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4-[4-(4-Amino-1,2,5-oxadiazol-3-yl)-1,2,5-oxadiazol-3-yl]-1,2,5-oxadiazol-3-amine

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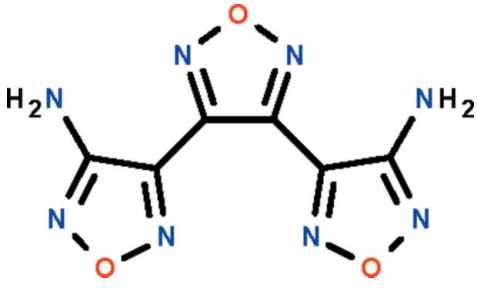
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.096; data-to-parameter ratio = 12.0.

The complete molecule of the compound, $C_6H_4N_8O_3$, is generated by a crystallographic twofold rotation axis that runs through the central ring. The flanking ring is twisted by 20.2 (1)° with respect to the central ring. One of the amino H atoms forms an intramolecular N—H···N hydrogen bond; adjacent molecules are linked by N—H···N hydrogen bonds forming a chain running along [10$\bar{2}$].

Related literature

For the synthesis, see: Kulikov & Kakhova (1994); Zhou *et al.* (2007).



Experimental

Crystal data

$C_6H_4N_8O_3$
M_r = 236.17
Monoclinic, $C2/c$
a = 7.1681 (9) Å
b = 10.8147 (13) Å
c = 12.3448 (18) Å
β = 103.155 (1)°
V = 931.9 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.33 × 0.26 × 0.17 mm

Data collection

Bruker SMART APEX diffractometer
2675 measured reflections
1047 independent reflections
933 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.096
S = 1.08
1047 reflections
87 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H1···N1	0.90 (2)	2.37 (2)	2.932 (2)	121 (1)
N4—H2···N3^{i}	0.87 (2)	2.23 (2)	3.070 (2)	162 (2)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT5881).

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supporting information

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4-[4-(4-Amino-1,2,5-oxadiazol-3-yl)-1,2,5-oxadiazol-3-yl]-1,2,5-oxadiazol-3-amine

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S1. Comment

We are interested in *N*-heterocyclic compounds having few hydrogen atoms as these compounds are a source of explosives. In the title compound (Scheme I), the hydrogen atoms constitute an amino group. In NH_2–C_2N_2O–C_2N_2O–C_2N_2O–NH_2, two amino-subsituted 1,2,5-oxadiazole rings flanking a central 1,2,5-oxadiazole ring; the molecule lies on a twofold rotation axis that relates one flanking ring to the other (Fig. 1). The flanking ring is twisted by 20.2 (1) ° with respect to the central ring. One of the amino H atoms forms an intramolecular hydrogen bond; adjacent molecules are linked by an N–H···N hydrogen bond (Table 1, Fig. 2). to form a chain running along [1 0 -2].

S2. Experimental

3,4-Bis(4′-aminofurazano-3′)furoxan was synthesized by using a literature procedure (Zhou *et al.*, 2007). The compound (7.5 g) was dissolved in acetic acid (30 ml). The solution was added to a reducing agent prepared from stannous chloride dihydrate (22.6 g. 100 mm mol) dissolved in acetic anhydride (20 ml), acetic acid (100 ml) and concentrated hydrochloric acid (20 ml). The reduction was performed according to an literature procedure (Kulikov & Kakhova, 1994). The mixture was heated atto 348 K for 8 h. The cool mixture was then poured into water (150 ml). The white precipitate that separated was collected and recrystallized from an ethyl acetate/ether mixture; yield 70%, m.pt. 456–457 K. The purity was established by HPLC to be 99.6%. CH&N elemental analysis. Calculated for $C_6H_4N_8O_3$ (%): C 30.51, N 47.46, H1.69. Found: C 30.41, N 47.58,H 1.61.

S3. Refinement

The H-atoms were located in a difference Fourier map, and were refined freely.

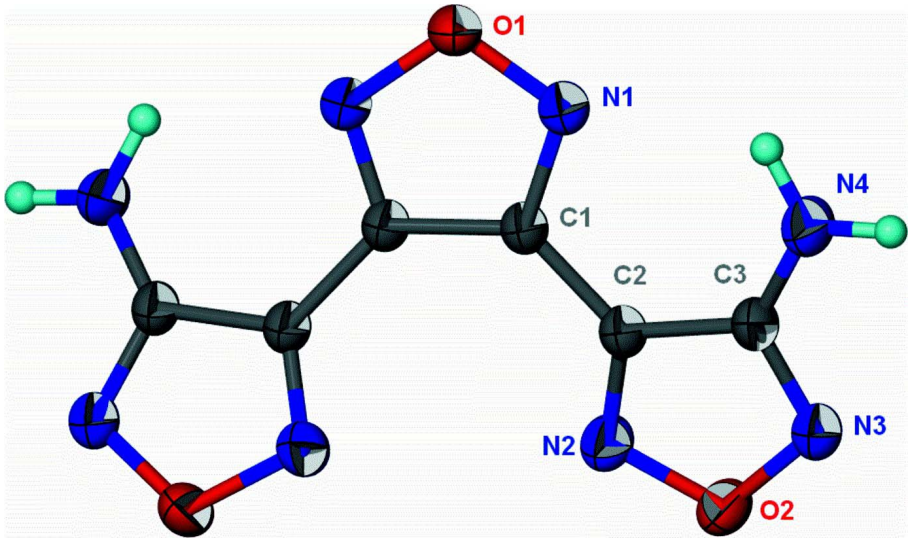

Figure 1

Anisotropic displacement ellipsoid plot (Barbour, 2001) of $C_6H_4N_8O_3$ at the 50% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The molecule is located on a twofold rotation axis; symmetry-related atoms are not labeled.

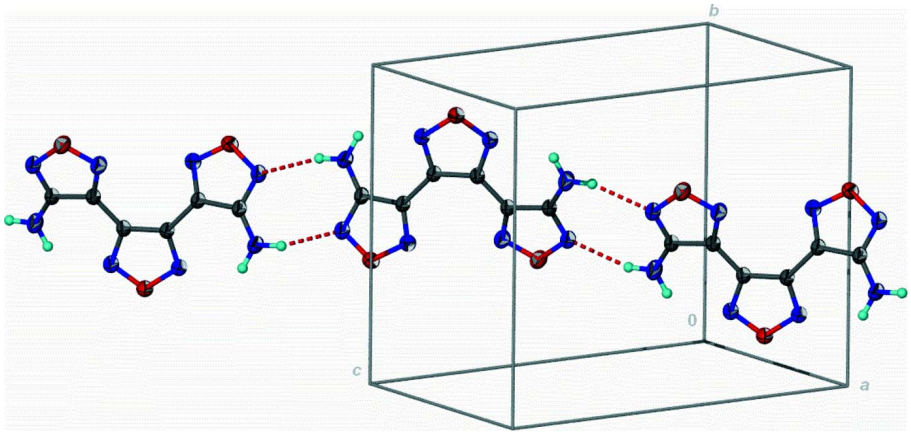

Figure 2

Hydrogen-bonded chain structure. The intermolecular H bond is drawn as a dashed line, the intramolecular H bond is not shown.

4-[4-(4-Amino-1,2,5-oxadiazol-3-yl)-1,2,5-oxadiazol-3-yl]-1,2,5-oxadiazol-3- amine

Crystal data

$C_6H_4N_8O_3$
M_r = 236.17
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 7.1681 (9) Å
b = 10.8147 (13) Å
c = 12.3448 (18) Å
β = 103.155 (1)°
V = 931.9 (2) Å^3
Z = 4
F(000) = 480
D_x = 1.683 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1575 reflections
θ = 3.4–27.7°
μ = 0.14 mm^{-1}

T = 293 K
Prism, colorless

0.33 × 0.26 × 0.17 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
2675 measured reflections
1047 independent reflections

933 reflections with $I > 2\sigma(I)$
R_{int} = 0.014
θ_{max} = 27.5°, θ_{min} = 3.4°
h = −9→9
k = −14→13
l = −15→8

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.096
S = 1.08
1047 reflections
87 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 0.219P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.020 (3)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
O1	0.0000	0.80892 (10)	0.7500	0.0471 (3)
O2	0.16242 (12)	0.35531 (8)	0.56878 (8)	0.0482 (3)
N1	0.09563 (14)	0.73719 (9)	0.68904 (8)	0.0431 (3)
N2	0.06415 (14)	0.41475 (9)	0.63634 (9)	0.0446 (3)
N3	0.29963 (15)	0.43342 (9)	0.54042 (9)	0.0453 (3)
N4	0.3974 (2)	0.63687 (11)	0.58898 (12)	0.0626 (4)
H1	0.372 (2)	0.7101 (14)	0.6165 (12)	0.059 (4)*
H2	0.480 (3)	0.6342 (15)	0.5474 (15)	0.064 (5)*
C1	0.06039 (15)	0.62259 (9)	0.71097 (9)	0.0349 (3)
C2	0.13553 (15)	0.52528 (10)	0.65104 (9)	0.0358 (3)
C3	0.28563 (16)	0.53785 (10)	0.59138 (10)	0.0393 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0644 (8)	0.0324 (6)	0.0520 (7)	0.000	0.0291 (6)	0.000
O2	0.0540 (5)	0.0385 (5)	0.0600 (6)	−0.0051 (4)	0.0291 (4)	−0.0103 (4)
N1	0.0529 (6)	0.0356 (5)	0.0471 (6)	−0.0024 (4)	0.0248 (5)	−0.0011 (4)
N2	0.0460 (5)	0.0397 (5)	0.0549 (6)	−0.0046 (4)	0.0255 (5)	−0.0068 (4)
N3	0.0513 (6)	0.0401 (5)	0.0526 (6)	0.0004 (4)	0.0287 (5)	−0.0004 (4)
N4	0.0745 (8)	0.0423 (6)	0.0915 (10)	−0.0118 (5)	0.0614 (8)	−0.0088 (6)
C1	0.0364 (5)	0.0344 (5)	0.0373 (5)	−0.0010 (4)	0.0151 (4)	0.0007 (4)

C2	0.0374 (5)	0.0349 (6)	0.0390 (6)	−0.0004 (4)	0.0166 (4)	0.0010 (4)
C3	0.0435 (6)	0.0366 (6)	0.0436 (6)	0.0017 (4)	0.0223 (5)	0.0022 (4)

Geometric parameters (Å, º)

O1—N1^{i}	1.3685 (11)	N4—C3	1.3419 (16)
O1—N1	1.3686 (11)	N4—H1	0.896 (16)
O2—N2	1.3684 (12)	N4—H2	0.871 (19)
O2—N3	1.4001 (13)	C1—C1^{i}	1.434 (2)
N1—C1	1.3055 (14)	C1—C2	1.4582 (15)
N2—C2	1.2967 (15)	C2—C3	1.4419 (15)
N3—C3	1.3077 (15)		
N1^{i}—O1—N1	110.94 (11)	N1—C1—C2	117.95 (9)
N2—O2—N3	110.93 (8)	C1^{i}—C1—C2	133.62 (6)
C1—N1—O1	106.22 (9)	N2—C2—C3	109.39 (10)
C2—N2—O2	106.07 (9)	N2—C2—C1	123.83 (9)
C3—N3—O2	105.40 (9)	C3—C2—C1	126.57 (10)
C3—N4—H1	121.5 (10)	N3—C3—N4	124.54 (11)
C3—N4—H2	118.5 (11)	N3—C3—C2	108.19 (10)
H1—N4—H2	118.6 (15)	N4—C3—C2	127.25 (11)
N1—C1—C1^{i}	108.31 (6)		
N1^{i}—O1—N1—C1	0.18 (6)	N1—C1—C2—C3	17.47 (17)
N3—O2—N2—C2	−0.32 (13)	C1^{i}—C1—C2—C3	−167.05 (15)
N2—O2—N3—C3	0.77 (13)	O2—N3—C3—N4	177.65 (12)
O1—N1—C1—C1^{i}	−0.43 (14)	O2—N3—C3—C2	−0.87 (13)
O1—N1—C1—C2	176.12 (8)	N2—C2—C3—N3	0.73 (14)
O2—N2—C2—C3	−0.23 (13)	C1—C2—C3—N3	−174.16 (11)
O2—N2—C2—C1	174.83 (10)	N2—C2—C3—N4	−177.73 (13)
N1—C1—C2—N2	−156.72 (11)	C1—C2—C3—N4	7.4 (2)
C1^{i}—C1—C2—N2	18.8 (2)		

Symmetry code: (i) −*x*, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H1···N1	0.90 (2)	2.37 (2)	2.932 (2)	121 (1)
N4—H2···N3^{ii}	0.87 (2)	2.23 (2)	3.070 (2)	162 (2)

Symmetry code: (ii) −*x*+1, −*y*+1, −*z*+1.