate or perchlorate (1 mmol) was added. The reaction mixture was stirred on a magnetic stirrer and heated at 70 °C. After 3 h the solid AgCl precipitate was filtered off and washed with H₂O (5 mL), and the filtrate was concentrated *in vacuo*. The salt (X = –NO₃) or (X = –ClO₄) was isolated as a crystalline yellow solid. The yields obtained by method B were on average: 65% for APATO nitrate and 52% for APATO perchlorate.

APATO nitrate: orange crystals (needles), m.p. 146–147 °C (decomposition with flash), C₈H₆N₉O₉:

– **anal.:**

(i) **found:** C(25.90) H(1.59) N(33.79),

(ii) **calc.:** C(25.82) H(1.62) N(33.87) O(38.69),

- **¹H NMR** (400 MHz, D₆-DMSO, 25 °C): δ = 8.92 (s, 2H, CH), 7.30 (br s, 5H, 3NH, NH₂) ppm,
 - **¹³C NMR** (100 MHz, D₆-DMSO, 25 °C): δ = 153.2 (1C, CNH₂), 150.6 (1C, CN), 140.8 (2C, CNO₂), 138.2 (1C, CNO₂), 135.7 (1C, CN), 126.3 (2C, CH) ppm.
- APATO perchlorate:** yellow crystals, m.p. 234 °C (decomposition), C₈H₆ClN₈O₁₀:
- **anal.:**
 - (i) **found:** C(23.39) H(1.52) Cl(8.38) N(27.43),
 - (ii) **calc.:** (23.46) H(1.48) Cl(8.65) N(27.35) O(39.06),
 - **¹H NMR** (400 MHz, D₆-DMSO, 25 °C): δ = 8.98 (s, 2H, CH), 7.80 (br s, 5H, 3NH, NH₂) ppm,
 - **¹³C NMR** (100 MHz, D₆-DMSO, 25 °C): δ = 152.6 (1C, CNH₂), 149.5 (1C, CN), 140.9 (2C, CNO₂), 138.3 (1C, CNO₂), 135.9 (1C, CN), 126.4 (2C, CH) ppm.

2.3 Thermal analysis

The thermal behaviour of energetic materials has an important bearing on their applications. The synthesized salts were analysed for their thermal stability by DSC and TG (10 °C/min). Only the perchlorate (APATO·HClO₄) of the cationic salts of APATO studied demonstrated good thermal stability. The thermal analysis results are shown in Figure 3.

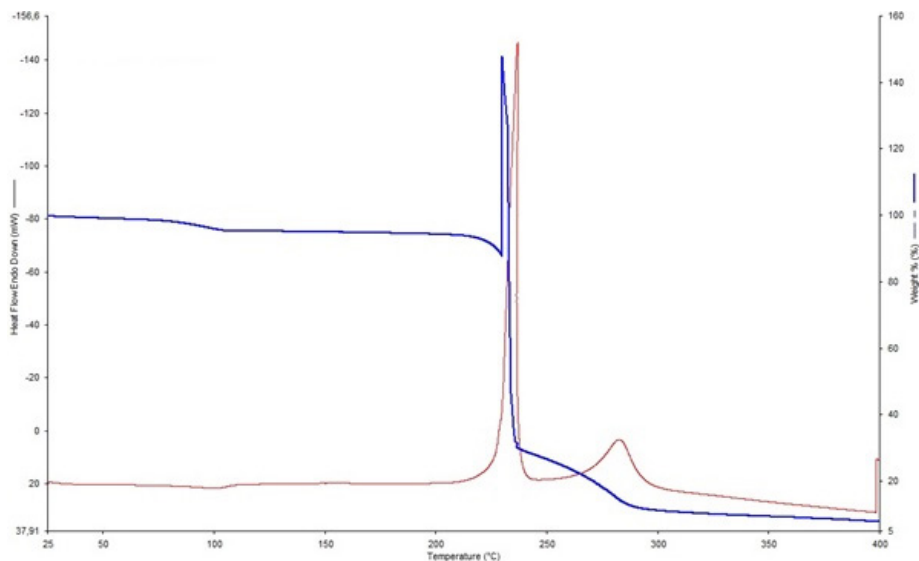


Figure 3. Thermal analysis results for the salt APATO perchlorate at 10 °C/min

The APATO perchlorate salt showed good heat resistance (decomposition started at 230 °C). Unfortunately, both of the other salts (Cl^- and NO_3^-) demonstrated a markedly decreased thermal stability. Decomposition of the nitrate salt started even on heating over 134 °C, while the chloride salt exhibited slow partial disproportionation/sublimation that began even over 100 °C, however a sharp decomposition point for this salt was reached only at 290 °C (and this value is near that of APATO base itself [25]). The data obtained in this work, showed that only the perchlorate salt can be of further interest for practical application as a potential energetic material. In our work, promising physico-chemical properties were found only for APATO·HClO₄. For this reason we have attempted a more detailed study of the structure of this salt.

2.4 Structure determination of 5-amino-3-[(2,4,6-trinitrophenyl)amino]-1H-1,2,4-triazol-4-ium perchlorate

2.4.1 APATO·HClO₄ crystal growing and structural analysis by X-Ray diffraction

APATO perchlorate crystals, suitable for X-ray structural analysis, were obtained by slow evaporation of a concentrated aqueous solution of the salt, containing dilute HClO₄, under ambient conditions (20 °C), as shown in Figure 4.

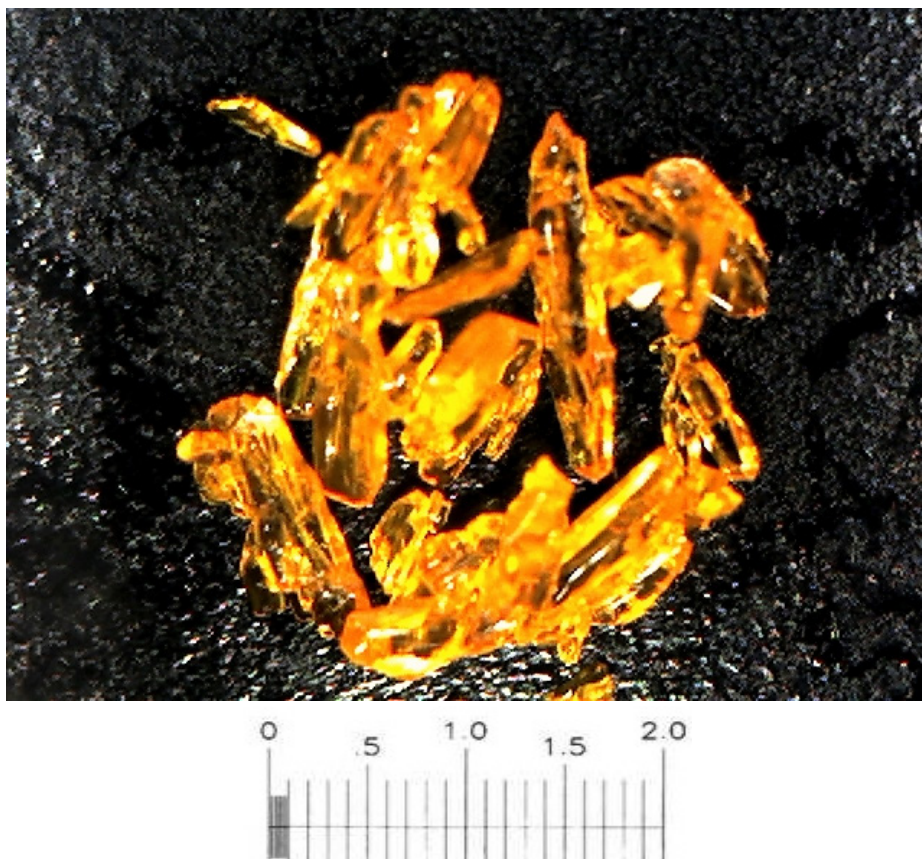


Figure 4. Microscope photo of APATO·HClO₄: amber yellow crystals – after 3x crystallization from H₂O (pH 1.0) (magnification ×20; full scale: 2 mm, one division: 0.1 mm)

2.4.2 X-ray structure determination of APATO·HClO₄

Selected main data from APATO·HClO₄ X-ray structural analysis are listed in Table 1 and ORTEP images are shown in Figures 5-7. Interestingly, in the APATO perchlorate structure, the picryl moiety is twisted with respect to the triazole ring plane (Figures 6-7). The crystal structure analysis revealed the presence of strong intramolecular (N6–H6···O14) and intermolecular hydrogen bonds in APATO·HClO₄ and these factors are mostly responsible for the high density of APATO·HClO₄ crystals.

Table 1. X-ray structure determination: selected data and parameters for APATO·HClO₄

Formula	C ₈ H ₆ N ₈ O ₆ ·HClO ₄ (C ₈ H ₇ ClN ₈ O ₁₀)
Form. weight [g/mol]	410.67
Crystal system	Orthorhombic
Space group	Pbc21 (Hall) / P2c-2b (H-M)
Colour/Shape	yellow block crystal from dil. HClO ₄
Size [mm]	0.41×0.29×0.26
<i>a</i> [Å]	8.6943(2)
<i>b</i> [Å]	9.3561(2)
<i>c</i> [Å]	17.9963(4)
α [°]	90
β [°]	90
γ [°]	90
<i>V</i> [Å ³]	1463.90(6)
<i>Z</i>	4
ρ [g/cm ⁻³]	1.863 (calc. from diff. an., at 173 K) 1.840 (pycnom., at 293 K)
μ [mm ⁻¹]	0.343
F(000)	832
λ _{MoKα} [Å]	0.71073
T [K]	173(2)
Limiting indices	-12:12, -13:13, -25:25
Reflections collected	3983
Independent reflections	2184
Absolute structure parameter	0.09(6)
R1(obs)	0.0174
wR2 (all data)	0.0859
GooF	1.023
Largest diff. peak and hole	0.427 and -0.373 e.Å ⁻³
Device type	Bruker-Nonius KappaCCD
Solution	SHELXS-97
Refinement	SHELXL-97
Absorbtion correction	none

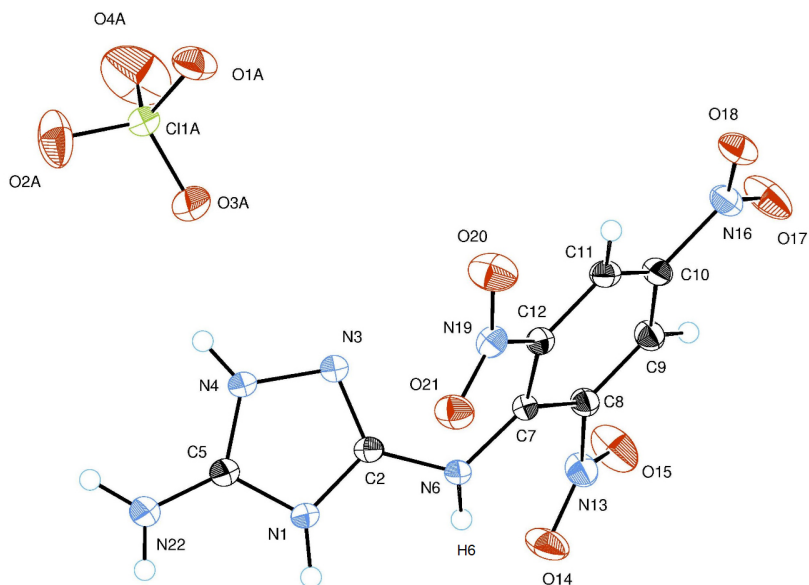


Figure 5. ORTEP image at the 50% probability of thermal ellipsoids for APATO·HClO₄; full data for this structure can be accessed on the Cambridge Crystallographic Data Centre: CCDC 1878763

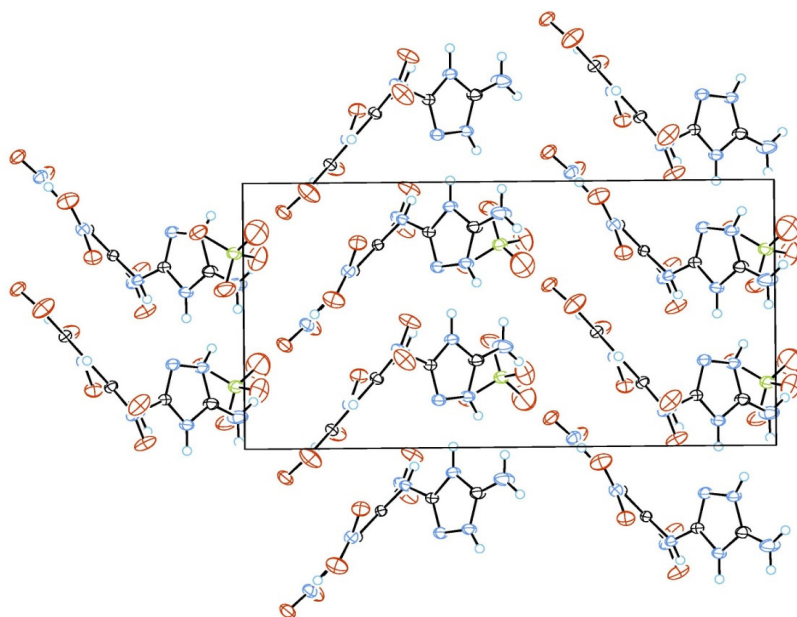


Figure 6. Crystal unit cell packing image for APATO·HClO₄ (Projection X)

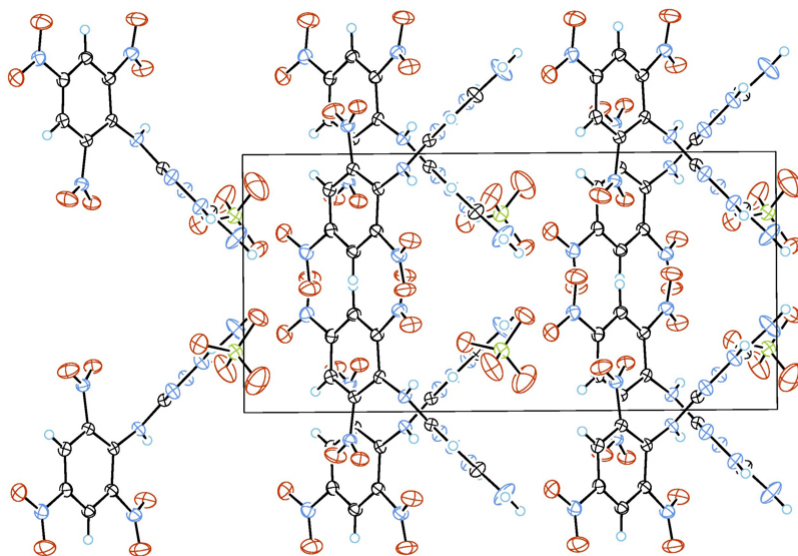


Figure 7. Crystal unit cell packing image for APATO·HClO₄ (Projection Y)

2.3 Calculated detonation performance of the synthesized APATO salts

The main calculated energetic properties and detonation performance of the newly synthesized salts, shown below, are listed in Table 5.

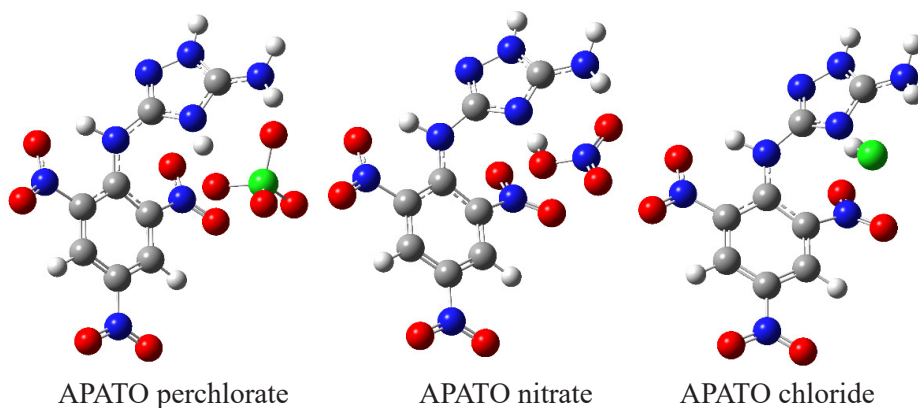


Table 5. Some calculated detonation properties of the synthesized 5-amino-3-[(2,4,6-trinitrophenyl)amino]-1*H*-1,2,4-triazol-4-ium salts

Compound	Molecular formula	ρ [g/cm ³]	Oxygen balance (OB_{CO_2}) [%]	Calc. velocity of detonation D [m/s]	Calc. detonation pressure P [kbar]
APATO	C ₈ H ₆ N ₈ O ₆	1.887 (calc.)	-67.06	6540	187.03
AATO chloride	C ₈ H ₇ ClN ₆ O ₆	1.587 (calc.)	-53.04	5790	145.16
APATO nitrate	C ₉ H ₈ N ₆ O ₇	1.650 (calc.)	-41.25	7090	222.09
APATO perchlorate	C ₁₀ H ₁₀ N ₆ O ₇	1.840 (exp.)	-37.01	7480	268.88
TNT (as standard)	C ₇ H ₅ N ₃ O ₆	1.630 (exp.)	-73.98	6900	210 (exp.)
Tetryl (as standard)	C ₇ H ₅ N ₅ O ₈	1.73 (exp.)	-47.40	7570	260, at 1.71 g/cm ³ (exp.)

The theoretical densities of the first three compounds were calculated using ACD Labs software (vers. 11.0) (based on the contributions of molecular fragments). The present data were calculated by applying the appropriate equations presented below [19]:

$$D = 1.9 + (-2.97a + 9.32b + 27.68c + 98.9d + \Delta H(g))\rho/M \quad (1)$$

$$P = -2.6 + (-1026a + 226b + 1031c + 3150d + 30.7\Delta H(g))\rho^2/M \quad (2)$$

where a , b , c , and d are the numbers of C, H, N and O, respectively, ρ is density, M is molar weight, $\Delta H(g)$ is gas phase heat of formation.

These data demonstrate that the calculated D and P of the new salts are almost equal or somewhat superior to the known standard TNT, however their characteristics are in general somewhat less when compared with the characteristics of another standard compound tetryl. Both the nitrate and perchlorate salts of APATO possess increased detonation characteristics when compared to the base (APATO), however the chloride salt exhibits somewhat decreased parameters.

3 Conclusions

- ◆ APATO (5-amino-3-picrylamino-1*H*-1,2,4-triazole), on direct reaction with dilute inorganic acids (HCl, HNO₃ and HClO₄) led to new cationic energetic salts. The nitrate and perchlorate salts were also obtained by another method: metathesis reactions.
- ◆ The synthesized new salts exhibit various densities (1.587-1.840 g/cm³). Their thermostability increased in order from nitrate to chloride and perchlorate (obtained from DSC-TG data). APATO perchlorate possess an excellent density ($\rho = 1.84 \text{ g/cm}^3$) and good thermostability (decomposition started at 230 °C). These parameters are comparable to the standard energetic material RDX.
- ◆ Both the nitrate and perchlorate salts of APATO possess better calculated detonation characteristics when compared to the base (APATO) and to the standard explosive, 2,4,6-trinitrotoluene (TNT), however the chloride salt showed somewhat decreased parameters.
- ◆ Hydrogen bonding in the packing of APATO·HClO₄ crystals is mostly responsible for the high crystal density.

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