

COOH and COO⁻ groups, Fig. 3. The bond lengths show clearly the difference between the acid group at C7 (O8 carries the hydrogen) and the anion at C12. The refinement showed more reasonable temperature parameters with the ammonium ion and the water molecule sited at N15 and O16 respectively than if these positions were reversed. Table 4 lists seven N...O and O...O contacts < 3.0 Å. Examination of angles and H-atom positions suggests that all of these are genuine hydrogen bonds, connecting the fragments into helices about the threefold axis. There are no other similar contacts < 3.3 Å. The angles between the normals to the planes of the carboxylate groups and the plane C1, C3, C4, C5 of the five-membered ring are 30.5 (5)° C(7) and 47.6 (6)° C(12).

References

- BARNES, J. C. & PATON, J. D. (1982). *Acta Cryst.* **B38**, 1588–1591.
 BARNES, J. C. & PATON, J. D. (1984). *Acta Cryst.* **C40**, 1809–1812.
 BARNES, J. C. & PATON, J. D. (1988). *Acta Cryst.* **C44**, 758–760.
 BUERGER, M. J. (1942). In *X-ray Crystallography*, pp. 498–504. New York: John Wiley.
 GOEBEL, W. F. & NOYES, W. A. (1923). *J. Am. Chem. Soc.* **45**, 3064–3070.
 MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
 ROBERTS, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

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Structure and Conformation of 5-Bromo-2',3'-dideoxyuridine

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Abstract. C₉H₁₁BrN₂O₄, *M_r* = 291.11, monoclinic, *P*2₁, *a* = 11.307 (1), *b* = 5.954 (1), *c* = 15.829 (2) Å, β = 93.25 (1)°, *V* = 1063.90 Å³, *Z* = 4, *D_x* = 1.82 g cm⁻³, λ(Cu *Kα*) = 1.54184 Å, μ = 53.58 cm⁻¹, *F*(000) = 584, *T* = 295 K, *R* = 0.034 for 1927 observed reflections [*I* > 3σ(*I*)]. The crystal structure contains two independent molecules forming a dimer linked by a pair of N3—H...O2 hydrogen bonds; the crystal structure is stabilized by four additional hydrogen bonds. Two of these are internal C6—H6...O5' hydrogen bonds, one in molecule *A* and another in molecule *B*. These two molecules exhibit two different conformations; their sugar ring puckers are 2'-*endo*-3'-*exo* for molecule *A* and 3'-*endo*-2'-*exo* for molecule *B*. The C1'—N1 distance, the χ_{CN} torsion angle and the glycosidic conformation are 1.464 (8) Å, -130.0° and -anticlinal for molecule *A* and 1.506 (8) Å, -168.9° and -antiperiplanar for molecule *B*, respectively.

Introduction. Some selected modification of the chemical structure of nucleosides has produced anticancer, antiviral and antibacterial agents (Bloch, 1975; Prusoff, Cheng & Neenan, 1973; Hamor, O'Leary & Walker, 1978). The 5-substituted uracils

and the corresponding nucleosides have attracted attention because of their antiviral activity (Sharma & Bobek, 1975; Cheng, Domin, Sharma & Bobek, 1976). The crystal structure of 5-iodo-2'-deoxyuridine has been studied by Camerman & Trotter (1965). The 2',3'-dideoxy nucleoside derivatives have also been studied as potential antibiotics and antiviral agents (Atkinson, Deutcher, Kornberg, Russell & Moffatt, 1969; Chu, Schinazi, Ahn, Ullas & Gu, 1989). We are studying a series of 2',3'-dideoxy nucleosides in order to delineate the structure-function relationship of antiviral compounds (Parthasarathy & Kim, 1988). In this connection, we have studied the crystal structure of 5-bromo-2',3'-dideoxyuridine (BrddU); no study on the biological activity of this compound seems to have been carried out. However, this molecule also shows the same ranges of conformations (Parthasarathy & Kim, 1988; Van Roey, Salerno, Chu & Schinazi, 1989; Low, Tollin, Howie & Wilson, 1988) formed for AZT (3'-azido-3'-deoxythymidine) [several independent studies have been published on the crystal structure of AZT; the earliest one seems to be that of Gurskaya, Tsapkina, Skaptsova, Kracvskii, Lindeman & Struchkov (1986)] and other compounds which are active against HIV-1 in peripheral blood mononuclear cells.

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Experimental. 5-Bromo-2',3'-dideoxyuridine (BrddU) was obtained from Aldrich Chemical Company in the form of transparent needles. The crystal chosen had dimensions $0.80 \times 0.15 \times 0.10$ mm. Unit-cell dimensions were determined by a least-squares refinement of the 2θ angles for 20 reflections with θ in the range $7\text{--}18^\circ$. Data were collected on an Enraf-Nonius CAD-4 diffractometer with Ni-filtered Cu $K\alpha$ radiation using the $\omega/2\theta$ scan technique with variable scan speed ($0.74\text{--}6.67^\circ \text{min}^{-1}$). Intensities of three standard reflections (700, 400, $\bar{1}\bar{3}\bar{2}$) were monitored: the total change in intensity was less than -3% as judged from $|F_{\text{obs}}|$ as a function of exposure time. No decay correction was applied. 4908 intensity data collected to $2\theta_{\text{max}} = 154^\circ$; $-14 \leq h \leq 14$, $0 \leq k \leq 7$, $-19 \leq l \leq 19$; corrected for Lorentz, polarization and absorption effects. Intensities of three reflections at $\chi \approx 90^\circ$ were measured for values of φ from 0 to 360° in steps of 10° and the resultant curve of transmissions as a function of φ was used to calculate the anisotropy of absorption for all reflections. The max. and the min. transmission factors were 0.99 and 0.82, respectively, and the average was 0.91. Equivalent reflections were averaged ($R_{\text{symm}} = 0.034$) to yield 2478 unique reflections. 1927 reflections with $I > 3\sigma(I)$ were used for all subsequent calculations. The structure was solved by direct methods using *SHELXS86* (Sheldrick, 1986) which revealed the positions of all the 32 non-H atoms. The remaining atoms were found from difference Fourier maps. Refinement by full-matrix least-squares calculations (on F) using individual anisotropic thermal parameters for non-H atoms with weights based on counting statistics reduced R to 0.059. A difference map calculated at this stage showed electron-density maxima of $0.3\text{--}0.6 \text{ e } \text{\AA}^{-3}$ in positions expected for the H atoms which, when included in the refinement using individual anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms (377 parameters including scale factor), resulted in $R = 0.034$ and $wR = 0.044$. The quantity minimized was $\sum w[|F_{\text{obs}}| - (1/k)|F_{\text{cal}}|]^2$, where $w = 1/\sigma^2(|F_{\text{obs}}|) = 4|F_{\text{obs}}|^2/\sigma^2(|F_{\text{obs}}|^2)$ and $\sigma(|F_{\text{obs}}|) = [\sigma^2(I) + 0.05 \times I^2]^{1/2}/Lp$, where k is the scale factor and $\sigma(I)$ is the standard deviation of intensity I ; shift/e.s.d. in the last cycle of refinement is 0.1 for non-H atoms and goodness of fit, $S = 1.29$. The strongest peaks in the final difference map had densities of 0.52 and $-0.60 \text{ e } \text{\AA}^{-3}$ near the Br atoms; the rest of the electron density excursions were less than $\pm 0.30 \text{ e } \text{\AA}^{-3}$. Computer programs used in this study were from the Enraf-Nonius (1979) *Structure Determination Package* and *ORTEPII* (Johnson, 1965). All calculations were carried out on a MicroVAXII. The atomic scattering factors are as given in Enraf-Nonius (1979) *Structure Determination Package* and

correspond to the values given in *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. The different conformations of molecules *A* and *B* and the atomic numbering are shown in Fig. 1. Atomic parameters are given in Table 1* and bond lengths and angles are given in Table 2. Both molecules *A* and *B* exhibit *anti* conformations across the glycosyl bond (C1'—N); χ_{CN} (O4'—C1'—N1—C2) = -130.04° ($-$ anticlinal) and -168.87° ($-$ antiperiplanar) respectively, for *A* and *B* (IUPAC-IUB Joint Commission on Biochemical Nomenclature, 1983). Molecule *A* has an unsymmetrical twist $2'$ -endo- $3'$ -exo, with major $2'$ -endo and minor $3'$ -exo sugar puckering mode, 2T_3 , with g^+ conformation ($+$ synclinal) about the C4'—C5' bond; molecule *B* also has an unsymmetrical twist, but with major $3'$ -endo and minor $3'$ -exo sugar puckering mode, $3'$ -endo- $2'$ -exo, 3T_2 , with g^+ conformation about the C4'—C5' bond. The values of the pseudorotation parameters, ν_m and P (Altona & Sundaralingam, 1972) and the conformation angle C3'—C4'—C5'—O5' are 37.09 , 167.17 , 61.41° for *A* and 37.74 , 7.53 , 62.02° for *B* respectively. The structure exhibits two internal C6—H6...O5' hydrogen bonds, one in molecule *A* and another in molecule *B* (Fig. 1). Molecules *A* and *B* are associated by two hydrogen bonds, N3—H3...O2, N3—H3 0.63 (7), H3...O2($1-x$, $\frac{3}{2}+y$, $1-z$) 2.29 (7) \AA , N3—H3...O2 152 (8) $^\circ$ for N3—H3 of molecule *A* to O2 of molecule *B*, and N3—H3 1.02 (7), H3...O2($1-x$, $-\frac{3}{2}+y$, $1-z$) 1.90 (7) \AA , N3—H3...O2 165 (5) $^\circ$ for N3—H3 of

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53921 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

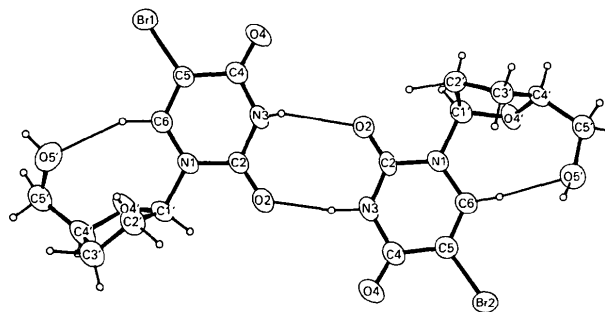


Fig. 1. Illustration of the conformational differences between molecule *A* and molecule *B* and the formation of a dimer of the two independent molecules, *A* and *B*, by utilizing two hydrogen bonds. Note the internal C6—H6...O5' hydrogen bond for molecule *A* and molecule *B*. The H6...O5 distance and the C6—H6...O5 angles are 2.28 (6) \AA and 170 (5) $^\circ$ for molecule *A* and 2.21 (6) \AA and 166 (6) $^\circ$ for molecule *B*.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters with *e.s.d.*'s in parentheses
$$B_{eq} = (4/3) \sum_i \sum_j \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
Br1	0.46752 (7)	0.241	0.57240 (4)	4.0 (1)
N1A	0.4793 (5)	0.650 (1)	0.7826 (3)	3.2 (1)
C2A	0.3969 (5)	0.818 (1)	0.7652 (4)	3.2 (1)
N3A	0.3335 (5)	0.797 (1)	0.6892 (3)	3.2 (1)
C4A	0.3448 (5)	0.639 (1)	0.6271 (4)	3.1 (1)
C5A	0.4361 (5)	0.471 (5)	0.6494 (4)	3.1 (1)
C6A	0.4965 (5)	0.484 (1)	0.7266 (4)	3.1 (1)
O2A	0.3792 (4)	0.970 (1)	0.8147 (3)	4.6 (1)
O4A	0.2882 (4)	0.643 (1)	0.5606 (3)	4.2 (1)
C1'A	0.5509 (6)	0.658 (2)	0.8626 (4)	3.6 (1)
C2'A	0.5268 (6)	0.460 (2)	0.9216 (4)	4.2 (2)
C3'A	0.6464 (6)	0.439 (2)	0.9690 (4)	4.4 (2)
C4'A	0.7337 (6)	0.494 (2)	0.9063 (4)	3.9 (2)
O4'A	0.6713 (4)	0.631 (1)	0.8432 (3)	4.5 (1)
C5'A	0.7886 (6)	0.298 (2)	0.8632 (4)	5.2 (2)
O5'A	0.7014 (5)	0.175 (1)	0.8180 (4)	6.4 (2)
Br2	0.95680 (8)	0.3467 (2)	0.08536 (5)	4.98 (2)
N1B	0.9303 (4)	-0.022 (1)	0.3022 (3)	3.0 (1)
C2B	0.8488 (5)	-0.187 (1)	0.2813 (4)	2.9 (1)
N3B	0.7996 (4)	-0.179 (1)	0.2014 (3)	3.4 (1)
C4B	0.8286 (6)	-0.035 (1)	0.1353 (4)	3.7 (1)
C5B	0.9135 (6)	0.134 (1)	0.1635 (4)	3.5 (1)
C6B	0.9594 (5)	0.139 (1)	0.2429 (4)	3.0 (1)
O2B	0.8237 (4)	-0.326 (1)	0.3326 (3)	4.1 (1)
O4B	0.7795 (5)	-0.055 (1)	0.0655 (3)	4.7 (1)
C1'B	0.9872 (5)	-0.022 (1)	0.3906 (4)	3.2 (1)
C2'B	1.0721 (6)	-0.212 (1)	0.4037 (4)	3.7 (1)
C3'B	1.1906 (5)	-0.106 (1)	0.3822 (4)	3.3 (1)
C4'B	1.1780 (5)	0.129 (1)	0.4176 (4)	3.1 (1)
O4'B	1.0492 (3)	0.1783 (9)	0.4053 (3)	3.12 (9)
C5'B	1.2474 (6)	0.308 (1)	0.3769 (4)	3.8 (1)
O5'B	1.2165 (4)	0.342 (1)	0.2907 (3)	4.4 (1)

molecule *B* to O2 of molecule *A* (Fig. 1). Fig. 1 illustrates the formation of a dimer of the two independent molecules (*A* and *B*) of BrddU. In addition, O5' of molecule *A* has a hydrogen bond with O5' of molecule *B*, O5'A—HO5'A 0.91 (8), HO5'A...O5'2(2 - *x*, -½ + *y*, 1 - *z*) 1.91 (8) Å, O5'A—HO5'A...O5'2 169 (7)° and O5'B hydrogen bonds to O4'A, O5'B—HO5'B 1.21 (12), HO5'B...O4'A(2 - *x*, -½ + *y*, 1 - *z*) 1.64 (12) Å, O5'B—HO5'B...O4'A 165 (9)°. Bases of molecules *A* and *B* are stacked with 3.4 Å between the uracil planes (Fig. 2). The base stacking does not extend beyond dimers. The stacking bases *A* and *B* are tilted by 16.92°. The Br1...Br1 distance between related molecules of *A* is 3.85 Å; for Br2...Br2 this distance is 4.17 Å (Fig. 2). The molecular dimensions of *A* and *B*, in general, agree well with one another and with other nucleosides (Saenger, 1983). The most pronounced difference occurs for the N1—C1' bonds [1.464 (8) for molecule *A* and 1.506 (8) Å for molecule *B*]; such pronounced differences in the N1—C1' bond distances are not uncommon in nucleosides that have different glycosyl conformations (Lo, Shefter & Cochran, 1975; Leeuw, Haasnoot & Altona, 1980). The C4'—O4' bond distance is appreciably longer than the C1—O4' bond distance, especially in molecule *B*; this is due to the well known bond-shortening anomeric effect (Berman, Chu & Jeffrey, 1967; Wolfe, Whangbo & Mitchell, 1979).

In conclusion, the crystal structure of BrddU has been determined from X-ray diffraction studies; it shows that the asymmetric unit contains two molecules. The crystal structure and molecular conformations are typical of those for antiviral and antibacterial agents that are dideoxynucleosides (Van Roey, Salerno, Chu & Schinazi, 1989).

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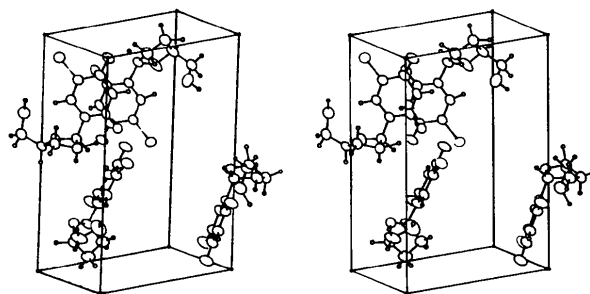


Fig. 2. Stereoview of the packing of the molecules in the unit cell. Molecule *A* stacks on molecule *B* (1 - *x*, ½ + *y*, 1 - *z*); similarly, molecule *B* stacks on molecule *A* (1 - *x*, ½ + *y*, 1 - *z*). *a* is horizontal, *c* is vertical and *b* is into the plane of the paper.

Table 2. Selected bond lengths (Å) and angles (°) with *e.s.d.*'s in parentheses

	Molecule <i>A</i>	Molecule <i>B</i>
Br—C5	1.884 (7)	1.854 (8)
N1—C2	1.385 (9)	1.373 (9)
C2—O2	1.223 (9)	1.205 (8)
C2—N3	1.369 (8)	1.353 (8)
N3—C4	1.372 (9)	1.405 (9)
C4—O4	1.201 (7)	1.214 (8)
C4—C5	1.463 (10)	1.446 (11)
C5—C6	1.368 (9)	1.333 (8)
C6—N1	1.348 (10)	1.394 (9)
N1—C1'	1.464 (8)	1.506 (8)
C1'—C2'	1.537 (12)	1.493 (11)
C1'—O4'	1.423 (8)	1.395 (9)
C2'—C3'	1.515 (10)	1.537 (9)
C3'—C4'	1.476 (10)	1.519 (11)
C4'—O4'	1.441 (9)	1.486 (7)
C4'—C5'	1.505 (13)	1.491 (11)
C5'—O5'	1.392 (11)	1.404 (8)
Br—C5—C4	119.8 (5)	117.8 (6)
Br—C5—C6	120.9 (9)	120.7 (6)
C2—N1—C6	121.2 (7)	120.6 (6)
N1—C2—N3	114.9 (6)	115.9 (6)
C2—N3—C4	128.7 (6)	127.9 (7)
N3—C4—C5	112.7 (6)	112.0 (7)
C4—C5—C6	119.1 (6)	121.4 (7)
O4—C4—C5	124.0 (7)	127.4 (7)
O4—C4—N3	123.0 (7)	120.3 (7)
C5—C6—N1	122.9 (7)	121.7 (7)
N1—C2—O2	122.7 (7)	120.5 (6)
N3—C2—O2	122.2 (7)	123.5 (7)
C6—N1—C1'	120.0 (6)	121.3 (6)
C2—N1—C1'	118.7 (7)	118.0 (6)
N1—C1'—C2'	113.0 (7)	111.5 (6)
N1—C1'—O4'	107.2 (6)	109.8 (6)
C2'—C1'—O4'	104.4 (7)	108.1 (7)
C1'—C2'—C3'	100.4 (7)	102.6 (7)
C2'—C3'—C4'	105.0 (7)	101.3 (7)
C3'—C4'—O4'	105.8 (7)	104.1 (6)
C3'—C4'—C5'	116.1 (8)	115.9 (7)
C4'—C5'—O5'	110.0 (8)	114.3 (7)

References

- ALTONA, C. & SUNDARALINGAM, M. (1972). *J. Am. Chem. Soc.* **94**, 8205–8212.
- ATKINSON, M. R., DEUTCHER, M. F., KORNBERG, A., RUSSELL, A. F. & MOFFATT, J. G. (1969). *Biochemistry*, **8**, 4897–4904.
- BERMAN, H. M., CHU, S. S. C. & JEFFREY, G. A. (1967). *Science*, **157**, 1576–1577.
- BLOCH, A. (1975). *Ann. N. Y. Acad. Sci.* **255**, 1–610.
- CAMERMAN, N. & TROTTER, J. (1965). *Acta Cryst.* **18**, 203–211.
- CHENG, Y. C., DOMIN, B. A., SHARMA, R. A. & BOBEK, M. (1976). *Antimicrob. Agents Chemother.* **10**, 119–122.
- CHU, C. K., SCHINAZI, R. F., AHN, M. K., ULLAS, G. V. & GU, Z. P. (1989). *J. Med. Chem.* **32**, 612–617.
- Enraf–Nonius (1979). *Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.
- GURSKAYA, G. V., TSAPKINA, E. N., SKAPTSOVA, N. V., KRACVSKII, A. A., LINDEMAN, S. V. & STRUCHKOV, YU. T. (1986). *Dokl. Akad. Nauk SSSR*, **291**, 854–859.
- HAMOR, T. A., O'LEARY, M. K. & WALKER, R. T. (1978). *Acta Cryst.* **B34**, 2799–2802.
- IUPAC–IUB JOINT COMMISSION ON BIOCHEMICAL NOMENCLATURE (1983). *Eur. J. Biochem.* **131**, 9–15.
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- LEEUW, H. P. M., HAASNOOT, C. A. G. & ALTONA, C. (1980). *Isr. J. Chem.* **20**, 108–126.
- LO, A., SHEFTER, E. & COCHRAN, T. G. (1975). *J. Pharm. Sci.* **64**, 1707–1710.
- LOW, J. N., TOLLIN, P., HOWIE, R. A. & WILSON, H. R. (1988). *Acta Cryst.* **C44**, 2109–2111.
- PARTHASARATHY, R. & KIM, H. (1988). *Biochem. Biophys. Res. Commun.* **151**, 351–358.
- PRUSOFF, W. H., CHENG, Y. C. & NEENAN, J. P. (1973). *Proc. Int. Congr. Chemother.* **2**, 881–888.
- SAENGER, W. (1983). *Principles of Nucleic Acid Structure*. New York: Springer-Verlag.
- SHARMA, R. A. & BOBEK, M. (1975). *J. Org. Chem.* **40**, 2377–2379.
- SHELDRIK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Gottingen, Germany.
- VAN ROEY, P., SALERNO, J. M., CHU, C. K. & SCHINAZI, R. F. (1989). *Proc. Natl Acad. Sci. USA*, **86**, 3929–3933.
- WOLFE, S., WHANGBO, H.-H. & MITCHELL, D. J. (1979). *Carbohydrate Res.* **69**, 1–26.

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9-(Cyclohepta-2,4,6-trien-1-yl)xanthen-9-ol. X-ray Crystal Structure and Computationally Optimized Structures Compared

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Abstract. The structure of the title compound (1) has been determined experimentally from X-ray data. This is compared with optimized structures computed with semi-empirical molecular orbital (*MNDO*, *AM1*, *PM3*) and molecular mechanics methods. $C_{20}H_{16}O_2$, $M_r = 288.35$, monoclinic, $P2_1/n$, $a = 12.690$ (3), $b = 6.140$ (1), $c = 19.660$ (4) Å, $\beta = 106.74$ (2)°, $V = 1466.9$ Å³, $Z = 4$, $D_x = 1.31$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å, $\mu = 0.8$ cm⁻¹, $F(000) = 608$, $T = 294$ (1) K, $R = 0.047$ for 1725 unique observed reflections. Although the molecules in the solid-state structure are linked by hydrogen bonds, there is little difference between the calculated gas-phase structures and that observed in the crystal.

Introduction. We have recently reported the synthesis of 9-(cyclohepta-2,4,6-trien-1-yl)xanthen-9-ol (1) (Badejo, Karaman, Lee, Lutz, Mamanta & Fry, 1989), together with some of its interesting chemical behavior (Badejo, Karaman & Fry, 1989; Karaman, Badejo & Fry, 1989). We have now determined its structure from both a single-crystal X-ray diffraction

study and from computations using semi-empirical molecular orbital and molecular mechanics energy-minimized methods.

Experimental.

X-ray

The title compound was obtained as pale-yellow elongated plates from THF/pentane. A crystal having approximate dimensions of 0.25 × 0.20 × 0.10 mm was mounted in a glass capillary. Preliminary examination and data collection were performed with Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å) on an Enraf–Nonius CAD-4 diffractometer. Cell constants were obtained from the setting angles of 25 reflections in the range $9 < \theta < 15^\circ$. As a check on crystal quality, ω -scans of several intense reflections were measured; the width at half-height was 0.20° indicating good crystal quality. From the systematic absences of $h0l$ $h+l = 2n+1$, $0k0$ $k = 2n+1$ the space group was determined to be $P2_1/n$. Data were collected ($\omega-2\theta$ scans) to a maximum 2θ of 60.0°.