Fig. 1. Molecule of $C_{10}H_{18}O_2$, 50% ellipsoids (Johnson, 1965).

structure of trans-1(7)-p-menthene-2,8-diol: Scott & Richards (1971); structure of menthyl trimethyl-ammonium iodide: Gabe & Grant (1962); systematics and classification of the genus Melampodium: Stuessy (1972).

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Structure of 3'-Azido-3'-deoxythymidine, AZT

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Abstract. $C_{10}H_{13}N_5O_4$, $M_r = 267.2$, monoclinic, $P2_1$, $a = 5.716$ (3), $b = 11.998$ (8), $c = 17.658$ (10) Å, $\beta = 94.26$ (4)°, $V = 1208$ Å^3, $Z = 4$, $D_x = 1.47$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 7.5$ cm^{-1}, $F(000) = 560$, $T = 293$ K. $R = 0.060$ for 2138 unique observed $[F > 4\sigma(F)]$ reflections. The N-glycosidic torsion angles χ have values -125.9 (5) and -172.0 (5)°, in the anti range. (Molecule-A values are given first throughout.) The sugar puckers are ^2_3T (C3'-exo/C2'-endo), with $P = 171$ (1)° and $\psi_m = 14$ (1)°, and ^4_3T (C4'-endo/C3'-exo), with $P = 213$ (1)° and $\psi_m = 11$ (1)°. The C4—C5 conformations, with $\gamma = 49.7$ (5) and 173.7 (5)°, are +sc (gauche–gauche) and ap (gauche–trans). The conformational parameters used follow the guidelines of the IUPAC–IUB Joint Commission on Biochemical Nomenclature [Pure Appl. Chem. (1983), 55, 1273–1280]. The molecules in the asymmetric unit form a hydrogen-bonded, base-paired dimer. The

bonding is as follows: N3A—0.973 Å—H3$A \cdots$ 1.790 Å$\cdots$O2B, N3$A\cdots$O2B 2.747 (8) Å, angle at H3A 167° and N3B—0.992 Å—H3$B\cdots$1.916 Å$\cdots$O2A, N3$B\cdots$O2A 2.894 (8) Å, angle at H3B 168°. The propeller twist between the bases is 5° [Wilson & Tollin (1987). Nucleosides Nucleotides, 6, 643–653].

Experimental. Crystals were obtained from aqueous solution. Space group and initial cell dimensions were obtained from Weissenberg photographs. Data were collected on a Nicolet P3 (four-circle) diffractometer in Aberdeen by RAH. The crystal had dimensions $0.6 \times 0.3 \times 0.2$ mm. Cell parameters were measured on the diffractometer using 14 reflections in the 2θ range 15–22°. Range of indices: $0 \le h \le 9$; $0 \le k \le 18$; $-26 \le l \le 26$. Data measured using $\theta/2\theta$ scans in the range $0 < 2\theta < 55°$. Standard reflections, 253 and 10$\bar{6}$, were measured every 50 reflections. No

Table 1. *Coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* $(\times 10^3)$ *for non-H atoms with e.s.d.'s in parentheses*

	x	y	z	U_{eq}†(Å²)
N1A	−5535 (7)	−8740	−1115 (2)	36 (1)
C2A	−4016 (8)	−8412 (4)	−1633 (2)	33 (1)
O2A	−2320 (6)	−7808 (3)	−1487 (2)	44 (1)
N3A	−4531 (7)	−8833 (4)	−2352 (2)	35 (1)
C4A	−6351 (8)	−9559 (4)	−2587 (3)	35 (1)
O4A	−6549 (6)	−9876 (3)	−3244 (2)	50 (1)
C5A	−7837 (8)	−9870 (4)	−1998 (3)	34 (1)
C7A	−9825 (9)	−10655 (5)	−2189 (3)	47 (1)
C6A	−7376 (8)	−9438 (4)	−1302 (3)	36 (1)
C1'A	−5202 (9)	−8334 (5)	−324 (2)	38 (1)
C2'A	−7176 (10)	−7609 (5)	−81 (3)	44 (1)
C3'A	−7262 (9)	−7888 (5)	750 (3)	42 (1)
N3'A	−5501 (10)	−7143 (5)	1173 (3)	66 (1)
N4'A	−5465 (8)	−7225 (4)	1872 (3)	45 (1)
N5'A	−5314 (9)	−7217 (5)	2511 (3)	61 (1)
C4'A	−6463 (9)	−9113 (5)	804 (3)	38 (1)
C5'A	−8473 (10)	−9939 (5)	795 (3)	50 (1)
O5'A	−10154 (7)	−9727 (4)	193 (2)	63 (1)
O4'A	−5105 (6)	−9289 (3)	154 (2)	40 (1)
N1B	1614 (6)	−7291 (3)	−3854 (2)	34 (1)
C2B	261 (8)	−7581 (5)	−3276 (3)	35 (1)
O2B	−1344 (6)	−8250 (3)	−3373 (2)	46 (1)
N3B	835 (7)	−7065 (4)	−2592 (2)	40 (1)
C4B	2621 (9)	−6315 (5)	−2435 (3)	39 (1)
O4B	2947 (7)	−5931 (4)	−1789 (2)	58 (1)
C5B	3941 (9)	−6025 (5)	−3070 (3)	41 (1)
C7B	5926 (10)	−5196 (5)	−2961 (3)	56 (2)
C6B	3416 (8)	−6527 (5)	−3741 (3)	38 (1)
C1'B	1098 (8)	−7893 (5)	−4586 (3)	38 (1)
C2'B	−1255 (8)	−7603 (6)	−4976 (3)	49 (1)
C3'B	−720 (9)	−7416 (5)	−5807 (3)	44 (1)
N3'B	−905 (9)	−8524 (5)	−6188 (3)	58 (1)
N4'B	−676 (8)	−8502 (4)	−6867 (3)	48 (1)
N5'B	−486 (11)	−8596 (5)	−7506 (3)	70 (2)
C4'B	1775 (9)	−6999 (5)	−5721 (3)	40 (1)
C5'B	1995 (11)	−5747 (5)	−5578 (3)	48 (1)
O5'B	4326 (7)	−5414 (4)	−5423 (2)	60 (1)
O4'B	2841 (5)	−7614 (3)	−5083 (2)	38 (1)

$$\dagger\ U_{eq}=\tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

Table 2. *Interatomic distances* (Å) *and angles* (°)

C2A–N1A	1·365 (6)	C2B–N1B	1·370 (6)
C6A–N1A	1·366 (6)	C6B–N1B	1·382 (6)
C1'A–N1A	1·478 (5)	C1'B–N1B	1·490 (6)
O2A–C2A	1·223 (6)	O2B–C2B	1·221 (6)
N3A–C2A	1·378 (6)	N3B–C2B	1·375 (6)
C4A–N3A	1·397 (6)	C4B–N3B	1·374 (7)
O4A–C4A	1·220 (6)	O4B–C4B	1·231 (6)
C5A–C4A	1·439 (7)	C5B–C4B	1·440 (7)
C7A–C5A	1·495 (7)	C7B–C5B	1·510 (8)
C6A–C5A	1·342 (6)	C6B–C5B	1·345 (7)
C2'A–C1'A	1·512 (8)	C2'B–C1'B	1·506 (7)
O4'A–C1'A	1·422 (6)	O4'B–C1'B	1·417 (6)
C3'A–C2'A	1·509 (7)	C3'B–C2'B	1·537 (7)
N3'A–C3'A	1·502 (7)	N3'B–C3'B	1·490 (8)
C4'A–C3'A	1·540 (8)	C4'B–C3'B	1·508 (7)
N4'A–N3'A	1·237 (6)	N4'B–N3'B	1·215 (7)
N5'A–N4'A	1·126 (7)	N5'B–N4'B	1·148 (8)
C5'A–C4'A	1·517 (8)	C5'B–C4'B	1·527 (8)
O4'A–C4'A	1·449 (6)	O4'B–C4'B	1·443 (6)
O5'A–C5'A	1·402 (7)	O5'B–C5'B	1·398 (7)

C6A–N1A–C2A	122·1 (4)	C6B–N1B–C2B	121·1 (4)
C1'A–N1A–C2A	119·6 (3)	C1'B–N1B–C2B	115·9 (4)
C1'A–N1A–C6A	118·3 (4)	C1'B–N1B–C6B	123·0 (4)
O2A–C2A–N1A	124·3 (4)	O2B–C2B–N1B	121·5 (4)
N3A–C2A–N1A	114·2 (4)	N3B–C2B–N1B	115·4 (4)
N3A–C2A–O2A	121·6 (4)	N3B–C2B–O2B	123·1 (4)
C4A–N3A–C2A	127·3 (4)	C4B–N3B–C2B	126·7 (4)
O4A–C4A–N3A	119·4 (4)	O4B–C4B–N3B	119·4 (5)
C5A–C4A–N3A	114·5 (4)	C5B–C4B–N3B	115·2 (4)
C5A–C4A–O4A	126·1 (4)	C5B–C4B–O4B	125·4 (5)
C7A–C5A–C4A	118·7 (4)	C7B–C5B–C4B	119·5 (5)
C6A–C5A–C4A	118·4 (4)	C6B–C5B–C4B	118·9 (5)
C6A–C5A–C7A	122·9 (4)	C6B–C5B–C7B	121·6 (5)
C5A–C6A–N1A	123·6 (4)	C5B–C6B–N1B	122·7 (5)
C2'A–C1'A–N1A	114·3 (4)	C2'B–C1'B–N1B	113·4 (4)
O4'A–C1'A–N1A	106·9 (4)	O4'B–C1'B–N1B	108·6 (4)
O4'A–C1'A–C2'A	107·0 (4)	O4'B–C1'B–C2'B	107·8 (4)
C3'A–C2'A–C1'A	103·1 (4)	C3'B–C2'B–C1'B	103·7 (4)
N3'A–C3'A–C2'A	106·4 (4)	N3'B–C3'B–C2'B	106·8 (5)
C4'A–C3'A–C2'A	104·0 (4)	C4'B–C3'B–C2'B	101·8 (4)
C4'A–C3'A–N3'A	110·6 (4)	C4'B–C3'B–N3'B	112·1 (4)
N4'A–N3'A–C3'A	114·2 (5)	N4'B–N3'B–C3'B	114·6 (5)
N5'A–N4'A–N3'A	173·9 (6)	N5'B–N4'B–N3'B	173·0 (6)
C5'A–C4'A–C3'A	113·7 (4)	C5'B–C4'B–C3'B	114·2 (5)
O4'A–C4'A–C3'A	105·3 (4)	O4'B–C4'B–C3'B	104·5 (4)
O4'A–C4'A–C5'A	110·2 (4)	O4'B–C4'B–C5'B	110·5 (4)
O5'A–C5'A–C4'A	111·4 (5)	O5'B–C5'B–C4'B	112·3 (5)
C4'A–O4'A–C1'A	110·6 (4)	C4'B–O4'B–C1'B	109·2 (3)

significant change in the intensities of these reflections was found throughout data collection. 2740 independent reflections measured, giving 2138 observed [$F >$ $4\sigma(F)$] reflections used in the refinement. The structure was solved using the *SHELXS*86 program (Sheldrick, 1986).

Blocked full-matrix refinement (on F) was carried out using the program *SHELX*76 (Sheldrick, 1976). The H atoms attached to the C atoms were included at calculated positions. Those attached to N3 and O5' were located on a difference Fourier map. All H atoms were given fixed isotropic temperature factors of 0·06 times that of the parent atom and allowed to ride on that atom. Anisotropic temperature factors were used for all non-H atoms, and the refinement converged at $R = 0·060$, $wR = 0·065$, $w = 1·6558[\sigma^2(F) +$ $0·00057F^2]$. 343 refined parameters; max. $\Delta/\sigma < 0·1$; max. and min. values of $\Delta\rho$ within $+0·33$, $-0·26$ e Å^{-3}.

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Also used were the program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978). All calculations were carried out on the Dundee University DEC-10 computer. The atomic numbering is shown in the perspective drawing (Fig. 1); final atomic
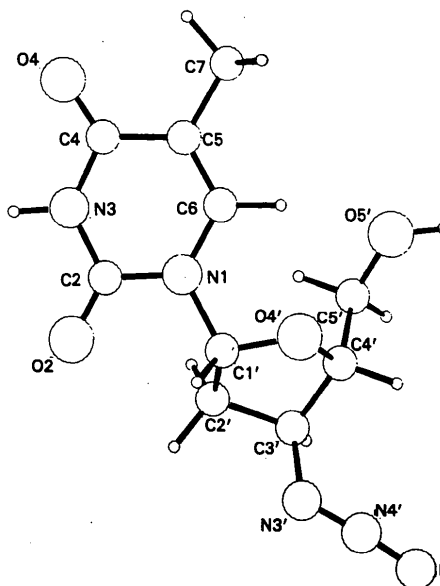

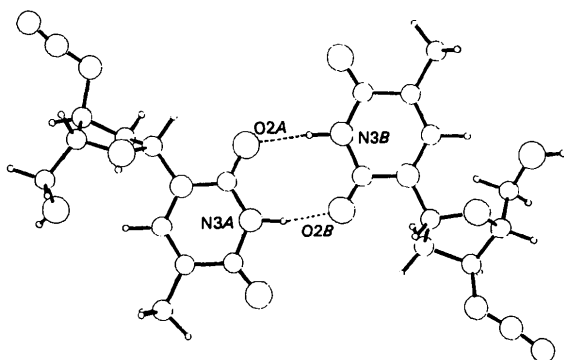Fig. 1. View of the molecule giving atomic numbering.

Fig. 2. The asymmetric unit showing the base pairing.

parameters are given in Table 1,* bond lengths and angles are given in Table 2. Fig. 2 shows the base-paired asymmetric unit.

Related literature. The related compound 3',5'-di-O-acetylthymidine has been studied recently (Wilson, Low, Tollin & Wilson, 1984). The conformations of substituted 2'-deoxyuridines are discussed by Kálmán, Czugler & Simon (1982) and by Párkányi, Kálmán, Czugler, Kovács & Walker (1987). Conformational parameters for nucleosides are discussed by Saenger (1984).

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Note added in proof: After this paper had been accepted for publication it was noted that the structure had already been solved and published independently by Gurskaya, Tsapkina, Skaptsova, Kraevskil, Lindeman & Struchkov (1986), Birnbaum, Giziewicz, Gabe, Lin & Prusoff (1987) and Camerman, Mastropaolo & Camerman (1987).

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Structure of 2,2',5,5'-Bis(tetramethylenedithio)di-1,3,4-thiadiazole

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Abstract. $C_{12}H_{16}N_4S_6$, $M_r = 408 \cdot 67$, triclinic, $P\bar{1}$, $a = 5 \cdot 731$ (1), $b = 8 \cdot 246$ (1), $c = 9 \cdot 913$ (1) Å, $\alpha = 100 \cdot 23$ (1), $\beta = 103 \cdot 77$ (1), $\gamma = 101 \cdot 33$ (1)°, $V = 433 \cdot 6$ (1) Å³, $Z = 1$, $D_x = 1 \cdot 565$, $D_m = 1 \cdot 59$ (2) g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1 \cdot 54184$ Å, $\mu = 72 \cdot 1$ cm⁻¹, $F(000) = 212$, $T = 296$ (1) K, 1787 unique reflections measured, final $R = 0 \cdot 049$ for 1585 reflections having $F_o > 6 \cdot 0\sigma(F_o)$. The centrosymmetric macrocyclic molecule consists of two 1,3,4-thiadiazole rings S-bonded at the 2 and 5 positions by two fully

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