

C21	0.3521 (6)	-0.0266 (4)	0.3614 (4)	0.0369 (9)
C22	0.2727 (6)	-0.0715 (4)	0.4789 (4)	0.0449 (10)
C23	0.1771 (6)	0.0211 (4)	0.5856 (4)	0.0449 (10)
C24	0.1606 (6)	0.1595 (4)	0.5721 (4)	0.0431 (10)
C25	0.2413 (5)	0.2030 (4)	0.4546 (3)	0.0316 (8)

Table 4. Selected geometric parameters (\AA , $^\circ$) for (II)

Os1—F1	1.923 (2)	C11—C12	1.370 (6)
Os1—C111	2.3353 (8)	C12—C13	1.361 (8)
Os1—C112	2.3488 (9)	C13—C14	1.365 (8)
Os2—F2	1.929 (2)	C14—C15	1.365 (6)
Os2—C121	2.3323 (9)	N2—C25	1.344 (4)
Os2—C122	2.3444 (9)	N2—C21	1.346 (4)
CM—N1	1.465 (4)	C21—C22	1.362 (5)
CM—N2	1.488 (4)	C22—C23	1.365 (6)
N1—C15	1.341 (5)	C23—C24	1.370 (6)
N1—C11	1.343 (5)	C24—C25	1.357 (5)
F1 ⁱ —Os1—C111	90.10 (6)	F2—Os2—C121	90.59 (7)
F1—Os1—C111	89.90 (6)	F2 ⁱⁱ —Os2—C122	90.44 (7)
F1—Os1—C112 ⁱ	90.31 (7)	F2—Os2—C122	89.56 (7)
F1—Os1—C112	89.69 (7)	C121 ⁱⁱ —Os2—C122	90.46 (3)
C111—Os1—C112	90.37 (3)	C121—Os2—C122	89.54 (3)
F2—Os2—C121 ⁱⁱ	89.41 (7)	N1—CM—N2	111.0 (3)

Symmetry codes: (i) $-x, -y, -z$; (ii) $1-x, 1-y, 1-z$.

In each compound the major residual features in the ΔF synthesis lie close to the metal atoms.

For both compounds, data collection: *CAD-4-PC* (Enraf–Nonius, 1993); cell refinement: *CAD-4-PC*; data reduction: *MolEN* (Fair, 1990); program(s) used to solve structures: *SIR92* (Altomare *et al.*, 1992); program(s) used to refine structures: *SHELXL93* (Sheldrick, 1993); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL93*.

Financial support from Fonds der Chemischen Industrie and Deutsche Forschungsgemeinschaft is gratefully acknowledged.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1027). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1992). *SIR92. Program for Crystal Structure Solution*. University of Bari, Italy.
- Alyoubi, O. A., Greenslade, D. J., Foster, M. J. & Preetz, W. (1990). *J. Chem. Soc. Dalton Trans.* pp. 381–383.
- Brüdgam, I. & Hartl, H. (1986). *Acta Cryst.* **C42**, 866–868.
- Bruhn, C. & Preetz, W. (1994a). *Acta Cryst.* **C50**, 1555–1557.
- Bruhn, C. & Preetz, W. (1994b). *Acta Cryst.* **C50**, 1687–1690.
- Bruhn, C. & Preetz, W. (1995a). *Acta Cryst.* **C51**, 865–867.
- Bruhn, C. & Preetz, W. (1995b). *Acta Cryst.* **C51**, 1112–1116.
- Bruhn, C., Drews, H. H., Meynhardt, B. & Preetz, W. (1995). *Z. Anorg. Allg. Chem.* **621**, 373–380.
- Enraf–Nonius (1993). *CAD-4-PC*. Version 1.5e. Enraf–Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
- Groth, Th. & Preetz, W. (1987). *Z. Anorg. Allg. Chem.* **548**, 76–88.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

- Keller, H.-L. & Homborg, H. (1976). *Z. Anorg. Allg. Chem.* **422**, 261–265.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Parzich, E., Peters, G. & Preetz, W. (1993). *Z. Naturforsch. Teil B*, **48**, 1169–1174.
- Preetz, W., Ruf, D. & Tensfeldt, D. (1984). *Z. Naturforsch. Teil B*, **39**, 1100–1109.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1996). **C52**, 325–328

Sodium 3 α ,7 α ,12 α -Trihydroxy-5 β -cholan-24-oate Ethanolate (Sodium Cholate Ethanolate)

MARK C. WAHLE, JOSEPH G. STOWELL AND STEPHEN R. BYRN

School of Pharmacy and Pharmacal Sciences, Purdue University, West Lafayette, Indiana 47907-1333, USA

(Received 2 November 1994; accepted 5 July 1995)

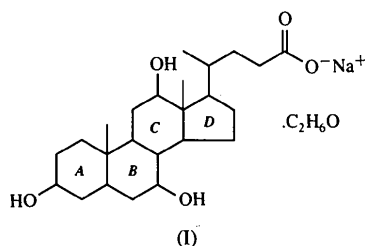
Abstract

The crystal structure of sodium cholate ethanolate, $\text{Na}^+\cdot\text{C}_{24}\text{H}_{39}\text{O}_5^-\cdot\text{C}_2\text{H}_6\text{O}$, recrystallized from absolute ethanol by slow evaporation, has been determined. This structure is the first report of a sodium cholate structure solely solvated by an organic molecule.

Comment

Our research into methyl rotation in the solid state led us to a number of compounds containing multiple methyl groups, including the two bile acids, cholic acid and deoxycholic acid. While most previous structural research has focused on deoxycholic acid (3 α ,12 α -dihydroxy-5 β -cholan-24-oic acid) and its many inclusion compounds (Giglio, 1984), more recent research has examined the many possible solvated structures of cholic acid (3 α ,7 α ,12 α -trihydroxy-5 β -cholan-24-oic acid). Many cholic acid structures have been solved, including cholic acid complexed with water (Lessinger, 1982; Lessinger & Low, 1993), acetophenone (Miki *et al.*, 1988), valerolactone (Miki, Kasai, Shibakami, Takemoto & Miyata, 1991), benzene (Nakano, Sada & Miyata, 1994), aniline (Caira, Nassimbeni & Scott, 1993; Shibakami & Sekiya, 1994), 3-fluoroaniline (Shibakami & Sekiya, 1994), nitrobenzene (Caira, Nassimbeni & Scott, 1993), acetonitrile (Scott, 1995), and a number of

alcohols (Johnson & Schaefer, 1972; Jones & Nassimbeni, 1990), aliphatic esters (Caira, Nassimbeni & Scott, 1994a) and ketones (Caira, Nassimbeni & Scott, 1994b). Recently, the structure of cholic acid without any guest molecules has been obtained (Miki *et al.*, 1990). Less attention, however, has been paid to cholic acid salt complexes. Until now, only three cholic acid salt structures have been solved, one with calcium (Hogan, Ealick, Bugg & Barnes, 1984), the other two with sodium (Norton & Haner, 1965; Cobblestick & Einstein, 1980). One of the sodium salts, (I), is reported to be anhydrous, while the other sodium salt and the calcium salt are both hydrated structures. In a search of the Cambridge Structural Database (Allen *et al.*, 1979), we found 1503 steroid structures. Of these structures, only 13 were salt complexes, of which only five involved sodium. In those five sodium salts, four were hydrates, and the fifth was an acetone/water solvate. Here we report the first cholic acid salt structure, (I), solvated solely with an organic molecule.



An ORTEPII (Johnson, 1976) diagram of the cholate salt structure is presented in Fig. 1. The rings have a geometry similar to the other cholic acid structures reported so far, with a *cis* ring juncture for the A/B rings and *trans* ring junctures for the B/C and C/D rings. When the monohydrated and ethanol-solvated sodium salts are overlaid using a least-squares fit, the B and C rings are quite similar, whereas the A and D rings deviate slightly from each other. The lack of similarity in the D rings produces a different tail orientation in both structures. Likewise, the cell dimensions for sodium

cholate ethanolate are quite different from those reported for sodium cholate monohydrate. The packing patterns are also noticeably different with the monohydrated salt adopting a layered-packing pattern, while the ethanol-solvated salt packs in a herringbone pattern. Fig. 2 presents the sodium cholate ethanolate packing diagram drawn using QUANTA4.0 (Molecular Simulations Inc., 1994).

We found that the methyl C atom of ethanol was disordered over two different positions, C92 and C93. Similar problems with disordered alcohol solvents were also encountered in the cholic acid structures (Jones & Nassimbeni, 1990). This disorder, we suspect, is due to the small size of the ethanol molecule when compared to the larger size of the cavity in which it sits. Hydrogen bonding between the ethanol molecule, the hydroxy O7 and O12 atoms and the carboxylate O24 atom holds the ethanol in this cavity, while additional hydrogen bonding occurs between the hydroxy O3 atom and the carboxylate O24 atom in the tail portion of the molecule (Table 2). All of the O...O distances fall within the normally accepted range for hydrogen bonds. The disorder in the ethanol molecule does not affect its ability to participate in hydrogen bonding with the cholate salt.

The Na⁺ atom has trigonal bipyramidal coordination involving five different O atoms from four different cholate molecules. These distances range from 2.266 (4) to 2.594 (4) Å (Table 3). Two of the coordinated O atoms are from hydroxy groups, while the other three are carboxylate O atoms. While the geometry around the Na⁺ atom is not octahedral, a pentacoordinated Na⁺ atom was also found earlier in the structure of sodium cholate monohydrate (Cobblestick & Einstein, 1980). The O25 carboxylate atom is not found to participate in hydrogen bonding since it coordinates to two different Na⁺ ions, whereas the O24 carboxylate atom only coordinates one Na⁺ ion while hydrogen bonding with the hydroxy O3 atoms. An anhydrous sodium salt structure (Norton & Haner, 1965) has been reported previously but no atomic coordinate data were published.

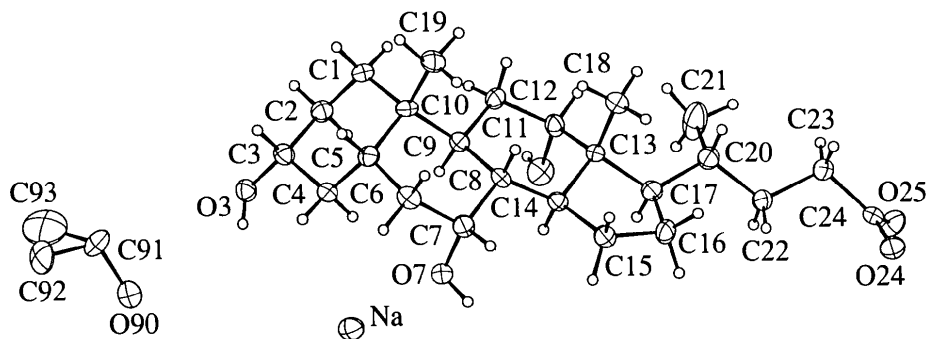


Fig. 1. ORTEPII (Johnson, 1976) diagram showing 50% probability displacement ellipsoids for non-H atoms. Ellipsoids for H atoms are drawn as spheres with $U = 1.0 \text{ \AA}^2$. The Na⁺ ion and disordered ethanol molecule are also included.

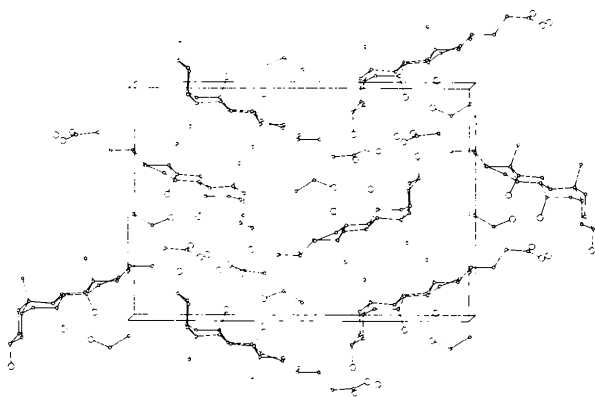


Fig. 2. Packing diagram for sodium cholate ethanolate viewed down the *a* axis.

Experimental

The title compound was prepared by recrystallization of sodium cholate (Sigma Chemical Co., St Louis, MO) by slow evaporation from absolute ethanol (Midwest Grain Products Co., Weston, MO).

Crystal data



$M_r = 476.63$

Orthorhombic

$P2_12_12_1$

$a = 7.7383$ (17) Å

$b = 15.098$ (2) Å

$c = 22.515$ (5) Å

$V = 2630.5$ (15) Å³

$Z = 4$

$D_x = 1.203$ Mg m⁻³

Cu $K\alpha$ radiation

$\lambda = 1.54184$ Å

Cell parameters from 23 reflections

$\theta = 20\text{--}43^\circ$

$\mu = 0.780$ mm⁻¹

$T = 295$ K

Plate

$0.30 \times 0.13 \times 0.06$ mm

Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer

ω - 2θ scans

Absorption correction: none

2738 measured reflections

2738 independent reflections

2737 observed reflections

$[I > 2\sigma(I)]$

$\theta_{\text{max}} = 68.13^\circ$

$h = 0 \rightarrow 9$

$k = 0 \rightarrow 18$

$l = 0 \rightarrow 27$

3 standard reflections

frequency: 83 min

intensity decay: none

Refinement

Refinement on F^2

$R(F) = 0.047$

$wR(F^2) = 0.109$

$S = 1.085$

2737 reflections

320 parameters

HO3, HO7, HO90, H5

refined isotropically, other

H atoms not refined ($U =$

$1.3U_{\text{eq}}$ of parent atom)

$w = 1/[\sigma^2(F) + (0.0560P)^2 + 0.5148P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.039$

$\Delta\rho_{\text{max}} = 0.24$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.23$ e Å⁻³

Extinction correction: none

Atomic scattering factors

from *International Tables*

for *Crystallography* (1992,

Vol. C, Tables 4.2.6.8 and

6.1.1.4)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

U_{iso} for H atoms; $U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$ for all other atoms.

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}/U_{\text{iso}}$
Na	0.7017 (3)	0.77502 (14)	0.52160 (8)	0.0440 (6)
O3	1.0495 (5)	0.7926 (2)	0.4010 (2)	0.0403 (10)
O7	0.6619 (5)	0.9397 (2)	0.5459 (2)	0.0403 (10)
O12	1.1355 (5)	1.0256 (2)	0.6455 (2)	0.0503 (10)
O24	0.7630 (4)	1.3002 (2)	0.9187 (2)	0.0407 (10)
O25	1.0170 (5)	1.2588 (3)	0.95257 (14)	0.0477 (12)
O90	0.9310 (5)	0.5834 (2)	0.3604 (2)	0.0473 (10)
C1	1.1388 (7)	1.0338 (3)	0.4235 (2)	0.0407 (15)
C2	1.1701 (7)	0.9345 (3)	0.4263 (2)	0.0390 (15)
C3	1.0198 (7)	0.8864 (3)	0.3977 (2)	0.0353 (15)
C4	0.8538 (6)	0.9119 (3)	0.4287 (2)	0.0340 (15)
C5	0.8172 (6)	1.0110 (3)	0.4266 (2)	0.0337 (15)
C6	0.6441 (7)	1.0314 (4)	0.4568 (2)	0.0383 (15)
C7	0.6487 (6)	1.0300 (3)	0.5249 (2)	0.0360 (15)
C8	0.7957 (6)	1.0882 (3)	0.5478 (2)	0.0300 (15)
C9	0.9702 (6)	1.0577 (3)	0.5215 (2)	0.0293 (10)
C10	0.9686 (7)	1.0665 (3)	0.4520 (2)	0.0330 (15)
C11	1.1277 (7)	1.1027 (4)	0.5501 (2)	0.0377 (15)
C12	1.1213 (6)	1.1106 (4)	0.6184 (2)	0.0343 (15)
C13	0.9511 (6)	1.1529 (3)	0.6385 (2)	0.0293 (15)
C14	0.8035 (6)	1.0938 (3)	0.6151 (2)	0.0300 (15)
C15	0.6427 (6)	1.1274 (3)	0.6479 (2)	0.0363 (15)
C16	0.7120 (6)	1.1608 (4)	0.7083 (2)	0.0423 (15)
C17	0.9131 (6)	1.1507 (4)	0.7068 (2)	0.0353 (15)
C18	0.9418 (8)	1.2481 (3)	0.6154 (2)	0.0427 (15)
C19	0.9453 (9)	1.1646 (3)	0.4325 (2)	0.0480 (17)
C20	1.0088 (7)	1.2161 (4)	0.7470 (2)	0.0417 (15)
C21	1.2051 (7)	1.2020 (6)	0.7473 (3)	0.087 (3)
C22	0.9395 (7)	1.2142 (4)	0.8106 (2)	0.0407 (15)
C23	0.9978 (7)	1.2921 (3)	0.8491 (2)	0.0417 (15)
C24	0.9235 (6)	1.2838 (3)	0.9117 (2)	0.0343 (15)
C91	0.9789 (12)	0.6184 (5)	0.3045 (3)	0.088 (3)
C92	0.908 (2)	0.5763 (10)	0.2558 (5)	0.067 (5)
C93	1.063 (3)	0.5751 (14)	0.2677 (9)	0.124 (9)
HO3	0.951 (7)	0.769 (4)	0.404 (3)	0.05200
H5	0.8050 (6)	1.0272 (3)	0.3847 (2)	0.04400
HO7	0.598 (8)	0.937 (4)	0.575 (2)	0.05200
HO90	0.855 (6)	0.614 (2)	0.3750 (12)	0.06100

Table 2. Hydrogen-bonding data (Å, °)

O90...O12 ⁱ	2.820 (5)	O90...O24 ⁱⁱⁱ	2.658 (5)
O90...O7 ⁱⁱ	2.787 (6)	O3...O24 ⁱⁱⁱ	2.823 (5)
O3—HO3...O24 ⁱⁱⁱ	172 (5)	C91—O90...HO7 ⁱⁱ	122.8 (17)
O90—HO90...O24 ⁱⁱⁱ	163 (4)	C91—O90...HO12 ⁱ	114.3 (16)
O7 ⁱⁱ —HO7 ⁱⁱ ...O90	172 (6)	C24 ⁱⁱⁱ —O24 ⁱⁱⁱ ...HO3	133.7 (16)
O12 ⁱ —HO12 ⁱ ...O90	156 (2)	C24 ⁱⁱⁱ —O24 ⁱⁱⁱ ...HO90	123.1 (12)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$; (ii) $\frac{1}{2} + x, \frac{3}{2} - y, 1 - z$; (iii) $\frac{3}{2} - x, 2 - y, z - \frac{1}{2}$.

Table 3. Sodium-ion coordination geometry (Å, °)

Na...O3 ⁱ	2.338 (4)	Na...O25 ⁱⁱ	2.354 (4)
Na...O7	2.565 (4)	Na...O25 ⁱⁱⁱ	2.266 (4)
Na...O24 ⁱⁱⁱ	2.594 (4)		
O3 ⁱ ...Na...O7	101.75 (14)	O7...Na...O25 ⁱⁱ	105.4 (2)
O3 ⁱ ...Na...O24 ⁱⁱⁱ	121.85 (14)	O7...Na...O25 ⁱⁱⁱ	99.5 (2)
O3 ⁱ ...Na...O25 ⁱⁱ	92.02 (15)	O24 ⁱⁱⁱ ...Na...O25 ⁱⁱ	52.50 (12)
O3 ⁱ ...Na...O25 ⁱⁱⁱ	104.20 (15)	O24 ⁱⁱⁱ ...Na...O25 ⁱⁱⁱ	94.63 (14)
O7...Na...O24 ⁱⁱⁱ	128.87 (14)	O25 ⁱⁱ ...Na...O25 ⁱⁱⁱ	146.78 (13)

Symmetry codes: (i) $x - \frac{1}{2}, \frac{3}{2} - y, 1 - z$; (ii) $\frac{3}{2} - x, 2 - y, z - \frac{1}{2}$; (iii) $2 - x, y - \frac{1}{2}, \frac{3}{2} - z$.

The methyl carbon in the ethanol solvent molecule had a site-occupation factor of 0.5. The disorder in the solvent molecule contributed to a large value for R .

Data collection: *CAD-4 Software* (Enraf–Nonius, 1977). 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: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* Johnson (1976); *QUANTA4.0* (Molecular Simulations Inc., 1994). Software used to prepare material for publication: *MolEN*.

The authors would like to thank Phillip Fanwick for his assistance with the refinement of the structure. Funding was provided by the Byrn/Zografi Program for the Study of the Effects of Water on Molecular Mobility of Crystalline and Amorphous Solids.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1178). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Allen, F. H., Bellard, S., Brice, M. D., Cartwright, B. A., Doubleday, A., Higgs, H., Hummelink, T., Hummelink-Peters, B. G., Kennard, O., Motherwell, W. D. S., Rodgers, J. R. & Watson, D. G. (1979). *Acta Cryst.* **B35**, 2331–2339.
- Caira, M. R., Nassimbeni, L. R. & Scott, J. L. (1993). *J. Chem. Soc. Chem. Commun.* pp. 612–614.
- Caira, M. R., Nassimbeni, L. R. & Scott, J. L. (1994a). *J. Chem. Soc. Perkin Trans. 2*, pp. 623–628.
- Caira, M. R., Nassimbeni, L. R. & Scott, J. L. (1994b). *J. Chem. Crystallogr.* **24**, 783–791.
- Cobbleddick, R. E. & Einstein, F. W. B. (1980). *Acta Cryst.* **B36**, 287–292.
- Enraf-Nonius (1977). *CAD-4 Operations Manual*. Enraf-Nonius, Delft, The Netherlands.
- Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf-Nonius, Delft, The Netherlands.
- Giglio, E. (1984). *Inclusion Compounds*, Vol. 2, edited by J. L. Atwood, J. E. D. Davies & D. D. MacNicol, pp. 207–229. London: Academic Press.
- Hogan, A., Ealick, S. E., Bugg, C. E. & Barnes, S. (1984). *J. Lipid Res.* **25**, 791–798.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Johnson, P. L. & Schaefer, J. P. (1972). *Acta Cryst.* **B28**, 3083–3088.
- Jones, E. L. & Nassimbeni, L. R. (1990). *Acta Cryst.* **B46**, 399–405.
- Lessinger, L. (1982). *Cryst. Struct. Commun.* **11**, 1787–1792.
- Lessinger, L. & Low, B. W. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 85–99.
- Miki, K., Kasai, N., Shibakami, M., Chirachanchai, S., Takemoto, K. & Miyata, M. (1990). *Acta Cryst.* **C46**, 2442–2445.
- Miki, K., Kasai, N., Shibakami, M., Takemoto, K. & Miyata, M. (1991). *J. Chem. Soc. Chem. Commun.* pp. 1757–1759.
- Miki, K., Masui, A., Kasai, N., Miyata, M., Shibakami, M. & Takemoto, K. (1988). *J. Am. Chem. Soc.* **110**, 6594–6596.
- Molecular Simulations Inc. (1994). *QUANTA4.0*. Molecular Simulations Inc., Burlington, USA.
- Nakano, K., Sada, K. & Miyata, M. (1994). *Chem. Lett.* pp. 137–140.
- Norton, D. A. & Haner, B. (1965). *Acta Cryst.* **19**, 477–478.
- Scott, J. L. (1995). *J. Chem. Soc. Perkin Trans. 2*, pp. 495–502.
- Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
- Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
- Shibakami, M. & Sekiya, A. (1994). *J. Chem. Soc. Chem. Commun.* pp. 429–430.
- Acta Cryst.* (1996). **C52**, 328–330

trans-Dichlorobis[tris(pentafluorophenyl)-phosphane]palladium(II)

BIRGIT BERTSCH-FRANK^a AND WALTER FRANK^b

^aDegussa AG, Werk Rheinfelden, Untere Kanalstraße 3, 79618 Rheinfelden, Germany, and ^bFachbereich Chemie, Universität Kaiserslautern, Erwin-Schrödinger-Straße, 67663 Kaiserslautern, Germany

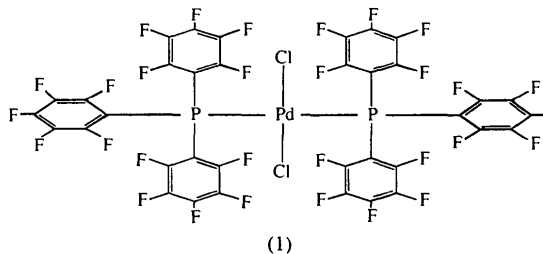
(Received 23 May 1995; accepted 10 August 1995)

Abstract

The crystal structure of the title compound, *trans*-[{(C₆F₅)₃P}₂PdCl₂], was determined from single-crystal X-ray diffraction data. Owing to the steric requirements of the bulky phosphane ligands, the centrosymmetric molecule shows a considerable angle distortion at the square-planar coordinated Pd atom [Cl—Pd—P 95.23 (4)°]. Pd—Cl and Pd—P distances are 2.291 (1) and 2.305 (1) Å, respectively. The metal–phosphorus distance is significantly shorter than in *trans*-dichlorobis(triphenylphosphane)palladium(II).

Comment

trans-[{(C₆F₅)₃P}₂PdCl₂], (1), is a catalyst for the synthesis of hydrogen peroxide from the elements (Dyer & Moseley, 1981). In the course of a study of the catalytic behavior of the compound, we were interested in comparing its structure with that of the corresponding triphenylphosphane derivative, *trans*-[{(C₆H₅)₃P}₂PdCl₂], (2) [(2) (Ferguson, McCrindle, McAlees & Masood, 1982); (2).0.5*p*-C₆H₄Cl₂ (Kitano, Kinoshita, Nakamura & Ashida, 1983)].



The crystals of (1) consist of discrete centrosymmetric molecules (Fig. 1) separated by van der Waals distances. The packing is like that of the isotopic platinum compound *trans*-[{(C₆H₅)₃P}₂PtCl₂] (Schaefer, Lyon, Labinger & Bercaw, 1992). The Pd—Cl distance lies in the middle of the range of values collected by Ferguson *et al.* (1982) for *trans*-dichlorobis(phosphane)palladium complexes [2.267–2.326 Å], whereas the Pd—P distance is slightly shorter than the shortest value given