to steric hindrance between the ester O atoms and the phenyl ring atoms. Three of the O···H contact distances involving H(1), H(8) and the four O atoms are close to 3·0 Å, but the fourth [O(16)···H(1)] is 2·548 (4) Å. Torsion angles O—C—O—C and C—C—O—C (Table 2) and least-squares planes reflect a higher degree of planarity for the ester group containing C(16) than for that containing C(18). The least-squares plane fitted to C(15)C(16)O(16)O(17)C(17) shows a maximum deviation of 0·015 (12) Å while a plane fitted to C(15)C(18)O(18)O(19)C(19) shows deviations larger than 0·1 Å; however, omission of C(19) results in a maximum deviation of 0·008 (6) Å. The angle between the two ester planes is 62·5 (6)° while the angles between these planes and C(9)C(11)C(14)C(15)C(16)-C(18) are 72·2 (6) and 48·2 (6)°.

The shortest contact distances between molecules translated along the *b* axis are of the type C···H [2·836 (2) to 3·070 (5) Å] and H···H (2·356 Å).

Based upon these results, it seems quite unlikely that simple thioxanthenylidenes bearing alkyl groups on the exocyclic double bond (*e.g.* thiothixene) will exist as sulfonium ylide vinyl analogs.

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Structure of 3′,5′-Di-*O*-acetylthymidine, $C_{14}H_{18}N_2O_7$

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Abstract. $M_r = 326{\cdot}3$, monoclinic, $P2_1$, $a = 9{\cdot}796$ (6), $b = 18{\cdot}758$ (8), $c = 8{\cdot}829$ (5) Å, $\beta = 100{\cdot}72$ (8)°, $U = 1594{\cdot}04$ Å^3, $Z = 4$, $D_x = 1{\cdot}36$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}070$ mm^{-1}, $F(000) = 688{\cdot}0$, $T = 293$ K, $R = 0{\cdot}086$ for 2232 observed reflexions. The hydrogen-bonded base-paired dimer has both molecules in the *anti* conformation, both displaying sugar pucker ^2E [C(2′)-*endo*]. The conformation about C(4′)—C(5′) for both molecules is *gauche–gauche* (g^+). A close contact of O(3′)1 with a neighbouring base ring occurs.

Introduction. This structure determination was undertaken as part of a series of studies of nucleoside analogues, including heavy-atom- and acetyl-substituted compounds. It is hoped that such determinations will aid conformational studies currently in progress.

Experimental. Crystals grown from aqueous ethanol solution. D_m not determined. Crystal approximately 0·25 × 0·35 × 0·3 mm. Cell dimensions obtained by diffractometry from crystals mounted along **a**,**b**, and **c**. Intensity data collected using the **a** and **c** crystals. Stoe STADI 2 diffractometer. Attempts to solve the structure using direct methods in *SHELX76* (Sheldrick, 1976) and *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) unsuccessful; structure finally solved using the direct-methods package *MITHRIL* (Gilmore, 1983), employing the modules *QUARTETS* and *YZARC*. *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) programs also used; all calculations performed on the Dundee University DEC-10 computer. Data collected from *a*-axis crystal in the range $h = 0$ to 8, $0 < 2\theta < 55°$, and *c*-axis crystal in the range $l = 0$ to 6, $0 < 2\theta < 55°$. Max. $\sin\theta/\lambda = 0{\cdot}77$ Å^{-1}.

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Absorption corrections not applied. Range of indices: $-13 < h < 12$, $0 < k < 24$, $0 < l < 12$. Standard reflexion measured every 100 reflexions on each layer line, random intensity variation (<5%). 11397 reflexions measured, 4049 unique, $R_{\text{int}} = 0{\cdot}041$; 2232 unique reflections $[F > 6\sigma(F)]$ used. Blocked full-matrix least-squares refinement with *SHELX*76 (Sheldrick, 1976). F magnitudes. All atoms except H refined using anisotropic temperature factors. A difference Fourier map revealed electron density at several of the expected H atom positions; for the best of these positions, the H atom fixed at that position; remaining H atoms included at calculated positions. H-atom temperature factors fixed at 1·5 times the isotropic value of the parent atom. Final refinement: 416 refined parameters, $R = 0{\cdot}086$, $wR = 0{\cdot}087$, $w = 12{\cdot}6130/[\sigma(F)^2 + 0{\cdot}000678F^2]$, $(\Delta/\sigma)_{\max} = 0{\cdot}02$; max. $\Delta\rho$ peak 0·46, min. $-0{\cdot}52$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. The numbering scheme used is shown in Fig. 1. Atomic coordinates are shown in Table 1,* with bond lengths and angles in Table 2 for the two molecules, *A* and *B*, in the asymmetric unit. These are linked (Fig. 2) as a base-paired dimer joined through hydrogen bonds between N(3) of one molecule and O(4) of the other, N(3)*A*···O(4)*B* being 2·834 (9), and N(3)*B*···O(4)*A* 2·866 (9) Å. The two hydrogen-bonded base rings are planar [max. deviations: for *A* C(4) at 0·032 (3) Å; for *B* N(1) at 0·011 (3) Å], and are tilted by 18·4 (4)° with respect to each other. In both molecules the acetyl groups are planar to within 0·01 Å. As can be seen from Table 2, the values of bond lengths and angles in molecules *A* and *B* agree well; the largest bond-length difference involves C(5)—C(7) [1·477 (11) Å in *A*, 1·531 (13) Å in *B*] and is significant. The bonds O(4′)—C(1′) [1·424 (8), 1·421 (8) Å] and O(4′)—C(4′) [1·445 (8), 1·452 (8) Å] exhibit the expected difference of 0·025 Å (de Kok, Romers, De Leeuw, Altona & van Boom, 1977), contrary to the situation found in 2′,3′,5′-tri-*O*-acetyluridine (TAU) (Low & Wilson, 1984). Also, while O(2)—C(2) [1·217 (10), 1·205 (11) Å] and O(4)—C(4) [1·217 (8), 1·221 (9) Å] exhibit bond lengths in good agreement with that expected for a conjugated C=O double bond (1·207 Å), once again we note (Low & Wilson, 1984) that the acetyl carbonyl distances are significantly shorter than this value. These bonds range from 1·144 (13) Å [O(3′)1—C(3′)1 in *B*] to 1·190 (11) Å [O(3′)1—C(3′)1 in *A*]. Other bond lengths and angles are normal.

Table 1. *Fractional atomic coordinates* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 ×10^3), *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Molecule *A*				
N(1)	2468 (5)	6102	4105 (6)	34 (1)
C(2)	3182 (7)	6392 (3)	3044 (10)	32 (2)
O(2)	2699 (5)	6419 (3)	1672 (7)	46 (1)
N(3)	4460 (5)	6648 (3)	3686 (7)	33 (2)
C(4)	5099 (7)	6643 (4)	5187 (9)	34 (2)
O(4)	6223 (5)	6925 (3)	5621 (6)	48 (1)
C(5)	4336 (7)	6279 (4)	6213 (8)	42 (2)
C(7)	4992 (8)	6189 (6)	7849 (11)	74 (4)
C(6)	3067 (7)	6049 (4)	5613 (9)	41 (2)
C(1′)	967 (6)	5958 (4)	3643 (8)	33 (2)
C(2′)	494 (7)	5212 (4)	3947 (8)	35 (2)
C(3′)	−914 (6)	5323 (3)	4318 (7)	32 (2)
O(3′)	−1895 (5)	5326 (3)	2888 (5)	41 (1)
C(3′)1	−3236 (6)	5314 (4)	3024 (10)	40 (2)
O(3′)1	−3621 (5)	5266 (3)	4222 (6)	47 (1)
C(3′)2	−4185 (7)	5375 (5)	1492 (9)	55 (2)
C(4′)	−854 (6)	6063 (4)	5039 (8)	34 (2)
C(5′)	−694 (8)	6086 (5)	6761 (10)	52 (3)
O(5′)	463 (5)	5633 (3)	7418 (7)	54 (2)
C(5′)1	474 (11)	5387 (6)	8870 (11)	70 (4)
O(5′)1	−409 (8)	5535 (5)	9556 (8)	98 (3)
C(5′)2	1713 (11)	4928 (5)	9326 (12)	83 (4)
O(4′)	300 (5)	6421 (2)	4556 (5)	38 (1)
Molecule *B*				
N(1)	9838 (5)	8212 (3)	3366 (7)	34 (1)
C(2)	8997 (7)	8033 (4)	4404 (11)	39 (2)
O(2)	9315 (5)	8149 (3)	5766 (7)	55 (2)
N(3)	7791 (6)	7710 (3)	3746 (8)	41 (2)
C(4)	7321 (7)	7536 (4)	2206 (11)	46 (2)
O(4)	6197 (6)	7247 (4)	1787 (6)	65 (2)
C(5)	8271 (8)	7731 (5)	1190 (9)	53 (3)
C(7)	7871 (10)	7534 (7)	−517 (12)	86 (4)
C(6)	9461 (7)	8043 (4)	1829 (10)	49 (3)
C(1′)	11222 (6)	8505 (3)	3959 (8)	32 (2)
C(2′)	12376 (7)	7991 (3)	3823 (8)	37 (2)
C(3′)	13544 (7)	8463 (3)	3541 (9)	37 (2)
O(3′)	14369 (5)	8643 (3)	5007 (6)	47 (1)
C(3′)1	15561 (7)	8998 (5)	4919 (13)	59 (3)
O(3′)1	15909 (6)	9179 (4)	3814 (8)	76 (2)
C(3′)2	16379 (8)	9096 (6)	6567 (11)	73 (3)
C(4′)	12809 (7)	9116 (4)	2718 (8)	38 (2)
C(5′)	12831 (8)	9136 (5)	997 (10)	56 (3)
O(5′)	12312 (6)	8456 (3)	351 (7)	57 (2)
C(5′)1	12665 (13)	8264 (6)	−956 (14)	85 (4)
O(5′)1	13375 (16)	8627 (6)	−1564 (14)	207 (7)
C(5′)2	12037 (14)	7569 (6)	−1487 (12)	96 (5)
O(4′)	11388 (4)	9084 (2)	2972 (5)	36 (1)

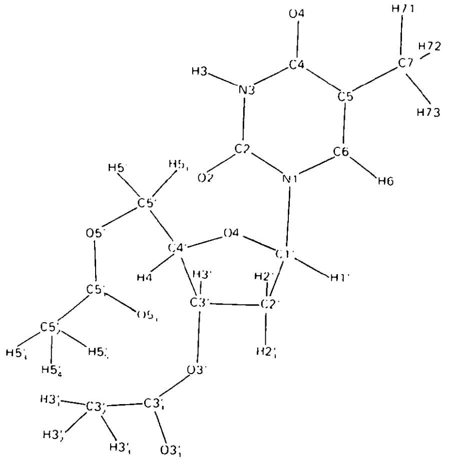

Fig. 1. Atomic numbering.

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* Lists of structure amplitudes, anisotropic thermal parameters, H-atom coordinates and Table 3 (short contacts) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39522 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

	Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*		Molecule *A*	Molecule *B*
C(2)–N(1)	1·381 (10)	1·382 (11)	C(7)–C(5)	1·477 (11)	1·531 (13)	O(3′)1–C(3′)1	1·190 (11)	1·144 (13)
C(6)–N(1)	1·355 (9)	1·375 (10)	C(6)–C(5)	1·330 (9)	1·333 (10)	C(3′)2–C(3′)1	1·496 (10)	1·536 (13)
C(1′)–N(1)	1·475 (7)	1·467 (8)	C(2′)–C(1′)	1·514 (10)	1·508 (9)	C(5′)–C(4′)	1·500 (11)	1·524 (11)
O(2)–C(2)	1·217 (10)	1·205 (11)	O(4′)–C(1′)	1·424 (8)	1·421 (8)	O(4′)–C(4′)	1·445 (8)	1·452 (8)
N(3)–C(2)	1·362 (8)	1·358 (8)	C(3′)–C(2′)	1·490 (9)	1·503 (10)	O(5′)–C(5′)	1·449 (9)	1·449 (10)
C(4)–N(3)	1·356 (9)	1·391 (11)	O(3′)–C(3′)	1·437 (7)	1·433 (8)	C(5′)1–O(5′)	1·361 (12)	1·315 (14)
O(4)–C(4)	1·217 (8)	1·221 (9)	C(4′)–C(3′)	1·523 (9)	1·535 (9)	O(5′)1–C(5′)1	1·178 (14)	1·172 (19)
C(5)–C(4)	1·448 (11)	1·454 (12)	C(3′)1–O(3′)	1·341 (8)	1·359 (9)	C(5′)2–C(5′)1	1·482 (15)	1·481 (16)
C(6)–N(1)–C(2)	120·9 (5)	121·0 (6)	C(6)–C(5)–C(7)	123·8 (7)	124·6 (8)	C(3′)2–C(3′)1–O(3′)	111·8 (7)	107·8 (8)
C(1′)–N(1)–C(2)	119·2 (5)	118·7 (6)	C(5)–C(6)–N(1)	124·8 (7)	124·9 (8)	C(3′)2–C(3′)1–O(3′)	124·3 (6)	125·9 (7)
C(1′)–N(1)–C(6)	119·1 (6)	119·9 (6)	C(2′)–C(1′)–N(1)	116·2 (5)	112·8 (5)	C(5′)–C(4′)–C(3′)	116·0 (6)	113·5 (6)
O(2)–C(2)–N(1)	122·7 (6)	122·8 (6)	O(4′)–C(1′)–N(1)	105·7 (5)	105·8 (5)	O(4′)–C(4′)–C(3′)	105·8 (5)	105·8 (5)
N(3)–C(2)–N(1)	113·5 (7)	113·8 (8)	O(4′)–C(1′)–C(2′)	106·0 (5)	105·5 (5)	O(4′)–C(4′)–C(5′)	110·2 (5)	110·3 (5)
N(3)–C(2)–O(2)	123·8 (7)	123·4 (8)	C(3′)–C(2′)–C(1′)	103·6 (5)	104·0 (5)	O(5′)–C(5′)–C(4′)	108·4 (6)	107·6 (7)
C(4)–N(3)–C(2)	128·7 (7)	128·7 (7)	O(3′)–C(3′)–C(2′)	107·6 (5)	108·0 (6)	C(5′)- O(5′)–C(5′)	116·4 (7)	117·1 (7)
O(4)–C(4)–N(3)	122·1 (7)	121·0 (8)	C(4′)–C(3′)–C(2′)	104·7 (5)	104·2 (5)	O(5′)1–C(5′)1–O(5′)	121·8 (9)	121·2 (11)
C(5)–C(4)–N(3)	114·6 (6)	114·3 (6)	C(4′)–C(3′)–O(3′)	109·4 (5)	112·0 (5)	C(5′)2–C(5′)1–O(5′)	108·4 (9)	111·0 (10)
C(5)–C(4)–O(4)	123·3 (7)	124·7 (8)	C(3′)1–O(3′)–C(3′)	115·3 (6)	114·2 (7)	C(5′)2–C(5′)1–O(5′)	129·8 (10)	127·8 (12)
C(7)–C(5)–C(4)	119·0 (6)	118·1 (7)	O(3′)1–C(3′)1–O(3′)	123·9 (6)	126·2 (8)	C(4′)–O(4′) C(1′)	110·5 (5)	110·4 (5)
C(6)–C(5)–C(4)	117·2 (7)	117·3 (8)						

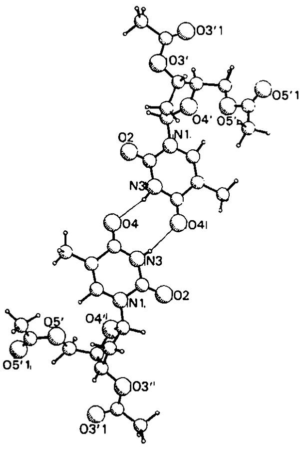

Fig. 2. View showing the hydrogen-bonded base pairing of the dimer; upper, molecule *B*; lower, *A*.

Conformation and stacking. Both molecules in the asymmetric unit have the usual *anti* conformation, with χ[C(2)–N(1)–C(1′)–O(4′)] values of −115·3 (3) and −136·3 (3)° for *A* and *B*, respectively. It is noticeable from this that molecule *A* resembles 3′,5′-di-*O*-acetyl-5-bromo-2′-deoxyuridine (ABDU) (Low, 1983) in conformation, and that molecule *B* resembles 3′,5′-di-*O*-acetyl-5-iodo-2′-deoxyuridine (AIDU) (Wilson, Low & Young, 1983). The possible growth of a dimeric co-crystal of ABDU–AIDU, which may resemble the present structure, is under investigation, and attempts are being made to grow non-dimeric crystals of the title compound. In both molecules *A* and *B*, the sugar-ring pucker is *E* [C(2′)-*endo*], with $P = 164{\cdot}3$ (5)°, $\varphi = 30{\cdot}1$ (5)° for *A*, and $P = 153{\cdot}2$ (5)°, $\varphi = 32{\cdot}3$ (5)° for *B*. The conformation about C(4′)–C(5′) is *gauche–gauche* (g^+) for both molecules. As in previous acetyl-substituted compounds investigated in this laboratory, AIDU and TAU, a close contact of an acetyl carbonyl dipole to a base ring is present. In this case, however, the close contact is between O(3′)1 of molecule *A* and the base in this molecule at $(x-1, y, z)$ and O(3′)1 of molecule *B* and the base in molecule *B* at $(x+1, y, z)$. Previously this contact has involved acetyl oxygen O(5′)1. The O atom is substantially to one side of the ring [towards N(3)] in molecule *B*, but is more centrally situated in molecule *A*, and is also closer in this case (see Table 3).* An O(3′)1 contact of this nature is more surprising than one involving O(5′)1, as the 3′ group is one atom shorter and should show less flexibility in bending towards the neighbouring ring.

O(5′)1*B temperature factor.* Upon completion of refinement, the thermal parameters of O(5′)1 in molecule *B* were found to be somewhat high (maximum values U_{11} 0·382, U_{13} 0·232 Å^2). To investigate the possibility of disorder, a Fourier map was examined in the region where O(5′)1 should be, with this latter atom removed. Two possible peaks were found, with heights in a ratio of 3:2. Upon insertion of these into a least-squares run, one of the positions refined to an occupancy of 1 at the original position of O(5′)1, the other site becoming virtually unoccupied (s.o.f. 0·004) after just two cycles of refinement. A difference Fourier map with O(5′)1 removed produced one dominant peak in the region, at a position in good agreement with the original O(5′)1 position. We conclude that the high temperature factor of O(5′)1 is a manifestation of large-amplitude thermal vibrations of this atom.

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* This table has been deposited; see deposition footnote. Closest contacts are O(3′)1*A*···C(4)*A* 3·061 (9), O(3′)1*A*···N(3)*A* 3·186 (10) and O(3′)1*B*···N(3)*B* 3·323 (9) Å.

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Acta Cryst. (1984). C**40**, 1715–1717

The Structure of Di(3,3′,4,4′-tetramethyl-2,2′,5,5′-tetraselenafulvalenium) Pentafluorosilicate,* $(C_{10}H_{12}Se_4)_2SiF_5$ at 293 and 125 K

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Abstract. (TMTSF)$_2$SiF$_5$: M_r = 1019·18, triclinic, $P\bar{1}$, Z = 1, F(000) = 475. At 293 K: a = 7·289 (2), b = 7·715 (2), c = 13·548 (4) Å, α = 83·53 (2), β = 86·20 (3), γ = 70·84 (2)°, V = 714·1 (4) Å^3, D_x = 2·370 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 102·1 cm^{-1}, $R(F)$ = 0·051, R_w = 0·032, S = 1·725 for 2951 reflections. At 125 K: a = 7·122 (2), b = 7·682 (1), c = 13·495 (3) Å, α = 83·72 (2), β = 86·90 (2), γ = 70·03 (2)°, V = 690·0 (2) Å^3, D_x = 2·452 g cm^{-3}, Mo $K\alpha$, λ = 0·71073 Å, μ = 105·8 cm^{-1}, $R(F)$ = 0·045, R_w = 0·024, S = 1·640 for 3969 reflections. No structural phase transition is observed between room temperature and 125 K. The structure is isomorphous with other highly electrically conducting (TMTSF)$_2X$ (X = monovalent anion) salts. The SiF$_5^-$ ion is disordered at both temperatures. This report represents the first structural study of a 2:1 TMTSF salt containing an anion that has a geometry intermediate between octahedral and tetrahedral.

Introduction. A considerable number of structures of electrically conducting and superconducting TMTSF salts (TMTSF = tetramethyltetraselenafulvalene) have been reported in the literature. Many of these have been of the type (TMTSF)$_2^+$.MX_6^- (MX_6^- = PF$_6^-$, AsF$_6^-$, TaF$_6^-$) and (TMTSF)$_2^+$.MX_4^- (MX_4^- = BF$_4^-$, ClO$_4^-$, BrO$_4^-$, ReO$_4^-$, FSO$_3^-$) (Thorup, Rindorf, Soling, Johannsen, Mortensen & Bechgaard, 1983), and all these structures are isomorphous, containing positively charged TMTSF sheets that are interconnected through short Se—Se contacts, with the anions located between the sheets. No salts containing an MX_5^- anion have been reported thus far. Since the pentafluorosilicate (SiF$_5^-$) salt has a geometry intermediate between MX_4^- and MX_6^- it was decided to determine the crystal structure at both room and low temperature. The electrical properties are, as yet, unknown and the structure is described below.

Experimental. Crystals prepared by electrolytic oxidation of TMTSF in 1,1,2-trichloroethane solution, in presence of (n-Bu$_4$N)(SiF$_5$). Syntex $P2_1$ automated diffractometer with graphite monochromator and a cold-stream device that allows crystal to be cooled to 125 K. At room temperature 4497 reflections including 230 Friedel pairs and at 125 K 7055 reflections including 2033 Friedel pairs. Unit-cell dimensions, in both cases, determined from least-squares refinement of setting angles of 25 reflections in 2θ range 15–20°. Further information about data collection and crystal parameters are given in Table 1. Intensities corrected for Lorentz and polarization factors and absorption. Latter correction used measured crystal dimensions and eight faces that bounded the crystal. Intensities of equivalent reflections averaged, $R_{\rm int}$ = 0·015. A starting model was easily obtained by taking the parameters for the four Se

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* Alternative nomenclature: di(4,4′,5,5′-tetramethyl-2,2′-bi-1,3-diselenolyliden)ium pentafluorosilicate.

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