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4′-Methyl-3-(4-nitrophenyl)-4-phenyl-4,5,1′,2′,3′,4′-hexahydrospiro[isoxazole-5,2′-naphthalen]-1′-one

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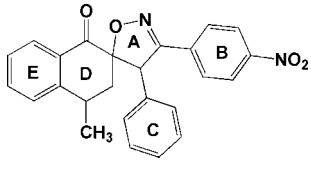
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.123; data-to-parameter ratio = 13.4.

The title compound, $C_{25}H_{20}N_2O_4$, is a new spiro-isoxazoline derivative. It contains a five-membered isoxazoline ring (*A*), a tetralone unit (*E* and *D*), a 4-nitrophenyl substituent (*B*), and a phenyl ring (*C*). The isoxazoline ring (*A*) has an envelope conformation, while the cyclohexenone ring (*D*) has an intermediate sofa/half-chair conformation. The aromatic ring of the 4-nitrophenyl substituent (*B*) is inclined at an angle of 78.97 (10)° to the phenyl ring (*C*). The rigid pharmacophore site, Osp^2—C—C—Osp^3, is characterized by an O···O distance of 3.113 (2) Å and an O—C—C—O torsion angle of 97.8 (2)°. In the crystal structure, molecules are linked by C—H···O contacts.

Related literature

For the crystal structure of a very similar compound, 3-(4-chlorophenyl)-4-(4-phenyl-3′,4′-dihydro-1′*H*,4*H*-spiro[isoxazole-5,2′-naphthalen]-1′-one, see: Subbiah Pandi *et al.* (2001). For related literature, see: Anaflous *et al.* (2004); Arnold *et al.* (1947); Bakavoli *et al.* (2005); Chaouni-Benabdallah *et al.* (2001); Chenera *et al.* (1993); Debaerdemaeker *et al.* (1977); Ellis (1997); Howe & Shelton (1990); Katritzky *et al.* (2003); Kerbal *et al.* (1990); Kooijman *et al.* (1984); Seifert *et al.* (1976); Smietana *et al.* (1999).



Experimental

Crystal data

$C_{25}H_{20}N_2O_4$
M_r = 412.43
Monoclinic, $P2_1/n$
a = 10.6567 (15) Å
b = 15.7071 (16) Å
c = 12.7259 (15) Å
β = 106.258 (10)°
V = 2045.0 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 173 (2) K
0.40 × 0.37 × 0.27 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: none
20256 measured reflections
3798 independent reflections
2837 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.123
S = 1.01
3798 reflections
283 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2B···O4^{i}	0.99	2.50	3.319 (3)	140
C20—H20···O1^{ii}	0.95	2.58	3.365 (2)	140

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

Data collection: *X-AREA* (Stoe & Cie, 2005); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2153).

References

Anaflous, A., Benchat1, N., Mimouni, Abouricha, M. S., Ben-Hadda, T., El-Bali, B., hakkou, A. & Hacht, B. (2004). *Lett. Drug. Des. Disc.* **1**, 35–44.
Arnold, T., Bruckley, J. S. & Richter, J. (1947). *J. Am. Chem. Soc.* **69**, 2323–2325.
Bakavoli, M., Bagherzadeh, G. & Rahimizadeh, M. (2005). *Mendeleev Commun.* **4**, 145–146.
Chaouni-Benabdallah, A., Galtier, C., Allouchi, H., Kherbeche, A., Debouzy, J. C., Teulade, J. C., Chavignon, O., Witvrouw, M., Pannecouque, C., Balzarini, J., De Clercq, E. & Enguehard, C. (2001). *Arch. Pharm. (Weinheim)*, **334**, 224–228.
Chenera, B., West, M. L., Finkelstein, J. A. & Dreyer, G. B. J. (1993). *J. Org. Chem.* **58**, 5605–5606.
Debaerdemaeker, T., Pohl, H. H. & Dimroth, K. (1977). *Chem. Ber. Recl*, **110**, 1497–1503.
Ellis, G. P. (1997). *Chromenes, Chromanones and Chromones*. New York: John Wiley and Sons, Inc.
Howe, R. K. & Shelton, B. R. (1990). *J. Org. Chem.* **55**, 4603–4607.

 doi:10.1107/S1600536807066470

Katritzky, A. R., Xu, Y. J. & Tu, H. B. (2003). *J. Org. Chem.* **68**, 4935–4937.
Kerbal, A., Tshiamala, K., Cerutti, E., Laude, B. & Vebrel, J. (1990). *Bull. Soc. Chim. Fr.* **127**, 252–257.
Kooijman, H., Spek, A. L., Kleijn, H., van Maanen, H. L., Jastrzebski, J. T. B. H. & van Kozikowski, A. P. (1984). *Acc. Chem. Res.* **17**, 410–416.
Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.
Seifert, W. J., Schaffer, O. & Dimroth, K. (1976). *Angew. Chem. Int. Ed. Engl.* **15**, 238–239.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Smietana, M., Gouverneur, V. & Mioskowski, C. (1999). *Tetrahedron Lett.* **40**, 1291–1294.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.
Stoe & Cie (2005). *X-AREA* (Version 1.25) and *X-RED32* (Version 1.26). Stoe & Cie GmbH, Darmstadt, Germany.
Subbiah Pandi, A., Banumathi, S., Velmurugan, D., Shanmuga Sundara Raj, S., Fun, H.-K. & Manikandan, S. (2001). *Acta Cryst.* C**57**, 819–820.

supporting information

Acta Cryst. (2008). E**64**, o509–o510 [doi:10.1107/S1600536807066470]

4′-Methyl-3-(4-nitrophenyl)-4-phenyl-4,5,1′,2′,3′,4′-hexahydrospiro-[isoxazole-5,2′-naphthalen]-1′-one

Ghali Alhouari, Abdelali Kerbal, Najib Ben Larbi, Brahim Bennani, Taibi Ben Hadda and Helen Stoeckli-Evans

S1. Comment

Spiroisoxazolines display interesting biological properties such as herbicidal, plant-growth regulatory and antitumour activities (Howe & Shelton, 1990; Smietana *et al.*, 1999). Many 4-chromanone derivatives are versatile intermediates for the synthesis of natural products such as brazillin, hematoxylin, ripariochromene, clausenin, calonlide A and inophyllum B (Kooijman *et al.*, 1984; Ellis *et al.*, 1997; Chenera *et al.*, 1993). Chromanone heterocycles have also attracted much attention owing to their important pharmacological properties (Chaouni-Benabdallah *et al.*, 2001). As a extention of our pharmacological studies on the structure-antituburcular activity relationships in 3-armed imidazo[1,2-*a*]pyrimidines (Debaerdemaeker *et al.*, 1977; Seifert *et al.*, 1976; Anaflous *et al.*, 2004), we synthesized 4′-methyl-4-(4-phenyl)-3-(4-nitrophenyl)-3′,4′- dihydro-1′*H*,4*H*-spiro[isoxazole-5,2′-naphthalen]-1′-one (III). Some analogous structures have been reported previously (Katritzky *et al.*, 2003; Bakavoli *et al.*, 2005). The title compound, (III), was prepared by the action of *para*-nitro- benzaldoxime (II), on 4-methyl-2-[(*E*)-phenylmethylidene]-3,4- dihydro-1(2*H*)-naphthalenone (I).

The molecular structure of compound (III) is illustrated in Fig. 1. It is composed of a five-membered isoxazoline ring (A), a tetralone moiety (E & D), a 4-nitrophenyl substituent (B), and a phenyl ring (C). Ring A [C1,O2,N1,C12,C11] has an envelope conformation, with atom C1 at the flap and lieing out of the best least-squares plane through the other four atoms by 0.259 (2) Å. Ring D [C1—C4,C9,C10] has an intermediate sofa/half-chair conformation with atoms C1 and C2 out of the best least-squares plane through the other four atoms [planar to within 0.028 (2) Å] by -0.111 (2) and 0.538 (2) Å, respectively. The best least-squares plane through atoms O2,N1,C12,C11 [planar to within 0.008 (2) Å], of the isoxazoline ring A, is inclined to the best least-squares planes through phenyl ring E (C4—C9) by 86.95 (11)°, and to the best least-squares plane through rings B (C13—C18), and C (C19—C24) by 7.47 (11) and 83.47 (11) °, respectively. Ring C is inclined to rings B and E by 78.97 (10)° and 78.66 (11) °, respectively.

The rigid pharmacophore site, O(sp2)-C—C—O(sp3), is characterized by an O···O spatial distance of 3.113 (2) Å (O1···O2), with a torsion angle (O2—C1—C10—O1) of 97.8 (2) °. The bond distances and angles are very similar to those observed in 3-(4-chlorophenyl)-4-(4-phenyl-3′,4′-dihydro- 1′*H*,4*H*-spiro[isoxazole-5,2′-naphthalen]-1′-one (Subbiah Pandi *et al.*, 2001).

In the crystal structure of (III) symmetry related molecules are linked by intermolecular C—H···O contacts (Table 1 and Fig. 2).

S2. Experimental

Compound (III) was synthesized by the action of *para*-nitro- benzaldoxime (II) on 4-methyl-2-[(*E*)-phenylmethyl-idene]-3,4-dihydro-1(2*H*)- naphthalenone (I). The latter was prepared according to the method described previously

(Arnold *et al.*, 1947). Its conformational analysis has also been reported (Kerbal *et al.*, 1990).

In a 100 ml flask, 2 mmoles (0.5 g) of (I) and 0.4 g (2.4 mmoles) mmoles of (II) were dissolved in 20 ml of chloroform. The mixture was cooled to 0°C under magnetic stirring in an ice bath. Then 15 ml of bleach at 18° was added in small doses without exceeding 5°C. The mixture was left under magnetic stirring for 16 h at room temperature, then washed with water until the pH was neutral. It was then dried on sodium sulfate. The solvent was evaporated with a rotating evaporator and the oily residue obtained dissolved in ethanol. The precipitated cycloadduct was then analysed by TLC, which indicated the limited formation of two of the 4 stereoisomers for (III) in a 9:1 ratio. The major stereoisomer, (III), was then crystallized in ethanol giving colourless block-like crystals (61% yield), *M*.p. = 150–153 °C.

S3. Refinement

The H atoms were included in calculated positons and treated as riding atoms with C—H = 0.95 - 1.00 Å and U_{iso}(H) = 1.2 or 1.5U_{eq}(C).

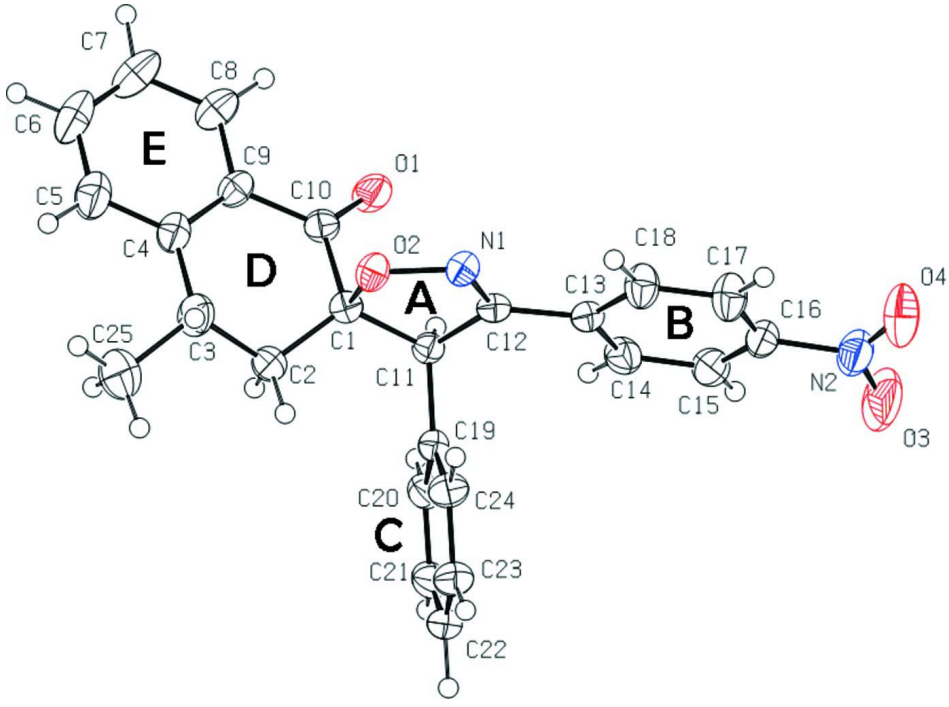

Figure 1

The molecular structure of (I), showing the crystallographic atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level.

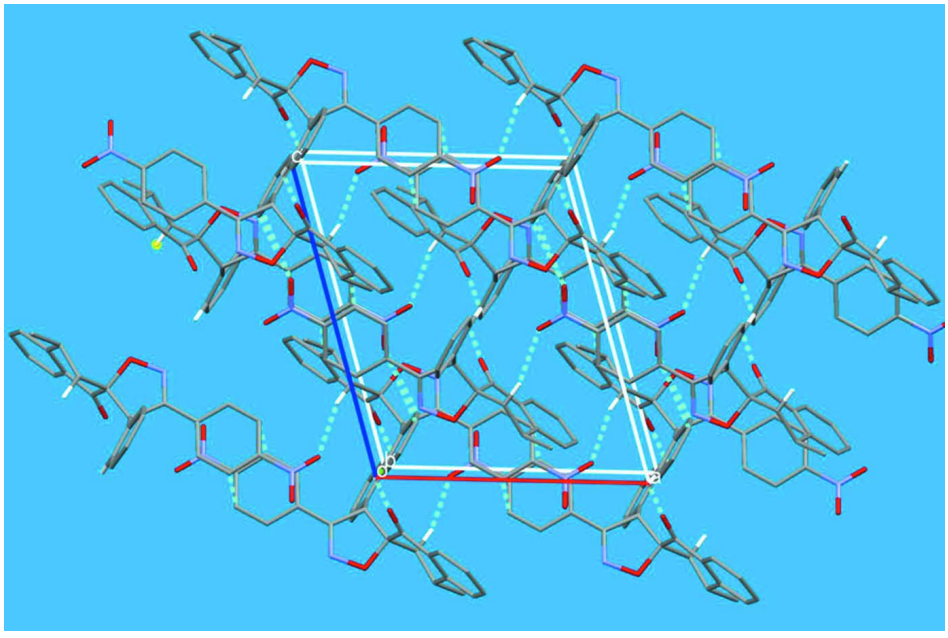

Figure 2

The crystal packing of (I), viewed down the *b* axis. The C—H···O contacts are shown as dashed lines. H atoms not involved in C—H···O contacts have been omitted for clarity.

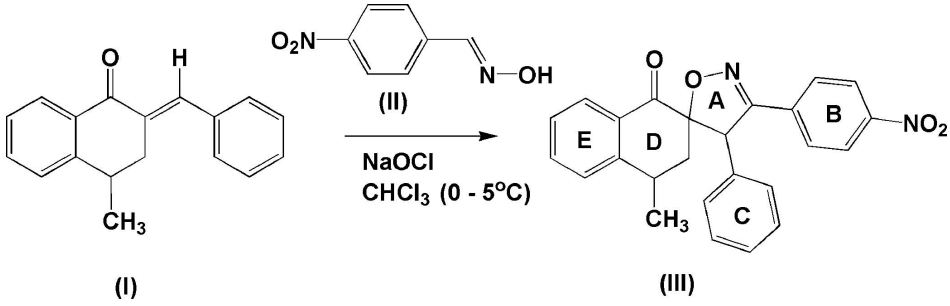

Figure 3

The formation of the title compound.

4′-Methyl-3-(4-nitrophenyl)-4-phenyl-4,5,1′,2′,3′,4′- hexahydrospiro[isoxazole-5,2′-naphthalen]-1′-one

Crystal data

$C_{25}H_{20}N_2O_4$
$M_r = 412.43$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 10.6567\ (15)$ Å
$b = 15.7071\ (16)$ Å
$c = 12.7259\ (15)$ Å
$\beta = 106.258\ (10)°$
$V = 2045.0\ (4)$ Å^3
$Z = 4$
$F(000) = 864$
$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 11994 reflections
$\theta = 1.7–29.4°$
$\mu = 0.09$ mm^{-1}
$T = 173$ K
Plate, colourless
0.40 × 0.37 × 0.27 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
20256 measured reflections
3798 independent reflections
2837 reflections with $I > 2\sigma(I)$
$R_{int} = 0.076$
$\theta_{max} = 25.5°$, $\theta_{min} = 2.1°$
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 19$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.123$
$S = 1.01$
3798 reflections
283 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0704P)^2 + 0.2039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.52$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0074 (14)

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

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 $F^2 > \sigma(F^2)$ 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	0.47898 (16)	0.55329 (9)	0.36239 (13)	0.0422 (5)
O2	0.32596 (13)	0.43601 (8)	0.17246 (10)	0.0297 (4)
O3	−0.1326 (2)	0.35498 (16)	0.60945 (19)	0.0861 (9)
O4	−0.26968 (17)	0.41232 (14)	0.47131 (16)	0.0632 (7)
N1	0.21389 (15)	0.44017 (10)	0.20955 (13)	0.0281 (5)
N2	−0.1598 (2)	0.38712 (13)	0.51976 (18)	0.0490 (8)
C1	0.44094 (18)	0.42092 (11)	0.26695 (15)	0.0248 (5)
C2	0.5302 (2)	0.36112 (12)	0.22914 (17)	0.0338 (7)
C3	0.5692 (2)	0.39637 (14)	0.13022 (19)	0.0415 (8)
C4	0.62172 (19)	0.48610 (13)	0.15022 (16)	0.0333 (6)
C5	0.7052 (2)	0.51874 (16)	0.09383 (19)	0.0464 (8)
C6	0.7473 (2)	0.60217 (18)	0.1075 (2)	0.0556 (9)
C7	0.7069 (3)	0.65532 (17)	0.1772 (2)	0.0541 (9)
C8	0.6247 (2)	0.62468 (14)	0.23505 (18)	0.0414 (7)
C9	0.58332 (19)	0.54037 (12)	0.22298 (15)	0.0295 (6)
C10	0.50110 (18)	0.50975 (12)	0.29134 (15)	0.0273 (6)

C11	0.38311 (18)	0.39083 (11)	0.35863 (15)	0.0239 (5)
C12	0.24282 (18)	0.41746 (11)	0.30968 (15)	0.0243 (5)
C13	0.13979 (19)	0.41296 (11)	0.36551 (16)	0.0268 (6)
C14	0.1696 (2)	0.39216 (13)	0.47532 (17)	0.0348 (6)
C15	0.0720 (2)	0.38411 (14)	0.52679 (18)	0.0395 (7)
C16	−0.0553 (2)	0.39756 (13)	0.46671 (18)	0.0352 (7)
C17	−0.0876 (2)	0.42136 (15)	0.35887 (19)	0.0424 (7)
C18	0.0101 (2)	0.42881 (14)	0.30805 (17)	0.0379 (7)
C19	0.39453 (18)	0.29696 (11)	0.38701 (15)	0.0247 (5)
C20	0.48296 (19)	0.26972 (12)	0.48285 (16)	0.0299 (6)
C21	0.4922 (2)	0.18425 (14)	0.50951 (18)	0.0388 (7)
C22	0.4143 (2)	0.12540 (13)	0.4410 (2)	0.0396 (7)
C23	0.3264 (2)	0.15185 (13)	0.34565 (19)	0.0395 (7)
C24	0.3160 (2)	0.23724 (12)	0.31901 (17)	0.0337 (6)
C25	0.6582 (3)	0.33530 (18)	0.0947 (2)	0.0652 (11)
H2A	0.48560	0.30570	0.20930	0.0410*
H2B	0.61000	0.35120	0.29010	0.0410*
H3	0.48710	0.39970	0.06850	0.0500*
H5	0.73390	0.48300	0.04490	0.0560*
H6	0.80480	0.62300	0.06830	0.0670*
H7	0.73530	0.71290	0.18550	0.0650*
H8	0.59630	0.66130	0.28330	0.0500*
H11	0.42200	0.42500	0.42630	0.0290*
H14	0.25810	0.38330	0.51580	0.0420*
H15	0.09240	0.36960	0.60220	0.0470*
H17	−0.17600	0.43250	0.31980	0.0510*
H18	−0.01110	0.44490	0.23320	0.0450*
H20	0.53740	0.30980	0.53040	0.0360*
H21	0.55270	0.16590	0.57560	0.0470*
H22	0.42150	0.06670	0.45970	0.0480*
H23	0.27270	0.11140	0.29810	0.0470*
H24	0.25450	0.25530	0.25340	0.0400*
H25A	0.62010	0.27810	0.08750	0.0980*
H25B	0.74370	0.33450	0.14950	0.0980*
H25C	0.66880	0.35350	0.02410	0.0980*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0591 (10)	0.0291 (7)	0.0472 (9)	−0.0119 (7)	0.0295 (8)	−0.0113 (7)
O2	0.0284 (7)	0.0377 (7)	0.0232 (7)	−0.0041 (6)	0.0074 (6)	0.0007 (5)
O3	0.0793 (15)	0.1206 (19)	0.0782 (15)	0.0189 (13)	0.0549 (13)	0.0445 (14)
O4	0.0351 (10)	0.0973 (14)	0.0626 (12)	−0.0111 (9)	0.0225 (9)	−0.0100 (10)
N1	0.0258 (9)	0.0308 (8)	0.0279 (9)	−0.0031 (6)	0.0080 (7)	0.0005 (7)
N2	0.0529 (14)	0.0509 (12)	0.0537 (13)	−0.0081 (10)	0.0320 (11)	−0.0020 (10)
C1	0.0249 (9)	0.0252 (9)	0.0233 (9)	−0.0006 (7)	0.0052 (8)	−0.0002 (7)
C2	0.0381 (12)	0.0280 (10)	0.0394 (12)	0.0007 (8)	0.0174 (10)	−0.0039 (9)
C3	0.0483 (14)	0.0440 (12)	0.0388 (13)	−0.0024 (10)	0.0229 (11)	−0.0049 (9)

C4	0.0285 (10)	0.0418 (11)	0.0302 (10)	−0.0004 (8)	0.0090 (9)	0.0048 (9)
C5	0.0398 (13)	0.0619 (15)	0.0413 (13)	0.0004 (11)	0.0177 (10)	0.0084 (11)
C6	0.0426 (14)	0.0763 (18)	0.0515 (15)	−0.0168 (12)	0.0191 (12)	0.0148 (13)
C7	0.0576 (16)	0.0547 (15)	0.0491 (15)	−0.0289 (12)	0.0134 (12)	0.0060 (12)
C8	0.0447 (13)	0.0410 (12)	0.0370 (12)	−0.0137 (10)	0.0091 (10)	0.0020 (9)
C9	0.0263 (10)	0.0333 (10)	0.0268 (10)	−0.0055 (8)	0.0039 (8)	0.0031 (8)
C10	0.0267 (10)	0.0266 (9)	0.0275 (10)	−0.0007 (7)	0.0058 (8)	−0.0012 (8)
C11	0.0247 (9)	0.0228 (9)	0.0241 (9)	−0.0021 (7)	0.0068 (8)	−0.0025 (7)
C12	0.0256 (10)	0.0192 (8)	0.0266 (10)	−0.0008 (7)	0.0051 (8)	0.0004 (7)
C13	0.0296 (10)	0.0221 (9)	0.0299 (10)	0.0002 (7)	0.0105 (8)	0.0013 (7)
C14	0.0343 (11)	0.0371 (11)	0.0330 (11)	0.0079 (9)	0.0096 (9)	0.0048 (9)
C15	0.0500 (14)	0.0390 (11)	0.0342 (12)	0.0073 (10)	0.0194 (10)	0.0086 (9)
C16	0.0361 (12)	0.0322 (10)	0.0428 (12)	−0.0045 (8)	0.0200 (10)	−0.0014 (9)
C17	0.0291 (11)	0.0594 (14)	0.0391 (12)	−0.0026 (10)	0.0103 (9)	−0.0002 (10)
C18	0.0284 (11)	0.0540 (13)	0.0305 (11)	0.0005 (9)	0.0069 (9)	0.0026 (9)
C19	0.0255 (9)	0.0250 (9)	0.0261 (9)	0.0005 (7)	0.0113 (8)	0.0003 (7)
C20	0.0279 (10)	0.0327 (10)	0.0297 (11)	0.0045 (8)	0.0090 (8)	0.0020 (8)
C21	0.0384 (12)	0.0413 (12)	0.0401 (12)	0.0150 (10)	0.0166 (10)	0.0130 (10)
C22	0.0469 (13)	0.0241 (10)	0.0578 (14)	0.0072 (9)	0.0310 (12)	0.0089 (10)
C23	0.0456 (13)	0.0269 (10)	0.0501 (14)	−0.0027 (9)	0.0201 (11)	−0.0021 (9)
C24	0.0357 (11)	0.0298 (11)	0.0338 (11)	−0.0027 (8)	0.0070 (9)	−0.0010 (8)
C25	0.080 (2)	0.0611 (16)	0.0703 (19)	0.0055 (14)	0.0470 (17)	−0.0079 (14)

Geometric parameters (Å, º)

O1—C10	1.208 (2)	C17—C18	1.376 (3)
O2—N1	1.403 (2)	C19—C20	1.384 (3)
O2—C1	1.476 (2)	C19—C24	1.387 (3)
O3—N2	1.207 (3)	C20—C21	1.381 (3)
O4—N2	1.226 (3)	C21—C22	1.378 (3)
N1—C12	1.275 (2)	C22—C23	1.373 (3)
N2—C16	1.464 (3)	C23—C24	1.380 (3)
C1—C2	1.508 (3)	C2—H2A	0.9900
C1—C10	1.531 (3)	C2—H2B	0.9900
C1—C11	1.539 (3)	C3—H3	1.0000
C2—C3	1.535 (3)	C5—H5	0.9500
C3—C4	1.512 (3)	C6—H6	0.9500
C3—C25	1.505 (4)	C7—H7	0.9500
C4—C5	1.388 (3)	C8—H8	0.9500
C4—C9	1.401 (3)	C11—H11	1.0000
C5—C6	1.380 (4)	C14—H14	0.9500
C6—C7	1.372 (4)	C15—H15	0.9500
C7—C8	1.379 (4)	C17—H17	0.9500
C8—C9	1.391 (3)	C18—H18	0.9500
C9—C10	1.477 (3)	C20—H20	0.9500
C11—C12	1.509 (3)	C21—H21	0.9500
C11—C19	1.515 (2)	C22—H22	0.9500
C12—C13	1.467 (3)	C23—H23	0.9500

C13—C14	1.383 (3)	C24—H24	0.9500
C13—C18	1.393 (3)	C25—H25A	0.9800
C14—C15	1.381 (3)	C25—H25B	0.9800
C15—C16	1.374 (3)	C25—H25C	0.9800
C16—C17	1.370 (3)		

N1—O2—C1	108.91 (13)	C20—C21—C22	120.5 (2)
O2—N1—C12	109.43 (15)	C21—C22—C23	119.8 (2)
O3—N2—O4	123.6 (2)	C22—C23—C24	119.9 (2)
O3—N2—C16	118.1 (2)	C19—C24—C23	120.73 (19)
O4—N2—C16	118.3 (2)	C1—C2—H2A	109.00
O2—C1—C2	107.10 (15)	C1—C2—H2B	109.00
O2—C1—C10	102.94 (14)	C3—C2—H2A	109.00
O2—C1—C11	104.46 (15)	C3—C2—H2B	109.00
C2—C1—C10	111.51 (16)	H2A—C2—H2B	108.00
C2—C1—C11	118.22 (15)	C2—C3—H3	107.00
C10—C1—C11	111.11 (15)	C4—C3—H3	107.00
C1—C2—C3	112.13 (16)	C25—C3—H3	106.00
C2—C3—C4	111.76 (18)	C4—C5—H5	119.00
C2—C3—C25	110.76 (19)	C6—C5—H5	119.00
C4—C3—C25	114.2 (2)	C5—C6—H6	120.00
C3—C4—C5	121.20 (19)	C7—C6—H6	120.00
C3—C4—C9	121.04 (18)	C6—C7—H7	120.00
C5—C4—C9	117.71 (19)	C8—C7—H7	120.00
C4—C5—C6	121.2 (2)	C7—C8—H8	120.00
C5—C6—C7	120.6 (2)	C9—C8—H8	120.00
C6—C7—C8	119.6 (2)	C1—C11—H11	109.00
C7—C8—C9	120.2 (2)	C12—C11—H11	109.00
C4—C9—C8	120.67 (19)	C19—C11—H11	109.00
C4—C9—C10	121.47 (17)	C13—C14—H14	120.00
C8—C9—C10	117.84 (18)	C15—C14—H14	120.00
O1—C10—C1	120.62 (18)	C14—C15—H15	121.00
O1—C10—C9	121.90 (18)	C16—C15—H15	121.00
C1—C10—C9	117.46 (16)	C16—C17—H17	121.00
C1—C11—C12	99.37 (15)	C18—C17—H17	121.00
C1—C11—C19	117.21 (15)	C13—C18—H18	120.00
C12—C11—C19	111.87 (16)	C17—C18—H18	120.00
N1—C12—C11	115.12 (17)	C19—C20—H20	120.00
N1—C12—C13	119.54 (18)	C21—C20—H20	120.00
C11—C12—C13	125.18 (16)	C20—C21—H21	120.00
C12—C13—C14	120.63 (19)	C22—C21—H21	120.00
C12—C13—C18	120.28 (18)	C21—C22—H22	120.00
C14—C13—C18	119.09 (19)	C23—C22—H22	120.00
C13—C14—C15	120.7 (2)	C22—C23—H23	120.00
C14—C15—C16	118.6 (2)	C24—C23—H23	120.00
N2—C16—C15	119.1 (2)	C19—C24—H24	120.00
N2—C16—C17	118.8 (2)	C23—C24—H24	120.00
C15—C16—C17	122.1 (2)	C3—C25—H25A	109.00

C16—C17—C18	118.9 (2)	C3—C25—H25B	109.00
C13—C18—C17	120.5 (2)	C3—C25—H25C	110.00
C11—C19—C20	120.03 (16)	H25A—C25—H25B	109.00
C11—C19—C24	121.02 (17)	H25A—C25—H25C	109.00
C20—C19—C24	118.94 (17)	H25B—C25—H25C	109.00
C19—C20—C21	120.07 (18)		

C1—O2—N1—C12	−9.41 (18)	C5—C6—C7—C8	0.8 (4)
N1—O2—C1—C2	142.07 (14)	C6—C7—C8—C9	0.2 (4)
N1—O2—C1—C10	−100.27 (15)	C7—C8—C9—C4	−1.6 (3)
N1—O2—C1—C11	15.89 (17)	C7—C8—C9—C10	176.8 (2)
O2—N1—C12—C11	−1.6 (2)	C4—C9—C10—O1	172.0 (2)
O2—N1—C12—C13	−177.30 (15)	C4—C9—C10—C1	−9.7 (3)
O3—N2—C16—C15	−10.8 (3)	C8—C9—C10—O1	−6.3 (3)
O3—N2—C16—C17	169.9 (2)	C8—C9—C10—C1	172.01 (18)
O4—N2—C16—C15	168.4 (2)	C1—C11—C12—N1	11.1 (2)
O4—N2—C16—C17	−10.9 (3)	C1—C11—C12—C13	−173.53 (16)
O2—C1—C2—C3	56.9 (2)	C19—C11—C12—N1	−113.37 (18)
C10—C1—C2—C3	−55.0 (2)	C19—C11—C12—C13	62.0 (2)
C11—C1—C2—C3	174.42 (17)	C1—C11—C19—C20	106.3 (2)
O2—C1—C10—O1	97.8 (2)	C1—C11—C19—C24	−74.7 (2)
O2—C1—C10—C9	−80.60 (19)	C12—C11—C19—C20	−139.91 (19)
C2—C1—C10—O1	−147.73 (19)	C12—C11—C19—C24	39.1 (3)
C2—C1—C10—C9	33.9 (2)	N1—C12—C13—C14	−177.13 (18)
C11—C1—C10—O1	−13.5 (3)	N1—C12—C13—C18	3.5 (3)
C11—C1—C10—C9	168.10 (16)	C11—C12—C13—C14	7.7 (3)
O2—C1—C11—C12	−15.17 (16)	C11—C12—C13—C18	−171.68 (18)
O2—C1—C11—C19	105.44 (17)	C12—C13—C14—C15	−177.04 (18)
C2—C1—C11—C12	−134.05 (17)	C18—C13—C14—C15	2.3 (3)
C2—C1—C11—C19	−13.4 (3)	C12—C13—C18—C17	177.39 (19)
C10—C1—C11—C12	95.16 (17)	C14—C13—C18—C17	−2.0 (3)
C10—C1—C11—C19	−144.23 (17)	C13—C14—C15—C16	−0.3 (3)
C1—C2—C3—C4	51.4 (2)	C14—C15—C16—N2	178.49 (19)
C1—C2—C3—C25	180.0 (2)	C14—C15—C16—C17	−2.2 (3)
C2—C3—C4—C5	155.9 (2)	N2—C16—C17—C18	−178.2 (2)
C2—C3—C4—C9	−26.9 (3)	C15—C16—C17—C18	2.6 (3)
C25—C3—C4—C5	29.1 (3)	C16—C17—C18—C13	−0.4 (3)
C25—C3—C4—C9	−153.6 (2)	C11—C19—C20—C21	179.13 (19)
C3—C4—C5—C6	176.3 (2)	C24—C19—C20—C21	0.1 (3)
C9—C4—C5—C6	−1.1 (3)	C11—C19—C24—C23	−179.6 (2)
C3—C4—C9—C8	−175.4 (2)	C20—C19—C24—C23	−0.6 (3)
C3—C4—C9—C10	6.3 (3)	C19—C20—C21—C22	0.3 (3)
C5—C4—C9—C8	2.0 (3)	C20—C21—C22—C23	−0.3 (3)
C5—C4—C9—C10	−176.32 (19)	C21—C22—C23—C24	−0.3 (3)
C4—C5—C6—C7	−0.3 (4)	C22—C23—C24—C19	0.7 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*B*···O4^{i}	0.99	2.50	3.319 (3)	140
C20—H20···O1^{ii}	0.95	2.58	3.365 (2)	140

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