Bermejo, Gayoso, Molleda, Hiller & Strähle, 1988; Castiñeiras, Hiller, Strähle, Carballo, Bermejo & Gayoso, 1990a,b).

References

BIRKER, P. J. M. W. L., HELDER, J., HENKEL, G., KREBS, B. & REEDIJK, J. (1982). *Inorg. Chem.* **21**, 357–363.
CASTIÑEIRAS, A., CARBALLO, R., BERMEJO, M. R., GAYOSO, M., MOLLEDA, C., HILLER, W. & STRÄHLE, J. (1988). *Z. Kristallogr.* **185**, 381.
CASTIÑEIRAS, A., HILLER, W., STRÄHLE, J., CARBALLO, R., BERMEJO, M. R. & GAYOSO, M. (1990a). *An. Quím.* In the press.
CASTIÑEIRAS, A., HILLER, W., STRÄHLE, J., CARBALLO, R., BERMEJO, M. R. & GAYOSO, M. (1990b). *Z. Naturforsch.* In the press.

CASTIÑEIRAS, A., HILLER, W., STRÄHLE, J., PAREDES, M. V. & SORDO J. (1985). *Acta Cryst.* C41, 41–43.
FRENZ, B. A. (1978). *The Enraf–Nonius CAD-4 SDP. A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution.* In *Computing in Crystallography*, edited by H. SCHENK, R. OLTHOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI. Delft Univ. Press.
International Tables for X-ray Crystallography (1974). Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
SHELDRICK, G. M. (1986). *SHELXS86. Program for the solution of crystal structures from diffraction data.* Univ. of Göttingen, Federal Republic of Germany.
SMITS, J. M. M., JANSSEN, R., BEURSKENS, P. T., VAN RIJN, J. & REEDIJK, J. (1987). *Polyhedron,* **6**, 1843–1847.
WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.
ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

Structure of Bis(1,1,1,5,5,5-hexafluoro-2,4-pentanedionato)-(2,5,8,11,14-pentaoxapentadecane)barium(II)

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Abstract. $[Ba(C_5HF_6O_2)_2(C_{10}H_{22}O_5)]$, $M_r = 773.71$, triclinic, $P\bar{1}$, $a = 9.506\,(1)$, $b = 10.969\,(1)$, $c = 15.085\,(1)$ Å, $\alpha = 84.10\,(1)$, $\beta = 88.10\,(1)$, $\gamma = 65.31\,(1)°$, $V = 1421.5\,(3)$ Å^3, $Z = 2$, $D_x = 1.808$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 15.1$ cm^{-1}, $F(000) = 760$, $T = 100$ K, $R = 0.020$ for 6391 unique observed diffractometer data $[I \geq 2.5\sigma(I)]$. The barium cation is coordinated by all nine O atoms of the three ligands with Ba—O distances ranging from 2.683 to 2.893 Å. The 2,5,8,11,14-pentaoxapentadecane ligand is wrapped around the Ba in a plane. One hexafluoroacetyl-acetonate anion is found on top of this plane and one below. Both anions have the ZZ conformation. The absence of short intermolecular interactions explains why the compound sublimes easily.

Experimental. Data were collected at 100 K on an Enraf–Nonius CAD-4F diffractometer for a transparent, colorless crystal (0.6 × 0.5 × 0.5 mm), mounted on a glass fiber. The cell parameters were

calculated by least-squares from the SET4 setting angles of 25 reflections with $16 \leq \theta \leq 18°$. 6755 reflections were scanned $[h \; -12{:}12, \; k \; -14{:}14, \; l \; -19{:}0; \; \theta \leq 27.5°; \; \omega/2\theta$-scan mode; $\Delta\omega = (0.60 + 0.35 \tan\theta)°$; Zr-filtered Mo $K\alpha$ radiation]. Three reference reflections ($04\bar{2}$, 440, $30\bar{4}$) showed no decay during 64 h of X-ray exposure time. The data were corrected for Lp and for absorption [*ABSORB* (Spek, 1983), correction factors 1.75–2.15] resulting in the unique set of 6391 reflections $[I > 2.5\sigma(I)]$ used in the structure determination. $\sigma^2(I) = \sigma^2_{cs}(I) + (pI)^2$ (McCandlish, Stout & Andrews, 1975) with $p = 0.015$. All non-H atoms were found by direct methods followed by peak optimization (*SHELXS*86; Sheldrick, 1986) in space group $P1$. The correct space group, $P\bar{1}$, was inferred during refinement. The structure was refined on F by full-matrix least-squares procedures using anisotropic thermal parameters for all non-H atoms (*SHELX*76; Sheldrick, 1976). All H atoms were introduced at calculated positions (C—H = 0.98 Å) and refined in the riding mode on their carrier atoms with three isotropic thermal parameters. Convergence was

Table 1. *Final coordinates and equivalent isotropic thermal parameters with their e.s.d.'s in parentheses*

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

	x	y	z	$U_{eq}(Å^2)$
Ba	0.04138 (1)	0.00565 (1)	0.23763 (1)	0.0149 (1)
F(1)	0.3409 (2)	0.3217 (2)	0.2120 (1)	0.0514 (6)
F(2)	0.2180 (2)	0.4705 (1)	0.1067 (1)	0.0466 (5)
F(3)	0.3603 (2)	0.2675 (2)	0.0781 (1)	0.0644 (7)
F(4)	-0.3082 (2)	0.6060 (1)	0.1189 (1)	0.0449 (5)
F(5)	-0.4000 (2)	0.4932 (1)	0.2080 (1)	0.0436 (4)
F(6)	-0.3867 (2)	0.4689 (1)	0.0687 (1)	0.0409 (4)
F(7)	0.5441 (2)	-0.2461 (2)	0.4503 (1)	0.0470 (5)
F(8)	0.6317 (1)	-0.3016 (1)	0.32090 (9)	0.0383 (4)
F(9)	0.6145 (2)	-0.4497 (1)	0.4185 (1)	0.0396 (4)
F(10)	0.1965 (2)	-0.5621 (1)	0.4074 (1)	0.0497 (5)
F(11)	0.0505 (2)	-0.4713 (2)	0.2924 (1)	0.0447 (5)
F(12)	-0.0318 (2)	-0.4057 (1)	0.4203 (1)	0.0432 (5)
O(1)	0.0767 (2)	0.1300 (2)	0.3839 (1)	0.0317 (5)
O(2)	-0.2002 (2)	0.1143 (2)	0.3645 (1)	0.0299 (5)
O(3)	-0.2471 (2)	-0.0125 (2)	0.2192 (1)	0.0276 (4)
O(4)	-0.0008 (2)	-0.1572 (1)	0.11418 (9)	0.0234 (4)
O(5)	0.2458 (2)	-0.0908 (1)	0.08952 (9)	0.0216 (4)
O(6)	0.1716 (2)	0.1764 (1)	0.1783 (1)	0.0242 (4)
O(7)	-0.1572 (2)	0.2572 (1)	0.17271 (9)	0.0220 (4)
O(8)	0.3170 (2)	-0.1616 (1)	0.3145 (1)	0.0240 (4)
O(9)	0.0534 (2)	-0.2244 (1)	0.3249 (1)	0.0250 (4)
C(1)	0.2249 (3)	0.1020 (3)	0.4206 (2)	0.0396 (8)
C(2)	-0.0448 (3)	0.1892 (2)	0.4446 (2)	0.0338 (7)
C(3)	-0.1959 (3)	0.2299 (2)	0.3971 (2)	0.0343 (7)
C(4)	-0.3498 (3)	0.1432 (3)	0.3278 (2)	0.0376 (7)
C(5)	-0.3455 (3)	0.0182 (3)	0.2957 (2)	0.0382 (8)
C(6)	-0.2318 (2)	-0.1352 (2)	0.1878 (2)	0.0296 (6)
C(7)	-0.1514 (3)	-0.1500 (2)	0.0996 (2)	0.0299 (6)
C(8)	0.0938 (3)	-0.1937 (2)	0.0367 (1)	0.0279 (6)
C(9)	0.2539 (3)	-0.2139 (2)	0.0601 (2)	0.0293 (6)
C(10)	0.3975 (2)	-0.0971 (2)	0.1018 (2)	0.0280 (6)
C(11)	0.2614 (3)	0.3413 (2)	0.1367 (2)	0.0317 (7)
C(12)	0.1274 (2)	0.2978 (2)	0.1529 (1)	0.0218 (5)
C(13)	-0.0220 (2)	0.3964 (2)	0.1376 (1)	0.0226 (6)
C(14)	-0.1512 (2)	0.3668 (2)	0.1507 (1)	0.0210 (5)
C(15)	-0.3123 (2)	0.4850 (2)	0.1359 (1)	0.0270 (6)
C(16)	0.5432 (2)	-0.3207 (2)	0.3855 (1)	0.0252 (6)
C(17)	0.3768 (2)	-0.2784 (2)	0.3526 (1)	0.0203 (5)
C(18)	0.3112 (2)	-0.3707 (2)	0.3703 (1)	0.0224 (5)
C(19)	0.1539 (2)	-0.3356 (2)	0.3525 (1)	0.0223 (6)
C(20)	0.0954 (3)	-0.4473 (2)	0.3692 (2)	0.0299 (7)

Table 2. *Bond distances (Å) and angles (°)*

Ba—O(1)	2.809 (2)	O(2)—C(4)	1.439 (4)
Ba—O(2)	2.872 (2)	O(3)—C(5)	1.437 (3)
Ba—O(3)	2.855 (2)	O(3)—C(6)	1.422 (3)
Ba—O(4)	2.855 (2)	O(4)—C(7)	1.424 (3)
Ba—O(5)	2.893 (1)	O(4)—C(8)	1.435 (3)
Ba—O(6)	2.713 (2)	O(5)—C(9)	1.435 (3)
Ba—O(7)	2.711 (1)	O(5)—C(10)	1.432 (3)
Ba—O(8)	2.706 (2)	O(6)—C(12)	1.238 (2)
Ba—O(9)	2.683 (2)	O(7)—C(14)	1.238 (2)
F(1)—C(11)	1.334 (3)	O(8)—C(17)	1.248 (2)
F(2)—C(11)	1.332 (2)	O(9)—C(19)	1.234 (2)
F(3)—C(11)	1.333 (3)	C(2)—C(3)	1.498 (4)
F(4)—C(15)	1.342 (2)	C(4)—C(5)	1.485 (4)
F(5)—C(15)	1.335 (3)	C(6)—C(7)	1.502 (3)
F(6)—C(15)	1.323 (3)	C(8)—C(9)	1.492 (4)
F(7)—C(16)	1.340 (3)	C(11)—C(12)	1.541 (4)
F(8)—C(16)	1.324 (3)	C(12)—C(13)	1.388 (3)
F(9)—C(16)	1.337 (2)	C(13)—C(14)	1.398 (3)
F(10)—C(20)	1.311 (3)	C(14)—C(15)	1.543 (3)
F(11)—C(20)	1.335 (3)	C(16)—C(17)	1.534 (3)
F(12)—C(20)	1.347 (3)	C(17)—C(18)	1.395 (3)
O(1)—C(1)	1.429 (4)	C(18)—C(19)	1.407 (3)
O(1)—C(2)	1.421 (3)	C(19)—C(20)	1.540 (3)
O(2)—C(3)	1.422 (3)		

Ba—O(1)—C(1)	122.5 (2)	O(6)—C(12)—C(11)	113.2 (2)
Ba—O(1)—C(2)	122.6 (2)	O(6)—C(12)—C(13)	129.5 (2)
C(1)—O(1)—C(2)	111.9 (2)	C(11)—C(12)—C(13)	117.3 (2)
Ba—O(2)—C(3)	110.6 (1)	C(12)—C(13)—C(14)	121.6 (2)
Ba—O(2)—C(4)	111.5 (1)	O(7)—C(14)—C(13)	129.4 (2)
C(3)—O(2)—C(4)	111.7 (2)	O(7)—C(14)—C(15)	113.2 (2)
Ba—O(3)—C(5)	115.1 (2)	C(13)—C(14)—C(15)	117.4 (2)
Ba—O(3)—C(6)	113.5 (1)	F(4)—C(15)—F(5)	106.2 (2)
C(5)—O(3)—C(6)	111.4 (2)	F(4)—C(15)—F(6)	107.2 (2)
Ba—O(4)—C(7)	119.7 (1)	F(4)—C(15)—C(14)	113.9 (2)
Ba—O(4)—C(8)	122.1 (1)	F(5)—C(15)—F(6)	107.2 (2)
C(7)—O(4)—C(8)	111.3 (2)	F(5)—C(15)—C(14)	110.9 (2)
Ba—O(5)—C(9)	115.2 (1)	F(6)—C(15)—C(14)	111.1 (2)
Ba—O(5)—C(10)	113.8 (1)	F(7)—C(16)—F(8)	107.0 (2)
C(9)—O(5)—C(10)	111.0 (2)	F(7)—C(16)—F(9)	106.8 (2)
Ba—O(6)—C(12)	137.5 (1)	F(7)—C(16)—C(17)	110.0 (2)
Ba—O(7)—C(14)	137.1 (1)	F(8)—C(16)—F(9)	106.6 (2)
Ba—O(8)—C(17)	137.2 (1)	F(8)—C(16)—C(17)	111.6 (2)
Ba—O(9)—C(19)	137.5 (1)	F(9)—C(16)—C(17)	114.5 (2)
O(1)—C(2)—C(3)	108.2 (2)	O(8)—C(17)—C(16)	113.7 (2)
O(2)—C(3)—C(2)	108.9 (2)	O(8)—C(17)—C(18)	128.7 (2)
O(2)—C(4)—C(5)	108.6 (2)	C(16)—C(17)—C(18)	117.6 (2)
O(3)—C(5)—C(4)	108.6 (2)	C(17)—C(18)—C(19)	121.8 (2)
O(3)—C(6)—C(7)	109.0 (2)	O(9)—C(19)—C(18)	128.2 (2)
O(4)—C(7)—C(6)	107.4 (2)	O(9)—C(19)—C(20)	114.2 (2)
O(4)—C(8)—C(9)	108.3 (2)	C(18)—C(19)—C(20)	117.6 (2)
O(5)—C(9)—C(8)	108.1 (2)	F(10)—C(20)—F(11)	107.9 (2)
F(1)—C(11)—F(2)	106.2 (2)	F(10)—C(20)—F(12)	107.9 (2)
F(1)—C(11)—F(3)	107.1 (2)	F(10)—C(20)—C(19)	114.8 (2)
F(1)—C(11)—C(12)	110.6 (2)	F(11)—C(20)—F(12)	105.7 (2)
F(2)—C(11)—F(3)	107.4 (2)	F(11)—C(20)—C(19)	110.1 (2)
F(2)—C(11)—C(12)	114.8 (2)	F(12)—C(20)—C(19)	110.0 (2)
F(3)—C(11)—C(12)	110.4 (2)		

reached at $R = 0.020$ [$wR = 0.032$, $w = 1.0/\sigma^2(F)$, $S = 0.82$, 383 parameters; $(\Delta/\sigma)_{max} = 0.2$, $\langle\Delta/\sigma\rangle = 0.04$]. No residual density outside the range -0.39–0.81 e Å^{-3}. Scattering factors from Cromer & Mann (1968), anomalous-dispersion corrections from Cromer & Liberman (1970). The program package *EUCLID* (Spek, 1982) was used for geometrical calculations and illustrations. All calculations were carried out on a microVAX II cluster. The final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1.* Bond distances and angles are given in Table 2. Fig. 1 shows the molecular structure with labeling scheme.

Related literature. Crystal structures of related compounds have been reported. 2,5,8,11,14-Pentaoxapentadecane–barium thiocyanate dihydrate (Wei,

* Lists of anisotropic thermal parameters, H-atom positions, bond angles and distances involving H atoms, torsion angles, and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52907 (47 pp.). Copies may be obtained through the Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
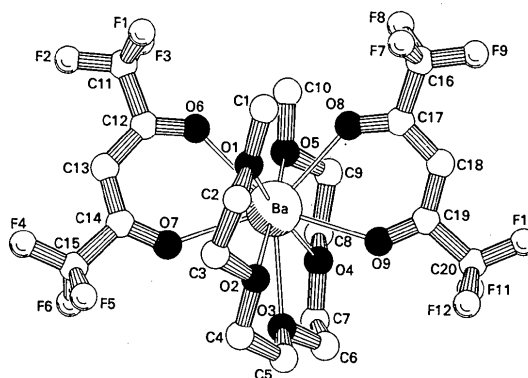

Fig. 1. *PLUTON* drawing of the molecular structure with adopted labeling.

Tinant, Declercq, van Meerssche & Dale, 1987) and *cis*-bis(acetylacetonato)diaquacalcium monohydrate (Sahbari & Olmstead, 1983). Details of the chemistry will be published elsewhere (Timmer, Meinema, van der Sluis & Spek, 1990).

References

CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

CROMER, D. T. & MANN, J. B. (1968). *Acta Cryst.* A24, 321–324.
McCANDLISH, L. E., STOUT, G. H. & ANDREWS, L. C. (1975). *Acta Cryst.* A31, 245–249.
SAHBARI, J. J. & OLMSTEAD, M. M. (1983). *Acta Cryst.* C39, 208–211.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELX*86. Program for the solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.
SPEK, A. L. (1982). *The EUCLID Package.* In *Computational Crystallography*, edited by D. SAYRE. Oxford: Clarendon Press.
SPEK, A. L. (1983). *ABSORB.* Program for absorption correction. ECM Abstracts Book, p. 283. Univ. of Utrecht, The Netherlands.
TIMMER, K., MEINEMA, H. A., VAN DER SLUIS, P. & SPEK, A. L. (1990). In preparation.
WEI, Y. Y., TINANT, B., DECLERCQ, J.-P., VAN MEERSSCHE, M. & DALE, J. (1987). *Acta Cryst.* C43, 1076–1080.

Structure of Bromo(4-iodo-1,2,3,4-tetraphenyl-1,3-butadienyl)diphenyltin(IV)

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Abstract. [SnBr(C$_6$H$_5$)$_2$(C$_{28}$H$_{20}$I)], $M_r = 836.19$, triclinic, $P\bar{1}$, $a = 10.336$ (4), $b = 10.582$ (5), $c = 16.615$ (8) Å, $\alpha = 94.65$ (4), $\beta = 98.80$ (3), $\gamma = 108.23$ (4)°, $V = 1690$ (1) Å^3, $Z = 2$, $D_x = 1.643$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 27.90$ cm^{-1}, $F(000) = 816$, $T = 298$ (2) K, $R = 0.032$ for 4718 unique observed reflections. The butadiene is σ-bound to the Sn atom and the pendent I substituent of the butadiene is involved in a intramolecular interaction with the Sn, having Sn···I = 3.8835 (5) Å (van der Waals radii for Sn and I are commonly 2.2 and 2.0 Å respectively). Whether this Sn···I interaction is attractive (bonding) or repulsive (steric), the presence of the I atom distorts the Sn coordination sphere from the ideal tetrahedron towards a trigonal bipyramid. The axial Br—Sn···I angle is 168.45 (2)°. Like all 1,2,3,4-tetraphenylbutadienes, this one is non-planar with 72.3 (1)° between the two olefinic planes. The C1—C2—C3—C4 torsion angle of the butadiene backbone is 78.7 (5)°. The phenyl groups on the 1, 2, 3 and 4 positions are canted with respect to their olefinic planes and the dihedral angles are 76.7 (2), −46.3 (3), 57.1 (4) and 69.8 (3)°, respectively.

Experimental. The title complex was prepared by the reaction of elemental iodine with hexaphenylstannole and tetrahydrofuran at 195 K under a nitrogen atmosphere and recrystallized from ether/trichloromethane. It should be noted that the presence of the Br atom was unexpected, as Br was not intentionally a part of the preparation and most likely part of an unknown impurity.

Colorless prismatic crystals, 0.25 × 0.25 × 0.40 mm; Nonius CAD-4 diffractometer, monochromated Mo $K\alpha$; $\theta/2\theta$ scans; $3 \le 2\theta \le 50°$; lattice parameters from 25 high-angle reflections ($2\theta > 20°$); Gaussian-integration absorption corrections applied with transmission coefficients 0.40–0.48; $0 \le h \le 12$, $-12 \le k \le 12$, $-19 \le l \le 19$; three standard reflections fluctuated 2.7%; 6093 total reflections, 5956 unique, 4718 observed with $I_o \ge 3.0\sigma(I)$. Patterson methods; full-matrix refinement *via* *SHELX*76 (Sheldrick, 1976) on F's, minimizing $\sum w(|F_o| - |F_c|)^2$; all C atoms anisotropic; H atoms were placed in calculated positions (C—H = 1.08 Å) and held invariant with U(H) set to 0.08 Å^2. Number of parameters varied was 388. For observed reflections $R = 0.032$, $wR = 0.045$, $S = 1.66$, $w = (\sigma_F)^{-2}$. In a