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2,2′-(4-Amino-4*H*-1,2,4-triazole-3,5-diyl)-diphenol

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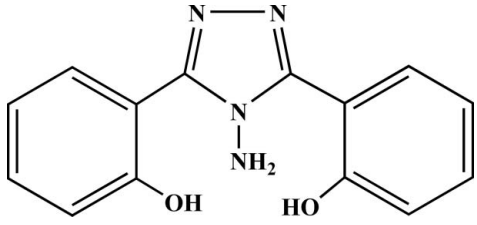
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.041; wR factor = 0.112; data-to-parameter ratio = 6.6.

The structure of the title compound, $C_{14}H_{12}N_4O_2$, was determined as part of a project on the coordination chemistry of 1,2,4-triazole derivatives. In the crystal structure, one of the two benzene rings is almost coplanar with the five-membered triazole ring (mean deviation = 0.019 Å), whereas the second benzene ring is rotated by 51.973 (2)°. The two N—C—N—N torsion angles [170.365 (2) and −170.942 (3)°] indicate that the amido group is slightly twisted away from the triazole plane. An intramolecular O—H···N hydrogen bond occurs. In the crystal structure, intermolecular N—H···O and O—H···N hydrogen bonding is found.

Related literature

For background information on tthe coordination chemistry of 1,2,4-triazole derivatives, see: Lavrenova *et al.* (1995).



Experimental

Crystal data

$C_{14}H_{12}N_4O_2$
M_r = 268.28
Orthorhombic, $P2_12_12_1$
a = 8.262 (2) Å
b = 9.384 (3) Å
c = 15.919 (4) Å
V = 1234.2 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 296 K
0.10 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.980, T_{max} = 0.992
6343 measured reflections
1276 independent reflections
1143 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.10
1276 reflections
192 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 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—H4A···O1^{i}	0.95 (5)	2.39 (6)	3.153 (4)	136 (4)
O1—H1···N1	0.82	1.87	2.598 (3)	148
O2—H2A···N2^{ii}	0.82	1.91	2.705 (3)	162

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NC2167).

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supporting information

Acta Cryst. (2010). E**66**, o330 [https://doi.org/10.1107/S1600536809055536]

2,2′-(4-Amino-4*H*-1,2,4-triazole-3,5-diyl)diphenol

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S1. Experimental

2-hydroxy-*N*-(2-hydroxybenzoyl)benzohydrazide and hydrazine monohydrate were purchased from Acros and used without further purification. 2-hydroxy-*N*-(2-hydroxybenzoyl)benzohydrazide (4.08 g, 0.15 mmol) and hydrazine monohydrate(7.27 ml, 0.15 mol) were transfered into a 50 ml round-bottom flask. The mixture was stirred and refluxed for 3 h. After cooling, the solution was poured into ice water and the resulting light pink precipitate was collected by filtration, and recrystallized from ethanol. colorless crystal, 1.812 g (46.6 %), ^{1}H NMR (DMSO, 400 MHz, p.p.m.): 11.27 (s, 2H), 8.0 (q, 2H), 7.4 (m, 2H), 7.0 (q, 4H), 6.14 (s, 2H). m.p = 532.3–533.2 K. Anal. Calcd for $C_{14}H_{12}O_2N_4$: C, 62.69; H, 4.48; N, 20.90%, Found: C, 62.58; H, 4.42; N, 20.83 %. GC—MS(m/*z*): 268 (M^+), 249, 221, 207, 117, 91, 77, 51, 32.

S2. Refinement

All C-H H atoms were placed in geometrically idealized positions (methyl H atoms allowed to rotate but not to tip) and constrained to ride on their parent atoms with C—H distances in the range of 0.93–0.98 Å, and with U_{iso}(H) = 1.2 U $_{eq}$ for aryl H atoms and 1.5 U_{eq} for the methyl H atoms. The O-H and N-H H atoms were located in difference map and were refined isotropic with varying coordinates. Because no strong anomalous scaterring atoms are present the absolute structure cannot be determined, Therefore, Friedel-opposites were merged in the refinement and the absolute structure was selected arbitrarily.

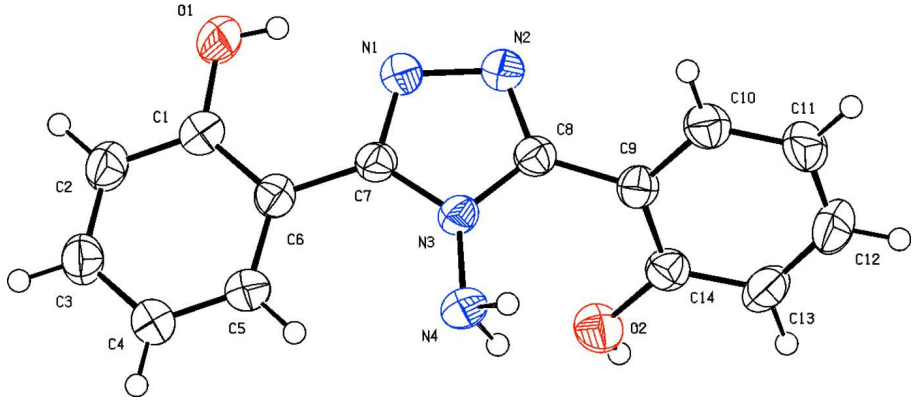

Figure 1

Crystal structure of the title compound showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

2,2′-(4-Amino-4*H*-1,2,4-triazole-3,5-diyl)diphenol

Crystal data

$C_{14}H_{12}N_4O_2$
M_r = 268.28
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
a = 8.262 (2) Å
b = 9.384 (3) Å
c = 15.919 (4) Å
V = 1234.2 (6) Å^3
Z = 4
F(000) = 560
D_x = 1.444 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2036 reflections
θ = 2.4–24.6°
μ = 0.10 mm^{-1}
T = 296 K
Block, colorless
0.10 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.980, T_{max} = 0.992
6343 measured reflections
1276 independent reflections
1143 reflections with $I > 2\sigma(I)$
R_{int} = 0.043
θ_{max} = 25.0°, θ_{min} = 2.5°
h = −9→9
k = −9→11
l = −18→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.112
S = 1.10
1276 reflections
192 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
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0693P)^2 + 0.078P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.005
$\Delta\rho_{max}$ = 0.18 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: 0.011 (4)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

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

	x	y	z	U_{iso}*/U_{eq}
C1	0.5404 (4)	0.5286 (4)	0.49447 (17)	0.0385 (7)
C2	0.4539 (4)	0.4330 (4)	0.54359 (19)	0.0449 (8)
H2	0.3416	0.4386	0.5450	0.054*

C3	0.5310 (4)	0.3309 (4)	0.5899 (2)	0.0457 (8)
H3	0.4711	0.2671	0.6220	0.055*
C4	0.6981 (4)	0.3225 (4)	0.5890 (2)	0.0479 (8)
H4	0.7509	0.2536	0.6208	0.057*
C5	0.7861 (4)	0.4166 (3)	0.5408 (2)	0.0442 (8)
H5	0.8985	0.4103	0.5406	0.053*
C6	0.7108 (4)	0.5211 (3)	0.49212 (17)	0.0339 (7)
C7	0.7987 (4)	0.6149 (3)	0.43411 (18)	0.0339 (7)
C8	0.9790 (3)	0.7037 (3)	0.34798 (17)	0.0333 (7)
C9	1.1311 (3)	0.7234 (3)	0.30083 (17)	0.0345 (7)
C10	1.1826 (4)	0.8612 (3)	0.2823 (2)	0.0422 (8)
H10	1.1286	0.9388	0.3055	0.051*
C11	1.3126 (4)	0.8838 (4)	0.2298 (2)	0.0521 (9)
H11	1.3482	0.9759	0.2186	0.062*
C12	1.3895 (4)	0.7686 (4)	0.1941 (2)	0.0586 (10)
H12	1.4744	0.7837	0.1568	0.070*
C13	1.3427 (4)	0.6316 (4)	0.2127 (2)	0.0482 (9)
H13	1.3981	0.5550	0.1894	0.058*
C14	1.2130 (4)	0.6075 (3)	0.26611 (18)	0.0371 (7)
N1	0.7263 (3)	0.7099 (3)	0.38646 (15)	0.0388 (6)
N2	0.8404 (3)	0.7657 (3)	0.33109 (15)	0.0395 (6)
N3	0.9594 (3)	0.6108 (3)	0.41328 (15)	0.0335 (6)
N4	1.0844 (3)	0.5384 (4)	0.45696 (19)	0.0427 (7)
O1	0.4530 (3)	0.6247 (3)	0.44960 (15)	0.0554 (7)
H1	0.5143	0.6734	0.4210	0.083*
O2	1.1615 (3)	0.4745 (2)	0.28606 (13)	0.0458 (6)
H2A	1.1785	0.4206	0.2465	0.069*
H4A	1.167 (7)	0.605 (5)	0.471 (3)	0.097 (16)*
H4B	1.131 (5)	0.486 (4)	0.419 (2)	0.059 (12)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0354 (17)	0.0409 (17)	0.0393 (15)	0.0028 (14)	−0.0007 (13)	−0.0023 (14)
C2	0.0329 (17)	0.052 (2)	0.0500 (17)	−0.0040 (15)	0.0028 (15)	0.0022 (16)
C3	0.0440 (19)	0.0430 (19)	0.0500 (18)	−0.0045 (16)	0.0092 (16)	0.0025 (15)
C4	0.0438 (18)	0.047 (2)	0.0531 (18)	0.0030 (16)	0.0074 (15)	0.0123 (16)
C5	0.0367 (17)	0.0430 (19)	0.0529 (18)	0.0052 (15)	0.0008 (14)	0.0087 (16)
C6	0.0321 (16)	0.0312 (16)	0.0385 (15)	−0.0029 (13)	0.0013 (12)	−0.0051 (13)
C7	0.0317 (15)	0.0291 (16)	0.0408 (15)	0.0000 (14)	0.0011 (12)	−0.0003 (13)
C8	0.0326 (15)	0.0280 (15)	0.0391 (14)	−0.0011 (13)	−0.0007 (12)	−0.0023 (13)
C9	0.0310 (15)	0.0344 (17)	0.0382 (15)	−0.0025 (13)	−0.0014 (12)	−0.0009 (13)
C10	0.0392 (17)	0.0343 (17)	0.0530 (18)	−0.0042 (14)	−0.0030 (15)	0.0003 (15)
C11	0.0391 (17)	0.046 (2)	0.071 (2)	−0.0101 (18)	−0.0009 (17)	0.0159 (18)
C12	0.0346 (18)	0.074 (3)	0.067 (2)	−0.0013 (19)	0.0118 (16)	0.013 (2)
C13	0.0326 (16)	0.057 (2)	0.0552 (19)	0.0087 (16)	0.0062 (15)	−0.0016 (18)
C14	0.0336 (15)	0.0377 (17)	0.0400 (15)	0.0003 (14)	−0.0047 (13)	0.0016 (14)
N1	0.0342 (13)	0.0348 (15)	0.0474 (13)	0.0007 (12)	0.0030 (11)	0.0056 (12)

N2	0.0368 (14)	0.0341 (14)	0.0477 (14)	0.0021 (11)	0.0044 (11)	0.0045 (12)
N3	0.0263 (12)	0.0335 (13)	0.0409 (12)	0.0003 (11)	0.0003 (10)	0.0009 (11)
N4	0.0313 (15)	0.0460 (17)	0.0507 (17)	0.0061 (13)	−0.0032 (12)	0.0065 (15)
O1	0.0302 (12)	0.0693 (18)	0.0666 (15)	0.0058 (12)	0.0032 (11)	0.0211 (14)
O2	0.0577 (15)	0.0305 (12)	0.0493 (12)	−0.0016 (11)	0.0076 (11)	−0.0071 (10)

Geometric parameters (Å, º)

C1—O1	1.358 (4)	C9—C10	1.393 (4)
C1—C2	1.388 (4)	C9—C14	1.395 (4)
C1—C6	1.410 (5)	C10—C11	1.377 (5)
C2—C3	1.366 (5)	C10—H10	0.9300
C2—H2	0.9300	C11—C12	1.376 (5)
C3—C4	1.383 (5)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.375 (5)
C4—C5	1.377 (5)	C12—H12	0.9300
C4—H4	0.9300	C13—C14	1.386 (4)
C5—C6	1.396 (4)	C13—H13	0.9300
C5—H5	0.9300	C14—O2	1.356 (4)
C6—C7	1.468 (4)	N1—N2	1.393 (3)
C7—N1	1.314 (4)	N3—N4	1.419 (3)
C7—N3	1.368 (4)	N4—H4A	0.95 (5)
C8—N2	1.312 (4)	N4—H4B	0.87 (4)
C8—N3	1.366 (4)	O1—H1	0.8200
C8—C9	1.475 (4)	O2—H2A	0.8200
O1—C1—C2	116.8 (3)	C14—C9—C8	121.2 (3)
O1—C1—C6	123.4 (3)	C11—C10—C9	120.6 (3)
C2—C1—C6	119.8 (3)	C11—C10—H10	119.7
C3—C2—C1	121.1 (3)	C9—C10—H10	119.7
C3—C2—H2	119.4	C12—C11—C10	119.3 (3)
C1—C2—H2	119.4	C12—C11—H11	120.3
C2—C3—C4	120.0 (3)	C10—C11—H11	120.3
C2—C3—H3	120.0	C13—C12—C11	121.0 (3)
C4—C3—H3	120.0	C13—C12—H12	119.5
C5—C4—C3	119.8 (3)	C11—C12—H12	119.5
C5—C4—H4	120.1	C12—C13—C14	120.1 (3)
C3—C4—H4	120.1	C12—C13—H13	119.9
C4—C5—C6	121.6 (3)	C14—C13—H13	119.9
C4—C5—H5	119.2	O2—C14—C13	122.4 (3)
C6—C5—H5	119.2	O2—C14—C9	118.2 (3)
C5—C6—C1	117.7 (3)	C13—C14—C9	119.4 (3)
C5—C6—C7	123.3 (3)	C7—N1—N2	108.2 (2)
C1—C6—C7	118.7 (3)	C8—N2—N1	107.1 (2)
N1—C7—N3	108.7 (2)	C8—N3—C7	106.4 (2)
N1—C7—C6	123.0 (3)	C8—N3—N4	126.3 (2)
N3—C7—C6	128.0 (3)	C7—N3—N4	126.9 (2)
N2—C8—N3	109.6 (3)	N3—N4—H4A	109 (3)

N2—C8—C9	125.7 (3)	N3—N4—H4B	105 (3)
N3—C8—C9	124.6 (3)	H4A—N4—H4B	103 (4)
C10—C9—C14	119.4 (3)	C1—O1—H1	109.5
C10—C9—C8	119.0 (3)	C14—O2—H2A	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N4—H4*A*···O1^{i}	0.95 (5)	2.39 (6)	3.153 (4)	136 (4)
O1—H1···N1	0.82	1.87	2.598 (3)	148
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