

Table 3. *Torsion angles in the ribose fragment* ($^{\circ}$)

| | A | B | C |
|---------------------|------|------|------|
| C(1)–C(2)–C(3)–O(3) | –167 | –166 | –155 |
| O(2)–C(2)–C(3)–O(3) | 69 | 72 | 54 |
| C(1)–C(2)–C(3)–C(4) | 75 | 74 | 90 |
| C(2)–C(3)–C(4)–C(5) | 177 | 176 | 179 |
| O(3)–C(3)–C(4)–O(4) | 178 | 179 | –178 |
| O(3)–C(3)–C(4)–C(5) | 57 | 56 | 60 |
| C(2)–C(3)–C(4)–O(4) | –62 | –60 | –60 |
| O(4)–C(4)–C(5)–O(5) | –65 | –64 | –67 |
| C(3)–C(4)–C(5)–O(5) | 55 | 59 | 55 |

References: A this work ($\sigma = 1^{\circ}$); B D-ribose *S,S'*-diethyl dithioacetal (Ducruix, Horton, Pascard, Wander & Prangé, 1978); C D-ribose *S,S'*-diphenyl dithioacetal (Ducruix, Horton, Pascard, Wander & Prangé, 1978).

Table 4. *Hydrogen-bond lengths* (\AA)

| | |
|----------------------------|-----------|
| O(1)···N(1) | 2.977 (5) |
| O(2)···O(3) | 2.741 (5) |
| O(2)···O(4 ⁱⁱ) | 2.736 (5) |
| O(3)···O(5 ⁱⁱ) | 2.805 (5) |
| O(4)···O(5) | 2.742 (5) |

Symmetry code: (i) $x+1, y, z$; (ii) $x, y+1, z$.

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Structure of 2,2-Dibromo-3,4-dihydro-1(2H)-naphthalenone

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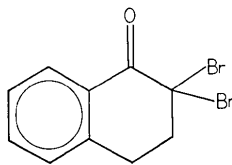
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Abstract. $C_{10}H_8Br_2O$, $M_r = 304.0$, monoclinic, $P2_1/n$, $a = 8.626$ (1), $b = 8.184$ (2), $c = 14.800$ (3) \AA , $\beta = 106.92$ (2) $^{\circ}$, $V = 999.6$ (6) \AA^3 , $Z = 4$, $D_x = 2.020$ g cm^{-3} , $\lambda(\text{Mo } K\alpha) = 0.71073$ \AA , $\mu = 85.5$ cm^{-1} , $F(000) = 584$, ambient temperature, 1013 observed reflections, $R = 0.038$. The C=O bond is essentially coplanar (3.2°) with the aromatic ring due to resonance of its π -system with that of the ring. The non-aromatic ring has a chair conformation, with C(3), the β carbon, displaced by 0.560 (9) \AA from the plane of the aromatic ring. One of the two C–Br bonds, C–Br(axial), is perpendicular to the aromatic ring. The Br(axial)–C–Br(equatorial) angle [107.1 (2) $^{\circ}$] is close to the sp^3 -hybrid angle. The Br(equatorial)–C–C(C=O) angle [109.6 (4) $^{\circ}$] is slightly larger than the Br(axial)–C–C(C=O) angle [104.0 (4) $^{\circ}$] due to repulsion of the Br(equatorial) by the carbonyl oxygen.

Introduction. α -Halocarbonyls have been used with limited success in the preparation of halohydrins irrespective of whether the reduction of α -halocarbonyls is catalytic (Augustine, 1965), nucleophilic

(Yoon & Brown, 1968), or electrophilic (Brown, 1972). Recently, Jensen, Jewett-Bronson, Hadley & French (1982) reported a high yield of aryldihaloalcohols by reduction of α,α -dihalo ketones by a variety of electrophilic reducing agents. Pertinent to this work, the reduction of the haloketone 2,2-dibromo-3,4-dihydro-1(2H)naphthalenone (2,2-dibromo- α -tetralone) with the electrophilic reducing agent diisobutylaluminium hydride gave an 84% yield of the corresponding alcohol. Extensive studies on the rearrangement of α -aryl-2,2-dihalo- α -alkanones with methylmagnesium iodide have been reported by De Kimpe, Verhe, De Buyck & Schamp (1979). The mechanism for the rearrangement proceeds *via* an initially formed carbonyl adduct, and leads to highly sterically hindered alcohols as proven by the synthesis of authentic reaction intermediates. At this department, attempts at the reduction of α -halo-carbonyls, and specifically 2,2-dihalo- α -carbonyls, seemed also to be stereospecific and in particular dependent on the disposition of the C=O bond relative to the C–X bonds (Dirania, 1979). In order to account for the steric factors involved in the reduction of

aryldihaloalkanones the determination of the absolute structure of 2,2-dibromo- α -tetralone was undertaken.



Experimental. 2,2-Dibromo- α -tetralone was prepared by direct bromination of α -tetralone in accordance with a modified Wilds procedure (Rutherford & Stevens, 1955). To an ether solution of α -tetralone (0.1 mol), bromine (0.21 mol) was added dropwise with stirring under a UV light. The product was salted out with petroleum ether (b.p. 330–350 K). Large transparent crystals suitable for X-ray diffraction work were obtained upon recrystallization from petroleum ether. Crystal dimensions center to face 0.042 (10 $\bar{1}$), 0.042 (101), 0.095 (001), 0.095 (00 $\bar{1}$), 0.165 (010), 0.160 (0 $\bar{1}$ 0) mm. Crystal volume 0.00516 mm³. Enraf-Nonius CAD-4 diffractometer, Mo K α radiation, graphite monochromator. Cell dimensions and crystal orientation by least-squares fit of setting angles of 25 reflections $\theta = 25$ –30°. One fourth of reciprocal lattice up to $h, k, \pm l$ of 9, 8, ± 15 , 1513 reflections with $2 < \theta < 22.5^\circ$, recorded by ω -2 θ scan method at scan rate 4.0–32.0° min⁻¹.

Data reduction and subsequent structure determination and refinement performed using computer programs of Enraf-Nonius SDP (Frenz & Okaya, 1980). Scattering factors those supplied internally by SDP. Data routinely corrected for background, Lorentz and polarization effects. Absorption within the crystal was found to be significant. A ψ -scan showed a change of transmission values between 20.16 and 52.09. To apply absorption corrections a crystal of dimensions 0.204 \times 0.325 \times 0.199 mm along direct crystal axes a, b, c was divided by a grid 0.01 mm in mesh size. An average decay of 6.7% occurred during data collection. The decay was anisotropic with reflections with high h index suffering most. The three reference reflections monitored for decay showed 24% reduction in intensity for 60 $\bar{2}$; 12% for 2,0,10 $\bar{0}$; and no significant reduction for 060. Decay corrections were applied to F_o by multiplying by a decay factor (1.3095–1.0000) depending on the exposure time and the reflection's h, k, l indices.

Phases determined by heavy atom (Patterson) and Fourier methods. H atoms not located and not positioned. Refinement of 1013 data with $I_o > 3.0\sigma(I_o)$ and 118 variables, anisotropic diagonal-matrix least squares on F , weights $w = 1/\sigma(|F_o|)^2$ and a fudge factor $p = 0.02$ converged with largest Δ/σ of 0.000 to $R = 0.0382$ and $wR = 0.0556$, $S = 2.635$. Highest residual peak in final difference electron density map

0.65 e \AA^{-3} . Final positional and thermal parameters with their standard deviations are listed in Table 1.*

* Lists of structure factors, anisotropic thermal parameters, mean planes and torsion angles and a stereoscopic packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42673 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>B</i> _{eq} * (Å ²) |
|-------|-------------|--------------|-------------|--|
| O | 0.2091 (9) | 0.5399 (8) | 0.5213 (4) | 5.1 (3) |
| Br(1) | 0.0475 (1) | 0.3917 (1) | 0.29689 (6) | 3.71 (4) |
| Br(2) | 0.3659 (1) | 0.5995 (1) | 0.36854 (7) | 4.79 (5) |
| C(1) | 0.2265 (9) | 0.4108 (10) | 0.4862 (6) | 3.3 (4) |
| C(2) | 0.2636 (9) | 0.4000 (10) | 0.3907 (6) | 3.0 (3) |
| C(3) | 0.3643 (9) | 0.2563 (11) | 0.3821 (6) | 3.7 (4) |
| C(4) | 0.2893 (10) | 0.0958 (10) | 0.4023 (6) | 3.8 (4) |
| C(5) | 0.2423 (10) | 0.1004 (9) | 0.4928 (6) | 3.1 (4) |
| C(6) | 0.2267 (11) | -0.0433 (13) | 0.5383 (7) | 4.1 (4) |
| C(7) | 0.1825 (10) | -0.0412 (12) | 0.6218 (7) | 4.5 (5) |
| C(8) | 0.1552 (11) | 0.1062 (12) | 0.6613 (7) | 5.0 (4) |
| C(9) | 0.1666 (10) | 0.2543 (13) | 0.6161 (6) | 3.9 (4) |
| C(10) | 0.2101 (9) | 0.2499 (11) | 0.5313 (6) | 3.0 (4) |

$$* B_{eq} = \frac{1}{3}(B_{11} + B_{22} + B_{33}).$$

Table 2. Bond lengths (Å), bond angles (°) and shortest contact distances (Å) with e.s.d.'s in parentheses

| | | | |
|-------------|-----------|------------------|-----------|
| O—C(1) | 1.205 (6) | O—C(1)—C(2) | 122.1 (5) |
| Br(1)—C(2) | 1.974 (5) | O—C(1)—C(10) | 122.6 (5) |
| Br(2)—C(2) | 1.930 (5) | C(2)—C(1)—C(10) | 115.4 (5) |
| C(1)—C(2) | 1.540 (8) | Br(1)—C(2)—Br(2) | 107.1 (2) |
| C(2)—C(3) | 1.489 (7) | Br(1)—C(2)—C(3) | 111.6 (4) |
| C(3)—C(4) | 1.533 (7) | Br(1)—C(2)—C(1) | 104.0 (4) |
| C(4)—C(5) | 1.509 (8) | Br(2)—C(2)—C(3) | 110.2 (4) |
| C(5)—C(6) | 1.381 (8) | Br(2)—C(2)—C(1) | 109.6 (4) |
| C(5)—C(10) | 1.410 (7) | C(1)—C(2)—C(3) | 114.0 (4) |
| C(6)—C(7) | 1.396 (9) | C(2)—C(3)—C(4) | 111.7 (4) |
| C(7)—C(8) | 1.390 (9) | C(3)—C(4)—C(5) | 112.9 (4) |
| C(8)—C(9) | 1.401 (8) | C(4)—C(5)—C(6) | 120.0 (5) |
| C(9)—C(10) | 1.412 (7) | C(4)—C(5)—C(10) | 121.0 (5) |
| C(10)—C(1) | 1.502 (7) | C(6)—C(5)—C(10) | 119.0 (6) |
| | | C(5)—C(6)—C(7) | 120.7 (6) |
| | | C(6)—C(7)—C(8) | 120.4 (6) |
| Br(1)⋯Br(2) | 3.141 (1) | C(7)—C(8)—C(9) | 120.4 (6) |
| Br(1)⋯O | 3.428 (4) | C(8)—C(9)—C(10) | 118.4 (6) |
| Br(2)⋯O | 2.993 (4) | C(5)—C(10)—C(9) | 121.0 (5) |
| Br(1)⋯C(3) | 2.877 (5) | C(5)—C(10)—C(1) | 121.8 (5) |
| Br(2)⋯C(3) | 2.816 (6) | C(9)—C(10)—C(1) | 117.1 (5) |

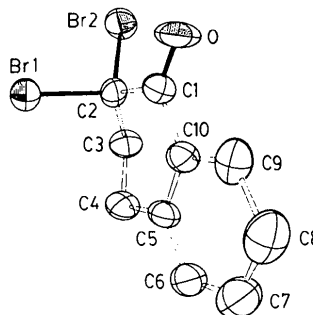


Fig. 1. ORTEP view (Johnson, 1965) of the molecular unit C₁₀H₈Br₂O showing labeling scheme.

Discussion. Bond distances and angles are given in Table 2, with the atom-labeling scheme shown in Fig. 1.

Although α -tetralone was prepared in the last century (Kipping & Hill, 1899), and its melting point (281 K) is not far from room temperature, its structure has not been reported, and consequently, no comparison of its structure with that of its dibromo-substituted analogue is possible. However, the structure of naphthalene, $C_{10}H_8$ (Abrahams, Robertson & White, 1949; Cruickshank & Robertson, 1953), and of anthraquinone, $C_{14}H_8O_2$ (Sen, 1948; Prakash, 1967) have long been known and could be used towards this end. The average aromatic C—C bond distance in this structure [1.398 (8) Å] is basically the same as that in naphthalene and in anthraquinone and in any aromatic ring. The C=O bond distance in this structure [1.205 (6) Å] is not significantly different from that in anthraquinone [1.213 (11) Å]. Furthermore, the two C=O bonds in anthraquinone are coplanar with the phenyl rings whereas in this structure, the C=O bond is slightly out of the plane of the phenyl ring (3.2°). Resonance stabilization of the C=O π -system with the phenyl ring's π -system is expected to maintain the coplanarity of the C=O and the phenyl ring. The slight deviation from coplanarity is probably due to the non-planarity of the non-aromatic ring.

Significant puckering exists in the saturated ring characteristic of sp^3 -hybridized C atoms. C(3), in particular, is 0.560 Å below the mean plane of the phenyl ring. Distances of other atoms from the mean plane of the phenyl ring are: C(1) -0.02 (1), C(2) 0.075 (9), C(3) -0.560 (9), C(4) -0.01 (1), O -0.089 (7), Br(1) 2.015 (1), Br(2) -0.689 (1) Å. The saturated ring could be thought of as having a chair conformation. The C—C bond lengths in the saturated part of the molecule (1.540–1.489 Å) are normal except that C(1)—C(2) [1.540 (8) Å] is longer than a normal sp^2 — sp^3 carbon bond, probably due to the bulky Br atoms attached to C(2).

The sum of the angles about C(1) [360.1 (5) $^\circ$] indicates the planarity of the O, C(1), C(2), C(10) fragment in agreement with an sp^2 -hybridized C(1). The Br(1)—C(2)—Br(2) angle [107.1 (2) $^\circ$] is slightly smaller than the tetrahedral angle in spite of the bulky Br atoms. This is probably due to a higher p -orbital contribution to the two hybrid orbitals directed towards the Br atoms. We are unaware of any reported structures with 2,2-dibromo- α -ketones to compare with this structure, but the structurally similar compound 2,2-dichloro-4,4-dimethylcyclohexanone (Lichanot, Petrisans, Hauw & Gaultier, 1974) also exhibits a Cl—C—Cl angle [106.6 (5) $^\circ$] which is slightly smaller than the tetrahedral angle. The same could be said for the structurally similar compound 2,2-dichloro-4-*tert*-butylcyclohexanone (Lectard, Petrisans & Hauw, 1973), where the Cl—C—Cl angle is 106.7 (5) $^\circ$. In 1,1-dichloro-2,2-bis(*p*-fluorophenyl)ethane (Smith,

Kennard & Whitnall, 1977), the Cl—C—Cl angle [107.6 (5) $^\circ$] is also less than the tetrahedral angle, although this compound is structurally different since it lacks a carbonyl group adjacent to the dihalogenated carbon. The Cl—C—Cl bond angle in 2,2-dichloro-3-phenylcyclobutanone (Hoge, Jager & Fisher, 1977), is 109.6 (6) $^\circ$, effectively equal to the tetrahedral angle, probably due to the strain caused by the four-membered carbon ring in the structure.

The C—Br bond length [av. 1.952 (5) Å] varies with the disposition of the Br atom. C(2)—Br(1)(axial) [1.974 (5) Å] is longer than C(2)—Br(2)(equatorial) [1.930 (5) Å] possibly due to closer contacts for Br(2) than for Br(1) (Table 2). The C(1)—C(2)—Br(1)(axial) bond angle [104.0 (4) $^\circ$] is smaller than the C(1)—C(2)—Br(2)(equatorial) angle [109.6 (4) $^\circ$] due to the repulsion of Br(2) by the carbonyl oxygen. The same variation in bond lengths and angles between the axial and equatorial positions is exhibited in the structurally similar compound 2,2-dichloro-4-*tert*-butylcyclohexane (Lectard *et al.*, 1973). For comparison purposes, the bromo- α -monoketone, 2 α ,4 α -dibromo-10 β -pinan-3-one (Williams, 1973), exhibits C—Br(axial) distances of 1.97 (2) and 2.03 (3) Å, while the bromo- α -alcohol, D-dibromomannitol (Simon & Sasvári, 1973), exhibits C—Br distances of 1.96 (1) and 2.08 (1) Å. It is also worth noting that the normal C(sp^2)—Br distance is about 0.09 Å shorter than C(sp^3)—Br(axial) and about 0.03 Å shorter than C(sp^3)—Br(equatorial), as is clearly shown upon comparison with the structures of 2,6-dibromonaphthalene (Chanh, Mcresse, Leroy & Geoffre, 1976), 4-bromo-4'-cyanobiphenyl and 4,4'-dibromobiphenyl (Kronebusch, Gleason & Britton, 1976).

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cis-Thymidine 3',5'-Cyclic *N,N*-Dimethylphosphoramidate Acetone Solvate, a Cyclic Nucleotide with an Axial Dimethylamino Substituent on Phosphorus

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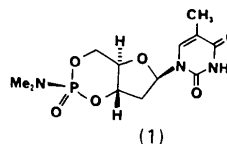
Abstract. $C_{12}H_{18}N_3O_6P.C_3H_6O$, $M_r = 389.34$, orthorhombic, $P2_12_1$, $a = 12.004$ (9), $b = 13.702$ (9), $c = 23.557$ (7) Å, $V = 3875$ (4) Å³, $Z = 8$, $D_x = 1.33$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu(\text{Mo } K\alpha) = 1.81$ cm⁻¹, $F(000) = 1648$, $T = 298$ K, $R = 0.091$ for 3306 unique data, 2531 of which were considered observed. There are two independent hydrogen-bonded (base-paired) nucleotide molecules and two acetone molecules in the asymmetric unit. The dioxaphosphorinane ring of each cyclic nucleotide adopts a distorted chair conformation with the pyramidal dimethylamino substituent axial. The pyrimidine ring exists in an *anti* conformation.

Introduction. There is considerable interest in the understanding of the effects of heteroatom substitution on the conformational properties of saturated six-membered rings. We have been concerned with the effects of various substituents on P on the solid-state conformations of 1,3,2-dioxaphosphorinanes (Day, Bentrude, Yee, Setzer, Deiters & Holmes, 1984; Warrent, Caughlan, Hargis, Yee & Bentrude, 1978; Haque, Caughlan, Hargis & Bentrude, 1970) and 1,3,2-oxazaphosphorinanes (Holmes, Day, Setzer, Sopchik & Bentrude, 1984; Bentrude, Day, Holmes, Quin, Setzer, Sopchik & Holmes, 1984; Newton, Pantaleo, Bentrude & Chandrasekaran 1982; Bajwa, Bentrude, Pantaleo, Newton & Hargis, 1979). Recently we have extended our

studies to include the conformational analysis of neutral derivatives of nucleoside 3',5'-cyclic monophosphates using NMR (Nelson, Sopchik & Bentrude, 1983; Sopchik, Bajwa, Nelson & Bentrude, 1981; Sopchik & Bentrude, 1980; Bajwa & Bentrude, 1978, 1980) and X-ray techniques (Newton, Pantaleo, Bajwa & Bentrude, 1977).

3',5'-Cyclic nucleotides play an important role in cell metabolism; adenosine 3',5'-cyclic monophosphate (cAMP) and guanosine 3',5'-cyclic monophosphate (cGMP) are important bioregulator molecules. There has been growing interest in the synthesis and biological activity of cyclic nucleotides in which the P centers are derivatized as triesters or phosphoramidates (Bajwa & Bentrude, 1978, and references therein). These derivatives may potentially act as cyclic nucleotide mimics, as antagonists of cyclic nucleotide action, or as storage forms of the natural diester nucleotides themselves (Nargeot, Nerbonne, Engels & Lester, 1983, and references therein).

This paper reports the crystal structure of *cis*-thymidine 3',5'-cyclic *N,N*-dimethylphosphoramidate, (1), a cyclic nucleotide with an axially disposed dimethylamino group on the P atom of the dioxaphosphorinane ring.



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