The Structure of the (2*R*)-3,3,3-Trifluoro-2-methoxy-2-phenylpropionic Acid Ester of *trans*-4-*tert*-Butylcyclohexanol

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Abstract

The crystal structure of the (2*R*)-3,3,3-trifluoro-2-methoxy-2-phenylpropionic acid (*R*-TMPPA) ester of *trans*-4-*tert*-butylcyclohexanol has been determined from Mo $K\alpha$ data. $C_{20}H_{27}F_3O_3$, $M_r = 372 \cdot 4$, is monoclinic, $P2_1$, with $Z = 2$, $a = 6 \cdot 993$ (5), $b = 11 \cdot 288$ (6), $c = 12 \cdot 303$ (6) Å, $\beta = 95 \cdot 51$ (5)°, $V = 967$ Å^3, $D_x = 1 \cdot 28$ Mg m^{-3}, $\mu = 0 \cdot 112$ mm^{-1}, m.p. 326 K. The structure was solved by a combination of *a priori* direct methods and an adapted *DIRDIF* version (application of direct methods when a qualitatively bad structural fragment is given) and refined by least squares to a final $R = 0 \cdot 047$ for 2003 reflections. The orientation of the *trans*-4-*tert*-butylcyclohexyl residue with respect to the *R*-TMPPA ester is different from that in other equatorial esters. The other geometrical parameters are unexceptional.

Introduction

The optical purity of type (1) chiral secondary alcohols can be determined by NMR analysis of their esters with (2*R*)-3,3,3-trifluoro-2-methoxy-2-phenylpropionic acid (*R*-TMPPA) (Merckx, Van de Wal, Lepoivre & Alderweireldt, 1978; Van de Wal, Merckx, Lemière, Lepoivre & Alderweireldt, 1978; Van Osselaer, Lemière, Merckx, Lepoivre & Alderweireldt, 1978; Sadozai, Lepoivre, Dommisse & Alderweireldt, 1980).
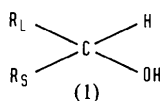
R_L = large alkyl
R_S = small alkyl

(1)

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Under certain circumstances, this method allows simultaneous assignment of the absolute configuration of the alcohols. After addition of Eu(fod)$_3$ (fod is 6,6,7,7,8,8,8-heptafluoro-2,2-dimethyl-3,5-octanedione), the trifluoromethyl resonances in the ^{19}F NMR spectrum of the diastereomeric *R*-TMPPA esters have different lanthanide-induced-shift (LIS) values. These differences in LIS gradients can be interpreted in terms of *R/S* absolute configurations. To investigate the molecular geometry of *R*-TMPPA ester/Eu(fod)$_3$ complexes, a substrate model was found in the *R*-TMPPA ester of *trans*-4-*tert*-butylcyclohexanol, which is crystalline. This ester is of special interest since the conformations in solution are limited, due to the presence of the *tert*-butyl substituent on the cyclohexane ring.

Experimental

Crystals were obtained by esterification of *trans*-4-*tert*-butylcyclohexanol with *S*-TMPPA chloride† and recrystallization from absolute alcohol.

Intensities were collected on an Enraf–Nonius CAD-4 diffractometer at 123 K from a crystal of dimensions 0·1 × 0·1 × 0·2 mm, using Zr-filtered Mo $K\alpha$ radiation. Of 2210 reflections collected ($\theta \le 27°$), 2003 with $I > 2\sigma(I)$ (counting statistics) were used in the analysis. Data were corrected for Lorentz–polarization effects. Absorption corrections were not applied.

† Experimental details for the synthesis of the *R*-TMPPA ester can be obtained from E. M. Merckx.

Structure determination

A first run of the direct-methods program *MULTAN* 78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) resulted in a 'best' phase set (CFOM

Table 1. *Fractional coordinates* ($\times 10^4$ *for non-*H *atoms,* $\times 10^3$ *for* H *atoms*) *and isotropic temperature factors* (Å^2 $\times 10^3$)

U_{eq} is as defined by Hamilton (1959).

	x	y	z	U_{eq}/U
C(1)	4536 (5)	2761 (4)	3757 (3)	33·3 (10)
C(2)	5865 (6)	3903 (4)	3894 (3)	35·2 (11)
C(3)	6822 (7)	3913 (4)	5020 (3)	38·5 (12)
C(4)	8160 (5)	2853 (4)	5193 (3)	31·5 (9)
C(5)	6933 (6)	1718 (4)	5037 (3)	36·2 (11)
C(6)	5799 (6)	1686 (4)	3901 (3)	35·8 (11)
C(7)	9498 (6)	2893 (5)	6277 (3)	39·9 (11)
C(8)	10791 (7)	3986 (5)	6277 (3)	45·7 (13)
C(9)	8366 (8)	2960 (6)	7283 (3)	51·5 (14)
C(10)	10798 (7)	1791 (5)	6357 (4)	48·0 (14)
C(11)	1172 (5)	3198 (4)	1238 (3)	30·3 (9)
C(12)	562 (7)	1091 (4)	1398 (4)	41·8 (12)
C(13)	−936 (5)	3624 (4)	1058 (3)	35·7 (10)
C(14)	2404 (5)	4023 (4)	615 (3)	29·7 (9)
C(15)	2897 (6)	5158 (5)	1024 (3)	42·9 (12)
C(16)	3997 (7)	5910 (5)	458 (4)	49·7 (14)
C(17)	4610 (6)	5552 (5)	−524 (4)	49·8 (14)
C(18)	4135 (6)	4447 (5)	−930 (3)	44·8 (13)
C(19)	3007 (5)	3670 (4)	−376 (3)	33·5 (10)
C(20)	1903 (5)	3281 (4)	2462 (3)	31·0 (9)
F(1)	−1169 (4)	4758 (3)	1300 (2)	42·7 (7)
F(2)	−2098 (3)	2989 (3)	1648 (2)	45·2 (7)
F(3)	−1590 (3)	3469 (3)	10 (2)	40·5 (7)
O(1)	3563 (4)	2697 (3)	2646 (2)	32·8 (7)
O(2)	1099 (4)	3816 (4)	3136 (2)	41·0 (8)
O(3)	1331 (4)	2056 (3)	823 (2)	33·3 (7)
H(1)	350 (6)	274 (5)	429 (4)	41
H(2A)	667 (7)	389 (5)	319 (4)	44
H(2B)	484 (7)	453 (5)	388 (4)	44
H(3A)	592 (8)	389 (5)	566 (4)	47
H(3B)	739 (8)	471 (5)	522 (4)	47
H(4)	895 (6)	295 (5)	456 (4)	38
H(5A)	763 (8)	102 (5)	523 (4)	43
H(5B)	608 (7)	167 (5)	561 (4)	43
H(6A)	688 (7)	166 (5)	330 (4)	44
H(6B)	513 (7)	92 (5)	386 (4)	44
H(8A)	1171 (8)	411 (6)	697 (5)	57
H(8B)	1001 (8)	474 (6)	622 (5)	57
H(8C)	1167 (8)	394 (6)	552 (4)	57
H(9A)	911 (8)	293 (6)	799 (5)	63
H(9B)	738 (8)	222 (6)	729 (5)	63
H(9C)	765 (9)	375 (6)	733 (5)	63
H(10A)	1145 (9)	161 (6)	572 (5)	63
H(10B)	1014 (9)	99 (6)	639 (5)	63
H(10C)	1174 (9)	192 (6)	696 (5)	63
H(12A)	106 (8)	36 (6)	118 (4)	55
H(12B)	95 (8)	113 (6)	218 (5)	55
H(12C)	−90 (9)	95 (6)	112 (4)	55
H(15)	248 (7)	533 (5)	175 (4)	52
H(16)	422 (8)	674 (6)	81 (5)	62
H(17)	522 (8)	607 (5)	−94 (4)	53
H(18)	448 (8)	414 (6)	−176 (4)	55
H(19)	260 (7)	290 (5)	−66 (4)	63

= 2·142), from which an E map was calculated. A fragment could be located, consisting of fifteen peaks. However, this model did not show any obvious resemblance to a molecular fragment of the proposed structure. To check the validity of the fragment and its position in the unit cell, strengthened translation functions (Doesburg & Beurskens, 1980) were used. Carbon-atom scattering power was assigned to the 15 peaks, and assuming a correct orientation, the position of the fragment in the unit cell was determined. The highest peak from this search (relative height 100) clearly indicated the correct positioning of the majority of the 'atoms' of the fragment, but as the absolute peak height was rather low compared to its optimum value (exact coincidence of structure and search fragment), the low quality of the model was confirmed. Having confirmed the validity of the fragment, we used it as input to *DIRDIF* (Beurskens, Bosman, Doesburg, Van den Hark, Prick & Gould, 1980). A large part of the molecule could then be recognized (24 out of 26 non-H atoms). Five peaks of the initial model now proved to be false. These were part of the well-known chicken-wire-type pattern often found in initial E maps. Peaks, corresponding to true atomic positions, had very low densities in this E map.

Subsequent weighted difference Fourier syntheses gave the positions of the two remaining non-H atoms and all H atoms. Anisotropic thermal parameters of non-H atoms and all positional parameters were refined by full-matrix least squares, minimizing $\sum w(|F_o| - k|F_c|)^2$; $w = [\sigma^2(F_o) + 0.0017F_o^2]^{-1}$. Refinement converged to $R = 0.047$ and $R_w = 0.070$. The highest peak in a final difference Fourier map was 0·23 e Å^{-3}. Mean and maximum shifts for non-H atoms were 0·07 and 0·31 σ, for H atoms 0·20 and 0·73 σ. Atomic parameters and isotropic temperature factors are given in Table 1.*

Scattering factors were those of Cromer & Mann (1968) for C, F, O and of Stewart, Davidson & Simpson (1965) for H. Routine calculations were performed with XRAY76 (Stewart, 1976).

Results and discussion

Bond distances and angles are listed in Table 2 and selected torsion angles are in Table 3. The atomic numbering is shown in Fig. 1. A view of the molecule in the minimum atomic overlap mode is shown in Fig. 2 and a stereoscopic view of the packing of the molecules, as seen along **b**, is shown in Fig. 3.

Table 2. *Bond distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

C(1)—C(2)	1·519 (6)	C(2)—C(1)—C(6)	111·9 (3)
C(2)—C(3)	1·530 (6)	C(1)—C(2)—C(3)	109·3 (3)
C(3)—C(4)	1·521 (6)	C(2)—C(3)—C(4)	112·4 (3)
C(4)—C(5)	1·544 (6)	C(3)—C(4)—C(5)	108·0 (3)
C(5)—C(6)	1·540 (6)	C(4)—C(5)—C(6)	111·4 (3)
C(6)—C(1)	1·502 (6)	C(5)—C(6)—C(1)	109·5 (3)
C(4)—C(7)	1·555 (5)	C(3)—C(4)—C(7)	114·1 (4)
		C(5)—C(4)—C(7)	114·4 (4)
C(7)—C(8)	1·529 (7)	C(4)—C(7)—C(8)	109·3 (3)
C(7)—C(9)	1·533 (6)	C(4)—C(7)—C(9)	112·2 (3)
C(7)—C(10)	1·539 (7)	C(4)—C(7)—C(10)	109·5 (4)
		C(8)—C(7)—C(9)	108·2 (4)
		C(8)—C(7)—C(10)	107·9 (4)
		C(9)—C(7)—C(10)	109·6 (4)
C(1)—O(1)	1·469 (4)	C(2)—C(1)—O(1)	109·7 (3)
C(20)—O(1)	1·336 (5)	C(6)—C(1)—O(1)	106·5 (3)
C(20)—O(2)	1·206 (5)	C(1)—O(1)—C(20)	116·8 (3)
C(20)—C(11)	1·546 (5)	O(1)—C(20)—O(2)	125·8 (3)
		O(1)—C(20)—C(11)	109·6 (3)
		O(2)—C(20)—C(11)	124·6 (3)
C(11)—O(3)	1·395 (6)	C(14)—C(11)—O(3)	108·2 (3)
C(12)—O(3)	1·431 (6)	C(20)—C(11)—O(3)	112·4 (3)
		C(13)—C(11)—O(3)	110·1 (3)
		C(11)—O(3)—C(12)	118·4 (3)
C(11)—C(13)	1·546 (6)	C(13)—C(11)—C(14)	108·4 (3)
C(13)—F(1)	1·328 (6)	C(20)—C(11)—C(13)	110·0 (3)
C(13)—F(2)	1·347 (5)	C(11)—C(13)—F(1)	113·8 (3)
C(13)—F(3)	1·38 (4)	C(11)—C(13)—F(2)	112·0 (3)
		C(11)—C(13)—F(3)	109·3 (3)
		F(1)—C(13)—F(2)	107·4 (3)
		F(1)—C(13)—F(3)	107·7 (3)
		F(2)—C(13)—F(3)	106·3 (3)
C(11)—C(14)	1·525 (6)	C(20)—C(11)—C(14)	107·7 (3)
C(14)—C(15)	1·407 (7)	C(11)—C(14)—C(15)	120·4 (3)
C(15)—C(16)	1·378 (7)	C(14)—C(15)—C(16)	120·6 (4)
C(16)—C(17)	1·381 (7)	C(15)—C(16)—C(17)	119·8 (5)
C(17)—C(18)	1·373 (8)	C(17)—C(18)—C(19)	121·3 (4)
C(18)—C(19)	1·400 (7)	C(18)—C(19)—C(14)	118·8 (4)
C(19)—C(14)	1·386 (5)	C(19)—C(14)—C(11)	120·1 (4)
		C(19)—C(14)—C(15)	119·5 (4)

Table 3. *Selected torsion angles*

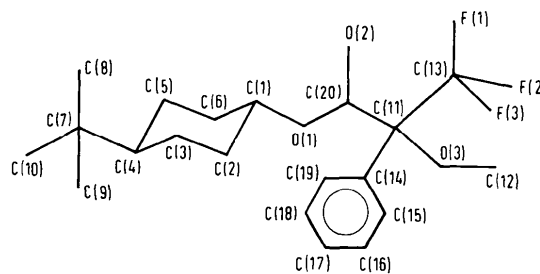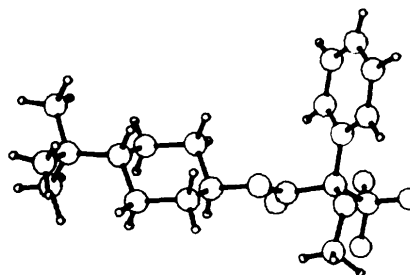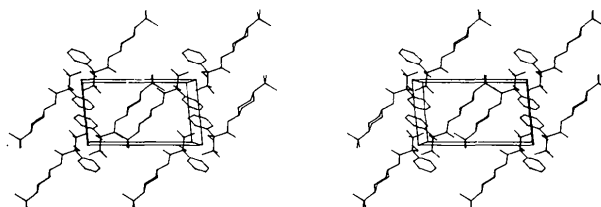|	
C(3)—C(4)—C(7)—C(8)	−61·3 (4)°
C(3)—C(4)—C(7)—C(9)	58·7 (4)
C(5)—C(4)—C(7)—C(9)	−66·3 (4)
C(5)—C(4)—C(7)—C(10)	55·7 (4)
C(3)—C(2)—C(1)—C(6)	−57·3 (4)
C(2)—C(1)—C(6)—C(5)	57·8 (4)
C(1)—C(6)—C(5)—C(4)	−58·1 (4)
C(6)—C(5)—C(4)—C(3)	57·4 (4)
C(5)—C(4)—C(3)—C(2)	−57·6 (4)
C(4)—C(3)—C(2)—C(1)	57·8 (4)
F(1)—C(13)—C(11)—C(20)	−63·9 (4)
F(1)—C(13)—C(11)—C(14)	53·6 (4)
F(3)—C(13)—C(11)—C(14)	−66·9 (4)
F(3)—C(13)—C(11)—O(3)	51·2 (4)
F(2)—C(13)—C(11)—O(3)	−66·3 (4)
F(2)—C(13)—C(11)—C(20)	58·1 (4)
C(13)—C(11)—C(20)—O(2)	13·8 (5)
C(14)—C(11)—C(20)—O(2)	−104·1 (4)
O(3)—C(11)—C(20)—O(2)	136·8 (3)

Fig. 1. Atomic numbering.

Fig. 2. Molecular conformation of the *R*-TMPPA ester of *trans*-4-*tert*-butylcyclohexanol.

4-*tert*-Butylcyclohexane moiety

The geometry of the 4-*tert*-butylcyclohexane moiety is compared with those of *trans*-4-*tert*-butylcyclohexyl *p*-toluenesulphonate (TOSTB), X-ray study (Johnson, Cheer, Schaeffer, James & Moore, 1972) and neutron study* (James & Moore, 1975), and of *trans*-4-*tert*-butylcyclohexyl *p*-bromobenzoate (PBTB) (Ohrt & Parthasarathy, 1972). Mean C—C bonds for *R*-TMPPA ester, TOSTB and PBTB, are 1·531, 1·526 and 1·532 Å, respectively. The shortening of C(1)—C(6) found in the *R*-TMPPA ester is similar to that in TOSTB, where it is considered to be a result of *sp* hybrid bonding with oxygen. The increased C(4)—C(7)

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* Because of small differences, the results of the two studies have been averaged.

Fig. 3. Packing of the molecules in the cell, a vertical, c horizontal.

bond length for the TMPPA ester and TOSTB is due to steric interactions of *tert*-butyl with ring H atoms. The distance is close to the theoretically calculated value of 1·57 Å (Altona & Sundaralingam, 1970). Mean C—C—C valence angles for the *R*-TMPPA ester, TOSTB and PBTB are 110·0, 111·2 and 110·2°. Mean endocyclic torsion angles for the three structures (same order) are 57·7, 55·9 and 58·3° (see Table 3). Comparison with the experimental mean torsion angle of 55·9° in gaseous cyclohexane (Buys & Geise, 1970) shows that there is no flattening effect on the ring by the equatorial *tert*-butyl group. However, the steric influence of this substituent reflects itself in the flattening of the C(3), C(4), C(5), C(7) pyramid. This is attained by pushing away the C(4)—C(7) bond from the C(3), C(4), C(5) plane thus yielding a small angle of 108·0 (3)° for C(3)—C(4)—C(5). This tendency was also noted for PBTB and TOSTB.

In acetates of cyclohexanol, with an equatorial ester group, eclipsing of the carbonyl group by the neighbouring axial H is a common observation in X-ray studies (Mathieson, 1965). In *R*-TMPPA ester such an eclipsed arrangement is not observed; the acute angle between vectors C(1)—H(1) and C(20)—O(2) is 32 (4)°, whereas in PBTB it is 5·8°. The O(2)···H(1) distance is 2·42 Å (corresponding distance in eclipsed arrangement ~2·22 Å).

TMPPA ester moiety

The average C—C(phenyl) bond length is 1·388 (7) Å [normal value 1·394 (5) Å]. The mean F—C—F bond angle is 107·1 (3)° which is normal. The mean C—F bond distance of 1·338 (5) Å is comparable to that in gaseous 1,1,1-trifluoroethane [1·340 (5) Å] (Beagley, Jones & Zanjansky, 1979), but the C(11)—C(13) distance [1·546 (6) Å] is considerably longer than the corresponding distance in this compound (1·494 Å). Other bond lengths in the TMPPA moiety also deviate significantly from the corresponding lengths in selected reference compounds (Table 4). An explanation for these anomalies might be found in the conformation of the TMPPA moiety. The con-

Table 4. *Bond lengths* (Å) *in the TMPPA-ester moiety and in reference compounds*

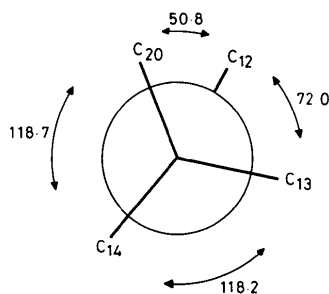|	Distance in TMPPA	Reference distance*	Reference compound
C(11)—C(13)	1·546 (6)	1·494 (3)	Trifluoroethane (Beagley *et al.*, 1979)
C(11)—O(3)	1·395 (6)	1·416 (5)	Dimethyl ether (Takemura *et al.*, 1979)
O(3)—C(12)	1·431 (6)	1·416 (6)	Dimethyl ether (Takemura *et al.*, 1979)
C(11)—C(20)	1·546 (5)	1·517 (3)	Acetone (Fijima, 1972)

* Determined by electron diffraction.

Fig. 4. Newman projection down the bond C(11)—O(3).

formation of the methoxy substituent is such that the interactions of the O(3) lone pairs are maximized with respect to the trifluoromethyl and carbonyl substituents (Fig. 4). This may give rise to anomeric effects, which result in a shortening of C(11)—O(3) and lengthening of C(11)—C(13), C(11)—C(20) and O(3)—C(12). The same phenomenon could explain (Williams, Scarsdale, Schäfer & Geise, 1981) why the C(11)—O(3)—C(12) angle has the high value of 118·4°, compared to 111° for the corresponding angle in dimethyl ether (Takemura, Tamagawa, Konaka & Kimura, 1979).

Packing

The packing seems to be determined by van der Waals forces. The shortest intermolecular contacts of 2·39 Å [H(2A)···F(2)] and 2·61 Å [H(4)···O(2)] occur between molecules related by an *a* translation. All other intermolecular contacts are well above the sum of their respective van der Waals radii.

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The Structures of Acetylcholine Hydrogen Tartrates*

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Abstract

The crystal structures of acetylcholine hydrogen ($\pm$)-tartrate, $C_7H_{16}NO_2^+.C_4H_5O_6^-$, and of two polymorphs of acetylcholine hydrogen (+)-tartrate have been determined. Crystals of the racemate are orthorhombic, space group $Pna2_1$, $a = 11\cdot853$ (2), $b = 8\cdot615$ (2), $c = 14\cdot111$ (3) Å, $Z = 4$. The two forms of the optically active salt are monoclinic, space group $P2_1$, $a = 6\cdot198$ (3), $b = 14\cdot530$ (9), $c = 8\cdot043$ (4) Å, $\beta = 97\cdot76$ (1)°, $Z = 2$, and $a = 12\cdot323$ (2), $b = 8\cdot518$ (2), $c = 13\cdot453$ (2) Å, $\beta = 90\cdot45$ (1)°, $Z = 4$, respectively. The final R values are 0·035 (1950 reflections), 0·030 (1426 reflections), and 0·069 (1672 reflections). The hydrogen tartrate ions in all three structures are interlinked *via* hydrogen bonds into infinite chains. The acetylcholine ions are not involved in hydrogen-bond formation in any of the structures, and no distinct contacts between the formally charged groups are found.

* IUPAC name: (acetoxyethyl)trimethylammonium hydrogen tartrate.

Introduction

The crystal structures of a series of acetylcholine tartrates and acetylcholine hydrogen tartrates have been investigated. The purpose was to examine interactions between the acetylcholine ion and the environment and to see whether the conformation of the flexible acetylcholine ion is influenced by these interactions. Tartrates and hydrogen tartrates were chosen because one could expect to get a series of six closely related structures (optically active, racemic, and *meso* as tartrate and hydrogen tartrate), in which a great number of potential hydrogen-bond donors are present. The hope was that the structures would constitute a material from which the packing forces could be analysed semiquantitatively, for instance by SCFF calculations. The problem has, however, been complicated by the finding that some of the structures are disordered. This paper describes the crystal structures of three hydrogen tartrates, *i.e.* the racemate, ARABIT, and two forms of the optically active salt, APBIT1 and APBIT2. A preliminary report on these salts was given in the abstract for the Fourth European Crystallo-