atoms (Table 2) are generally larger than the previously determined values in well refined, ordered silicate structures (Burnham, 1965). Again, this may be partly because of the disordering of the crystal and partly because of the inherent internal strain which induces the distortion of the ideal structure (Vousden, 1954; Lin, 1971). In order to confirm that atoms originally located on the mirror plane in the ideal structure ($I4/m$) do migrate away from the mirror plane when refined according to $P4_2/n$, a vertical electron-density section was prepared and this shows a round (Na,Ca,K) peak with the centre clearly located away from the mirror plane.

In this marialitic scapolite, nearly one fourth of the central cavities are occupied by a carbonate group. This is shown in both the Fourier synthesis and the difference synthesis in which four peaks elongated parallel to the c axis and symmetrical around the origin (000) and with a density maximum on the (001) plane are present (Fig. 6). This has been previously described and explained by Papike & Stephenson (1966). However, some of the CO_3 groups are believed to be tilted from the (001) plane, as evidenced by the elongated peaks present on the vertical electron-density maps and the large temperature factors. In conclusion, the crystal structure of this sodium and chlorine-rich scapolite has successfully been refined in the newly determined space group $P4_2/n$. The overall structure is shown to be pseudo body-centred, contrary to previous reports.

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The Crystal and Molecular Structure of the Dimethyl Ester of *meso*-Tartaric Acid

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Crystals of the dimethyl ester of *meso*-tartaric acid are triclinic, space group $P\bar{1}$, with $a = 8\cdot117$, $b = 9\cdot619$, $c = 5\cdot606$ Å, $\alpha = 96\cdot8$, $\beta = 103\cdot0$ and $\gamma = 73\cdot9°$ ($Z = 2$). Intensity data were measured on an automatic diffractometer. The structure was solved by direct methods and refined to an R of 0·096. The molecular conformation is identical with the dissymmetric conformation found in *meso*-tartaric acid and its derivatives. The hydrogen atoms of the methyl groups show a disordering over a staggered and eclipsed position with respect to the C–O bond of the ester group. The hydrogen-bond scheme is the same as in racemic acid dihydrate and two modifications of *meso*-tartaric acid.

Introduction

In *meso*-tartaric acid and its salts the *meso*-tartaric acid molecule has a dissymmetric conformation (Boots-ma & Schoone 1967; Kroon & Kanters 1972; Kroon, Peerdeman & Bijvoet 1965) and both conformational antipodes are present in the unit cell, thus constituting a racemate. In order to acquire knowledge of the effect

of esterification of the carboxyl group on the molecular conformation the structure analysis of the dimethyl ester of *meso*-tartaric acid was undertaken.

Experimental

meso-Tartaric dimethyl ester crystals were obtained by sublimation in high vacuum. The crystals are triclinic, $a = 8\cdot117$, $b = 9\cdot619$, $c = 5\cdot606$ Å, $\alpha = 96\cdot8$, $\beta = 103\cdot0$, $\gamma = 73\cdot9°$. The Delaunay reduced cell may be obtained under the transformation matrix $(001/010/\overline{1}0\overline{1})$ with cell constants $a' = 5\cdot606$, $b' = 9\cdot619$, $c' = 8\cdot762$ Å, $\alpha' = 100\cdot4$, $\beta' = 115\cdot5$ and $\gamma' = 96\cdot8°$. The density calculated is $1\cdot46$ g cm^{-3} $(Z = 2)$.

From a crystal of dimensions $0\cdot6 \times 0\cdot4 \times 0\cdot2$ mm sealed in a thin-walled capillary 1207 non-zero intensities (65 % of the copper $K\alpha$ sphere) were collected with a CAD-3 Nonius diffractometer $(\lambda_{\text{Cu }K\alpha} = 1\cdot5418$ Å) according to the ω-scan technique. No absorption correction was applied. The reduced intensities were put on an absolute scale with Wilson's (1942) method.
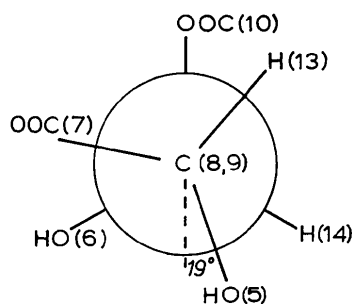

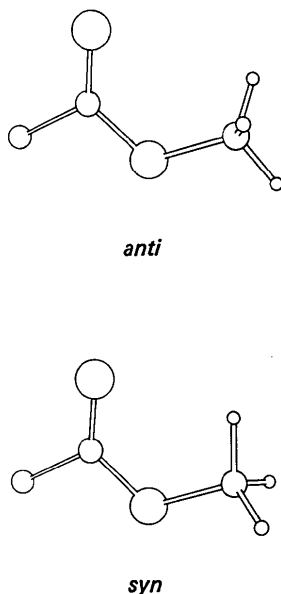Fig. 1. Newman projection along C(8)–C(9).

anti

syn

Fig. 2. *Anti* and *syn* positions of the methyl groups in the ester groups.

The ratio test of Ramachandran & Srinivasan (1959) yields a value of 0·78 and indicates the space group $P\overline{1}$.

Structure determination

A symbolic procedure (Spek, 1970) was applied to 2295 origin invariants $E_{H1}E_{H2}E_{H1+H2}$ that were generated from 343 E values such that two E values were larger than 1·20 and the third one larger than 0·75. Four sets of signs for all 343 structure factors were obtained. The structure could easily be deduced from the Fourier synthesis of one of these sets. The further course of the structure analysis is summarized in Table 1.

Table 1. *Least-squares refinement of the dimethyl ester of meso-tartaric acid*

Parameters refined		R value
(a)	Non-hydrogen atoms: coordinates and anisotropic thermal parameters.	0·121
(b)	As (a), and coordinates of non-methyl hydrogen atoms. Methyl hydrogen atoms fixed at *anti* positions. Isotropic B values for the hydrogen atoms kept fixed at values corresponding to peak heights in the difference electron density map.	0·100
(c)	As (a), and coordinates of non-methyl hydrogen atoms. Methyl hydrogen atoms fixed at *anti* and *syn* positions with a 50:50 occupancy. Thermal parameters for the hydrogen atoms as in (b).	0·096
(d)	As (c), and coordinates of all hydrogen atoms.	0·094

Inspection of the difference electron density synthesis calculated at an R value of 0·121 revealed maxima at the expected positions of the non-methyl hydrogen atoms. As for the hydrogen atoms of the methyl groups the synthesis points to a disordering. The positions of the maxima do not deviate much from those calculated for the *anti* and *syn* positions of the CH$_3$-hydrogen atoms. The peak heights indicate a 50:50 occupancy for both methyl groups (Table 2). Refinement of the disordered model gives only a slight lowering of the R value (Table 1), but results in an unreasonable geometry, which is not surprising for a refinement of hardly separated hydrogen atoms with half scattering power. Table 3 gives final positional and thermal parameters and Table 4 the observed and calculated structure factors $(R = 0\cdot096)$.

Discussion

The conformation of the molecule (*synclical*) is the same as that found in the *meso*-tartaric acids (Bootsma & Schoone, 1967), potassium *meso*-tartrate dihydrate (Kroon, Peerdeman & Bijvoet, 1965) and potassium

Table 2. *Electron-density maxima in the difference Fourier synthesis with all terms $F_o - F_c$ (non-hydrogen atoms)*

Atom	Bonded to	Maximum (e Å^{-3})
H(13)	C(8)	0·7
H(14)	C(9)	0·7
H(15)	O(5)	0·6
H(16)	O(6)	0·4
H(17)		0·4
H(18) *anti*	C(11)	0·3
H(19)		0·4
H(23)		0·4
H(24) *syn*	C(11)	0·3
H(25)		0·3
H(20)		0·3
H(21) *anti*	C(12)	0·3
H(22)		0·4
H(26)		0·3
H(27) *syn*	C(12)	0·3
H(28)		0·5

hydrogen *meso*-tartrate (Kroon & Kanters, 1972). In the unit cell both conformational antipodes are present. The non-hydrogen atoms of both the glycollic acid ester parts of the molecule are coplanar. The dihedral

angles regarding the conformation around bonds are summarized in Table 5. A Newman projection along the central C–C bond is given in Fig. 1.

The hydrogen atoms of the methyl groups are distributed in approximately equal weights over the *anti* and *syn* conformation (Fig. 2). At first sight the occurrence
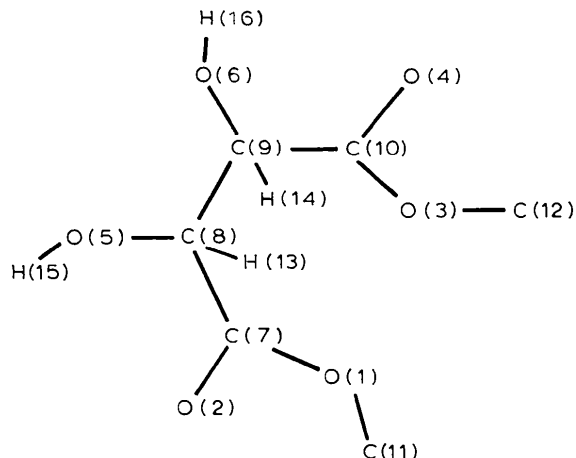

Fig. 3. Numbering of the atoms in the molecule of the dimethyl ester of *meso*-tartaric acid.

Table 3. *Final positional ($\times 10^4$) and thermal parameters with e.s.d's in parentheses*

The anisotropic temperature factor is of the form $\exp(-10^{-4} \sum_i \sum_j h_i h_j \beta_{ij})$.

	x	y	z	β_{11}	β_{22}	β_{33}	$2\beta_{12}$	$2\beta_{23}$	$2\beta_{31}$
O(1)	3300 (5)	6134 (4)	6534 (8)	165	119	413	−69	164	−31
O(2)	728 (7)	6642 (6)	3887 (10)	203	235	544	−213	248	−201
O(3)	6224 (5)	8082 (5)	6930 (7)	102	162	282	−55	98	68
O(4)	5002 (5)	8318 (5)	10229 (7)	134	159	242	−67	167	43
O(5)	1555 (5)	8834 (5)	2169 (7)	106	198	220	19	185	69
O(6)	1776 (5)	9490 (4)	7563 (7)	122	128	253	23	131	90
C(7)	2119 (7)	6868 (7)	4730 (10)	132	139	283	−62	68	− 3
C(8)	2784 (7)	8067 (6)	4086 (9)	101	123	234	−11	99	70
C(9)	3192 (7)	9090 (6)	6317 (10)	111	104	260	−14	105	102
C(10)	4897 (7)	8451 (6)	8098 (9)	115	103	255	−78	125	0
C(11)	2775 (10)	5044 (8)	7550 (15)	233	127	579	−91	199	36
C(12)	7940 (8)	7490 (8)	8397 (13)	98	182	446	−26	156	37

	x	y	z	B (Å^2)	Bonded to	Conformation
H(13)	0·390 (8)	0·760 (6)	0·346 (11)	2	C(8)	
H(14)	0·322 (8)	1·002 (6)	0·571 (10)	2	C(9)	
H(15)	0·058 (8)	0·924 (7)	0·262 (12)	3	O(5)	
H(16)	0·178 (8)	0·897 (7)	0·835 (12)	3	O(6)	
H(17)	0·248	0·429	0·621	4		
H(18)	0·374	0·454	0·887	4	C(11)	*anti*
H(19)	0·170	0·550	0·826	4		
H(20)	0·823	0·822	0·976	4		
H(21)	0·796	0·658	0·914	4	C(12)	*anti*
H(22)	0·884	0·725	0·736	4		
H(23)	0·154	0·502	0·670	4		
H(24)	0·358	0·406	0·731	4	C(11)	*syn*
H(25)	0·280	0·527	0·935	4		
H(26)	0·784	0·746	1·015	4		
H(27)	0·874	0·812	0·836	4	C(12)	*syn*
H(28)	0·846	0·649	0·775	4		

of an eclipsed conformation is surprising, and leads one to speculate on the possible influence of an interaction between a carbonyl oxygen atom and a hydrogen atom in the methyl group in the *syn* position, their distances being 2·21 and 2·23 Å in the respective ester groups. It would be worth while to have evidence of similar configurations in other organic acid methyl esters.

Bond lengths and angles are given in Table 6 (the

Table 4. *Observed and calculated structure factors*

The columns are h, k, l, $|10F_o|$, $10F_c$.

h	k	l	|10Fo|	10Fc
	-6	0	53	-62
	-5		21	-17
	-4		24	-11
	-3		46	52
	-2		30	20
	-1		49	-50
	0		49	-50
	1		30	-33
-8	-8	0	10	-19
	-6		48	50
	-5		74	77
	-4		6	-17
	-3		100	-103
	-1		29	21
	1		13	-8
	2		74	-75
	3		52	-54
-7	-8	0	28	26
	-7		30	-25
	-6		28	-21
	-5		35	-27
	-4		40	34
	-3		45	50
	-2		125	129
	-1		71	75
	0		72	-73
	1		34	-45
	2		17	15
	3		73	-77
	4		29	-18
	5		21	-16
-6	-9	0	31	-32
	-8		24	19
	-7		43	40
	-6		39	-52
	-5		6	19
	-3		31	-36
	-2		49	-56
	-1		30	31
	0		138	141
	1		174	176
	2		106	107
	3		45	-49
	4		126	-125
	6		13	-7
-5	-11	0	12	-16
-4	-11	0	14	-1
	-10		38	-34
	-9		14	-37
	-8		45	-40
	-6		125	132
	-5		53	-58
	-3		131	-124
	-2		193	-186
	-1		91	82
	1		112	-114
	2		61	60
	3		28	-23
	4		80	83
	5		8	15
	7		59	54
-3	-10	0	73	68
	-9		53	48
	-8		19	23
	-7		122	-127
	-6		83	-81
	-5		53	61
	-4		107	111
	-3		84	92
	-2		58	-56
	-1		288	-284
	0		160	-158
	1		194	-200
	2		128	129
	3		34	31
	4		45	-43
	5		66	67
	6		10	3
	7		20	-15
	8		5	7
-2	-10	0	19	-19
	-9		14	-9
	-8		14	-30
	-7		182	172
	-6		87	86
	-5		34	35
	-4		37	-37
	-3		54	-45
	-2		9	21
	-1		198	207
	0		317	-314
	1		77	-76
	2		255	-258
	3		119	-114
	4		145	-139
	5		72	66
	6		81	77
	7		13	-9
	8		8	-4
-1	-10	0	16	-15
	-9		27	22
	-8		18	-4
	-6		51	-45
	-5		207	-207
	-4		243	242
	-3		697	719
	-2		150	154
	-1		216	214
	0		164	-160
	1		354	-360
	2		44	39
	3		19	-14
	4		115	-119
0	-9	0	41	-45
	-8		6	-26
	-7		54	-55
	-6		86	84
	-5		17	10
	-4		159	-163
	-3		197	-200
	-2		194	-202
	-1		218	229
1	-9	0	57	53
	-7		27	-17
	-6		73	-75
	-5		106	-111
2	-10	0	41	-45
-9	-7	1	49	-55
	-6		25	-24
	-5		18	5
	-4		26	35
	-2		15	0
	-1		53	59
	1		8	-15
-8	-7	1	43	40
	-5		34	-38
	-4		95	-95
	-3		59	-55
	-2		18	-30
	-1		55	58
	0		58	56
	1		15	9
-7	-9	1	43	46
	-7		36	39
	-5		76	-73
	-4		59	57
	-3		72	67
	-2		50	-50
	-1		155	-160
	0		92	-91
	1		28	12
-7	2	1	46	51
	3		91	48
	4		22	-17
	5		46	-44
-6	-10	1	38	-34
	-6		175	174
	-5		12	21
	-3		16	-24
	-2		89	-83
	-1		44	41
	0		128	126
	1		30	-35
	2		113	-108
	3		103	-101
	4		23	26
	5		22	-1
-5	-11	1	19	12
	-8		13	14
	-7		83	-61
	-6		45	-37
	-5		89	88
	-4		5	-11
	-3		178	182
	-2		83	81
	-1		45	-43
	1		38	-40
	2		46	42
	3		117	124
	6		90	-90
	7		34	-40
-4	-11	1	28	-23
	-10		28	-24
	-8		42	-37
	-6		39	-39
	-5		103	96
	-4		54	49
	-3		67	-67
	-1		20	13
	0		33	29
	1		165	161
	3		26	38
	4		28	-29
	5		49	50
	6		101	103
	8		38	-40
-3	-11	1	25	17
	-9		40	-28
	-8		24	-36
	-7		115	-109
	-6		16	21
	-5		194	-197
	-4		25	-22
	-3		166	164
	-2		220	213
	-1		7	5
	0		153	-148
	1		253	-247
	2		37	38
	3		63	62
	4		115	117
	5		27	-2
	6		33	26
	7		38	20
	8		33	26
	9		40	39
-2	-11	1	16	13
	-10		38	27
	-9		32	-22
	-8		70	62
	-7		36	4
	-5		122	-117
	-4		413	-405
	-2		145	137
	-1		160	-152
	0		402	401
	1		70	66
	2		199	-181
	3		98	-99
	4		68	-61
	6		48	44
	7		36	38
-1	-11	1	13	9
	-10		14	27
	-9		83	87
	-8		20	24
	-7		26	21
	-6		76	-82
	-5		211	210
	-4		36	44
	-3		143	-148
	-2		293	-304
	-1		408	-416
	0		129	-132
	1		556	592
	2		300	-312
	3		29	-24
	5		77	-82
	6		26	-38
0	-10	1	73	-70
	-8		60	64
	-7		64	62
	-6		118	117
	-5		107	104
	-4		4	-4
	-3		171	-171
	-2		116	120
	-1		344	342
	0		203	-201
	1		49	-57
	2		41	-44
	3		266	-269
	4		69	63
	5		68	-73
	6		86	-80
	7		41	39
	9		15	-23
	10		17	-7
1	-10	1	27	17
	-9		50	-50
	-8		18	22
	-7		59	-59
	-6		79	-74
	-5		85	81
	-4		141	145
	-3		43	43
	-2		302	299
	-1		62	-54
	0		116	-108
	1		133	-133
	2		611	622
	3		214	199
	4		49	-50
	5		106	-105
	6		131	-129
	7		58	-62
	8		34	39
	10		23	15
	11		4	-11
2	-10	1	20	16
	-9		34	-29
	-8		82	-79
	-7		33	48
	-6		20	24
	-5		14	32
	-3		293	-292
	-2		34	-30
	-1		194	190
	0		52	70
	1		198	200
	2		133	-124
	3		23	-11
2	4	1	139	132
	5		229	241
	6		129	126
	7		57	-64
	8		106	-101
	9		13	-23
	11		17	13
3	-8	1	25	-32
	-7		32	-30
	-6		57	-56
	-5		100	-97
	-4		70	65
	-3		225	214
	-2		54	-60
	-1		112	-95
	0		442	-424
	1		58	-55
	2		112	107
	3		21	17
	4		102	104
	5		26	36
	6		30	-35
	7		96	104
	8		41	29
	9		31	31
	10		18	-9
	11		25	-25
4	-8	1	39	43
	-7		25	-22
	-6		39	-51
	-5		27	-34
	-4		17	-34
	-3		28	-7
	0		147	133
	1		62	-52
	2		59	-70
	3		228	-221
	4		105	-96
	5		80	75
	6		32	24
	7		40	39
	8		64	61
	9		46	-44
5	-7	1	34	38
	-6		19	19
	-4		23	2
	-3		125	-127
	-2		32	29
	-1		83	87
	1		85	-89
	3		44	37
	5		36	-39
	6		60	-63
	7		78	-77
	8		37	50
	9		26	30
	10		14	-4
6	-6	1	37	38
	-5		17	20
	-4		33	28
	-2		55	-57
	-1		90	95
	0		15	-3
	1		24	-23
	2		55	59
	3		25	-36
	4		89	-97
	5		28	12
	6		24	31
	8		13	-40
	10		20	-17
7	-4	1	15	16
	-3		65	65
	-2		32	-29
	-1		41	-41
	0		55	54
	1		24	29
	2		76	79
	3		53	54
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	1		28	-6
	2		14	4
-2	-5	6	9	-5
	-4		82	-82
	-1		27	34
	0		72	70
	1		47	56
	2		38	-3
	3		22	-18
-1	-5	6	19	-26
	-3		11	-15
	-2		29	-31
	-1		35	-39
	0		51	-57
	1		27	7
	3		31	13
	4		20	10
0	-5	6	30	33
	-4		17	12
	-3		19	1
	-2		56	-61
	4		22	-32
1	-4	6	18	5
	-2		27	35
	3		29	37
2	-2	6	20	16
	-1		21	4
	0		29	-9
	2		27	-6

Table 5. *Conformations in the molecule of the dimethyl ester of meso-tartaric acid*

Plane through	Plane through	Dihedral angle	Conformation about
O(3)O(4)C(9)C(10)	O(6)C(9)C(10)	0°	C(9)–C(10)
O(1)O(2)C(7)C(8)	O(5)C(7)C(8)	2	C(7)–C(8)
O(1)C(7)C(11)	O(1)C(7)O(2)	3	O(1)–C(7)
O(3)C(10)C(12)	O(3)C(10)O(4)	1	O(3)–C(10)
O(5)C(8)C(9)	C(8)C(9)C(10)	19	C(8)–C(9)

Table 6. *Bond lengths and angles*

C(7)—O(1)	1·341 (8) Å	O(1)—C(7)—O(2)	124·2 (6)°	
C(7)—O(2)	1·190 (8)	O(1)—C(7)—C(8)	109·5 (5)	
C(11)—O(1)	1·450 (9)	C(11)—O(1)—C(7)	116·3 (5)	
C(10)—O(3)	1·329 (7)	O(2)—C(7)—C(8)	126·2 (6)	
C(10)—O(4)	1·198 (7)	O(3)—C(10)—O(4)	125·7 (5)	
C(12)—O(3)	1·448 (8)	O(3)—C(10)—C(9)	109·9 (4)	
C(8)—O(5)	1·411 (7)	O(4)—C(10)—C(9)	124·4 (5)	
C(9)—O(6)	1·418 (7)	C(12)—O(3)—C(10)	116·3 (4)	
C(7)—C(8)	1·511 (8)	O(5)—C(8)—C(7)	109·8 (5)	
C(8)—C(9)	1·531 (8)	O(5)—C(8)—C(9)	111·0 (4)	
C(9)—C(10)	1·530 (8)	O(6)—C(9)—C(8)	109·6 (4)	
C(8)—H(13)	1·01 (6)	O(6)—C(9)—C(10)	109·9 (4)	
C(9)—H(14)	0·99 (6)	C(7)—C(8)—C(9)	111·8 (4)	
O(5)—H(15)	0·86 (7)	C(8)—C(9)—C(10)	113·5 (4)	
O(6)—H(16)	0·70 (7)	C(8)—O(5)—H(15)	111 (4)	
C–H (methyl)	1·00*	C(9)—O(6)—H(16)	114 (5)	
		C(7)—C(8)—H(13)	108 (3)	
		C(9)—C(8)—H(13)	109 (3)	
		O(5)—C(8)—H(13)	108 (3)	
		C(8)—C(9)—H(14)	108 (3)	
		C(10)-C(9)—H(14)	111 (3)	
		O(6)—C(9)—H(14)	104 (3)	

O–C–H (methyl) ⎱ Tetrahedral
H–C–H ⎰ angle*

* Assumed

numbering in the molecule is shown in Fig. 3). It is of interest to note the geometry in the ester group. The carboxyl group C–O and C=O distances are 1·34 and 1·19 Å respectively, which is different from values in carboxylic acids: 1·31 and 1·23 Å (Kanters, Kroon, Peerdeman & Schoone, 1967). The bond angles C–C–O (110°) and C–C=O (125°) also differ from those in carboxylic acids: 113 and 123° (Kanters *et al.*, 1967). These bond angles as well as the bond lengths reflect a sharper distinction between single and double bonds of the
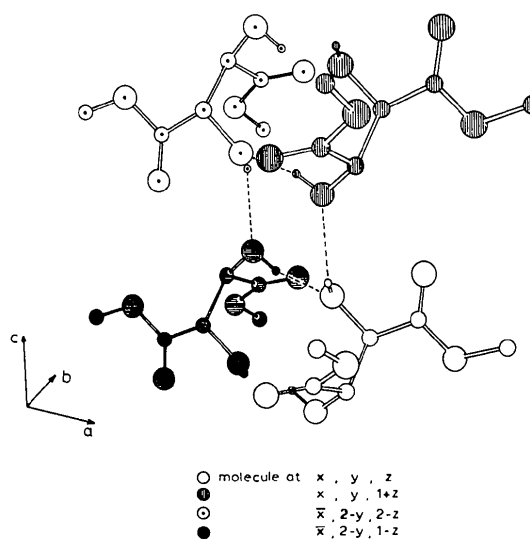

O molecule at	x , y , z
◐	x , y , 1+z
⊙	x̄ , 2-y , 2-z
●	x̄ , 2-y , 1-z

Fig. 4. Hydrogen bonding in the crystal structure of dimethyl *meso*-tartrate.

Table 7. *Geometry in several ester groups*

$$\begin{array}{c} \text{O(5)} \\ | \quad \quad // \\ -\text{C(1)}-\text{C(2)} \\ | \quad \quad \backslash \\ \quad \quad \text{O(4)}-\text{C(3)} \end{array}$$

	Bond lengths (Å)		Bond angles (°)		Reference
Compound	C(2)–O(4)	C(2)–O(5)	C(1)C(2)O(4)	C(1)C(2)O(5)	
1. Dimethyl oxalate	1·31 (2)	1·19 (2)	109 (2)	125 (2)	(1)
2. Methyl stearate	1·36 (2)	1·21 (2)	113 (2)	131 (2)	(2)
3. Ethyl carbamate* (room temperature)	1·333 (4)	1·223 (3)	114·2 (5)	122·4 (5)	(3)
4. 2,5-Diphenyl-1,3a,4,6a-tetra-azapentalene-3,6-dicarboxylic acid dimethyl ester	1·34 (2)	1·22 (2)	114 (1)	125 (1)	(4)
	1·32 (2)	1·20 (2)	112 (1)	124 (1)	
5. Ethyl *p*-azoxybenzoate	1·318 (7)	1·191 (7)	111·9 (5)	122·8 (5)	(5)
	1·324 (7)	1·196 (7)	112·6 (5)	123·2 (5)	
6. Propargyl 2-bromo-3-nitrobenzoate	1·32 (1)	1·18 (1)	110·0 (9)	126·0 (10)	(6)
7. L-5-Methoxycarbonyl-7-formyl-1,2,5,6-tetrahydro-3*H*-pyrrolo[1,2*a*]azepin-3-one	1·348 (10)	1·164 (10)	108·7 (6)	126·6 (6)	(7)
8. *p*-Nitrophenyl acetate	1·359 (14)	1·183 (15)	111·0 (8)	126·3 (9)	(8)
	1·359 (16)	1·193 (16)	109·9 (10)	125·8 (10)	
9. Ethyl-3,5-dinitrobenzoate	1·305 (4)	1·186 (6)	111·3 (3)	123·4 (3)	(9)
Title compound	1·341 (8)	1·190 (8)	109·5 (5)	126·2 (5)	
	1·329 (7)	1·198 (7)	109·9 (4)	124·4 (5)	

* Here C(1) is replaced by N.

References:
(1) Dougill & Jeffrey (1953)
(2) MacGillavry & Wolthuis-Spuy (1970)
(3) Bracher & Small (1967)
(4) Brufani, Casini, Fedeli, Mazza & Vàciago (1971)
(5) Kriegbaum & Barber (1971)

(6) Calabrese, McPhail & Sim (1966)
(7) Karle, Karle & Estlin (1967)
(8) Guttormson & Robertson (1972)
(9) Hughes & Trotter (1971)

Table 8. *Geometry in the hydrogen bonds*

Donor (D)–H	Acceptor (A)			Distance $D \cdots A$	Distance $H \cdots A$	Angle D–$H \cdots A$
O(5)–H(15)	O(6)	$-x$ $2-y$	$1-z$	2·764 (6) Å	1·95 (7) Å	158 (6)°
O(6)–H(16)	O(5)	x y	$1+z$	2·785 (6)	2·21 (7)	140 (6)

carboxyl group moiety in the ester when compared with free acids in general. These findings are corroborated by other analyses of esters as Table 7 shows.

The hydrogen-bonding system in the *meso*-tartaric acid ester is very similar to that found in the racemic acid dihydrate (Parry, 1951) and the triclinic modifications of *meso*-tartaric acid (Bootsma & Schoone, 1967). Each hydroxyl group takes part in two hydrogen bonds, as shown in Fig. 4. One hydroxyl group is connected to a hydroxyl group of an antipode molecule [O(5)–H(15)$\cdots$O'(6)] and the other one with a hydroxyl group of a molecule of the same configuration shifted one period along the c axis [O(6)–H(16)$\cdots$O''(5)] (Table 8).

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The Crystal and Molecular Structure of Diiodobis(triphenylphosphine)palladium(II)

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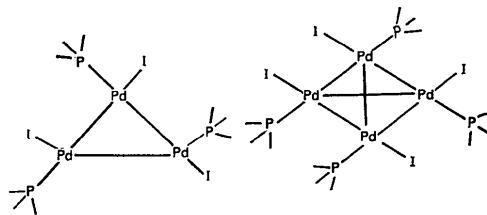
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The compound crystallizes in the form of monoclinic yellow needles; $a = 11·922$, $b = 20·432$, $c = 8·35$ Å, $\beta = 95·18°$, space group $P2_1/c$. The palladium atom is on an inversion centre which is the centre of an octahedron formed by the atoms of iodine, phosphorus and the hydrogen on a β carbon of a phenyl ring.

Introduction

Schmid & Weber (1971) have pointed out the interest of the X-ray structure analysis of a compound for which the chemical analysis of carbon, iodine and hydrogen had suggested the formula $[(C_6H_5)_3PPdI]_n$, in which n was unknown but greater than 2, and probably equal to 3 or 4, where the molecules were linked together by metal–metal bonds. The structures were supposed to be of the form:

Usually, palladium(II) with a d^8 configuration forms quadratic complexes with 4 ligands. This is the case