Acta Cryst. (1976). B32, 1021

2,7-Dimethyl-9,10-*o*-(*m*-methyl)benzeno-9-phospha-10-azaanthracene

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(*Received* 23 *July* 1975; *accepted* 25 *July* 1975)

$C_{21}H_{18}NP$, monoclinic, $P2_1/n$, $a=13{\cdot}105$ (5), $b=24{\cdot}483$ (6), $c=11{\cdot}468$ (3) Å, $\beta=108{\cdot}57$ (2)°, $M=315{\cdot}36$, $Z=8$, $D_x=1{\cdot}20$ g cm^{-3}. There are two independent molecules in the unit cell, both with approximate C_2 symmetry. The restrictions imposed by the caged structure lead to a narrowing of the C–P–C angle to a mean of 93·3 (5)° with a corresponding mean P–C distance of 1·823 (8) Å. By contrast the mean C–N–C angle of 107·0 (5)°, with a mean N–C distance of 1·460 (13) Å, approaches the tetrahedral value.

Introduction

Cell dimensions were determined by a least-squares fit to the settings for 15 reflexions ($\pm hkl$) on a Syntex $P2_1$ four-circle diffractometer (Mo $K\alpha$,

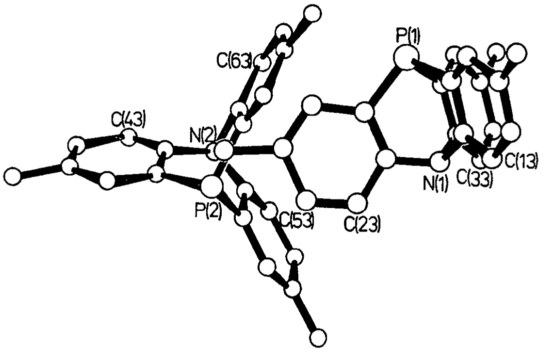

Fig. 1. The two independent molecules of (I) with the atom numbering system.

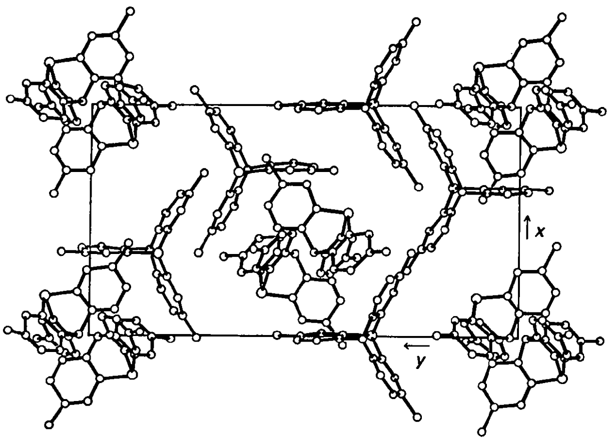

Fig. 2. Projection of the cell contents perpendicular to [001].

Table 1. *Atom positional parameters* (× 10⁴)

	x	*y*	*z*
P(1)	2625 (2)	985 (1)	3308 (3)
N(1)	1087 (5)	217 (3)	3690 (6)
C(11)	2059 (7)	1060 (4)	4546 (8)
C(12)	1331 (7)	641 (4)	4625 (9)
C(13)	859 (8)	668 (5)	5551 (10)
C(14)	1133 (8)	1078 (5)	6407 (10)
C(15)	1869 (8)	1476 (4)	6384 (9)
C(16)	2338 (7)	1464 (4)	5454 (10)
C(21)	2984 (7)	265 (4)	3559 (8)
C(22)	2100 (6)	9952 (5)	3762 (8)
C(23)	2218 (10)	9391 (5)	3981 (9)
C(24)	3155 (11)	9140 (5)	3943 (9)
C(25)	3981 (8)	9450 (5)	3715 (9)
C(26)	3859 (9)	19 (5)	3565 (8)
C(31)	1305 (8)	843 (5)	2151 (9)
C(32)	678 (8)	469 (4)	2538 (10)
C(33)	9669 (8)	325 (5)	1714 (12)
C(34)	9336 (10)	564 (6)	583 (13)
C(35)	9939 (10)	931 (6)	166 (10)
C(36)	967 (9)	1092 (5)	994 (11)
C(151)	2207 (9)	1901 (4)	7415 (11)
C(251)	4991 (8)	9131 (5)	3676 (10)
C(351)	9524 (11)	1198 (6)	8874 (11)
P(2)	1504 (2)	3451 (1)	5067 (3)
N(2)	2104 (6)	3713 (3)	7693 (7)
C(41)	721 (7)	3214 (4)	6046 (9)
C(42)	1107 (8)	3372 (4)	7254 (9)
C(43)	609 (9)	3229 (4)	8074 (10)
C(44)	9645 (10)	2904 (5)	7637 (13)
C(45)	9269 (8)	2739 (4)	6437 (14)
C(46)	9797 (8)	2892 (4)	5633 (10)
C(51)	2785 (7)	3245 (4)	6179 (10)
C(52)	2960 (8)	3418 (4)	7374 (10)
C(53)	3912 (10)	3316 (5)	8329 (11)
C(54)	4695 (10)	3032 (6)	7974 (17)
C(55)	4564 (11)	2822 (6)	6790 (18)
C(56)	3597 (10)	2946 (4)	5877 (12)
C(61)	1595 (7)	4146 (4)	5637 (10)
C(62)	1874 (7)	4206 (4)	6913 (10)
C(63)	1956 (8)	4705 (5)	7483 (10)
C(64)	3242 (8)	191 (5)	8325 (13)
C(65)	3492 (7)	128 (5)	9542 (13)
C(66)	1397 (7)	4602 (4)	4883 (10)
C(451)	8228 (10)	2369 (5)	6006 (13)
C(551)	9549 (11)	7507 (6)	8482 (17)
C(651)	3665 (9)	657 (5)	321 (12)

0·71069 Å). Intensities from a prismatic crystal, elongated *a*, with approximate dimensions 0·5 × 0·2 × 0·1 mm, were collected on the diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Measurements were carried out in θ–2θ mode ($3{\cdot}0 \leq 2\theta \leq 50{\cdot}0^\circ$) at scan speeds varying linearly between 2·93° min^{-1} (150 c.p.s. and below) and 8·37° min^{-1} (5000 c.p.s. and above). Scan and background times were equal. Three standard reflexions, monitored at regular intervals, showed no significant variations. Lorentz and polarization but no absorption [$\mu(\mathrm{Mo}\ K\alpha) = 1{\cdot}18\ \mathrm{cm}^{-1}$] corrections were applied. From the reflexions recorded (one quadrant of reciprocal space), 2066 were retained for use in the structure analysis, after application of the criterion $I \geq 2{\cdot}5\sigma(I)$ and averaging, where $\sigma(I)$ is based on the counting statistics.

The structure (Figs. 1 and 2) was solved by conventional Patterson and difference Fourier methods. Refinement with anisotropic temperature factors for all non-hydrogen atoms was by full-matrix least squares, $\sum w\Delta^2$ being minimized. The coordinates of the H atoms were revealed by difference syntheses and included as fixed parameters in the final cycles. Group isotropic temperature factors were introduced for the methyl and benzene H atoms of the two independent molecules. The final value of $R_G = [\sum w\Delta^2/\sum wF_o^2]^{1/2}$ was 0·100, with $R_w = \sum w^{1/2}\Delta/\sum w^{1/2}F_o = 0{\cdot}084$ and $R = 0{\cdot}083$. The weights were given by $w = k/[\sigma^2(F_o) + gF_o^2]$ where k and g refined to 2·3606 and 0·00218 respectively. In the final cycle the largest observed shift/e.s.d. was 0·053. A final difference synthesis displayed no peaks or troughs greater than 0·39 e $\mathrm{Å}^{-3}$. Complex

Table 2. *Anisotropic temperature factors* ($\mathrm{Å}^2 \times 10^3$)

$T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{31}lhc^*a^* + 2U_{12}hka^*b^*)]$.

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
P(1)	42 (2)	67 (2)	74 (2)	3 (2)	21 (1)	−9 (2)
N(1)	31 (4)	31 (4)	40 (5)	−3 (4)	5 (4)	−10 (4)
C(11)	51 (6)	43 (7)	55 (7)	−13 (5)	0 (5)	16 (5)
C(12)	44 (6)	68 (8)	62 (7)	12 (6)	6 (6)	−2 (6)
C(13)	62 (7)	84 (9)	62 (7)	−17 (7)	23 (6)	−4 (6)
C(14)	54 (7)	92 (10)	80 (9)	−3 (7)	23 (6)	−5 (7)
C(15)	61 (7)	49 (7)	50 (7)	−8 (5)	6 (6)	17 (6)
C(16)	37 (6)	65 (8)	83 (8)	10 (7)	12 (6)	12 (5)
C(21)	20 (5)	90 (9)	51 (7)	−1 (6)	−2 (5)	13 (6)
C(22)	59 (7)	63 (8)	53 (7)	−16 (6)	7 (6)	4 (6)
C(23)	78 (9)	77 (10)	65 (8)	−3 (7)	9 (6)	4 (7)
C(24)	96 (10)	72 (9)	58 (8)	−5 (6)	3 (7)	13 (8)
C(25)	55 (7)	78 (9)	44 (7)	−15 (6)	−6 (5)	9 (7)
C(26)	69 (8)	81 (10)	45 (7)	−9 (6)	1 (5)	−13 (7)
C(31)	48 (6)	84 (9)	46 (7)	1 (6)	4 (5)	33 (6)
C(32)	48 (7)	54 (7)	85 (9)	−20 (6)	17 (7)	−5 (6)
C(33)	43 (7)	107 (10)	82 (9)	−20 (8)	8 (7)	3 (7)
C(34)	58 (8)	118 (12)	88 (11)	−28 (9)	15 (8)	20 (8)
C(35)	59 (8)	113 (11)	61 (8)	−31 (8)	9 (7)	34 (8)
C(36)	81 (9)	81 (10)	96 (10)	−16 (8)	42 (8)	29 (7)
C(151)	100 (9)	70 (9)	98 (9)	−25 (7)	30 (7)	−3 (7)
C(251)	59 (7)	128 (12)	92 (9)	−45 (8)	−15 (6)	44 (7)
C(351)	122 (11)	146 (13)	68 (9)	−14 (8)	9 (8)	41 (10)
P(2)	57 (2)	62 (2)	64 (2)	−4 (2)	15 (1)	−3 (2)
N(2)	56 (5)	42 (5)	57 (5)	3 (4)	14 (4)	−10 (4)
C(41)	50 (6)	35 (6)	62 (7)	−10 (5)	22 (5)	−5 (5)
C(42)	65 (7)	36 (6)	61 (7)	−14 (5)	26 (6)	4 (5)
C(43)	81 (8)	74 (8)	69 (8)	−6 (6)	41 (7)	−2 (7)
C(44)	91 (10)	81 (9)	114 (11)	−10 (8)	63 (9)	−3 (8)
C(45)	44 (7)	64 (9)	131 (12)	−14 (8)	31 (8)	−8 (6)
C(46)	42 (6)	71 (8)	87 (9)	−12 (6)	15 (6)	4 (6)
C(51)	42 (6)	56 (7)	69 (8)	9 (6)	16 (6)	−1 (5)
C(52)	57 (7)	55 (7)	57 (7)	11 (6)	2 (6)	−5 (6)
C(53)	61 (8)	93 (10)	88 (9)	17 (7)	−7 (7)	−15 (7)
C(54)	43 (8)	84 (12)	186 (17)	63 (11)	14 (11)	−11 (8)
C(55)	61 (10)	96 (11)	178 (17)	41 (11)	52 (12)	−4 (8)
C(56)	68 (8)	60 (8)	120 (11)	19 (7)	40 (8)	−14 (7)
C(61)	37 (6)	64 (8)	74 (8)	11 (7)	9 (5)	8 (5)
C(62)	38 (6)	65 (8)	70 (8)	−12 (7)	10 (5)	−10 (5)
C(63)	78 (8)	59 (8)	91 (9)	−32 (7)	24 (7)	−22 (7)
C(64)	47 (7)	87 (10)	97 (10)	−2 (8)	19 (7)	8 (6)
C(65)	30 (6)	80 (10)	98 (10)	−20 (8)	12 (6)	−1 (6)
C(66)	34 (6)	53 (7)	89 (8)	7 (7)	7 (5)	−4 (5)
C(451)	82 (9)	93 (11)	202 (15)	−24 (10)	75 (16)	−32 (8)
C(551)	108 (11)	110 (12)	331 (22)	−107 (13)	152 (14)	−69 (9)
C(651)	76 (9)	86 (10)	133 (12)	−33 (9)	12 (8)	−7 (7)

neutral-atom scattering factors (Cromer & Waber, 1965; Cromer & Liberman, 1970) were employed. The heavy-atom coordinates are listed in Table 1 and their anisotropic temperature factors in Table 2; the fixed H atom positional parameters are summarized in Table 3. The full covariance matrix was used in the estimation of the bond lengths and bond angles. These are presented in Tables 4 and 5.*

Discussion

The ^{31}P chemical shifts (upfield from an 85% H_3PO_4 external reference) for triphenylphosphine (8 p.p.m.) (Van Wazer, Callis, Shoolery & Jones, 1956), 1,6-diphosphatriptycene (II) (43 p.p.m.) (Weinberg & Whipple, 1971) and azaphosphatriptycene (III) (80 p.p.m.) (Hellwinkel & Schenk, 1969) reflect the progressive distortion of the C–P–C angle required by the caged structures (II) and (III). A structure determination of (II) (Schomburg & Sheldrick, 1975) has demonstrated that the C–P–C angle in (II) is reduced to 97·0 (2) compared to angles in the range 102·1–103·6 (2)° for Ph_3P (Daly, 1966). The mean librationally corrected P–C distance in (II) of 1·845 (2) is slightly longer than those of 1·822–1·831 (5) Å in Ph_3P. This structural investigation of an azaphosphatriptycene derivative, the title compound (I), has been undertaken to investigate the extent of the further distortion at P necessitated by the introduction of N at the 10-anthracene position.

There are two independent molecules of (I) in the cell. Although significant differences are observed between the two molecules for the individual N–C distances, this is not the case for the C–N–C angles. The following mean lengths and angles are obtained: molecule 1 [P(1)]P–C 1·820 (14), N–C 1·437 (19) Å, C–P–C 93·1 (10)°, C–N–C 107·9 (11)°; molecule 2 [P(2)] P–C 1·826 (8), N–C 1·482 (7) Å, C–P–C 93·5 (2)°, C–N–C 106·1 (14)°. The very small C–P–C angle

Table 3. *Hydrogen atom coordinates* (× 10^4)

H(C13)	271	366	5591
H(C14)	762	1088	7124
H(C16)	2926	1770	5434
H(C23)	1591	9157	4172
H(C24)	3256	8705	4092
H(C26)	4507	265	3457
H(C33)	9164	33	1975
H(C34)	8548	458	9969
H(C36)	1458	1393	736
H(C43)	922	3355	9025
H(C44)	9216	2790	8263
H(C46)	9499	2763	4683
H(C53)	4035	3444	9266
H(C54)	5469	2973	8665
H(C56)	3466	2817	4940
H(C63)	2071	5035	8136
H(C64)	3040	110	7351
H(C66)	1207	4878	4109
H(C151)1	1434	1834	7846
H(C151)2	2273	2283	7295
H(C151)3	2987	2047	8364
H(C251)1	4507	8862	2821
H(C251)2	5666	9477	3839
H(C251)3	5316	8934	4583
H(C351)1	9023	926	8717
H(C351)2	9988	1651	9000
H(C351)3	9322	816	8320
H(C451)1	2120	7540	3460
H(C451)2	1700	8000	4300
H(C451)3	2141	7336	4731
H(C551)1	4570	7780	2950
H(C551)2	4600	7450	4390
H(C551)3	4100	7700	3000
H(C651)1	8340	4060	4680
H(C651)2	9290	4270	5970
H(C651)3	8820	4400	5950

The following group isotropic temperature factors were obtained: molecule (1), H(benzene) 0·099 (9), H(methyl) 0·171 (14); molecule (2), H(benzene) 0·193 (16), H(methyl) 0·182 (15) Å^2.

Table 4. *Bond lengths* (Å)

P(1)—C(11)	1·809 (11)	P(2)—C(41)	1·839 (11)
P(1)—C(21)	1·802 (11)	P(2)—C(51)	1·826 (9)
P(1)—C(31)	1·848 (9)	P(2)—C(61)	1·812 (11)
N(1)—C(12)	1·454 (12)	N(2)—C(42)	1·496 (12)
N(1)—C(22)	1·456 (13)	N(2)—C(52)	1·475 (14)
N(1)—C(32)	1·401 (13)	N(2)—C(62)	1·476 (13)
C(11)–C(12)	1·422 (14)	C(41)–C(42)	1·370 (14)
C(12)–C(13)	1·390 (17)	C(42)–C(43)	1·350 (17)
C(13)–C(14)	1·371 (16)	C(43)–C(44)	1·442 (16)
C(14)–C(15)	1·376 (16)	C(44)–C(45)	1·367 (20)
C(15)–C(16)	1·390 (17)	C(45)–C(46)	1·369 (20)
C(16)–C(11)	1·398 (14)	C(46)–C(41)	1·397 (13)
C(21)–C(22)	1·371 (16)		
C(22)–C(23)	1·396 (17)	C(51)–C(52)	1·383 (15)
C(23)–C(24)	1·385 (20)	C(52)–C(53)	1·395 (14)
C(24)–C(25)	1·413 (19)	C(53)–C(54)	1·404 (22)
C(25)–C(26)	1·407 (18)	C(54)–C(55)	1·410 (28)
C(26)–C(21)	1·398 (15)	C(55)–C(56)	1·396 (18)
C(31)–C(32)	1·394 (16)	C(56)–C(51)	1·422 (17)
C(32)–C(33)	1·403 (14)	C(61)–C(62)	1·398 (16)
C(33)–C(34)	1·362 (19)	C(62)–C(63)	1·373 (16)
C(34)–C(35)	1·379 (21)	C(63)–C(64)	1·479 (18)
C(35)–C(36)	1·434 (15)	C(64)–C(65)	1·337 (20)
C(36)–C(31)	1·398 (16)	C(65)–C(66)	1·434 (17)
		C(66)–C(61)	1·384 (15)
C(15)–C(151)	1·532 (15)		
C(25)–C(251)	1·550 (17)	C(45)–C(451)	1·579 (16)
C(35)–C(351)	1·551 (17)	C(55)–C(551)	1·509 (18)
		C6(5)–C(651)	1·548 (18)
C(151)–H(1511)	1·27	C(451)–H(4511)	0·91
C(151)–H(1512)	0·96	C(451)–H(4512)	0·98
C(151)–H(1513)	1·28	C(451)–H(4513)	1·09
C(251)–H(2511)	1·19	C(551)–H(5511)	0·93
C(251)–H(2512)	1·19	C(551)–H(5512)	1·04
C(251)–H(2513)	1·08	C(551)–H(5513)	0·84
C(351)–H(3511)	0·91	C(651)–H(6511)	1·01
C(351)–H(3512)	1·25	C(651)–H(6512)	0·93
C(351)–H(3513)	1·12	C(651)–H(6513)	0·69

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31296 (14 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

correlates nicely with the trend in the ^{31}P chemical shifts. However, the mean P–C distance of 1·823 (8) Å for the two molecules is similar to those in Ph$_3$P and only slightly smaller than in (II). As was observed for (II), considerable distortion of the trigonal angles at C(11)–C(16) is necessary to enable the achievement of the mean C–P–C angle of 93·3 (5)°. Thus the mean value for the internal P–C–C angles, within the P(CC)$_3$N cage, is 115·6 (4) [(II) 117·4 (2)] with an external angle of 124·0 (4)° [(II) 122·6 (2)°].

Table 5. *Bond angles* (°)

C(11)P(1)C(21)	94·7 (0·5)	C(41)P(2)C(51)	93.1 (0·5)
C(11)P(1)C(31)	93·4 (0·5)	C(41)P(2)C(61)	93·5 (0·5)
C(21)P(1)C(31)	91·3 (0·5)	C(51)P(2)C(61)	93·9 (0·4)
C(12)N(1)C(22)	107·1 (0·6)	C(42)N(2)C(52)	107·2 (0·7)
C(12)N(1)C(32)	108·0 (0·7)	C(42)N(2)C(62)	105·3 (0·6)
C(22)N(1)C(32)	108·6 (0·8)	C(52)N(2)C(62)	105·9 (0·8)
P(1)C(11)C(12)	115·3 (0·7)	P(2)C(41)C(42)	115·5 (0·7)
P(1)C(11)C(16)	125·5 (0·8)	P(2)C(41)C(46)	124·3 (0·8)
C(12)C(11)C(16)	118·9 (1·0)	C(42)C(41)C(46)	120·3 (1·1)
N(1)C(12)C(11)	117·5 (1·0)	N(2)C(42)C(41)	119·9 (1·0)
N(1)C(12)C(13)	123·4 (0·9)	N(2)C(42)C(43)	118·3 (0·9)
C(11)C(12)C(13)	119·0 (1·0)	C(41)C(42)C(43)	121·8 (0·9)
C(12)C(13)C(14)	120·3 (1·1)	C(42)C(43)C(44)	117·7 (1·0)
C(13)C(14)C(15)	121·9 (1·2)	C(43)C(44)C(45)	120·6 (1·3)
C(14)C(15)C(16)	119·0 (1·0)	C(44)C(45)C(46)	120·0 (1·1)
C(14)C(15)C(151)	119·9 (1·1)	C(44)C(45)C(451)	118·9 (1·3)
C(16)C(15)C(151)	121·1 (1·0)	C(46)C(45)C(451)	121·2 (1·2)
C(15)C(16)C(11)	120·8 (1·0)	C(45)C(46)C(41)	119·7 (1·0)
P(1)C(21)C(22)	117·0 (0·8)	P(1)C(51)C(52)	115·6 (0·8)
P(1)C(21)C(26)	123·5 (0·8)	P(2)C(51)C(56)	124·4 (0·8)
C(22)C(21)C(26)	119·4 (1·0)	C(52)C(51)C(56)	119·9 (0·9)
N(1)C(22)C(21)	117·9 (0·9)	N(2)C(52)C(51)	119·8 (0·8)
N(1)C(22)C(23)	119·8 (1·0)	N(2)C(52)C(53)	116·9 (1·0)
C(21)C(22)C(23)	122·3 (1·1)	C(51)C(52)C(53)	123·3 (1·1)
C(22)C(23)C(24)	118·6 (1·2)	C(52)C(53)C(54)	114·5 (1·2)
C(23)C(24)C(25)	120·5 (1·1)	C(53)C(54)C(55)	125·7 (1·2)
C(24)C(25)C(26)	119·3 (1·1)	C(54)C(55)C(65)	116·5 (1·4)
C(24)C(25)C(251)	116·5 (1·1)	C(54)C(55)C(551)	121·6 (1·3)
C(26)C(25)C(251)	124·1 (1·1)	C(56)C(55)C(551)	121·8 (1·7)
C(25)C(26)C(21)	119·7 (1·1)	C(55)C(56)C(51)	120·0 (1·3)
P(1)C(31)C(32)	114·0 (0·7)	P(2)C(61)C(62)	116·1 (0·8)
P(1)C(31)C(36)	122·8 (0·9)	P(2)C(61)C(66)	123·6 (0·9)
C(32)C(31)C(36)	123·2 (0·9)	C(62)C(61)C(66)	120·3 (1·0)
N(1)C(32)C(31)	120·1 (0·8)	N(2)C(62)C(61)	118·9 (0·9)
N(1)C(32)C(33)	121·3 (1·0)	N(2)C(62)C(63)	118·0 (1·0)
C(31)C(32)C(33)	118·4 (1·0)	C(61)C(62)C(63)	123·1 (1·0)
C(32)C(33)C(34)	118·7 (1·2)	C(62)C(63)C(64)	116·4 (1·1)
C(33)C(34)C(35)	124·4 (1·1)	C(63)C(64)C(65)	122·1 (1·2)
C(34)C(35)C(36)	118·1 (1·1)	C(64)C(65)C(66)	120·3 (1·2)
C(34)C(35)C(351)	122·7 (1·0)	C(64)C(65)C(651)	116·7 (1·1)
C(36)C(35)C(351)	119·1 (1·2)	C(66)C(65)C(651)	121·7 (1·2)
C(35)C(36)C(31)	117·1 (1·1)	C(65)C(66)C(61)	119·0 (1·1)

This work was carried out within the technology programme of the Ministry for research and technology of the Federal Republic of Germany. The calculations were carried out on the GMBF PDP 10 computer. We are indebted to Professor D. Hellwinkel for supplying the compound and to Professor R. Schmutzler for his support.

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