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

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Keywords: crystal structure; imidazole; phenol; intramolecular hydrogen bond.

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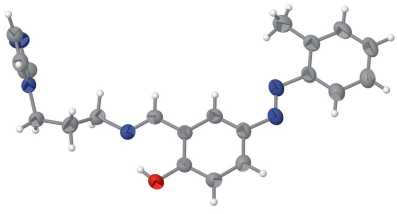
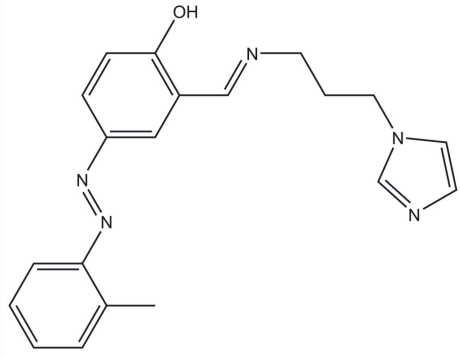
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{20}H_{21}N_5O$, an intramolecular O—H···N hydrogen bond forms between the –OH substituent of the phenol ring and the adjacent iminomethyl N atom, enclosing an *S*6 ring. The dihedral angles between the imidazole ring and the methylphenyl and phenol rings are 86.93 (14) and 88.00 (13)°, respectively, while that between the methylphenyl and phenol rings is 2.18 (12)°.

3D view



Chemical scheme



Structure description

In the title compound (Fig. 1), an *S*6 ring motif is formed by an intramolecular O1—H1···N3 hydrogen bond (Table 1). The dihedral angles between the imidazole ring and the methylphenyl and phenol rings are 86.93 (14) and 88.00 (13)°, respectively. In contrast, the methylphenyldiazenylphenol segment of the molecule is almost planar, with a dihedral angle of 2.18 (12)° between the C1–C6 and C7–C12 benzene rings.

Synthesis and crystallization

A diazonium salt solution was prepared by dissolving *o*-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, 0.01 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. *N*-(3-Aminopropyl)imidazole (0.5 g, 4 mmol) was next added to an ethanol solution (30 ml) of 2-hydroxy-5-(*o*-tolyldiazenyl)benzaldehyde (0.96 g, 4 mmol). The mixture was refluxed for 2 h and cooled to room temperature. The solvent was removed under

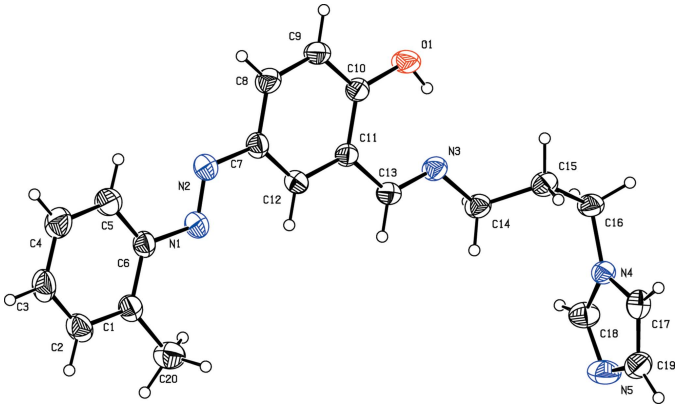


Figure 1
The structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres with arbitrary radii.

vacuum. The final product obtained after extraction was recrystallized from a mixture of ethanol and diethyl ether, the solution being allowed to evaporate slowly at a constant ambient temperature to give coulourless good-quality crystals after 3 d.

Refinement

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

References

Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N3	0.82	1.80	2.534 (2)	148

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{21}N_5O$
M_r	347.42
Crystal system, space group	Orthorhombic, $P2_12_12$
Temperature (K)	293
a, b, c (Å)	9.7570 (3), 32.0691 (13), 5.8643 (2)
V (Å^3)	1834.93 (11)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.25 × 0.15 × 0.12
Data collection	
Diffractometer	Bruker APEXII CCD detector
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	25352, 3590, 2978
R_{int}	0.026
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.617
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.037, 0.105, 1.04
No. of reflections	3590
No. of parameters	238
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.16
Absolute structure	Flack x determined using 1099 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.4 (4)

Computer programs: *APEX2* and *SAINT* (Bruker, 2005), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

full crystallographic data

IUCrData (2017). **2**, x171477 [https://doi.org/10.1107/S2414314617014778]

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

Crystal data

$C_{20}H_{21}N_5O$
M_r = 347.42
Orthorhombic, $P2_12_12$
a = 9.7570 (3) Å
b = 32.0691 (13) Å
c = 5.8643 (2) Å
V = 1834.93 (11) Å^3
Z = 4
F(000) = 736
D_x = 1.258 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 245 reflections
θ = 0.2–52°
μ = 0.08 mm^{-1}
T = 293 K
Prism, colourless
0.25 × 0.15 × 0.12 mm

Data collection

Bruker APEXII CCD detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
25352 measured reflections
3590 independent reflections
2978 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 26.0°, θ_{min} = 1.3°
h = −12→11
k = −39→39
l = −7→7

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.105
S = 1.04
3590 reflections
238 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0669P)^2 + 0.0698P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Extinction correction: SHELXL2014 (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.011 (3)
Absolute structure: Flack x determined using 1099 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.4 (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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C12	0.3769 (2)	0.07393 (7)	0.3773 (4)	0.0467 (5)
H12	0.4141	0.0641	0.2415	0.056*
O1	0.41943 (18)	0.15905 (5)	0.7861 (3)	0.0678 (5)
H1	0.4841	0.1685	0.7134	0.102*
C11	0.4278 (2)	0.11065 (6)	0.4741 (4)	0.0418 (5)
C13	0.5404 (2)	0.13322 (7)	0.3685 (4)	0.0469 (5)
H13	0.5787	0.1233	0.2339	0.056*
N1	0.26508 (19)	−0.00103 (6)	0.2286 (3)	0.0531 (5)
N3	0.58696 (18)	0.16637 (5)	0.4584 (3)	0.0494 (5)
C8	0.2161 (2)	0.06705 (7)	0.6845 (4)	0.0555 (6)
H8	0.1450	0.0525	0.7539	0.067*
N2	0.2132 (2)	0.01415 (6)	0.4030 (3)	0.0541 (5)
C6	0.2023 (2)	−0.03952 (7)	0.1584 (4)	0.0489 (5)
C14	0.7004 (2)	0.18966 (7)	0.3569 (4)	0.0503 (6)
H14A	0.6653	0.2138	0.2761	0.060*
H14B	0.7487	0.1722	0.2484	0.060*
C9	0.2645 (2)	0.10291 (7)	0.7834 (4)	0.0563 (6)
H9	0.2260	0.1125	0.9186	0.068*
C7	0.2719 (2)	0.05208 (7)	0.4815 (4)	0.0471 (5)
C1	0.2536 (2)	−0.05815 (7)	−0.0377 (4)	0.0507 (6)
C5	0.0958 (3)	−0.05797 (8)	0.2794 (5)	0.0662 (7)
H5	0.0629	−0.0454	0.4115	0.079*
N4	0.99745 (18)	0.22455 (5)	0.3059 (3)	0.0441 (4)
C10	0.3715 (2)	0.12496 (6)	0.6814 (4)	0.0478 (5)
C17	1.1092 (2)	0.20035 (7)	0.3481 (4)	0.0542 (6)
H17	1.1344	0.1885	0.4866	0.065*
C20	0.3712 (3)	−0.03963 (9)	−0.1689 (5)	0.0686 (7)
H20A	0.3643	−0.0098	−0.1669	0.103*
H20B	0.3684	−0.0494	−0.3236	0.103*
H20C	0.4562	−0.0480	−0.0998	0.103*
C3	0.0873 (3)	−0.11327 (8)	0.0100 (6)	0.0672 (7)
H3	0.0485	−0.1380	−0.0415	0.081*
C16	0.8941 (2)	0.23810 (7)	0.4694 (4)	0.0552 (6)
H16A	0.8410	0.2605	0.4022	0.066*
H16B	0.9397	0.2491	0.6034	0.066*
C19	1.1760 (3)	0.19709 (8)	0.1467 (5)	0.0622 (7)
H19	1.2566	0.1822	0.1248	0.075*
C18	1.0022 (3)	0.23486 (8)	0.0844 (4)	0.0596 (6)
H18	0.9374	0.2516	0.0123	0.072*

C15	0.7975 (2)	0.20352 (7)	0.5421 (4)	0.0538 (6)
H15A	0.8512	0.1797	0.5914	0.065*
H15B	0.7443	0.2131	0.6718	0.065*
N5	1.1094 (2)	0.21854 (8)	−0.0191 (4)	0.0712 (6)
C4	0.0388 (3)	−0.09479 (8)	0.2045 (6)	0.0714 (8)
H4	−0.0325	−0.1071	0.2857	0.086*
C2	0.1933 (3)	−0.09525 (7)	−0.1093 (5)	0.0618 (7)
H2	0.2254	−0.1082	−0.2408	0.074*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C12	0.0489 (11)	0.0477 (11)	0.0437 (11)	0.0006 (10)	−0.0008 (10)	−0.0074 (10)
O1	0.0660 (11)	0.0584 (10)	0.0792 (12)	−0.0179 (8)	0.0269 (10)	−0.0315 (9)
C11	0.0404 (11)	0.0399 (10)	0.0451 (11)	0.0019 (8)	0.0003 (9)	−0.0026 (9)
C13	0.0470 (12)	0.0482 (11)	0.0456 (11)	0.0012 (10)	0.0048 (10)	−0.0057 (10)
N1	0.0543 (11)	0.0472 (10)	0.0579 (11)	−0.0060 (9)	−0.0022 (9)	−0.0101 (9)
N3	0.0454 (10)	0.0446 (10)	0.0581 (11)	−0.0036 (8)	0.0082 (9)	−0.0046 (8)
C8	0.0496 (12)	0.0528 (13)	0.0641 (15)	−0.0096 (11)	0.0106 (13)	−0.0070 (11)
N2	0.0536 (10)	0.0492 (11)	0.0595 (12)	−0.0063 (9)	−0.0010 (10)	−0.0111 (9)
C6	0.0450 (11)	0.0439 (11)	0.0577 (14)	−0.0012 (10)	−0.0072 (11)	−0.0080 (10)
C14	0.0494 (12)	0.0492 (12)	0.0524 (13)	−0.0049 (10)	0.0098 (11)	−0.0001 (10)
C9	0.0542 (14)	0.0571 (14)	0.0577 (14)	−0.0081 (11)	0.0152 (12)	−0.0164 (11)
C7	0.0454 (11)	0.0441 (11)	0.0517 (12)	−0.0043 (10)	−0.0044 (11)	−0.0041 (10)
C1	0.0504 (12)	0.0459 (11)	0.0557 (13)	0.0054 (10)	−0.0091 (11)	−0.0050 (10)
C5	0.0625 (14)	0.0585 (14)	0.0775 (18)	−0.0102 (12)	0.0078 (15)	−0.0180 (13)
N4	0.0449 (9)	0.0440 (9)	0.0435 (9)	−0.0058 (8)	0.0065 (9)	−0.0005 (8)
C10	0.0427 (11)	0.0439 (12)	0.0568 (13)	−0.0009 (9)	0.0031 (11)	−0.0103 (10)
C17	0.0508 (12)	0.0512 (12)	0.0606 (14)	−0.0055 (11)	−0.0064 (12)	0.0033 (11)
C20	0.0768 (17)	0.0621 (15)	0.0668 (16)	0.0011 (13)	0.0108 (15)	−0.0055 (13)
C3	0.0579 (14)	0.0482 (13)	0.096 (2)	−0.0028 (12)	−0.0175 (16)	−0.0191 (14)
C16	0.0547 (13)	0.0569 (14)	0.0539 (13)	−0.0120 (11)	0.0166 (12)	−0.0108 (11)
C19	0.0528 (13)	0.0512 (13)	0.0827 (19)	−0.0042 (11)	0.0115 (14)	−0.0136 (13)
C18	0.0678 (15)	0.0632 (15)	0.0480 (13)	0.0051 (13)	0.0092 (13)	0.0098 (11)
C15	0.0531 (12)	0.0587 (14)	0.0496 (13)	−0.0088 (11)	0.0104 (11)	0.0029 (11)
N5	0.0828 (15)	0.0772 (14)	0.0535 (12)	−0.0035 (13)	0.0235 (13)	−0.0049 (11)
C4	0.0605 (15)	0.0572 (15)	0.097 (2)	−0.0141 (12)	0.0049 (15)	−0.0138 (15)
C2	0.0670 (15)	0.0505 (13)	0.0679 (17)	0.0053 (12)	−0.0091 (14)	−0.0165 (12)

Geometric parameters (Å, º)

C12—C7	1.383 (3)	C5—C4	1.377 (3)
C12—C11	1.398 (3)	C5—H5	0.9300
C12—H12	0.9300	N4—C18	1.341 (3)
O1—C10	1.338 (3)	N4—C17	1.361 (3)
O1—H1	0.8200	N4—C16	1.458 (3)
C11—C10	1.410 (3)	C17—C19	1.353 (4)
C11—C13	1.454 (3)	C17—H17	0.9300

C13—N3	1.271 (3)	C20—H20A	0.9600
C13—H13	0.9300	C20—H20B	0.9600
N1—N2	1.240 (3)	C20—H20C	0.9600
N1—C6	1.439 (3)	C3—C4	1.370 (4)
N3—C14	1.462 (3)	C3—C2	1.376 (4)
C8—C9	1.372 (3)	C3—H3	0.9300
C8—C7	1.395 (3)	C16—C15	1.517 (3)
C8—H8	0.9300	C16—H16A	0.9700
N2—C7	1.421 (3)	C16—H16B	0.9700
C6—C1	1.389 (3)	C19—N5	1.357 (4)
C6—C5	1.390 (3)	C19—H19	0.9300
C14—C15	1.508 (3)	C18—N5	1.317 (3)
C14—H14A	0.9700	C18—H18	0.9300
C14—H14B	0.9700	C15—H15A	0.9700
C9—C10	1.396 (3)	C15—H15B	0.9700
C9—H9	0.9300	C4—H4	0.9300
C1—C2	1.392 (3)	C2—H2	0.9300
C1—C20	1.504 (4)		

C7—C12—C11	120.7 (2)	O1—C10—C9	118.6 (2)
C7—C12—H12	119.7	O1—C10—C11	121.68 (19)
C11—C12—H12	119.7	C9—C10—C11	119.74 (19)
C10—O1—H1	109.5	C19—C17—N4	105.7 (2)
C12—C11—C10	119.04 (19)	C19—C17—H17	127.1
C12—C11—C13	120.97 (19)	N4—C17—H17	127.1
C10—C11—C13	119.95 (19)	C1—C20—H20A	109.5
N3—C13—C11	120.7 (2)	C1—C20—H20B	109.5
N3—C13—H13	119.7	H20A—C20—H20B	109.5
C11—C13—H13	119.7	C1—C20—H20C	109.5
N2—N1—C6	113.5 (2)	H20A—C20—H20C	109.5
C13—N3—C14	121.94 (19)	H20B—C20—H20C	109.5
C9—C8—C7	121.0 (2)	C4—C3—C2	120.1 (2)
C9—C8—H8	119.5	C4—C3—H3	120.0
C7—C8—H8	119.5	C2—C3—H3	120.0
N1—N2—C7	115.98 (19)	N4—C16—C15	113.40 (19)
C1—C6—C5	120.6 (2)	N4—C16—H16A	108.9
C1—C6—N1	116.9 (2)	C15—C16—H16A	108.9
C5—C6—N1	122.5 (2)	N4—C16—H16B	108.9
N3—C14—C15	109.41 (19)	C15—C16—H16B	108.9
N3—C14—H14A	109.8	H16A—C16—H16B	107.7
C15—C14—H14A	109.8	C17—C19—N5	110.8 (2)
N3—C14—H14B	109.8	C17—C19—H19	124.6
C15—C14—H14B	109.8	N5—C19—H19	124.6
H14A—C14—H14B	108.2	N5—C18—N4	112.1 (3)
C8—C9—C10	120.1 (2)	N5—C18—H18	124.0
C8—C9—H9	120.0	N4—C18—H18	124.0
C10—C9—H9	120.0	C14—C15—C16	113.8 (2)
C12—C7—C8	119.5 (2)	C14—C15—H15A	108.8

C12—C7—N2	126.1 (2)	C16—C15—H15A	108.8
C8—C7—N2	114.4 (2)	C14—C15—H15B	108.8
C6—C1—C2	117.7 (2)	C16—C15—H15B	108.8
C6—C1—C20	121.9 (2)	H15A—C15—H15B	107.7
C2—C1—C20	120.4 (2)	C18—N5—C19	104.6 (2)
C4—C5—C6	120.2 (3)	C3—C4—C5	119.8 (3)
C4—C5—H5	119.9	C3—C4—H4	120.1
C6—C5—H5	119.9	C5—C4—H4	120.1
C18—N4—C17	106.8 (2)	C3—C2—C1	121.6 (2)
C18—N4—C16	126.0 (2)	C3—C2—H2	119.2
C17—N4—C16	127.2 (2)	C1—C2—H2	119.2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.82	1.80	2.534 (2)	148