The ring containing S1 is disordered such that the S atom is 80% at the site labelled S1 and 20% at the site labelled C3. Appropriately averaged scattering factors were used for the two positions.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1988). Cell refinement: *CAD-4 Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *MolEN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HA1104). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(5-acetyl-3-thienyl)methane–Cyclononanone (1/1) Inclusion Compound

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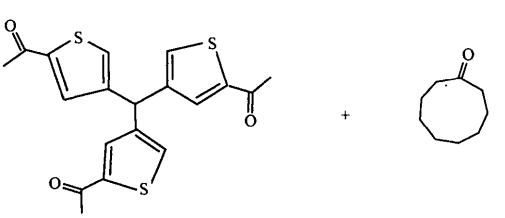
Abstract

Tris(5-acetyl-3-thienyl)methane (TATM) forms an inclusion compound with cyclononanone in a host/guest ratio of 1/1, $C_{19}H_{16}O_3S_3.C_9H_{16}O$. The crystal belongs to the monoclinic system. The cavity formed by the TATM molecule is of a zigzag-channel type, parallel to the *c* axis. The cyclononanone guest molecule occupies two orientationally disordered positions with occupation factors of 25 and 75%. The two cyclononanone molecules each have the ($g^-g^-ggg^-g^-sg^-s$) conformation.

Comment

The tris(5-acetyl-3-thienyl)methane (TATM) molecule forms host/guest inclusion compounds with a large number of organic molecules (Bin Din & Meth-Cohn, 1977). The crystal structures of a few TATM inclusion compounds have been published recently, with ethyl acetate (Van Rooyen & Roos, 1991*a*), benzene (van Rooyen & Roos, 1991*b*), *n*-hexane (Roos & Dillen, 1992) and ethanol (Dillen & Roos, 1992) as guest molecules. These compounds belong to the triclinic system and have a host/guest ratio of 2/1. The crystal obtained in this work for the cyclononanone inclusion compound (1) is monoclinic $P2_1/c$ and has a host/guest ratio of 1/1. The cyclononanone guest molecule is larger than the other guest molecules whose structures were reported with 2/1 stoichiometry. This leads to a rearrangement of the structural units, which now crystallize with a host/guest ratio of 1/1. Similar changes in stoichiometry are often encountered in inclusion compounds (Atwood, Davies & MacNicol, 1984). There are four TATM host molecules and four cyclononanone guest molecules in the unit cell. The TATM host molecules are ordered, but the cyclononanone guest molecules are disordered over two orientations with 25 and 75% occupancy; they have nearly the same conformation and very similar orientations (Fig. 1).



(1)

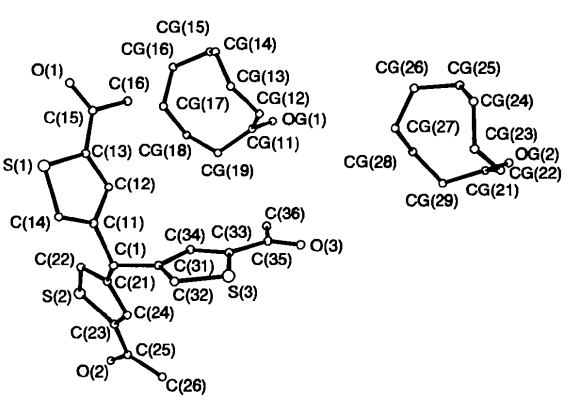


Fig. 1. The molecular structure of the title complex with the atomic numbering scheme. The guest molecule on the right is the major component.

The stereoscopic pair in Fig. 2 shows that the TATM molecules form channel-type cavities along the *c* axis in which the cyclononanone guest molecules are disordered in a zigzag way. The two cyclononanone molecules have nearly the same conformation so that the disorder effect is of the orientational type. It can be seen from Table 2 that both guest molecules are in the ($g^-g^-ggg^-g^-sg^-s$) conformation, which is in agreement with that predicted for the cyclononane molecule (Dale, 1973). Slightly higher thermal factors of the guest molecule is a normal occurrence considering their disorder.

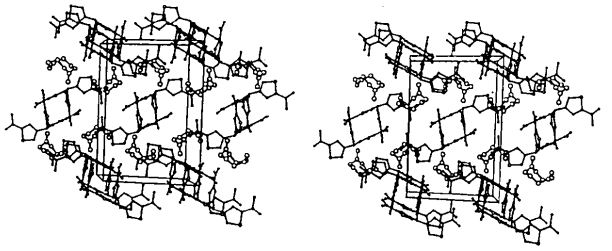


Fig. 2. Stereoscopic view of the unit-cell contents of TATM–cyclononanone down the *a* axis (the minor orientation of cyclononanone has been omitted for clarity). The *c* axis is horizontal.

Experimental

The host molecule, TATM, was synthesized according the method described by Yakubov, Sudarushkin, Belenkii & Gold'farb (1973), and characterized by ^{1}H NMR. The cyclononanone inclusion compound was formed by recrystallization of the host molecule from the guest solvent. The host/guest ratio was determined by repeated crystal density measurements (flotation in KI/H_2O). A well developed single crystal was selected and mounted on the tip of a glass fibre for data collection at 220 K.

Crystal data

$C_{19}H_{16}O_3S_3.C_9H_{16}O$
$M_r = 528.7$
Monoclinic
$P2_1/c$
$a = 10.994$ (2) Å
$b = 19.464$ (4) Å
$c = 13.417$ (1) Å
$\beta = 109.40$ (1)°
$V = 2708.1$ (8) Å^3
$Z = 4$
$D_x = 1.30$ Mg m^{-3} (220 K)
$D_m = 1.24$ (2) Mg m^{-3} (room temperature)

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 20.0$–$22.5°$
$\mu = 2.71$ mm^{-1}
$T = 220$ K
Platelet
0.61 × 0.23 × 0.09 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: by integration from crystal shape
$T_{min} = 0.739$, $T_{max} = 0.943$
10 110 measured reflections
5138 independent reflections
4256 observed reflections [$I > 1.96\sigma(I)$]

$R_{int} = 0.043$
$\theta_{max} = 70°$
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 23$
$l = -16 \rightarrow 16$
5 standard reflections monitored every 400 reflections
intensity variation: 1.3%

Refinement

Refinement on F
$R = 0.055$
$wR = 0.059$
$S = 2.44$
4255 reflections
414 parameters
$w = 1/[\sigma^2(F) + 0.0001F^2]$
$(\Delta/\sigma)_{max} = 0.61$

$\Delta\rho_{max} = 0.58$ e Å^{-3}
$\Delta\rho_{min} = -0.38$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

U_{iso} for guest molecule I; $U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	U_{eq}/U_{iso}
Host molecule				
S(1)	1.11335 (8)	−0.05284 (4)	0.12793 (7)	0.0414 (3)
S(2)	0.79164 (8)	−0.24288 (4)	0.39006 (6)	0.0409 (3)
S(3)	0.41166 (7)	−0.09612 (4)	−0.02957 (6)	0.0403 (3)
O(1)	1.1516 (2)	0.0964 (1)	0.1733 (2)	0.0496 (8)
O(2)	0.8103 (3)	−0.1640 (1)	0.5816 (2)	0.0680 (10)
O(3)	0.3355 (2)	−0.0348 (1)	−0.2449 (2)	0.0423 (7)
C(1)	0.7847 (3)	−0.1473 (1)	0.1219 (2)	0.0295 (9)
C(11)	0.8940 (3)	−0.0983 (2)	0.1278 (2)	0.0308 (9)
C(12)	1.0061 (3)	−0.1180 (2)	0.1135 (2)	0.0384 (10)
C(13)	1.0080 (3)	0.0045 (2)	0.1528 (2)	0.0325 (9)
C(14)	0.8958 (3)	−0.0272 (2)	0.1499 (2)	0.0315 (9)
C(15)	1.0445 (3)	0.0771 (2)	0.1702 (2)	0.0376 (10)
C(16)	0.9461 (3)	0.1269 (2)	0.1805 (3)	0.0552 (12)
C(21)	0.7836 (3)	−0.1681 (2)	0.2307 (2)	0.0302 (9)
C(22)	0.7886 (3)	−0.2347 (2)	0.2626 (2)	0.0342 (9)
C(23)	0.7869 (3)	−0.1550 (2)	0.4035 (2)	0.0343 (9)
C(24)	0.7818 (3)	−0.1218 (2)	0.3118 (2)	0.0324 (9)
C(25)	0.7972 (3)	−0.1261 (2)	0.5067 (3)	0.0442 (11)
C(26)	0.7950 (4)	−0.0499 (2)	0.5181 (3)	0.0602 (12)
C(31)	0.6549 (3)	−0.1198 (1)	0.0520 (2)	0.0296 (9)
C(32)	0.5414 (3)	−0.1267 (2)	0.0711 (2)	0.0358 (9)
C(33)	0.5090 (3)	−0.0715 (2)	−0.1004 (2)	0.0317 (9)
C(34)	0.6364 (3)	−0.0874 (2)	−0.0471 (2)	0.0320 (9)
C(35)	0.4529 (3)	−0.0374 (2)	−0.2026 (2)	0.0365 (9)
C(36)	0.5421 (3)	−0.0058 (2)	−0.2520 (3)	0.0567 (12)
Guest molecule I (occupancy 0.25)				
OG(1)	0.7136 (15)	0.3047 (7)	0.4013 (13)	0.031 (2)
CG(11)	0.7278 (12)	0.2440 (7)	0.3916 (16)	0.046 (3)
CG(12)	0.8548 (12)	0.2095 (9)	0.4503 (11)	0.070 (3)
CG(13)	0.8611 (17)	0.1592 (10)	0.5365 (13)	0.110 (3)
CG(14)	0.7952 (14)	0.1659 (11)	0.6170 (13)	0.111 (3)
CG(15)	0.6744 (14)	0.2084 (9)	0.6010 (15)	0.129 (3)
CG(16)	0.5488 (14)	0.1677 (11)	0.5544 (11)	0.123 (3)
CG(17)	0.5382 (18)	0.1315 (8)	0.4496 (11)	0.113 (3)
CG(18)	0.5094 (12)	0.1817 (9)	0.3589 (11)	0.072 (3)
CG(19)	0.6216 (12)	0.1991 (9)	0.3200 (11)	0.065 (3)
Guest molecule II (occupancy 0.75)				
OG(2)	0.7167 (9)	0.3024 (4)	0.3967 (7)	0.084 (2)
CG(21)	0.7097 (5)	0.2409 (3)	0.3882 (6)	0.053 (1)
CG(22)	0.8329 (6)	0.2007 (3)	0.4133 (4)	0.070 (2)
CG(23)	0.8497 (5)	0.1374 (2)	0.4797 (4)	0.054 (1)
CG(24)	0.8661 (5)	0.1495 (3)	0.5969 (3)	0.056 (1)

CG(25)	0.7626 (5)	0.1908 (3)	0.6224 (4)	0.061 (1)
CG(26)	0.6434 (5)	0.1488 (3)	0.6105 (5)	0.084 (2)
CG(27)	0.5570 (6)	0.1301 (3)	0.5022 (5)	0.094 (2)
CG(28)	0.5087 (5)	0.1880 (3)	0.4230 (5)	0.089 (2)
CG(29)	0.5818 (5)	0.2037 (3)	0.3455 (5)	0.072 (2)

Table 2. *Selected geometric parameters* (Å, °)

Host molecule			
S(1)—C(12)	1.698 (3)	C(13)—C(14)	1.368 (4)
S(1)—C(13)	1.719 (3)	C(13)—C(15)	1.466 (5)
S(2)—C(22)	1.707 (3)	C(15)—C(16)	1.493 (5)
S(2)—C(23)	1.723 (3)	C(21)—C(22)	1.361 (4)
S(3)—C(32)	1.713 (3)	C(21)—C(24)	1.418 (4)
S(3)—C(33)	1.719 (3)	C(23)—C(24)	1.374 (4)
O(1)—C(15)	1.223 (4)	C(23)—C(25)	1.464 (5)
O(2)—C(25)	1.216 (4)	C(25)—C(26)	1.492 (5)
O(3)—C(35)	1.226 (4)	C(31)—C(32)	1.361 (5)
C(1)—C(11)	1.515 (4)	C(31)—C(34)	1.424 (4)
C(1)—C(21)	1.519 (4)	C(33)—C(34)	1.380 (4)
C(1)—C(31)	1.521 (4)	C(33)—C(35)	1.463 (4)
C(11)—C(12)	1.364 (5)	C(35)—C(36)	1.487 (5)
C(11)—C(14)	1.414 (4)		
Guest molecule I			
OG(1)—CG(11)	1.20 (2)	CG(14)—CG(15)	1.52 (2)
CG(11)—CG(12)	1.51 (2)	CG(15)—CG(16)	1.53 (2)
CG(11)—CG(19)	1.52 (2)	CG(16)—CG(17)	1.54 (2)
CG(12)—CG(13)	1.50 (2)	CG(17)—CG(18)	1.51 (2)
CG(13)—CG(14)	1.49 (2)	CG(18)—CG(19)	1.53 (2)
Guest molecule II			
OG(2)—CG(21)	1.202 (10)	CG(24)—CG(25)	1.522 (8)
CG(21)—CG(22)	1.502 (9)	CG(25)—CG(26)	1.507 (8)
CG(21)—CG(29)	1.515 (9)	CG(26)—CG(27)	1.493 (9)
CG(22)—CG(23)	1.495 (7)	CG(27)—CG(28)	1.519 (9)
CG(23)—CG(24)	1.540 (6)	CG(28)—CG(29)	1.541 (8)
Host molecule			
C(12)—S(1)—C(13)	91.1 (2)	S(2)—C(22)—C(21)	112.9 (2)
C(22)—S(2)—C(23)	91.2 (2)	S(2)—C(23)—C(24)	111.5 (2)
C(32)—S(3)—C(33)	91.4 (2)	S(2)—C(23)—C(25)	119.0 (2)
C(11)—C(1)—C(21)	112.1 (2)	C(24)—C(23)—C(25)	129.3 (3)
C(11)—C(1)—C(31)	112.1 (2)	C(21)—C(24)—C(23)	112.4 (3)
C(21)—C(1)—C(31)	112.4 (2)	O(2)—C(25)—C(23)	120.0 (3)
C(1)—C(11)—C(12)	123.6 (3)	O(2)—C(25)—C(26)	121.3 (3)
C(1)—C(11)—C(14)	125.6 (3)	C(23)—C(25)—C(26)	118.7 (3)
C(12)—C(11)—C(14)	110.8 (3)	C(1)—C(31)—C(32)	125.5 (3)
S(1)—C(12)—C(11)	113.6 (2)	C(1)—C(31)—C(34)	123.1 (3)
S(1)—C(13)—C(14)	111.2 (2)	C(32)—C(31)—C(34)	111.3 (3)
S(1)—C(13)—C(15)	119.4 (2)	S(3)—C(32)—C(31)	113.1 (2)
C(14)—C(13)—C(15)	129.5 (3)	S(3)—C(33)—C(34)	111.2 (2)
C(11)—C(14)—C(13)	113.4 (3)	S(3)—C(33)—C(35)	119.9 (2)
O(1)—C(15)—C(13)	120.7 (3)	C(34)—C(33)—C(35)	128.8 (3)
O(1)—C(15)—C(16)	121.1 (3)	C(31)—C(34)—C(33)	112.9 (3)
C(13)—C(15)—C(16)	118.2 (3)	O(3)—C(35)—C(33)	120.3 (3)
C(1)—C(21)—C(22)	123.0 (3)	O(3)—C(35)—C(36)	121.7 (3)
C(1)—C(21)—C(24)	125.1 (3)	C(33)—C(35)—C(36)	118.1 (3)
C(22)—C(21)—C(24)	111.9 (3)		
Guest molecule I			
OG(1)—CG(11)—CG(12)	121. (2)	CG(14)—CG(15)—CG(16)	114. (1)
OG(1)—CG(11)—CG(19)	122. (2)	CG(15)—CG(16)—CG(17)	113. (1)
CG(12)—CG(11)—CG(19)	117. (1)	CG(16)—CG(17)—CG(18)	112. (1)
CG(11)—CG(12)—CG(13)	119. (1)	CG(17)—CG(18)—CG(19)	116. (1)
CG(12)—CG(13)—CG(14)	126. (2)	CG(11)—CG(19)—CG(18)	117. (1)
CG(13)—CG(14)—CG(15)	124. (2)		
Guest molecule II			
OG(2)—CG(21)—CG(22)	118.2 (7)	CG(24)—CG(25)—CG(26)	112.2 (5)
OG(2)—CG(21)—CG(29)	122.3 (7)	CG(25)—CG(26)—CG(27)	119.0 (5)
CG(22)—CG(21)—CG(29)	119.4 (6)	CG(26)—CG(27)—CG(28)	117.5 (5)
CG(21)—CG(22)—CG(23)	119.1 (5)	CG(27)—CG(28)—CG(29)	118.7 (5)
CG(22)—CG(23)—CG(24)	115.6 (4)	CG(21)—CG(29)—CG(28)	117.8 (5)
CG(23)—CG(24)—CG(25)	117.9 (4)		
Guest molecule I			
OG(1)—CG(11)—CG(12)—CG(13)		112 (2)	
OG(1)—CG(11)—CG(19)—CG(18)		−74 (2)	
CG(19)—CG(11)—CG(12)—CG(13)		−69 (2)	
CG(11)—CG(12)—CG(13)—CG(14)		−40 (2)	
CG(12)—CG(11)—CG(19)—CG(18)		107 (2)	
CG(12)—CG(13)—CG(14)—CG(15)		25 (3)	
CG(13)—CG(14)—CG(15)—CG(16)		92 (2)	
CG(14)—CG(15)—CG(16)—CG(17)		−55 (2)	
CG(15)—CG(16)—CG(17)—CG(18)		−74 (2)	
CG(16)—CG(17)—CG(18)—CG(19)		105 (2)	
CG(17)—CG(18)—CG(19)—CG(11)		−72 (2)	
Guest molecule II			
OG(2)—CG(21)—CG(22)—CG(23)		134 (8)	
OG(2)—CG(21)—CG(29)—CG(28)		−83.5 (9)	
CG(29)—CG(21)—CG(22)—CG(23)		−49.8 (8)	
CG(21)—CG(22)—CG(23)—CG(24)		−70.0 (7)	
CG(22)—CG(21)—CG(29)—CG(28)		101.2 (7)	
CG(22)—CG(23)—CG(24)—CG(25)		55.1 (6)	
CG(23)—CG(24)—CG(25)—CG(26)		78.0 (6)	
CG(24)—CG(25)—CG(26)—CG(27)		−72.0 (7)	
CG(25)—CG(26)—CG(27)—CG(28)		−53.4 (8)	
CG(26)—CG(27)—CG(28)—CG(29)		96.0 (7)	
CG(27)—CG(28)—CG(29)—CG(21)		−75.2 (7)	

The structure was solved by direct methods with *MULTAN*80 (Main, Fiske, Hall, Lessinger, Germain, Declercq & Woolfson, 1980) and refined by full-matrix least squares, initially using the *NRCVAX* program (Gabe, Le Page, Charland, Lee & White, 1989). The disordered structure was refined by constraining the C—C distances to 1.53 (1) Å, C═O distances to 1.21 (1) Å, and the non-bonded C···C distance to 2.58 (2) Å, using *SHELX*76 (Sheldrick, 1976). All non-H atoms of the TATM molecule were refined with anisotropic displacement parameters. The structure of the two guest molecules was established from difference Fourier syntheses. Only the non-H atoms of the major molecule were refined anisotropically. The occupancy factors of 25 and 75% were arrived at after several refinement cycles keeping the displacement parameters constant. The positions of all the H atoms were calculated (C—H = 0.95 Å, C—C—H = 105–125°) except for one H atom of each CH_3 group of the TATM molecule which was found from the difference Fourier syntheses and refined. The two remaining H-atom positions were calculated. H atoms were refined isotropiocally. One reidual peak of 0.58 e $Å^{-3}$ was located at 0.88 Å from S(2) atom of the TATM molecule. All other peaks were less than 0.40 e $Å^{-3}$.

The National Sciences and Engineering Research Council of Canada supported this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and least-squares-planes data have been deposited with the IUCr (Reference: CR1100). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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DL-Valine NCA

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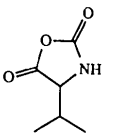
Abstract

The crystal of *N*-carboxy-DL-valine anhydride (DL-valine NCA), $C_6H_9NO_3$, has a similar layer structure to that observed in the crystal of L-valine NCA [Kanazawa, Ohashi & Sasada (1984). *Acta Cryst.* C**40**, 1094–1096]. However, the D and L molecules are connected alternately by N—H···O hydrogen bonds in each layer.

Comment

The *N*-carboxy anhydrides (NCA) of amino acids are useful monomers for the synthesis of polypeptides. These compounds are generally unstable to moisture and heat. A series of studies has been made on crystal structures of these compounds in order to explain their polymerizability in the crystalline state: glycine NCA (Kanazawa *et al.*, 1976*a*), L-alanine NCA (Kanazawa *et al.*, 1976*b*), γ-benzyl-L-glutamate (BLG) NCA (Kanazawa, Ohashi, Sasada & Kawai, 1978*a*), L-leucine NCA (Kanazawa, Ohashi, Sasada & Kawai, 1978*b*) and L-valine NCA (Kanazawa, Ohashi & Sasada, 1984). There are four types of hydrogen bonding in these compounds: the N1···O1 dimer type (glycine NCA), the N1···O1 layer type (L-leucine NCA and L-valine NCA), the N1···O3 type (L-alanine NCA) and the N1···O4 type (BLG NCA). In this paper, the crystal structure of DL-valine NCA, (I), is determined and compared with those of the related compounds.



(I)

The crystals were prepared in a way similar to those of the related compounds. The molecular structure is shown in Fig. 1. Bond distances and angles are consistent with the corresponding ones in L-valine NCA. The bond distances C1—O2 and C2—O2 are in good agreement with those in the N1···O1 dimer and layer-type compounds. This means that O1 is likely to have a negative charge and O2 a positive charge as a result of the resonance in the five-membered ring caused by the intermolecular hydrogen bond (Kanazawa, Ohashi, Sasada & Kawai, 1978*a*). In this case, CO_2 can be readily cleaved from the five-membered ring.

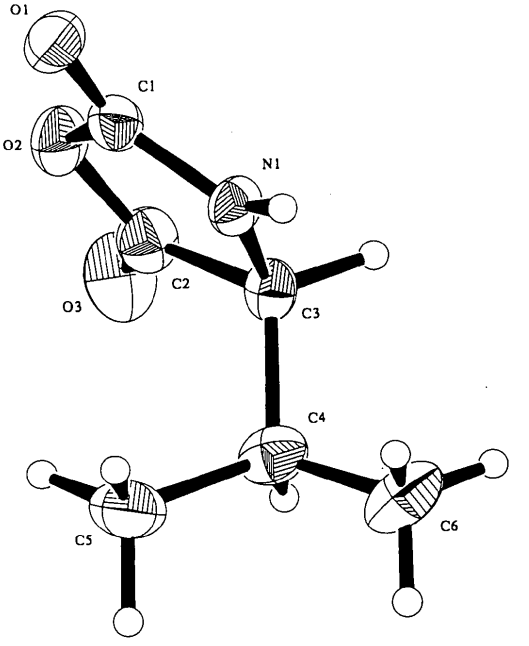


Fig. 1. View of the L isomer of DL-valine NCA showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 30% probability level, H atoms are drawn as small circles of arbitrary radii.

The crystal structure is shown in Fig. 2. The molecules are connected along the *c* axis by the intermolecular hydrogen bond N1···O1 of 2.935 (6) Å as indicated by dashed lines in Fig. 2. The polymerizing moiety, the five-membered ring, forms a layer structure parallel to the *c* axis and this layer is interwoven with the hydrophobic side-chain layers. Such layer structures are observed in L-leucine NCA and L-valine NCA, *i.e.* the N1···O1 layer-type compounds. The reactivities of

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