IUCrData

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2-((*E*)-{[3-(1*H*-Imidazol-1-yl)propyl]imino}methyl)-4-[(*E*)-(4-methylphenyl)diazenyl]phenol

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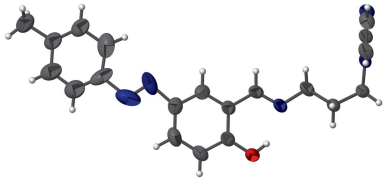
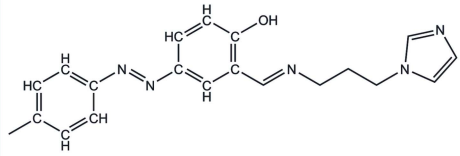
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{20}H_{21}N_5O$, the dihedral angles between the central phenol ring and pendant toluyl and imidazole rings are 3.20 (16) and 81.44 (14)°, respectively; the dihedral angle between the pendant rings is 84.39 (16)°. An intramolecular O—H···N hydrogen bond occurs. A Hirshfeld fingerprint analysis indicates that H···H contacts account for 48.6% of the surface.

3D view



Chemical scheme



Structure description

As a continuation of our studies of compounds based on azo Schiff base derivatives (Slassi *et al.*, 2017), we report herein the synthesis and structure of the title compound.

The molecular structure of the title compound is shown in Fig. 1. The bond lengths and angles of the imidazol moiety are comparable with those reported for similar compounds (Slassi *et al.*, 2017). The dihedral angles between the phenol ring and pendant toluyl and imidazole rings are 3.20 (16) and 81.44 (14)°, respectively; the dihedral angle between the pendant rings is 84.39 (16)°. The N1—N2 and N3—C14 bond lengths [1.174 (4) and 1.272 (4) Å, respectively] confirm their double-bond character, whereas the N4—C18 and N4—C19 values [1.347 (3) and 1.370 (3) Å, respectively] are shorter than (nominal) isolated C—N bonds (1.46 Å) due to conjugation. An intramolecular O—H···N hydrogen bond occurs (Table 1) but no directional interactions beyond van der Waals' contacts could be identified in the packing (Fig. 2).

Two-dimensional Hirshfeld fingerprint plots (McKinnon *et al.*, 2007) indicate that 48.6% of the surface is due to H···H contacts, followed by C···H, N···H and O···H interactions, which contribute 28.5, 15.2 and 6.1%, respectively.

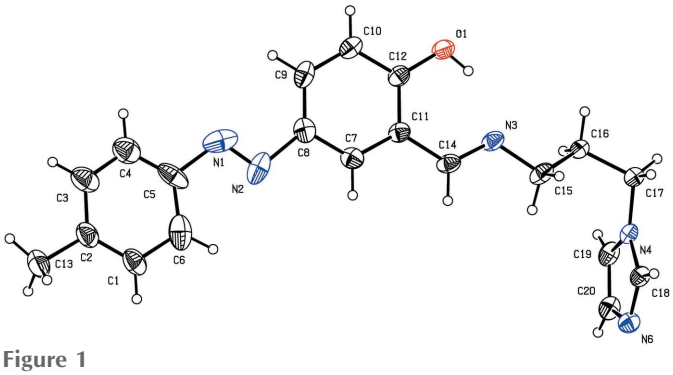


Figure 1
The molecular structure with displacement ellipsoids drawn at the 30% probability level.

Synthesis and crystallization

A diazonium-salt solution was prepared by dissolving *p*-toluidine amine (1.23 g, 0.01 mol) in a mixture of water and concentrated hydrochloric acid (8 and 3 ml, respectively). The resulting solution was cooled to 273 K, treated with aqueous (1.0 *M*) sodium nitrate (15 ml) dropwise and stirred for 15 min. Salicylaldehyde (2.2 g, 0.010 mol) was dissolved in 10% sodium hydroxide (50 ml). The diazonium solution was then added dropwise to initiate the coupling reaction. After the mixture had been stirred for 1 h at 273–278 K, the precipitate was filtered off. Crystals were obtained by recrystallization from ethanol solution. *N*-(3-Aminopropyl)imidazole (4.0 mmol) was added into a methanol solution (30 ml) of 2-hydroxy-5-(*p*-tolyldiazenyl)benzaldehyde (4.0 mmol) prepared in the first step of the reaction. The mixture was refluxed for 2 h and cooled to room temperature.

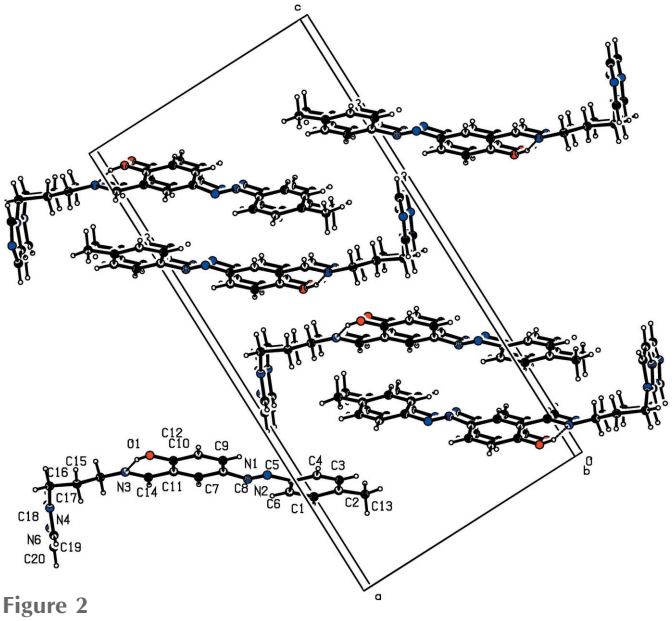


Figure 2
The packing viewed down [010].

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3	0.82	1.86	2.588 (3)	148

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{21}N_5O$
M_r	347.42
Crystal system, space group	Monoclinic, *P*2/*n*
Temperature (K)	293
a, *b*, *c* (Å)	12.2410 (4), 5.9386 (2), 25.2112 (9)
β (°)	90.317 (3)
V (Å^3)	1832.69 (11)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.65
Crystal size (mm)	0.35 × 0.22 × 0.13
Data collection	
Diffractometer	Agilent SuperNova CCD
Absorption correction	–
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5548, 3487, 3037
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.622
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.084, 0.245, 1.08
No. of reflections	3487
No. of parameters	236
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.02, −0.68

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

The solvent was removed on a rotatory evaporator and the orange product was rinsed and recrystallized from mixed solvents of methanol and ether to give orange crystals after one week.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

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full crystallographic data

IUCrData (2019). **4**, x190036 [https://doi.org/10.1107/S2414314619000361]

2-((*E*)-{[3-(1*H*-Imidazol-1-yl)propyl]imino}methyl)-4-[(*E*)-(4-methylphenyl)-diazenyl]phenol

Siham Slassi, Oleh Stetsiuk, Amina Amine, Mohammed Aarjane, Abdelkrim El-Ghayoury, Hafid Zouihri and Khalid Yamni

(I)

Crystal data

$C_{20}H_{21}N_5O$
M_r = 347.42
Monoclinic, $P2/n$
a = 12.2410 (4) Å
b = 5.9386 (2) Å
c = 25.2112 (9) Å
β = 90.317 (3)°
V = 1832.69 (11) Å^3
Z = 4
$F(000)$ = 736
D_x = 1.259 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 235 reflections
θ = 0.7–32.0°
μ = 0.65 mm^{-1}
T = 293 K
Prism, orange
0.35 × 0.22 × 0.13 mm

Data collection

Agilent SuperNova CCD diffractometer
Radiation source: fine-focus sealed tube
ω and φ scans
5548 measured reflections
3487 independent reflections
3037 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 73.5°, θ_{min} = 4.0°
h = −14→14
k = −4→7
l = −30→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.084
$wR(F^2)$ = 0.245
S = 1.08
3487 reflections
236 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1176P)^2 + 1.9097P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.02 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}
Extinction correction: SHELXL2014/7 (Sheldrick 2014, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0012 (6)

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. Hydrogen atoms were placed in calculated positions and treated as riding: (O—H = 0.82 Å, N—H = and C —H = 0.93 to 0.97 Å) and refined using a riding model, with U_{iso}(H) = 1.2Ueq(C) and 1.5Ueq(O).

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C13	0.8079 (4)	0.4904 (8)	0.15663 (17)	0.0903 (14)
H13A	0.7426	0.5753	0.1499	0.135*
H13B	0.8425	0.4547	0.1236	0.135*
H13C	0.7897	0.3537	0.1749	0.135*
C14	1.4876 (2)	1.0267 (5)	0.40967 (12)	0.0478 (7)
H14	1.5059	0.8854	0.3965	0.057*
C15	1.6512 (2)	1.0082 (5)	0.45962 (14)	0.0530 (8)
H15A	1.6768	0.9101	0.4315	0.064*
H15B	1.6357	0.9160	0.4904	0.064*
C16	1.7384 (2)	1.1766 (5)	0.47322 (12)	0.0463 (7)
H16A	1.7086	1.2866	0.4976	0.056*
H16B	1.7595	1.2556	0.4412	0.056*
C17	1.8392 (2)	1.0701 (5)	0.49814 (12)	0.0495 (7)
H17A	1.8915	1.1870	0.5070	0.059*
H17B	1.8189	0.9943	0.5307	0.059*
C18	1.8979 (2)	0.6838 (4)	0.46977 (10)	0.0405 (6)
H18	1.8738	0.6112	0.5002	0.049*
C19	1.9335 (2)	0.9502 (5)	0.41319 (11)	0.0476 (7)
H19	1.9400	1.0892	0.3965	0.057*
C20	1.9650 (2)	0.7472 (5)	0.39414 (11)	0.0501 (7)
H20	1.9972	0.7252	0.3612	0.060*
N3	1.55155 (17)	1.1238 (4)	0.44235 (11)	0.0484 (6)
N4	1.89023 (16)	0.9079 (4)	0.46217 (8)	0.0380 (5)
N6	1.9431 (2)	0.5788 (4)	0.42948 (9)	0.0468 (6)
O1	1.42088 (16)	1.4663 (3)	0.44393 (9)	0.0535 (6)
H1	1.4761	1.3929	0.4505	0.080*
C1	0.9844 (3)	0.5444 (6)	0.20574 (12)	0.0658 (9)
H2	1.0071	0.4026	0.1947	0.079*
C2	0.8845 (3)	0.6273 (6)	0.19032 (12)	0.0644 (9)
C3	0.8533 (4)	0.8425 (7)	0.20665 (14)	0.0763 (11)
H3	0.7858	0.8970	0.1954	0.092*
C4	0.9149 (4)	0.9741 (8)	0.23747 (15)	0.0763 (11)
H4	0.8913	1.1168	0.2474	0.092*
C5	1.0114 (4)	0.8946 (6)	0.25356 (12)	0.0703 (11)
C6	1.0528 (3)	0.6788 (8)	0.23901 (13)	0.0785 (12)
H6	1.1208	0.6285	0.2506	0.094*
C7	1.3171 (2)	1.0200 (5)	0.35781 (11)	0.0489 (7)
H7	1.3370	0.8779	0.3457	0.059*
C8	1.2196 (2)	1.1128 (6)	0.34087 (11)	0.0520 (7)
C9	1.1896 (2)	1.3256 (6)	0.35955 (12)	0.0556 (8)
H9	1.1237	1.3890	0.3486	0.067*
C10	1.2569 (2)	1.4428 (5)	0.39417 (12)	0.0511 (7)

H10	1.2358	1.5839	0.4065	0.061*
C11	1.3862 (2)	1.1332 (5)	0.39245 (11)	0.0433 (6)
C12	1.3566 (2)	1.3504 (5)	0.41079 (11)	0.0450 (6)
N1	1.0795 (3)	1.0496 (5)	0.28969 (13)	0.0807 (11)
N2	1.1576 (2)	0.9503 (6)	0.30336 (11)	0.0681 (9)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C13	0.116 (4)	0.081 (3)	0.074 (2)	0.007 (3)	−0.042 (2)	−0.015 (2)
C14	0.0355 (13)	0.0398 (14)	0.0683 (18)	0.0006 (11)	0.0102 (12)	−0.0050 (13)
C15	0.0353 (14)	0.0440 (15)	0.080 (2)	0.0054 (12)	0.0043 (13)	0.0064 (14)
C16	0.0420 (14)	0.0410 (14)	0.0557 (16)	0.0095 (11)	−0.0084 (12)	−0.0055 (12)
C17	0.0467 (15)	0.0449 (15)	0.0568 (16)	0.0099 (12)	−0.0092 (12)	−0.0075 (13)
C18	0.0407 (13)	0.0396 (13)	0.0411 (13)	0.0024 (10)	0.0025 (10)	0.0091 (10)
C19	0.0426 (14)	0.0500 (16)	0.0503 (15)	0.0007 (12)	0.0023 (11)	0.0206 (13)
C20	0.0462 (15)	0.0629 (18)	0.0412 (14)	0.0048 (13)	0.0065 (11)	0.0100 (13)
N3	0.0309 (11)	0.0414 (12)	0.0728 (15)	0.0034 (9)	0.0024 (10)	−0.0025 (11)
N4	0.0321 (10)	0.0372 (11)	0.0447 (11)	0.0030 (8)	−0.0030 (8)	0.0064 (9)
N6	0.0483 (13)	0.0435 (13)	0.0486 (13)	0.0037 (10)	0.0030 (10)	0.0032 (10)
O1	0.0425 (10)	0.0406 (11)	0.0772 (14)	0.0046 (8)	−0.0049 (9)	−0.0094 (10)
C1	0.091 (3)	0.067 (2)	0.0394 (15)	−0.0114 (19)	−0.0074 (15)	0.0012 (14)
C2	0.089 (2)	0.065 (2)	0.0395 (15)	−0.0021 (18)	−0.0112 (15)	−0.0008 (14)
C3	0.109 (3)	0.068 (2)	0.0517 (19)	−0.005 (2)	0.0041 (19)	−0.0053 (17)
C4	0.095 (3)	0.074 (2)	0.060 (2)	−0.004 (2)	0.005 (2)	−0.0018 (19)
C5	0.118 (3)	0.058 (2)	0.0353 (15)	−0.033 (2)	0.0192 (17)	−0.0083 (14)
C6	0.074 (2)	0.121 (3)	0.0412 (16)	−0.019 (2)	−0.0011 (15)	0.032 (2)
C7	0.0419 (14)	0.0543 (16)	0.0507 (15)	−0.0049 (12)	0.0113 (12)	−0.0046 (13)
C8	0.0432 (15)	0.0693 (19)	0.0436 (14)	−0.0089 (14)	0.0083 (11)	0.0039 (14)
C9	0.0393 (14)	0.077 (2)	0.0506 (16)	0.0053 (14)	0.0014 (12)	0.0181 (15)
C10	0.0449 (15)	0.0520 (16)	0.0566 (17)	0.0102 (13)	0.0075 (12)	0.0098 (13)
C11	0.0327 (12)	0.0440 (14)	0.0532 (15)	−0.0010 (10)	0.0075 (11)	−0.0006 (12)
C12	0.0370 (13)	0.0450 (15)	0.0531 (15)	0.0013 (11)	0.0055 (11)	0.0044 (12)
N1	0.106 (3)	0.0599 (18)	0.076 (2)	0.0242 (18)	0.0444 (19)	0.0206 (16)
N2	0.0388 (13)	0.099 (2)	0.0666 (17)	0.0065 (14)	0.0089 (12)	0.0411 (16)

Geometric parameters (Å, º)

C13—C2	1.501 (5)	O1—C12	1.336 (3)
C13—H13A	0.9600	O1—H1	0.8200
C13—H13B	0.9600	C1—C2	1.372 (5)
C13—H13C	0.9600	C1—C6	1.427 (5)
C14—N3	1.272 (4)	C1—H2	0.9300
C14—C11	1.457 (4)	C2—C3	1.397 (5)
C14—H14	0.9300	C3—C4	1.333 (6)
C15—N3	1.464 (3)	C3—H3	0.9300
C15—C16	1.500 (4)	C4—C5	1.333 (6)
C15—H15A	0.9700	C4—H4	0.9300

C15—H15B	0.9700	C5—C6	1.426 (6)
C16—C17	1.520 (4)	C5—N1	1.537 (5)
C16—H16A	0.9700	C6—H6	0.9300
C16—H16B	0.9700	C7—C8	1.381 (4)
C17—N4	1.465 (3)	C7—C11	1.386 (4)
C17—H17A	0.9700	C7—H7	0.9300
C17—H17B	0.9700	C8—C9	1.399 (5)
C18—N6	1.317 (3)	C8—N2	1.547 (5)
C18—N4	1.347 (3)	C9—C10	1.385 (5)
C18—H18	0.9300	C9—H9	0.9300
C19—C20	1.354 (4)	C10—C12	1.401 (4)
C19—N4	1.370 (3)	C10—H10	0.9300
C19—H19	0.9300	C11—C12	1.417 (4)
C20—N6	1.367 (4)	N1—N2	1.174 (4)
C20—H20	0.9300		

C2—C13—H13A	109.5	C18—N6—C20	103.9 (2)
C2—C13—H13B	109.5	C12—O1—H1	109.5
H13A—C13—H13B	109.5	C2—C1—C6	119.0 (4)
C2—C13—H13C	109.5	C2—C1—H2	120.5
H13A—C13—H13C	109.5	C6—C1—H2	120.5
H13B—C13—H13C	109.5	C1—C2—C3	119.3 (4)
N3—C14—C11	121.1 (3)	C1—C2—C13	121.2 (4)
N3—C14—H14	119.5	C3—C2—C13	119.4 (4)
C11—C14—H14	119.5	C4—C3—C2	123.6 (4)
N3—C15—C16	110.2 (2)	C4—C3—H3	118.2
N3—C15—H15A	109.6	C2—C3—H3	118.2
C16—C15—H15A	109.6	C3—C4—C5	117.8 (4)
N3—C15—H15B	109.6	C3—C4—H4	121.1
C16—C15—H15B	109.6	C5—C4—H4	121.1
H15A—C15—H15B	108.1	C4—C5—C6	123.7 (4)
C15—C16—C17	113.1 (2)	C4—C5—N1	116.4 (4)
C15—C16—H16A	109.0	C6—C5—N1	119.9 (4)
C17—C16—H16A	109.0	C5—C6—C1	116.5 (4)
C15—C16—H16B	109.0	C5—C6—H6	121.8
C17—C16—H16B	109.0	C1—C6—H6	121.8
H16A—C16—H16B	107.8	C8—C7—C11	121.6 (3)
N4—C17—C16	111.5 (2)	C8—C7—H7	119.2
N4—C17—H17A	109.3	C11—C7—H7	119.2
C16—C17—H17A	109.3	C7—C8—C9	119.0 (3)
N4—C17—H17B	109.3	C7—C8—N2	111.1 (3)
C16—C17—H17B	109.3	C9—C8—N2	129.8 (3)
H17A—C17—H17B	108.0	C10—C9—C8	120.7 (3)
N6—C18—N4	112.8 (2)	C10—C9—H9	119.7
N6—C18—H18	123.6	C8—C9—H9	119.7
N4—C18—H18	123.6	C9—C10—C12	120.4 (3)
C20—C19—N4	105.6 (2)	C9—C10—H10	119.8
C20—C19—H19	127.2	C12—C10—H10	119.8

N4—C19—H19	127.2	C7—C11—C12	119.4 (3)
C19—C20—N6	111.3 (2)	C7—C11—C14	119.5 (3)
C19—C20—H20	124.4	C12—C11—C14	121.1 (3)
N6—C20—H20	124.4	O1—C12—C10	119.6 (3)
C14—N3—C15	119.3 (3)	O1—C12—C11	121.5 (2)
C18—N4—C19	106.4 (2)	C10—C12—C11	118.9 (3)
C18—N4—C17	126.2 (2)	N2—N1—C5	108.1 (3)
C19—N4—C17	127.3 (2)	N1—N2—C8	105.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.82	1.86	2.588 (3)	148