Hickel & Colleter, 1981; Leger, Goursolle & Carpy, 1984; Gadret, Goursolle, Leger & Colleter, 1975; Weber & Petcher, 1977). In contrast to τ_3, the conformations around C(1)–O(1), O(1)–C(13) and C(14)–C(15) are probably strongly preferred as only conformations $\tau_1 \simeq 0$, $\tau_2 \simeq 180$ and $\tau_4 \simeq 180°$ are uniformly found in the crystals; in (–)-VUL 111 $\tau_1 = -4{\cdot}6$ (9), $\tau_2 = 177{\cdot}8$ (6) and $\tau_4 = 164{\cdot}9$ (6)°. The coplanarity of the O(1)–C(13) bond with the adjacent aromatic system, a feature well known from the structures of aromatic alkoxy compounds (Domiano, Nardelli, Balsamo, Macchia & Macchia, 1979), is rationalized on electronic grounds due to some degree of conjugation between the O(1) non-bonding orbital and the aromatic π system. This is further supported by a widening of the C(1)–O(1)–C(13) bond angle [118·4 (5)°], suggesting an essentially sp^2 hybridization state of O(1).

The molecular packing, as can be seen from Fig. 2, is influenced by hydrogen bonding. The (–)-VUL 111 molecules form infinite chains along the screw axes (at $x, \frac{1}{4}, \frac{1}{2}$ and $x, \frac{3}{4}, 0$), the main intrachain interactions being N(1)···Cl$^-$ and O(2)···Cl$^-$ hydrogen bonds. Apart from these hydrogen bonds there are no distances between non-hydrogen atoms shorter than 3·5 Å.

All calculations were performed on a Siemens 4004/150 computer at the Research Computing Centre of Comenius University. We are grateful to Dr J. Soldánová for measurements of X-ray diffraction intensities on a Syntex $P2_1$ diffractometer.

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Structures of 2,6-Bis(benzylidene)cyclohexanone (III) and 3,5-Bis(4-dimethylaminobenzylidene)-1-methyl-4-piperidone (IV)

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Abstract. (III): $C_{20}H_{18}O$, $M_r = 274{\cdot}36$, monoclinic, $P2_1/c$, $a = 10{\cdot}096$ (1), $b = 18{\cdot}393$ (2), $c = 9{\cdot}4731$ (9) Å, $\beta = 121{\cdot}388$ (8)°, $V = 1501{\cdot}79$ Å^3, $Z = 4$, D_m(by flotation) = 1·202, $D_x = 1{\cdot}213$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0{\cdot}57$ cm^{-1}, $F(000) = 584$, $T = 287$ K, $R = 0{\cdot}040$ ($wR = 0{\cdot}044$) for 2580 observed reflections. (IV): $C_{24}H_{29}N_3O$, $M_r = 375{\cdot}52$, monoclinic, $P2_1/n$, $a = 16{\cdot}098$ (2), $b = 6{\cdot}1533$ (6), $c = 20{\cdot}606$ (3) Å, $\beta = 96{\cdot}75$ (1)°, $V = 2027{\cdot}06$ Å^3, $Z = 4$, D_m(by flotation) = 1·233, $D_x = 1{\cdot}230$ g cm^{-3},

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λ(Cu $K\alpha$) = 1·5418 Å, μ = 0·60 cm^{-1}, $F(000)$ = 808, T = 287 K, R = 0·051 (wR = 0·049) for 2055 observed reflections. Most bond distances and angles show good correspondence between (III) and (IV) and are also close to those of (I) and (II) which were recently reported [Jia, Quail, Arora & Dimmock (1988). *Acta Cryst.* C**44**, 2114–2117]. The central nonheterocyclic ring in (III) exhibits a chair conformation but the heterocyclic ring in (IV) exhibits a boat conformation. However, both rings are obviously flattened at the C4 end due to the conjugated dienone system. For both molecules the steric repulsions between the H atoms attached to C2/C6 and those on the phenyl rings cause increases in the bond angles at the C atoms joining the rings and rotations of the phenyl groups about the C8–C9/C7–C15 bonds.

Introduction. The present paper describes the third and fourth structures to be reported in a series of cyclic conjugated bis(benzylidene)ketones which have much higher cytotoxicity to P388 leukemia cells than the clinically useful drug, BCNU (Warrington, Fang, Dimmock & Arora, 1987). 3,5-Bis(benzylidene)-4-piperidone hydrochloride (I) has a little over 50 times the activity of its *N*-methyl analog (II) (Jia, Quail, Arora & Dimmock, 1988). Replacement of the basic center of (I) and (II) by a methylene group gives rise to (III) with 25 times the potency of (II) whereas introduction of a 4-dimethylamino group into the aryl rings of (II) to give (IV) doubled the bioactivity. In order to ascertain if there is a structural basis for the differences in observed biological activities of these compounds, the crystal structures were determined.

Experimental. Compound synthesized by: (III) a literature procedure (Smith, Dimmock & Turner, 1973) in a yield of 62%, crystallized from EtOH as dark-yellow fluffy needles, m.p. 389–390 K (literature m.p. 390–391 K; Vorlander & Kunze, 1926), (IV) a literature procedure (McElvain & Rorig, 1948) in a yield of 60% except that 95% EtOH was used as the solvent, crystallized from 95% EtOH as reddish-orange needles, m.p. 496–498 K (literature m.p. 496–498 K; Leonard & Locke, 1955). Good quality crystal used for data collection from: (III) mixture of 2-propanol and 95% EtOH, (IV) mixture of MeOH and hexane. Crystal: (III) light yellow, 0·18 × 0·23 × 0·48 mm, (IV) yellow, 0·056 × 0·16 × 0·25 mm. Space group: (III) $P2_1/c$, (IV) $P2_1/n$ (equivalent positions: x, y, z; $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; $-x, -y, -z$; $-\frac{1}{2}+x, -\frac{1}{2}-y, -\frac{1}{2}+z$, equivalent standard setting: $P2_1/c$). Cell parameters by least squares using 25 reflections with $10{\cdot}96 < \theta < 25{\cdot}14°$ (III), $11{\cdot}02 < \theta < 30{\cdot}04°$ (IV). CAD-4 Enraf–Nonius diffractometer. Data collection: (III) 3055 unique reflections, $0 \le h \le 12$, $0 \le k \le 23$, $-11 \le l \le 11$, $(\sin\theta)/\lambda = 0{\cdot}6265$ Å^{-1}, (IV) 2990 unique reflections, $-19 \le h \le 19$, $0 \le k \le 6$, $0 \le l \le 23$, $(\sin\theta)/\lambda = 0{\cdot}6265$ Å^{-1}. 2580 (III), 2055 (IV) reflections with $I > 2\sigma(I)$ used in refinement. Three intensity and orientation monitor reflections for both (III) and (IV), intensity fluctuation within 2%. No absorption or extinction correction applied. Merging R based on intensities 0·0075 for 106 (III), 0·0103 for 286 (IV) replicate reflections. Structure solved by direct methods using *XTAL*2·4 (Hall & Stewart, 1988), all non-H atoms found on E map and refined anisotropically. H atoms: (III) found on difference map and refined isotropically; (IV) calculated and refined isotropically. R = 0·040, wR = 0·044 [$w = 1/\sigma^2(F)$], S = 2·510 for 2580 observed reflections (III) and R = 0·051, wR = 0·049 [$w = 1/\sigma^2(F)$], S = 1·972 for 2055 observed reflections (IV); 263 (III), 370 (IV) parameters refined. F magnitudes used in LS refinement. Final $(\Delta/\sigma)_{av}$ = 0·012 (III), 0·016 (IV), $(\Delta/\sigma)_{max}$ = 0·57 (III), 0·24 (IV). $\Delta\rho$ in final difference map within +0·14 and −0·14 e Å^{-3} (III), +0·25 and −0·24 e Å^{-3} (IV). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed on a VAX 8650 computer at the University of Saskatchewan.

Discussion. The atomic parameters are summarized in Tables 1 and 2.* Bond distances, angles, selected torsion angles and some interatomic distances which may be of importance in understanding the bioactivities of (III) and (IV) are listed in Table 3. Figs. 1 and 2 are *ORTEP* drawings (Johnson, 1976) of (III) and (IV), respectively. Most atom numbers are assigned as previously described for (I) and (II) (Jia *et al.*, 1988).

Most bond distances and angles of (III) and (IV) display a close correspondence to each other and also to those of (I) and (II). The central nonheterocyclic ring in (III) shows a chair conformation but the heterocyclic ring in (IV) exhibits a boat conformation, with both rings flattened obviously at the C4 end because of the conjugated dienone system. Due to the electron-donating effect of dimethylamino groups, the dienone system in (IV) displays a higher degree of conjugation that is demonstrated by the bond distances, the closer coplanarity of the phenyl groups with the dienone moiety and the more flattened chair conformation with respect to the C4 end. The angles between planes C2–C1–C6 (or C2–N1–C6), C3, C4, O4, C5 and reference plane C2, C3, C5, C6 (A) are 51·8 (2) and 10·6 (1)° (III), 56·6 (3) and 6·5 (2)° (IV), respectively.

The steric repulsion between the aromatic rings and the central ring is mainly ascribed to the short intramolecular nonbonded H2*a*···H14 and H6*a*···H20

* Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51413 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters* ($\times 10^3$) *for* (III), *with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3\sin^2\beta$.

	x	y	z	U_{eq}(Å^2)
O4	0·8988 (1)	0·11369 (5)	0·8052 (1)	61·8
C1	0·6388 (2)	−0·07087 (8)	0·7293 (2)	55
C2	0·6128 (2)	−0·00696 (7)	0·8143 (2)	55
C3	0·6755 (2)	0·06181 (7)	0·7851 (2)	43
C4	0·8276 (2)	0·05776 (7)	0·7935 (2)	44
C5	0·8899 (1)	−0·01521 (7)	0·7862 (2)	42
C6	0·8104 (2)	−0·08259 (7)	0·7963 (2)	50
C7	1·0104 (2)	−0·01409 (7)	0·7636 (2)	48
C8	0·6030 (2)	0·12608 (7)	0·7431 (2)	49
C9	0·4570 (2)	0·14707 (7)	0·7298 (2)	47
C10	0·3568 (2)	0·19501 (8)	0·6037 (2)	59
C11	0·2177 (2)	0·2154 (1)	0·5864 (2)	63
C12	0·1764 (2)	0·19042 (9)	0·6955 (2)	65
C13	0·2768 (2)	0·14591 (8)	0·8247 (2)	69
C14	0·4153 (2)	0·12438 (8)	0·8423 (2)	58
C15	1·0962 (2)	−0·07411 (7)	0·7445 (2)	46
C16	1·1652 (2)	−0·06030 (9)	0·6518 (2)	56
C17	1·2419 (2)	−0·1142 (1)	0·6214 (2)	64
C18	1·2550 (2)	−0·1827 (1)	0·6864 (2)	68
C19	1·1917 (2)	−0·19707 (9)	0·7833 (2)	65
C20	1·1138 (2)	−0·14325 (8)	0·8132 (2)	54

Table 2. *Fractional coordinates and equivalent isotropic thermal parameters* ($\times 10^3$) *for* (IV), *with e.s.d.'s in parentheses*

$U_{eq} = (U_{11} + U_{22}\sin^2\beta + U_{33} + 2U_{13}\cos\beta)/3\sin^2\beta$.

	x	y	z	U_{eq}(Å^2)
O4	0·5198 (1)	0·1272 (3)	0·7974 (1)	68
N1	0·3667 (1)	0·6264 (4)	0·7544 (1)	49
N2	0·6505 (2)	0·6523 (5)	0·4501 (1)	73
N3	0·3248 (2)	0·4496 (4)	1·1425 (1)	64
C1	0·3145 (2)	0·8194 (6)	0·7405 (2)	65
C2	0·4419 (2)	0·6423 (5)	0·7213 (2)	49
C3	0·4933 (2)	0·4393 (5)	0·7306 (1)	45
C4	0·4854 (2)	0·3066 (5)	0·7900 (1)	50
C5	0·4373 (2)	0·3983 (5)	0·8402 (1)	45
C6	0·3900 (2)	0·6050 (5)	0·8240 (2)	50
C7	0·4407 (2)	0·2918 (5)	0·8978 (2)	50
C8	0·5478 (2)	0·3702 (5)	0·6904 (2)	50
C9	0·5733 (2)	0·4573 (5)	0·6306 (1)	50
C10	0·6235 (2)	0·3292 (6)	0·5954 (2)	57
C11	0·6502 (2)	0·3913 (6)	0·5375 (2)	62
C12	0·6273 (2)	0·5916 (6)	0·5093 (2)	55
C13	0·5791 (2)	0·7247 (6)	0·5452 (2)	63
C14	0·5539 (2)	0·6621 (5)	0·6033 (2)	61
C15	0·4075 (2)	0·3423 (4)	0·9582 (1)	47
C16	0·4092 (2)	0·1815 (5)	1·0063 (2)	55
C17	0·3830 (2)	0·2127 (5)	1·0662 (2)	56
C18	0·3514 (2)	0·4146 (5)	1·0831 (1)	50
C19	0·3489 (2)	0·5790 (6)	1·0352 (2)	58
C20	0·3773 (2)	0·5431 (5)	0·9760 (2)	55
C1N2	0·7083 (3)	0·5233 (9)	0·4184 (2)	85
C2N2	0·6261 (4)	0·8603 (8)	0·4214 (3)	88
C1N3	0·3280 (3)	0·2787 (6)	1·1912 (2)	65
C2N3	0·2882 (3)	0·6556 (7)	1·1582 (2)	72

contacts: 2·34 (3) and 2·22 (2) Å (III), 2·08 (4) and 2·03 (4) Å (IV), respectively. Ample evidence for short distances between H atoms has been reported (Nyburg & Faerman, 1986). This steric repulsion is reduced in two ways: (1) by expansion of the angles C2–C3–C8, C3–C8–C9, C8–C9–C14 and C6–C5–C7, C5–C7–C15, C7–C15–C20; (2) by rotation about

Table 3. *Bond distances* (Å), *angles* (°), *selected torsion angles* (°) *and some interatomic distances* (Å) *for* (III) *and* (IV), *with e.s.d.'s in parentheses*

	Compound (III)	Compound (IV)
O4–C4	1·226 (2)	1·236 (4)
N1–C1	—	1·463 (5)
N1–C2	—	1·461 (4)
N1–C6	—	1·446 (4)
N2–C1N2	—	1·437 (7)
N2–C2N2	—	1·444 (6)
N2–C12	—	1·370 (4)
N3–C1N3	—	1·451 (5)
N3–C2N3	—	1·451 (5)
N3–C18	—	1·360 (4)
C1–C2	1·523 (3)	—
C1–C6	1·518 (2)	—
C2–C3	1·503 (2)	1·498 (4)
C3–C4	1·498 (2)	1·489 (4)
C3–C8	1·337 (2)	1·345 (4)
C4–C5	1·499 (2)	1·476 (4)
C5–C6	1·507 (2)	1·500 (4)
C5–C7	1·341 (3)	1·352 (4)
C8–C9	1·465 (3)	1·446 (4)
C9–C10	1·403 (2)	1·392 (5)
C9–C14	1·397 (3)	1·401 (5)
C9–C15	1·471 (2)	1·446 (5)
C10–C11	1·378 (3)	1·369 (5)
C11–C12	1·380 (4)	1·393 (3)
C12–C13	1·380 (2)	1·397 (5)
C13–C14	1·378 (3)	1·364 (5)
C15–C16	1·399 (3)	1·398 (5)
C15–C20	1·397 (2)	1·392 (4)
C16–C17	1·378 (3)	1·366 (5)
C17–C18	1·378 (3)	1·401 (5)
C18–C19	1·390 (4)	1·412 (5)
C19–C20	1·382 (3)	1·369 (5)
C1–N1–C2	—	110·2 (3)
C1–N1–C6	—	110·1 (3)
C2–N1–C6	—	109·7 (2)
C12–N2–C1N2	—	120·9 (3)
C12–N2–C2N2	—	121·1 (4)
C1N2–N2–C2N2	—	117·6 (4)
C18–N3–C1N3	—	121·2 (3)
C18–N3–C2N3	—	121·3 (3)
C1N3–N3–C2N3	—	117·5 (3)
C2–C1–C6	111·2 (1)	—
N1–C2–C3	—	111·1 (3)
C1–C2–C3	110·3 (2)	—
C2–C3–C4	117·8 (1)	117·7 (3)
C2–C3–C8	125·5 (2)	125·1 (3)
C4–C3–C8	116·6 (1)	117·3 (3)
C3–C4–C5	119·0 (1)	118·0 (3)
O4–C4–C3	120·0 (1)	120·7 (3)
O4–C4–C5	121·0 (2)	121·3 (3)
N1–C5–C6	—	111·3 (3)
C4–C5–C6	118·9 (1)	117·6 (3)
C4–C5–C7	115·5 (1)	117·4 (3)
C6–C5–C7	125·5 (1)	125·0 (3)
C1–C6–C5	112·7 (1)	—
C5–C7–C15	130·5 (1)	132·2 (3)
C3–C8–C9	129·0 (2)	132·8 (3)
C8–C9–C10	119·0 (2)	118·3 (3)
C8–C9–C14	123·0 (1)	126·7 (3)
C10–C9–C14	117·9 (2)	114·9 (3)
C9–C10–C11	120·7 (2)	123·9 (3)
C10–C11–C12	120·5 (2)	121·0 (3)
N2–C12–C11	—	121·7 (3)
N2–C12–C13	—	122·5 (3)
C11–C12–C13	119·5 (2)	115·8 (3)
C12–C13–C14	120·6 (2)	122·7 (3)
C9–C14–C13	120·8 (1)	122·1 (3)
C7–C15–C16	117·3 (1)	118·6 (3)
C7–C15–C20	124·5 (2)	126·4 (3)
C16–C15–C20	118·3 (2)	114·9 (3)
C15–C16–C17	121·2 (2)	124·0 (3)

Table 3 (*cont.*)

	Compound (III)	Compound (IV)
C16–C17–C18	119·9 (2)	120·6 (3)
N3–C18–C17	—	121·7 (3)
N3–C18–C19	—	122·0 (3)
C17–C18–C19	119·9 (2)	116·3 (3)
C18–C19–C20	120·4 (2)	121·5 (3)
C15–C20–C19	120·3 (2)	122·8 (3)
C1N2–N2–C12–C11	—	−8·0 (5)
C2N2–N2–C12–C11	—	−0·2 (8)
C1N3–N3–C18–C17	—	0·1 (7)
C2N3–N3–C18–C19	—	3·2 (5)
N1–C2–C3–C4	—	−26·1 (4)
C1–C2–C3–C4	−41·0 (2)	—
C2–C3–C8–C9	5·9 (2)	0·0 (9)
C4–C5–C6–N1	—	27·4 (4)
C4–C5–C6–C1	28·5 (2)	—
C6–C5–C7–C15	−0·5 (3)	−3·8 (5)
C5–C7–C15–C20	−28·7 (2)	−14·8 (5)
C3–C8–C9–C14	40·1 (2)	9·6 (5)
C8–C9–C14–C13	179·7 (2)	−177·7 (3)
C7–C15–C20–C19	177·6 (1)	−178·1 (3)
N1–C7	—	3·684 (4)
N1–C8	—	3·692 (4)
C1–C7	3·742 (3)	—
C1–C8	3·649 (2)	—
O4–C7	2·722 (2)	2·747 (4)
O4–C8	2·737 (2)	2·746 (4)
C7–C8	4·772 (3)	4·818 (5)
C12–C18	12·910 (3)	13·155 (5)

C8–C9, C7–C15 bonds at the expense of the conjugation energy of the system. Similar effects have been observed previously (Tokuno, Matsui, Miyoshi, Asao, Ohashi & Kihara, 1986). For example, the opening of the angle C5–C7–C15 leads to a value of 130·5 (1)° (III), 132·2 (3)° (IV). (III) shows more rotation about the bonds but less expansion of the bond angles than (IV) whose aryl planes are therefore less tilted from *A*. The angles between the aromatic planes attached to C8, C7 and plane *A* are 29·0 (1) and 49·2 (1)° (III), 17·3 (1) and 8·8 (1)° (IV), respectively. The different ways of adjusting to steric repulsions result in significant differences between (III) and (IV) with respect to torsion angles (Table 3).

Some aromatic bond angles show deviations from 120° at the substituted atoms C12 and C18 in (IV) where smaller values are observed. This deviation from 120° is consistent with the observation that the aromatic substitution by an electron-donating group leads to an internal angle smaller than 120° on the substituted C atom (Carter, McPhail & Sim, 1966; Hope, 1969). However, the significant deviation at C9 and C15 in both (III) and (IV) may be mainly due to a steric effect since the dienone system is by no means an electron-donating group. The H6*a*···H20 and H7···H16 [2·24 (3) Å in (IV)] repulsions, for instance, compress the angle C16–C15–C20 from both sides, causing it to be substantially smaller than 120°. In the aromatic rings of (IV) most internal angles, except those at the C atoms attached to a substituent, are larger than 120° in order to compensate for the narrowing at C9 and C12, C15 and C18.

In summary, (I) and (IV) are similar; they both have boat conformations in the central rings and show more expansion of the angles and less rotation about the bonds. In contrast, (II) is similar to (III); both exhibit chair conformations and show less expansion of the angles and more rotation about the bonds. It has not been possible to correlate these observations with the differences in bioactivity of this series of compounds. However, when further structural determinations and screening results are available, some structure–activity relationship may become apparent.

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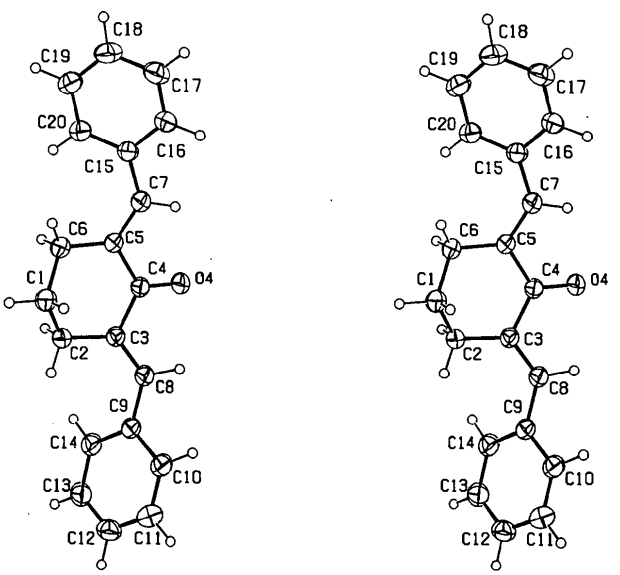

Fig. 1. Stereoscopic *ORTEP* view (Johnson, 1976) of (III) with atomic numbering.

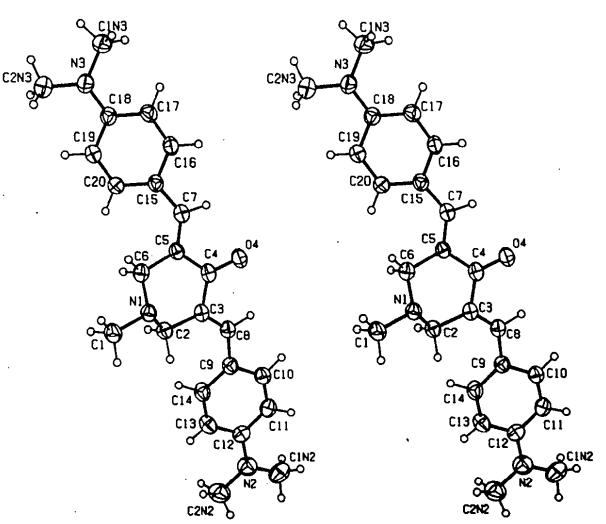

Fig. 2. Stereoscopic *ORTEP* view (Johnson, 1976) of (IV) with atomic numbering.

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Structure of a Chiral Chloropropyl Bicyclic Phosphonamide

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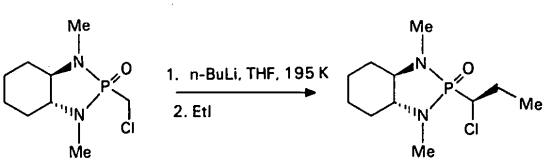
Abstract. 2-(1-Chloropropyl-2,3,3a,4,5,6,7,7a-octahydro-1,3-dimethyl-1*H*-1,3,2-benzodiazaphosphole 2-oxide, $C_{11}H_{22}ClN_2OP$, $M_r = 264{\cdot}74$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}862\ (3)$, $b = 9{\cdot}492\ (4)$, $c = 18{\cdot}976\ (7)$ Å, $V = 1416{\cdot}11$ Å^3, $D_x = 1{\cdot}242$ Mg m^{-3}, $Z = 4$, $\lambda(\text{Cu } K\bar{\alpha}) = 1{\cdot}54178$ Å, $\mu(\text{Cu } K\bar{\alpha}) = 3{\cdot}381$ mm^{-1}, $F(000) = 568$, $T = 293$ K, $R = 0{\cdot}077$, $wR = 0{\cdot}049$ for 1554 observed reflections. The title compound contains a five-membered ring of the envelope type including a tetrahedrally coordinated P atom. A chloropropyl group and an O atom are attached to the phosphorus. The cyclohexane ring, in the chair conformation, is *trans*-fused to the five-membered ring.

Introduction. Chiral, non-racemic phosphonamides (Hanessian, Delorme, Beaudoin & Leblanc, 1985) can be obtained from chloromethyl phosphonamides by anion formation and alkylation (Hanessian & Bennani, 1989). Although alkyl phosphonamides behave similarly and the products can be rationalized based on precedents (Hanessian, Delorme, Beaudoin & Leblanc, 1984), definitive evidence for the structure of the alkylchloro phosphonamides was not available. We now report the crystal structure of the product (2) resulting from the treatment of the chloromethyl phosphonamide (1) with ethyl iodide.

It is of interest to note that very high selectivity results from this alkylation, since the product (2) has an optical purity in excess of 90%. Previously, we had also resorted to X-ray crystallography to elucidate the structure and stereochemistry of related alkyl phosphonamides (Bélanger-Gariépy, Delorme, Hanessian & Brisse, 1986).



(1) (2)

Experimental. Crystals of $C_{11}H_{22}ClN_2OP$ recrystallized from hexane solution, bounded by {001}, {010}, {100}, dimensions $0{\cdot}04 \times 0{\cdot}09 \times 0{\cdot}42$ mm. Unit-cell dimensions from 25 well centered reflections in the range $40 \le 2\theta \le 50°$. Nonius CAD-4 diffractometer, graphite-monochromatized Cu $K\bar{\alpha}$ radiation, ω–2θ scan, $\Delta\omega = (1{\cdot}00 + 0{\cdot}14\tan\theta)°$, $2\theta_{max} = 140{\cdot}0°$, $-9 \le h \le 9$, $0 \le k \le 11$, $0 \le l \le 23$. Orientation monitored every 200 measurements, the intensities of seven standard reflections (checked every hour) decreased regularly by about 15% over the duration of the data collection. This is due to the slow decomposition of the compound. 2679 measured reflections of which 1554

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