

Table 2. Bond distances (Å) and angles (°)

C2—C1	1.525 (8)	C6—C1	1.574 (7)
C8—C1	1.548 (6)	C3—C2	1.521 (8)
C4—C3	1.537 (8)	C5—C4	1.537 (8)
C6—C5	1.554 (8)	C7—C6	1.522 (7)
C8—C7	1.533 (7)	O21—C7	1.421 (6)
C22—C7	1.455 (8)	N9—C8	1.476 (6)
C10—N9	1.474 (7)	S11—N9	1.646 (4)
C19—C14	1.378 (7)	O12—S11	1.438 (4)
C17—C16	1.388 (8)	O13—S11	1.434 (4)
C17—C18	1.402 (8)	C14—S11	1.776 (5)
C20—C17	1.515 (8)	C15—C14	1.389 (7)
C22—O21	1.457 (8)	C16—C15	1.378 (8)
		C19—C18	1.391 (8)
C6—C1—C2	118.9 (5)	C8—C1—C2	119.9 (5)
C8—C1—C6	87.2 (4)	C3—C2—C1	113.3 (5)
C4—C3—C2	110.1 (6)	C5—C4—C3	109.5 (5)
C6—C5—C4	111.8 (5)	C5—C6—C1	112.5 (5)
C7—C6—C1	86.8 (4)	C7—C6—C5	112.6 (5)
C8—C7—C6	89.6 (4)	O21—C7—C6	126.5 (5)
O21—C7—C8	124.9 (5)	C22—C7—C6	129.0 (6)
C22—C7—C8	130.2 (5)	C22—C7—O21	60.9 (4)
C7—C8—C1	87.4 (4)	N9—C8—C1	122.8 (4)
N9—C8—C7	117.8 (4)	C10—N9—C8	118.1 (4)
S11—N9—C8	117.9 (3)	S11—N9—C10	116.9 (4)
O12—S11—N9	106.6 (2)	O13—S11—N9	106.5 (2)
O13—S11—O12	120.4 (3)	C14—S11—N9	107.0 (2)
C14—S11—O12	108.3 (3)	C14—S11—O13	107.5 (3)
C15—C14—S11	119.4 (5)	C19—C14—S11	119.9 (4)
C19—C14—C15	120.7 (5)	C16—C15—C14	119.1 (6)
C17—C16—C15	121.9 (6)	C18—C17—C16	117.9 (6)
C20—C17—C16	121.4 (6)	C20—C17—C18	120.7 (6)
C19—C18—C17	120.8 (6)	C18—C19—C14	119.6 (5)
C22—O21—C7	60.7 (4)	O21—C22—C7	58.4 (4)

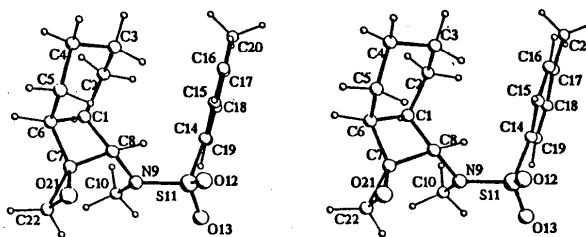


Fig. 1. Stereoscopic view of the molecule with atom numbering.

Related literature. In a preceding paper we determined the structure of 8-(*N*-methyl-*N*-*p*-tolylsulfonfylamino)bicyclo[4.2.0]octan-7-one (Tinant, Declercq & Gobeaux, 1990). In this compound the configuration at C8 is also *exo* and the puckering in the cyclobutane ring is 31 (1) and 29 (1)° in the two independent molecules, respectively.

JFD and JPD thank the Fonds de Développement Scientifique Catholic University of Louvain for its financial support.

References

- COREY, E. J. & CHAYKOVSKY, M. (1965). *J. Am. Chem. Soc.* **87**, 1353–1364.
- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1985). *SHELXS86*. In *Crystallographic Computing 3*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
- TINANT, B., DECLERCQ, J.-P. & GOBEAUX, B. (1990). *Acta Cryst.* **C46**, 1944–1946.

The atomic parameters are given in Table 1.* Fig. 1 is a stereoscopic view of the molecule showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55612 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1011]

Acta Cryst. (1993). **C49**, 562–565

Structure of the *p*-Nitrobenzoate Ester of 3-*endo*-(*p*-Methoxybenzyl)isoborneol

BY IL-HWAN SUH,* BYUNG-ICK SEO, DAVID E. LEWIS* AND WILLIAM P. JENSEN

Department of Chemistry, South Dakota State University, Brookings, SD 57007, USA

AND ROBERT A. JACOBSON

Ames Laboratory, USDOE and Department of Chemistry, Iowa State University, Ames IA 50011, USA

(Received 28 February 1992; accepted 21 July 1992)

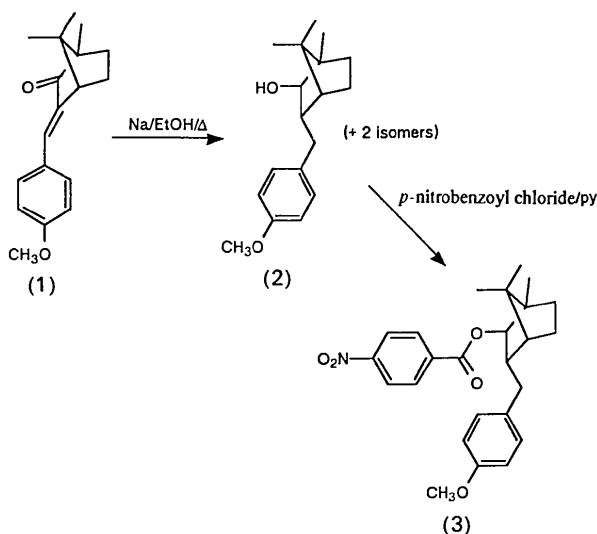
Abstract. (1*R*,2*R*,3*S*,4*R*)-3-[(4-Methoxyphenyl)methyl]-1,7,7-trimethylbicyclo[2.2.1]hept-2-yl 4-nitro-

benzoate, C₂₅H₂₉NO₅, *M_r* = 423.5, orthorhombic, *P*2₁2₁2₁, *a* = 12.104 (3), *b* = 26.712 (5), *c* = 7.138 (1) Å, *V* = 2308.0 (8) Å³, *Z* = 4, *D_x* = 1.22 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 0.8 cm⁻¹,

* To whom correspondence should be addressed.

$F(000) = 904$, room temperature, $R = 0.065$ for 1421 observed out of 1770 reflections. Maximum deviations from planarity of the two aromatic rings are 0.018 Å each; the dihedral angle formed by the two rings is 29.5 (3)°, with a distance of closest approach of 3.827 (8) Å. The X-ray structure determination establishes the orientation of the aromatic rings and confirms the steric shielding of the *Si* face of the ester carbonyl group.

Experimental. Bouveault–Blanc reduction (House, 1972) of 3-(*p*-methoxybenzylidene)camphor (1) gives three stereoisomeric alcohols (2) which were converted to their *p*-nitrobenzoates. Fractional crystallization of the 3-*endo*-(*p*-methoxybenzyl)isobornyl *p*-nitrobenzoate from the mixture of esters gave the desired compound (3) stereochemically homogeneous (Seo, Wall, Lee, Buttrum & Lewis, 1993; Wall, 1990).



A colorless crystal of (3) was obtained by slow evaporation of ethanol. Crystal size was 0.4 × 0.4 × 0.5 mm. Space group was determined from systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd. Cell dimensions were obtained from setting angles of nine reflections with $10 < \theta < 12^\circ$. Data were collected on a Rigaku AFC-6R diffractometer, with Mo $K\alpha$ radiation and graphite monochromator; scan technique was ω - 2θ . One octant of reflections having $2 < 2\theta < 50^\circ$, corresponding to h 0 to 14, k 0 to 31, l 0 to 8, was collected. Of 1770 reflections collected, 1421 with $I > 2.5\sigma(I)$ were observed and used in the structure solution. Intensities of three standard reflections (0, $\bar{1}$, 0, 124 and 600), monitored every 6000 s, showed no systematic variation; no decay correction was applied. Data were corrected for Lorentz and polarization effects, but not for absorption. The structure was solved by

direct methods using the PC version of *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). The *E* map revealed most of the non-H atoms; the handedness corresponded to the known natural enantiomorph. All the remaining atoms except the methyl H atoms were revealed by difference map, positions of one H atom of each methyl group were obtained by difference map and the positions of the remaining H atoms were calculated. Refinement was by full-matrix least-squares methods initially with isotropic thermal parameters for all atoms, and then with anisotropic thermal parameters for all atoms. In the final rounds of calculations, a secondary-extinction coefficient of 0.020 (1) mm was applied and the positions of the H atoms were fixed on geometric grounds (C—H 1.08 Å). H atoms were assigned the same anisotropic thermal parameters as the corresponding C atoms; H-atom anisotropic thermal parameters were not refined. The final cycles of least-squares refinement included 60 atoms and 281 parameters. $R = 0.065$, $wR = 0.063$, goodness of fit = 2.41 and $w = 1/\sigma^2(F_o)$. The maximum shift/e.s.d. ratio in the final cycle was 0.031; the final difference map showed no chemically significant features and no peak higher than 0.22 e \AA^{-3} . Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Atomic coordinates are given in Table 1.* Details of the molecular geometry are given in Table 2. Fig. 1 is a view of the molecule prepared using *ORTEPII* (Johnson, 1976).

Related literature. Camphor-based chiral auxiliaries have proved to be very powerful chiral adjuvants in organic synthesis (Helmchen & Wierschokuski, 1984; Oppolzer, 1987). The Bouveault–Blanc reduction of the benzylidenecamphors and fractional crystallization of the *p*-nitrobenzoate ester of the 3-*endo*-2-*exo* isomer provides multigram quantities of the 3-*endo*-arylmethylisoborneol, which we propose to use as a chiral auxiliary and as a platform for the development of chiral reagents. This procedure is simpler than the method of Richer & Rossi (1972). The compound adopts a conformation in the crystal in which the planes of two aromatic rings are as close to parallel as possible; the key bond torsion angles are C1—C2—O1—C18 109.3 (6), C4—C3—C11—C12 178.1 (7) and C3—C11—C12—C17 115.4 (7)°. This conformation cannot be explained on the basis of crystal packing constraints because the two close approaches between molecules occur in a region

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55592 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1009]

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²)

B_{eq} is the mean of the principal axes of the anisotropic mean square displacement tensor.

	x	y	z	B_{eq}
O1	0.9083 (3)	0.8512 (2)	0.2180 (6)	5.0 (2)
O2	1.1270 (5)	1.0243 (2)	-0.1126 (9)	8.9 (3)
O3	1.0227 (3)	0.8349 (2)	-0.0230 (7)	6.3 (2)
O4	1.3341 (4)	0.8948 (2)	0.8159 (8)	7.7 (3)
O5	1.4519 (4)	0.8715 (2)	0.6089 (7)	7.2 (3)
N	1.3565 (5)	0.8790 (2)	0.6596 (9)	5.7 (3)
C1	0.7474 (5)	0.7956 (2)	0.1604 (8)	4.9 (3)
C2	0.8089 (4)	0.8441 (2)	0.1038 (8)	4.2 (3)
C3	0.7273 (4)	0.8864 (2)	0.1512 (9)	4.5 (3)
C4	0.6215 (4)	0.8569 (2)	0.2082 (9)	4.8 (3)
C5	0.5758 (5)	0.8303 (3)	0.035 (1)	5.9 (3)
C6	0.6627 (5)	0.7886 (2)	-0.000 (1)	5.6 (3)
C7	0.6704 (5)	0.8133 (2)	0.3247 (9)	5.0 (3)
C8	0.7319 (5)	0.8290 (3)	0.4997 (9)	6.5 (4)
C9	0.5862 (6)	0.7733 (3)	0.3859 (9)	7.0 (4)
C10	0.8205 (5)	0.7505 (3)	0.195 (1)	6.8 (4)
C11	0.7144 (5)	0.9259 (2)	-0.004 (1)	5.5 (3)
C12	0.8218 (5)	0.9526 (2)	-0.040 (1)	5.2 (3)
C13	0.8733 (7)	0.9811 (3)	0.099 (1)	7.4 (4)
C14	0.9717 (7)	1.0056 (3)	0.070 (1)	7.6 (4)
C15	1.0272 (6)	1.0006 (3)	-0.097 (1)	6.8 (4)
C16	0.9798 (6)	0.9733 (3)	-0.241 (1)	6.6 (4)
C17	0.8773 (6)	0.9493 (3)	-0.206 (1)	6.2 (4)
C18	1.0074 (5)	0.8452 (2)	0.141 (1)	4.7 (3)
C19	1.0971 (5)	0.8532 (2)	0.2796 (9)	4.6 (3)
C20	1.2068 (5)	0.8497 (3)	0.2205 (9)	5.6 (3)
C21	1.2918 (5)	0.8574 (3)	0.346 (1)	5.6 (4)
C22	1.2664 (5)	0.8696 (2)	0.5280 (9)	4.3 (3)
C23	1.1596 (5)	0.8726 (3)	0.5926 (9)	5.2 (3)
C24	1.0745 (5)	0.8640 (2)	0.4655 (9)	4.9 (3)
C25	1.1878 (6)	1.0178 (3)	-0.283 (2)	9.4 (5)

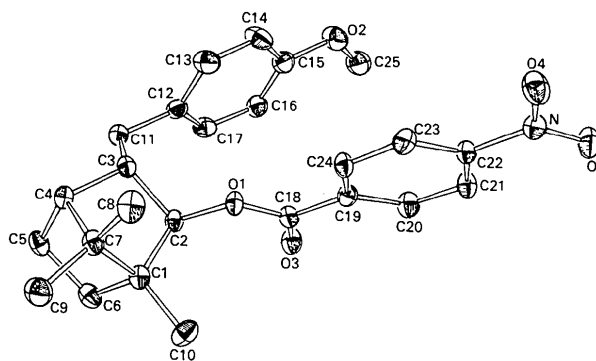


Fig. 1. ORTEPII (Johnson, 1976) view of 3-endo-(*p*-methoxybenzyl)isobornyl *p*-nitrobenzoate. Thermal ellipsoids are scaled to 50% probability.

remote from the critical atoms (C3 and C11). The C6-endo H atom of one molecule approaches the C8 methyl H atoms of the adjacent molecule at $1 + z$ at 2.09 (2) Å, significantly less than the sum of the van der Waals radii of the H atoms (Pauling, 1960). The ¹H NMR spectrum of the ester is consistent with the view that the solution conformation of the molecule is identical to the crystal conformation. ¹H NMR analysis of the conformations of the alcohol and its esters (Seo, Suh, Jensen, Lewis, Wall & Jacobson, 1992) reveals that esterification of the hydroxyl group causes a conformational change such that the *p*-methoxyphenyl group moves from a position where the C4—C3—C11—C12 torsion angle is approximately 120° to a position where it is approximately 180°, a change which we attribute to the favorable through-space interaction between the ester carbonyl group and the electron-rich *p*-methoxyphenyl ring. This is confirmed by the X-ray crystal structure determination.

The authors gratefully acknowledge the financial support of this work, in part, by the Robert A. Welch Foundation (DEL; Grant #AA-936), the National Science Foundation (DEL; Grant #STI-8902066) and the US Department of Energy, Office of Basic Energy Sciences, Materials Sciences Division (RAJ; contract W-74-5-Eng-82).

References

- GABE, E. J., LE PAGE, Y., CHARLAND, J.-P., LEE, F. L. & WHITE, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
 HELMCHEN, G. & WIERSCHOKUSKI, R. (1984). *Angew. Chem. Int. Ed. Engl.* **20**, 60–61.
 HOUSE, H. O. (1972). *Modern Synthetic Reactions*, 2nd ed., pp. 152–160. New York: Benjamin.
 JOHNSON, C. K. (1976). ORTEPII. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
 OPPOLZER, W. (1987). *Tetrahedron*, **43**, 1969–2004, and references therein.
 PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed., pp. 257–264. Ithaca, New York: Cornell Univ. Press.

Table 2. Bond distances (Å) and bond angles (°)

O1—C2	1.468 (7)	C5—C6	1.55 (1)
O1—C18	1.328 (7)	C7—C8	1.513 (9)
O2—C15	1.369 (9)	C7—C9	1.540 (9)
O2—C25	1.43 (1)	C11—C12	1.504 (9)
O3—C18	1.217 (8)	C12—C13	1.40 (1)
O4—N	1.223 (9)	C12—C17	1.37 (1)
O5—N	1.227 (8)	C13—C14	1.37 (1)
N—C22	1.461 (9)	C14—C15	1.38 (1)
C1—C2	1.549 (9)	C15—C16	1.39 (1)
C1—C6	1.548 (9)	C16—C17	1.42 (1)
C1—C7	1.571 (8)	C18—C19	1.484 (9)
C1—C10	1.514 (9)	C19—C20	1.396 (8)
C2—C3	1.539 (8)	C19—C24	1.385 (9)
C3—C4	1.558 (8)	C20—C21	1.38 (1)
C3—C11	1.54 (1)	C21—C22	1.38 (1)
C4—C5	1.53 (1)	C22—C23	1.375 (9)
C4—C7	1.548 (9)	C23—C24	1.392 (9)
C2—O1—C18	119.6 (5)	C4—C7—C9	114.9 (5)
C15—O2—C25	117.8 (7)	C8—C7—C9	106.5 (5)
O4—N—O5	122.3 (6)	C3—C11—C12	111.0 (5)
O4—N—C22	118.7 (6)	C11—C12—C13	121.6 (6)
O5—N—C22	119.0 (6)	C11—C12—C17	122.9 (7)
C2—C1—C6	102.9 (5)	C13—C12—C17	115.5 (6)
C2—C1—C7	103.3 (5)	C12—C13—C14	122.9 (7)
C2—C1—C10	115.2 (5)	C13—C14—C15	120.4 (7)
C6—C1—C7	101.3 (5)	O2—C15—C14	117.1 (8)
C6—C1—C10	114.2 (5)	O2—C15—C16	123.3 (8)
C7—C1—C10	117.8 (5)	C14—C15—C16	119.6 (7)
O1—C2—C1	110.7 (5)	C15—C16—C17	117.9 (7)
O1—C2—C3	107.9 (4)	C12—C17—C16	123.8 (7)
C1—C2—C3	104.2 (4)	O1—C18—O3	124.2 (6)
C2—C3—C4	102.4 (5)	O1—C18—C19	111.6 (5)
C2—C3—C11	114.1 (5)	O3—C18—C19	124.2 (6)
C4—C3—C11	116.9 (5)	C18—C19—C20	119.0 (6)
C3—C4—C5	108.7 (5)	C18—C19—C24	121.6 (5)
C3—C4—C7	101.9 (4)	C20—C19—C24	119.4 (6)
C5—C4—C7	103.0 (5)	C19—C20—C21	120.2 (6)
C4—C5—C6	102.6 (5)	C20—C21—C22	118.8 (6)
C1—C6—C5	104.0 (5)	N—C22—C21	118.8 (6)
C1—C7—C4	93.0 (5)	N—C22—C23	118.4 (6)
C1—C7—C8	114.1 (5)	C21—C22—C23	122.8 (6)
C1—C7—C9	113.2 (5)	C22—C23—C24	117.9 (6)
C4—C7—C8	115.0 (5)	C19—C24—C23	120.8 (6)

RICHER, J.-C. & ROSSI, A. (1972). *Can. J. Chem.* **50**, 1376–1385.
 SEO, B.-I., SUH, I.-H., JENSEN, W. P., LEWIS, D. E., WALL, L. K. &
 JACOBSON, R. A. (1992). *Tetrahedron Asymmetry*, **3**, 367–370.

SEO, B.-I., WALL, L. K., LEE, H., BUTTRUM, J. & LEWIS, D. E.
 (1993). *Synth. Commun.* **23**, In the press.
 WALL, L. K. (1990). MS Thesis. Baylor Univ., Waco, Texas, USA.

Acta Cryst. (1993). **C49**, 565–567

Structure of a Highly Potent Anxiolytic Dipeptoid

BY T. DEBAERDEMAEKER

Sektion für Röntgen- und Elektronenbeugung, Universität Ulm, D-7900 Ulm, Germany

AND T. HEIGL AND K. STEINER

Chemische Forschung und Entwicklung, GÖDECKE AG, D-7800 Freiburg, Germany

(Received 30 November 1991; accepted 28 May 1992)

Abstract. [R-(R*,R*)]-4-[[2-[[3-(1*H*-Indol-3-yl)-2-methyl-1-oxo-2-[[tricyclo[3.3.1.1^{3,7}]dec-2-yloxy)carbonyl]amino]propyl]amino]-1-phenylethyl]amino]-4-oxobutanoic acid, C₃₅H₄₂N₄O₆, *M_r* = 614.748, orthorhombic, *P*2₁2₁2₁, *a* = 13.341 (2), *b* = 13.643 (2), *c* = 18.222 (2) Å, *V* = 3316.61 Å³, *Z* = 4, *D_m* = 1.24, *D_x* = 1.23 Mg m⁻³, Mo *K*α radiation, λ = 0.71069 Å, μ = 0.050 mm⁻¹, *F*(000) = 1312, room temperature, *R* = 0.0497 for 5121 observed reflections. The analysis confirms the expected (1*R*,17*R*) configuration. In addition, four hydrogen bonds (one intra- and three intermolecular) are identified.

Experimental. The dipeptoid (internal code CI-988, free acid) was synthesized in a ten-step synthesis starting from D-(–)-2-amino-2-phenylethanol and D-α-methyltryptophan methyl ester according to a procedure developed by Horwell *et al.* (1991). Prismatic colourless crystals (0.2 × 0.2 × 0.4 mm) were obtained by dissolving 300 mg of CI-988 (free acid) in 60 ml ethyl acetate at 348 K. The solution was cooled slowly from 345 to 293 K during 108 h (LAUDA RKS/RK20, LAUDA PM351). The crystals were dried in vacuum at 303 K.

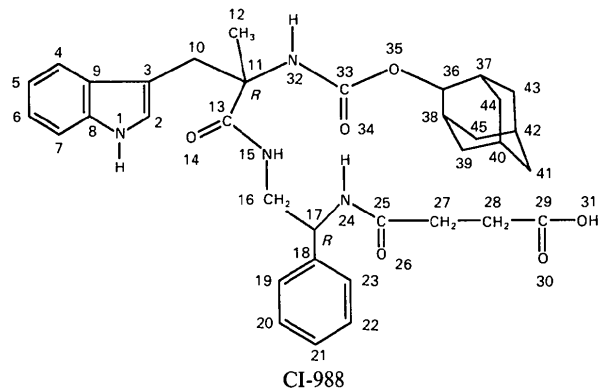


Table 1. Final fractional coordinates and equivalent isotropic thermal parameters (Å²)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
N(1)	0.6931 (1)	-0.2760 (1)	0.9902 (1)	0.055 (2)
C(2)	0.6200 (2)	-0.2078 (1)	0.9742 (1)	0.050 (2)
C(3)	0.5580 (1)	-0.1952 (1)	1.0330 (1)	0.038 (1)
C(4)	0.5633 (1)	-0.2832 (1)	1.1602 (1)	0.045 (2)
C(5)	0.6150 (2)	-0.3526 (2)	1.2001 (1)	0.057 (2)
C(6)	0.7005 (2)	-0.3999 (2)	1.1697 (1)	0.060 (2)
C(7)	0.7316 (1)	-0.3799 (1)	1.1005 (1)	0.054 (2)
C(8)	0.6794 (1)	-0.3094 (1)	1.0599 (1)	0.043 (2)
C(9)	0.5947 (1)	-0.2600 (1)	1.0894 (1)	0.037 (1)
C(10)	0.4695 (1)	-0.1275 (1)	1.0396 (1)	0.040 (1)
C(11)	0.4990 (1)	-0.0210 (1)	1.0601 (1)	0.034 (1)
C(12)	0.5671 (1)	-0.0197 (1)	1.1283 (1)	0.043 (2)
C(13)	0.4049 (1)	0.0396 (1)	1.0782 (1)	0.035 (1)
O(14)	0.3238 (1)	0.0003 (1)	1.0938 (1)	0.049 (1)
N(15)	0.4184 (1)	0.1371 (1)	1.0815 (1)	0.039 (1)
C(16)	0.3365 (2)	0.2039 (1)	1.1002 (1)	0.045 (2)
C(17)	0.3078 (1)	0.2711 (1)	1.0356 (1)	0.042 (2)
C(18)	0.3931 (2)	0.3438 (1)	1.0187 (1)	0.054 (2)
C(19)	0.4023 (4)	0.4350 (2)	1.0572 (2)	0.095 (5)
C(20)	0.4917 (4)	0.4924 (3)	1.0421 (3)	0.103 (5)
C(21)	0.5593 (3)	0.4657 (4)	0.9947 (2)	0.096 (5)
C(22)	0.5512 (2)	0.3826 (3)	0.9578 (2)	0.087 (4)
C(23)	0.4688 (2)	0.3211 (2)	0.9697 (2)	0.064 (3)
N(24)	0.2814 (1)	0.2130 (1)	0.9716 (1)	0.038 (1)
C(25)	0.2097 (1)	0.2400 (1)	0.9245 (1)	0.044 (2)
O(26)	0.1555 (1)	0.3120 (1)	0.9348 (1)	0.069 (2)
C(27)	0.2001 (2)	0.1786 (1)	0.8559 (1)	0.047 (2)
C(28)	0.2087 (3)	0.2440 (2)	0.7895 (1)	0.066 (3)
C(29)	0.2075 (2)	0.1938 (1)	0.7158 (1)	0.045 (2)
O(30)	0.2255 (2)	0.2377 (1)	0.6597 (1)	0.079 (2)
O(31)	0.1836 (2)	0.1010 (1)	0.7177 (1)	0.069 (2)
N(32)	0.5546 (1)	0.0246 (1)	0.9995 (1)	0.037 (1)
C(33)	0.5085 (1)	0.0505 (1)	0.9369 (1)	0.036 (1)
O(34)	0.4179 (1)	0.0441 (1)	0.9261 (0)	0.039 (1)
O(35)	0.5745 (1)	0.0871 (1)	0.8877 (0)	0.048 (1)
C(36)	0.5323 (1)	0.1159 (1)	0.8160 (1)	0.045 (2)
C(37)	0.6115 (3)	0.1803 (2)	0.7792 (1)	0.067 (3)
C(38)	0.5109 (1)	0.0262 (2)	0.7690 (1)	0.052 (2)
C(39)	0.6079 (2)	-0.0295 (2)	0.7528 (2)	0.068 (3)
C(40)	0.6824 (2)	0.0369 (2)	0.7129 (1)	0.064 (3)
C(41)	0.6374 (2)	0.0731 (3)	0.6413 (1)	0.071 (3)
C(42)	0.5431 (2)	0.1295 (3)	0.6572 (1)	0.072 (3)
C(43)	0.5634 (3)	0.2165 (2)	0.7063 (2)	0.087 (4)
C(44)	0.7059 (2)	0.1240 (3)	0.7631 (1)	0.073 (3)
C(45)	0.4664 (2)	0.0630 (3)	0.6963 (1)	0.081 (4)

D_m was determined by the flotation method. Intensity data were collected with an automatic Philips PW1100 four-circle diffractometer with graphite monochromator, using 2θ scans to 2θ_{max} = 50°. Cell parameters were determined by least-squares fit of 88