trigonal-planar geometry in $[Au(Ph_3P)_3][B_9H_{19}S]$ [Au—P 2.345, 2.384, 2.389 Å; P—Au—P 112.3, 121.5, 124.1°, Au 0.20 Å from the $P_3$ plane (Guggenberger, 1974)] and in $[Au(Ph_3P)_3][BPh_4]$ [Au—P 2.365, 2.384, 2.403 Å, P—Au—P 115.2, 119.3, 125.4°, Au 0.06 Å from the $P_3$ plane (Jones, 1980)].

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# Two New Crystal Morphologies for Tetraphenyldibismuthine, $Bi_2Ph_4$

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**Abstract.** Tetraphenyldibismuthine, $Bi_2Ph_4$, $M_r$ = 726.38. (1) monoclinic, $P2_1/n$, $a$ = 11.531 (2), $b$ = 5.801 (3), $c$ = 16.691 (2) Å, $\beta$ = 104.87 (2)°, $V$ = 1079.2 (6) Å$^3$, $Z$ = 2, $D_x$ = 2.24 g cm$^{-3}$, Mo $K\alpha$, $\lambda$ = 0.7107 Å, $\mu$ = 162.42 cm$^{-1}$, $F(000)$ = 660, $T$ = 296 K, $R$ = 0.034 for 1468 reflections with $I > 3\sigma(I)$. Two identical pyramidal $Ph_2Bi$ fragments, related by an inversion center, are joined with a Bi—Bi bond [2.988 (1) Å]. The average Bi—C bond distance is 2.26 Å with Bi—Bi—$C_{ave}$ 93.6° and C—Bi—C 93.3 (4)°. (2) Tetragonal, $I4_1/a$, $a$ = 28.07 (1), $c$ = 10.83 (9) Å, $V$ = 8533 (4) Å$^3$, $Z$ = 16, $D_x$ = 2.26 g cm$^{-3}$, Mo $K\alpha$, $\lambda$ = 0.7107 Å, $\mu$ = 164.15 cm$^{-1}$, $F(000)$ = 5280, $T$ = 296 K, $R$ = 0.038 for 1287 reflections with $I > 3\sigma(I)$. The structure is essentially as for the monoclinic form with $d_{Bi—Bi}$ = 2.984 (2) Å, $d_{Bi—C(ave)}$ = 2.24 Å, Bi—Bi—$C_{ave}$ 92.7 and C—Bi—$C_{ave}$ 96.7°.

**Experimental.** $Bi_2Ph_4$ was obtained as the by-product of the reaction of $Fe(CO)_5$ with $Na^+.BiPh_2^-$ in $NH_3$ (1). Crystals which grew from hexane solution gave crystals with two new cell morphologies: monoclinic and tetragonal. Data on the monoclinic form were measured from a needle-like crystal of dimensions 0.1 × 0.1 × 0.5 mm using a Rigaku AFC5S fully automated single-crystal X-ray diffractometer over the range $h$ 0 to 14, $k$ 0 to 7, $l$ −21 to 20. The unit cell was determined from 23 reflections over the range $9.9 \le 2\theta \le 17.3°$ and later refined using reflections in the range $23 \le 2\theta \le 29°$. 2849 reflections were collected of which 2720 were unique ($R_{int}$ = 3.4%) and 1468 were classified as observed [$I > 3\sigma(I)$]. The standards showed significant decay during data collection (40%). The structure was solved using the program *MITHRIL* (Gilmore, 1983) which located the Bi atoms, followed by full-matrix least squares and Fourier syntheses to find the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic $U$ values while H atoms were included in calculated positions but not refined. The data were corrected for Lorentz–polarization effects and absorption ($\psi$ scans, transmission range 0.8167–1.000) (*DIFABS*; Walker & Stuart, 1983). Refinement of 118 variables (Molecular Structure Corporation, 1988) was carried out on $F$ with final $R$ = 0.034, $wR$ = 0.040, $S$ = 1.02 and a maximum $\Delta/\sigma$ of 0.0003. The final difference map showed peaks $\Delta\rho_{min}$ = −1.56, $\Delta\rho_{max}$ = 1.12 e Å$^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974). The molecule sits on a crystallographic inversion center lying at the midpoint of the Bi—Bi bond and consequently only half of the molecule is

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Table 1. *Positional parameters and* $B_{eq}$ *for* $Bi_2Ph_4$, *monoclinic form*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | x | y | z | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Bi | 0.44449 (4) | 0.18905 (8) | 0.53830 (2) | 3.24 (2) |
| C(1) | 0.337 (1) | −0.062 (2) | 0.5926 (6) | 3.3 (4) |
| C(2) | 0.391 (1) | −0.236 (2) | 0.6445 (7) | 3.9 (5) |
| C(3) | 0.324 (1) | −0.391 (2) | 0.6775 (8) | 5.0 (6) |
| C(4) | 0.205 (1) | −0.373 (3) | 0.661 (1) | 6.0 (8) |
| C(5) | 0.146 (1) | −0.200 (3) | 0.609 (1) | 6.2 (7) |
| C(6) | 0.212 (1) | −0.046 (3) | 0.5755 (8) | 5.2 (6) |
| C(7) | 0.293 (1) | 0.255 (2) | 0.4248 (6) | 3.2 (4) |
| C(8) | 0.228 (1) | 0.457 (2) | 0.4225 (7) | 4.6 (5) |
| C(9) | 0.131 (1) | 0.499 (2) | 0.3562 (8) | 4.8 (6) |
| C(10) | 0.099 (1) | 0.348 (2) | 0.2918 (7) | 4.4 (6) |
| C(11) | 0.161 (1) | 0.149 (2) | 0.2919 (7) | 4.7 (6) |
| C(12) | 0.259 (1) | 0.103 (2) | 0.3592 (7) | 4.3 (5) |

Table 2. *Selected intramolecular distances* (Å) *and angles* (°) *for* $Bi_2Ph_4$, *monoclinic form*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Bi | Bi$^i$ | | 2.988 (1) | Bi | C(1) | | 2.25 (1) |
| Bi | C(7) | | 2.26 (1) | | | | |
| Bi$^i$ | Bi | C(1) | 92.2 (3) | Bi$^i$ | Bi | C(7) | 95.1 (3) |
| C(1) | Bi | C(7) | 93.3 (4) | | | | |

Table 3. *Positional parameters and* $B_{eq}$ *for* $Bi_2Ph_4$, *tetragonal form*

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

| | x | y | z | $B_{eq}$(Å$^2$) |
|---|---|---|---|---|
| Bi(1) | 0.07849 (3) | 0.09708 (3) | 0.11539 (8) | 4.13 (5) |
| Bi(2) | 0.03058 (3) | 0.02517 (3) | 0.27569 (8) | 4.34 (5) |
| C(11) | 0.0116 (7) | 0.1189 (8) | 0.017 (2) | 3.8 (5) |
| C(12) | 0.0036 (8) | 0.1676 (8) | −0.000 (2) | 4.4 (5) |
| C(13) | −0.0358 (9) | 0.1819 (8) | −0.075 (2) | 5.8 (6) |
| C(14) | −0.0658 (8) | 0.1503 (8) | −0.114 (2) | 4.5 (5) |
| C(15) | −0.0618 (8) | 0.1030 (9) | −0.097 (2) | 5.4 (6) |
| C(16) | −0.0227 (8) | 0.0877 (8) | −0.030 (2) | 4.3 (5) |
| C(21) | 0.1019 (7) | 0.0395 (7) | −0.018 (2) | 3.6 (5) |
| C(22) | 0.0853 (8) | 0.0383 (8) | −0.140 (2) | 4.6 (5) |
| C(23) | 0.1032 (8) | 0.0036 (8) | −0.219 (2) | 5.3 (6) |
| C(24) | 0.1363 (8) | −0.0261 (8) | −0.181 (2) | 4.3 (5) |
| C(25) | 0.1549 (8) | −0.0265 (8) | −0.064 (2) | 4.8 (6) |
| C(26) | 0.1352 (8) | 0.0072 (8) | 0.017 (2) | 4.4 (5) |
| C(31) | 0.0065 (7) | 0.0815 (7) | 0.408 (2) | 3.6 (5) |
| C(32) | −0.0286 (8) | 0.1122 (9) | 0.374 (2) | 5.5 (6) |
| C(33) | −0.0460 (9) | 0.1485 (9) | 0.454 (2) | 6.1 (6) |
| C(34) | −0.026 (1) | 0.154 (1) | 0.565 (2) | 7.0 (7) |
| C(35) | 0.0087 (8) | 0.1226 (8) | 0.607 (2) | 4.7 (5) |
| C(36) | 0.0245 (7) | 0.0875 (7) | 0.528 (2) | 3.8 (5) |
| C(41) | 0.0976 (7) | 0.0073 (8) | 0.377 (2) | 4.1 (5) |
| C(42) | 0.1325 (8) | 0.0407 (8) | 0.403 (2) | 4.7 (5) |
| C(43) | 0.1758 (8) | 0.0274 (8) | 0.466 (2) | 4.8 (6) |
| C(44) | 0.1796 (8) | −0.0197 (9) | 0.496 (2) | 5.5 (6) |
| C(45) | 0.1467 (9) | −0.0535 (8) | 0.470 (2) | 5.8 (6) |
| C(46) | 0.1045 (7) | −0.0398 (7) | 0.410 (2) | 3.6 (5) |

Table 4. *Selected intramolecular distances* (Å) *and angles* (°) *for* $Bi_2Ph_4$, *tetragonal form*

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Bi(1) | Bi(2) | | 2.984 (2) | Bi(1) | C(11) | | 2.24 (2) |
| Bi(1) | C(21) | | 2.27 (2) | Bi(2) | C(31) | | 2.24 (2) |
| Bi(2) | C(41) | | 2.23 (2) | | | | |
| Bi(2) | Bi(1) | C(11) | 94.8 (5) | Bi(2) | Bi(1) | C(21) | 91.1 (5) |
| C(11) | Bi(1) | C(21) | 97.7 (7) | Bi(1) | Bi(2) | C(31) | 91.7 (5) |
| Bi(1) | Bi(2) | C(41) | 93.3 (5) | C(31) | Bi(2) | C(41) | 95.7 (8) |

unique. Atomic coordinates are given in Table 1 and selected bonded metricals in Table 2.* An *ORTEP* diagram (Johnson, 1976) is presented in Fig. 1 and a packing diagram in Fig. 2.

Data on the tetragonal form were obtained from a cube-shaped crystal of dimensions 0.2 × 0.2 × 0.2 mm as above over the range $h$ 0 to 22, $k$ 0 to 30, $l$ 0 to 11 (maximum $\sin\theta/\lambda = 0.54$ Å$^{-1}$). The unit cell was determined from 25 reflections over the range $7.0 \le 2\theta \le 15.0°$. 2471 reflections were collected of which 2352 were unique ($R_{int} = 4.6\%$) and 1287 were classified as observed [$I > 3\sigma(I)$]. The standards showed some decay during data collection (16%). The structure was solved using the program *MITHRIL* (Gilmore, 1983) which located the Bi atoms, followed by full-matrix least squares and Fourier syntheses to find the remaining non-hydrogen atoms. The Bi atoms were refined anisotropically, the C atoms isotropically and the H atoms

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and complete distances and angles for both forms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54746 (46 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

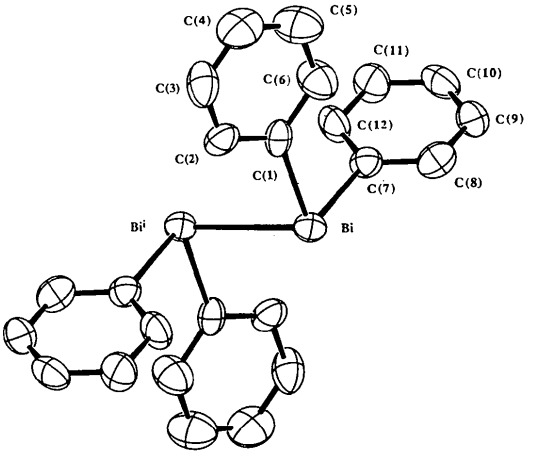


Fig. 1. *ORTEP* diagram and atom-labeling scheme of $Bi_2Ph_4$, monoclinic form, showing 50% probability ellipsoids.

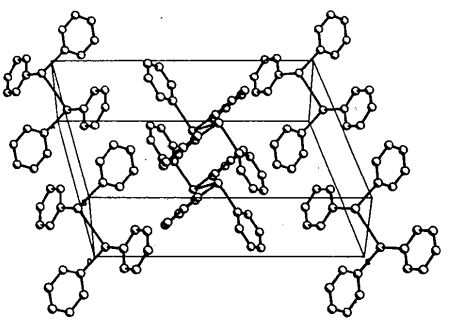

Fig. 2. Packing diagram of $Bi_2Ph_4$, monoclinic form.

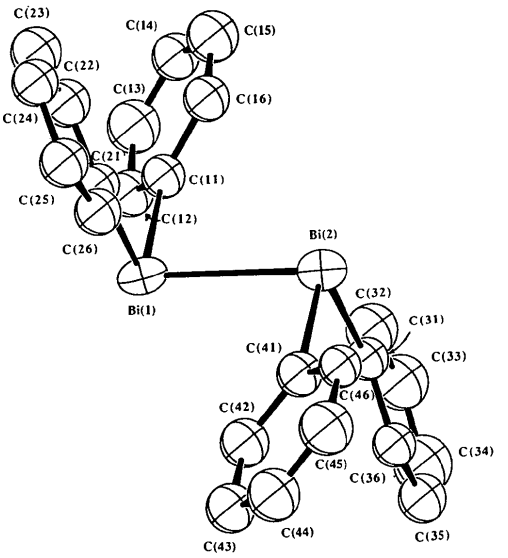


Fig. 3. *ORTEP* diagram and atom-labeling scheme of $Bi_2Ph_4$, tetragonal form, showing 50% probability ellipsoids.

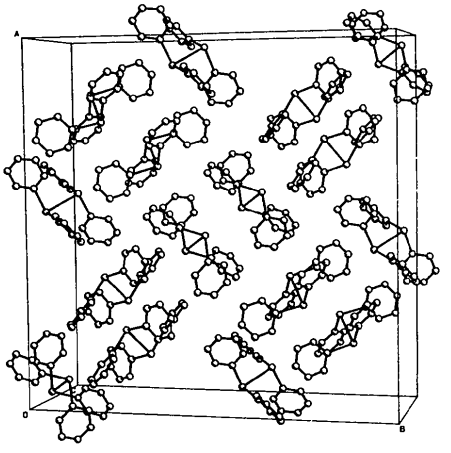

Fig. 4. Packing diagram of $Bi_2Ph_4$, tetragonal form.

were included in calculated positions but not refined. The data were corrected for Lorentz–polarization effects and absorption (*DIFABS*, transmission range 0.901–1.238). Refinement of 115 variables (Molecular Structure Corporation, 1988) was carried out on $F$ with final $R = 0.038$, $wR = 0.039$, $S = 1.21$ and a maximum $\Delta/\sigma$ of 0.0001. The final difference map showed peaks $\Delta\rho_{min} = -0.84$, $\Delta\rho_{max} = 0.93$ e Å$^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography*. Atomic coordinates are given in Table 3 and selected bond metricals in Table 4. An *ORTEP* diagram is presented in Fig. 3 and a packing diagram in Fig. 4.

**Related literature.** Bond metricals are similar to those reported for $Bi_2Ph_4$ in a triclinic cell (Calderazzo, Morvillo, Pelizzi & Poli, 1983; Calderazzo, Poli & Pelizzi, 1984).

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# Structure of 2-Chloroimidazolium Aquatrichlorocuprate(II)

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**Abstract.** $C_3H_4ClN_2^+.[CuCl_3(H_2O)]^-$, $M_r = 291.5$, monoclinic, $P2_1/c$, $a = 9.023$ (1), $b = 13.881$ (2), $c = 7.341$ (1) Å, $\beta = 97.8$ (2)°, $V = 910.9$ (5) Å$^3$, $Z = 4$, $D_x = 2.13$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.7107 Å, $\mu = 33.72$ cm$^{-1}$, $F(000) = 572$, room temperature, $R = 0.053$ for 1106 reflections with $F > 7\sigma(F)$. The struc-

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