

Synthesis and Properties of a New Explosive, 4- Amino-3,5-dinitro-1H- Pyrazole (LLM-116)

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Synthesis and Properties of a New Explosive, 4-Amino-3,5-dinitro-1*H*-pyrazole (LLM-116)

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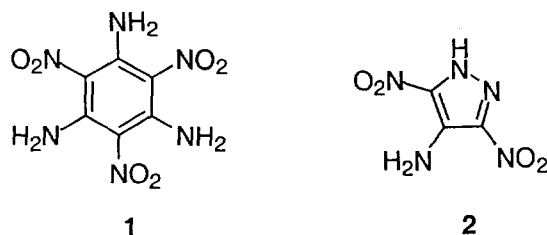
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Abstract: A novel synthesis of the title compound was achieved by direct amination using Vicarious Nucleophilic Substitution (VNS) methodology. Reaction of 1,1,1-trimethylhydrazinium iodide with 3,5-dinitropyrazole in DMSO produces 4-amino-3,5-dinitro-1*H*-pyrazole as a 1:1 crystal solvate with DMSO. Recrystallization from water yields the monohydrated crystal. Recrystallization of the monohydrate from butyl acetate yields the compound in pure form. Crystallographic data and results of small-scale safety tests are reported. These data indicate that LLM-116 is a promising candidate as an insensitive high explosive.

Introduction

The compound 1,3,5-triamino-2,4,6-trinitrobenzene (TATB, **1**) is an explosive material with a remarkably low detonation sensitivity to impact, friction and spark, and an explosive power comparable to that of 2,4,6-trinitrotoluene (TNT). The unique insensitivity of TATB has been attributed to the high degree of hydrogen bonding afforded by its alternating amino and nitro functional groups, along with the molecular symmetry, which allows the molecule to form a graphite-like crystal structure [1]. It has also been suggested that the thermal stability of TATB may be, at least in part, due to alternating ortho amino and nitro groups, which allow the formation of benzofurazan and benzofuroxan rings in the first step of decomposition [2].



By analogy, many ideas for new insensitive explosive compounds have been based upon the assumption that introduction of amino groups into nitroaromatic molecules might decrease the sensitivity of the product relative to the unaminated starting material [3]. It is also generally true that the explosive power of solid explosives is increased by increasing the density, oxygen balance and/or heat of formation of the material [4].

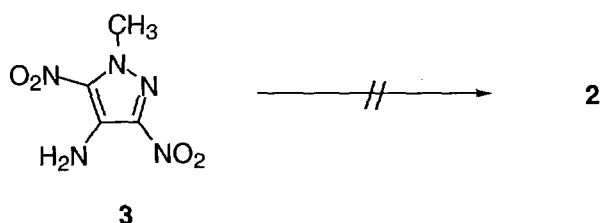
With this in mind, one goal of our group's effort has been to synthesize compounds that may be considered as high-energy analogues of TATB. One target molecule that features the alternating amino-nitro aromatic moiety is 4-amino-3,5-dinitro-1*H*-pyrazole (**2**, abbreviated as

"ADNP" and given the LLNL designation "LLM-116".) Relative to TATB, **2** has a better oxygen balance and, because of its smaller ring size and the presence of nitrogen atoms in the ring, should have a higher density and heat of formation. Until recently, no reports of the successful synthesis of **2** have been published, although the existence of **2** was suggested as early as 1993 [5]. However, Shevelev makes a footnote mention of an approach to the synthesis of **2** [6]. In this report, we report a novel approach to **2** using the technique of amination by Vicarious Nucleophilic Substitution of hydrogen (VNS) [7], as well as results of crystallographic, analytical and small-scale safety characterization of this material.

Discussion

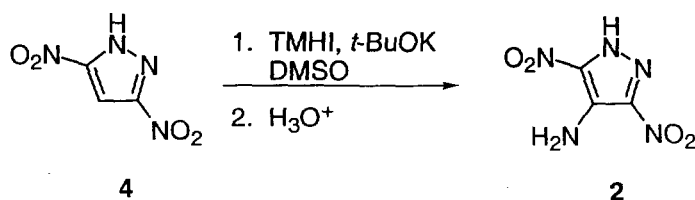
Our original efforts at preparing **2** were based on the attempted deprotection of N-protected analogues of **2**, including 4-amino-3,5-dinitro-1-methylpyrazole (**3**) [8] (Scheme 1). Oxidation followed by decarboxylation [9] was unsuccessful. Attempts to prepare the 1-*t*-butyl analogue of **3** for subsequent deprotection were also unsuccessful.

Scheme 1



At about this same time, we discovered a powerful new VNS reagent for the direct amination of nitrobenzenes, 1,1,1-trimethylhydrazinium iodide (TMHI) [7]. Although TMHI had never been tested on a nitroheterocyclic compound, we were encouraged by successes with other VNS aminating reagents on heterocycles [10]. The synthesis of TATB by amination of 1,3,5-trinitrobenzene with TMHI suggested a mechanism in which each nitro group was available to stabilize a negative charge formed by reaction with the TMHI nucleophile, allowing the formation of an intermediate, tri-anionic species. This mechanism led us to investigate the amination of 3,5-dinitropyrazole (**4**) [11], which carries an acidic hydrogen, to give 4-amino-3,5-dinitropyrazole (**2**). We reasoned that the acidic proton on **4** would initially react with one equivalent of base to form a stable nitronate anion leaving the second nitro-group available to participate in the VNS amination. This would allow the synthesis of **2** without the need of a protecting group for the pyrazole proton. We found the reaction of **4** with TMHI in the presence of excess potassium *tert*-butoxide gave **2** in 70% yield (Scheme 2). This reaction proceeds rapidly at room temperature, producing after acidic work-up a good yield of flat, golden crystals.

Scheme 2



X-ray crystallography [12] revealed that the product recovered from this reaction is actually a 1:1 solvate of **2** with DMSO, with a crystal density of 1.608 g/cc. (See Appendix A-2.) Recrystallization from acetonitrile yielded, surprisingly, the same 1:1 DMSO solvate, whereas recrystallization from water resulted in a 1:1 hydrate (Appendix A-3). After some experimentation, it was discovered that recrystallization of the monohydrate from butyl acetate/heptane yielded pure crystals of **2**, the structure of which was confirmed by x-ray crystallography (see Appendix A-1). The pure compound **2** is a yellow crystal formed of aggregates of plates, which melts at 175-8°C (uncorrected), and which has a density (X-ray) of 1.900 g/cc.

Synthesis and Characterization

Melting points were measured on a Mel-Temp instrument and are uncorrected. Unless otherwise noted, proton and ¹³C nmr spectra were recorded on a Bruker 300MHz nmr Spectrometer using DMSO-*d*₆ as the solvent with tetramethylsilane as the internal standard. The infrared spectra were obtained using a Nicolet 730 ftir spectrophotometer. Positive ion electron impact mass spectrum was recorded on a Finnigan GCQ-MS instrument, using a direct insertion probe (DIP) to introduce the sample. The starting material 3,5-dinitropyrazole was prepared according to the method of Habraken [11]. Trimethylhydrazinium iodide was prepared as previously reported [7]. Dimethyl sulfoxide (anhydrous, 99.8%) and solid potassium *t*-butoxide (95%) were obtained from Aldrich, and were used as received. Elemental analysis was performed by Midwest Microlab LLC, Indianapolis, Indiana.

Procedure: 3,5-Dinitropyrazole (**4**) (0.355 g, 2.25 mmol) and 1,1,1-trimethylhydrazinium iodide (0.504 g, 2.49 mmol) were dissolved in 12.0 mL DMSO. Solid potassium *t*-butoxide (0.741 g, 6.60 mmol) was then added in one portion with stirring. The clear yellow solution immediately turned to a dark crimson red color as the base dissolved, and the odor of trimethylamine was noted. The reaction mixture was stirred at room temperature for 4 hours, after which it was poured onto 12 g of ice and acidified to pH 3 with 10% HCl. The resulting solids were suction filtered, washed with cold water and air dried to yield 0.382 g of a yellow powder (DMSO monosolvate of **2**). The product was soluble in DMSO, hot water, methanol, ethanol and hot acetonitrile. ¹H nmr: δ 7.13 (broad s, 2H, NH₂), 2.51 (s, 6H, DMSO CH₃). ¹³C nmr: δ 137.8 (s), 128.8 (s), 39.5 (septet). ir (KBr): 3446(s), 3255(m), 3200(m), 3145(w), 3009(w), 1641(s), 1510(s), 1486(s), 1463(s), 1327(s), 1309(s), 1141(w), 1018(m), 936(m).

Recrystallization of crude product from acetonitrile produced small, yellow needles, which were washed with diethyl ether and air-dried (mp 160-162°C, with decomposition). NMR and IR spectra were identical to those of the starting crude product. Crystallographic analysis confirmed the structure **2**-DMSO (monosolvate).

When the crude **2**-DMSO (0.10g) was placed in water (2 mL), heated until dissolved and cooled overnight, small, yellow iridescent plates formed (mp 169-171°C). ¹H nmr (90MHz): δ 7.15 (very broad s, NH₂). ir (KBr): 3437(s), 3327(s), 3172(s, br), 1641(s), 1577(m), 1513(s), 1477(s), 1438(m), 1323(s), 1236(w), 1214(w), 1095(w), 945(m). Crystallographic analysis confirmed the structure **2**-H₂O (monosolvate).

When 2-H₂O was recrystallized from hot butyl acetate/heptane, pure 2 recrystallized as yellow plates (mp 175-178°C). ¹H nmr: δ 7.13 (broad s, 2H, NH₂). ¹³C nmr (500MHz): δ 137.7 (s), 128.6 (s). ¹⁵N nmr (500MHz, CrAcAc added): δ -24.4 (s) (NO₂), -317.4 (s) (NH₂). FT-IR (KBr): 3437(m), 3325(m), 1641(s), 1581(w), 1510(s), 1476(s), 1440(w), 1328(s), 1237(w), 1208(w), 847(m), 831(w). MS (amu): 173 (M+), 143 (M-NO), 112, 80, 54. Differential Scanning Calorimetry (10°C/min): endotherm @ 175.70-176.97°C (66.51 J/g), exotherm (peak) @ 183.63°C (996.1 J/g). Elemental analysis calculated for C₃H₃N₅O₄: C, 20.81; H, 1.75; N, 40.46. Found: C, 20.91; H, 1.84; N, 39.94.

DSC trace and Mass Spectrogram are reproduced in Appendices A and D, respectively.

Small Scale Safety Test Data

1. Impact Sensitivity: Type 12 drop hammer tests were conducted on 35mg piles of material. Ambient temperature was 70°F, and relative humidity was 61%. DH₅₀ was calculated from 15 impact runs, and standard deviation was ±0.002. Result:
DH₅₀ = 167.5 cm
2. Electrostatic Sensitivity: Electrostatic spark test was conducted under the following conditions: 20KpF capacitance, 10.0 kV, 510 ohm resistance, 0.007 in. gap, 1.0 Joule spark energy, 68°F, 67% relative humidity. Results: **0 of 10** showed reaction to spark. (Not spark sensitive.)
3. Friction Sensitivity: The Friction B.A.M test was conducted under the following conditions: 28.8 kg applied mass, 70°F ambient temperature, 67% relative humidity. Results: **1 of 10** showed sensitivity to friction.
4. Chemical Reactivity Test (CRT): CRT's were performed on 0.25g samples at 80 & 120°C. Pure ADNP produced **0.002cc/0.25g** gas on both of 2 runs. ADNP monohydrate produced 0.001-0.002 cc/0.25g. At 120°C, pure ADNP decomposed rapidly during this test.

Complete data sheets for all of the above tests are included in the appendices.

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APPENDIX A: Crystallographic Data

A-1. Pure ADNP (LLM-116)

All atoms in **2** are essentially coplanar, with an average deviation from the plane of only 0.03Å.

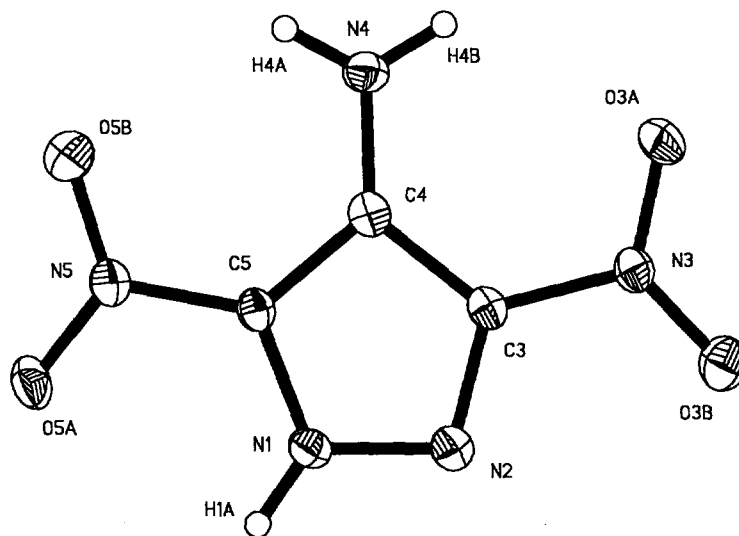


Figure A-1a. ORTEP drawing of neat **2** from X-ray analysis.

This facilitates a dense packing scheme, shown in Figure A-1b, which resembles that of TATB insofar as there are two strong hydrogen bonds within each molecule and six strong intermolecular hydrogen bonds about the perimeter of each molecule. However, it also differs. TATB forms endless, essentially flat two-dimensional sheets in its crystals [13] through amino-nitro hydrogen bonding. These sheets then stack on top of, and parallel to, one another, leading to an assembly of all-parallel molecules resembling graphite. The crystal assembly in **2** contains sub-elements of parallel-molecule stacking (see Fig. 2), described here as stacked-ribbons, but the ribbons in adjacent stacks are decidedly not parallel, and a view down the *c* axis of the crystal through two or more stacks would show an almost orthogonal grid of criss-crossed “ribbons”. This difference in the supramolecular assembly patterns would be expected to lead to differences in the mechanical properties of the two materials.

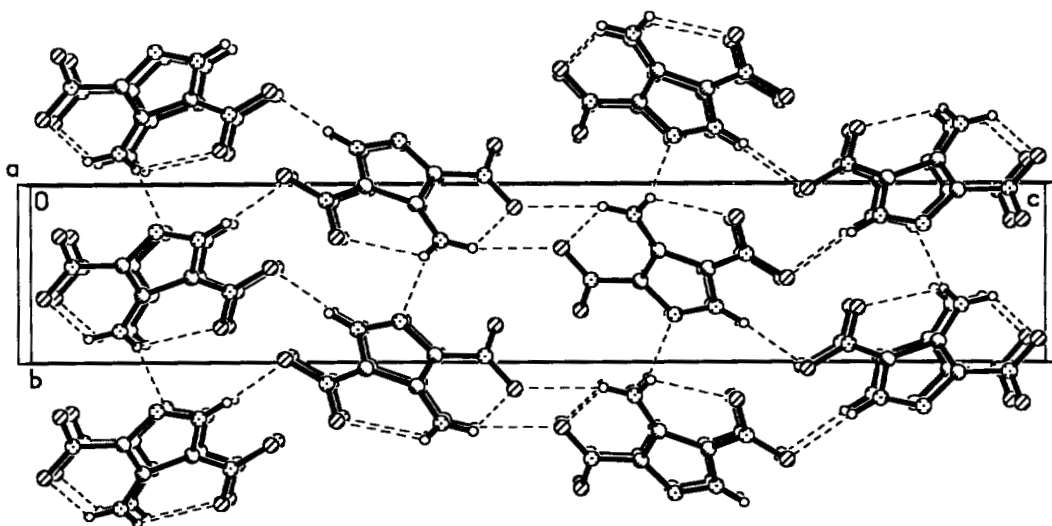


Figure 2. A view down unit cell axis **a**, with **b** (vertical) and **c** (horizontal) in the plane of the paper. The molecules in vertical columns, along **b**, are parallel, and resemble, from the side, stacked ribbons. Molecules in adjacent columns differ in their inclination out of the plane of the paper, and so are not parallel, but all molecules are hydrogen bonded across each column junction. The strong hydrogen bonds listed in Table 2c, and their symmetry equivalents, are shown here as dashed lines.

Table A-1a. Crystal data and structure refinement for LLM-116 (2).

Identification code	ADNP	
Empirical formula	C ₃ H ₃ N ₅ O ₄	
Formula weight	173.10	
Temperature	294(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 4.7257(5) Å	α = 90°.
	b = 4.7312(6) Å	β = 90°.
	c = 27.063(4) Å	γ = 90°.
Volume	605.07(13) Å ³	
Z	4	
Density (calculated)	1.900 Mg/m ³ at 21C	
Absorption coefficient	1.555 mm ⁻¹	
F(000)	352	
Crystal size	.03 x .28 x .25 mm ³	
Theta range for data collection	3.27 to 57.97°.	
Index ranges	-5 ≤ h ≤ 5, 0 ≤ k ≤ 5, 0 ≤ l ≤ 29	
Reflections collected	1036	
Reflections 'observed'	835 [I > 2σ(I)]	
Independent reflections	851 [R(int) = 0.0174]	
Completeness to theta = 57.97°	100.0 %	
Absorption correction	Integration	
Max. and min. transmission	0.9474 and 0.6735	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	851 / 0 / 111	
Goodness-of-fit on F ²	1.100	
Final R indices [I > 2σ(I)]	R1 = 0.0329, wR2 = 0.0868	
R indices (all data)	R1 = 0.0338, wR2 = 0.0880	
Absolute structure parameter	-0.1(7)	
Extinction coefficient	0.013(2)	
Largest diff. peak and hole	0.157 and -0.170 e.Å ⁻³	

Table A-1b. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for LLM-116. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	$U(\text{eq})$
N(1)	7192(6)	3103(6)	1720(1)	32(1)
N(2)	8789(6)	2668(6)	1325(1)	34(1)
C(3)	7808(7)	4520(7)	988(1)	29(1)
N(3)	9087(6)	4594(6)	512(1)	31(1)
O(3A)	8170(6)	6375(5)	226(1)	41(1)
O(3B)	10967(6)	2912(6)	413(1)	51(1)
C(4)	5548(7)	6209(7)	1156(1)	29(1)
N(4)	4090(6)	8223(6)	924(1)	36(1)
C(5)	5230(7)	5211(7)	1636(1)	29(1)
N(5)	3273(6)	6021(6)	1996(1)	34(1)
O(5A)	3188(6)	4689(5)	2388(1)	44(1)
O(5B)	1734(6)	8037(6)	1896(1)	49(1)

Table A-1c. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$).

	x	y	z	$U(\text{eq})$
H(1A)	7356	2191	1993	39
H(4A)	2745	9092	1074	43
H(4B)	4494	8652	623	43

Table A-1d. Hydrogen bonding parameters^[a]

Donor	Acceptor	Symmetry op. (A)	$[\text{D}-\text{H}\cdots\text{A}]^\circ$	$[\text{H}\cdots\text{A}] \text{\AA}$	$[\text{D}\cdots\text{A}] \text{\AA}$
N4-H4a	O5b	intramolecular	116.9	2.258	2.860
N4-H4b	O3a	intramolecular	116.5	2.238	2.836
N1-H1a	O5a	(1 - x, y - $\frac{1}{2}$, - z)	165.7	1.922	2.910
N4-H4a	N2	(x - 1, y + 1, z)	163.3	2.468	3.446
N4-H4b	O3a	(x - $\frac{1}{2}$, 1 - y, - z)	146.6	2.256	3.148

[a] Only the shortest H-bonds are tabulated here, those for which $\text{H}\cdots\text{A}$ is at least 0.2\AA less than the sums of the vdW radii [14], which are 2.72\AA for $\text{H}\cdots\text{O}$, 2.75\AA for $\text{H}\cdots\text{N}$. For the calculations in this Table, the experimental N-H distances were first normalized to match the average neutron diffraction distance, 1.009\AA , used in Ref. 14 to derive the van der Waals radii from the CCDC X-ray database.

Table A-1e. Bond lengths [Å] and angles [°] for LLM-116.

N(1)-N(2)	1.325(4)
N(1)-C(5)	1.380(5)
N(2)-C(3)	1.346(4)
C(3)-C(4)	1.409(5)
C(3)-N(3)	1.424(4)
N(3)-O(3B)	1.223(4)
N(3)-O(3A)	1.223(4)
C(4)-N(4)	1.334(5)
C(4)-C(5)	1.390(4)
C(5)-N(5)	1.398(4)
N(5)-O(5B)	1.229(4)
N(5)-O(5A)	1.234(4)
N(2)-N(1)-C(5)	111.2(3)
N(1)-N(2)-C(3)	104.4(3)
N(2)-C(3)-C(4)	114.4(3)
N(2)-C(3)-N(3)	118.8(3)
C(4)-C(3)-N(3)	126.8(3)
O(3B)-N(3)-O(3A)	124.6(3)
O(3B)-N(3)-C(3)	119.4(3)
O(3A)-N(3)-C(3)	116.0(3)
N(4)-C(4)-C(5)	128.9(3)
N(4)-C(4)-C(3)	130.2(3)
C(5)-C(4)-C(3)	101.0(3)
N(1)-C(5)-C(4)	109.1(3)
N(1)-C(5)-N(5)	121.8(3)
C(4)-C(5)-N(5)	129.1(3)
O(5B)-N(5)-O(5A)	124.5(3)
O(5B)-N(5)-C(5)	116.8(3)
O(5A)-N(5)-C(5)	118.8(3)

Table A-1f. Torsion angles [°] for LLM-116.

C(5)-N(1)-N(2)-C(3)	-0.2(3)
N(1)-N(2)-C(3)-C(4)	0.0(4)
N(1)-N(2)-C(3)-N(3)	-179.8(3)
N(2)-C(3)-N(3)-O(3B)	2.7(5)
C(4)-C(3)-N(3)-O(3B)	-177.0(3)
N(2)-C(3)-N(3)-O(3A)	-177.9(3)
C(4)-C(3)-N(3)-O(3A)	2.4(5)
N(2)-C(3)-C(4)-N(4)	-179.9(3)
N(3)-C(3)-C(4)-N(4)	-0.1(6)
N(2)-C(3)-C(4)-C(5)	0.2(4)
N(3)-C(3)-C(4)-C(5)	180.0(3)
N(2)-N(1)-C(5)-C(4)	0.4(4)
N(2)-N(1)-C(5)-N(5)	179.0(3)
N(4)-C(4)-C(5)-N(1)	179.8(3)
C(3)-C(4)-C(5)-N(1)	-0.3(3)
N(4)-C(4)-C(5)-N(5)	1.3(6)
C(3)-C(4)-C(5)-N(5)	-178.8(3)
N(1)-C(5)-N(5)-O(5B)	175.6(3)
C(4)-C(5)-N(5)-O(5B)	-6.1(5)
N(1)-C(5)-N(5)-O(5A)	-4.6(5)
C(4)-C(5)-N(5)-O(5A)	173.7(3)

Table A-1g. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for LLM-116. The anisotropic displacement factor exponent takes the form:

$$-2\pi^2 [h^2 a^2 U^{11} + \dots + 2 h k a^* b^* U^{12}]$$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
N(1)	36(2)	33(2)	28(1)	5(1)	-2(1)	1(1)
N(2)	36(1)	35(2)	31(1)	0(1)	0(1)	-1(1)
C(3)	32(2)	27(2)	27(2)	-1(1)	1(1)	-5(1)
N(3)	36(1)	32(2)	25(1)	-3(1)	3(1)	-3(1)
O(3A)	47(2)	47(2)	29(1)	10(1)	1(1)	1(1)
O(3B)	55(2)	51(2)	45(1)	-4(1)	14(1)	12(2)
C(4)	29(2)	29(2)	29(1)	-4(1)	-3(1)	-7(1)
N(4)	40(2)	34(2)	34(1)	3(1)	-3(1)	4(1)
C(5)	32(2)	32(2)	24(2)	-4(1)	-1(1)	-2(1)
N(5)	34(1)	36(2)	31(2)	-4(1)	2(1)	-4(1)
O(5A)	60(2)	44(2)	26(1)	2(1)	9(1)	-3(1)
O(5B)	50(2)	49(2)	47(1)	0(1)	5(2)	11(2)

A-2. ADNP-DMSO Complex

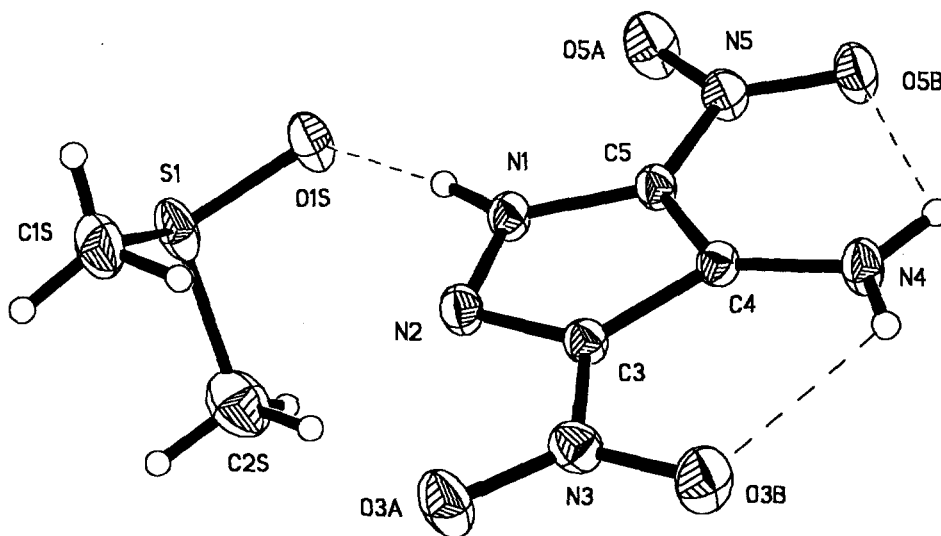


Figure A-2a. A thermal ellipsoid drawing of the ADNP, DMSO (1:1) crystal complex.

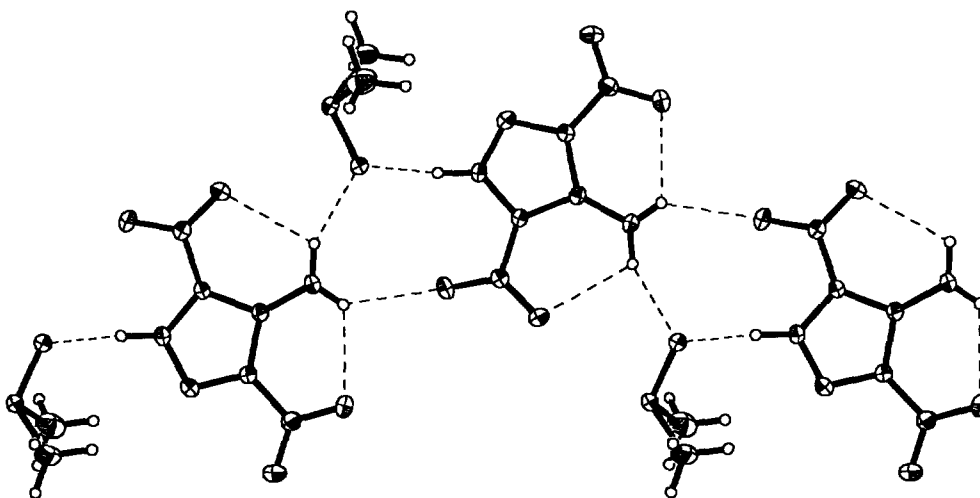


Figure A-2b. A view of the hydrogen bonding in ADNP:DMSO, which links the two moieties into ribbons that are essentially planar, but for the projecting methyl groups.

Table A-2a. Crystal data and structure refinement for ADNP*DMSO.

Identification code	liv08a	
Empirical formula	C5 H9 N5 O5 S	
Formula weight	251.23	
Temperature	295(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 10.8977(8) Å	= 90°.
	b = 14.1398(11) Å	= 96.453(6)°.
	c = 6.7767(6) Å	= 90°.
Volume	1037.61(14) Å ³	
Z	4	
Density (calculated)	1.608 Mg/m ³	
Absorption coefficient	0.330 mm ⁻¹	
F(000)	520	
Crystal size	.08 x .20 x .48 mm ³	
Theta range for data collection	2.37 to 25.00°.	
Index ranges	-12<=h<=12, -16<=k<=1, 0<=l<=8	
Reflections collected	2235	
Reflections 'observed'	1300 [I>2sigma(I)]	
Independent reflections	1822 [R(int) = 0.0151]	
Completeness to theta = 25.00°	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	1822 / 0 / 182	
Goodness-of-fit on F ²	1.028	
Final R indices [I>2sigma(I)]	R1 = 0.0423, wR2 = 0.0926	
R indices (all data)	R1 = 0.0695, wR2 = 0.1053	
Extinction coefficient	0.0061(12)	
Largest diff. peak and hole	0.255 and -0.245 e.Å ⁻³	

Table A-2b. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ADNP*DMSO. $U(\text{eq})$ is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	$U(\text{eq})$
S(1)	8009(1)	3533(1)	3924(2)	55(1)
O(1S)	6739(2)	3987(1)	3727(4)	53(1)
C(1S)	8843(4)	4079(3)	6017(7)	65(1)
C(2S)	8777(4)	4051(4)	2028(7)	76(1)
N(1)	6584(2)	5868(2)	3742(4)	38(1)
N(2)	7620(2)	6297(2)	4425(4)	38(1)
C(3)	7360(2)	7224(2)	4250(4)	32(1)
N(3)	8282(2)	7898(2)	4872(4)	40(1)
O(3A)	9295(2)	7634(2)	5653(3)	56(1)
O(3B)	8002(2)	8737(1)	4596(4)	55(1)
C(4)	6130(2)	7411(2)	3426(4)	30(1)
N(4)	5567(2)	8243(2)	3074(4)	40(1)
C(5)	5684(2)	6496(2)	3126(4)	32(1)
N(5)	4502(2)	6188(2)	2321(4)	42(1)
O(5A)	4307(2)	5334(2)	2166(4)	64(1)
O(5B)	3731(2)	6798(1)	1803(3)	52(1)
	x	y	z	$U(\text{iso})$
H(1S3)	8740(30)	4770(30)	5850(60)	85(13)
H(1S2)	9650(40)	3870(30)	6090(60)	83(13)
H(1S1)	8460(50)	3890(30)	7000(70)	104(19)
H(2S3)	9620(40)	3850(30)	2220(60)	106(16)
H(2S2)	8670(40)	4740(30)	2050(60)	89(14)
H(2S1)	8380(50)	3860(40)	790(80)	123(19)
H(1)	6580(30)	5220(20)	3730(40)	52(9)
H(4A)	4770(30)	8270(20)	2530(50)	63(10)
H(4B)	5980(30)	8730(20)	3310(50)	53(10)

Table A-2c. Bond lengths [Å] and angles [°] for ADNP*DMSO.

S(1)-O(1S)	1.519(2)	N(2)-C(3)	1.344(3)
S(1)-C(2S)	1.769(5)	C(3)-N(3)	1.414(3)
S(1)-C(1S)	1.773(4)	C(3)-C(4)	1.417(3)
C(1S)-H(1S3)	0.99(4)	N(3)-O(3A)	1.227(3)
C(1S)-H(1S2)	0.92(4)	N(3)-O(3B)	1.235(3)
C(1S)-H(1S1)	0.87(5)	C(4)-N(4)	1.335(3)
C(2S)-H(2S3)	0.96(5)	C(4)-C(5)	1.390(4)
C(2S)-H(2S2)	0.98(4)	N(4)-H(4A)	0.91(3)
C(2S)-H(2S1)	0.94(5)	N(4)-H(4B)	0.83(3)
N(1)-N(2)	1.319(3)	C(5)-N(5)	1.409(3)
N(1)-C(5)	1.354(3)	N(5)-O(5B)	1.227(3)
N(1)-H(1)	0.92(3)	N(5)-O(5A)	1.229(3)
O(1S)-S(1)-C(2S)	105.20(19)	N(2)-C(3)-N(3)	119.6(2)
O(1S)-S(1)-C(1S)	105.45(18)	N(2)-C(3)-C(4)	113.4(2)
C(2S)-S(1)-C(1S)	99.1(2)	N(3)-C(3)-C(4)	126.9(2)
S(1)-C(1S)-H(1S3)	107(2)	O(3A)-N(3)-O(3B)	123.6(2)
S(1)-C(1S)-H(1S2)	108(2)	O(3A)-N(3)-C(3)	119.9(2)
H(1S3)-C(1S)-H(1S2)	114(3)	O(3B)-N(3)-C(3)	116.5(2)
S(1)-C(1S)-H(1S1)	103(3)	N(4)-C(4)-C(5)	130.4(2)
H(1S3)-C(1S)-H(1S1)	110(4)	N(4)-C(4)-C(3)	129.0(2)
H(1S2)-C(1S)-H(1S1)	114(4)	C(5)-C(4)-C(3)	100.6(2)
S(1)-C(2S)-H(2S3)	108(3)	C(4)-N(4)-H(4A)	121(2)
S(1)-C(2S)-H(2S2)	109(2)	C(4)-N(4)-H(4B)	118(2)
H(2S3)-C(2S)-H(2S2)	114(4)	H(4A)-N(4)-H(4B)	121(3)
S(1)-C(2S)-H(2S1)	109(3)	N(1)-C(5)-C(4)	109.6(2)
H(2S3)-C(2S)-H(2S1)	112(4)	N(1)-C(5)-N(5)	121.0(2)
H(2S2)-C(2S)-H(2S1)	105(4)	C(4)-C(5)-N(5)	129.3(2)
N(2)-N(1)-C(5)	111.6(2)	O(5B)-N(5)-O(5A)	124.0(2)
N(2)-N(1)-H(1)	117.9(19)	O(5B)-N(5)-C(5)	117.4(2)
C(5)-N(1)-H(1)	130.4(19)	O(5A)-N(5)-C(5)	118.6(2)
N(1)-N(2)-C(3)	104.7(2)		

Table A-2d. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ADNP*DMSO. The anisotropic displacement factor exponent takes the form: $-2_{-2}^2 [h^2 a^* U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³ U ¹²	
S(1)	34(1)	28(1)	98(1)	-3(1)	-10(1)	4(1)
O(1S)	32(1)	31(1)	91(2)	-2(1)	-8(1)	1(1)
C(1S)	44(2)	73(3)	72(3)	11(2)	-15(2)	1(2)
C(2S)	65(3)	93(4)	71(3)	-17(3)	7(2)	15(2)
N(1)	35(1)	24(1)	53(2)	3(1)	3(1)	2(1)
N(2)	34(1)	33(1)	47(2)	2(1)	1(1)	5(1)
C(3)	31(1)	30(1)	35(2)	-1(1)	3(1)	0(1)
N(3)	35(1)	39(1)	45(2)	-4(1)	3(1)	-3(1)
O(3A)	34(1)	56(1)	75(2)	-5(1)	-12(1)	1(1)
O(3B)	52(1)	31(1)	80(2)	-1(1)	-4(1)	-5(1)
C(4)	33(1)	27(1)	30(2)	-1(1)	4(1)	2(1)
N(4)	36(1)	25(1)	56(2)	1(1)	-3(1)	3(1)
C(5)	30(1)	26(1)	40(2)	1(1)	2(1)	2(1)
N(5)	35(1)	31(1)	58(2)	-1(1)	1(1)	-2(1)
O(5A)	49(1)	29(1)	111(2)	-8(1)	-5(1)	-7(1)
O(5B)	35(1)	40(1)	78(2)	6(1)	-8(1)	5(1)

Table A-2e. Torsion angles [$^\circ$] for ADNP*DMSO.

C(5)-N(1)-N(2)-C(3)	0.6(3)
N(1)-N(2)-C(3)-N(3)	179.5(2)
N(1)-N(2)-C(3)-C(4)	-0.3(3)
N(2)-C(3)-N(3)-O(3A)	-3.5(4)
C(4)-C(3)-N(3)-O(3A)	176.4(3)
N(2)-C(3)-N(3)-O(3B)	177.1(3)
C(4)-C(3)-N(3)-O(3B)	-3.0(4)
N(2)-C(3)-C(4)-N(4)	179.1(3)
N(3)-C(3)-C(4)-N(4)	-0.8(5)
N(2)-C(3)-C(4)-C(5)	0.0(3)
N(3)-C(3)-C(4)-C(5)	-179.9(3)
N(2)-N(1)-C(5)-C(4)	-0.7(3)
N(2)-N(1)-C(5)-N(5)	179.3(2)
N(4)-C(4)-C(5)-N(1)	-178.7(3)
C(3)-C(4)-C(5)-N(1)	0.4(3)
N(4)-C(4)-C(5)-N(5)	1.4(5)
C(3)-C(4)-C(5)-N(5)	-179.6(3)
N(1)-C(5)-N(5)-O(5B)	179.6(3)
C(4)-C(5)-N(5)-O(5B)	-0.4(5)
N(1)-C(5)-N(5)-O(5A)	-0.8(4)
C(4)-C(5)-N(5)-O(5A)	179.1(3)

Table A-2f. Hydrogen bonds for ADNP*DMSO [\AA and $^\circ$].

D-H...A	$d(D-H)$	$d(H...A)$	$d(D...A)$	$\angle(DHA)$
N(1)-H(1)...O(1S)	0.92(3)	1.75(3)	2.665(3)	174(3)
N(4)-H(4B)...O(3B)	0.83(3)	2.28(3)	2.824(3)	123(3)
N(4)-H(4A)...O(5B)	0.91(3)	2.39(3)	2.921(3)	117(3)
N(4)-H(4A)...O(1S)#1	0.91(3)	2.03(3)	2.868(3)	152(3)
N(4)-H(4B)...O(5A)#1	0.83(3)	2.30(3)	2.966(3)	137(3)

Symmetry transformations used to generate equivalent atoms:

#1 $-x+1, y+1/2, -z+1/2$

A-3. ADNP-H₂O Complex.

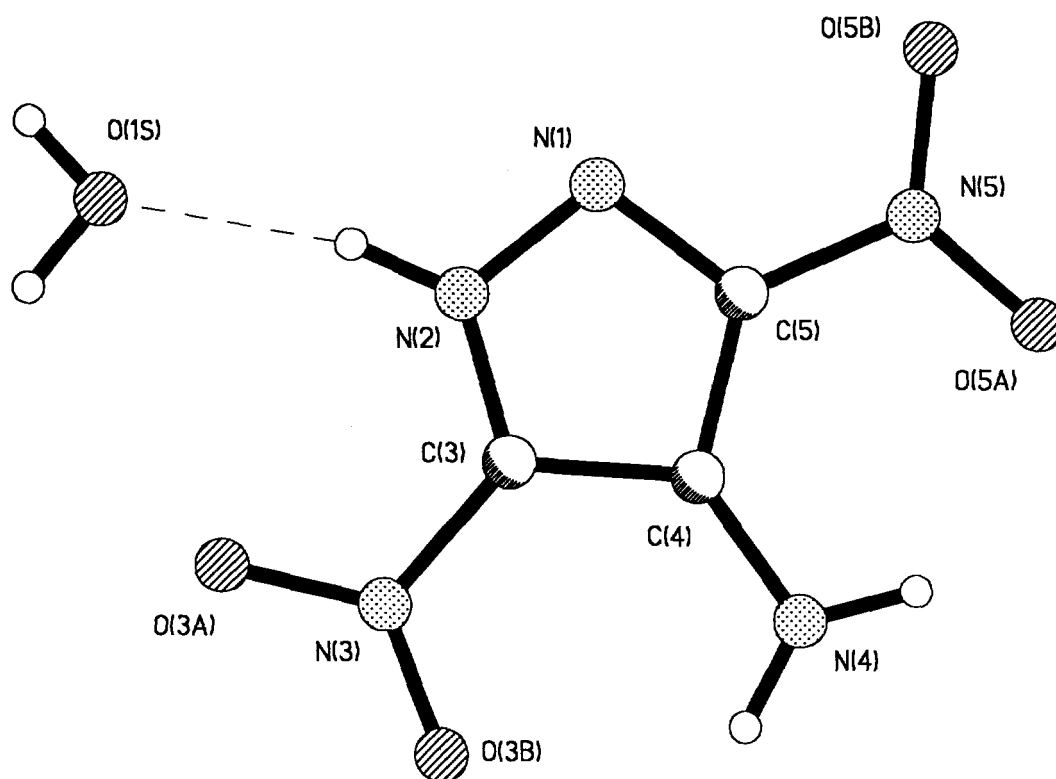


Figure A-3a. A drawing of the crystal structure results found for the ADNP hydrate. The results from this analysis are not of high accuracy, because the hydrate was deteriorating during data collection, but they are sufficient to identify the non-hydrogen atom locations clearly, and to define the unit cell dimensions and crystal density accurately.

Table A-3a. Crystal data and structure refinement for ADNP*H₂O.

Identification code	liv11	
Empirical formula	C ₃ H ₅ N ₅ O ₅	
Formula weight	191.12	
Temperature	295(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 4.658(4) Å	α = 90°.
	b = 5.010(4) Å	β = 90°.
	c = 30.54(2) Å	γ = 90°.
Volume	712.8(9) Å ³	
Z	4	
Density (calculated)	1.781 Mg/m ³	
Absorption coefficient	0.167 mm ⁻¹	
F(000)	392	
Crystal size	.02 x .40 x .80 mm ³	
Theta range for data collection	2.67 to 22.50°.	
Index ranges	0 ≤ h ≤ 5, -5 ≤ k ≤ 5, 0 ≤ l ≤ 32	
Reflections collected	1104	
Reflections 'observed'	673 [I > 2σ(I)]	
Independent reflections	924 [R(int) = 0.0500]	
Completeness to theta = 22.50°	99.7 %	
Absorption correction	Integration	
Max. and min. transmission	0.9934 and 0.9404	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	924 / 29 / 119	
Goodness-of-fit on F ²	1.509	
Final R indices [I > 2σ(I)]	R1 = 0.1465, wR2 = 0.3475	
R indices (all data)	R1 = 0.1895, wR2 = 0.3867	
Absolute structure parameter	-6(10)	
Extinction coefficient	0.04(3)	
Largest diff. peak and hole	0.487 and -0.553 e.Å ⁻³	

Note: The results from this analysis are not of high accuracy, because the hydrate was deteriorating (losing water) during data collection, but they are sufficient to identify the non-hydrogen atom locations clearly, and to define the unit cell dimensions and crystal density accurately.

Table A-3b. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ADNP*H₂O. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	-20(20)	2120(20)	1348(3)	35(3)
O(3B)	-5070(30)	8990(20)	837(4)	57(3)
O(5B)	-350(20)	10(20)	2150(3)	56(3)
O(5A)	-3970(30)	2630(20)	2307(3)	51(3)
C(5)	-1940(30)	3030(30)	1642(4)	33(3)
C(4)	-3520(30)	5280(30)	1496(4)	29(3)
N(5)	-2060(30)	1830(20)	2059(4)	33(3)
O(3A)	-1820(30)	7300(20)	398(3)	60(3)
N(4)	-5560(30)	6680(20)	1690(4)	45(3)
N(2)	-410(30)	3680(20)	1009(4)	38(3)
C(3)	-2430(30)	5530(30)	1077(4)	32(3)
N(3)	-3190(30)	7380(20)	752(4)	38(3)
O(1S)	2640(30)	3450(20)	225(3)	61(4)

Table A-3c. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ADNP*H₂O.

	x	y	z	U(iso)
H(4A)	-6405	7945	1551	54
H(4B)	-6038	6317	1955	54
H(2A)	533	3523	768	46
H(1SB)	2771	5105	170	92
H(1SA)	4110	2876	89	92

Table A-3d. Bond lengths [Å] and angles [°] for ADNP*H₂O.

N(1)-N(2)	1.309(15)
N(1)-C(5)	1.344(17)
O(3B)-N(3)	1.220(16)
O(5B)-N(5)	1.240(15)
O(5A)-N(5)	1.235(15)
C(5)-N(5)	1.410(16)
C(5)-C(4)	1.42(2)
C(4)-N(4)	1.321(17)
C(4)-C(3)	1.381(18)
O(3A)-N(3)	1.256(15)
N(4)-H(4A)	0.8600
N(4)-H(4B)	0.8600
N(2)-C(3)	1.339(18)
N(2)-H(2A)	0.8600
C(3)-N(3)	1.403(17)
O(1S)-H(1SB)	0.8500
O(1S)-H(1SA)	0.8501

N(2)-N(1)-C(5)	103.6(11)
N(1)-C(5)-N(5)	119.1(12)
N(1)-C(5)-C(4)	113.8(12)
N(5)-C(5)-C(4)	126.9(12)
N(4)-C(4)-C(3)	129.1(13)
N(4)-C(4)-C(5)	130.7(12)
C(3)-C(4)-C(5)	100.0(12)
O(5A)-N(5)-O(5B)	124.3(12)
O(5A)-N(5)-C(5)	116.4(11)
O(5B)-N(5)-C(5)	119.2(12)
C(4)-N(4)-H(4A)	120.0
C(4)-N(4)-H(4B)	120.0
H(4A)-N(4)-H(4B)	120.0
N(1)-N(2)-C(3)	112.9(12)
N(1)-N(2)-H(2A)	123.6
C(3)-N(2)-H(2A)	123.6
N(2)-C(3)-C(4)	109.7(12)
N(2)-C(3)-N(3)	121.8(13)
C(4)-C(3)-N(3)	128.5(13)
O(3B)-N(3)-O(3A)	124.7(12)
O(3B)-N(3)-C(3)	117.9(12)
O(3A)-N(3)-C(3)	117.4(12)
H(1SB)-O(1S)-H(1SA)	100.0

Table A-3e. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for ADNP*H₂O. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + \dots + 2 h k a^* b^* U^{12}]$

	U ¹¹	U ²²	U ³³	U ²³	U ¹³	U ¹²
N(1)	37(6)	24(6)	43(5)	0(4)	-3(4)	-1(4)
O(3B)	57(8)	27(6)	86(8)	1(5)	-10(5)	11(4)
O(5B)	55(8)	33(6)	80(7)	16(5)	-8(6)	4(5)
O(5A)	65(7)	32(6)	55(5)	0(5)	13(4)	-2(5)
C(5)	41(8)	13(5)	44(5)	-4(4)	-5(4)	-5(5)
C(4)	25(7)	19(6)	41(5)	-8(4)	-13(4)	-7(4)
N(5)	40(7)	12(5)	48(5)	0(4)	-4(4)	-12(4)
O(3A)	80(8)	44(6)	55(5)	18(5)	7(5)	6(7)
N(4)	56(8)	21(6)	57(7)	-6(5)	4(6)	7(5)
N(2)	40(7)	24(6)	51(5)	4(4)	5(5)	2(4)
C(3)	17(7)	30(7)	51(5)	6(5)	-6(5)	-9(5)
N(3)	47(7)	17(6)	51(5)	-3(4)	-6(5)	-1(5)
O(1S)	73(9)	31(6)	79(8)	-3(5)	27(6)	1(7)

Table A-3f. Torsion angles [$^\circ$] for ADNP*H₂O.

N(2)-N(1)-C(5)-N(5)-178	.4(11)
N(2)-N(1)-C(5)-C(4)	-2.4(15)
N(1)-C(5)-C(4)-N(4)	179.4(13)
N(5)-C(5)-C(4)-N(4)	-5(2)
N(1)-C(5)-C(4)-C(3)	2.8(14)
N(5)-C(5)-C(4)-C(3)	178.4(13)
N(1)-C(5)-N(5)-O(5A)	-176.0(12)
C(4)-C(5)-N(5)-O(5A)	8.6(18)
N(1)-C(5)-N(5)-O(5B)	2.1(18)
C(4)-C(5)-N(5)-O(5B)	-173.3(12)
C(5)-N(1)-N(2)-C(3)	0.9(15)
N(1)-N(2)-C(3)-C(4)	0.8(16)
N(1)-N(2)-C(3)-N(3)	-179.5(11)
N(4)-C(4)-C(3)-N(2)	-178.8(13)
C(5)-C(4)-C(3)-N(2)	-2.0(14)
N(4)-C(4)-C(3)-N(3)	2(2)
C(5)-C(4)-C(3)-N(3)	178.3(14)
N(2)-C(3)-N(3)-O(3B)	179.2(13)
C(4)-C(3)-N(3)-O(3B)	-1(2)
N(2)-C(3)-N(3)-O(3A)	-2.5(19)
C(4)-C(3)-N(3)-O(3A)	177.2(13)

Table A-3g. Hydrogen bonds for ADNP*H₂O [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(4)-H(4A)...O(3B)	0.86	2.33	2.860(17)	120.4
N(4)-H(4B)...O(5A)	0.86	2.34	2.864(16)	119.3
N(2)-H(2A)...O(1S)	0.86	1.93	2.787(15)	176.0
N(4)-H(4B)...O(5A)#1	0.86	2.35	3.109(15)	147.7
O(1S)-H(1SB)...O(3A)#2	0.85	2.17	2.866(14)	138.4
O(1S)-H(1SA)...O(1S)#3	0.85	2.02	2.865(12)	179.1

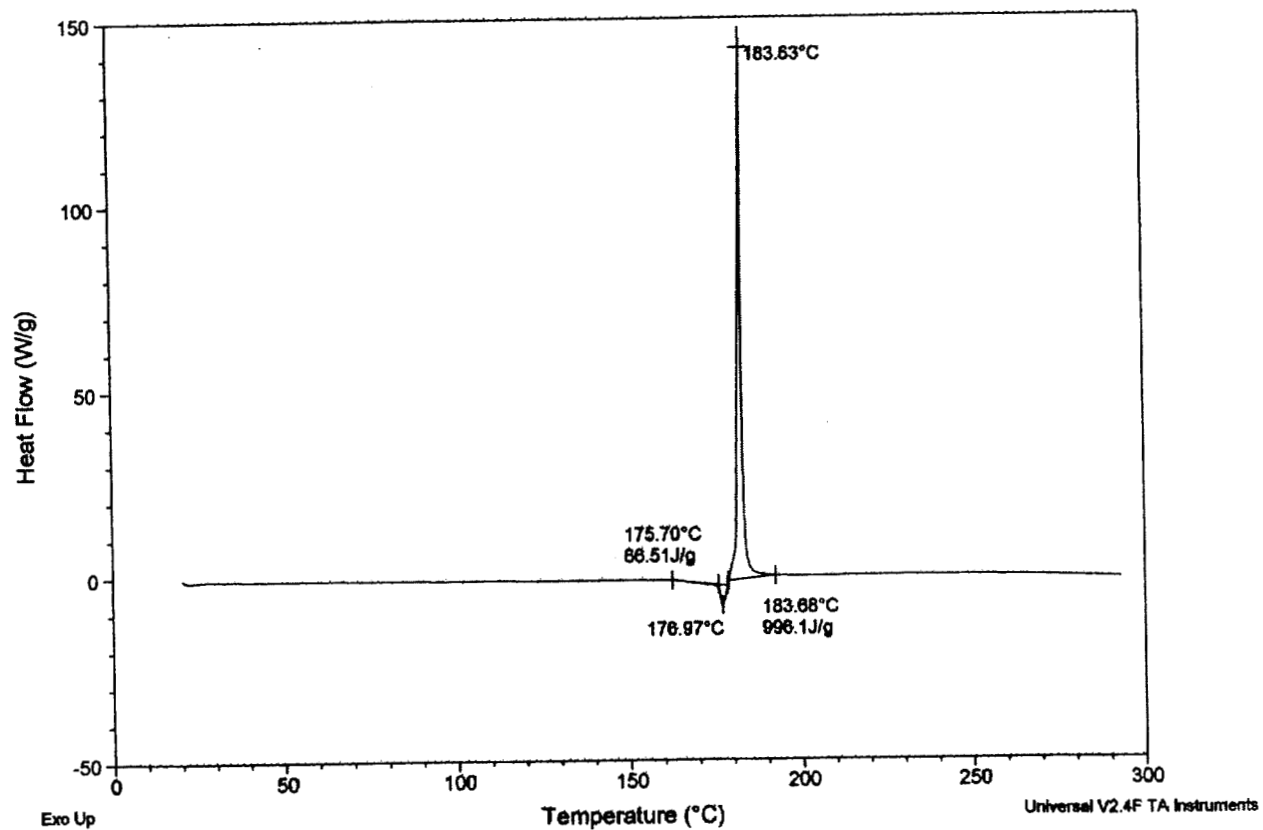
Symmetry transformations used to generate equivalent atoms:

#1 $-x-1, y+1/2, -z+1/2$ #2 $x+1/2, -y+3/2, -z$ #3 $x+1/2, -y+1/2, -z$

APPENDIX B: Differential Scanning Calorimetry (DSC) plot of LLM-116

Sample: ADNP RDS-III-67-3
Size: 0.3060 mg
Method: RT_to_450C
Comment: RT to 450C @ 10° C MIN

DSC



APPENDIX C: Small Scale Safety Test Data Sheets for LLM-116

C-1. Drop Hammer Data Sheet

DROP HAMMER CALCULATION — Dh ₅₀		
Name of Explosive:	ADNP	
Identification Number:	RDS-III-83	
Date of Test:	5/15/01	
Operator:	S. Weber	
Requester:	R. Schmidt	
Account Number:	6862-12	
Sample Description:	35 mg PILE	
Test Type:	12	
Temperature:	70 °F	
Humidity:	61%	
Book Reference:	19-11	
Number of X's	2	
Number of O's	13	
+/- 0.5	-0.5	
Least Height for X's or O's	177.4	
LOG of Least Height	2.2490	
LOG of Interval	0.05	
# of Events at Least Height	2	177.4
# of Events at Next Height	0	199.0
# of Events at Next Height	0	223.3
# of Events at Next Height	0	250.6
# of Events at Next Height	0	281.2
# of Events at Next Height	0	315.5
<u>Dh₅₀ CALCULATION</u>		
Sum (N _i * i)	0	
Sum of Events for Calculation	2	
LOG of Dh ₅₀	2.2240	
Dh₅₀		167.5 cm
<u>STANDARD DEVIATION CALCULATION</u>		
Sum (N _i * i ²)	0	
STANDARD DEVIATION		0.002
MAXIMUM Dh₅₀	0.9	168.4 cm
MINIMUM Dh₅₀	-0.9	166.6 cm

C-2. Spark Sensitivity Data Sheet

ELECTROSTATIC SPARK TEST					
REQUESTOR: <u>R. Schmidt</u>			DATE: <u>5/15/01</u>		
ACCOUNT NO.: <u>6862-12</u>			TEMP./R.H.: <u>68°F/67%</u>		
MATERIAL: <u>ADNP</u>			OPERATOR: <u>S. Weber</u>		
IDENTIFICATION NO.: <u>RDS-III-83</u>					
CAP. (pF)	VOLTS (kV)	RESISTANCE (ohms)	GAP (in.)	ENERGY (Joules)	REACTION (YES or NO)
<u>20k_pF</u>	<u>10.0</u>	<u>510</u>	<u>0.007</u>	<u>1.0</u>	<u>NO</u>
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
↓	↓	↓	↓	↓	"
<u>0</u> OF 10 TESTS @ <u>1.0</u> JOULES WITH 510-OHM RESISTANCE <input type="checkbox"/> IS SPARK SENSITIVE <input checked="" type="checkbox"/> IS NOT SPARK SENSITIVE					
REMARKS:					

C-3. Friction Sensitivity Data Sheet

FRICTION B.A.M. TEST	
Requester: R. Schmidt	Date: 5/15/01
Account Number: 6862.12	Book Reference: II-100
Material: ADNP	Operator: S. Weber
Identification Number: RDS-II-83	
Test Results: 1/10 @ 28.8 kg	
Room conditions at time of test	
Temperature: 70 °F	
Relative Humidity: 67%	

C-4. Chemical Reactivity Test Data Sheet

MATERIAL	WEIGHT (grams)	TEMP (deg C)	He	N2	O2	Ar	CO	NO	CO2	N2O	TOTAL (cc/.25g)	DATE	JOB NO.	REMARKS	ADJ CO2	ADJ TOTAL (cc/.25g)
ADNP (LLM-116)	0.25	80		0.002					0.004		0.006	5/10/02	2468	RDS-III-83	-0.008	0.002
ADNP (LLM-116)	0.25	80		0.002					0.005		0.007	5/10/02	2468	RDS-III-83	-0.008	0.002
ADNP monohydrate	2500	80		0.001					0.008		0.009	1/7/99	2239	GSL-II-133-A	-0.009	0.001
ADNP monohydrate	2500	80		0.002					0.005		0.007	1/7/99	2239	GSL-II-133-A	-0.009	0.002

APPENDIX D: Mass Spectrum (+EI) for LLM-116

