2392 measured reflections
2108 independent reflections
1615 observed reflections
[$F > 4\sigma(F)$]

2 standard reflections
monitored every 50 reflections
intensity decay: none

Refinement

Refinement on F
$R = 0.061$
$wR = 0.105$
$S = 1.17$
1615 reflections
199 parameters
H atoms located by difference synthesis and refined isotropically

$w = 1/[\sigma^2(F) + 0.0070F^2]$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.34$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1991)

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

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	0.1739 (3)	0.0863 (2)	0.9600 (2)	0.046 (1)
C(2)	0.3011 (4)	0.0208 (3)	1.0287 (2)	0.057 (1)
C(3)	0.4164 (4)	0.0746 (3)	1.1074 (3)	0.066 (1)
C(4)	0.4056 (4)	0.1915 (3)	1.1164 (2)	0.066 (1)
C(5)	0.0467 (3)	0.0346 (2)	0.8742 (2)	0.049 (1)
C(6)	0.0305 (4)	−0.0777 (3)	0.8368 (2)	0.058 (1)
C(7)	−0.1139 (4)	−0.0826 (3)	0.7517 (3)	0.068 (1)
C(8)	−0.1823 (4)	0.0247 (3)	0.7374 (2)	0.066 (1)
C(9)	0.2628 (5)	0.3753 (3)	1.0589 (3)	0.070 (1)
C(10)	0.1409 (7)	0.4014 (3)	1.1183 (4)	0.105 (2)
N(1)	0.2828 (3)	0.2488 (2)	1.0498 (2)	0.054 (1)
N(2)	0.1647 (3)	0.2003 (2)	0.9710 (2)	0.049 (1)
N(3)	−0.0853 (3)	0.0954 (2)	0.8124 (2)	0.057 (1)
B(1)	0.6048 (4)	0.2063 (3)	0.4014 (3)	0.054 (1)
F(1)	0.5199 (3)	0.1057 (2)	0.3894 (2)	0.107 (1)
F(2)	0.4931 (3)	0.2943 (2)	0.3656 (2)	0.094 (1)
F(3)	0.7145 (3)	0.2028 (2)	0.3405 (2)	0.108 (1)
F(4)	0.6862 (4)	0.2260 (3)	0.5052 (2)	0.117 (1)

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

C(1)—N(2)	1.335 (3)	C(7)—C(8)	1.358 (4)
N(1)—N(2)	1.330 (3)	C(8)—N(3)	1.355 (4)
N(1)—C(4)	1.318 (4)	N(3)—C(5)	1.359 (3)
C(4)—C(3)	1.366 (5)	N(1)—C(9)	1.484 (4)
C(3)—C(2)	1.345 (4)	C(9)—C(10)	1.456 (7)
C(2)—C(1)	1.400 (4)	B(1)—F(1)	1.347 (4)
C(1)—C(5)	1.435 (3)	B(1)—F(2)	1.368 (4)
C(5)—C(6)	1.385 (4)	B(1)—F(3)	1.354 (5)
C(6)—C(7)	1.386 (5)	B(1)—F(4)	1.352 (4)
C(1)—N(2)—N(1)	116.8 (2)	C(8)—N(3)—C(5)	109.3 (2)
N(2)—N(1)—C(4)	124.1 (2)	N(3)—C(5)—C(6)	107.2 (2)
N(1)—C(4)—C(3)	120.1 (3)	N(2)—N(1)—C(9)	114.0 (2)
C(4)—C(3)—C(2)	118.5 (3)	N(1)—C(9)—C(10)	110.8 (3)
C(3)—C(2)—C(1)	118.9 (3)	F(1)—B(1)—F(2)	109.1 (3)
C(2)—C(1)—N(2)	121.6 (2)	F(1)—B(1)—F(3)	108.4 (3)
N(2)—C(1)—C(5)	116.5 (2)	F(1)—B(1)—F(4)	111.2 (3)
C(1)—C(5)—C(6)	130.2 (2)	F(2)—B(1)—F(3)	108.5 (3)
C(5)—C(6)—C(7)	107.3 (3)	F(2)—B(1)—F(4)	108.4 (3)
C(6)—C(7)—C(8)	107.8 (3)	F(3)—B(1)—F(4)	111.1 (3)
C(7)—C(8)—N(3)	108.4 (2)		

Data collection: *P*3 (Siemens, 1989). Cell refinement: *P*3. Data reduction: *XPREP* in *SHELXTL-Plus* (Sheldrick, 1991). Structure solution: *XS* in *SHELXTL-Plus*. Structure refinement: *XLS* in *SHELXTL-Plus*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *SHELXTL-Plus*. Preparation of material for publication: *XPUB* in *SHELXTL-Plus*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: PT1023). (00 pp.). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Domingo, L. R. (1993). Unpublished data. University of Valencia, Spain.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Karatza, M.-E. (1992). PhD thesis, University of East Anglia, England.

Sheldrick, G. M. (1991). *SHELXTL-Plus*. Release 4.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Siemens (1989). *P3/PC Diffractometer Program*. Version 3.13. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Whitmore, A. P. (1994). PhD thesis, University of East Anglia, England.

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Trichotomine Dimethyl Ester

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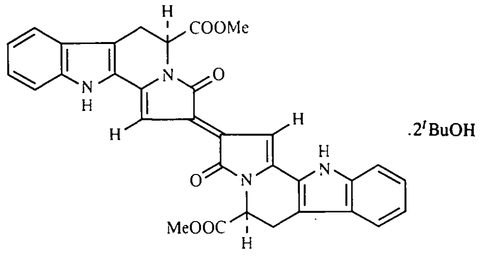
Abstract

The title compound, methyl 2-[6,11-dihydro-5-(methoxycarbonyl)-3-oxo-3*H*-indolizino[8,7-*b*]indol-2(5*H*)-ylidene]-2,5,6,11-tetrahydro-3-oxo-3*H*-indolizino[8,7-*b*]indole-5-carboxylate, $C_{32}H_{24}N_4O_6$, crystallized from *tert*-butanol as the disolvate. The compound has approximate C_2 symmetry. The two central five-membered rings are linked by a C═C double bond and are approximately coplanar. The two lateral indole rings are canted slightly out of this plane by 5 and 8°. The molecular skeleton is thus essentially planar, except for the methoxycarbonyl groups, which are nearly perpendicular to the rest of the molecule.

Comment

Trichotomine, isolated from *Clerodendron trichotomum Thunb*, has an H-type chromophore similar to that of indigo (Iwadera, Shizuri, Sasaki & Hirata, 1974). It is reported that indigo is planar, but *N*,*N'*-dimethylindigo shows a torsion about the central double bond (Miehe *et al.*, 1991). The 1,1′-bis(ethoxycarbonyl)trichotomine derivative shows a similar twisting about the central double bond (Irikawa *et al.*, 1994). The present study of trichotomine dimethyl ester *tert*-butanol solvate, (I), was undertaken in order to estimate the effect of substitution on the central double bond in trichotomine derivatives.



(I)

The structure determination of the title compound shows that the two five-membered rings linked through the central double bond are coplanar, with a maximum deviation of 0.05 Å from the best plane (Fig. 1). Each indole ring is also planar within 0.02 Å, though they are canted both with respect to the central plane (5 and 8°) and with respect to each other (13°). This inclination of the indole rings probably results from the non-planar six-membered rings linking them to the central core. The overall appearance of the molecular skeleton, however, is planar, except for the methoxycarbonyl groups (Fig. 2). These groups are perpendicular to the molecular plane and the whole molecule has approximate C_2 symmetry. The C(1)—C(2), C(20)—C(21), C(22)—C(23) and C(41)—C(42) bond distances are shorter by 0.1 Å than normal C—C single bonds. On the other hand, the C(1)—C(21), C(2)—C(23) and C(22)—C(42) bonds are longer by about 0.03 Å than typical C═C double bonds.

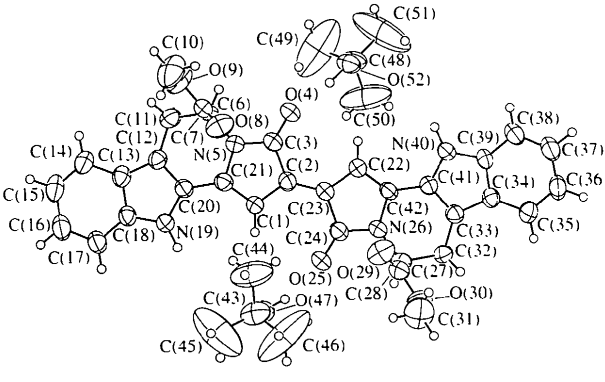


Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the title compound and the solvent molecules with the atomic numbering scheme. Displacement ellipsoids of non-H atoms are shown at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

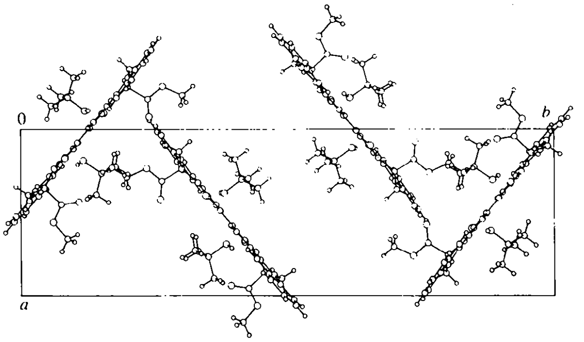


Fig. 2. The crystal structure viewed along the *c* axis, with the *a* axis vertical and the *b* axis horizontal.

The trichotomine derivative with ethoxycarbonyl groups at the C(1) and C(22) positions has a bent propeller shape (Irikawa *et al.*, 1994). The central double bond is 0.01 Å shorter than that of the present molecule. The steric hindrance between the O(4) atom and the bulky substituent at the C(22) position obviously causes a twisting and bending of the central double bond, resulting in a decrease in conjugation within the system.

Two molecules of *tert*-butanol co-crystallize with the trichotomine ester. One of these solvent molecules links two molecules of the ester through hydrogen bonding: O(47)···O(25) 2.874 (2) and O(47)···N(40′) 2.804 (2) Å [atom N(40′) is in the molecule translated one unit along the *c* axis]. The second solvent molecule also links two molecules of the ester related by translation in the opposite direction along the *c* axis: O(52)···O(4) 2.789 (2) and O(52)···N(19″) 2.873 (2) Å. Molecular columns are thus formed along the *c* axis. There are no unusually short distances between these columns.

Experimental

The title compound was prepared according to the method of Irikawa *et al.* (1994) and recrystallized from *tert*-butanol. The density D_m was measured by flotation in aqueous KI.

Crystal data

$C_{32}H_{24}N_4O_6.2C_4H_{10}O$
$M_r = 708.0$
Orthorhombic
$P2_12_12_1$
$a = 10.740 (2)$ Å
$b = 35.173 (5)$ Å
$c = 10.057 (2)$ Å
$V = 3799.0 (9)$ Å^3
$Z = 4$
$D_x = 1.24$ Mg m^{-3}
$D_m = 1.22$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 28 reflections
$\theta = 28$–$30°$
$\mu = 0.67$ mm^{-1}
$T = 296$ K
Block
0.45 × 0.30 × 0.20 mm
Blue–black

Data collection

Mac Science MXC3 diffractometer
$2\theta/\omega$ scans
Absorption correction: none
3643 measured reflections
3611 independent reflections
3125 observed reflections $[I > 2\sigma(I)]$

$\theta_{\max} = 65°$
$h = 0 \rightarrow 12$
$k = -41 \rightarrow 0$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 100 reflections
intensity decay: <1%

Refinement

Refinement on *F*
$R = 0.0493$
$wR = 0.0514$
$S = 1.000$
3125 reflections
469 parameters
H-atom parameters not refined
$w = \exp[15\sin^2(\theta)/\lambda^2]/\sigma^2(F_o)$

$(\Delta/\sigma)_{\max} = 0.04$
$\Delta\rho_{\max} = 0.45$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ 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 equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
C(1)	−0.04321 (12)	0.13593 (3)	0.72941 (12)	0.046 (1)
C(2)	−0.00882 (11)	0.12849 (3)	0.59432 (12)	0.046 (1)
C(3)	−0.09376 (12)	0.15157 (4)	0.50995 (12)	0.049 (1)
O(4)	−0.09703 (11)	0.15523 (3)	0.38892 (9)	0.064 (1)
N(5)	−0.17399 (10)	0.16950 (3)	0.59581 (10)	0.048 (1)
C(6)	−0.25802 (12)	0.20013 (4)	0.55799 (12)	0.052 (1)
C(7)	−0.18447 (13)	0.23726 (4)	0.54174 (12)	0.055 (1)
O(8)	−0.07512 (12)	0.24002 (4)	0.55041 (15)	0.080 (1)
O(9)	−0.26157 (13)	0.26492 (4)	0.50879 (17)	0.090 (1)
C(10)	−0.2071 (3)	0.3019 (1)	0.4852 (3)	0.120 (2)
C(11)	−0.36477 (12)	0.20417 (4)	0.65898 (14)	0.057 (1)
C(12)	−0.31538 (12)	0.20110 (4)	0.79945 (13)	0.049 (1)
C(13)	−0.36178 (12)	0.21484 (3)	0.92176 (13)	0.049 (1)
C(14)	−0.46514 (13)	0.23769 (4)	0.96026 (17)	0.059 (1)
C(15)	−0.48345 (17)	0.24437 (4)	1.09269 (19)	0.070 (1)
C(16)	−0.40279 (19)	0.22986 (4)	1.18985 (16)	0.070 (1)
C(17)	−0.30208 (17)	0.20795 (4)	1.15740 (14)	0.061 (1)
C(18)	−0.28268 (12)	0.20043 (3)	1.02167 (12)	0.047 (1)
N(19)	−0.19129 (10)	0.17887 (3)	0.96240 (10)	0.046 (1)
C(20)	−0.21235 (11)	0.17923 (3)	0.82741 (12)	0.044 (1)
C(21)	−0.14066 (11)	0.16110 (3)	0.72779 (11)	0.043 (1)
C(22)	0.11769 (11)	0.09727 (3)	0.41094 (12)	0.046 (1)
C(23)	0.08206 (11)	0.10483 (3)	0.54464 (11)	0.044 (1)
C(24)	0.16822 (12)	0.08233 (4)	0.63100 (12)	0.047 (1)
O(25)	0.17828 (10)	0.08150 (3)	0.75176 (9)	0.058 (1)
N(26)	0.24042 (10)	0.06117 (3)	0.54526 (10)	0.046 (1)
C(27)	0.35772 (12)	0.04377 (4)	0.58441 (11)	0.047 (1)
C(28)	0.45590 (13)	0.07487 (4)	0.60026 (13)	0.055 (1)
O(29)	0.43973 (13)	0.10767 (3)	0.57856 (16)	0.082 (1)
O(30)	0.56182 (11)	0.05957 (4)	0.64419 (14)	0.076 (1)
C(31)	0.6617 (2)	0.0860 (1)	0.6639 (3)	0.109 (2)
C(32)	0.39747 (11)	0.01297 (3)	0.48391 (12)	0.048 (1)
C(33)	0.37544 (12)	0.02659 (3)	0.34409 (11)	0.045 (1)
C(34)	0.43121 (12)	0.01552 (3)	0.22176 (12)	0.046 (1)
C(35)	0.52728 (14)	−0.01000 (4)	0.18603 (15)	0.056 (1)
C(36)	0.55957 (17)	−0.01285 (4)	0.05437 (16)	0.067 (1)
C(37)	0.49876 (18)	0.00876 (4)	−0.04384 (15)	0.066 (1)
C(38)	0.40495 (15)	0.03361 (4)	−0.01260 (12)	0.057 (1)
C(39)	0.37110 (13)	0.03660 (3)	0.12169 (12)	0.047 (1)
N(40)	0.28170 (11)	0.05937 (3)	0.17867 (10)	0.047 (1)
C(41)	0.28673 (11)	0.05321 (3)	0.31458 (11)	0.043 (1)
C(42)	0.21326 (12)	0.07174 (3)	0.41255 (11)	0.046 (1)
C(43)	0.2690 (2)	0.1506 (1)	1.0119 (2)	0.085 (1)
C(44)	0.2299 (4)	0.1761 (1)	0.9034 (4)	0.145 (3)
C(45)	0.2358 (7)	0.1695 (1)	1.1404 (4)	0.244 (5)
C(46)	0.3970 (3)	0.1403 (1)	1.0102 (8)	0.254 (4)
O(47)	0.19164 (13)	0.11649 (3)	1.00964 (13)	0.075 (1)
C(48)	−0.1937 (2)	0.0872 (1)	0.1400 (2)	0.074 (1)
C(49)	−0.3161 (3)	0.0988 (1)	0.1805 (8)	0.249 (4)
C(50)	−0.1432 (4)	0.0570 (1)	0.2216 (5)	0.184 (2)
C(51)	−0.2003 (7)	0.0747 (1)	0.0009 (3)	0.259 (5)
O(52)	−0.11241 (12)	0.11987 (3)	0.14113 (10)	0.066 (1)

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

C(1)—C(2)	1.432 (2)	C(22)—C(23)	1.423 (2)
C(1)—C(21)	1.371 (2)	C(22)—C(42)	1.364 (2)
C(2)—C(3)	1.487 (2)	C(23)—C(24)	1.496 (2)
C(2)—C(23)	1.377 (2)	C(24)—O(25)	1.220 (2)
C(3)—O(4)	1.224 (2)	C(24)—N(26)	1.378 (2)
C(3)—N(5)	1.373 (2)	N(26)—C(27)	1.455 (2)
N(5)—C(6)	1.456 (2)	N(26)—C(42)	1.416 (2)
N(5)—C(21)	1.406 (2)	C(27)—C(28)	1.528 (2)
C(6)—C(7)	1.535 (2)	C(27)—C(32)	1.542 (2)
C(6)—C(11)	1.538 (2)	C(28)—O(29)	1.187 (2)
C(7)—O(8)	1.182 (2)	C(28)—O(30)	1.334 (2)
C(7)—O(9)	1.320 (2)	O(30)—C(31)	1.432 (3)
O(9)—C(10)	1.446 (3)	C(32)—C(33)	1.504 (2)
C(11)—C(12)	1.513 (2)	C(33)—C(34)	1.423 (2)
C(12)—C(13)	1.412 (2)	C(33)—C(41)	1.368 (2)
C(12)—C(20)	1.377 (2)	C(34)—C(35)	1.414 (2)
C(13)—C(14)	1.424 (2)	C(34)—C(39)	1.407 (2)
C(13)—C(18)	1.410 (2)	C(35)—C(36)	1.372 (3)
C(14)—C(15)	1.367 (3)	C(36)—C(37)	1.407 (3)
C(15)—C(16)	1.402 (3)	C(37)—C(38)	1.370 (3)
C(16)—C(17)	1.368 (3)	C(38)—C(39)	1.403 (2)
C(17)—C(18)	1.406 (2)	C(39)—N(40)	1.375 (2)
C(18)—N(19)	1.376 (2)	N(40)—C(41)	1.385 (2)
N(19)—C(20)	1.376 (2)	C(41)—C(42)	1.421 (2)
C(20)—C(21)	1.415 (2)		

C(2)—C(1)—C(21)	107.7 (2)	C(23)—C(22)—C(42)	108.3 (2)
C(1)—C(2)—C(3)	106.5 (1)	C(2)—C(23)—C(22)	130.3 (2)
C(1)—C(2)—C(23)	129.6 (2)	C(2)—C(23)—C(24)	123.2 (2)
C(3)—C(2)—C(23)	123.9 (2)	C(22)—C(23)—C(24)	106.5 (1)
C(2)—C(3)—O(4)	130.0 (2)	C(23)—C(24)—O(25)	130.2 (2)
C(2)—C(3)—N(5)	106.1 (1)	C(23)—C(24)—N(26)	105.7 (1)
O(4)—C(3)—N(5)	124.0 (2)	O(25)—C(24)—N(26)	124.1 (2)
C(3)—N(5)—C(6)	124.4 (2)	C(24)—N(26)—C(27)	123.0 (1)
C(3)—N(5)—C(21)	109.7 (2)	C(24)—N(26)—C(42)	109.4 (2)
C(6)—N(5)—C(21)	124.0 (1)	C(27)—N(26)—C(42)	123.0 (1)
N(5)—C(6)—C(7)	109.8 (2)	N(26)—C(27)—C(28)	108.9 (2)
N(5)—C(6)—C(11)	111.0 (2)	N(26)—C(27)—C(32)	111.0 (1)
C(7)—C(6)—C(11)	112.0 (2)	C(28)—C(27)—C(32)	112.4 (2)
C(6)—C(7)—O(8)	125.0 (2)	C(27)—C(28)—O(29)	125.2 (2)
C(6)—C(7)—O(9)	109.3 (2)	C(27)—C(28)—O(30)	109.5 (2)
O(8)—C(7) O(9)	125.6 (2)	O(29)—C(28)—O(30)	125.3 (2)
C(7)—O(9)—C(10)	116.8 (2)	C(28)—O(30)—C(31)	115.0 (2)
C(6)—C(11)—C(12)	110.4 (2)	C(27)—C(32)—C(33)	110.2 (1)
C(11)—C(12)—C(13)	131.7 (2)	C(32)—C(33)—C(34)	130.9 (2)
C(11)—C(12)—C(20)	120.8 (2)	C(32)—C(33)—C(41)	122.0 (2)
C(13)—C(12)—C(20)	107.3 (2)	C(34)—C(33)—C(41)	107.0 (1)
C(12)—C(13)—C(14)	134.8 (2)	C(33)—C(34)—C(35)	134.5 (2)
C(12)—C(13)—C(18)	106.6 (2)	C(33)—C(34)—C(39)	106.3 (2)
C(14)—C(13)—C(18)	118.6 (2)	C(35)—C(34)—C(39)	119.2 (2)
C(13)—C(14)—C(15)	118.3 (2)	C(34)—C(35)—C(36)	118.4 (2)
C(14)—C(15)—C(16)	121.9 (2)	C(35)—C(36)—C(37)	121.4 (2)
C(15)—C(16)—C(17)	121.8 (2)	C(36)—C(37)—C(38)	121.7 (2)
C(16)—C(17)—C(18)	117.1 (2)	C(37)—C(38)—C(39)	117.3 (2)
C(13)—C(18)—C(17)	122.3 (2)	C(34)—C(39)—C(38)	122.0 (2)
C(13)—C(18)—N(19)	108.6 (2)	C(34)—C(39)—N(40)	109.2 (2)
C(17)—C(18)—N(19)	129.0 (2)	C(38)—C(39)—N(40)	128.7 (2)
C(18)—N(19)—C(20)	107.8 (1)	C(39)—N(40)—C(41)	107.0 (2)
C(12)—C(20)—N(19)	109.8 (2)	C(33)—C(41)—N(40)	110.4 (2)
C(12)—C(20)—C(21)	123.0 (2)	C(33)—C(41)—C(42)	123.4 (2)
N(19)—C(20)—C(21)	127.2 (2)	N(40)—C(41)—C(42)	126.2 (2)
C(1)—C(21)—N(5)	110.0 (2)	C(22)—C(42)—N(26)	109.8 (2)
C(1)—C(21)—C(20)	134.3 (2)	C(22)—C(42)—C(41)	135.4 (2)
N(5)—C(21)—C(20)	115.8 (2)	N(26)—C(42)—C(41)	114.8 (2)

The structure of the title compound was solved by direct methods and refined by full-matrix least-squares calculations using *CRYSTAN-GM* (Gilmore & Brown, 1988). H atoms attached to *tert*-butanol O atoms were found from the difference Fourier map and fixed in the refinement. Other H atoms were placed at geometrically idealized positions with N—H and C—H distances of 0.96 Å, and were restricted to their parent atoms with the corresponding equivalent isotropic displacement parameters. The absolute configuration was assigned to agree with the known chirality at the C(6) and C(27) atoms arising from L-tryptophan methyl ester.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including contact distances, have been deposited with the IUCr (Reference: TA1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Gilmore, C. J. & Brown, S. R. (1988). *Acta Cryst.* A**44**, 1018–1021.
Irikawa, H., Enomoto, M., Shimoda, Y., Atsumi, T., Okumura, Y. & Iijima, K. (1994). *Bull. Chem. Soc. Jpn*, **67**, 1931–1935.
Iwadera, S., Shizuri, Y., Sasaki, K. & Hirata, Y. (1974). *Tetrahedron*, **30**, 4105–4111.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Miehe, G., Süsse, P., Kupcik, V., Egert, E., Nieger, M., Kunz, G., Gerke, R., Knieriem, B., Niemeyer, M. & Lüttke, W. (1991). *Angew. Chem. Int. Ed. Engl.* **30**, 964–967.

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5-Cyclohexylamino-2-phenyl-4-thiazolecarbonitrile

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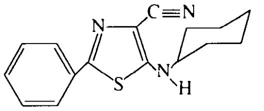
Abstract

$C_{16}H_{17}N_3S$ contains a planar thiazole ring. The C—S bond length is shorter than that found in the tetrahydrothiophene [Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19]. The phenyl and the thiazole rings are conjugated.

Comment

The title compound (I) was synthesized by the treatment of methyl *N*-cyanomethylbenzimidate with lithium diisopropylamine at 203 K followed by the action of cyclohexylisothiocyanate (Kammoun, Hajjem & Baccar, 1996). Theoretically, the reaction could lead to the formation of imidazole or to thiazole derivatives. Single-crystal X-ray diffraction allowed the determination of the molecular structure. The thiazole ring is planar, as shown by the torsion angles C7—S—C9—C8 = 1.4 (2) and C9—S—C7—N1 = −1.2 (2)°. The C—S bond lengths in the thiazole ring [C9—S 1.729 (2) Å, C7—S 1.761 (2) Å] are similar to the C—S bond distances in the thiophene ring (Allen *et al.*, 1987) indicating a partial π-bond character. This results from the delocalization of the negative density charge through the thiazole ring. The phenyl-ring plane is twisted about 7.9 (2)° out of the thiazole ring. The low value of this twist angle and the short C1—C7 distance [1.468 (3) Å] could explain the conjugation between the two rings.



(I)

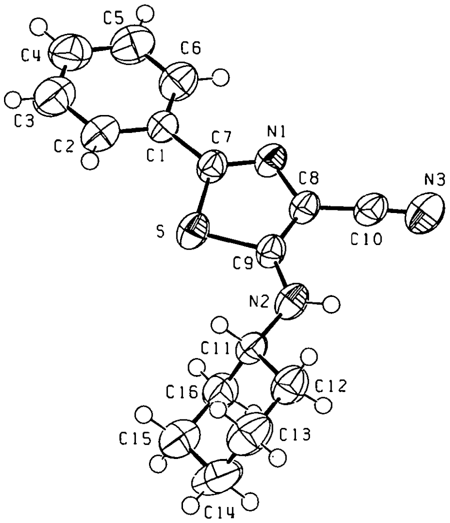


Fig. 1. *ORTEP*II (Johnson, 1976) view of $C_{16}H_{17}N_3S$ showing the labelling of the non-H atoms. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

Experimental

The title compound was synthesized according to Kammoun, Hajjem & Baccar (1996) (see *Comment*).