Small Molecule Crystallography Lab
Department of Chemistry and Biochemistry
University of Oklahoma
101 Stephenson Parkway
Norman, OK 73019-5251

Sample: Calcuim Picolinate (D_2O) Lab ID: 12136m
User: Kieth Thomas
Formula: $C_7 H_3 Ca D_6 N O_7$
Date: January 29, 2014

For Prof. Charles Rice

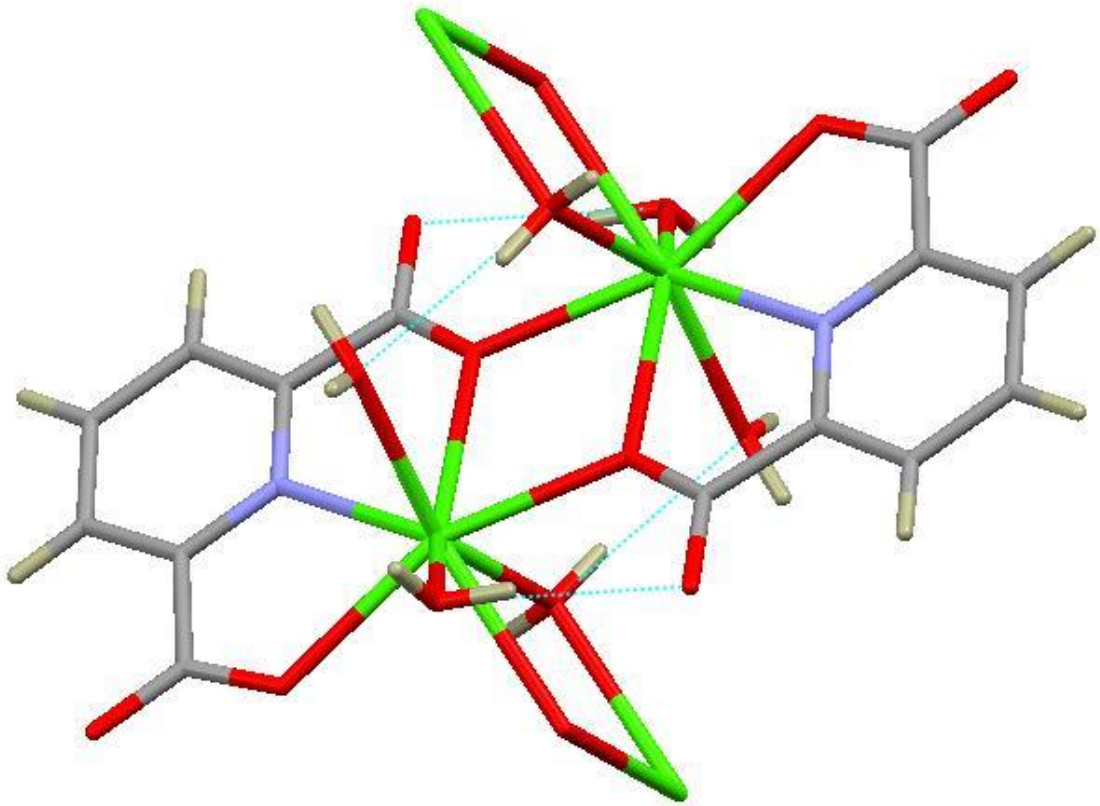
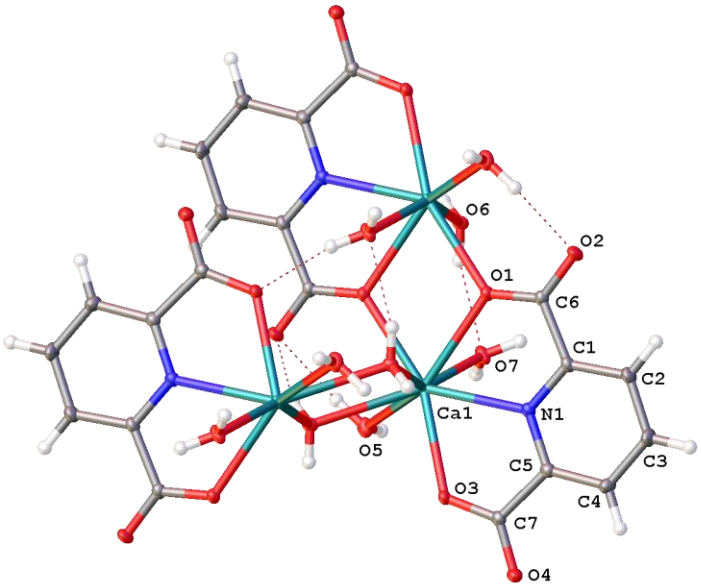
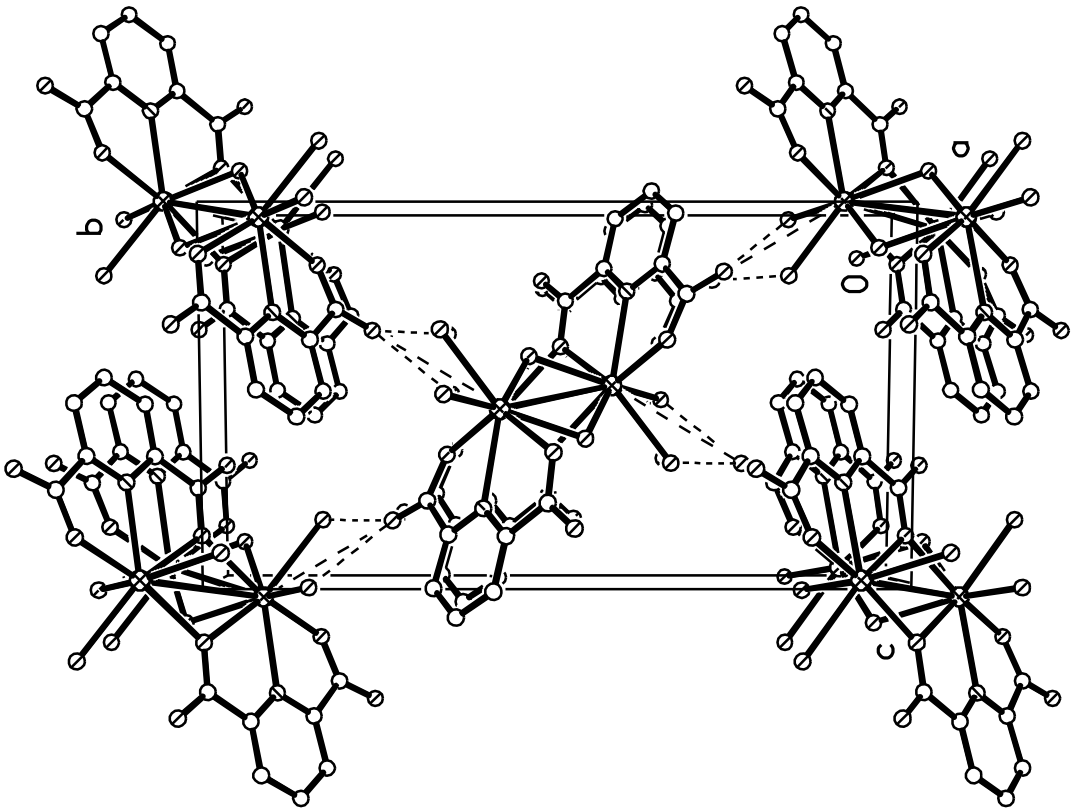


Table of Contents


O6
O2
O1
C6
C1
C2
O7
Ca1
N1
O5
C5
C3
O3
C4
C7
O4


b
a
c

Comment

The displacement ellipsoids were drawn at the 50% probability level (4).

Experimental

A colorless needle-shaped crystal of dimensions 0.350 x 0.080 x 0.060 mm was selected for structural analysis. Intensity data for this compound were collected using a diffractometer with a Bruker APEX ccd area detector (1) and graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The sample was cooled to 100(2) K. Cell parameters were determined from a non-linear least squares fit of 5599 peaks in the range 2.27 < θ < 26.06°. A total of 9838 data were measured in the range 2.270 < θ < 26.139° using ϕ and ω oscillation frames. The data were corrected for absorption by the empirical method (2) giving minimum and maximum transmission factors of 0.808 and 0.963. The data were merged to form a set of 1907 independent data with R(int) = 0.0334 and a coverage of 98.9 %.

The monoclinic space group $P2_1/n$ was determined by systematic absences and statistical tests and verified by subsequent refinement. The structure was solved by direct methods and refined by full-matrix least-squares methods on F^2 (3). The positions of hydrogens bonded to carbons were initially determined by geometry and were refined using a riding model. Hydrogens and deuteriums bonded to oxygens were located on a difference map, and their positions were refined independently. Non-hydrogen atoms were refined with anisotropic displacement parameters. Hydrogen atom displacement parameters were set to 1.2 times the isotropic equivalent displacement parameters of the bonded atoms. A total of 163 parameters were refined against 1907 data to give wR(F^2) = 0.0862 and S = 1.003 for weights of w = 1/[σ^2 (F^2) + (0.0520 P)^2 + 0.8000 P], where P = [F_o^2 + 2F_c^2] / 3. The final R(F) was 0.0317 for the 1746 observed, [$F > 4\sigma(F)$], data. The largest shift/s.u. was 0.001 in the final refinement cycle. The final difference map had maxima and minima of 0.385 and -0.271 e/Å^3, respectively.

Acknowledgment

The authors thank the National Science Foundation (grant CHE-0130835) and the University of Oklahoma for funds to purchase of the X-ray instrument and computers. This structure was determined by Douglas R. Powell.

References

(1) (a) Data Collection: Bruker (2007). SMART Bruker AXS Inc., Madison, Wisconsin, USA. (b) Data Reduction: Bruker (2007). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.

(2) Bruker (2002). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.

(3) G. M. Sheldrick (2013). SHELXL University of Gottingen.

Table 1. Crystal data and structure refinement for 12136m.

Empirical formula	$C_7 H_3 Ca D_6 N O_7$	
Formula weight	265.27	
Crystal system	monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 5.8532(13) Å	α= 90°
	b = 17.942(4) Å	β= 97.974(2)°
	c = 9.813(2) Å	γ= 90°
Volume	1020.6(4) Å^3	
Z, Z'	4, 1	
Density (calculated)	1.726 Mg/m^3	
Wavelength	0.71073 Å	
Temperature	100(2) K	
F(000)	536	
Absorption coefficient	0.636 mm^{-1}	
Absorption correction	semi-empirical from equivalents	
Max. and min. transmission	0.963 and 0.808	
Theta range for data collection	2.270 to 26.139°	
Reflections collected	9838	
Independent reflections	1907 [R(int) = 0.0334]	
Data / restraints / parameters	1907 / 0 / 163	
wR(F^2 all data)	$wR2$ = 0.0862	
R(F obsd data)	$R1$ = 0.0317	
Goodness-of-fit on F^2	1.003	
Observed data [I > 2σ(I)]	1746	
Largest and mean shift / s.u.	0.001and 0.000	
Largest diff. peak and hole	0.385 and -0.271 e/Å^3	

$wR2 = \{ \Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2] \}^{1/2}$

$R1 = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$

Table 2. Atomic coordinates and equivalent isotropic displacement parameters for 12136m. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
Ca(1)	0.73169(6)	0.42166(2)	0.46942(4)	0.00911(14)
O(1)	0.3819(2)	0.49524(7)	0.36354(13)	0.0114(3)
O(2)	0.1202(2)	0.52307(7)	0.18163(14)	0.0155(3)
O(3)	0.9836(2)	0.34589(7)	0.35130(13)	0.0130(3)
O(4)	1.0299(2)	0.26821(7)	0.17853(14)	0.0165(3)
O(5)	0.8598(2)	0.33845(8)	0.67262(15)	0.0155(3)
D(5OA)	0.789(4)	0.3006(14)	0.691(2)	0.019
D(5OB)	0.852(4)	0.3668(13)	0.742(3)	0.019
O(6)	0.8763(2)	0.53934(7)	0.39374(15)	0.0118(3)
D(6OA)	0.789(4)	0.5745(13)	0.398(2)	0.014
D(6OB)	0.921(4)	0.5358(12)	0.321(3)	0.014
O(7)	0.4019(2)	0.34810(7)	0.50721(15)	0.0139(3)
D(7OA)	0.429(4)	0.3110(13)	0.551(2)	0.017
D(7OB)	0.278(4)	0.3424(12)	0.454(2)	0.017
N(1)	0.5952(2)	0.39966(8)	0.21967(16)	0.0107(3)
C(1)	0.4040(3)	0.43158(10)	0.15478(19)	0.0119(4)
C(2)	0.3135(3)	0.41507(10)	0.0192(2)	0.0153(4)
C(3)	0.4289(3)	0.36317(11)	-0.0505(2)	0.0178(4)
C(4)	0.6296(3)	0.33037(10)	0.0154(2)	0.0156(4)
C(5)	0.7073(3)	0.35027(9)	0.15040(19)	0.0119(4)
C(6)	0.2917(3)	0.48766(10)	0.23953(19)	0.0109(4)
C(7)	0.9250(3)	0.31841(10)	0.23278(19)	0.0115(4)

Table 3. Bond lengths [Å] and angles [°] for 12136m.

Ca(1)-O(1)#1	2.3780(13)	O(6)-Ca(1)#2	2.5890(14)
Ca(1)-O(7)	2.4091(14)	O(6)-D(6OA)	0.82(2)
Ca(1)-O(3)	2.4166(13)	O(6)-D(6OB)	0.80(2)
Ca(1)-O(6)	2.4291(14)	O(7)-D(7OA)	0.80(2)
Ca(1)-N(1)	2.5014(16)	O(7)-D(7OB)	0.84(3)
Ca(1)-O(5)	2.5204(14)	N(1)-C(1)	1.337(2)
Ca(1)-O(1)	2.5344(13)	N(1)-C(5)	1.342(2)
Ca(1)-O(6)#2	2.5890(14)	C(1)-C(2)	1.394(3)
Ca(1)-Ca(1)#1	4.0090(9)	C(1)-C(6)	1.513(3)
Ca(1)-Ca(1)#2	4.1960(9)	C(2)-C(3)	1.386(3)
O(1)-C(6)	1.265(2)	C(2)-H(2)	0.9500
O(1)-Ca(1)#1	2.3780(13)	C(3)-C(4)	1.391(3)
O(2)-C(6)	1.255(2)	C(3)-H(3)	0.9500
O(3)-C(7)	1.266(2)	C(4)-C(5)	1.386(3)
O(4)-C(7)	1.250(2)	C(4)-H(4)	0.9500
O(5)-D(5OA)	0.83(3)	C(5)-C(7)	1.523(2)
O(5)-D(5OB)	0.85(3)		

O(1)#1-Ca(1)-O(7)	86.03(5)	O(6)-Ca(1)-O(6)#2	66.56(5)
O(1)#1-Ca(1)-O(3)	158.58(5)	N(1)-Ca(1)-O(6)#2	133.66(5)
O(7)-Ca(1)-O(3)	108.72(5)	O(5)-Ca(1)-O(6)#2	66.72(4)
O(1)#1-Ca(1)-O(6)	78.95(5)	O(1)-Ca(1)-O(6)#2	132.78(4)
O(7)-Ca(1)-O(6)	147.55(5)	O(1)#1-Ca(1)-Ca(1)#1	36.62(3)
O(3)-Ca(1)-O(6)	94.62(5)	O(7)-Ca(1)-Ca(1)#1	77.76(4)
O(1)#1-Ca(1)-N(1)	133.83(5)	O(3)-Ca(1)-Ca(1)#1	159.52(4)
O(7)-Ca(1)-N(1)	84.87(5)	O(6)-Ca(1)-Ca(1)#1	73.02(4)
O(3)-Ca(1)-N(1)	64.82(5)	N(1)-Ca(1)-Ca(1)#1	97.27(4)
O(6)-Ca(1)-N(1)	85.13(5)	O(5)-Ca(1)-Ca(1)#1	115.47(4)
O(1)#1-Ca(1)-O(5)	84.66(5)	O(1)-Ca(1)-Ca(1)#1	34.03(3)
O(7)-Ca(1)-O(5)	73.17(5)	O(6)#2-Ca(1)-Ca(1)#1	107.58(4)
O(3)-Ca(1)-O(5)	84.95(5)	O(1)#1-Ca(1)-Ca(1)#2	75.92(3)
O(6)-Ca(1)-O(5)	132.74(5)	O(7)-Ca(1)-Ca(1)#2	160.89(4)
N(1)-Ca(1)-O(5)	134.53(5)	O(3)-Ca(1)-Ca(1)#2	87.19(4)
O(1)#1-Ca(1)-O(1)	70.65(5)	O(6)-Ca(1)-Ca(1)#2	34.48(3)
O(7)-Ca(1)-O(1)	74.31(5)	N(1)-Ca(1)-Ca(1)#2	112.14(4)
O(3)-Ca(1)-O(1)	127.47(5)	O(5)-Ca(1)-Ca(1)#2	98.57(4)
O(6)-Ca(1)-O(1)	73.61(4)	O(1)-Ca(1)-Ca(1)#2	104.81(3)
N(1)-Ca(1)-O(1)	63.29(4)	O(6)#2-Ca(1)-Ca(1)#2	32.08(3)
O(5)-Ca(1)-O(1)	140.14(5)	Ca(1)#1-Ca(1)-Ca(1)#2	90.99(2)
O(1)#1-Ca(1)-O(6)#2	77.61(5)	C(6)-O(1)-Ca(1)#1	127.67(11)
O(7)-Ca(1)-O(6)#2	137.70(5)	C(6)-O(1)-Ca(1)	122.97(11)
O(3)-Ca(1)-O(6)#2	81.08(5)	Ca(1)#1-O(1)-Ca(1)	109.35(5)

C(7)-O(3)-Ca(1)	123.66(11)	C(3)-C(2)-C(1)	117.85(17)
Ca(1)-O(5)-D(5OA)	123.9(16)	C(3)-C(2)-H(2)	121.1
Ca(1)-O(5)-D(5OB)	103.4(16)	C(1)-C(2)-H(2)	121.1
D(5OA)-O(5)-D(5OB)	103(2)	C(2)-C(3)-C(4)	119.54(18)
Ca(1)-O(6)-Ca(1)#2	113.44(5)	C(2)-C(3)-H(3)	120.2
Ca(1)-O(6)-D(6OA)	114.3(15)	C(4)-C(3)-H(3)	120.2
Ca(1)#2-O(6)-D(6OA)	106.4(16)	C(5)-C(4)-C(3)	118.65(17)
Ca(1)-O(6)-D(6OB)	112.2(16)	C(5)-C(4)-H(4)	120.7
Ca(1)#2-O(6)-D(6OB)	96.2(17)	C(3)-C(4)-H(4)	120.7
D(6OA)-O(6)-D(6OB)	113(2)	N(1)-C(5)-C(4)	122.37(17)
Ca(1)-O(7)-D(7OA)	116.0(16)	N(1)-C(5)-C(7)	114.26(16)
Ca(1)-O(7)-D(7OB)	127.5(16)	C(4)-C(5)-C(7)	123.37(16)
D(7OA)-O(7)-D(7OB)	109(2)	O(2)-C(6)-O(1)	125.59(17)
C(1)-N(1)-C(5)	118.51(16)	O(2)-C(6)-C(1)	117.97(16)
C(1)-N(1)-Ca(1)	122.09(12)	O(1)-C(6)-C(1)	116.43(15)
C(5)-N(1)-Ca(1)	119.23(11)	O(4)-C(7)-O(3)	125.85(16)
N(1)-C(1)-C(2)	123.07(17)	O(4)-C(7)-C(5)	117.94(16)
N(1)-C(1)-C(6)	114.96(16)	O(3)-C(7)-C(5)	116.21(15)
C(2)-C(1)-C(6)	121.98(16)		

Symmetry transformations used to generate equivalent atoms:
#1 -x+1,-y+1,-z+1 #2 -x+2,-y+1,-z+1

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 12136m. The anisotropic displacement factor exponent takes the form:
$-2\ \pi^2[\ h^2\ a^{*2}\ U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ca(1)	7(1)	11(1)	8(1)	0(1)	0(1)	0(1)
O(1)	10(1)	15(1)	9(1)	-1(1)	0(1)	1(1)
O(2)	12(1)	22(1)	12(1)	-1(1)	-2(1)	7(1)
O(3)	10(1)	17(1)	11(1)	-2(1)	-2(1)	3(1)
O(4)	13(1)	18(1)	18(1)	-6(1)	-2(1)	5(1)
O(5)	15(1)	15(1)	16(1)	2(1)	0(1)	-4(1)
O(6)	11(1)	14(1)	11(1)	1(1)	3(1)	1(1)
O(7)	11(1)	14(1)	16(1)	4(1)	-3(1)	-1(1)
N(1)	10(1)	12(1)	10(1)	1(1)	2(1)	-1(1)
C(1)	10(1)	15(1)	10(1)	2(1)	-1(1)	1(1)
C(2)	13(1)	20(1)	12(1)	0(1)	-2(1)	4(1)
C(3)	20(1)	22(1)	10(1)	-3(1)	-4(1)	4(1)
C(4)	18(1)	17(1)	12(1)	-2(1)	1(1)	5(1)
C(5)	11(1)	12(1)	13(1)	0(1)	1(1)	0(1)
C(6)	10(1)	14(1)	9(1)	0(1)	1(1)	-1(1)
C(7)	10(1)	12(1)	12(1)	0(1)	2(1)	-1(1)

Table 5. Hydrogen and deuterium coordinates and isotropic displacement parameters for 12136m.

	x	y	z	U(eq)
D(5OA)	0.789(4)	0.3006(14)	0.691(2)	0.019
D(5OB)	0.852(4)	0.3668(13)	0.742(3)	0.019
D(6OA)	0.789(4)	0.5745(13)	0.398(2)	0.014
D(6OB)	0.921(4)	0.5358(12)	0.321(3)	0.014
D(7OA)	0.429(4)	0.3110(13)	0.551(2)	0.017
D(7OB)	0.278(4)	0.3424(12)	0.454(2)	0.017
H(2)	0.1770	0.4386	-0.0240	0.018
H(3)	0.3714	0.3501	-0.1427	0.021
H(4)	0.7118	0.2950	-0.0311	0.019

Table 6. Torsion angles [°] for 12136m.

C(5)-N(1)-C(1)-C(2)	1.1(3)
Ca(1)-N(1)-C(1)-C(2)	-174.12(14)
C(5)-N(1)-C(1)-C(6)	-178.77(15)
Ca(1)-N(1)-C(1)-C(6)	6.0(2)
N(1)-C(1)-C(2)-C(3)	-0.1(3)
C(6)-C(1)-C(2)-C(3)	179.69(17)
C(1)-C(2)-C(3)-C(4)	-0.7(3)
C(2)-C(3)-C(4)-C(5)	0.6(3)
C(1)-N(1)-C(5)-C(4)	-1.2(3)
Ca(1)-N(1)-C(5)-C(4)	174.15(14)
C(1)-N(1)-C(5)-C(7)	178.88(15)
Ca(1)-N(1)-C(5)-C(7)	-5.80(19)
C(3)-C(4)-C(5)-N(1)	0.4(3)
C(3)-C(4)-C(5)-C(7)	-179.70(17)
Ca(1)#1-O(1)-C(6)-O(2)	-0.8(3)
Ca(1)-O(1)-C(6)-O(2)	-179.29(13)
Ca(1)#1-O(1)-C(6)-C(1)	178.40(10)
Ca(1)-O(1)-C(6)-C(1)	-0.1(2)
N(1)-C(1)-C(6)-O(2)	175.46(16)
C(2)-C(1)-C(6)-O(2)	-4.4(3)
N(1)-C(1)-C(6)-O(1)	-3.8(2)
C(2)-C(1)-C(6)-O(1)	176.36(17)
Ca(1)-O(3)-C(7)-O(4)	-165.59(14)
Ca(1)-O(3)-C(7)-C(5)	14.6(2)
N(1)-C(5)-C(7)-O(4)	175.07(16)
C(4)-C(5)-C(7)-O(4)	-4.9(3)
N(1)-C(5)-C(7)-O(3)	-5.1(2)
C(4)-C(5)-C(7)-O(3)	174.95(17)

Symmetry transformations used to generate equivalent atoms:
#1 -x+1, -y+1, -z+1

Table 7. Hydrogen bonds for 12136m[Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)-D(5OA)...O(4)#3	0.83(3)	1.95(3)	2.7250(19)	156(2)
O(5)-D(5OB)...O(2)#1	0.85(3)	2.11(3)	2.861(2)	146(2)
O(6)-D(6OA)...O(7)#1	0.82(2)	2.08(2)	2.849(2)	156(2)
O(6)-D(6OB)...O(2)#4	0.80(2)	1.93(2)	2.698(2)	162(2)
O(7)-D(7OA)...O(4)#3	0.80(2)	1.93(3)	2.719(2)	170(2)
O(7)-D(7OB)...O(3)#5	0.84(3)	1.87(3)	2.7014(18)	169(2)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1, -y+1, -z+1 #3 x-1/2, -y+1/2, z+1/2 #4 x+1, y, z #5 x-1, y, z