Supplementary Material (ESI) for Chemical Communications

X-ray Structure Report

March 2, 2009

Experimental

Data Collection

A colorless unknown crystal of $C_{28}H_{33}NO_3$ having approximate dimensions of 0.68 x 0.46 x 0.27 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Cu-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 90 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 16.2710(3) Å
b = 8.83139(16) Å β = 102.9857(8)^o
c = 17.4864(3) Å
V = 2448.45(8) Å^3

For Z = 4 and F.W. = 431.57, the calculated density is 1.171 g/cm^3. The systematic absences of:

h0l: l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of 23 ± 1^{o}C to a maximum 2θ value of 136.4^o. A total of 30 oscillation images were collected. A sweep of data was done using ω scans from 80.0 to 260.0^o in 30.0^o step, at χ=54.0^o and ϕ = 0.0^o. The exposure rate was 360.0 [sec./^{o}]. A second sweep was performed using ω scans from 80.0 to 260.0^o in 30.0^o step, at χ=54.0^o and ϕ = 90.0^o. The exposure rate was 360.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^o in 30.0^o step, at χ=54.0^o and ϕ = 180.0^o. The exposure rate was 360.0 [sec./^{o}]. Another sweep was performed using

ω scans from 80.0 to 260.0^{o} in 30.0^{o} step, at χ=54.0^{o} and ϕ = 270.0^{o}. The exposure rate was 360.0 [sec./^{o}]. Another sweep was performed using ω scans from 80.0 to 260.0^{o} in 30.0^{o} step, at χ=0.0^{o} and ϕ = 0.0^{o}. The exposure rate was 360.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 23572 reflections that were collected, 4462 were unique (R_{int} = 0.050); equivalent reflections were merged.

The linear absorption coefficient, μ, for Cu-Kα radiation is 5.930 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.608 to 0.852. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction[1] was applied (coefficient = 299.450012).

Structure Solution and Refinement

The structure was solved by direct methods[2] and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[4] on F^2 was based on 4462 observed reflections and 323 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0500$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1743$$

The standard deviation of an observation of unit weight[5] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w (|Fo| - |Fc|)^2 versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.29 and -0.27 e$^-$/Å^3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10,11]

crystallographic software package.

References

(1) Larson, A.C. (1970), Crystallographic Computing, 291-294. F.R. Ahmed, ed. Munksgaard, Copenhagen (equation 22, with V replaced by the cell volume).

(2) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(3) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(5) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D.,

Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{28}H_{33}NO_3$
Formula Weight	431.57
Crystal Color, Habit	colorless, unknown
Crystal Dimensions	0.68 X 0.46 X 0.27 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 16.2710(3) Å b = 8.83139(16) Å c = 17.4864(3) Å β = 102.9857(8) ^{o} V = 2448.45(8) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.171 g/cm^3
F_{000}	928.00
μ(CuKα)	5.930 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	CuKα (λ = 1.54187 Å) graphite monochromated
Detector Aperture	460 mm x 256 mm
Data Images	30 exposures
ω oscillation Range (χ=54.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	360.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=90.0)	80.0 - 260.0^{o}
Exposure Rate	360.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=180.0)	80.0 - 260.0^{o}
Exposure Rate	360.0 sec./^{o}
ω oscillation Range (χ=54.0, ϕ=270.0)	80.0 - 260.0^{o}
Exposure Rate	360.0 sec./^{o}
ω oscillation Range (χ=0.0, ϕ=0.0)	80.0 - 260.0^{o}
Exposure Rate	360.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	136.4^{o}
No. of Reflections Measured	Total: 23572 Unique: 4462 (R_{int} = 0.050)

Corrections

Lorentz-polarization
Absorption
(trans. factors: 0.608 - 0.852)
Secondary Extinction
(coefficient: 2.99450e+002)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0030Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	136.4^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4462
No. Variables	323
Reflection/Parameter Ratio	13.81
Residuals: R1 (I>2.00σ(I))	0.0500
Residuals: R (All reflections)	0.0613
Residuals: wR2 (All reflections)	0.1743
Goodness of Fit Indicator	1.004
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.29 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.27 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
O(2)	0.14744(8)	0.34684(14)	0.42940(7)	4.67(2)
O(8)	0.07675(9)	-0.08820(17)	0.28334(8)	5.84(3)
O(16)	0.02695(10)	-0.2144(2)	0.37454(9)	6.64(4)
N(1)	0.22600(8)	0.18863(15)	0.52367(7)	3.62(2)
C(3)	0.20971(10)	0.22697(19)	0.44141(9)	3.84(3)
C(4)	0.12151(10)	-0.0055(2)	0.41288(10)	4.10(3)
C(5)	0.19538(10)	0.0244(2)	0.62518(9)	4.04(3)
C(6)	0.23167(11)	0.34173(19)	0.55916(9)	4.03(3)
C(7)	0.18156(11)	0.09766(19)	0.38450(9)	4.07(3)
C(9)	0.16516(12)	0.4368(2)	0.49972(10)	4.61(3)
C(10)	0.31393(11)	0.4234(2)	0.56226(9)	4.15(3)
C(11)	0.29994(13)	0.5609(2)	0.52293(11)	4.72(4)
C(12)	0.15743(10)	0.1029(2)	0.54813(9)	3.94(3)
C(13)	0.11317(10)	-0.0048(2)	0.48653(10)	4.24(3)
C(14)	0.25566(12)	-0.0870(2)	0.62759(11)	4.65(4)
C(15)	0.39562(12)	0.3772(2)	0.60014(10)	4.53(3)
C(17)	0.25860(12)	0.0083(2)	0.37060(12)	5.01(4)
C(18)	0.06979(12)	-0.1156(2)	0.35677(11)	4.77(4)
C(19)	0.36698(16)	0.6564(2)	0.52006(13)	5.74(5)
C(20)	0.41228(12)	0.2241(2)	0.64006(12)	5.09(4)
C(21)	0.46098(14)	0.4763(2)	0.59723(13)	5.59(4)
C(22)	0.20793(14)	0.5869(2)	0.48938(12)	5.37(4)
C(23)	0.17273(14)	0.0646(2)	0.69419(11)	5.36(4)
C(24)	0.44744(16)	0.6137(2)	0.55831(14)	6.15(5)
C(25)	0.29355(14)	-0.1558(2)	0.69713(13)	5.86(5)
C(26)	0.03064(15)	-0.1853(3)	0.22104(13)	6.73(5)
C(27)	0.21079(17)	-0.0039(3)	0.76396(12)	6.78(6)
C(28)	0.27113(17)	-0.1135(2)	0.76599(13)	6.63(5)
C(29)	0.44486(19)	0.1131(3)	0.58792(18)	7.70(7)
C(30)	0.05824(16)	-0.1490(4)	0.14976(14)	8.67(8)
C(31)	0.47121(15)	0.2339(3)	0.72044(15)	7.68(6)
C(32)	0.3042(2)	0.0815(3)	0.3153(2)	9.51(9)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.5166	0.4479	0.6231	6.66
H(2)	0.0773	-0.0788	0.5012	4.99
H(3)	0.2707	-0.1159	0.5802	5.49
H(4)	0.3575	0.7492	0.4920	7.07
H(5)	0.4939	0.6785	0.5575	7.55
H(6)	0.1305	0.1395	0.6929	6.52
H(7)	0.3347	-0.2327	0.6981	6.75
H(8)	0.1950	0.0243	0.8111	8.15
H(9)	0.2975	-0.1588	0.8146	7.63
H(10)	0.2192	0.3384	0.6097	4.75
H(11)	0.1532	0.1389	0.3355	4.78
H(12)	0.1153	0.4515	0.5184	5.41
H(13)	0.1174	0.1745	0.5577	4.66
H(14)	0.2967	-0.0043	0.4200	6.04
H(15)	0.2400	-0.0881	0.3495	6.01
H(16)	0.1981	0.6117	0.4352	6.29
H(17)	0.1875	0.6661	0.5168	6.27
H(18)	0.0433	-0.2884	0.2341	7.82
H(19)	-0.0284	-0.1692	0.2136	7.80
H(20)	0.3598	0.1855	0.6466	5.90
H(21)	0.3998	0.0621	0.5536	9.14
H(22)	0.4801	0.0412	0.6200	9.10
H(23)	0.4769	0.1679	0.5579	9.09
H(24)	0.0230	-0.0713	0.1226	10.28
H(25)	0.0553	-0.2352	0.1168	10.26
H(26)	0.1148	-0.1137	0.1639	10.28
H(27)	0.5277	0.2217	0.7153	8.82
H(28)	0.4581	0.1569	0.7537	8.83
H(29)	0.4650	0.3303	0.7425	8.81
H(30)	0.2810	0.0494	0.2631	12.34
H(31)	0.3623	0.0560	0.3293	12.32
H(32)	0.2979	0.1880	0.3189	12.31
H(33)	0.2602	0.2678	0.4311	4.50

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	0.0694(7)	0.0522(7)	0.0474(6)	0.0154(5)	-0.0045(5)	-0.0036(5)
O(8)	0.0782(9)	0.0863(10)	0.0512(7)	-0.0158(7)	0.0016(6)	-0.0175(6)
O(16)	0.0854(10)	0.0863(10)	0.0739(9)	-0.0250(8)	0.0036(7)	-0.0105(7)
N(1)	0.0500(7)	0.0465(7)	0.0385(6)	0.0026(5)	0.0049(5)	-0.0014(5)
C(3)	0.0532(8)	0.0493(9)	0.0409(7)	0.0083(7)	0.0050(6)	0.0013(6)
C(4)	0.0482(8)	0.0539(9)	0.0490(8)	0.0052(7)	0.0009(6)	-0.0040(7)
C(5)	0.0549(9)	0.0536(9)	0.0436(8)	-0.0038(7)	0.0083(7)	-0.0009(7)
C(6)	0.0591(9)	0.0502(9)	0.0417(8)	0.0047(7)	0.0068(7)	-0.0041(6)
C(7)	0.0577(9)	0.0527(9)	0.0411(8)	0.0049(7)	0.0046(6)	-0.0021(6)
C(9)	0.0651(10)	0.0563(10)	0.0502(9)	0.0156(8)	0.0056(7)	-0.0060(7)
C(10)	0.0640(10)	0.0499(9)	0.0417(8)	0.0006(7)	0.0075(7)	-0.0066(6)
C(11)	0.0826(12)	0.0475(9)	0.0500(9)	0.0053(8)	0.0164(8)	-0.0063(7)
C(12)	0.0487(8)	0.0547(9)	0.0450(8)	0.0041(7)	0.0080(6)	-0.0015(7)
C(13)	0.0471(8)	0.0577(10)	0.0532(9)	-0.0007(7)	0.0043(7)	0.0013(7)
C(14)	0.0618(10)	0.0565(10)	0.0554(10)	0.0015(8)	0.0070(8)	0.0026(7)
C(15)	0.0606(10)	0.0579(10)	0.0510(9)	-0.0026(8)	0.0072(7)	-0.0042(7)
C(17)	0.0636(10)	0.0611(11)	0.0652(11)	0.0052(8)	0.0137(9)	-0.0121(8)
C(18)	0.0543(9)	0.0643(11)	0.0569(10)	-0.0014(8)	0.0002(7)	-0.0060(8)
C(19)	0.1026(16)	0.0489(10)	0.0722(12)	-0.0025(10)	0.0319(11)	-0.0026(9)
C(20)	0.0548(9)	0.0669(11)	0.0649(11)	0.0000(8)	-0.0004(8)	0.0097(9)
C(21)	0.0671(11)	0.0695(13)	0.0746(12)	-0.0056(10)	0.0131(10)	-0.0061(10)
C(22)	0.0908(13)	0.0484(10)	0.0594(10)	0.0156(9)	0.0057(9)	-0.0037(8)
C(23)	0.0829(13)	0.0717(12)	0.0517(10)	0.0000(10)	0.0210(9)	0.0004(8)
C(24)	0.0881(15)	0.0627(12)	0.0887(15)	-0.0159(11)	0.0324(12)	-0.0098(11)
C(25)	0.0735(12)	0.0652(12)	0.0737(13)	0.0003(10)	-0.0053(10)	0.0141(10)
C(26)	0.0769(13)	0.1063(18)	0.0639(12)	-0.0154(13)	-0.0022(10)	-0.0326(12)
C(27)	0.123(2)	0.0885(16)	0.0470(10)	-0.0086(15)	0.0210(11)	0.0052(10)
C(28)	0.1040(17)	0.0823(15)	0.0548(12)	-0.0155(14)	-0.0049(11)	0.0200(10)
C(29)	0.108(2)	0.0695(15)	0.1085(19)	0.0159(14)	0.0115(15)	0.0007(13)
C(30)	0.0771(15)	0.180(3)	0.0671(14)	0.0007(17)	0.0045(11)	-0.0488(17)
C(31)	0.0846(15)	0.117(2)	0.0763(15)	0.0034(15)	-0.0118(12)	0.0155(14)
C(32)	0.133(2)	0.112(2)	0.145(2)	0.0230(19)	0.091(2)	0.0079(19)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
O(2)	C(3)	1.448(2)	O(2)	C(9)	1.437(2)
O(8)	C(18)	1.336(2)	O(8)	C(26)	1.455(2)
O(16)	C(18)	1.200(2)	N(1)	C(3)	1.4431(19)
N(1)	C(6)	1.482(2)	N(1)	C(12)	1.488(2)
C(3)	C(7)	1.516(2)	C(4)	C(7)	1.500(2)
C(4)	C(13)	1.325(2)	C(4)	C(18)	1.499(2)
C(5)	C(12)	1.518(2)	C(5)	C(14)	1.383(2)
C(5)	C(23)	1.385(2)	C(6)	C(9)	1.565(2)
C(6)	C(10)	1.511(2)	C(7)	C(17)	1.546(2)
C(9)	C(22)	1.527(2)	C(10)	C(11)	1.389(2)
C(10)	C(15)	1.406(2)	C(11)	C(19)	1.389(3)
C(11)	C(22)	1.498(2)	C(12)	C(13)	1.495(2)
C(14)	C(25)	1.375(2)	C(15)	C(20)	1.518(2)
C(15)	C(21)	1.387(3)	C(17)	C(32)	1.491(4)
C(19)	C(24)	1.382(3)	C(20)	C(29)	1.513(3)
C(20)	C(31)	1.516(2)	C(21)	C(24)	1.385(3)
C(23)	C(27)	1.378(2)	C(25)	C(28)	1.386(3)
C(26)	C(30)	1.452(3)	C(27)	C(28)	1.373(3)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(3)	H(33)	0.950	C(6)	H(10)	0.950
C(7)	H(11)	0.950	C(9)	H(12)	0.950
C(12)	H(13)	0.950	C(13)	H(2)	0.950
C(14)	H(3)	0.950	C(17)	H(14)	0.950
C(17)	H(15)	0.950	C(19)	H(4)	0.950
C(20)	H(20)	0.950	C(21)	H(1)	0.950
C(22)	H(16)	0.950	C(22)	H(17)	0.950
C(23)	H(6)	0.950	C(24)	H(5)	0.950
C(25)	H(7)	0.950	C(26)	H(18)	0.950
C(26)	H(19)	0.950	C(27)	H(8)	0.950
C(28)	H(9)	0.950	C(29)	H(21)	0.950
C(29)	H(22)	0.950	C(29)	H(23)	0.950
C(30)	H(24)	0.950	C(30)	H(25)	0.950
C(30)	H(26)	0.950	C(31)	H(27)	0.950
C(31)	H(28)	0.950	C(31)	H(29)	0.950
C(32)	H(30)	0.950	C(32)	H(31)	0.950
C(32)	H(32)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	O(2)	C(9)	106.29(11)	C(18)	O(8)	C(26)	118.15(17)
C(3)	N(1)	C(6)	100.58(11)	C(3)	N(1)	C(12)	115.10(11)
C(6)	N(1)	C(12)	108.89(13)	O(2)	C(3)	N(1)	106.38(13)
O(2)	C(3)	C(7)	110.93(11)	N(1)	C(3)	C(7)	115.99(13)
C(7)	C(4)	C(13)	122.38(14)	C(7)	C(4)	C(18)	119.30(15)
C(13)	C(4)	C(18)	118.32(16)	C(12)	C(5)	C(14)	119.76(15)
C(12)	C(5)	C(23)	121.42(16)	C(14)	C(5)	C(23)	118.79(15)
N(1)	C(6)	C(9)	104.03(11)	N(1)	C(6)	C(10)	114.95(14)
C(9)	C(6)	C(10)	104.05(13)	C(3)	C(7)	C(4)	111.31(14)
C(3)	C(7)	C(17)	110.58(13)	C(4)	C(7)	C(17)	110.71(14)
O(2)	C(9)	C(6)	104.34(13)	O(2)	C(9)	C(22)	112.80(15)
C(6)	C(9)	C(22)	106.18(14)	C(6)	C(10)	C(11)	110.75(14)
C(6)	C(10)	C(15)	127.93(15)	C(11)	C(10)	C(15)	121.30(17)
C(10)	C(11)	C(19)	120.41(17)	C(10)	C(11)	C(22)	111.47(17)
C(19)	C(11)	C(22)	128.10(17)	N(1)	C(12)	C(5)	107.81(11)
N(1)	C(12)	C(13)	112.79(13)	C(5)	C(12)	C(13)	112.51(14)
C(4)	C(13)	C(12)	124.43(16)	C(5)	C(14)	C(25)	121.01(19)
C(10)	C(15)	C(20)	121.83(16)	C(10)	C(15)	C(21)	116.82(17)
C(20)	C(15)	C(21)	121.32(16)	C(7)	C(17)	C(32)	114.81(18)
O(8)	C(18)	O(16)	124.06(17)	O(8)	C(18)	C(4)	110.80(16)
O(16)	C(18)	C(4)	125.13(18)	C(11)	C(19)	C(24)	118.91(19)
C(15)	C(20)	C(29)	110.51(19)	C(15)	C(20)	C(31)	112.71(18)
C(29)	C(20)	C(31)	111.19(19)	C(15)	C(21)	C(24)	122.17(19)
C(9)	C(22)	C(11)	105.23(15)	C(5)	C(23)	C(27)	120.3(2)
C(19)	C(24)	C(21)	120.4(2)	C(14)	C(25)	C(28)	119.7(2)
O(8)	C(26)	C(30)	107.8(2)	C(23)	C(27)	C(28)	120.6(2)
C(25)	C(28)	C(27)	119.6(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(2)	C(3)	H(33)	107.5	N(1)	C(3)	H(33)	107.7
C(7)	C(3)	H(33)	108.0	N(1)	C(6)	H(10)	110.8
C(9)	C(6)	H(10)	111.9	C(10)	C(6)	H(10)	110.7
C(3)	C(7)	H(11)	108.5	C(4)	C(7)	H(11)	108.1
C(17)	C(7)	H(11)	107.5	O(2)	C(9)	H(12)	110.3
C(6)	C(9)	H(12)	111.2	C(22)	C(9)	H(12)	111.7
N(1)	C(12)	H(13)	107.5	C(5)	C(12)	H(13)	107.8
C(13)	C(12)	H(13)	108.2	C(4)	C(13)	H(2)	117.6
C(12)	C(13)	H(2)	118.0	C(5)	C(14)	H(3)	119.0
C(25)	C(14)	H(3)	120.0	C(7)	C(17)	H(14)	107.6
C(7)	C(17)	H(15)	108.6	C(32)	C(17)	H(14)	108.9
C(32)	C(17)	H(15)	107.4	H(14)	C(17)	H(15)	109.5
C(11)	C(19)	H(4)	120.3	C(24)	C(19)	H(4)	120.8
C(15)	C(20)	H(20)	107.6	C(29)	C(20)	H(20)	106.6
C(31)	C(20)	H(20)	107.9	C(15)	C(21)	H(1)	118.2
C(24)	C(21)	H(1)	119.6	C(9)	C(22)	H(16)	109.8
C(9)	C(22)	H(17)	111.0	C(11)	C(22)	H(16)	110.8
C(11)	C(22)	H(17)	110.4	H(16)	C(22)	H(17)	109.5
C(5)	C(23)	H(6)	119.3	C(27)	C(23)	H(6)	120.4
C(19)	C(24)	H(5)	119.9	C(21)	C(24)	H(5)	119.7
C(14)	C(25)	H(7)	120.4	C(28)	C(25)	H(7)	119.9
O(8)	C(26)	H(18)	109.7	O(8)	C(26)	H(19)	110.4
C(30)	C(26)	H(18)	109.1	C(30)	C(26)	H(19)	110.4
H(18)	C(26)	H(19)	109.5	C(23)	C(27)	H(8)	119.9
C(28)	C(27)	H(8)	119.6	C(25)	C(28)	H(9)	120.6
C(27)	C(28)	H(9)	119.8	C(20)	C(29)	H(21)	111.3
C(20)	C(29)	H(22)	108.9	C(20)	C(29)	H(23)	108.2
H(21)	C(29)	H(22)	109.5	H(21)	C(29)	H(23)	109.5
H(22)	C(29)	H(23)	109.5	C(26)	C(30)	H(24)	108.7
C(26)	C(30)	H(25)	111.1	C(26)	C(30)	H(26)	108.5
H(24)	C(30)	H(25)	109.5	H(24)	C(30)	H(26)	109.5
H(25)	C(30)	H(26)	109.5	C(20)	C(31)	H(27)	109.3
C(20)	C(31)	H(28)	110.2	C(20)	C(31)	H(29)	109.0
H(27)	C(31)	H(28)	109.5	H(27)	C(31)	H(29)	109.5
H(28)	C(31)	H(29)	109.5	C(17)	C(32)	H(30)	110.3
C(17)	C(32)	H(31)	110.0	C(17)	C(32)	H(32)	108.0
H(30)	C(32)	H(31)	109.5	H(30)	C(32)	H(32)	109.5

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
H(31)	C(32)	H(32)	109.5				

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(3)	O(2)	C(9)	C(6)	-10.36(17)	C(3)	O(2)	C(9)	C(22)	104.44(16)
C(9)	O(2)	C(3)	N(1)	34.52(16)	C(9)	O(2)	C(3)	C(7)	161.49(14)
C(18)	O(8)	C(26)	C(30)	-171.26(19)	C(26)	O(8)	C(18)	O(16)	-1.0(3)
C(26)	O(8)	C(18)	C(4)	179.81(16)	C(3)	N(1)	C(6)	C(9)	35.46(16)
C(3)	N(1)	C(6)	C(10)	-77.63(14)	C(6)	N(1)	C(3)	O(2)	-43.64(14)
C(6)	N(1)	C(3)	C(7)	-167.52(14)	C(3)	N(1)	C(12)	C(5)	159.81(13)
C(3)	N(1)	C(12)	C(13)	34.99(18)	C(12)	N(1)	C(3)	O(2)	73.18(16)
C(12)	N(1)	C(3)	C(7)	-50.70(19)	C(6)	N(1)	C(12)	C(5)	-88.19(14)
C(6)	N(1)	C(12)	C(13)	146.98(13)	C(12)	N(1)	C(6)	C(9)	-85.87(14)
C(12)	N(1)	C(6)	C(10)	161.04(12)	O(2)	C(3)	C(7)	C(4)	-81.78(16)
O(2)	C(3)	C(7)	C(17)	154.72(14)	N(1)	C(3)	C(7)	C(4)	39.71(19)
N(1)	C(3)	C(7)	C(17)	-83.80(18)	C(7)	C(4)	C(13)	C(12)	3.9(2)
C(13)	C(4)	C(7)	C(3)	-16.5(2)	C(13)	C(4)	C(7)	C(17)	106.90(18)
C(7)	C(4)	C(18)	O(8)	-9.5(2)	C(7)	C(4)	C(18)	O(16)	171.32(18)
C(18)	C(4)	C(7)	C(3)	164.52(14)	C(18)	C(4)	C(7)	C(17)	-72.05(18)
C(13)	C(4)	C(18)	O(8)	171.54(16)	C(13)	C(4)	C(18)	O(16)	-7.7(2)
C(18)	C(4)	C(13)	C(12)	-177.10(15)	C(12)	C(5)	C(14)	C(25)	177.39(17)
C(14)	C(5)	C(12)	N(1)	-64.67(19)	C(14)	C(5)	C(12)	C(13)	60.3(2)
C(12)	C(5)	C(23)	C(27)	-177.17(19)	C(23)	C(5)	C(12)	N(1)	113.50(18)
C(23)	C(5)	C(12)	C(13)	-121.51(19)	C(14)	C(5)	C(23)	C(27)	1.0(3)
C(23)	C(5)	C(14)	C(25)	-0.8(2)	N(1)	C(6)	C(9)	O(2)	-15.91(18)
N(1)	C(6)	C(9)	C(22)	-135.30(15)	N(1)	C(6)	C(10)	C(11)	122.30(15)
N(1)	C(6)	C(10)	C(15)	-59.4(2)	C(9)	C(6)	C(10)	C(11)	9.22(19)
C(9)	C(6)	C(10)	C(15)	-172.53(17)	C(10)	C(6)	C(9)	O(2)	104.80(15)
C(10)	C(6)	C(9)	C(22)	-14.59(18)	C(3)	C(7)	C(17)	C(32)	-78.5(2)
C(4)	C(7)	C(17)	C(32)	157.64(18)	O(2)	C(9)	C(22)	C(11)	-99.01(17)
C(6)	C(9)	C(22)	C(11)	14.7(2)	C(6)	C(10)	C(11)	C(19)	178.50(18)
C(6)	C(10)	C(11)	C(22)	-0.01(18)	C(6)	C(10)	C(15)	C(20)	4.8(2)
C(6)	C(10)	C(15)	C(21)	-177.18(18)	C(11)	C(10)	C(15)	C(20)	-177.09(18)
C(11)	C(10)	C(15)	C(21)	0.9(2)	C(15)	C(10)	C(11)	C(19)	0.1(2)
C(15)	C(10)	C(11)	C(22)	-178.40(17)	C(10)	C(11)	C(19)	C(24)	-1.3(3)
C(10)	C(11)	C(22)	C(9)	-9.5(2)	C(19)	C(11)	C(22)	C(9)	172.1(2)
C(22)	C(11)	C(19)	C(24)	176.9(2)	N(1)	C(12)	C(13)	C(4)	-12.3(2)
C(5)	C(12)	C(13)	C(4)	-134.50(17)	C(5)	C(14)	C(25)	C(28)	0.1(2)
C(10)	C(15)	C(20)	C(29)	100.2(2)	C(10)	C(15)	C(20)	C(31)	-134.67(19)
C(10)	C(15)	C(21)	C(24)	-0.7(3)	C(20)	C(15)	C(21)	C(24)	177.3(2)
C(21)	C(15)	C(20)	C(29)	-77.7(2)	C(21)	C(15)	C(20)	C(31)	47.4(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(11)	C(19)	C(24)	C(21)	1.5(3)
C(5)	C(23)	C(27)	C(28)	-0.5(3)
C(23)	C(27)	C(28)	C(25)	-0.3(3)
C(15)	C(21)	C(24)	C(19)	-0.5(3)
C(14)	C(25)	C(28)	C(27)	0.5(3)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(2)	C(26)[1)]	3.460(2)	O(2)	C(27)[2)]	3.563(2)
O(2)	C(30)[1)]	3.322(2)	O(16)	C(23)[3)]	3.463(2)
C(21)	C(24)[4)]	3.474(3)	C(23)	O(16)[3)]	3.463(2)
C(24)	C(21)[4)]	3.474(3)	C(24)	C(24)[4)]	3.562(3)
C(26)	O(2)[5)]	3.460(2)	C(27)	O(2)[6)]	3.563(2)
C(30)	O(2)[5)]	3.322(2)			

Symmetry Operators:

(1) -X,Y+1/2,-Z+1/2
(2) X,-Y+1/2,Z+1/2-1
(3) -X,-Y,-Z+1
(4) -X+1,-Y+1,-Z+1
(5) -X,Y+1/2-1,-Z+1/2
(6) X,-Y+1/2,Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(2)	H(8)[1)]	2.625	O(2)	H(19)[2)]	2.802
O(2)	H(24)[2)]	2.812	O(2)	H(25)[2)]	3.296
O(8)	H(6)[3)]	3.519	O(8)	H(18)[2)]	3.264
O(16)	H(6)[3)]	2.652	O(16)	H(13)[3)]	2.879
O(16)	H(16)[4)]	3.148	O(16)	H(17)[4)]	3.347
O(16)	H(24)[5)]	3.258	C(3)	H(8)[1)]	3.136
C(4)	H(16)[4)]	3.594	C(4)	H(17)[4)]	3.462
C(5)	H(25)[6)]	3.404	C(7)	H(19)[2)]	3.387
C(9)	H(8)[1)]	3.457	C(9)	H(24)[2)]	3.318
C(9)	H(26)[7)]	3.523	C(11)	H(3)[8)]	3.097
C(11)	H(7)[8)]	3.500	C(13)	H(2)[3)]	3.239
C(13)	H(17)[4)]	3.146	C(13)	H(25)[6)]	3.509
C(14)	H(4)[4)]	3.497	C(14)	H(17)[4)]	2.962
C(14)	H(25)[6)]	3.584	C(17)	H(4)[4)]	3.286
C(17)	H(9)[9)]	3.341	C(18)	H(6)[3)]	3.186
C(18)	H(16)[4)]	3.279	C(18)	H(17)[4)]	3.578
C(18)	H(18)[2)]	3.599	C(19)	H(1)[10)]	3.583
C(19)	H(3)[8)]	2.889	C(19)	H(7)[8)]	3.416
C(19)	H(9)[1)]	3.516	C(19)	H(14)[8)]	3.528
C(19)	H(23)[10)]	3.505	C(21)	H(5)[10)]	3.260
C(21)	H(28)[11)]	3.087	C(22)	H(3)[8)]	3.119
C(22)	H(8)[1)]	3.230	C(23)	H(18)[6)]	3.397
C(23)	H(19)[3)]	3.267	C(23)	H(25)[6)]	3.575
C(23)	H(32)[7)]	3.418	C(24)	H(1)[10)]	3.397
C(24)	H(5)[10)]	3.542	C(24)	H(23)[10)]	3.240
C(24)	H(28)[11)]	3.323	C(25)	H(17)[4)]	3.599
C(25)	H(26)[6)]	3.490	C(25)	H(27)[12)]	3.157
C(26)	H(6)[3)]	3.329	C(26)	H(11)[5)]	3.317
C(27)	H(16)[7)]	3.193	C(27)	H(18)[6)]	3.229
C(27)	H(19)[3)]	3.439	C(27)	H(32)[7)]	3.178
C(27)	H(33)[7)]	3.533	C(28)	H(15)[6)]	3.110
C(28)	H(16)[7)]	3.428	C(28)	H(27)[12)]	3.529
C(29)	H(5)[10)]	3.463	C(29)	H(31)[13)]	3.482
C(30)	H(12)[1)]	3.183	C(30)	H(17)[1)]	3.471
C(31)	H(7)[11)]	3.174	C(31)	H(31)[7)]	3.424
C(32)	H(29)[1)]	3.252	H(1)	C(19)[10)]	3.583
H(1)	C(24)[10)]	3.397	H(1)	H(5)[10)]	3.319

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(1)	H(9)[11)]	3.122	H(1)	H(28)[11)]	2.795
H(2)	C(13)[3)]	3.239	H(2)	H(2)[3)]	2.866
H(2)	H(13)[3)]	3.215	H(2)	H(17)[4)]	2.854
H(2)	H(25)[6)]	2.689	H(3)	C(11)[4)]	3.097
H(3)	C(19)[4)]	2.889	H(3)	C(22)[4)]	3.119
H(3)	H(4)[4)]	2.603	H(3)	H(16)[4)]	3.502
H(3)	H(17)[4)]	2.469	H(4)	C(14)[8)]	3.497
H(4)	C(17)[8)]	3.286	H(4)	H(3)[8)]	2.603
H(4)	H(9)[1)]	3.139	H(4)	H(14)[8)]	2.597
H(4)	H(15)[8)]	3.128	H(4)	H(21)[8)]	2.989
H(4)	H(23)[10)]	3.101	H(5)	C(21)[10)]	3.260
H(5)	C(24)[10)]	3.542	H(5)	C(29)[10)]	3.463
H(5)	H(1)[10)]	3.319	H(5)	H(22)[8)]	3.408
H(5)	H(23)[10)]	2.565	H(5)	H(28)[11)]	3.222
H(5)	H(31)[10)]	3.579	H(6)	O(8)[3)]	3.519
H(6)	O(16)[3)]	2.652	H(6)	C(18)[3)]	3.186
H(6)	C(26)[3)]	3.329	H(6)	H(11)[7)]	3.126
H(6)	H(18)[6)]	3.552	H(6)	H(19)[3)]	2.594
H(6)	H(32)[7)]	3.449	H(7)	C(11)[4)]	3.500
H(7)	C(19)[4)]	3.416	H(7)	C(31)[12)]	3.174
H(7)	H(27)[12)]	2.438	H(7)	H(28)[12)]	3.429
H(7)	H(29)[12)]	3.240	H(7)	H(30)[6)]	3.213
H(8)	O(2)[7)]	2.625	H(8)	C(3)[7)]	3.136
H(8)	C(9)[7)]	3.457	H(8)	C(22)[7)]	3.230
H(8)	H(11)[7)]	3.102	H(8)	H(16)[7)]	2.471
H(8)	H(18)[6)]	3.279	H(8)	H(19)[3)]	2.941
H(8)	H(32)[7)]	3.029	H(8)	H(33)[7)]	2.811
H(9)	C(17)[6)]	3.341	H(9)	C(19)[7)]	3.516
H(9)	H(1)[12)]	3.122	H(9)	H(4)[7)]	3.139
H(9)	H(14)[6)]	3.502	H(9)	H(15)[6)]	2.550
H(9)	H(16)[7)]	2.962	H(9)	H(27)[12)]	3.185
H(9)	H(30)[6)]	3.560	H(10)	H(26)[7)]	3.228
H(10)	H(30)[7)]	2.824	H(11)	C(26)[2)]	3.317
H(11)	H(6)[1)]	3.126	H(11)	H(8)[1)]	3.102
H(11)	H(18)[2)]	3.219	H(11)	H(19)[2)]	2.636
H(12)	C(30)[7)]	3.183	H(12)	H(24)[2)]	2.948
H(12)	H(24)[7)]	2.814	H(12)	H(25)[7)]	3.308

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	H(26)$^{7)}$	2.921	H(13)	O(16)$^{3)}$	2.879
H(13)	H(2)$^{3)}$	3.215	H(14)	C(19)$^{4)}$	3.528
H(14)	H(4)$^{4)}$	2.597	H(14)	H(9)$^{9)}$	3.502
H(15)	C(28)$^{9)}$	3.110	H(15)	H(4)$^{4)}$	3.128
H(15)	H(9)$^{9)}$	2.550	H(15)	H(16)$^{4)}$	3.193
H(16)	O(16)$^{8)}$	3.148	H(16)	C(4)$^{8)}$	3.594
H(16)	C(18)$^{8)}$	3.279	H(16)	C(27)$^{1)}$	3.193
H(16)	C(28)$^{1)}$	3.428	H(16)	H(3)$^{8)}$	3.502
H(16)	H(8)$^{1)}$	2.471	H(16)	H(9)$^{1)}$	2.962
H(16)	H(15)$^{8)}$	3.193	H(17)	O(16)$^{8)}$	3.347
H(17)	C(4)$^{8)}$	3.462	H(17)	C(13)$^{8)}$	3.146
H(17)	C(14)$^{8)}$	2.962	H(17)	C(18)$^{8)}$	3.578
H(17)	C(25)$^{8)}$	3.599	H(17)	C(30)$^{7)}$	3.471
H(17)	H(2)$^{8)}$	2.854	H(17)	H(3)$^{8)}$	2.469
H(17)	H(25)$^{7)}$	3.121	H(17)	H(26)$^{7)}$	3.097
H(18)	O(8)$^{5)}$	3.264	H(18)	C(18)$^{5)}$	3.599
H(18)	C(23)$^{9)}$	3.397	H(18)	C(27)$^{9)}$	3.229
H(18)	H(6)$^{9)}$	3.552	H(18)	H(8)$^{9)}$	3.279
H(18)	H(11)$^{5)}$	3.219	H(18)	H(19)$^{5)}$	3.508
H(19)	O(2)$^{5)}$	2.802	H(19)	C(7)$^{5)}$	3.387
H(19)	C(23)$^{3)}$	3.267	H(19)	C(27)$^{3)}$	3.439
H(19)	H(6)$^{3)}$	2.594	H(19)	H(8)$^{3)}$	2.941
H(19)	H(11)$^{5)}$	2.636	H(19)	H(18)$^{2)}$	3.508
H(20)	H(30)$^{7)}$	3.528	H(20)	H(32)$^{7)}$	3.565
H(21)	H(4)$^{4)}$	2.989	H(22)	H(5)$^{4)}$	3.408
H(22)	H(29)$^{12)}$	3.013	H(22)	H(31)$^{13)}$	2.659
H(23)	C(19)$^{10)}$	3.505	H(23)	C(24)$^{10)}$	3.240
H(23)	H(4)$^{10)}$	3.101	H(23)	H(5)$^{10)}$	2.565
H(23)	H(31)$^{13)}$	3.510	H(24)	O(2)$^{5)}$	2.812
H(24)	O(16)$^{2)}$	3.258	H(24)	C(9)$^{5)}$	3.318
H(24)	H(12)$^{5)}$	2.948	H(24)	H(12)$^{1)}$	2.814
H(25)	O(2)$^{5)}$	3.296	H(25)	C(5)$^{9)}$	3.404
H(25)	C(13)$^{9)}$	3.509	H(25)	C(14)$^{9)}$	3.584
H(25)	C(23)$^{9)}$	3.575	H(25)	H(2)$^{9)}$	2.689
H(25)	H(12)$^{1)}$	3.308	H(25)	H(17)$^{1)}$	3.121
H(26)	C(9)$^{1)}$	3.523	H(26)	C(25)$^{9)}$	3.490
H(26)	H(10)$^{1)}$	3.228	H(26)	H(12)$^{1)}$	2.921

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(26)	H(17)[1)]	3.097	H(27)	C(25)[11)]	3.157
H(27)	C(28)[11)]	3.529	H(27)	H(7)[11)]	2.438
H(27)	H(9)[11)]	3.185	H(27)	H(29)[12)]	3.531
H(27)	H(31)[13)]	3.233	H(28)	C(21)[12)]	3.087
H(28)	C(24)[12)]	3.323	H(28)	H(1)[12)]	2.795
H(28)	H(5)[12)]	3.222	H(28)	H(7)[11)]	3.429
H(28)	H(29)[12)]	3.139	H(28)	H(31)[7)]	3.395
H(28)	H(32)[7)]	3.362	H(29)	C(32)[7)]	3.252
H(29)	H(7)[11)]	3.240	H(29)	H(22)[11)]	3.013
H(29)	H(27)[11)]	3.531	H(29)	H(28)[11)]	3.139
H(29)	H(30)[7)]	3.271	H(29)	H(31)[7)]	2.691
H(29)	H(32)[7)]	3.292	H(30)	H(7)[9)]	3.213
H(30)	H(9)[9)]	3.560	H(30)	H(10)[1)]	2.824
H(30)	H(20)[1)]	3.528	H(30)	H(29)[1)]	3.271
H(31)	C(29)[13)]	3.482	H(31)	C(31)[1)]	3.424
H(31)	H(5)[10)]	3.579	H(31)	H(22)[13)]	2.659
H(31)	H(23)[13)]	3.510	H(31)	H(27)[13)]	3.233
H(31)	H(28)[1)]	3.395	H(31)	H(29)[1)]	2.691
H(32)	C(23)[1)]	3.418	H(32)	C(27)[1)]	3.178
H(32)	H(6)[1)]	3.449	H(32)	H(8)[1)]	3.029
H(32)	H(20)[1)]	3.565	H(32)	H(28)[1)]	3.362
H(32)	H(29)[1)]	3.292	H(33)	C(27)[1)]	3.533
H(33)	H(8)[1)]	2.811			

Symmetry Operators:

(1) X,-Y+1/2,Z+1/2-1
(2) -X,Y+1/2,-Z+1/2
(3) -X,-Y,-Z+1
(4) X,Y-1,Z
(5) -X,Y+1/2-1,-Z+1/2
(6) X,-Y+1/2-1,Z+1/2
(7) X,-Y+1/2,Z+1/2
(8) X,Y+1,Z
(9) X,-Y+1/2-1,Z+1/2-1
(10) -X+1,-Y+1,-Z+1
(11) -X+1,Y+1/2,-Z+1/2+1
(12) -X+1,Y+1/2-1,-Z+1/2+1
(13) -X+1,-Y,-Z+1