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(*E*)-2-Methyl-4-[(4-nitrophenyl)diazenyl]-phenol dimethylformamide solvate

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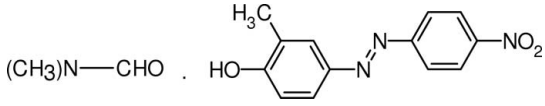
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.142; data-to-parameter ratio = 13.2.

The title compound, $C_{13}H_{11}N_3O_3{\cdot}C_3H_7NO$, displays an intermolecular O—H···O hydrogen bond between the hydroxyl group and the carbonyl O atom of the solvent molecule. The configuration of the N=N double bond is *trans* and the dihedral angle between the two aromatic rings is 24.85 (6)°.

Related literature

For related compounds, see: Koşar *et al.* (2004*a*,*b*).



Experimental

Crystal data

$C_{13}H_{11}N_3O_3{\cdot}C_3H_7NO$
M_r = 330.34
Orthorhombic, *Pbca*
a = 7.1951 (6) Å
b = 11.4501 (7) Å
c = 40.252 (2) Å
V = 3316.1 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.50 × 0.40 × 0.09 mm

Data collection

STOE IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.473, T_{max} = 0.877
28423 measured reflections
2925 independent reflections
1177 reflections with $I > 2\sigma(I)$
R_{int} = 0.134

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.143
S = 0.84
2925 reflections
221 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O4	0.82	1.84	2.595 (3)	153

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2612).

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Koşar, B., Albayrak, Ç., Odabaşoğlu, M. & Büyükgüngör, O. (2004*b*). *Acta Cryst.* E**60**, o246–o247.
Sheldrick, G. M. (1997). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
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  doi:10.1107/S1600536807059454

supplementary materials

Acta Cryst. (2007). E**63**, o4772 [**doi:10.1107/S1600536807059454**]

(*E*)-2-Methyl-4-[(4-nitrophenyl)diazenyl]phenol dimethylformamide solvate

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Comment

Azo compounds are used as dyes in textile, printing, paper manufacturing, pharmaceutial and food industries. All of them contain at least one azo group, which links two sp^2-hybridized C atoms. In our ongoing work, these C atoms are part of aromatic systems.

Experimental

A mixture of 4-nitroaniline (4.47 g, 32.4 mmol), water (50 ml) and concentrated hydrochloric acid (8.14 ml, 97.2 mmol) was stirred until a clear solution was obtained. This solution was cooled to 273–278 K and a solution of sodium nitrite (3,13 g, 45.36 mmol) in water was added dropwise while the temperature was maintained below 278 K. The resulting mixture was stirred for 30 min in an ice bath. An *o*-Cresol (3.5 g, 32.4 mmol) solution (pH 9) was gradually added to a cooled solution of 4-nitrobenzenediazonium chloride, prepared as described above, and the resulting mixture was stirred at 273–278 K for 60 min in ice bath. The product was recrystallized from ethyl alcohol to obtain solid (*E*)-2-methyl-4-[(4-nitrophenyl)diazenyl]phenol. Crystals were obtained by slow evaporation from DMF (yield %75, m.p. 480–482 K).

Refinement

All H atoms were positioned geometrically with C—H ranging from 0.93 to 0.96Å and O—H = 0.82Å and U(H)=1.2U_{eq}(C,*O*) and refined using a riding model.

Figures

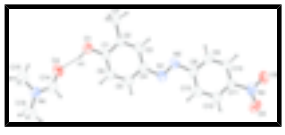


Fig. 1. A view of the molecule, with atom labels and 30% probability displacement ellipsoids for non-H atoms.

(*E*)-2-Methyl-4-[(4-nitrophenyl)diazenyl]phenol dimethylformamide solvate

Crystal data

$C_{13}H_{11}N_3O_3·C_3H_7NO$	D_x = 1.323 Mg m^{-3}
M_r = 330.34	Mo *K*α radiation λ = 0.71073 Å
Orthorhombic, *Pbca*	Cell parameters from 1369 reflections
a = 7.1951 (6) Å	θ = 1.8–24.9º
b = 11.4501 (7) Å	μ = 0.10 mm^{-1}
c = 40.252 (2) Å	*T* = 293 (2) K

V = 3316.1 (4) Å^3	Plate, red
Z = 8	0.50 × 0.40 × 0.09 mm
F_{000} = 1392	

Data collection

STOE IPDS 2 diffractometer	2925 independent reflections
Radiation source: fine-focus sealed tube	1177 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.134
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 25.1º
T = 293(2) K	θ_{min} = 3.0º
ω scan	h = −8→8
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	k = −13→13
T_{min} = 0.473, T_{max} = 0.877	l = −45→47
28423 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.052	H-atom parameters constrained
$wR(F^2)$ = 0.143	$w = 1/[\sigma^2(F_o^2) + (0.0656P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.84	$(\Delta/\sigma)_{max} < 0.001$
2925 reflections	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
221 parameters	$\Delta\rho_{min}$ = −0.14 e Å^{-3}
2 restraints	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0035 (5)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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	0.5794 (4)	0.5894 (3)	0.35445 (8)	0.0724 (9)
C2	0.5428 (4)	0.7036 (2)	0.36521 (8)	0.0720 (8)
C3	0.5668 (4)	0.7278 (3)	0.39844 (8)	0.0739 (9)
H3	0.5447	0.8031	0.4061	0.089*
C4	0.6239 (4)	0.6415 (3)	0.42095 (8)	0.0724 (8)
C5	0.6587 (4)	0.5303 (3)	0.40948 (8)	0.0795 (9)
H5	0.6969	0.4726	0.4242	0.095*
C6	0.6374 (5)	0.5044 (3)	0.37665 (8)	0.0794 (9)
H6	0.6619	0.4292	0.3691	0.095*
C7	0.4750 (5)	0.7943 (2)	0.34091 (8)	0.0906 (11)
H7A	0.3609	0.7683	0.3310	0.136*
H7B	0.5668	0.8057	0.3239	0.136*
H7C	0.4540	0.8666	0.3524	0.136*
C8	0.6317 (4)	0.7776 (3)	0.49993 (8)	0.0721 (8)
C9	0.6518 (5)	0.8906 (3)	0.51015 (8)	0.0849 (10)
H9	0.6685	0.9491	0.4944	0.102*
C10	0.6479 (5)	0.9190 (3)	0.54313 (8)	0.0800 (9)
H10	0.6615	0.9963	0.5498	0.096*
C11	0.6239 (4)	0.8322 (3)	0.56618 (7)	0.0700 (8)
C12	0.6051 (4)	0.7175 (3)	0.55705 (9)	0.0807 (9)
H12	0.5899	0.6594	0.5730	0.097*
C13	0.6093 (4)	0.6899 (3)	0.52346 (9)	0.0798 (9)
H13	0.5972	0.6126	0.5167	0.096*
C14	0.5340 (6)	0.2559 (4)	0.30409 (9)	0.0985 (11)
H14	0.6045	0.2312	0.3221	0.118*
C15	0.3192 (7)	0.2085 (5)	0.26042 (11)	0.1502 (18)
H15A	0.3223	0.2916	0.2573	0.225*
H15B	0.1943	0.1843	0.2652	0.225*
H15C	0.3615	0.1705	0.2405	0.225*
C16	0.4454 (7)	0.0558 (3)	0.29793 (13)	0.1434 (17)
H16A	0.5206	0.0489	0.3175	0.215*
H16B	0.4983	0.0095	0.2804	0.215*
H16C	0.3219	0.0287	0.3026	0.215*
N1	0.6423 (4)	0.6580 (2)	0.45645 (6)	0.0793 (7)
N2	0.6258 (4)	0.7616 (2)	0.46413 (6)	0.0795 (8)
N3	0.6146 (4)	0.8638 (3)	0.60128 (8)	0.0869 (8)
N4	0.4384 (4)	0.1770 (2)	0.28767 (7)	0.0891 (8)
O1	0.5537 (3)	0.56890 (17)	0.32164 (5)	0.0928 (7)
H1	0.5784	0.5005	0.3175	0.139*
O2	0.6458 (4)	0.9649 (2)	0.60921 (6)	0.1128 (9)
O3	0.5750 (4)	0.7869 (2)	0.62114 (6)	0.1149 (9)
O4	0.5362 (5)	0.3596 (2)	0.29714 (7)	0.1331 (11)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.074 (2)	0.080 (2)	0.063 (2)	−0.0053 (16)	0.0035 (16)	−0.0091 (16)
C2	0.075 (2)	0.0710 (18)	0.070 (2)	−0.0002 (15)	0.0017 (17)	−0.0048 (16)
C3	0.067 (2)	0.0716 (18)	0.083 (3)	−0.0058 (16)	0.0047 (17)	−0.0120 (17)
C4	0.061 (2)	0.0793 (19)	0.0768 (15)	−0.0057 (16)	0.0004 (17)	0.0016 (17)
C5	0.084 (2)	0.076 (2)	0.079 (3)	0.0004 (16)	0.0020 (19)	0.0042 (17)
C6	0.090 (2)	0.0761 (19)	0.072 (2)	−0.0020 (18)	0.0029 (19)	−0.0085 (18)
C7	0.115 (3)	0.0817 (19)	0.076 (2)	0.0060 (19)	−0.007 (2)	0.0028 (17)
C8	0.064 (2)	0.080 (2)	0.0721 (14)	0.0041 (16)	−0.0040 (17)	0.0034 (17)
C9	0.097 (3)	0.082 (2)	0.076 (3)	0.0024 (19)	−0.0035 (19)	0.0017 (17)
C10	0.094 (3)	0.0737 (19)	0.072 (2)	−0.0006 (17)	0.000 (2)	−0.0055 (17)
C11	0.069 (2)	0.082 (2)	0.059 (2)	0.0024 (16)	0.0004 (16)	−0.0065 (17)
C12	0.082 (3)	0.084 (2)	0.077 (3)	0.0038 (18)	−0.0007 (18)	0.0031 (17)
C13	0.079 (3)	0.0688 (19)	0.092 (3)	0.0039 (16)	−0.0053 (19)	−0.0145 (19)
C14	0.114 (3)	0.109 (3)	0.072 (3)	0.002 (3)	0.007 (2)	−0.004 (2)
C15	0.142 (4)	0.208 (5)	0.101 (4)	0.022 (4)	−0.030 (3)	−0.013 (3)
C16	0.164 (5)	0.088 (3)	0.178 (5)	−0.016 (3)	0.020 (4)	0.008 (3)
N1	0.0747 (18)	0.0811 (17)	0.0820 (15)	−0.0049 (14)	−0.0012 (15)	−0.0062 (14)
N2	0.089 (2)	0.0741 (16)	0.0752 (14)	0.0021 (14)	0.0016 (15)	−0.0015 (13)
N3	0.074 (2)	0.111 (2)	0.076 (2)	0.0067 (17)	0.0012 (16)	−0.0116 (19)
N4	0.095 (2)	0.0920 (19)	0.080 (2)	0.0020 (17)	−0.0018 (17)	−0.0043 (16)
O1	0.125 (2)	0.0827 (13)	0.0706 (16)	0.0053 (13)	−0.0023 (13)	−0.0112 (11)
O2	0.140 (2)	0.1082 (18)	0.0901 (19)	0.0084 (16)	−0.0070 (16)	−0.0325 (14)
O3	0.135 (2)	0.140 (2)	0.0699 (17)	−0.0055 (18)	0.0115 (15)	0.0098 (15)
O4	0.201 (3)	0.0948 (17)	0.104 (2)	−0.0107 (18)	0.011 (2)	−0.0246 (15)

Geometric parameters (Å, °)

C1—O1	1.354 (3)	C10—H10	0.9300
C1—C6	1.386 (4)	C11—C12	1.370 (4)
C1—C2	1.402 (4)	C11—N3	1.460 (4)
C2—C3	1.377 (4)	C12—C13	1.389 (4)
C2—C7	1.508 (4)	C12—H12	0.9300
C3—C4	1.402 (4)	C13—H13	0.9300
C3—H3	0.9300	C14—O4	1.219 (4)
C4—C5	1.377 (4)	C14—N4	1.314 (4)
C4—N1	1.447 (4)	C14—H14	0.9300
C5—C6	1.363 (4)	C15—N4	1.438 (5)
C5—H5	0.9300	C15—H15A	0.9600
C6—H6	0.9300	C15—H15B	0.9600
C7—H7A	0.9600	C15—H15C	0.9600
C7—H7B	0.9600	C16—N4	1.449 (4)
C7—H7C	0.9600	C16—H16A	0.9600
C8—C9	1.365 (4)	C16—H16B	0.9600
C8—C13	1.390 (4)	C16—H16C	0.9600
C8—N2	1.453 (4)	N1—N2	1.231 (3)

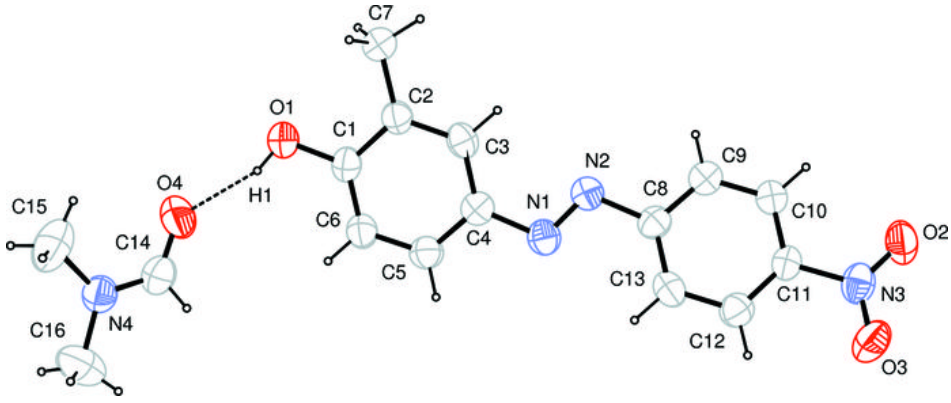
C9—C10	1.367 (4)	N3—O2	1.222 (3)
C9—H9	0.9300	N3—O3	1.222 (3)
C10—C11	1.371 (4)	O1—H1	0.8200
O1—C1—C6	123.2 (3)	C12—C11—C10	121.8 (3)
O1—C1—C2	116.0 (3)	C12—C11—N3	119.5 (3)
C6—C1—C2	120.8 (3)	C10—C11—N3	118.7 (3)
C3—C2—C1	117.6 (3)	C11—C12—C13	118.5 (3)
C3—C2—C7	122.2 (3)	C11—C12—H12	120.8
C1—C2—C7	120.2 (3)	C13—C12—H12	120.8
C2—C3—C4	121.6 (3)	C12—C13—C8	120.1 (3)
C2—C3—H3	119.2	C12—C13—H13	119.9
C4—C3—H3	119.2	C8—C13—H13	119.9
C5—C4—C3	119.2 (3)	O4—C14—N4	124.1 (4)
C5—C4—N1	115.8 (3)	O4—C14—H14	117.9
C3—C4—N1	125.0 (3)	N4—C14—H14	117.9
C6—C5—C4	120.4 (3)	N4—C15—H15A	109.5
C6—C5—H5	119.8	N4—C15—H15B	109.5
C4—C5—H5	119.8	H15A—C15—H15B	109.5
C5—C6—C1	120.4 (3)	N4—C15—H15C	109.5
C5—C6—H6	119.8	H15A—C15—H15C	109.5
C1—C6—H6	119.8	H15B—C15—H15C	109.5
C2—C7—H7A	109.5	N4—C16—H16A	109.5
C2—C7—H7B	109.5	N4—C16—H16B	109.5
H7A—C7—H7B	109.5	H16A—C16—H16B	109.5
C2—C7—H7C	109.5	N4—C16—H16C	109.5
H7A—C7—H7C	109.5	H16A—C16—H16C	109.5
H7B—C7—H7C	109.5	H16B—C16—H16C	109.5
C9—C8—C13	119.4 (3)	N2—N1—C4	111.4 (3)
C9—C8—N2	114.9 (3)	N1—N2—C8	111.6 (2)
C13—C8—N2	125.5 (3)	O2—N3—O3	123.7 (3)
C8—C9—C10	121.1 (3)	O2—N3—C11	118.6 (3)
C8—C9—H9	119.5	O3—N3—C11	117.7 (3)
C10—C9—H9	119.5	C14—N4—C15	121.6 (3)
C9—C10—C11	119.1 (3)	C14—N4—C16	119.8 (4)
C9—C10—H10	120.4	C15—N4—C16	118.6 (4)
C11—C10—H10	120.4	C1—O1—H1	109.5
O1—C1—C2—C3	179.8 (3)	C9—C10—C11—N3	−178.3 (3)
C6—C1—C2—C3	0.1 (5)	C10—C11—C12—C13	−0.6 (5)
O1—C1—C2—C7	1.3 (4)	N3—C11—C12—C13	178.3 (3)
C6—C1—C2—C7	−178.4 (3)	C11—C12—C13—C8	−0.1 (5)
C1—C2—C3—C4	−0.7 (4)	C9—C8—C13—C12	0.9 (5)
C7—C2—C3—C4	177.8 (3)	N2—C8—C13—C12	−175.6 (3)
C2—C3—C4—C5	0.7 (4)	C5—C4—N1—N2	173.7 (3)
C2—C3—C4—N1	−176.3 (3)	C3—C4—N1—N2	−9.1 (4)
C3—C4—C5—C6	−0.2 (5)	C4—N1—N2—C8	176.1 (2)
N1—C4—C5—C6	177.2 (3)	C9—C8—N2—N1	167.4 (3)
C4—C5—C6—C1	−0.4 (5)	C13—C8—N2—N1	−16.0 (4)
O1—C1—C6—C5	−179.3 (3)	C12—C11—N3—O2	174.4 (3)

C2—C1—C6—C5	0.4 (5)	C10—C11—N3—O2	−6.7 (4)
C13—C8—C9—C10	−0.9 (5)	C12—C11—N3—O3	−5.9 (4)
N2—C8—C9—C10	175.9 (3)	C10—C11—N3—O3	173.0 (3)
C8—C9—C10—C11	0.2 (5)	O4—C14—N4—C15	−2.9 (6)
C9—C10—C11—C12	0.5 (5)	O4—C14—N4—C16	179.9 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O4	0.82	1.84	2.595 (3)	153

Fig. 1


C7
C2
O1
C1
C3
N2
C9
O4
H1
C8
C10
O2
C15
C14
C6
C4
N1
C5
C13
C11
N3
C16
N4
C12
O3