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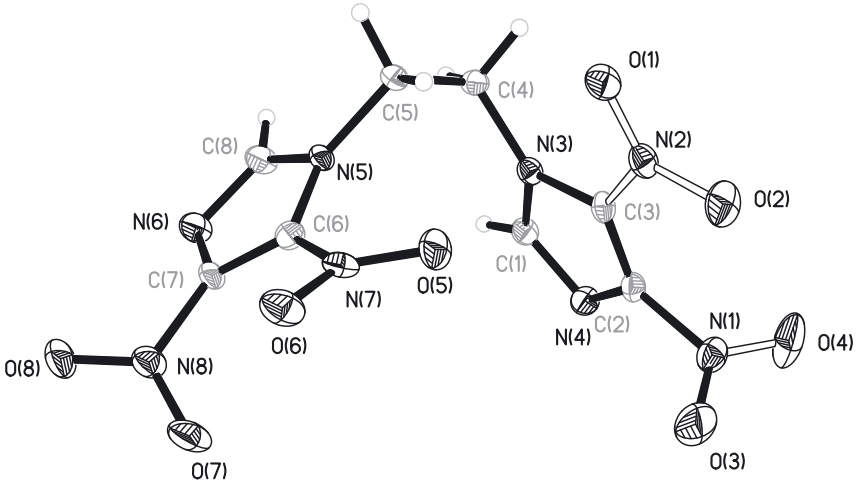
Crystal structure of 1,2-bis(4,5-dinitro-1*H*-imidazol-1-yl)ethane, $C_8H_6N_8O_8$



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Abstract

$C_8H_6N_8O_8$, orthorhombic, $P2_12_12_1$ (no. 19), a = 6.7563(9) Å, b = 7.2512(12) Å, c = 25.884(3) Å, V = 1268.1(3) Å^3, β = 90°, Z = 4, $R_{gt}(F)$ = 0.0473, w$R_{ref}(F^2)$ = 0.1170, T = 170 K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.15 × 0.07 × 0.04 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.16 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	26.4°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	9675, 2589, 0.088
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2044
$N(param)_{refined}$:	218
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

Source of material

All of reagents were purchased with analysis grade. In a representative experiments, 5 ml HNO_3 and 15 ml H_2SO_4 were mixed, then 7.9 g (50 mmol) 1,2-bis(1*H*-imidazol-1-yl) ethane was added at 293 K. The target compound was obtained by reaction at 353 K for 10 h. The crystal of the title compound was obtained by slow evaporation at 293 K.

Experimental details

A suitable crystal was selected and on a 'Bruker APEX-II CCD' diffractometer. The crystal was kept at 170 K during data collection. Using Olex2 [2], the structure was solved with the SHELXT [3] structure solution program and refined

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2743 (5)	0.2916 (5)	0.48509 (12)	0.0368 (8)
O2	0.4950 (5)	0.4987 (5)	0.50361 (12)	0.0446 (9)
O3	0.7068 (5)	0.7629 (7)	0.42673 (18)	0.0722 (14)
O4	0.5122 (6)	0.9274 (6)	0.47318 (18)	0.0684 (13)
O5	0.4961 (5)	0.2121 (5)	0.38525 (12)	0.0406 (8)
O6	0.6119 (4)	0.0237 (5)	0.32811 (13)	0.0413 (8)
O7	0.6130 (4)	0.1753 (6)	0.23068 (12)	0.0521 (10)
O8	0.3949 (5)	0.0355 (5)	0.18278 (13)	0.0471 (9)
N1	0.5446 (5)	0.8081 (6)	0.44187 (16)	0.0357 (9)
N2	0.3581 (5)	0.4366 (5)	0.47752 (13)	0.0294 (8)
N3	0.1336 (5)	0.5227 (4)	0.40404 (12)	0.0234 (7)
N4	0.2710 (5)	0.7910 (5)	0.38133 (14)	0.0293 (8)
N5	0.1296 (5)	0.1981 (5)	0.33184 (12)	0.0231 (7)
N6	0.1064 (5)	0.1908 (6)	0.24530 (13)	0.0346 (9)
N7	0.4866 (5)	0.1300 (5)	0.34495 (13)	0.0278 (8)
N8	0.4451 (5)	0.1192 (6)	0.22178 (14)	0.0351 (9)
C1	0.1274 (6)	0.6692 (6)	0.37223 (15)	0.0262 (9)
H1	0.030609	0.684333	0.345953	0.031*
C2	0.3722 (6)	0.7141 (6)	0.42056 (15)	0.0252 (9)
C3	0.2963 (6)	0.5520 (6)	0.43599 (15)	0.0238 (9)
C4	−0.0153 (6)	0.3762 (6)	0.40475 (16)	0.0272 (9)
H4A	−0.128576	0.414793	0.383035	0.033*
H4B	−0.064496	0.361850	0.440557	0.033*
C5	0.0573 (6)	0.1913 (6)	0.38589 (16)	0.0260 (9)
H5A	0.165845	0.148523	0.408616	0.031*
H5B	−0.051883	0.100686	0.388304	0.031*
C6	0.3145 (5)	0.1582 (6)	0.31236 (16)	0.0257 (9)
C7	0.2948 (6)	0.1542 (6)	0.26058 (15)	0.0271 (10)
C8	0.0118 (6)	0.2155 (6)	0.28979 (16)	0.0322 (10)
H8	−0.125494	0.242814	0.291899	0.039*

with the ShelXL [4] refinement package. All H atoms were positioned geometrically and treated as riding, with C–H bond 0.95 Å, and U_{iso}(H) = 1.2U_{eq}(C).

Comment

Imidazole has arised great attention of chemists. A series of compounds such as nitroimidazole, bisimidazole, benzimidazole have been synthesized [5–8]. The title compound is been reported for the first time in this paper (cf. the figure).

The asymmetric unit of the title structure contains an ethan-1,2-diyl-bridge connecting two dinitroimidazolyl moieties forming the title molecule. Both dinitroimidazole rings are almost flat, and the dihedral angle of the two imidazole rings is 38.37°.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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