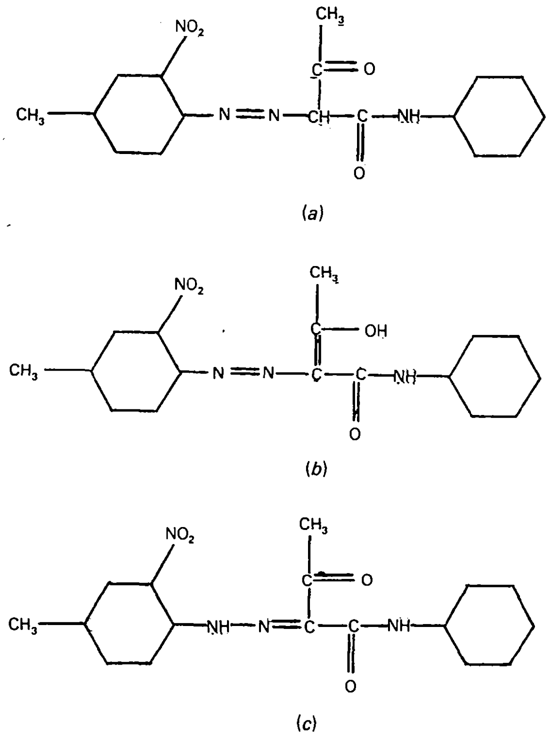

Fig. 2. Possible double-bond formulations around the azo group: (*a*) azo formula; (*b*) hydroxyazo formula; (*c*) hydrazone formula.

The relative merits of the hydroxyazo (*b*) and hydrazone (*c*) tautomeric forms have been discussed in detail by Prendergrass, Paul & Curtin (1972), by Kuder (1972) and by Guggenberger & Teufer (1975). Prendergrass *et al.* list the ranges of bond lengths to be expected for the two tautomers by comparison with those found in a series of closely related compounds, and our values generally lie intermediate between their two extremes. Berrie, Hampson, Longworth & Mathias (1968) have studied the problem by NMR spectroscopy and give quantitative estimates of the amounts of the tautomers. They concluded that a resonance hybrid would be the best representation of the formula. Our X-ray results tend to favour the hydrazone (*c*) formulation, and this agrees with ideas of extensive π-electron resonance throughout the molecule which maintains geometric planarity and consequential exceptional stability and light fastness of the pigment.

References

Ahmed, F. R., Hall, S. R., Pippy, M. E. & Huber, C. P. (1970). NRC crystallographic programs for the IBM/360 system. National Research Council, Ottawa, Canada.
Allen, R. L. M. (1971). *Colour Chemistry*, p. 247. London: T. Nelson & Sons.
Berrie, A. H., Hampson, P., Longworth, S. W. & Mathias, A. (1968). *J. Chem. Soc. B*, pp. 1308–1310.
Brown, C. J. (1967). *J. Chem. Soc. A*, pp. 405–409.
Brown, C. J. & Yadav, H. R. (1984). *Acta Cryst.* C**40**, 95–96.
Colour Index (1971). 3rd ed. Bradford: Society of Dyers and Colourists.
Guggenberger, L. J. & Teufer, G. (1975). *Acta Cryst.* B**31**, 785–790.
International Tables for X-ray Crystallography (1962). Vol. III, pp. 202–207. Birmingham: Kynoch Press.
Kuder, J. E. (1972). *Tetrahedron*, **28**, 1973–1981.
Mez, H.-C. (1968). *Ber. Bunsenges. Phys. Chem.* **72**, 389–393.
Prendergrass, D. B., Paul, I. C. & Curtin, D. Y. (1972). *J. Am. Chem. Soc.* **94**, 8730–8737.
Yagi, Y. (1963). *Bull. Chem. Soc. Jpn*, **36**, 487–500.

Acta Cryst. (1984). C**40**, 566–569

Ester of *cis*-(1*R*,3*S*)-3-*tert*-Butylcyclohexanol and (2*R*)-3,3,3-Trifluoro-2-methoxy-2-phenylpropionic Acid, $C_{20}H_{27}F_3O_3$

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(*Received* 2 *August* 1983; *accepted* 12 *December* 1983)

Abstract. $M_r = 372\cdot4$, orthorhombic, $P2_12_12_1$, $Z = 4$, $a = 7\cdot805\ (2)$, $b = 15\cdot150\ (7)$, $c = 16\cdot85\ (1)$ Å, $V = 1992\cdot4$ Å^3, $D_x = 1\cdot241$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0\cdot7107$ Å, $\mu = 0\cdot109$ mm^{-1}, $F(000) = 792$, room temperature. $R_w = 0\cdot046$ for 1448 reflections with $I > 2\sigma(I)$, out of 2038 independent measurements. The cyclohexane ring has the chair conformation with the *tert*-butyl and the propionate groups at equatorial positions. The molecular geometry of the title compound is very

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similar to that of the (2*R*)-3,3,3-trifluoro-2-methoxy-2-phenylpropionic acid ester of *trans*-4-*tert*-butylcyclohexanol. In the propionate moieties of both compounds the anomeric effect plays a key role in determining the geometry. The major difference is found in the cyclohexane ring, where the change of the *tert*-butyl group from a *para* to a *meta* transposition with respect to the O*R* group results in a flattening of the ring.

Introduction. Optical purity and sometimes the absolute configuration of *inter alia* alkyl-substituted cyclohexanols can be determined in solution from their esters with (2*R*)-3,3,3-trifluoro-2-methoxy-2-phenylpropionic acid (henceforth abbreviated as 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). 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, which may be interpreted in terms of absolute configurations (Merckx, 1982).

In an attempt to rationalize the ^{19}F NMR observations, the conformation and geometry of the ester/Eu(fod)$_3$ complexes in solution have been investigated from both ^{1}H and ^{13}C LIS experiments (Vanhoeck, Merckx, Bossaerts, Lepoivre & Alderweireldt, 1983; Vanhoeck, Bossaerts, Dommisse, Lepoivre & Alderweireldt, 1983). Thus it would be of interest to have the geometry of the ester as a point of reference. Therefore, the X-ray analysis of the title compound (Fig. 1) was undertaken. The presence of the *tert*-butyl group is believed to introduce sufficient structural rigidity in the ester so that the geometry observed in the solid state will also be representative of the geometry in solution.

Experimental. Optically pure *cis*-(1*R*,3*S*)-3-*tert*-butylcyclohexanol was prepared in three steps. First, racemic 3-*tert*-butylcyclohexanone was reduced enzymatically using horse-liver alcohol dehydrogenase to the (1*S*,3*S*)-alcohol following procedures described by Merckx (1982) and Van Osselaer, Lemière, Merckx, Lepoivre & Alderweireldt (1978). Second, the alcohol was oxidized to the pure (3*S*)-*tert*-butylcyclohexanone according to Corey & Suggs (1973). Third, the (3*S*) ketone was reduced with LiAlH$_4$ resulting in a mixture of the (1*R*,3*S*) and (1*S*,3*S*) alcohols, which was separated by liquid chromatography. Then, the title compound was prepared by esterification of *cis*-(1*R*,3*S*)-3-*tert*-butylcyclohexanol with *R*-TMPPA (Merckx, 1982). Single crystals in the form of thin, transparent plates were obtained by slow evaporation of a methanol solution at room temperature.

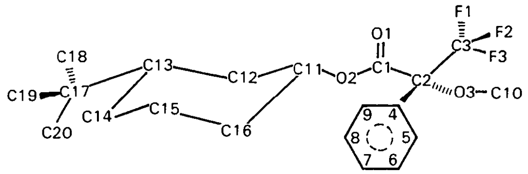

Fig. 1. Structural formula, conformation and numbering of atoms.

Cell dimensions from setting angles of 25 reflections, 2038 independent reflections ($0 \leq \theta \leq 27°$) collected; three intensity- and orientation-control reflections measured every 120 min and 200 reflections, respectively, showed no significant drift; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo radiation and ω/θ scan; absorption correction omitted because of the small size of the crystal ($0{\cdot}2 \times 0{\cdot}15 \times 0{\cdot}1$ mm) and the low linear absorption coefficient, structure solved with *MULTAN* (Germain, Main & Woolfson, 1971), *E* map with highest combined figure of merit (2·999) revealing all non-hydrogen atoms; refinement on *F* by Gauss–Seidel block method (Sparks, 1974), in which each reflection was given a weight based on counting statistics; 18 out of 27 H atoms located from difference Fourier maps, the remainder placed at expected positions; H-atom positions not refined, their Debye–Waller temperature parameters fixed at 6 Å^2, the value of the overall *B* in the Wilson plot being 5 Å^2; $R_w = 0{\cdot}046$, $R = 0{\cdot}044$, $S = 5{\cdot}2$, $(\Delta/\sigma)_{\max} = 0{\cdot}04$, final difference-Fourier map peaks $\leq 0{\cdot}1$ e Å^{-3}; no extinction correction applied, atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Relevant parameters* are listed in Table 1, the numbering of the atoms is given in Fig. 1. Note that the crystallographic numbering scheme is used in what follows. Since we did not determine the absolute configuration the coordinates of Table 1 happen to give the mirror image of the true configuration. The numbers given in the *Discussion*, however, are transformed into those of the C(2)*R*, C(11)*R*, C(13)*S* configuration.

As could be expected the cyclohexane ring is in the chair conformation with the TMPPA-ester moiety and the *tert*-butyl group on equatorial valencies. Bond distances, valence angles and selected torsion angles are given in Tables 2, 3 and 4, respectively. The values are to be compared to those of the (2*R*)-TMPPA ester of *trans*-4-*tert*-butylcyclohexanol (Doesburg, Petit & Merckx, 1982). The agreement is striking, while most of the few discrepancies can be accounted for.

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* Lists of structure factors, H-atom positions and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39077 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Positional parameters in fractions of the cell edges and isotropic temperature factors* (Å²)

The e.s.d.'s given in parentheses refer to the last significant digit. Isotropic temperature factors are calculated from anisotropic temperature parameters assuming equal volume of the 50% probability region. $B_{\rm iso}$ was calculated according to Lipson & Cochran (1966): $B_{\rm iso} = 8\pi^2\,(U^{\circ}_{11}U^{\circ}_{22}U^{\circ}_{33})^{1/3}$. All anisotropic thermal parameters were physically acceptable.

	x	y	z	$B_{\rm iso}$
O(1)	0·3928 (6)	0·0114 (3)	0·3836 (2)	6·41
O(2)	0·2326 (5)	0·1297 (2)	0·4022 (2)	3·74
O(3)	0·4400 (5)	0·1790 (2)	0·5209 (2)	3·98
F(1)	0·6377 (4)	0·0594 (2)	0·5945 (2)	6·44
F(2)	0·5422 (5)	−0·0504 (2)	0·5281 (2)	6·51
F(3)	0·6938 (4)	0·0486 (3)	0·4716 (2)	6·92
C(1)	0·3464 (7)	0·0710 (3)	0·4224 (3)	3·78
C(2)	0·4088 (6)	0·0894 (3)	0·5078 (3)	3·51
C(3)	0·5733 (7)	0·0360 (3)	0·5251 (3)	5·02
C(4)	0·2726 (6)	0·0616 (3)	0·5670 (3)	2·96
C(5)	0·2736 (7)	0·0994 (3)	0·6420 (3)	3·93
C(6)	0·1523 (8)	0·0749 (4)	0·6975 (3)	5·02
C(7)	0·0327 (7)	0·0130 (4)	0·6796 (3)	5·30
C(8)	0·0295 (7)	−0·0252 (4)	0·6066 (3)	5·37
C(9)	0·1508 (7)	−0·0012 (4)	0·5505 (3)	4·49
C(10)	0·5437 (8)	0·2257 (4)	0·4662 (3)	6·24
C(11)	0·1567 (7)	0·1246 (3)	0·3228 (3)	3·69
C(12)	0·2183 (7)	0·2028 (3)	0·2768 (3)	4·05
C(13)	0·1358 (7)	0·2077 (3)	0·1940 (3)	3·89
C(14)	−0·0570 (8)	0·2051 (4)	0·2028 (3)	5·05
C(15)	−0·1197 (8)	0·1284 (4)	0·2515 (3)	5·84
C(16)	−0·0339 (8)	0·1245 (4)	0·3329 (3)	4·71
C(17)	0·2034 (8)	0·2860 (3)	0·1432 (3)	4·81
C(18)	0·3963 (9)	0·2823 (4)	0·1378 (3)	6·83
C(19)	0·1343 (10)	0·2801 (4)	0·0601 (3)	7·68
C(20)	0·1536 (10)	0·3738 (4)	0·1776 (4)	7·62

Table 2. *Bond lengths* (Å) *with e.s.d.'s in parentheses for the title compound and the* (2*R*)-*MTPPA ester of trans-4-tert-butyl cyclohexanol* (*Doesburg et al.*, 1982)

The first column contains values for the title compound, the second column, those for the ester of *trans*-4-*tert*-butylcyclohexanol.

O(1)—C(1)	1·173 (3)	1·206 (5)	C(6)—C(7)	1·357 (4)	1·373 (8)
O(2)—C(1)	1·302 (3)	1·336 (5)	C(7)—C(8)	1·361 (4)	1·400 (7)
O(2)—C(11)	1·465 (3)	1·469 (4)	C(8)—C(9)	1·386 (4)	1·386 (5)
O(3)—C(2)	1·396 (3)	1·395 (6)	C(11)—C(12)	1·495 (3)	1·502 (6)
O(3)—C(10)	1·417 (3)	1·431 (6)	C(11)—C(16)	1·497 (4)	1·519 (6)
C(3)—F(1)	1·321 (3)	1·347 (5)	C(12)—C(13)	1·539 (3)	1·540 (6)
C(3)—F(2)	1·332 (3)	1·38 (4)	C(13)—C(14)	1·512 (4)	1·544 (6)
C(3)—F(3)	1·317 (3)	1·328 (6)	C(14)—C(15)	1·512 (4)	1·521 (6)
C(1)—C(2)	1·545 (3)	1·546 (5)	C(15)—C(16)	1·527 (4)	1·530 (6)
C(2)—C(3)	1·546 (4)	1·546 (6)	C(13)—C(17)	1·555 (4)	1·555 (5)
C(2)—C(4)	1·517 (3)	1·525 (6)	C(17)—C(18)	1·510 (4)	1·539 (7)
C(4)—C(5)	1·388 (3)	1·407 (7)	C(17)—C(19)	1·504 (4)	1·533 (6)
C(4)—C(9)	1·373 (3)	1·378 (7)	C(17)—C(20)	1·502 (4)	1·529 (7)
C(5)—C(6)	1·380 (3)	1·381 (7)			

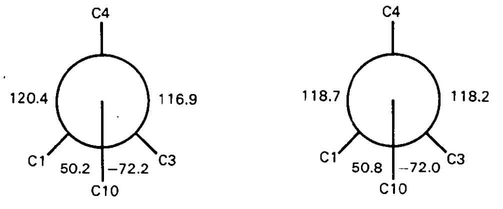

Fig. 2. Newman projection along C(2)—O(3) of the title compound (left) and the 4-*tert*-butylcyclohexanol analogue (right).

The largest differences in the bond lengths are found at the periphery of the molecules (*e.g.* CC in *tert*-butyl group and CF) and are possibly due to shrinkage effects. Thermal movements in the title compound (room-temperature data) are larger than in the other TMPPA ester (123 K data) and consequently the bond lengths in the title compound tend to be smaller. Discrepancies in the valence angles are observed at C(13) and C(14), being respectively tertiary and secondary C atoms in the title compound but respectively secondary and tertiary C atoms in the other ester. Indeed in both compounds the valence angles at the tertiary *C* atoms are *ca* 109° and at the secondary C atoms *ca* 112°.

The agreement between these two compounds shows the rigidity of their molecular structures and points to a similarity of the geometry in the solid state with that in solution. At the same time it confirms a number of features tentatively described by Doesburg *et al.* (1982) as typical of TMPPA esters.

First, the anomeric effect – maximization of overlap between the O(3) lone pairs with the antibonding orbitals of the electronegative groups CF_3 and C(1)OO (Fig. 2) – explains the large value of C(2)—O(3)—C(10) (118·9°) as well as the shortening of C(2)—O(3) with respect to the longer distance C(10)—O(3). It is probably also responsible for the geometrical similarity (Fig. 2) in the ester groups. Second, the phenyl group is nearly perpendicular to the C(2)C(1)O(1)O(2) plane, the actual value in the title compound being 85 (1)°. Third, values of *ca* −14 (1)° are found for the dihedral angle C(3)—C(2)—C(1)—O(1). PCILO-calculations showed (Merckx, Vanhoeck, Lepoivre, Alderweireldt, Van der Veken, De Tollenaere & Raymaeckers, 1983) that the energy content is hardly influenced by deviations from the eclipsed conformation along C(1)—C(2). Fourth, the bonds C(11)—C(12) and C(11)—C(16) are shorter than a normal $C(sp^3)$—$C(sp^3)$ bond, whereas C(13)—C(17) is longer. The latter value (1·555 Å) is close to the theoretically calculated value of Altona & Sundaralingam (1970) for the corresponding bond in *tert*-butylcyclohexane.

Finally we note some differences between the title compound and its *trans*-4-*tert*-butyl analogue. In the title compound the puckering of the cyclohexyl ring is somewhat smaller as follows from the smaller averaged endocyclic torsion angle (55·2 *vs* 57·7°) and the larger averaged endocyclic valence angle (110·7 *vs* 110·0°). In acetates of cyclohexanol derivatives, 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). The angle between C(1)—O(1) and C(11)—H is 7 (2)° in the title compound, but 32 (4)° in the 4-*tert*-butyl analogue.

One of us (MPG) is grateful to the Board of the University of Antwerp (UIA) and to the Belgian

Table 3. *Valence angles (°) with e.s.d.'s in parentheses for the title compound and the (2R)-MTPPA ester of trans-*4-*tert-butylcyclohexanol*

The first column contains values for the title compound, the second column, those for the ester of *trans*-4-*tert*-butylcyclohexanol.

C(1)–O(2)–C(11)	118·6 (2)	116·8 (3)	F(2)–C(3)–F(3)	107·4 (3)	106·3 (3)	C(11)–C(12)–C(13)	112·0 (2)	109·5 (3)
C(2)–O(3)–C(10)	118·9 (2)	118·4 (3)	F(2)–C(3)–C(2)	110·7 (3)	112·0 (3)	C(12)–C(13)–C(14)	109·1 (2)	111·4 (3)†
O(1)–C(1)–O(2)	126·3 (3)	125·8 (3)	F(3)–C(3)–C(2)	112·9 (3)	113·8 (3)	C(12)–C(13)–C(17)	113·2 (2)	114·1 (4)*
O(1)–C(1)–C(2)	124·1 (3)	124·6 (3)	C(2)–C(4)–C(5)	118·7 (3)	120·4 (3)	C(14)–C(13)–C(17)	114·0 (3)	114·4 (4)*
O(2)–C(1)–C(2)	109·6 (2)	109·6 (3)	C(2)–C(4)–C(9)	123·0 (3)	120·1 (4)	C(13)–C(14)–C(15)	113·0 (3)	108·0 (3)†
O(3)–C(2)–C(1)	112·1 (2)	112·4 (3)	C(5)–C(4)–C(9)	118·3 (3)	119·5 (4)	C(14)–C(15)–C(16)	112·0 (3)	112·4 (3)
O(3)–C(2)–C(3)	109·5 (3)	110·1 (3)	C(4)–C(5)–C(6)	120·2 (3)	120·6 (4)	C(15)–C(16)–C(11)	109·5 (2)	109·3 (3)
O(3)–C(2)–C(4)	106·8 (2)	108·2 (3)	C(5)–C(6)–C(7)	120·5 (3)	119·8 (5)	C(13)–C(17)–C(18)	110·1 (3)	109·3 (3)
C(1)–C(2)–C(3)	110·1 (2)	110·0 (3)	C(6)–C(7)–C(8)	120·5 (3)		C(13)–C(17)–C(19)	110·3 (3)	109·5 (4)
C(1)–C(2)–C(4)	110·0 (2)		C(7)–C(8)–C(9)	119·5 (3)	121·3 (4)	C(13)–C(17)–C(20)	112·0 (3)	112·2 (3)
C(3)–C(2)–C(4)	108·3 (2)	108·4 (3)	C(4)–C(9)–C(8)	121·1 (3)	118·8 (4)	C(18)–C(17)–C(19)	107·4 (3)	108·2 (4)
F(1)–C(3)–F(2)	107·4 (3)	107·4 (3)	O(2)–C(11)–C(12)	107·5 (2)	106·5 (3)	C(18)–C(17)–C(20)	108·3 (3)	107·9 (4)
F(1)–C(3)–F(3)	107·2 (3)	107·7 (3)	O(2)–C(11)–C(16)	107·4 (2)	109·7 (3)	C(19)–C(17)–C(20)	108·6 (3)	109·6 (4)
F(1)–C(3)–C(2)	110·0 (3)	109·3 (3)	C(12)–C(11)–C(16)	112·3 (3)	111·9 (3)			

* The values reported refer to the angles C(ring)—C(ring)—C(Bu^t).

† C(13) is a tertiary C atom in the title compound, but a secondary C atom in the *trans*-4-Bu^t-ester. On the other hand C(14) is a secondary C atom in the title compound, but a tertiary C atom in the *trans*-4-Bu^t-ester.

Table 4. *Selected torsion angles (°) in the title compound and in the (2R)-MTPPA ester of trans-*4-*tert-butylcyclohexanol*

E.s.d.'s are 0·5 and 0·4°, respectively.

	Title compound	4-Bu^t analogue
C(16)–C(11)–C(12)–C(13)	58·0	57·8
C(11)–C(12)–C(13)–C(14)	−54·5	−58·1
C(12)–C(13)–C(14)–C(15)	53·1	57·4
C(13)–C(14)–C(15)–C(16)	−54·6	−57·6
C(14)–C(15)–C(16)–C(11)	54·4	57·8
C(15)–C(16)–C(11)–C(12)	−56·5	−57·3
C(1)–C(2)–O(3)–C(10)	50·2	50·8
C(3)–C(2)–O(3)–C(10)	−72·2	−72·0
C(12)–C(13)–C(17)–C(18)	−54·5	
C(12)–C(13)–C(17)–C(20)	66·0	
C(14)–C(13)–C(17)–C(19)	61·5	
C(14)–C(13)–C(17)–C(20)	−59·5	

National Science Foundation (NFWO) for a Visiting Professorship during the tenure of which the present work was done. LV thanks the Belgian Organization 'Instituut voor Wetenschappelijk Onderzoek in Nijverheid en Landbouw' (IWONL) for a predoctoral grant. The work was partly supported by NATO, Research Grant Number 0314/82.

References

ALTONA, C. & SUNDARALINGAM, M. (1970). *Tetrahedron*, **26**, 925–939.

COREY, E. J. & SUGGS, J. W. (1973). *Tetrahedron Lett.* **31**, 2647–2650.

DOESBURG, H. M., PETIT, G. H. & MERCKX, E. M. (1982). *Acta Cryst.* B**38**, 1181–1185.

GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.

LIPSON, H. & COCHRAN, W. (1966). *The Determination of Crystal Structures*, pp. 300*ff.* London: Bell.

MATHIESON, A. McL. (1965). *Tetrahedron, Lett.* pp. 4137–3144.

MERCKX, E. M. (1982). PhD Thesis, Univ. of Antwerp.

MERCKX, E. M., VAN DE WAL, A. J., LEPOIVRE, J. A. & ALDERWEIRELDT, F. C. (1978). *Bull. Soc. Chim. Belg.* **87**, 21–25.

MERCKX, E. M., VANHOECK, L., LEPOIVRE, J. A., ALDERWEIRELDT, F. C., VAN DER VEKEN, B. J., DETOLLENAERE, J. & RAYMAECKERS, L. A. (1983). *Spectrosc. Int. J.* In the press.

SADOZAI, S. K., LEPOIVRE, J. A., DOMMISSE, R. A. & ALDERWEIRELDT, F. C. (1980). *Bull. Soc. Chim. Belg.* **89**, 637–642.

SPARKS, R. A. (1974). Least-squares tutorial, Proc. Am. Crystallogr. Assoc. Meet., Berkeley, CA, pp. 99*ff.*

VAN DE WAL, A. J., MERCKX, E. M., LEMIÈRE, G. L., LEPOIVRE, J. A. & ALDERWEIRELDT, F. C. (1978). *Bull. Soc. Chim. Belg.* **87**, 545–552.

VANHOECK, L., BOSSAERTS, J., DOMMISSE, R. A., LEPOIVRE, J. A. & ALDERWEIRELDT, F. C. (1983). *Org. Magn. Reson.* In the press.

VANHOECK, L., MERCKX, E. M., BOSSAERTS, J., LEPOIVRE, J. A. & ALDERWEIRELDT, F. C. (1983). *Org. Magn. Reson.* **21**, 214–216.

VAN OSSELAER, T. A., LEMIÈRE, G. L., MERCKX, E. M., LEPOIVRE, J. A. & ALDERWEIRELDT, F. C. (1978). *Bull. Soc. Chim. Belg.* **87**, 799–800.