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One-pot synthesis of 4,6,8-trinitro-4,5,7,8-tetrahydro-6*H*-furazano[3,4-*f*]-1,3,5-triazepine in ionic liquids

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Table 1 Conditions for preparation of compound **1** from diamine **1** in ILs.

Entry	IL systems	Nitration step				Condensation step			Yield of **1** (%)
		HNO_3 (equiv.)	Ac_2O (equiv.)	*T* /°C	t / h	**6a** or **6b** (equiv.)	*T* /°C	t / h	
1	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:1)	1	-	0	1	**6a** (1)	20	8	43
2	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:1)	1	-	0	1	**6b** (1)	20	8	54
3	[emim][HSO_4] [bmim][PF_6] (1:1)	1	-	0	1	**6b** (1)	20	8	51
4	[emim][HSO_4] [bmim][BF_4] (1:1)	1	-	0	1	**6b** (1)	20	8	41
5	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1	-	0	1	**6b** (1)	20	8	59
6	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1	-	20	1	**6b** (1)	20	8	31
7	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	-	0	1	**6b** (1)	20	8	73
8	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	-	0	2	**6b** (1)	20	8	69
9	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	-	0	1	**6b** (1)	20	16	74
10	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.5	-	0	1	**6b** (1)	20	8	72
11	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	-	0	1	**6b** (1.5)	20	8	85
12	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	1.2	0	1	**6b** (1.5)	20	8	83
13	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	1.2	0	1	**6a** (1.5)	20	8	57
14	[emim][HSO_4] [bmpyrr][CF_3SO_3] (1:2)	1.2	-	0	1	**6b** (1.5)	40	8	84

Table 2 Selected geometry characteristics of compound **1**; X-ray *versus ab initio* data.

Parameter	Unprimed molecule A	Primed molecule A'	M052X/ aug-cc-pvdz
Interplanar angle (°) (N3, C3, C4, N5) (N5,C1,C2,N3)	33.47(5)	34.96(5)	30.43
Interplanar angle (°) (N3, C3, C4, N5) (C3,N4,C4)	66.46(8)	68.71(8)	65.94
Sum of bond angles at N3 (°)	359.0(2)	359.1(2)	357.5
Sum of bond angles at N4 (°)	354.9(2)	351.1(2)	349.8
Sum of bond angles at N5 (°)	358.0(2)	359.5(2)	357.5
Intramolecular contacts (Å)			
O3…O4	3.400(1)	3.178(1)	2.939
O5…O6	2.958(1)	2.968(1)	2.939
Intermolecular contacts (Å)			
O6…O5'(0.5-*x*, 0.5+*y*, *z*)	2.866(1)		–
O5…O7'	2.873(1)		

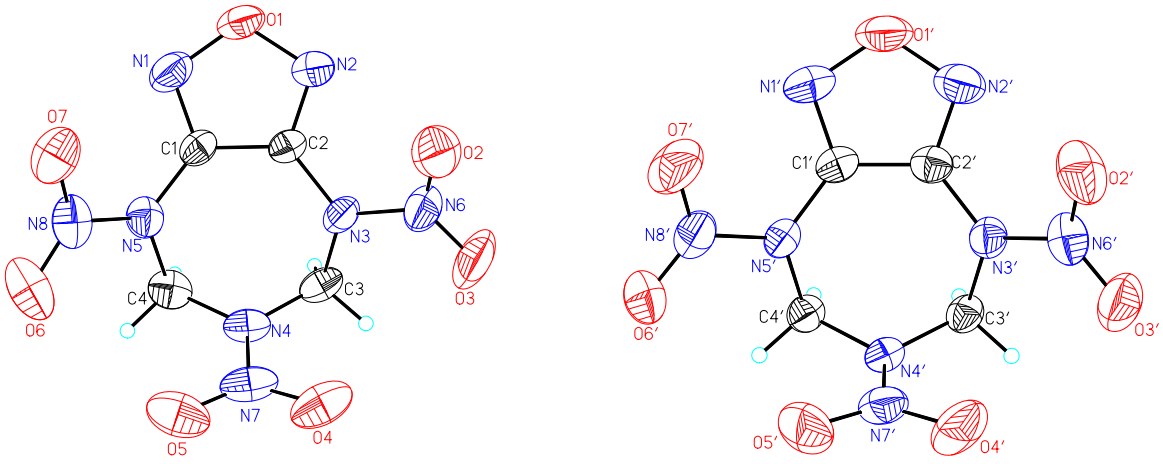
Table 3 Experimental and calculated intramolecular C-H…O contacts in compound **1** (interatomic distances in Å, angles in degrees).

H-bond	Unprimed molecule A			Primed molecule A'			M052X/aug-cc-pvdz		
	C…O	H…O	<CHO	C…O	H…O	<CHO	C…O	H…O	<CHO
C3-H3A…O3	2.668(1)	2.24	104	2.662(1)	2.21	106	2.691	2.21	106
C4-H4A…O6	2.696(1)	2.25	106	2.681(1)	2.23	106	2.691	2.21	106

*Crystal data and structure refinement for compound **1** obtained at room temperature (293K).*

Identification code	rt169	
Empirical formula	C4 H4 N8 O7	
Formula weight	276.15	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P b c a	
Unit cell dimensions	a = 12.174(11) Å	α= 90°.
	b = 13.952(12) Å	β= 90°.
	c = 22.83(2) Å	γ = 90°.
Volume	3878(6) $Å^3$	
Z	16	

Density (calculated)	1.892 Mg/m^3
Absorption coefficient	0.179 mm^{-1}
F(000)	2240
Crystal size	0.34 x 0.26 x 0.18 mm^3
Theta range for data collection	1.78 to 29.00°.
Index ranges	-16<=h<=16, -17<=k<=19, -31<=l<=24
Reflections collected	22276
Independent reflections	5135 [R(int) = 0.0326]
Completeness to theta = 29.00°	99.7 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	5135 / 0 / 343
Goodness-of-fit on F^2	0.993
Final R indices [for 3551 rfln with I>2sigma(I)]	R1 = 0.0405, wR2 = 0.0978
R indices (all data)	R1 = 0.0662, wR2 = 0.1135
Largest diff. peak and hole	0.200 and -0.211 $e.Å^{-3}$



ORTEP view of the compound **1**; atomic displacement ellipsoids drawn at 50% probability level (left) unprimed molecule A; (right) primed molecule A'