Supporting Information

Belonging to the manuscript

Energetic salts of substituted 1,2,4-triazolium and tetrazolium 3,5-dinitro-1,2,4-triazolates

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Experimental:

Crystals of compound **16** were removed from the flask and covered with a layer of hydrocarbon oil. A suitable crystal was selected, attached to a glass fiber and placed in the low-temperature nitrogen stream.[1] Data for **16** were collected at 87(2) K using a Bruker/Siemens SMART APEX instrument (Mo Kα radiation, λ = 0.71073 Å) equipped with a Cryocool NeverIce low temperature device. Data were measured using omega scans of 0.3 ° per frame for 30 seconds, and a full sphere of data was collected. A total of 2450 frames were collected with a final resolution of 0.83 Å. The first 50 frames were recollected at the end of data collection to monitor for decay. Cell parameters were retrieved using SMART[2] software and refined using SAINTPlus[3] on all observed reflections. Data reduction and correction for Lp and decay were performed using the SAINTPlus software. Absorption corrections were applied using SADABS.[4] The structure was solved by direct methods and refined by least squares method on F^2 using the SHELXTL program package.[5] The structure was solved in the space group P2(1)/c (# 14) by analysis of systematic absences. All non-hydrogen atoms were refined anisotropically. No decomposition was observed during data collection. Details of the data collection and refinement are given in Table 1. Further details are provided in the Supporting Information.

Acknowledgement

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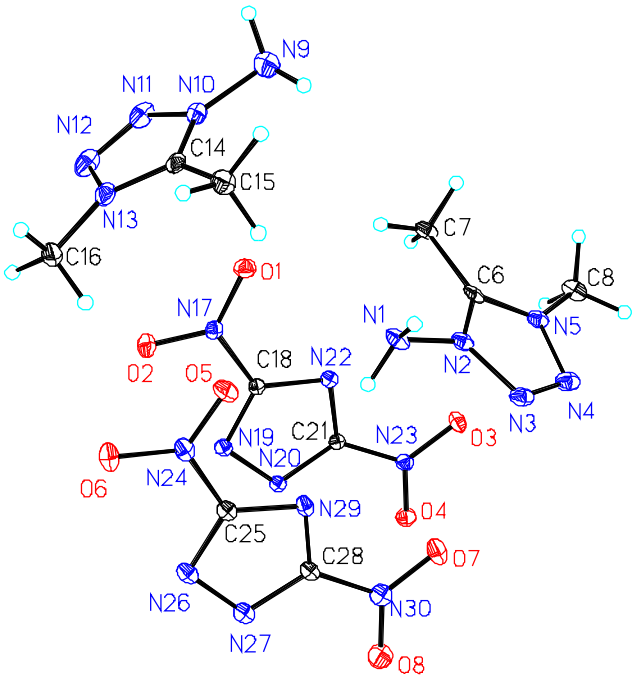
[1] Hope, H. *Prog. Inorg. Chem.*, **1994**, *41*, 1.
[2] SMART: v.5.626, Bruker Molecular Analysis Research Tool, Bruker AXS, Madison, WI, **2002**.
[3] SAINTPlus: v. 6.45a, Data Reduction and Correction Program, Bruker AXS, Madison, WI, **2003**.
[4] SADABS: v.2.01, an empirical absorption correction program, Bruker AXS Inc., Madison, WI, **2004**.
[5] SHELXTL: v. 6.10, Structure Determination Software Suite, Sheldrick, G.M., Bruker AXS Inc., Madison, WI, **2001**.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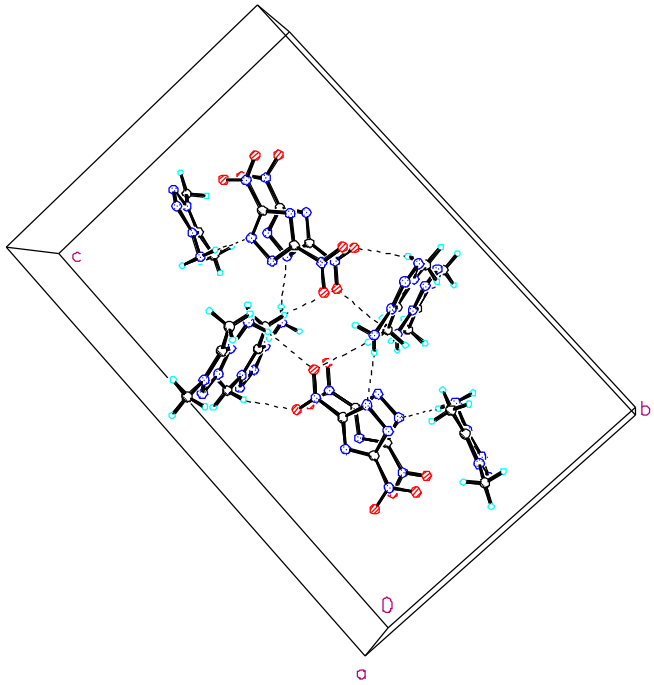
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Table 1. Crystal data and structure refinement for 4, 5-dimethyl-1-aminotetrazolium 3, 5-dinitro-1, 2, 4-triazolate (**16**).

Identification code	**16**	
Empirical formula	C5 H8 N10 O4	
Formula weight	272.21	
Temperature	87(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/c	
Unit cell dimensions	a = 6.8731(4) Å	α= 90°.
	b = 14.3073(8) Å	β= 90.4460(10)°.
	c = 22.1427(12) Å	γ = 90°.
Volume	2177.3(2) $Å^3$	
Z	8	
Density (calculated)	1.661 Mg/m^3	
Absorption coefficient	0.143 mm^{-1}	
F(000)	1120	
Crystal size	0.30 x 0.16 x 0.04 mm^3	
Crystal color and habit	colorless plate	
Diffractometer	Bruker/Siemens SMART APEX	
Theta range for data collection	1.84 to 25.24°.	
Index ranges	-8<=h<=8, -17<=k<=17, -26<=l<=26	
Reflections collected	24316	
Independent reflections	3942 [R(int) = 0.0704]	
Completeness to theta = 25.24°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9943 and 0.9584	
Solution method	XS, Bruker SHELXTL v. 6.12	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3942 / 0 / 347	
Goodness-of-fit on F^2	1.061	
Final R indices [I>2sigma(I)]	R1 = 0.0555, wR2 = 0.1210	
R indices (all data)	R1 = 0.0845, wR2 = 0.1330	
Largest diff. peak and hole	0.465 and -0.347 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **16**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(6)	1378(4)	1156(2)	3248(1)	17(1)
C(7)	-96(4)	1381(2)	2784(1)	21(1)
C(8)	-803(5)	781(2)	4124(2)	27(1)
C(14)	2842(5)	2064(2)	682(1)	20(1)
C(15)	4302(5)	1346(2)	836(2)	25(1)
C(16)	4973(5)	3412(2)	409(2)	28(1)
C(18)	2622(4)	3931(2)	2669(1)	13(1)
C(21)	2343(4)	3906(2)	3568(1)	13(1)
C(25)	7496(4)	3752(2)	2582(1)	16(1)
C(28)	7289(4)	3792(2)	3484(1)	16(1)
N(1)	4329(3)	1404(2)	2687(1)	21(1)
N(2)	3294(3)	1168(2)	3201(1)	18(1)
N(3)	4148(4)	925(2)	3736(1)	22(1)
N(4)	2775(4)	765(2)	4112(1)	24(1)
N(5)	1063(3)	905(2)	3813(1)	19(1)
N(9)	-284(4)	1215(2)	872(1)	37(1)
N(10)	907(4)	1996(2)	701(1)	20(1)
N(11)	58(4)	2812(2)	540(1)	28(1)
N(12)	1427(5)	3389(2)	419(1)	31(1)
N(13)	3148(4)	2938(2)	504(1)	24(1)
N(17)	2558(3)	3688(2)	2038(1)	17(1)
N(19)	3366(3)	4747(2)	2851(1)	16(1)
N(20)	3168(3)	4736(2)	3458(1)	15(1)
N(22)	1945(3)	3361(2)	3095(1)	14(1)
N(23)	1884(3)	3631(2)	4177(1)	17(1)
N(24)	7393(3)	3453(2)	1960(1)	20(1)
N(26)	8252(3)	4577(2)	2730(1)	20(1)
N(27)	8115(3)	4610(2)	3339(1)	20(1)
N(29)	6849(3)	3220(2)	3034(1)	17(1)
N(30)	6877(4)	3566(2)	4108(1)	22(1)
O(1)	1674(3)	2985(1)	1882(1)	22(1)
O(2)	3418(3)	4210(2)	1686(1)	26(1)

O(3)	1122(3)	2861(1)	4256(1)	22(1)
O(4)	2272(3)	4179(1)	4588(1)	26(1)
O(5)	6710(3)	2676(2)	1853(1)	27(1)
O(6)	7976(3)	3992(2)	1571(1)	26(1)
O(7)	5978(3)	2842(2)	4209(1)	30(1)
O(8)	7459(3)	4110(2)	4498(1)	30(1)

Table 3. Bond lengths [Å] and angles [°] for **16**.

C(6)-N(5)	1.320(4)	N(2)-N(3)	1.364(3)
C(6)-N(2)	1.322(4)	N(3)-N(4)	1.284(4)
C(6)-C(7)	1.474(4)	N(4)-N(5)	1.361(3)
C(7)-H(7A)	0.9800	N(9)-N(10)	1.438(4)
C(7)-H(7B)	0.9800	N(9)-H(9A)	0.9019
C(7)-H(7C)	0.9800	N(9)-H(9B)	0.9052
C(8)-N(5)	1.471(4)	N(10)-N(11)	1.351(3)
C(8)-H(8A)	0.9800	N(11)-N(12)	1.282(4)
C(8)-H(8B)	0.9800	N(12)-N(13)	1.360(4)
C(8)-H(8C)	0.9800	N(17)-O(1)	1.224(3)
C(14)-N(13)	1.328(4)	N(17)-O(2)	1.234(3)
C(14)-N(10)	1.334(4)	N(19)-N(20)	1.351(3)
C(14)-C(15)	1.474(4)	N(23)-O(4)	1.228(3)
C(15)-H(15A)	0.9800	N(23)-O(3)	1.233(3)
C(15)-H(15B)	0.9800	N(24)-O(6)	1.226(3)
C(15)-H(15C)	0.9800	N(24)-O(5)	1.229(3)
C(16)-N(13)	1.443(4)	N(26)-N(27)	1.353(4)
C(16)-H(16A)	0.9800	N(30)-O(8)	1.228(3)
C(16)-H(16B)	0.9800	N(30)-O(7)	1.229(3)
C(16)-H(16C)	0.9800		
C(18)-N(22)	1.333(3)	N(5)-C(6)-N(2)	104.4(2)
C(18)-N(19)	1.335(4)	N(5)-C(6)-C(7)	127.1(3)
C(18)-N(17)	1.440(4)	N(2)-C(6)-C(7)	128.5(3)
C(21)-N(22)	1.332(3)	C(6)-C(7)-H(7A)	109.5
C(21)-N(20)	1.340(4)	C(6)-C(7)-H(7B)	109.5
C(21)-N(23)	1.442(4)	H(7A)-C(7)-H(7B)	109.5
C(25)-N(26)	1.330(4)	C(6)-C(7)-H(7C)	109.5
C(25)-N(29)	1.335(4)	H(7A)-C(7)-H(7C)	109.5
C(25)-N(24)	1.444(4)	H(7B)-C(7)-H(7C)	109.5
C(28)-N(29)	1.323(4)	N(5)-C(8)-H(8A)	109.5
C(28)-N(27)	1.341(4)	N(5)-C(8)-H(8B)	109.5
C(28)-N(30)	1.448(4)	H(8A)-C(8)-H(8B)	109.5
N(1)-N(2)	1.388(3)	N(5)-C(8)-H(8C)	109.5
N(1)-H(1A)	0.8999	H(8A)-C(8)-H(8C)	109.5
N(1)-H(1B)	0.9009	H(8B)-C(8)-H(8C)	109.5

N(13)-C(14)-N(10)	103.8(3)	N(3)-N(4)-N(5)	107.1(2)
N(13)-C(14)-C(15)	128.0(3)	C(6)-N(5)-N(4)	110.7(2)
N(10)-C(14)-C(15)	128.2(3)	C(6)-N(5)-C(8)	128.8(3)
C(14)-C(15)-H(15A)	109.5	N(4)-N(5)-C(8)	120.5(3)
C(14)-C(15)-H(15B)	109.5	N(10)-N(9)-H(9A)	105.2
H(15A)-C(15)-H(15B)	109.5	N(10)-N(9)-H(9B)	103.5
C(14)-C(15)-H(15C)	109.5	H(9A)-N(9)-H(9B)	123.6
H(15A)-C(15)-H(15C)	109.5	C(14)-N(10)-N(11)	110.9(3)
H(15B)-C(15)-H(15C)	109.5	C(14)-N(10)-N(9)	129.3(3)
N(13)-C(16)-H(16A)	109.5	N(11)-N(10)-N(9)	119.7(2)
N(13)-C(16)-H(16B)	109.5	N(12)-N(11)-N(10)	107.2(3)
H(16A)-C(16)-H(16B)	109.5	N(11)-N(12)-N(13)	107.7(2)
N(13)-C(16)-H(16C)	109.5	C(14)-N(13)-N(12)	110.4(3)
H(16A)-C(16)-H(16C)	109.5	C(14)-N(13)-C(16)	128.8(3)
H(16B)-C(16)-H(16C)	109.5	N(12)-N(13)-C(16)	120.8(3)
N(22)-C(18)-N(19)	117.1(2)	O(1)-N(17)-O(2)	123.9(2)
N(22)-C(18)-N(17)	122.1(2)	O(1)-N(17)-C(18)	119.0(2)
N(19)-C(18)-N(17)	120.8(2)	O(2)-N(17)-C(18)	117.1(2)
N(22)-C(21)-N(20)	117.4(2)	C(18)-N(19)-N(20)	104.4(2)
N(22)-C(21)-N(23)	122.1(2)	C(21)-N(20)-N(19)	103.7(2)
N(20)-C(21)-N(23)	120.5(2)	C(21)-N(22)-C(18)	97.4(2)
N(26)-C(25)-N(29)	116.9(3)	O(4)-N(23)-O(3)	123.8(2)
N(26)-C(25)-N(24)	121.0(3)	O(4)-N(23)-C(21)	118.1(2)
N(29)-C(25)-N(24)	122.1(2)	O(3)-N(23)-C(21)	118.1(2)
N(29)-C(28)-N(27)	117.0(3)	O(6)-N(24)-O(5)	124.0(3)
N(29)-C(28)-N(30)	122.4(2)	O(6)-N(24)-C(25)	118.0(2)
N(27)-C(28)-N(30)	120.6(3)	O(5)-N(24)-C(25)	117.9(2)
N(2)-N(1)-H(1A)	108.8	C(25)-N(26)-N(27)	104.3(2)
N(2)-N(1)-H(1B)	108.4	C(28)-N(27)-N(26)	104.0(2)
H(1A)-N(1)-H(1B)	121.1	C(28)-N(29)-C(25)	97.8(2)
C(6)-N(2)-N(3)	110.5(2)	O(8)-N(30)-O(7)	124.6(3)
C(6)-N(2)-N(1)	125.8(3)	O(8)-N(30)-C(28)	117.7(2)
N(3)-N(2)-N(1)	123.7(2)	O(7)-N(30)-C(28)	117.7(2)
N(4)-N(3)-N(2)	107.2(2)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **16**. The anisotropic displacement factor exponent takes the form: $-2\ ^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(6)	15(2)	5(1)	31(2)	-6(1)	0(1)	0(1)
C(7)	13(2)	13(1)	36(2)	-3(1)	-5(1)	1(1)
C(8)	23(2)	24(2)	34(2)	-7(2)	5(1)	-4(1)
C(14)	30(2)	18(2)	12(2)	-3(1)	0(1)	1(1)
C(15)	24(2)	22(2)	30(2)	0(1)	-2(1)	2(1)
C(16)	36(2)	27(2)	22(2)	2(1)	3(1)	-11(2)
C(18)	7(1)	14(1)	16(2)	2(1)	-1(1)	3(1)
C(21)	13(1)	12(1)	15(2)	-1(1)	0(1)	2(1)
C(25)	10(1)	14(2)	24(2)	0(1)	1(1)	4(1)
C(28)	11(1)	12(1)	25(2)	-1(1)	2(1)	2(1)
N(1)	15(1)	14(1)	35(2)	-4(1)	3(1)	0(1)
N(2)	14(1)	13(1)	27(1)	-4(1)	-1(1)	1(1)
N(3)	21(1)	18(1)	28(2)	-7(1)	-5(1)	1(1)
N(4)	21(1)	21(1)	30(2)	-6(1)	-5(1)	3(1)
N(5)	17(1)	13(1)	28(2)	-6(1)	-1(1)	2(1)
N(9)	36(2)	37(2)	38(2)	4(1)	-5(1)	-7(1)
N(10)	22(2)	20(1)	18(1)	1(1)	0(1)	2(1)
N(11)	37(2)	25(2)	23(2)	-3(1)	-2(1)	14(1)
N(12)	46(2)	25(2)	22(2)	-1(1)	-3(1)	13(1)
N(13)	36(2)	20(1)	16(1)	-1(1)	1(1)	1(1)
N(17)	14(1)	17(1)	18(1)	2(1)	-1(1)	3(1)
N(19)	11(1)	17(1)	20(1)	2(1)	-1(1)	2(1)
N(20)	14(1)	14(1)	17(1)	0(1)	0(1)	0(1)
N(22)	11(1)	13(1)	17(1)	-1(1)	1(1)	3(1)
N(23)	19(1)	13(1)	20(1)	-2(1)	-3(1)	1(1)
N(24)	15(1)	17(1)	28(2)	4(1)	1(1)	2(1)
N(26)	13(1)	15(1)	31(2)	1(1)	1(1)	1(1)
N(27)	16(1)	17(1)	26(2)	-1(1)	1(1)	2(1)
N(29)	12(1)	16(1)	25(1)	5(1)	2(1)	1(1)
N(30)	18(1)	21(1)	27(2)	-1(1)	1(1)	6(1)
O(1)	25(1)	21(1)	21(1)	-3(1)	-2(1)	-1(1)
O(2)	28(1)	29(1)	21(1)	6(1)	4(1)	-4(1)

O(3)	27(1)	15(1)	25(1)	1(1)	4(1)	-4(1)
O(4)	36(1)	22(1)	19(1)	-4(1)	-2(1)	-5(1)
O(5)	31(1)	19(1)	31(1)	-3(1)	-2(1)	-8(1)
O(6)	28(1)	22(1)	29(1)	8(1)	7(1)	2(1)
O(7)	30(1)	24(1)	35(1)	7(1)	6(1)	-5(1)
O(8)	36(1)	26(1)	29(1)	-5(1)	0(1)	2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **16**.

	x	y	z	U(eq)
H(7A)	553	1559	2408	31
H(7B)	-903	1901	2923	31
H(7C)	-919	832	2710	31
H(8A)	-579	460	4509	40
H(8B)	-1681	406	3871	40
H(8C)	-1390	1394	4199	40
H(15A)	4706	1420	1259	38
H(15B)	5435	1417	574	38
H(15C)	3735	724	778	38
H(16A)	5838	3304	756	43
H(16B)	4737	4084	364	43
H(16C)	5583	3171	42	43
H(1A)	4932	890	2548	32
H(1B)	4996	1934	2763	32
H(9A)	-900	1037	530	55
H(9B)	548	842	1080	55

Table 6. Hydrogen bonds for **16** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1A)...N(19)#1	0.90	2.20	3.096(3)	173.3
N(1)-H(1B)...N(29)	0.90	2.31	3.213(3)	174.6
N(1)-H(1B)...O(5)	0.90	2.57	3.074(3)	115.9
N(9)-H(9A)...O(8)#2	0.90	2.55	3.436(4)	168.1
N(9)-H(9B)...N(27)#1	0.91	2.36	3.241(4)	163.3

Symmetry transformations used to generate equivalent atoms:

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