

C4—C5	1.540 (5)	C1'—C6'	1.396 (5)
C4—C15	1.503 (6)	C2'—C3'	1.385 (5)
C5—C6	1.543 (5)	C3'—C4'	1.388 (7)
C5—C10	1.554 (4)	C4'—C5'	1.377 (6)
C5—C14	1.553 (4)	C5'—C6'	1.368 (5)
C6—C7	1.521 (5)	C7'—C8'	1.494 (6)
C5'—O2—C7'	115.8 (3)	C10—C9—C12	113.4 (3)
C2—C1—C10	108.1 (3)	C11—C9—C12	105.2 (3)
C1—C2—C3	111.5 (3)	C1—C10—C5	108.8 (3)
C2—C3—C4	126.3 (3)	C1—C10—C9	114.5 (3)
C3—C4—C5	121.9 (3)	C5—C10—C9	117.1 (3)
C3—C4—C15	120.7 (3)	C9—C11—C1'	119.4 (3)
C5—C4—C15	117.4 (3)	C11—C1'—C2'	121.7 (3)
C4—C5—C6	110.5 (3)	C11—C1'—C6'	121.0 (3)
C4—C5—C10	107.0 (2)	C2'—C1'—C6'	117.2 (3)
C4—C5—C14	106.6 (3)	O1—C2'—C1'	117.6 (3)
C6—C5—C10	108.1 (2)	O1—C2'—C3'	120.9 (4)
C6—C5—C14	108.8 (3)	C1'—C2'—C3'	121.5 (4)
C10—C5—C14	115.8 (3)	C2'—C3'—C4'	120.5 (4)
C5—C6—C7	111.3 (3)	C3'—C4'—C5'	117.7 (4)
C6—C7—C8	112.0 (3)	O2—C5'—C4'	118.4 (3)
C7—C8—C9	111.7 (3)	O2—C5'—C6'	119.0 (3)
C7—C8—C13	109.4 (3)	C4'—C5'—C6'	122.5 (4)
C9—C8—C13	114.6 (3)	C1'—C6'—C5'	120.5 (3)
C8—C9—C10	106.9 (2)	O2—C7'—O3	124.2 (4)
C8—C9—C11	110.1 (3)	O2—C7'—C8'	111.3 (4)
C8—C9—C12	111.5 (3)	O3—C7'—C8'	124.5 (4)
C10—C9—C11	109.6 (2)		
C5'—O2—C7'—O3	0.9 (5)	C8—C9—C11—C1'	66.1 (4)
C2—C3—C4—C5	0.6 (6)	C9—C11—C1'—C2'	109.8 (4)
C4—C5—C10—C9	-172.0 (3)	C6'—C1'—C2'—O1	-173.0 (3)
C14—C5—C10—C9	69.3 (4)	O2—C5'—C6'—C1'	-178.0 (3)
C13—C8—C9—C12	-53.9 (4)		

The structure was solved using the *MULTAN11/82* package (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). The weighting scheme was according to Killean & Lawrence (1969). H atoms were located on the basis of geometrical considerations and ΔF map suggestions (for hydroxyl and methyl groups). All H atoms were included as fixed atoms in the final refinement, with isotropic displacement parameters set equal to B_{eq} of the parent atoms. Calculations were performed using Enraf-Nonius *SDP* software (B. A. Frenz & Associates, Inc., 1985) on a MicroVAX 3100 computer.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1126). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- B. A. Frenz & Associates, Inc. (1985). *SDP Structure Determination Package*. College Station, Texas, USA, and Enraf-Nonius, Delft, The Netherlands.
- Cimino, G., Crispino, A., Epifanio, R. de A., Madaio, A., Mattia, C. A., Mazzarella, L., Puliti, R., Trivellone, E. & Uriz, M. (1992). *Tetrahedron*, **48**, 9013–9022.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1361.
- Crispino, A., De Giulio, A., De Rosa, S. & Strazzullo, G. (1989). *J. Nat. Prod.* **52**, 646–648.
- De Giulio, A., De Rosa, S., Strazzullo, G., Diliberto, L., Obino, P., Marongiu, M. E., Pani, A. & La Colla, P. (1991). *Antivir. Chem. Chemother.* **2**, 223–227.
- De Rosa, S., Puliti, R., Crispino, A., De Giulio, A., Mattia, C. A. & Mazzarella, L. (1994). *J. Nat. Prod.* **57**, 256–262.
- Duax, W. L., Weeks, C. M. & Rohrer, D. C. (1976). *Top. Stereochem.* **9**, 271–383.

- Ferrándiz, M. L., Sanz, M. J., Bustos, G., Payá, M., Alcaraz, M. J. & De Rosa, S. (1994). *Eur. J. Pharmacol.* **253**, 75–82.
- Giordano, F. & Puliti, R. (1987). *Acta Cryst.* **C43**, 985–988.
- Killean, R. C. G. & Lawrence, J. L. (1969). *Acta Cryst.* **B25**, 1750–1752.
- Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- Puliti, R., De Rosa, S. & Mattia, C. A. (1994). *Acta Cryst.* **C50**, 830–833.
- Puliti, R., Fontana, A., Cimino, G., Mattia, C. A. & Mazzarella, L. (1993). *Acta Cryst.* **C49**, 1373–1376.
- Puliti, R., Gavagnin, M., Cimino, G., Mattia, C. A. & Mazzarella, L. (1992). *Acta Cryst.* **C48**, 2145–2147.
- Stout, G. H. & Jensen, L. H. (1968). *X-ray Structure Determination*, pp. 409–412. New York: Macmillan.

Acta Cryst. (1995). **C51**, 1198–1201

The 2:1 Adduct of an α -L-Altropyranose Derivative with Hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone at 173 K

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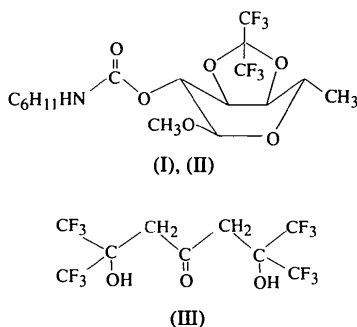
Abstract

As reported earlier [Miethchen, Rentsch & Michalik (1994). *Liebigs Ann. Chem.* pp. 219–222], the structure of methyl 2-*O*-(cyclohexylcarbonyl)-6-deoxy-3,4-

O-(hexafluoroisopropylidene)- α -L-altropyranoside, C₁₇H₂₃F₆NO₆, could be determined by X-ray analysis of a species which was obtained by co-crystallization with 1,1,1,7,7,7-hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone, C₉H₆F₁₂O₃, in a 2:1 molar ratio. While the pure α -L-altropyranose derivative forms needles that are not suitable for single-crystal X-ray investigations, the co-crystallization product yielded well shaped monoclinic prisms. The L-altropyranose derivative adopts a ring conformation which is intermediate between a boat and a twisted-boat conformation. The structure is stabilized by four intermolecular hydrogen bonds.

Comment

The reaction of methyl α -L-rhamnopyranoside with hexafluoroacetone (HFA) affords cyclic 3,4-acetals accompanied by simultaneous inversion of configuration at position C3 when *N,N'*-dicyclohexylcarbodiimide (DCC) is added as a coreagent. The main product of this reaction is methyl 6-deoxy-3,4-*O*-(hexafluoroisopropylidene)- α -L-altropyranoside in 88% yield, while the carbamoyl derivative is only formed in 3% yield (Miethchen, Rentsch & Stoll, 1992). In order to characterize the resulting reaction products unambiguously, an X-ray structure determination was undertaken. Suitable single crystals of the title α -L-altropyranose derivative [molecules (I) and (II)] were only obtained in the presence of 1,1,1,7,7,7-hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone, (III).



The asymmetric unit consists of two molecules of methyl 2-*O*-(cyclohexylcarbamoyl)-6-deoxy-3,4-*O*-(hexafluoroisopropylidene)- α -L-altropyranoside ($M_r = 451.36$; Figs. 1 and 2) and one molecule of 1,1,1,7,7,7-hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone ($M_r = 390.13$; Fig. 3). The structure of hexafluoro-2,6-dihydroxy-2,6-bis(trifluoromethyl)-4-heptanone has been reported recently (Vittal & Willis, 1993). The positions of all H atoms were refined with distance restraints for the C—H distances (Sheldrick, 1993). Details of the hydrogen-bonding scheme are given in Table 2.

In the case of the pure hexafluoro compound (Vittal & Willis, 1993), both hydroxy groups form intramolecular hydrogen bonds to the ketonic carbonyl O atom. In the present structure, intermolecular hydrogen bonds are formed resulting in a different non-zigzag molecular conformation. Other distances and angles in the two molecules are generally as expected.

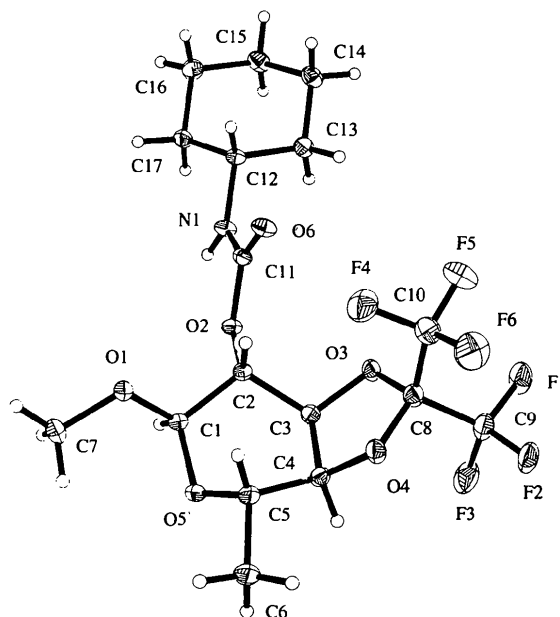


Fig. 1. *SHELXTL-Plus* (Sheldrick, 1992) drawing of the molecular conformation of molecule (I) showing displacement ellipsoids of 30% probability and the atomic numbering scheme.

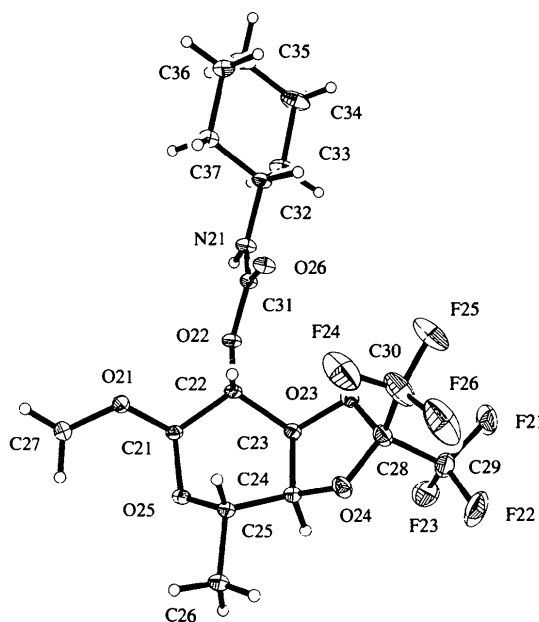


Fig. 2. *SHELXTL-Plus* (Sheldrick, 1992) drawing of molecule (II) showing displacement ellipsoids of 30% probability and the atomic numbering scheme.

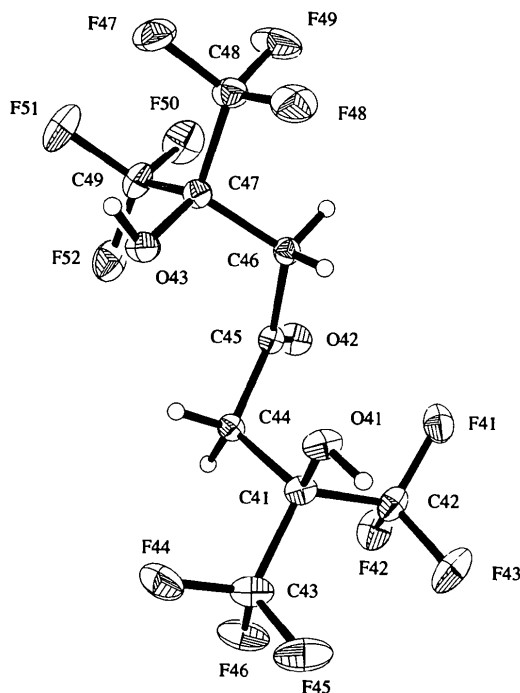


Fig. 3. *SHELXTL-Plus* (Sheldrick, 1992) drawing of molecule (III) showing displacement ellipsoids of 30% probability and the atomic numbering scheme.

The two molecules of the α -L-altropyranose derivative have a pyranoid conformation which lies between a boat ($\theta = 90^\circ$, $\Phi = 120^\circ$) and a twisted-boat ($\theta = 90^\circ$, $\Phi = 150^\circ$) conformation. While the puckering parameters (Cremer & Pople, 1975) of molecule (I) [$Q = 0.771$ (2) Å, $\theta = 95.4$ (1), $\Phi = 142.5$ (2) $^\circ$] point more to the twisted-boat conformation, the puckering parameters of molecule (II) [$Q = 0.768$ (2) Å, $\theta = 93.3$ (1), $\Phi = 135.7$ (2) $^\circ$] show a ring conformation which lies almost ideally between the twisted-boat and boat conformations. A similar conformation was reported for methyl 2,6-dichloro-2,6-dideoxy-3,4-isopropylidene- α -D-altropyranoside (Lin, Sundaralingam & Jackobs, 1973). Since this structure has the D-altro configuration, the puckering parameters ($Q = 0.776$ Å, $\theta = 84.8$, $\Phi = 320.1^\circ$) are 'inverted' compared to the α -L-altropyranose derivative.

The puckering parameters of the five-membered isopropylidene rings are: $Q = 0.195$ (3) Å, $\Phi = 91.4$ (7) $^\circ$ for (I) and $Q = 0.186$ (3) Å, $\Phi = 103.6$ (8) $^\circ$ for (II). This means that both isopropylidene rings of the two molecules of the α -L-altropyranose derivative adopt rather flat conformations. While the ring conformation of molecule (I) corresponds to a C^3T_{C4} twist ($\Phi = 90^\circ$) conformation, the ring conformation of molecule (II) points more to an E_{C24} envelope ($\Phi = 108^\circ$) conformation.

All bond lengths and angles in both molecules of the title α -L-altropyranose derivative are very similar. There is, however, a difference in the general conformation of

the carbamoyl moiety. The cyclohexyl ring twist around the N—C bond for molecule (II) differs by 57.4 (3) $^\circ$ from the same twist in molecule (I).

The program used to calculate these special molecular-geometry details was *PLATON* (Spek, 1990).

Experimental

Crystal data

2C₁₇H₂₃F₆NO₆·C₉H₆F₁₂O₃

$M_r = 1292.87$

Monoclinic

$P2_1$

$a = 11.623$ (1) Å

$b = 20.861$ (2) Å

$c = 12.570$ (1) Å

$\beta = 116.47$ (1) $^\circ$

$V = 2728.3$ (4) Å³

$Z = 2$

$D_x = 1.574$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.54178$ Å

Cell parameters from 25

reflections

$\theta = 42.0$ – 50.9°

$\mu = 1.522$ mm⁻¹

$T = 173$ (1) K

Transparent prisms

$0.5 \times 0.4 \times 0.4$ mm

Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

$2\theta/\omega$ scans

Absorption correction:

none

12 030 measured reflections

11 034 independent reflections

10 719 observed reflections [$I > 2\sigma(I)$]

$R_{int} = 0.0384$

$\theta_{max} = 76.5^\circ$

$h = 0 \rightarrow 14$

$k = 0 \rightarrow 26$

$l = -15 \rightarrow 15$

3 standard reflections

frequency: 120 min

intensity decay: 1%

Refinement

Refinement on F^2

$R(F) = 0.0475$

$wR(F^2) = 0.1284$

$S = 1.038$

11 027 reflections

816 parameters

H atoms with riding model

$w = 1/[\sigma^2(F_o^2) + (0.0886P)^2 + 0.9226P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.003$

$\Delta\rho_{max} = 0.386$ e Å⁻³

$\Delta\rho_{min} = -0.243$ e Å⁻³

Extinction correction:

SHELXL93 (Sheldrick, 1993)

Extinction coefficient:

0.0013 (2)

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Absolute configuration:

$\chi = 0.12$ (8) (Flack, 1983)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i\cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
F1	1.0418 (2)	0.41370 (9)	0.7559 (2)	0.0519 (5)
F2	1.0042 (2)	0.32336 (9)	0.6656 (2)	0.0477 (4)
F3	0.8763 (2)	0.40222 (11)	0.5876 (2)	0.0583 (5)
F4	0.8653 (2)	0.30308 (12)	0.9169 (2)	0.0591 (5)
F5	1.0299 (2)	0.36034 (13)	0.9469 (2)	0.0656 (6)
F6	1.0032 (2)	0.26878 (12)	0.8617 (2)	0.0677 (6)

O1	0.4098 (2)	0.34501 (8)	0.75136 (14)	0.0257 (3)
O2	0.59332 (15)	0.45879 (7)	0.81527 (13)	0.0225 (3)
O3	0.8053 (2)	0.40702 (8)	0.7670 (2)	0.0285 (3)
O4	0.7735 (2)	0.30937 (8)	0.6774 (2)	0.0301 (4)
O5	0.4396 (2)	0.34682 (8)	0.58026 (13)	0.0255 (3)
O6	0.7393 (2)	0.42587 (8)	0.99906 (14)	0.0284 (3)
N1	0.6453 (2)	0.52504 (9)	0.9658 (2)	0.0252 (4)
C1	0.4547 (2)	0.38101 (10)	0.6828 (2)	0.0220 (4)
C2	0.5951 (2)	0.39757 (10)	0.7637 (2)	0.0207 (4)
C3	0.6699 (2)	0.40340 (10)	0.6903 (2)	0.0230 (4)
C4	0.6542 (2)	0.34338 (11)	0.6142 (2)	0.0239 (4)
C5	0.5426 (2)	0.30220 (11)	0.6042 (2)	0.0253 (4)
C6	0.4975 (3)	0.25427 (13)	0.5028 (2)	0.0341 (5)
C7	0.2751 (3)	0.3312 (2)	0.6873 (2)	0.0384 (6)
C8	0.8650 (2)	0.35312 (12)	0.7503 (2)	0.0292 (5)
C9	0.9494 (3)	0.37352 (13)	0.6898 (3)	0.0373 (6)
C10	0.9427 (3)	0.3207 (2)	0.8714 (3)	0.0437 (6)
C11	0.6678 (2)	0.46723 (10)	0.9351 (2)	0.0219 (4)
C12	0.7155 (2)	0.55360 (10)	1.0840 (2)	0.0234 (4)
C13	0.8352 (2)	0.58867 (11)	1.0944 (2)	0.0284 (5)
C14	0.9034 (2)	0.62308 (12)	1.2147 (2)	0.0315 (5)
C15	0.8124 (3)	0.66928 (12)	1.2334 (2)	0.0334 (5)
C16	0.6933 (3)	0.63451 (14)	1.2235 (2)	0.0361 (6)
C17	0.6252 (2)	0.59954 (12)	1.1047 (2)	0.0297 (5)
F21	1.2112 (2)	0.03538 (10)	0.3456 (2)	0.0617 (6)
F22	1.2480 (2)	0.12659 (13)	0.4325 (3)	0.0911 (10)
F23	1.1589 (2)	0.05208 (12)	0.4876 (2)	0.0637 (6)
F24	0.9092 (3)	0.15164 (14)	0.1215 (2)	0.0842 (9)
F25	1.0396 (3)	0.07822 (13)	0.1252 (2)	0.0759 (7)
F26	1.1137 (4)	0.16938 (12)	0.2056 (4)	0.0961 (11)
O21	0.5262 (2)	0.11489 (8)	0.21893 (13)	0.0248 (3)
O22	0.6767 (2)	0.00391 (7)	0.20063 (13)	0.0240 (3)
O23	0.9440 (2)	0.05132 (8)	0.2798 (2)	0.0304 (4)
O24	0.9932 (2)	0.15173 (8)	0.3567 (2)	0.0339 (4)
O25	0.7066 (2)	0.13492 (9)	0.39784 (14)	0.0290 (3)
O26	0.6891 (2)	0.02351 (8)	0.02745 (14)	0.0283 (3)
N21	0.6262 (2)	-0.07331 (9)	0.06888 (2)	0.0264 (4)
C21	0.6416 (2)	0.08932 (10)	0.3078 (2)	0.0218 (4)
C22	0.7227 (2)	0.06642 (10)	0.2457 (2)	0.0215 (4)
C23	0.8630 (2)	0.06235 (10)	0.3373 (2)	0.0224 (4)
C24	0.9108 (2)	0.12582 (11)	0.4037 (2)	0.0244 (4)
C25	0.8019 (2)	0.17210 (11)	0.3811 (2)	0.0258 (4)
C26	0.8420 (3)	0.22749 (13)	0.4672 (2)	0.0360 (6)
C27	0.4333 (2)	0.12494 (12)	0.2635 (2)	0.0299 (5)
C28	1.0299 (3)	0.10199 (12)	0.3053 (2)	0.0325 (5)
C29	1.1651 (3)	0.0786 (2)	0.3941 (3)	0.0486 (8)
C30	1.0224 (4)	0.1256 (2)	0.1862 (4)	0.0594 (10)
C31	0.6651 (2)	-0.01301 (11)	0.0911 (2)	0.0226 (4)
C32	0.6063 (2)	-0.10821 (11)	-0.0396 (2)	0.0268 (4)
C33	0.6279 (3)	-0.17932 (13)	-0.0078 (2)	0.0393 (6)
C34	0.5992 (4)	-0.22093 (13)	-0.1176 (3)	0.0473 (7)
C35	0.4663 (3)	-0.2077 (2)	-0.2149 (3)	0.0502 (8)
C36	0.4498 (3)	-0.1369 (2)	-0.2485 (3)	0.0434 (7)
C37	0.4747 (3)	-0.09481 (15)	-0.1401 (2)	0.0380 (6)
F41	2.0299 (2)	-0.02570 (9)	0.6405 (2)	0.0463 (4)
F42	1.9662 (2)	-0.11517 (10)	0.54889 (15)	0.0476 (4)
F43	2.1468 (2)	-0.10990 (13)	0.7057 (2)	0.0593 (5)
F44	1.8996 (2)	-0.18972 (9)	0.8222 (2)	0.0528 (5)
F45	2.0914 (2)	-0.19119 (11)	0.8388 (2)	0.0644 (6)
F46	1.9311 (2)	-0.21168 (8)	0.6699 (2)	0.0584 (5)
F47	1.6547 (2)	0.12612 (9)	0.8539 (2)	0.0525 (5)
F48	1.8493 (2)	0.12135 (10)	0.8771 (2)	0.0545 (5)
F49	1.7030 (3)	0.16087 (8)	0.7178 (2)	0.0626 (6)
F50	1.5386 (2)	0.07959 (12)	0.5536 (2)	0.0593 (5)
F51	1.4786 (2)	0.05117 (13)	0.6860 (2)	0.0648 (6)
F52	1.5442 (2)	-0.01904 (11)	0.6019 (2)	0.0511 (4)
O41	2.0138 (2)	-0.06560 (9)	0.83787 (15)	0.0335 (4)
O42	1.7486 (2)	-0.01594 (10)	0.5085 (2)	0.0377 (4)
O43	1.7255 (2)	0.00083 (9)	0.8293 (2)	0.0343 (4)
C41	1.9580 (2)	-0.10292 (12)	0.7351 (2)	0.0280 (5)
C42	2.0262 (2)	-0.08870 (14)	0.6563 (2)	0.0351 (5)
C43	1.9704 (3)	-0.17478 (14)	0.7668 (2)	0.0420 (6)
C44	1.8141 (2)	-0.08706 (11)	0.6716 (2)	0.0252 (4)
C45	1.7830 (2)	-0.02153 (11)	0.6147 (2)	0.0243 (4)
C46	1.7998 (2)	0.03804 (11)	0.6897 (2)	0.0258 (4)
C47	1.7035 (2)	0.04745 (12)	0.7423 (2)	0.0274 (5)
C48	1.7270 (3)	0.11469 (13)	0.7983 (2)	0.0365 (6)
C49	1.5650 (3)	0.0401 (2)	0.6451 (3)	0.0393 (6)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
O41—H41...O6 ⁱ	0.84 (2)	1.89 (2)	2.688 (3)	158 (3)
O43—H43...O26 ⁱⁱ	0.84 (3)	1.94 (3)	2.747 (3)	161 (1)
N1—H11...O21 ⁱⁱⁱ	0.880 (3)	2.096 (3)	2.958 (3)	166.2 (3)
N21—H211...O1 ^{iv}	0.879 (3)	2.133 (3)	3.005 (3)	171.6 (3)

Symmetry codes: (i) $3 - x, y - \frac{1}{2}, 2 - z$; (ii) $1 + x, y, 1 + z$; (iii) $1 - x, \frac{3}{2} + y, 1 - z$; (iv) $1 - x, y - \frac{1}{2}, 1 - z$.

Friedel opposites were collected in the ranges $h - 14$ to 0 , $k - 26$ to 0 and $l - 15$ to 15 . These Friedel pairs are regarded as symmetry independent and were not merged. Since the α -L-altropyranose derivative crystallizes in a polar space group, the origin needs to be fixed in y . Therefore, polar axis restraints were applied by the method of Flack & Schwarzenbach (1988).

The structure was solved using *SHELXS90* (Sheldrick, 1990) and a certain amount of brute force (16 h CPU time on an IBM RS6000/32H, equivalent to about 0.15 VAX years). 50 000 random starting phase sets for 600 high E values gave four correct solutions ($R\alpha = 0.067$, $NQUAL = -0.76$). The top 84 peaks of the resulting E map gave the complete structure.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-SHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS90* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1992). Software used to prepare material for publication: *CIF2TEX* (Kopf, 1992a) and *FCF2FOC* (Kopf, 1992b).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1073). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5. Enraf-Nonius, Delft, The Netherlands.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Flack, H. D. & Schwarzenbach, D. (1988). *Acta Cryst.* **A44**, 499–506.
- Kopf, J. (1987). *CAD-SHEL. Program for Data Reduction of Enraf-Nonius CAD-4 Data*. Univ. of Hamburg, Germany.
- Kopf, J. (1992a). *CIF2TEX. Program for Automatic Preparation of Deposition Data*. Univ. of Hamburg, Germany.
- Kopf, J. (1992b). *FCF2FOC. Program for Printing F_o^2 and F_o^2* . Univ. of Hamburg, Germany.
- Lin, G. H., Sundaralingam, M. & Jackobs, J. (1973). *Carbohydr. Res.* **29**, 439–449.
- Miethchen, R., Rentsch, D. & Michalik, M. (1994). *Liebigs Ann. Chem.* pp. 219–222.
- Miethchen, R., Rentsch, D. & Stoll, N. (1992). *Tetrahedron*, **48**, 8393–8400.
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1992). *SHELXTL-Plus*. Release 4.21/V. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. Univ. of Göttingen, Germany.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-31.
- Vital, J. J. & Willis, C. J. (1993). *Acta Cryst.* **C49**, 310–313.