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5-(4-Methoxyphenyl)-1-(4-methylphenyl)-1*H*-pyrazol-3-ol

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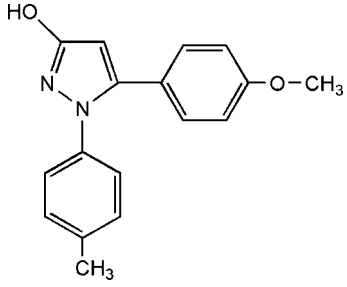
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.077; wR factor = 0.225; data-to-parameter ratio = 15.2.

The asymmetric unit of the title compound, $C_{17}H_{16}N_2O_2$, contains two crystallographically independent molecules, with methylphenyl and methoxyphenyl substituents in different orientations with respect to the pyrazole ring. In the crystal structure, intermolecular O—H···N and C—H···O hydrogen bonds link the molecules; the O—H···N bonds generate centrosymmetric dimers, and these are connected by C—H···O bonds to generate one-dimensional double-stranded chains.

Related literature

For bond-length data, see: Allen *et al.* (1987). For related literature, see: Masahiko & Kazuyoshi (1999); Zhu *et al.* (2004).



Experimental

Crystal data

$C_{17}H_{16}N_2O_2$
M_r = 280.32
Triclinic, $P\overline{1}$
a = 9.2520 (19) Å
b = 10.160 (2) Å
c = 16.338 (3) Å
α = 90.72 (3)°
β = 91.97 (3)°
γ = 107.34 (3)°
V = 1464.7 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 (2) K
0.40 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.975, T_{max} = 0.990
6130 measured reflections
5754 independent reflections
2899 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.077
$wR(F^2)$ = 0.225
S = 1.09
5754 reflections
379 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1*E*···N1^{i}	0.85	1.88	2.702 (5)	164
O3—H3*C*···N3^{ii}	0.82	1.91	2.728 (5)	175
C18—H18*B*···O1^{i}	0.96	2.50	3.380 (7)	152

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); 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: HK2281).

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supplementary materials

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5-(4-Methoxyphenyl)-1-(4-methylphenyl)-1*H*-pyrazol-3-ol

Y.-F. Sun, H.-S. Jia, S. Liu and H.-J. Zhu

Comment

In the process of synthesis, we obtained the title compound, (I), and we herein report its crystal structure.

The asymmetric unit of the title compound, (I), contains two crystallographically independent molecules, with methylphenyl and methoxyphenyl substituents in different orientations with respect to the pyrazole ring (Fig. 1). The bond lengths and angles are generally within normal ranges (Allen *et al.*, 1987).

The rings A (N1/N2/C8—C10), B (C2—C7), C (C11—C16) and A' (N3/N4/C25—C27), B' (C19—C24), C' (C28—C33) are, of course, planar and they are oriented at diheral angles of A/B = 53.93 (2)°, A/C = 39.63 (3)°, B/C = 64.37 (2)° and A'/B' = 45.45 (2)°, A'/C' = 48.66 (3)°, B'/C' = 51.98 (3)°.

In the crystal structure, intermolecular O—H···N and C—H···O hydrogen bonds (Table 1) link the molecules, in which O—H···N bonds generate the centrosymmetric dimers, and they are connected by C—H···O bonds to generate one-dimensional double-stranded chains (Fig. 2).

Experimental

For the preparation of the title compound, (I), 5-(4-Methoxyphenyl)-1-p -tolylpyrazolidin-3-one, (II), was firstly prepared and recrystallized from ethanol, according to the literature method (Masahiko & Kazuyoshi, 1999). Then, compound (II) (4.2 g, 15 mmol) was dissolved in dimethyl- formamide (100 ml) and mixed with $FeCl_3$ (0.3 g, 1.5 mmol) (Zhu *et al.*, 2004). The mixture was heated to 353 K and maintained at that temperature for 1 h, and then stirred for 12 h without heating. The reaction mixture was then poured into water (500 ml). The precipitate which formed was filtered off, washed with water and dried under reduced pressure. The crude product was then crystallized from ethanol to give (I) (yield; 2.8 g, 10 mmol). Crystals of (I) suitable for X-ray analysis were grown by slow evaporation of a solution in acetic ether at room temperature about 7 d.

Refinement

H atoms were positioned geometrically, with O—H = 0.82 Å (for OH) and C—H = 0.93 and 0.96 Å for aromatic and methyl H atoms, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C,*O*), where x = 1.2 for aromatic H, and x = 1.5 for all other H atoms.

Figures

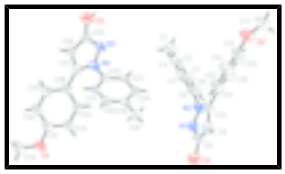

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 40% probability level.

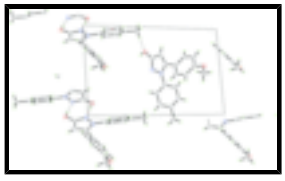

Fig. 2. A packing diagram of (I). Hydrogen bonds are shown as dashed lines.

5-(4-Methoxyphenyl)-1-(4-methylphenyl)-1*H*-pyrazol-3-ol

Crystal data

$C_{17}H_{16}N_2O_2$	$Z = 4$
$M_r = 280.32$	$F_{000} = 592$
Triclinic, $P\overline{1}$	$D_x = 1.271$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 515 K
$a = 9.2520$ (19) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 10.160$ (2) Å	Cell parameters from 25 reflections
$c = 16.338$ (3) Å	$\theta = 9$–12º
$\alpha = 90.72$ (3)º	$\mu = 0.09$ mm^{-1}
$\beta = 91.97$ (3)º	$T = 298$ (2) K
$\gamma = 107.34$ (3)º	Needle, colorless
$V = 1464.7$ (6) Å^3	0.40 × 0.10 × 0.10 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.056$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 1.3$º
$T = 298$(2) K	$h = -11 \rightarrow 11$
ω/2θ scans	$k = -12 \rightarrow 12$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 20$
$T_{min} = 0.975$, $T_{max} = 0.990$	3 standard reflections
6130 measured reflections	every 120 min
5754 independent reflections	intensity decay: none
2899 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.077$	H-atom parameters constrained
$wR(F^2) = 0.225$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 2.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.002$
5754 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
379 parameters	$\Delta\rho_{min} = -0.28$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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}
O1	0.3812 (4)	0.3092 (3)	0.4980 (3)	0.0922 (13)
H1E	0.4660	0.3544	0.5206	0.111*
O2	−0.4318 (4)	0.4092 (4)	0.2242 (2)	0.0857 (11)
N1	0.3451 (4)	0.5152 (4)	0.4561 (3)	0.0688 (11)
N2	0.2291 (4)	0.5472 (4)	0.4138 (2)	0.0651 (11)
C1	0.3833 (6)	1.1069 (5)	0.3103 (4)	0.0936 (18)
H1B	0.4116	1.1693	0.3568	0.140*
H1C	0.3011	1.1249	0.2797	0.140*
H1D	0.4684	1.1197	0.2760	0.140*
C2	0.3336 (5)	0.9587 (5)	0.3397 (3)	0.0626 (12)
C3	0.2867 (5)	0.8513 (5)	0.2808 (3)	0.0646 (13)
H3A	0.2808	0.8708	0.2255	0.078*
C4	0.2493 (5)	0.7155 (5)	0.3057 (3)	0.0639 (13)
H4A	0.2175	0.6435	0.2673	0.077*
C5	0.2593 (5)	0.6883 (5)	0.3867 (3)	0.0611 (12)
C6	0.3034 (5)	0.7945 (5)	0.4455 (3)	0.0662 (13)
H6A	0.3060	0.7753	0.5010	0.079*
C7	0.3429 (5)	0.9281 (5)	0.4198 (4)	0.0709 (14)

H7A	0.3770	0.9997	0.4585	0.085*
C8	0.2978 (6)	0.3788 (5)	0.4612 (3)	0.0725 (14)
C9	0.1579 (5)	0.3230 (5)	0.4217 (3)	0.0740 (14)
H9A	0.1031	0.2301	0.4164	0.089*
C10	0.1164 (5)	0.4329 (5)	0.3919 (3)	0.0663 (13)
C11	−0.0252 (5)	0.4331 (5)	0.3476 (3)	0.0617 (12)
C12	−0.0911 (5)	0.3309 (5)	0.2886 (3)	0.0726 (14)
H12A	−0.0419	0.2663	0.2754	0.087*
C13	−0.2264 (5)	0.3234 (5)	0.2498 (3)	0.0763 (15)
H13A	−0.2710	0.2515	0.2126	0.092*
C14	−0.2975 (5)	0.4228 (5)	0.2658 (3)	0.0670 (13)
C15	−0.2348 (6)	0.5255 (6)	0.3244 (4)	0.0844 (17)
H15A	−0.2834	0.5907	0.3373	0.101*
C16	−0.0983 (5)	0.5290 (5)	0.3634 (3)	0.0760 (15)
H16A	−0.0547	0.5993	0.4017	0.091*
C17	−0.5285 (6)	0.4835 (7)	0.2533 (4)	0.101 (2)
H17A	−0.6190	0.4625	0.2188	0.151*
H17B	−0.4771	0.5806	0.2523	0.151*
H17C	−0.5547	0.4576	0.3084	0.151*
O3	0.5366 (4)	0.9271 (4)	−0.1042 (2)	0.0901 (12)
H3C	0.4917	0.9760	−0.0826	0.135*
O4	1.2470 (4)	0.5589 (4)	0.0961 (2)	0.0795 (10)
N3	0.6276 (4)	0.9259 (4)	0.0300 (2)	0.0618 (10)
N4	0.7377 (4)	0.8768 (4)	0.0654 (2)	0.0609 (10)
C18	0.8710 (7)	0.9521 (6)	0.4095 (3)	0.0938 (18)
H18A	0.9787	0.9828	0.4203	0.141*
H18B	0.8286	0.8649	0.4348	0.141*
H18C	0.8269	1.0186	0.4315	0.141*
C19	0.8381 (5)	0.9361 (5)	0.3186 (3)	0.0607 (12)
C20	0.9493 (5)	0.9620 (5)	0.2624 (3)	0.0629 (12)
H20A	1.0497	0.9923	0.2817	0.076*
C21	0.9209 (5)	0.9459 (5)	0.1793 (3)	0.0586 (12)
H21A	1.0001	0.9647	0.1435	0.070*
C22	0.7713 (5)	0.9008 (4)	0.1499 (3)	0.0532 (11)
C23	0.6570 (5)	0.8759 (5)	0.2060 (3)	0.0597 (12)
H23A	0.5563	0.8462	0.1870	0.072*
C24	0.6890 (5)	0.8938 (5)	0.2871 (3)	0.0687 (13)
H24A	0.6099	0.8775	0.3229	0.082*
C25	0.6305 (6)	0.8974 (5)	−0.0495 (3)	0.0649 (13)
C26	0.7356 (6)	0.8242 (5)	−0.0640 (3)	0.0691 (13)
H26A	0.7548	0.7895	−0.1141	0.083*
C27	0.8022 (5)	0.8151 (5)	0.0092 (3)	0.0593 (12)
C28	0.9197 (5)	0.7482 (5)	0.0309 (3)	0.0578 (11)
C29	1.0511 (5)	0.7743 (5)	−0.0123 (3)	0.0696 (13)
H29A	1.0642	0.8343	−0.0559	0.083*
C30	1.1634 (5)	0.7144 (5)	0.0070 (3)	0.0706 (14)
H30A	1.2509	0.7346	−0.0227	0.085*
C31	1.1442 (5)	0.6256 (5)	0.0699 (3)	0.0602 (12)
C32	1.0140 (5)	0.5950 (5)	0.1143 (3)	0.0623 (12)

H32A	1.0013	0.5335	0.1570	0.075*
C33	0.9034 (5)	0.6563 (5)	0.0946 (3)	0.0574 (11)
H33A	0.8161	0.6357	0.1246	0.069*
C34	1.3763 (5)	0.5759 (6)	0.0480 (4)	0.0854 (17)
H34A	1.4398	0.5259	0.0718	0.128*
H34B	1.4321	0.6721	0.0465	0.128*
H34C	1.3443	0.5413	−0.0067	0.128*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.060 (2)	0.061 (2)	0.148 (4)	0.0090 (18)	−0.023 (2)	0.020 (2)
O2	0.057 (2)	0.087 (3)	0.109 (3)	0.018 (2)	−0.025 (2)	−0.006 (2)
N1	0.053 (2)	0.059 (3)	0.091 (3)	0.012 (2)	−0.013 (2)	0.006 (2)
N2	0.052 (2)	0.052 (2)	0.087 (3)	0.0116 (19)	−0.018 (2)	0.003 (2)
C1	0.078 (4)	0.059 (3)	0.135 (5)	0.007 (3)	0.003 (4)	0.000 (3)
C2	0.039 (2)	0.054 (3)	0.094 (4)	0.013 (2)	−0.002 (2)	−0.002 (3)
C3	0.047 (3)	0.072 (3)	0.076 (3)	0.021 (2)	0.000 (2)	−0.004 (3)
C4	0.050 (3)	0.062 (3)	0.079 (3)	0.019 (2)	−0.011 (2)	−0.018 (3)
C5	0.047 (3)	0.060 (3)	0.075 (3)	0.015 (2)	−0.008 (2)	−0.002 (3)
C6	0.068 (3)	0.064 (3)	0.066 (3)	0.019 (3)	−0.006 (2)	−0.008 (3)
C7	0.057 (3)	0.053 (3)	0.100 (4)	0.015 (2)	−0.010 (3)	−0.017 (3)
C8	0.058 (3)	0.067 (3)	0.093 (4)	0.021 (3)	−0.016 (3)	0.004 (3)
C9	0.053 (3)	0.061 (3)	0.099 (4)	0.003 (2)	−0.003 (3)	0.004 (3)
C10	0.051 (3)	0.062 (3)	0.083 (3)	0.014 (2)	−0.005 (2)	−0.005 (3)
C11	0.046 (2)	0.065 (3)	0.070 (3)	0.012 (2)	−0.009 (2)	−0.006 (2)
C12	0.057 (3)	0.065 (3)	0.095 (4)	0.020 (3)	−0.010 (3)	−0.008 (3)
C13	0.057 (3)	0.065 (3)	0.102 (4)	0.014 (3)	−0.019 (3)	−0.015 (3)
C14	0.048 (3)	0.068 (3)	0.083 (3)	0.017 (2)	−0.011 (2)	0.000 (3)
C15	0.062 (3)	0.081 (4)	0.112 (5)	0.026 (3)	−0.014 (3)	−0.023 (3)
C16	0.053 (3)	0.075 (4)	0.096 (4)	0.016 (3)	−0.017 (3)	−0.022 (3)
C17	0.063 (4)	0.110 (5)	0.136 (6)	0.039 (4)	−0.018 (4)	−0.006 (4)
O3	0.100 (3)	0.116 (3)	0.072 (2)	0.064 (3)	−0.023 (2)	−0.017 (2)
O4	0.057 (2)	0.088 (3)	0.102 (3)	0.0357 (19)	0.0013 (19)	0.014 (2)
N3	0.043 (2)	0.072 (3)	0.075 (3)	0.0259 (19)	−0.0095 (18)	−0.007 (2)
N4	0.050 (2)	0.065 (3)	0.068 (3)	0.0202 (19)	−0.0099 (19)	−0.002 (2)
C18	0.092 (4)	0.085 (4)	0.088 (4)	0.005 (3)	−0.019 (3)	−0.004 (3)
C19	0.056 (3)	0.058 (3)	0.068 (3)	0.019 (2)	−0.009 (2)	−0.006 (2)
C20	0.040 (2)	0.071 (3)	0.073 (3)	0.012 (2)	−0.009 (2)	−0.007 (3)
C21	0.044 (2)	0.070 (3)	0.060 (3)	0.015 (2)	−0.005 (2)	−0.002 (2)
C22	0.046 (2)	0.048 (3)	0.064 (3)	0.013 (2)	0.000 (2)	−0.003 (2)
C23	0.039 (2)	0.071 (3)	0.070 (3)	0.018 (2)	−0.003 (2)	−0.001 (2)
C24	0.051 (3)	0.087 (4)	0.066 (3)	0.017 (3)	0.006 (2)	0.002 (3)
C25	0.067 (3)	0.073 (3)	0.058 (3)	0.028 (3)	−0.011 (2)	−0.006 (2)
C26	0.070 (3)	0.085 (4)	0.054 (3)	0.026 (3)	0.000 (2)	−0.010 (3)
C27	0.048 (3)	0.059 (3)	0.071 (3)	0.015 (2)	0.003 (2)	−0.010 (2)
C28	0.048 (3)	0.054 (3)	0.071 (3)	0.015 (2)	0.004 (2)	0.001 (2)
C29	0.064 (3)	0.071 (3)	0.079 (3)	0.027 (3)	0.013 (3)	0.013 (3)

C30	0.048 (3)	0.065 (3)	0.102 (4)	0.022 (2)	0.012 (3)	0.007 (3)
C31	0.050 (3)	0.054 (3)	0.076 (3)	0.017 (2)	−0.007 (2)	−0.006 (2)
C32	0.050 (3)	0.064 (3)	0.073 (3)	0.018 (2)	0.002 (2)	0.001 (2)
C33	0.046 (2)	0.059 (3)	0.067 (3)	0.014 (2)	0.000 (2)	0.000 (2)
C34	0.051 (3)	0.089 (4)	0.117 (5)	0.025 (3)	−0.005 (3)	−0.007 (3)

Geometric parameters (Å, °)

O1—C8	1.327 (5)	O3—C25	1.325 (5)
O1—H1E	0.8500	O3—H3C	0.8200
O2—C14	1.365 (5)	O4—C31	1.382 (5)
O2—C17	1.420 (6)	O4—C34	1.423 (6)
N1—C8	1.328 (6)	N3—C25	1.329 (6)
N1—N2	1.378 (5)	N3—N4	1.374 (5)
N2—C10	1.344 (6)	N4—C27	1.355 (5)
N2—C5	1.454 (6)	N4—C22	1.407 (5)
C1—C2	1.526 (7)	C18—C19	1.502 (7)
C1—H1B	0.9600	C18—H18A	0.9600
C1—H1C	0.9600	C18—H18B	0.9600
C1—H1D	0.9600	C18—H18C	0.9600
C2—C7	1.356 (7)	C19—C20	1.372 (6)
C2—C3	1.403 (6)	C19—C24	1.395 (6)
C3—C4	1.388 (6)	C20—C21	1.372 (6)
C3—H3A	0.9300	C20—H20A	0.9300
C4—C5	1.360 (6)	C21—C22	1.388 (6)
C4—H4A	0.9300	C21—H21A	0.9300
C5—C6	1.392 (6)	C22—C23	1.391 (6)
C6—C7	1.372 (7)	C23—C24	1.346 (6)
C6—H6A	0.9300	C23—H23A	0.9300
C7—H7A	0.9300	C24—H24A	0.9300
C8—C9	1.382 (6)	C25—C26	1.413 (6)
C9—C10	1.374 (6)	C26—C27	1.344 (6)
C9—H9A	0.9300	C26—H26A	0.9300
C10—C11	1.475 (6)	C27—C28	1.480 (6)
C11—C16	1.368 (6)	C28—C29	1.386 (6)
C11—C12	1.392 (6)	C28—C33	1.389 (6)
C12—C13	1.365 (6)	C29—C30	1.382 (6)
C12—H12A	0.9300	C29—H29A	0.9300
C13—C14	1.387 (6)	C30—C31	1.356 (6)
C13—H13A	0.9300	C30—H30A	0.9300
C14—C15	1.385 (7)	C31—C32	1.384 (6)
C15—C16	1.385 (6)	C32—C33	1.379 (6)
C15—H15A	0.9300	C32—H32A	0.9300
C16—H16A	0.9300	C33—H33A	0.9300
C17—H17A	0.9600	C34—H34A	0.9600
C17—H17B	0.9600	C34—H34B	0.9600
C17—H17C	0.9600	C34—H34C	0.9600
C8—O1—H1E	118.4	C25—O3—H3C	109.5
C14—O2—C17	118.8 (4)	C31—O4—C34	116.5 (4)

C8—N1—N2	104.5 (4)	C25—N3—N4	104.2 (4)
C10—N2—N1	111.3 (4)	C27—N4—N3	111.7 (4)
C10—N2—C5	130.5 (4)	C27—N4—C22	129.9 (4)
N1—N2—C5	116.9 (4)	N3—N4—C22	118.4 (4)
C2—C1—H1B	109.5	C19—C18—H18A	109.5
C2—C1—H1C	109.5	C19—C18—H18B	109.5
H1B—C1—H1C	109.5	H18A—C18—H18B	109.5
C2—C1—H1D	109.5	C19—C18—H18C	109.5
H1B—C1—H1D	109.5	H18A—C18—H18C	109.5
H1C—C1—H1D	109.5	H18B—C18—H18C	109.5
C7—C2—C3	119.4 (5)	C20—C19—C24	116.3 (4)
C7—C2—C1	122.0 (5)	C20—C19—C18	123.2 (4)
C3—C2—C1	118.4 (5)	C24—C19—C18	120.6 (5)
C4—C3—C2	119.4 (5)	C21—C20—C19	123.8 (4)
C4—C3—H3A	120.3	C21—C20—H20A	118.1
C2—C3—H3A	120.3	C19—C20—H20A	118.1
C5—C4—C3	119.7 (5)	C20—C21—C22	118.5 (4)
C5—C4—H4A	120.1	C20—C21—H21A	120.8
C3—C4—H4A	120.1	C22—C21—H21A	120.8
C4—C5—C6	121.1 (5)	C21—C22—C23	118.5 (4)
C4—C5—N2	120.7 (4)	C21—C22—N4	120.2 (4)
C6—C5—N2	118.2 (4)	C23—C22—N4	121.3 (4)
C7—C6—C5	118.5 (5)	C24—C23—C22	121.4 (4)
C7—C6—H6A	120.8	C24—C23—H23A	119.3
C5—C6—H6A	120.8	C22—C23—H23A	119.3
C2—C7—C6	121.8 (5)	C23—C24—C19	121.5 (5)
C2—C7—H7A	119.1	C23—C24—H24A	119.2
C6—C7—H7A	119.1	C19—C24—H24A	119.2
N1—C8—O1	122.1 (4)	O3—C25—N3	122.1 (4)
N1—C8—C9	111.5 (4)	O3—C25—C26	126.8 (4)
O1—C8—C9	126.3 (5)	N3—C25—C26	110.9 (4)
C10—C9—C8	105.9 (5)	C27—C26—C25	105.9 (4)
C10—C9—H9A	127.1	C27—C26—H26A	127.0
C8—C9—H9A	127.1	C25—C26—H26A	127.0
N2—C10—C9	106.8 (4)	C26—C27—N4	107.2 (4)
N2—C10—C11	124.3 (4)	C26—C27—C28	129.7 (4)
C9—C10—C11	128.9 (5)	N4—C27—C28	123.1 (4)
C16—C11—C12	117.6 (4)	C29—C28—C33	117.1 (4)
C16—C11—C10	122.6 (4)	C29—C28—C27	121.0 (4)
C12—C11—C10	119.8 (4)	C33—C28—C27	121.9 (4)
C13—C12—C11	121.3 (5)	C30—C29—C28	122.2 (5)
C13—C12—H12A	119.3	C30—C29—H29A	118.9
C11—C12—H12A	119.3	C28—C29—H29A	118.9
C12—C13—C14	120.0 (5)	C31—C30—C29	119.2 (5)
C12—C13—H13A	120.0	C31—C30—H30A	120.4
C14—C13—H13A	120.0	C29—C30—H30A	120.4
O2—C14—C15	123.4 (5)	C30—C31—O4	125.4 (4)
O2—C14—C13	116.7 (5)	C30—C31—C32	120.7 (4)
C15—C14—C13	119.8 (4)	O4—C31—C32	114.0 (4)

C14—C15—C16	118.5 (5)	C33—C32—C31	119.5 (5)
C14—C15—H15A	120.7	C33—C32—H32A	120.2
C16—C15—H15A	120.7	C31—C32—H32A	120.2
C11—C16—C15	122.6 (5)	C32—C33—C28	121.3 (4)
C11—C16—H16A	118.7	C32—C33—H33A	119.4
C15—C16—H16A	118.7	C28—C33—H33A	119.4
O2—C17—H17A	109.5	O4—C34—H34A	109.5
O2—C17—H17B	109.5	O4—C34—H34B	109.5
H17A—C17—H17B	109.5	H34A—C34—H34B	109.5
O2—C17—H17C	109.5	O4—C34—H34C	109.5
H17A—C17—H17C	109.5	H34A—C34—H34C	109.5
H17B—C17—H17C	109.5	H34B—C34—H34C	109.5
C8—N1—N2—C10	−1.7 (6)	C25—N3—N4—C27	−2.3 (5)
C8—N1—N2—C5	−169.9 (4)	C25—N3—N4—C22	175.2 (4)
C7—C2—C3—C4	0.6 (7)	C24—C19—C20—C21	−1.4 (7)
C1—C2—C3—C4	176.4 (4)	C18—C19—C20—C21	178.9 (5)
C2—C3—C4—C5	−0.4 (7)	C19—C20—C21—C22	0.1 (7)
C3—C4—C5—C6	1.5 (7)	C20—C21—C22—C23	0.8 (7)
C3—C4—C5—N2	−176.4 (4)	C20—C21—C22—N4	−177.1 (4)
C10—N2—C5—C4	−45.2 (7)	C27—N4—C22—C21	42.1 (7)
N1—N2—C5—C4	120.3 (5)	N3—N4—C22—C21	−134.9 (4)
C10—N2—C5—C6	136.8 (5)	C27—N4—C22—C23	−135.7 (5)
N1—N2—C5—C6	−57.7 (6)	N3—N4—C22—C23	47.2 (6)
C4—C5—C6—C7	−2.7 (7)	C21—C22—C23—C24	−0.4 (7)
N2—C5—C6—C7	175.3 (4)	N4—C22—C23—C24	177.5 (4)
C3—C2—C7—C6	−2.0 (7)	C22—C23—C24—C19	−1.0 (8)
C1—C2—C7—C6	−177.5 (5)	C20—C19—C24—C23	1.8 (7)
C5—C6—C7—C2	3.0 (7)	C18—C19—C24—C23	−178.5 (5)
N2—N1—C8—O1	179.6 (5)	N4—N3—C25—O3	178.5 (4)
N2—N1—C8—C9	1.7 (6)	N4—N3—C25—C26	3.1 (5)
N1—C8—C9—C10	−1.0 (7)	O3—C25—C26—C27	−178.1 (5)
O1—C8—C9—C10	−178.9 (5)	N3—C25—C26—C27	−2.9 (6)
N1—N2—C10—C9	1.1 (6)	C25—C26—C27—N4	1.4 (6)
C5—N2—C10—C9	167.2 (5)	C25—C26—C27—C28	179.2 (5)
N1—N2—C10—C11	178.4 (4)	N3—N4—C27—C26	0.5 (5)
C5—N2—C10—C11	−15.4 (8)	C22—N4—C27—C26	−176.6 (4)
C8—C9—C10—N2	−0.1 (6)	N3—N4—C27—C28	−177.5 (4)
C8—C9—C10—C11	−177.2 (5)	C22—N4—C27—C28	5.3 (7)
N2—C10—C11—C16	−38.6 (8)	C26—C27—C28—C29	50.0 (7)
C9—C10—C11—C16	138.1 (6)	N4—C27—C28—C29	−132.4 (5)
N2—C10—C11—C12	142.9 (5)	C26—C27—C28—C33	−129.5 (6)
C9—C10—C11—C12	−40.4 (8)	N4—C27—C28—C33	48.1 (7)
C16—C11—C12—C13	−2.2 (8)	C33—C28—C29—C30	−1.1 (7)
C10—C11—C12—C13	176.3 (5)	C27—C28—C29—C30	179.4 (5)
C11—C12—C13—C14	3.2 (8)	C28—C29—C30—C31	0.7 (8)
C17—O2—C14—C15	−13.9 (8)	C29—C30—C31—O4	−179.7 (4)
C17—O2—C14—C13	163.2 (5)	C29—C30—C31—C32	0.1 (7)
C12—C13—C14—O2	179.5 (5)	C34—O4—C31—C30	−6.1 (7)
C12—C13—C14—C15	−3.4 (8)	C34—O4—C31—C32	174.2 (4)

O2—C14—C15—C16	179.5 (5)	C30—C31—C32—C33	−0.5 (7)
C13—C14—C15—C16	2.6 (8)	O4—C31—C32—C33	179.3 (4)
C12—C11—C16—C15	1.5 (8)	C31—C32—C33—C28	0.1 (7)
C10—C11—C16—C15	−177.0 (5)	C29—C28—C33—C32	0.6 (7)
C14—C15—C16—C11	−1.7 (9)	C27—C28—C33—C32	−179.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1E···N1^{i}	0.85	1.88	2.702 (5)	164
O3—H3C···N3^{ii}	0.82	1.91	2.728 (5)	175
C18—H18B···O1^{i}	0.96	2.50	3.380 (7)	152

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) −*x*+1, −*y*+2, −*z*.

Fig. 1

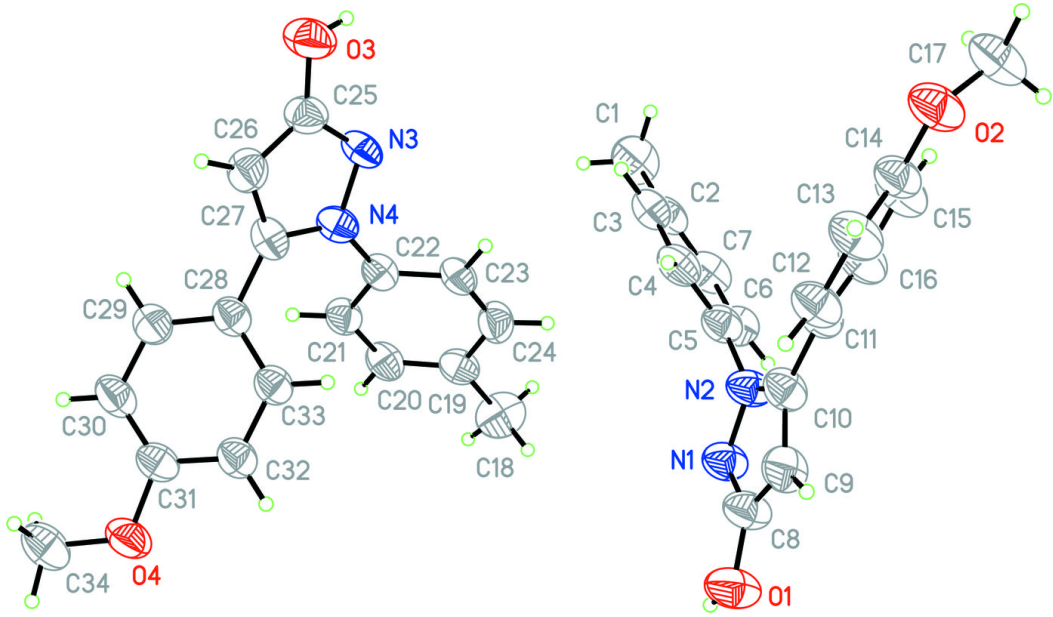
O3
C25
C26
N3
C27
N4
C22
C23
C28
C29
C21
C24
C20
C19
C33
C30
C18
C32
C31
O4
C34
C17
O2
C1
C14
C2
C13
C15
C3
C7
C12
C16
C4
C6
C5
C11
N2
C10
N1
C9
C8
O1

Fig. 2