Related literature. See Cromer, Hall, Lee & Ryan (1988) for further triazole and small explosive molecule references.

References

CROMER, D. T., HALL, J., LEE, K.-Y. & RYAN, R. R. (1988). *Acta Cryst.* C**44**, 1144–1147, 2206–2208.

CROMER, D. T. & STORM, C. (1991). C**47**, 2476–2478.

GARCIA, E. & LEE, K.-Y. (1992). *Acta Cryst.* C**48**, 1682–1683.

LARSON, A. C. (1969). In *Crystallographic Computing*, edited by F. R. AHMED, pp. 291–296. Copenhagen: Munksgaard.

LEE, K.-Y. & STORM, C. (1990). Report LA-11907-MS. Los Alamos National Laboratory, Los Alamos, NM 87545, USA.

SHELDRICK, G. M. (1990). *SHELXTL-Plus*. Siemens Analytical X-ray Instruments, Madison, Wisconsin, USA.

Acta Cryst. (1992). C**48**, 1685–1686

Structure of 4,5-Di-*O*-acetyl-6-*S*-acetyl-2,3-di-*S*-ethyl-2,3,6-trithio-D-allose Diethyl Dithioacetal

BY V. DIVJAKOVIĆ

Institute of Physics, Faculty of Sciences, Trg D. Obradovića 4, *University of Novi Sad*, 21000 *Novi Sad, Yugoslavia*

D. MILJKOVIĆ

Institute of Chemistry, Faculty of Sciences, Trg D. Obradovića 5, *University of Novi Sad*, 21000 *Novi Sad, Yugoslavia*

S. LAJŠIĆ

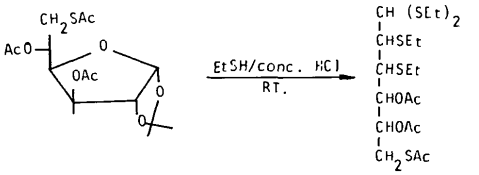
Institute for Microbiological Processes and Applied Chemistry, Faculty of Technology, Bulevar Avnoj-a 1, *University of Novi Sad*, 21000 *Novi Sad, Yugoslavia*

AND U. KLEMENT

Institut für Anorganische Chemie, Universität Regensburg, Universitätsst. 31, 8400 *Regensburg, Germany*

(*Received* 16 *October* 1991; *accepted* 14 *January* 1992)

Abstract. $C_{20}H_{36}O_5S_5$, $M_r = 516.82$, monoclinic, $P2_1$, $a = 8.670$ (1), $b = 17.149$ (1), $c = 9.224$ (1) Å, $\beta = 95.88$ (1)°, $V = 1364.2$ (3) Å^3, $Z = 2$, $D_m = 1.26$, $D_x = 1.27$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 40.8$ cm^{-1} $F(000) = 552.0$, $T = 296$ K, final $R = 0.066$, $wR = 0.065$ for 3478 observed reflections. The purpose of the structure determination was to establish the position of the substituents and the molecular conformation of the title compound (2) obtained by acid catalyzed ethanethiolysis of 3,5-di-*O*-acetyl-6-*S*-acetyl-1,2-isopropylidene-6-thio-α-D-glucofuranose (1). Since the product (2) was planned as a starting synthone in a sequence of reactions, its stereochemistry was of essential importance to us.



(1) (2)

Experimental. The compound (2) was isolated as the only reaction product in almost quantitative yield. A colourless plate-like crystal, 0.16 × 0.24 × 0.44 mm, was mounted on an Enraf–Nonius CAD-4 diffractometer, and data collected with monochromated Cu $K\alpha$ radiation and $\omega/2\theta$ scans. 4650 reflections were measured ($-10 \le h \le 10$; $-20 \le k \le 20$; $0 \le l \le 10$) to $2\theta_{max} = 60°$. Three check reflections showed no significant intensity variation. 4285 unique reflections ($R_{int} = 0.019$) with $I > 3\sigma(I)$ were used for structure solution. Data were corrected for Lorentz and polarization effects, as well as for absorption (*DIFABS*; Walker & Stuart, 1983), maximum and minimum transmission 1.00 and 0.83. Cell constants were refined from $\pm\theta$ values of 25 reflections in the range 11–21°. An extinction correction was not necessary. Structure solution was by direct methods (Sheldrick, 1986), and refinement was by the full-matrix least-squares method based on F; $w = 1/\sigma^2$ (Sheldrick, 1976). Final $R = 0.066$, $wR = 0.065$ for 3478 observations and 272 variables in the last cycle. All non-H atoms were anisotropically refined; H

0108-2701/92/091685-02$06.00

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($Å^2 \times 10^3$) *for the non-*H *atoms with e.s.d.'s in parentheses*

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
S1	2781 (3)	2331*	1242 (3)	94 (1)
S'1	2485 (3)	3999 (2)	2151 (3)	84 (1)
S2	5597 (3)	3142 (2)	3512 (3)	81 (1)
S3	1364 (2)	3009 (2)	5883 (3)	76 (1)
S6	7695 (3)	2412 (2)	8186 (3)	82 (1)
O4	4227 (6)	3843 (3)	7731 (6)	67 (2)
O43	6401 (12)	4449 (6)	7348 (11)	175 (4)
O5	4499 (6)	1833 (3)	6723 (5)	58 (1)
O53	3225 (7)	1116 (4)	8228 (7)	83 (2)
O63	7194 (7)	929 (4)	8568 (9)	103 (2)
C1	2447 (9)	2998 (5)	2701 (8)	65 (2)
C2	3700 (8)	2858 (5)	4037 (8)	57 (2)
C3	3298 (8)	3308 (4)	5415 (7)	52 (2)
C4	4510 (9)	3227 (4)	6724 (8)	55 (2)
C5	4446 (9)	2492 (5)	7684 (8)	55 (2)
C6	5781 (8)	2439 (6)	8850 (8)	68 (2)
C11	1302 (25)	1672 (10)	1149 (19)	294 (11)
C12	904 (22)	1146 (10)	2173 (23)	271 (12)
C11′	963 (14)	4053 (8)	698 (11)	124 (4)
C12′	−634 (14)	3978 (9)	1102 (14)	140 (5)
C21	6791 (13)	2300 (9)	3951 (13)	140 (5)
C22	6880 (24)	1681 (10)	3085 (16)	226 (9)
C31	456 (17)	3861 (10)	6398 (16)	181 (7)
C32	−65 (21)	4444 (11)	5441 (18)	240 (9)
C41	5303 (14)	4422 (6)	7970 (12)	91 (3)
C42	4834 (12)	4968 (6)	9112 (12)	106 (3)
C51	3859 (11)	1158 (5)	7122 (11)	70 (3)
C52	4098 (13)	513 (5)	6065 (12)	98 (4)
C61	8014 (11)	1414 (6)	8115 (11)	80 (3)
C62	−563 (11)	1159 (6)	7303 (14)	114 (4)

* Origin-defining coordinate.

atoms were placed in geometrically calculated positions and isotropically refined. Maximum and minimum heights in the final $\Delta\rho$ map were 0.85 and -0.51 e $Å^{-3}$, respectively; maximum shift/e.s.d. = 1.62 (z coordinate of C31 and U_{22} temperature factor of C32). Owing to high thermal motion two ethyl groups (at S1 and S3) were refined with constrained bond distances. No evidence was found for multiple sites for ethyl groups. The atomic coordinates and equivalent isotropic temperature factors are given in Table 1;* bond lengths and angles are listed in Table 2; Fig. 1 depicts the molecule.

Related literature. The process of ethanethiolysis of certain 1,2-*O*-isopropylidene-3,5,6-tri-*O*-substituted-D-glucose derivatives has been studied in detail by Bethel & Ferrier (1972, 1973). In these papers, based on a possible mechanism, it was assumed that the stereochemistry of the main reaction products corresponded to the D-*allo*-configuration.

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55057 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0300]

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

S1—C1	1.812 (8)	O53—C51	1.209 (12)
S1—C11	1.704 (20)	O63—C61	1.196 (12)
S'1—C1	1.792 (9)	C1—C2	1.576 (10)
S'1—C11′	1.785 (11)	C2—C3	1.557 (10)
S2—C2	1.825 (8)	C3—C4	1.524 (10)
S2—C21	1.799 (14)	C4—C5	1.545 (11)
S3—C3	1.844 (7)	C5—C6	1.500 (10)
S3—C31	1.748 (17)	C11—C12	1.375 (27)
S6—C6	1.825 (8)	C11′—C12′	1.474 (18)
S6—C61	1.736 (11)	C21—C22	1.336 (22)
O4—C4	1.444 (9)	C31—C32	1.379 (24)
O4—C41	1.365 (12)	C41—C42	1.496 (16)
O5—C5	1.440 (10)	C51—C52	1.503 (14)
O5—C51	1.351 (10)		

C1—S1—C11	106.3 (7)	O4—C4—C3	107.2 (6)
C1—S'1—C11′	103.2 (5)	O4—C4—C5	102.1 (6)
C2—S2—C21	103.8 (5)	C3—C4—C5	117.8 (6)
C3—S3—C31	106.0 (6)	O5—C5—C4	106.4 (6)
C6—S6—C61	101.0 (4)	O5—C5—C6	108.9 (6)
C4—O4—C41	118.6 (7)	C4—C5—C6	113.0 (7)
C5—O5—C51	117.8 (6)	S6—C6—C5	114.9 (6)
S1—C1—S'1	112.7 (4)	S1—C11—C12	129.8 (15)
S1—C1—C2	109.6 (5)	S'1—C11′—C12′	116.5 (9)
S'1—C1—C2	109.7 (5)	S2—C21—C22	124.8 (12)
S2—C2—C1	108.6 (5)	S3—C31—C32	124.0 (13)
S2—C2—C3	111.5 (5)	O4—C41—C42	109.9 (9)
C1—C2—C3	111.7 (6)	O5—C51—O53	121.0 (8)
S3—C3—C2	109.7 (5)	O5—C51—C52	111.5 (8)
S3—C3—C4	111.0 (5)	O53—C51—C52	127.4 (9)
C2—C3—C4	114.1 (6)	S6—C61—O63	124.8 (8)

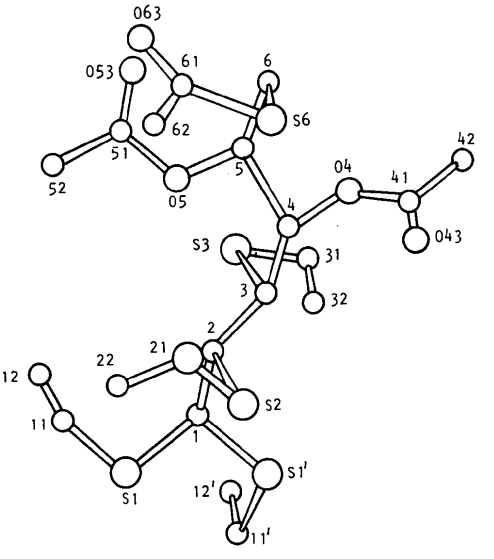


Fig. 1. The structure of the molecule with the atom-numbering scheme (C atoms are labeled with a number only).

References

BETHEL, G. S. & FERRIER, R. J. (1972). *J. Chem. Soc. Perkin Trans.* 1, pp. 1033–1037.

BETHEL, G. S. & FERRIER, R. J. (1973). *J. Chem. Soc. Perkin Trans.* 1, pp. 1400–1405.

SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS*86. *Crystallographic Computing* 3, edited by G. M. SHELDRICK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 158–166.