[A similar conformation has been reported for the Cr(CO)₃ complex of 2-methoxyacetophenone (Dusausoy, Protas, Bensancon & Tirouflet, 1973).] The aromatic ring is symmetrically bonded to the chromium atom with an average Cr—C$_{arene}$ bond length of 2.23 Å typical of (η^6-arene)tricarbonylchromium(0) complexes. Likewise, the average Cr—C$_{co}$ bond length of 1.83 Å and the average C—O bond length of 1.15 Å are typical values. Interestingly, it is observed that the methoxy and both methyl substituents remain in the plane of the ring, whereas the C(7)—N(1) bond of the amide substituent is nearly perpendicular to the plane of the ring. Moreover, C(9) in one of the ethyl groups bonded to N(1) points back towards and above the aromatic ring while the other ethyl group faces away from the ring. These different conformations for the ethyl groups in the solid state imply different magnetic environments. Correspondingly, in the ¹H NMR (solution phase) spectrum, the methylene protons attached to C(9) have more shielding interaction with the π-electron density of the aromatic ring, and occurred at higher field (δ_H 3.38) than the methylene protons attached to C(8) (δ_H 3.47). Similarly, the C(11) methyl protons resonated at higher field (δ_H 1.13) than the C(10) methyl protons (δ_H 1.20).

References

CAMBIE, R. C., ERSON, A. D., GOURDIE, A. C., RUTLEDGE, P. S. & WOODGATE, P. D. (1988). *J. Organomet. Chem.* **348**, 317–331.
DUSAUSOY, Y., PROTAS, J., BENSANCON, J. & TIROUFLET, J. (1973). *Acta Cryst.* B29, 469–476.
ENRAF-NONIUS (1981). *Structure Determination Package.* Enraf-Nonius, Delft, The Netherlands.
JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
KELLER, A. G. & SZCZECINSKI, P. (1988). *Magn. Reson. Chem.* **26**, 990–991.
MEURS, F. VAN & VAN KONINGSVELD, H. (1977). *J. Organomet. Chem.* **131**, 423–428.
REES, B. & COPPENS, P. (1973). *Acta Cryst.* B29, 2516–2528.
SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

[8,8-(PPh₃)₂-9-(OEt)-8,7-RhSB₉H₉].0.95(CH₂Cl₂)

By Michael Murphy and Trevor R. Spalding*

Department of Chemistry, University College, Cork, Ireland

and George Ferguson* and John F. Gallagher

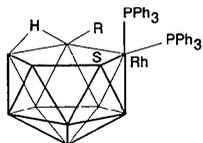*Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1*

Abstract. 9-Ethoxy-8,8-di(triphenylphosphine)-9,10-μH-8-rhoda-7-thia-*nido*-undecaborane(10) dichloromethane solvate, C₃₈H₄₄B₉OP₂RhS.0.95(CH₂Cl₂), M_r = 891.7, triclinic, P1̄, a = 10.271 (4), b = 11.401 (3), c = 19.426 (4) Å, α = 74.86 (2), β = 88.51 (3), γ = 83.51 (3)°, V = 2182 (2) Å³, Z = 2, D_x = 1.357 g cm⁻³, graphite-monochromated Mo $K\alpha$ radiation, λ = 0.71073 Å, μ = 6.5 cm⁻¹, $F(000)$ = 912, T = 294 K, R = 0.038 for 3984 observed reflections. The title compound contains an 11-atom RhSB₉ *nido*-structured cage with Rh and S atoms adjacent in the open RhSB₃ face. An ethoxy group is bonded to the B atom adjacent to Rh in the open face with Rh—B9 2.119 (6) and B9—O 1.387 (9) Å. The phosphine ligands are bonded to the Rh atom with one Rh—P bond [2.278 (2) Å] *trans* to the S atom and the other [2.417 (1) Å] located perpendicular to the open face of the cage.

Introduction. We have recently described a rhodathiaborane complex which had a *nido* structure but a *closo* electron count according to Wade's rules (Ferguson, Jennings, Lough, Coughlan, Spalding, Kennedy, Fontaine & Stibr, 1990). The compound (1a), [8,8-(PPh₃)₂-8,7-RhSB₉H₁₀], had a gross cage geometry of a dodecahedron minus one vertex. The Rh and S atoms were adjacent and located in an open RhSB₃ face. One of the PPh₃ ligands was bonded to Rh in a position *trans* to the Rh—S vector and the other Rh—P bond was perpendicular to the open face of the cage. The P—Rh—P angle was 98.50 (2)°. We now report the structural analysis of an ethoxy derivative obtained from the reaction

* E-mail addresses: GF   CHMFERG@VM.UOGUELPH.CA
   TRS   STCH8006@IRUCCVAX

between ethanol and [8,8-(PPh₃)₂-8,7-RhSB₉H₁₀], and show that the reaction affords the 9-(OEt)-compound (1*b*).

(1*a*) R = H
(1*b*) R = OEt

Experimental. A reddish-brown needle-like crystal of what was shown to be the title compound, of size $0.09 \times 0.13 \times 0.30$ mm, was mounted on a glass fibre with its long axis roughly parallel to the φ axis of the goniometer. Accurate cell dimensions and crystal orientation matrix were determined on a CAD-4 diffractometer from a least-squares refinement of data collected using the setting angles of 25 reflections in the range $9 < \theta < 15°$. Intensities of reflections with indices h 0 to 13, k -14 to 14, l -24 to 24 with $4 < 2\theta < 50°$ were measured [ω–2θ scans; ω-scan width $(0.6 + 0.35\tan\theta)°$] using graphite-monochromated Mo $K\alpha$ radiation. The intensities of three reflections measured at 120 min intervals remained constant within experimental error throughout data collection and no decay correction was required. Lorentz, polarization and absorption corrections [*DIFABS*; Walker & Stuart (1983); correction range 0.88 to 1.10] were applied. 8113 unique reflections were measured of which only the 3984 with $I > 3\sigma(I)$ were labelled observed and used in structure solution and refinement. Space group $P\bar{1}$ was assumed and confirmed after cell reduction and successful refinement. The structure was solved using the Patterson heavy-atom method; the remaining atoms were located in succeeding difference Fourier syntheses. Terminal H atoms were visible in a difference map computed at an intermediate stage of the refinement and were allowed for (in geometrically idealized positions with C—H 0.95, B—H 1.08 Å and B_{iso} values appropriate to the atoms to which they were bonded; details are in the deposition material)* but not refined in subsequent calculations. The coordinates for the H atom bridging B9 and B10 were obtained from a difference synthesis and were included and refined isotropically. Refinement was by full-matrix least-squares calculations on F using *SHELX76* (Sheldrick, 1976) initially with isotropic and finally with anisotropic thermal parameters for non-H atoms. It became obvious from difference maps that not only was there CH_2Cl_2 solvent of crystallization trapped in what would have been voids in the crystal lattice, but this solvent was disordered over two sites with markedly unequal population parameters [0.85 (1) and 0.10 (1) from refinement]. In the final cycles of refinement, all non-H atoms of (1*b*) (R = OEt) were allowed anisotropic motion as were the atoms of the major component of the solvent molecule. At convergence, $R =$ 0.038, $wR = 0.041$, goodness of fit 1.12, $w = 1/[\sigma^2(F_o) + 0.0004(F_o)^2]$. Maximum shift/e.s.d. 0.01; number of parameters in the final cycles was 512; density range in final difference map from -0.30 to 0.36 e Å^{-3}; no chemically significant features. Scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on a PDP11/73 computer using *SDP-Plus* (B. A. Frenz & Associates, Inc., 1983) and on an IBM 3081-K using *SHELX76*. Atomic coordinates and selected bond lengths and angles are given in Tables 1 and 2, respectively. Fig. 1 is a view of molecule (1*b*) prepared using *ORTEPII* (Johnson, 1976).

Discussion. The *nido*-RhSBX₉ cage structure and the conformation of the phosphine ligands in (1*b*) are essentially the same as in the precursor compound [8,8-(PPh₃)₂-8,7-RhSB₉H₁₀], (1*a*), (Ferguson, Jennings *et al.*, 1990). Thus compound (1*b*) is another example of a non-Wadian rhodathiaborane.

The Rh—S distance in the compound (1*b*) of 2.375 (2) Å is essentially identical with the corresponding distance in (1*a*) of 2.3769 (6) Å. The other Rh–cage distances in (1*b*) are Rh8—B9 2.119 (6), Rh8—B4 2.210 (5) and Rh8—B3 2.238 (7) Å which may be compared with values in (1*a*) of 2.146 (3), 2.236 (3) and 2.242 (4) Å, respectively; the substitution of the terminal H atom at B9 by the ethoxy group has thus caused the Rh8—B9 and Rh8—B4 distances to shorten slightly but significantly.

The distance between the S atom and the adjacent B atom in the open face of (1*b*) [S7—B11 1.907 (6) Å] is significantly shorter than the other S—B bond lengths [S7—B2 1.976 (6), S7—B3 2.031 (6) Å]. A similar situation was observed in (1*a*) (Ferguson, Jennings *et al.*, 1990). The cage B—B distances in (1*b*) are in the range 1.700 (9) to 1.916 (10) Å (Table 2). The range of B—B distances in (1*a*) was 1.713 (6) to 1.887 (5) Å. Both these ranges are within the wide range of values reported in metallaborane and heteroborane compounds (Kennedy, 1986).

The B9—O distance of 1.387 (8) Å is closer to values typical of $B(sp^2)$—O bonds rather than

---

* Lists of structure factors, anisotropic thermal parameters, calculated H-atom coordinates and full details of molecular dimensions have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54737 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0064]

Table 1. Atomic coordinates and equivalent isotropic thermal parameters (Å²) with e.s.d.'s in parentheses

$B_{eq} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}]$. Atoms C11′, C1S′ and C12′ were refined isotropically. The site occupancy factor is 1.0 unless otherwise specified.

	x	y	z	B_{eq}	Site occupancy
Rh8	0.30850 (4)	−0.00012 (4)	0.20244 (2)	2.11 (1)	
S7	0.2590 (1)	−0.0932 (1)	0.11167 (7)	3.09 (3)	
P1	0.4026 (1)	0.1024 (1)	0.27068 (7)	2.33 (3)	
P2	0.1152 (1)	0.1387 (1)	0.15881 (7)	2.32 (3)	
O1	0.2337 (4)	−0.1292 (3)	0.3480 (2)	3.70 (9)	
C1E	0.1742 (9)	−0.2145 (7)	0.4030 (4)	9.1 (2)	
C2E	0.1275 (11)	−0.1661 (10)	0.4590 (5)	12.3 (3)	
C11	0.5302 (5)	0.0340 (5)	0.3373 (3)	2.8 (1)	
C12	0.6528 (6)	0.0691 (6)	0.3304 (3)	4.8 (2)	
C13	0.7451 (7)	0.0192 (8)	0.3843 (4)	7.1 (2)	
C14	0.7137 (7)	−0.0622 (7)	0.4442 (4)	5.8 (2)	
C15	0.5923 (7)	−0.0963 (6)	0.4522 (3)	5.9 (2)	
C16	0.5002 (6)	−0.0478 (6)	0.3989 (3)	5.1 (2)	
C21	0.3036 (5)	0.1925 (5)	0.3211 (3)	2.7 (1)	
C22	0.1772 (6)	0.1646 (5)	0.3415 (3)	2.9 (1)	
C23	0.1011 (6)	0.2295 (6)	0.3814 (3)	4.1 (2)	
C24	0.1473 (7)	0.3256 (6)	0.3992 (3)	4.6 (2)	
C25	0.2721 (7)	0.3526 (5)	0.3809 (3)	4.5 (2)	
C26	0.3504 (6)	0.2863 (5)	0.3433 (3)	3.7 (1)	
C31	0.4843 (5)	0.2070 (4)	0.1997 (3)	2.2 (1)	
C32	0.4540 (5)	0.3324 (5)	0.1799 (3)	3.1 (1)	
C33	0.5125 (6)	0.4036 (5)	0.1213 (3)	4.2 (2)	
C34	0.5985 (6)	0.3502 (6)	0.0813 (3)	4.3 (2)	
C35	0.6301 (6)	0.2260 (5)	0.0986 (3)	3.6 (1)	
C36	0.5738 (5)	0.1554 (5)	0.1577 (3)	2.9 (1)	
C41	0.0270 (5)	0.2589 (4)	0.1941 (3)	2.3 (1)	
C42	−0.0994 (5)	0.2562 (5)	0.2194 (3)	3.6 (1)	
C43	−0.1594 (6)	0.3508 (5)	0.2447 (3)	4.6 (2)	
C44	−0.0973 (6)	0.4486 (5)	0.2455 (3)	4.3 (2)	
C45	0.0285 (6)	0.4542 (5)	0.2198 (3)	3.9 (1)	
C46	0.0905 (5)	0.3604 (5)	0.1938 (3)	3.0 (1)	
C51	0.1441 (5)	0.2269 (5)	0.0683 (3)	2.6 (1)	
C52	0.0437 (6)	0.3052 (5)	0.0280 (3)	3.8 (1)	
C53	0.0685 (7)	0.3769 (6)	−0.0388 (3)	4.9 (2)	
C54	0.1943 (7)	0.3739 (6)	−0.0654 (3)	5.4 (2)	
C55	0.2922 (6)	0.2995 (6)	−0.0263 (3)	4.7 (2)	
C56	0.2681 (5)	0.2249 (5)	0.0406 (3)	3.3 (1)	
C61	−0.0091 (5)	0.0403 (4)	0.1550 (3)	2.6 (1)	
C62	−0.0590 (5)	0.0267 (5)	0.0908 (3)	3.3 (1)	
C63	−0.1457 (6)	−0.0584 (5)	0.0932 (3)	4.1 (1)	
C64	−0.1883 (6)	−0.1261 (5)	0.1568 (4)	4.4 (2)	
C65	−0.1433 (6)	−0.1135 (5)	0.2199 (3)	3.9 (1)	
C66	−0.0516 (6)	−0.0326 (5)	0.2197 (3)	3.4 (1)	
B1	0.4668 (6)	−0.2780 (5)	0.2026 (4)	3.1 (2)	
B2	0.3686 (7)	−0.2518 (6)	0.1268 (4)	3.6 (2)	
B3	0.4421 (6)	−0.1260 (6)	0.1543 (3)	2.9 (1)	
B4	0.4469 (6)	−0.1636 (5)	0.2479 (3)	2.7 (1)	
B5	0.3693 (7)	−0.2972 (6)	0.2810 (4)	3.4 (2)	
B6	0.3170 (7)	−0.3462 (6)	0.2072 (4)	3.7 (2)	
B9	0.2795 (6)	−0.1570 (6)	0.2857 (3)	2.8 (1)	
B10	0.2001 (7)	−0.2793 (6)	0.2553 (4)	3.6 (2)	
B11	0.1979 (7)	−0.2460 (6)	0.1581 (4)	3.5 (2)	
C1S	0.8347 (4)	0.6033 (4)	0.4080 (2)	13.3 (1)	0.85
C1S	0.672 (1)	0.598 (1)	0.3998 (7)	11.8 (5)	0.85
C12	0.6369 (6)	0.4443 (6)	0.4273 (4)	26.8 (3)	0.85
C11′	0.599 (3)	0.546 (3)	0.464 (2)	12.3 (9)	0.10
C1S′	0.75 (1)	0.48 (1)	0.451 (7)	17 (5)	0.10
C12′	0.643 (2)	0.441 (1)	0.3967 (8)	4.2 (3)	0.10

Table 2. Selected bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

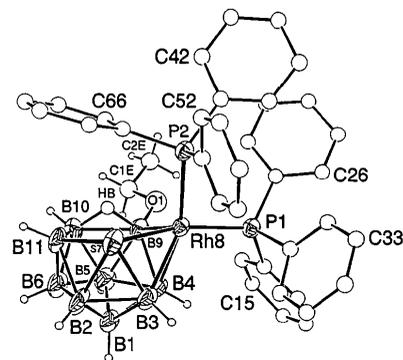|			
Rh8—S7	2.375 (2)	B1—B3	1.735 (8)
Rh8—P1	2.278 (2)	B1—B4	1.745 (10)
Rh8—P2	2.417 (1)	B1—B5	1.779 (9)
Rh8—B3	2.238 (7)	B1—B6	1.791 (10)
Rh8—B4	2.210 (5)	B2—B3	1.891 (10)
Rh8—B9	2.119 (6)	B2—B6	1.757 (9)
S7—B2	1.976 (6)	B2—B11	1.841 (9)
S7—B3	2.031 (6)	B3—B4	1.757 (9)
S7—B11	1.907 (6)	B4—B5	1.762 (9)
P1—C11	1.825 (5)	B4—B9	1.854 (9)
P1—C21	1.813 (6)	B5—B6	1.783 (11)
P1—C31	1.826 (5)	B5—B9	1.776 (9)
P2—C41	1.829 (5)	B5—B10	1.795 (10)
P2—C51	1.818 (5)	B6—B10	1.732 (10)
P2—C61	1.806 (6)	B6—B11	1.700 (9)
O1—C1E	1.421 (8)	B9—B10	1.916 (10)
O1—B9	1.387 (8)	B10—B11	1.826 (10)
C1E—C2E	1.394 (13)	B9—HB	1.31 (7)
B1—B2	1.748 (10)	B10—HB	1.23 (6)
S7—Rh8—P1	164.78 (5)	B2—S7—B3	56.3 (3)
S7—Rh8—P2	84.20 (5)	B2—S7—B11	56.6 (3)
S7—Rh8—B3	52.1 (2)	B3—S7—B11	98.3 (3)
S7—Rh8—B4	88.1 (2)	Rh8—P1—C11	124.6 (2)
S7—Rh8—B9	93.4 (2)	Rh8—P1—C21	121.2 (2)
P1—Rh8—P2	101.35 (5)	Rh8—P1—C31	98.4 (2)
P1—Rh8—B3	117.1 (2)	C11—P1—C21	98.3 (2)
P1—Rh8—B4	89.5 (2)	C11—P1—C31	104.7 (2)
P1—Rh8—B9	96.7 (2)	C21—P1—C31	108.0 (2)
P2—Rh8—B3	133.0 (2)	Rh8—P2—C41	130.6 (2)
P2—Rh8—B4	164.6 (2)	Rh8—P2—C51	109.3 (2)
P2—Rh8—B9	116.4 (2)	Rh8—P2—C61	104.5 (2)
B3—Rh8—B4	46.5 (2)	C41—P2—C51	99.6 (2)
B3—Rh8—B9	86.2 (2)	C41—P2—C61	103.3 (2)
B4—Rh8—B9	50.7 (2)	C51—P2—C61	107.8 (3)
Rh8—S7—B2	108.6 (2)	C1E—O1—B9	122.2 (5)
Rh8—S7—B3	60.5 (2)	O1—C1E—C2E	113.2 (7)
Rh8—S7—B11	107.0 (2)		

Fig. 1. A view of the [8,8-(PPh₃)₂-9-(OEt)-8,7-RhSB₉H₉] molecule. For clarity the C, H and O atoms are shown as spheres of an arbitrary size. The thermal ellipsoids shown for the Rh, P, S and B atoms are at the 50% probability level.

B(sp^3)—O ones (Ferguson, Lough, Sheehan & Spalding, 1990). A number of molecular structures containing (cage)B—O*R* groups have been reported (Kennedy, 1986); in these, the B—O distances range from 1.371 to 1.409 Å.

The P—Rh—P angle in (1*b*) is 101.35 (5) ° and the Rh—P distances are significantly different: 2.278 (2) Å for the P1—Rh8 bond *trans* to the S7 atom and 2.417 (1) Å for P2—Rh8 *cis* to S7. Similar values were observed in compound (1*a*) [2.2906 (5)

and 2.4197 (5) Å, respectively]. All distances and angles in the PPh₃, ethyl and CH₂Cl₂ moieties are normal and there were no untoward intermolecular contacts.

References

B. A. FRENZ & ASSOCIATES, INC. (1983). *SDP-Plus Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.

FERGUSON, G., JENNINGS, M. C., LOUGH, A. J., COUGHLAN, S., SPALDING, T. R., KENNEDY, J. D., FONTAINE, X. L. R. & STIBR, B. (1990). *J. Chem. Soc. Chem. Commun.* pp. 891–894.

FERGUSON, G., LOUGH, A. J., SHEEHAN, J. P. & SPALDING, T. R. (1990). *Acta Cryst.* C46, 2390–2392.

JOHNSON, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

KENNEDY, J. D. (1986). *Prog. Inorg. Chem.* 34, 211–434.

SHELDRICK, G. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

WALKER, D. & STUART, N. (1983). *Acta Cryst.* A39, 158–166.

Dimeric Ethyl(2,6-di-*tert*-butylphenoxy)zinc

BY MASOOD PARVEZ, GRETCHEN L. BERGSTRESSER AND HERMAN G. RICHEY JR

Department of Chemistry, The Pennsylvania State University, University Park, PA 16802, USA

Abstract. Bis(μ-2,6-di-*tert*-butylphenoxy)-bis(ethylzinc), $[Zn(\mu\text{-}C_{14}H_{21}O)(C_2H_5)]_2$, $M_r = 599.51$, monoclinic, $P2_1/n$, $a = 10.244\,(2)$, $b = 9.844\,(1)$, $c = 15.532\,(3)$ Å, $\beta = 92.10\,(1)°$, $V = 1565.2\,(8)$ Å^3, $Z = 2$, $D_x = 1.272$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 1.594$ mm^{-1}, $F(000) = 640$, $T = 293\,(1)$ K, $R = 0.028$ for 2656 observed reflections with $I > 3\sigma(I)$. The centrosymmetric structure has tricoordinate Zn atoms. The two Zn atoms are bonded to the O atoms of the same two 2,6-di-*tert*-butylphenoxides to form a planar four-membered ring of alternating Zn and O atoms with Zn—O 1.970 (1) and 1.990 (1) Å, Zn···Zn 3.000 (1) Å, Zn—O—Zn 98.44 (5)°, and O—Zn—O 81.56 (5)°. Each Zn is also bonded to an ethyl group, Zn—C 1.949 (2) Å.

Introduction. As part of an effort to determine if organozinc cations could be synthesized by interaction of organozinc compounds with crown ethers or cryptands, we wanted to study the reaction of an alkyl(aryloxy)zinc compound (*RZnOAr*) with crown ethers. To maximize the possibility that $^-$OAr would be lost [*e.g.* $RZnOAr + \text{crown} \rightleftharpoons RZn(\text{crown})^+ + \,^-OAr$] and not incorporated into a larger Zn-containing anion [*e.g.* $RZn(OAr)_2^-$], we also wanted the O atom to be in a sterically hindered environment. Therefore we synthesized ethyl(2,6-di-*tert*-butylphenoxy)zinc. Because the structure of this phenoxide should be interesting and also because of possible comparisons with structures of some related compounds, we undertook the structural determination of this solid.

Experimental. A hexane solution of diethylzinc (5.0 mL, 1.0 *M*, 5.0 mmol) was added to a solution of 2,6-di-*tert*-butylphenol (1.03 g, 5.0 mmol) in hexane (10 mL). The solution was stirred for 5 min and then the vial left undisturbed. Within several hours, crystals suitable for single-crystal X-ray analysis had formed (1.42 g, 4.74 mmol, 95%): m.p. *ca* 363 K with decomposition. ^{1}H NMR (200 MHz, benzene-d_6, internal standard benzene-d_5 assumed to absorb at δ 7.15): δ 0.54 (q, 2 H, $J = 8.2$ Hz, CH$_2$), 1.07 (t, 3 H, $J = 8.2$ Hz, CH_3CH$_2$), 1.59 [s, 18 H, (CH$_3$)$_3$C], 6.91 (t, 1 H, $J = 8.2$ Hz, p-H), 7.31 (d, 2 H, $J = 8.2$ Hz, m-H atoms). ^{13}C NMR [50 MHz, EtOEt–benzene-d_6 (9:1, $v:v$), internal standard benzene-d_6 assumed to absorb at δ 128.00]: δ 2.08 (CH$_2$); 11.61 (CH$_3$CH$_2$); 32.73 [(CH$_3$)$_3$C]; 35.91 [*C*(CH$_3$)$_3$]; 121.10, 126.29, 140.41 and 160.27 (C atoms of aryl ring).

A prismatic crystal with approximate dimensions $0.30 \times 0.45 \times 0.50$ mm, handled under a nitrogen atmosphere, was sealed in a glass capillary. Diffraction data were collected with an Enraf–Nonius CAD-4 automatic diffractometer equipped with a graphite monochromator. Unit-cell parameters indicated a monoclinic crystal system, and systematic absences indicated the space group. Lattice constants were obtained by least-squares refinement of the setting angles of 25 reflections with $10 < \theta < 15°$. Intensity data were collected by the $\omega/2\theta$ scan technique, scan width $(0.80 + 0.35\tan\theta)°$, using variable scan speed (1.5–5.5° min^{-1}) in the range $2 < \theta < 27°$ with $h\ 0 \rightarrow 13$, $k\ 0 \rightarrow 12$, and $l\ -19 \rightarrow 19$. The intensities of 3401 unique reflections (3806 total) were measured, of which 2656 had $I > 3\sigma(I)$ and were used; $R_f = 0.018$. The intensities of three reflections, chosen as standards and measured at regular intervals, decreased by 4.5%; the data were corrected for decay by appropriate scaling. Data were also corrected for Lorentz and polarization effects and for