must be assumed to have greater than the tetrahedral proportion of *p* character; hence, the lone pair or P–M orbital must have a larger share of *s* character. Since the *s* and *p* orbitals of the phosphorus atom have distinctly different radii (Coulson & Gianturco, 1968) with the *s* orbital smaller by a value on the order of perhaps 0·3 Å, there will be an apparent shortening of the P–M bond, if the length of the latter is predicted directly from a 'radius' derived from the P–C bonds themselves.

In $Cr(CO)_4$(diphos), the Cr–P–C angles have an average value of 115°, so an effect of the kind just described is to be expected. We do not, however, consider it worthwhile to attempt a quantitative estimate of its magnitude, because of the marked irregularity of the set of C–P–C angles, which range from 122 to 108°.

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A Refinement of the Crystal Structure of Bis(triphenylphosphine)(ethylene)nickel

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Dreissig & Dietrich (*Acta Cryst.* (1968). B**24**, 108) published the result of a crystal structure determination of bis(triphenylphosphine) (ethylene) nickel at a time when the authors' analysis was unrefined. The fully refined results are reported. The transformation from Dreissig & Dietrich's triclinic axes is given and normal probability plot analyses used to compare the estimated standard deviations of the two sets of atomic parameters. These analyses indicate that the e.s.d.'s of Dreissig & Dietrich's parameters are about three times as large as the present e.s.d.'s.

Some time ago we published a short note on the unrefined X-ray crystal-structure analysis of bis(triphenylphosphine)(ethylene)nickel. (Cook, Koo, Nyburg & Shiomi, 1967). We gave the approximate atomic geometry for the nickel environment and stated that the ethylene-carbon atoms were not coplanar with the P(1)–Ni–P(2) plane; they were twisted by about 12°.

While the data were being refined, another structure analysis was published independently by Dreissig & Dietrich (1968), (hereafter D & D). This analysis, although more refined than ours at that time, involved fewer intensity measurements (2112 observed as against 4050) and although the authors did not give the angle of twist of ethylene from the P(1)–Ni–P(2) plane, their listed coordinates give a twist of 8·4°.

The triclinic axial system used by us was different from that used by D & D and, because our cell dimensions were unrefined, did not give quite the same cell volume. This has led to the identity of the two substances being questioned (McGinnety & Mays, 1968). As we now show, the substances are certainly identical.

Refinement

The cell dimensions were carefully remeasured by calibrating Weissenberg photographs at 25°C with aluminum wire. Parameters are given in the first column of Table 1. The transformation to the axes used by D & D is **b**, **a**+**c**, −**a**. This transformation yields the parameters given in the second column of Table 1, which are compared with those given by D & D in the third column. There are some significant discrepancies but not sufficient to throw any doubt on the identity

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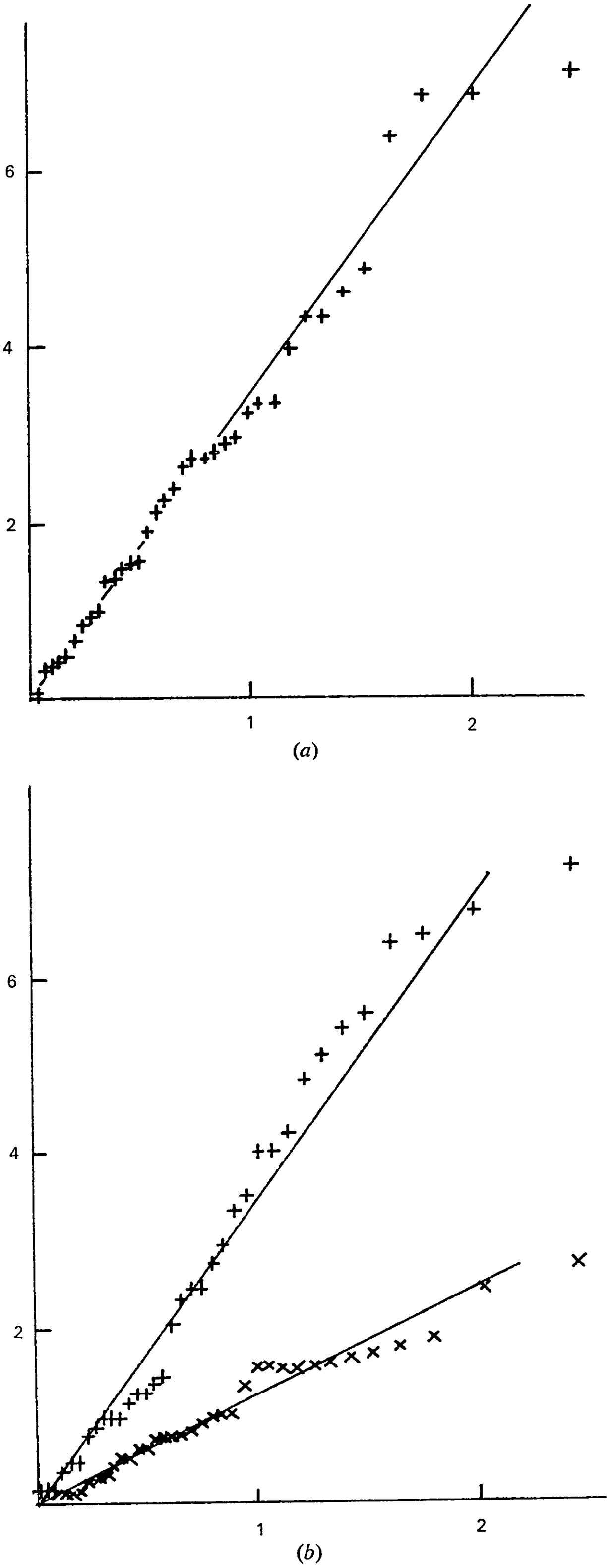

Fig. 1. Half normal probability plots comparing x parameters for phenyl carbon atoms: (*a*) D & D *vs.* present paper, (*b*) + D & D *vs.* best molecular fit parameters, × present paper *vs.* best molecular fit.

of the specimens. Reliability estimates of two sets of parameters are not easily made, but note that γ^* of D & D is the same angle as our original α^*. We have carefully remeasured our $0kl$ zero-level Weissenberg photograph and confirmed that α^* is 82·9(1)°. The γ^* angle of 81·4° given by D & D would correspond to an easily detectable displacement of the central row lines on our photograph of 0·75 mm. Note that, by coincidence, the spacing of the planes (100) and ($0\bar{1}0$) on our axes is identical. This can be clearly seen on the $h0l$ Weissenberg photograph. Because of this accidental coincidence, a^* on our axes and c^* on D & D axes are identical in length.

Table 1. *Cell parameters*

First column: refined cell dimensions on original choice of axes. Second column: data in first column transformed to axes given by D & D. Third column: data cited by D & D, except reciprocal angles (calculated from their cell parameters).

a	10·089 (10) Å	10·448 (10) Å	10·40 (2) Å
b	10·448 (10)	17·561 (10)	17·58 (10)
c	18·193 (10)	10·089 (10)	10·04 (2)
α	105·1 (1)°	102·9 (1)°	102·6 (3)°
β	109·8 (1)	117·6 (1)	116·9 (7)
γ	62·4 (1)	90·2 (1)	91·8 (3)
a^*	0·11567 (11) Å	0·10885 (10) Å	0·10905 (20) Å
b^*	0·10885 (10)	0·05888 (3)	0·05895 (3)
c^*	0·05888 (3)	0·11565 (11)	0·11569 (23)
α^*	82·9 (1)°	75·2 (1)°	74·9 (1)°
β^*	75·2 (1)	61·6 (1)	61·9 (1)
γ^*	114·4 (1)	82·9 (1)	81·4 (1)

Starting with the original atomic coordinates (Shiomi, 1967) we carried out several cycles of full-matrix least-squares refinement by the *XFLS* routine (Ellison, 1962). Hartree–Fock scattering factors (Cromer & Mann, 1968) were used, and corrections were made to the real part of the scattering factor due to anomalous dispersion. The Hughes (1941) scheme was used for weighting F_o. Final conventional R was 0·095.

Final positional and anisotropic thermal parameters, using the D & D axial system† are given in Table 2. No hydrogen contributions were used in F_c.

Some problems arise when trying to make a direct comparison of our results with those of D & D. They carried out their refinement using anisotropic factors for Ni, P(1) and P(2) atoms only. Not unexpectedly, although the thermal parameters for Ni are comparable with ours, those for phosphorus are not. D & D do not give the source of scattering factors or the weighting scheme used in refinement. In their published list of F_o *vs.* F_c a number of reflections are given the same indices (and same F_c) but different F_o, for example $\bar{3}\bar{1}4$, and several of type $h\bar{1}7$ and $h\bar{2}7$. (Although D & D claim to have listed 2505 observed reflections, only 2464 could be counted.) We used their published data (including both values of F_o when

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† A table of F_o *vs.* F_c (with our original indexing) can be obtained on application to the authors.

duplicated) to carry out a comparison of the fit between our parameters and theirs. Of course, we calculated F_c using the same scattering factors. Using our parameters (Table 3) we found that the conventional R value for D & D's observed data was 0·114. However, when the positional and thermal parameters of D & D were used we found a conventional R of 0·141. This should be compared with the residual given by D & D as 0·119, a difference which may be due to the use of different scattering factors but seems unlikely. On checking through the F's listed by D & D we found eight with quite bad agreement between F_o and F_c (in particular $\bar{1}\bar{1}1$ with $F_o=43{\cdot}4$, $F_c=105{\cdot}4$; $4\bar{3}0$ with $F_o=68{\cdot}9$, $F_c=29{\cdot}6$). In each case the agreement between our F_o and F_c was better.

Normal probability plot analysis

Where two or more sets of variates are available for comparison an instructive procedure is to carry out a normal probability plot analysis (Abrahams & Keve, 1971). This tests graphically whether the variates have the expected values based on their assumed variance. Briefly, if there are no systematic errors in either of the two sets of variates the plot should be a straight line; if the two sets are properly scaled the straight line should also pass through the origin; lastly, if the as-

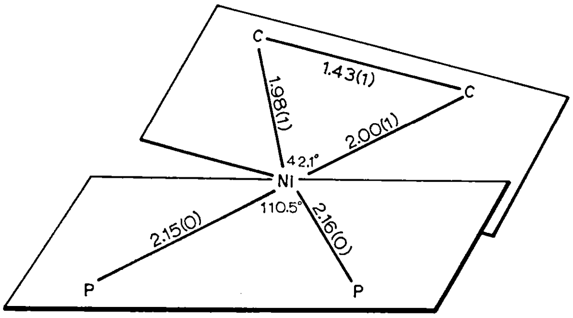

Fig. 2. Bond lengths and angles for the central part of the molecule.

Table 2. *Atomic fractional coordinates* ($\times 10^4$) *and anisotropic thermal parameters* ($\times 10^5$)

$T(hkl)=\exp[(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)]$

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Ni	3997 (1)	2278 (1)	120 (2)	1078 (18)	290 (5)	1088 (8)	−19 (6)	648 (9)	64 (3)
P(1)	2371 (2)	1370 (1)	−307 (3)	1208 (21)	254 (7)	1164 (13)	−6 (10)	683 (9)	68 (3)
P(2)	4153 (2)	3288 (1)	1182 (3)	900 (27)	257 (8)	881 (14)	45 (11)	448 (14)	116 (4)
C(1)	487 (7)	1582 (4)	−1134 (8)	1019 (86)	210 (25)	717 (37)	71 (37)	338 (35)	77 (14)
C(2)	−674 (7)	1019 (5)	−1472 (14)	823 (59)	468 (36)	1119 (47)	−14 (45)	369 (40)	156 (19)
C(3)	−2111 (9)	1233 (6)	−2057 (16)	1120 (110)	589 (45)	1420 (52)	199 (58)	438 (49)	254 (24)
C(4)	−2359 (10)	1978 (6)	−2391 (17)	1490 (139)	656 (52)	1571 (55)	388 (72)	595 (50)	343 (24)
C(5)	−1247 (11)	2495 (5)	−2122 (16)	1884 (151)	439 (42)	1896 (79)	428 (67)	865 (67)	302 (24)
C(6)	198 (10)	2317 (4)	−1495 (13)	1878 (133)	300 (31)	1413 (59)	266 (53)	905 (60)	209 (20)
C(7)	2165 (7)	388 (4)	−1589 (12)	980 (89)	234 (27)	1091 (48)	45 (38)	577 (43)	77 (17)
C(8)	1921 (8)	379 (5)	−3074 (14)	1223 (104)	471 (39)	950 (50)	127 (51)	492 (46)	93 (18)
C(9)	1669 (9)	−342 (6)	−4168 (17)	1401 (124)	512 (44)	1434 (71)	146 (61)	738 (64)	10 (30)
C(10)	1660 (9)	−1043 (5)	−3734 (17)	1445 (121)	434 (42)	1755 (78)	151 (58)	786 (71)	−32 (31)
C(11)	1918 (10)	−1048 (5)	−2274 (17)	1715 (139)	364 (40)	2391 (96)	240 (59)	1194 (85)	168 (33)
C(12)	2184 (9)	−321 (4)	−1157 (14)	1730 (130)	221 (29)	1800 (76)	93 (48)	836 (71)	141 (22)
C(13)	2780 (7)	1135 (4)	1517 (12)	1084 (92)	255 (27)	643 (38)	−51 (40)	273 (38)	71 (14)
C(14)	1827 (8)	1150 (4)	2117 (13)	1454 (110)	236 (28)	1103 (56)	56 (43)	764 (54)	79 (18)
C(15)	2298 (10)	1007 (5)	3586 (14)	1964 (142)	412 (36)	1268 (44)	179 (59)	976 (50)	304 (16)
C(16)	3698 (10)	849 (5)	4423 (14)	1913 (142)	373 (35)	953 (46)	72 (58)	544 (55)	126 (18)
C(17)	4686 (9)	829 (5)	3842 (15)	1333 (118)	484 (40)	1411 (54)	205 (58)	492 (49)	315 (21)
C(18)	4211 (8)	954 (5)	2371 (14)	1170 (104)	420 (36)	1216 (40)	45 (50)	347 (38)	305 (17)
C(19)	3378 (10)	3237 (4)	3196 (14)	2100 (139)	173 (26)	1615 (64)	107 (48)	1351 (68)	80 (21)
C(20)	1870 (10)	3129 (5)	2576 (17)	1431 (133)	380 (37)	2100 (97)	−24 (56)	357 (19)	−63 (34)
C(21)	1230 (12)	3048 (5)	3499 (19)	2719 (190)	359 (38)	3037 (124)	−29 (69)	2275 (122)	−33 (41)
C(22)	2155 (12)	3068 (6)	5075 (19)	2680 (198)	550 (47)	2736 (96)	455 (80)	2079 (101)	510 (34)
C(23)	3670 (13)	3195 (7)	5723 (20)	2814 (234)	935 (68)	2605 (71)	719 (103)	2111 (79)	855 (23)
C(24)	4289 (11)	3275 (6)	4771 (17)	2313 (175)	643 (49)	1864 (53)	388 (79)	1364 (59)	550 (20)
C(25)	3389 (9)	4134 (4)	1144 (14)	1357 (113)	305 (32)	1690 (66)	55 (48)	700 (61)	218 (22)
C(26)	2915 (14)	4721 (4)	1984 (19)	3685 (270)	434 (41)	3488 (150)	684 (88)	2214 (144)	536 (39)
C(27)	2315 (17)	5358 (7)	1298 (25)	4265 (320)	596 (59)	4400 (150)	868 (117)	2656 (171)	901 (51)
C(28)	2305 (15)	5426 (7)	−27 (13)	2755 (243)	577 (55)	3810 (127)	220 (96)	1416 (136)	809 (44)
C(29)	2826 (12)	4869 (7)	−802 (20)	1946 (172)	821 (63)	2255 (63)	143 (90)	589 (57)	810 (26)
C(30)	3371 (11)	4211 (6)	−215 (18)	1774 (148)	562 (46)	2168 (63)	120 (69)	825 (66)	559 (25)
C(31)	6062 (8)	3685 (4)	3296 (13)	1445 (113)	250 (29)	1162 (56)	24 (46)	770 (54)	12 (20)
C(32)	7034 (10)	3132 (6)	3800 (17)	1573 (130)	372 (37)	1674 (71)	6 (58)	847 (71)	128 (29)
C(33)	8483 (10)	3386 (6)	4885 (18)	1288 (116)	669 (59)	1873 (65)	55 (69)	561 (53)	279 (27)
C(34)	9008 (10)	4166 (7)	5424 (20)	1516 (145)	617 (52)	1800 (81)	−47 (74)	741 (73)	120 (36)
C(35)	8062 (13)	4723 (6)	4955 (20)	2399 (190)	584 (51)	1972 (103)	−473 (82)	1167 (109)	−174 (45)
C(36)	6578 (10)	4483 (4)	3852 (15)	1872 (136)	257 (33)	1659 (77)	−139 (53)	1006 (79)	−77 (28)
C(37)	5617 (9)	2565 (6)	−249 (18)	1203 (118)	712 (54)	2390 (86)	30 (66)	1267 (75)	285 (36)
C(38)	4866 (10)	1808 (6)	−1230 (18)	1555 (136)	697 (52)	1753 (81)	−63 (69)	1168 (75)	29 (34)

Table 3. *Bond lengths and angles*

Central part of molecule

Bond lengths:			
Ni——P(1)	2·157 (4) Å	P(1)–C(19)	1·862 (13) Å
Ni——P(2)	2·147 (4)	P(1)–C(31)	1·846 (8)
Ni——C(37)	1·980 (12)	P(1)–C(25)	1·831 (8)
Ni——C(38)	2·002 (15)	P(2)–C(13)	1·831 (9)
C(37)–C(38)	1·431 (15)	P(2)–C(7)	1·853 (9)
		P(2)–C(1)	1·825 (8)
Interbond angles:			
P(1)—Ni——P(2)	110·5 (2)°	Ni——P(1)–C(25)	114·0 (4)°
P(1)—Ni——C(37)	102·5 (4)	Ni——P(2)–C(13)	109·2 (3)
P(1)—Ni——C(38)	144·6 (3)	Ni——P(2)–C(7)	119·7 (3)
P(2)—Ni——C(37)	146·5 (4)	Ni——P(2)–C(1)	117·2 (3)
P(2)—Ni——C(38)	104·8 (3)	C(19)–P(1)–C(31)	100·9 (5)
C(37)–Ni——C(38)	42·1 (5)	C(19)–P(1)–C(25)	102·1 (5)
Ni——C(37)–C(38)	69·8 (7)	C(31)–P(1)–C(25)	103·2 (4)
Ni——C(38)–C(37)	68·1 (8)	C(13)–P(2)–C(7)	102·4 (4)
Ni——P(1)–C(19)	122·3 (3)	C(13)–P(2)–C(1)	105·9 (4)
Ni——P(1)—C(31)	112·0 (4)	C(7)—P(2)–C(1)	100·7 (4)
P(1)–C(19)–C(20)	118·5 (8)°	P(2)–C(13)–C(14)	125·0 (5)°
P(1)–C(19)–C(24)	120·1 (9)	P(2)–C(13)–C(18)	115·9 (7)
P(1)–C(31)–C(32)	116·7 (6)	P(2)–C(7)—C(8)	116·0 (5)
P(1)–C(31)–C(36)	123·9 (6)	P(2)–C(7)—C(12)	123·6 (7)
P(1)–C(25)–C(30)	118·0 (8)	P(2)–C(1)—C(2)	121·7 (5)
P(1)–C(25)–C(26)	120·8 (8)	P(2)–C(17)–C(6)	117·8 (6)

Phenyl rings

Bond lengths:			
C(1)—C(2)	1·422 (10) Å	C(19)–C(20)	1·393 (14) Å
C(2)—C(3)	1·423 (12)	C(20)–C(21)	1·404 (22)
C(3)—C(4)	1·418 (13)	C(21)–C(22)	1·491 (16)
C(4)—C(5)	1·356 (15)	C(22)–C(23)	1·399 (17)
C(5)—C(6)	1·409 (14)	C(23)–C(24)	1·413 (22)
C(6)—C(1)	1·415 (19)	C(24)–C(19)	1·409 (13)
C(7)—C(8)	1·395 (14)	C(25)–C(26)	1·424 (18)
C(8)—C(9)	1·412 (16)	C(26)–C(27)	1·440 (15)
C(9)—C(10)	1·397 (13)	C(27)–C(28)	1·364 (27)
C(10)–C(11)	1·371 (18)	C(28)–C(29)	1·386 (24)
C(11)–C(12)	1·429 (14)	C(29)–C(30)	1·417 (14)
C(12)–C(7)	1·406 (10)	C(30)–C(25)	1·399 (18)
C(13)–C(14)	1·382 (15)	C(31)–C(32)	1·408 (12)
C(14)–C(11)	1·412 (13)	C(32)–C(33)	1·389 (12)
C(15)–C(16)	1·376 (13)	C(33)–C(34)	1·363 (15)
C(16)–C(17)	1·401 (17)	C(34)–C(35)	1·392 (15)
C(17)–C(18)	1·399 (13)	C(35)–C(36)	1·417 (14)
C(18)–C(13)	1·411 (10)	C(36)–C(31)	1·390 (10)
Interbond angles:			
C(1)—C(2)—C(3)	118·8 (7)°	C(19)–C(20)–C(21)	120·8 (1·0)°
C(2)—C(3)—C(4)	119·1 (8)	C(20)–C(21)–C(22)	118·5 (1·1)
C(3)—C(4)—C(5)	121·1 (9)	C(21)–C(22)–C(23)	121·4 (1·4)
C(4)—C(5)—C(6)	121·5 (8)	C(22)–C(23)–C(24)	119·1 (1·1)
C(5)—C(6)—C(1)	118·9 (8)	C(23)–C(24)–C(19)	119·8 (1·0)
C(6)—C(1)—C(2)	120·3 (7)	C(24)–C(19)–C(20)	120·4 (1·2)
C(7)—C(8)—C(9)	120·5 (7)	C(25)–C(26)–C(27)	116·6 (1·4)
C(8)—C(9)—C(10)	118·6 (1·1)	C(26)–C(27)–C(28)	121·5 (1·6)
C(9)—C(10)–C(11)	121·9 (1·0)	C(27)–C(28)–C(29)	121·4 (1·1)
C(10)–C(11)–C(12)	119·8 (8)	C(28)–C(29)–C(30)	119·5 (1·3)
C(11)–C(12)–C(7)	118·9 (9)	C(29)–C(30)–C(25)	119·8 (1·3)
C(12)–C(7)—C(8)	120·3 (8)	C(30)–C(25)–C(26)	121·1 (8)
C(13)–C(14)–C(15)	119·8 (7)	C(31)–C(32)–C(33)	120·1 (9)
C(14)–C(15)–C(16)	120·5 (1·1)	C(32)–C(33)–C(34)	121·1 (9)
C(15)–C(16)–C(17)	120·9 (9)	C(33)–C(34)–C(35)	119·7 (9)
C(16)–C(17)–C(18)	118·3 (7)	C(34)–C(35)–C(36)	120·5 (9)
C(17)–C(18)–C(13)	121·3 (1·0)	C(35)–C(36)–C(31)	119·2 (7)
C(18)–C(13)–C(14)	119·1 (8)	C(36)–C(31)–C(32)	119·4 (7)

sumed variances are correct the line should have unit slope.

We have made use of this method to estimate the reliability of the atomic parameters cited by D & D and those found by us. We have confined our attention to the x coordinates of the phenyl-ring carbon atoms. First we plotted $\delta m_i = |\Delta x_i|/(\sigma_i^2+\sigma_i'^2)^{1/2}$ against expected values of $|\Delta x_i|$ where $|\Delta x_i|$ is the difference between the D & D parameter and ours for the ith carbon atom, σ_i is the estimated standard deviation given in Table 2 and σ_i' is taken to be 0·01 Å for each of D & D's parameters. (D & D do not give e.s.d's so we have taken the average of ours.) The half normal probability plot is shown in Fig. 1(*a*). Although not very straight, it shows no obvious systematic trends. The slope of 3·5 shows, however, that on average the σ's have been underestimated by this factor. To gain some insight into the partitioning of the variance between the two sets of parameters we proceeded as follows. Our parameters and those of D & D both correspond to phenyl rings with average C---C length of 1·40 Å. Accordingly, using a best molecular fit procedure (Nyburg, 1970) we determined the positions which the idealized phenyl rings would occupy to give separately the best least-squares fit with both D & D's and our own parameters. We assumed the best molecular fit data to be error-free and plotted $|\Delta x_i|/\sigma_i = 0{\cdot}01$ Å for D & D's data and the estimated σ_i's for our own. The two plots are shown together in Fig. 1(*b*). They are both reasonably linear but indicate that whereas our σ's are underestimated by about 25% those of D & D are underestimated by about 350%. Thus the e.s.d's on the x parameters given by D & D are about 0·035 Å and those given by us in Table 2 should be multiplied by about 1·25. These three normal probability plots are self-consistent; the variances on the latter agree quite well with those on the former, namely on average:

$$(0{\cdot}035^2+0{\cdot}01^2)^{1/2}=0{\cdot}36.$$

Discussion of the structure

The differences between our atomic fractional coordinates and those of D & D are not large enough for there to be differences in the general molecular structure or mode of molecular packing. However, there are important differences in detail. Bond lengths and angles are given in Table 3 and are illustrated for the central part of the molecule in Fig. 2.

The atomic positions in the central part changed substantially between the publication of our original note (isotropic thermal parameters) and full refinement. These coordinate changes are such as to give an ethylene-bond twist of 5·0° out of the P(1)–Ni–P(2) plane compared with our original estimate of 12°.

No other structural features are worthy of special comment.

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