$S = 1.131$
4092 reflections
582 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1035P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Si	0.5975 (2)	0.23695 (6)	0.0623 (2)	0.082
O1	0.6155 (3)	0.1885 (1)	0.4087 (4)	0.071
O2	0.6256 (2)	0.2377 (1)	0.2126 (3)	0.063
O3	0.7329 (3)	0.3028 (1)	0.2451 (5)	0.085
O4	0.3709 (4)	0.3466 (2)	0.3817 (7)	0.153
O5	0.3778 (3)	0.2938 (2)	0.5976 (6)	0.113
O6	0.7477 (3)	0.3006 (2)	0.6419 (6)	0.097
C1	0.6077 (3)	0.2342 (2)	0.4397 (5)	0.051
C2	0.5809 (3)	0.2584 (2)	0.3141 (5)	0.054
C3	0.5906 (4)	0.3086 (2)	0.3092 (5)	0.059
C4	0.6639 (4)	0.3290 (2)	0.2688 (6)	0.070
C5	0.6680 (6)	0.3748 (2)	0.2516 (7)	0.101
C6	0.5968 (9)	0.3995 (3)	0.2791 (9)	0.128
C7	0.5289 (7)	0.3813 (2)	0.3236 (7)	0.107
C8	0.5187 (4)	0.3353 (2)	0.3411 (6)	0.074
C9	0.4395 (4)	0.3177 (2)	0.3991 (7)	0.084
C10	0.4511 (4)	0.3129 (2)	0.5441 (6)	0.072
C11	0.5310 (3)	0.2873 (2)	0.5708 (5)	0.052
C12	0.6009 (3)	0.3093 (2)	0.6040 (5)	0.049
C13	0.6843 (3)	0.2871 (2)	0.5885 (6)	0.057
C14	0.6910 (3)	0.2506 (2)	0.4936 (5)	0.056
C15	0.5376 (3)	0.2379 (2)	0.5413 (5)	0.057
C16	0.8085 (5)	0.3226 (3)	0.199 (1)	0.123
C17	0.3134 (4)	0.3361 (3)	0.2950 (9)	0.103
C18	0.3566 (6)	0.3081 (3)	0.7131 (9)	0.117
C19	0.6060 (4)	0.3558 (2)	0.6502 (6)	0.069
C20	0.5663 (5)	0.2139 (2)	0.6650 (6)	0.074
C21	0.4551 (4)	0.2144 (2)	0.5002 (7)	0.087
C22	0.6082 (9)	0.2898 (3)	−0.0184 (9)	0.156
C23	0.6668 (9)	0.1956 (4)	−0.0084 (9)	0.177
C24	0.4873 (9)	0.2200 (6)	0.052 (1)	0.245
Si′	0.5401 (2)	0.06524 (7)	0.2815 (2)	0.086
O1′	0.6288 (3)	0.1022 (1)	0.6106 (4)	0.066
O2′	0.5310 (3)	0.0735 (1)	0.4359 (3)	0.070
O3′	0.3724 (3)	0.0718 (2)	0.5052 (6)	0.110
O4′	0.5594 (3)	−0.0987 (1)	0.6087 (4)	0.088
O5′	0.6495 (3)	−0.0591 (1)	0.7978 (4)	0.077
O6′	0.3885 (4)	0.0787 (2)	0.8712 (6)	0.113
C1′	0.5702 (3)	0.0699 (2)	0.6536 (5)	0.051
C2′	0.5412 (4)	0.0425 (2)	0.5361 (5)	0.055
C3′	0.4642 (3)	0.0123 (2)	0.5475 (5)	0.053
C4′	0.3826 (4)	0.0270 (2)	0.5232 (7)	0.083
C5′	0.3146 (5)	−0.0016 (3)	0.5194 (9)	0.113
C6′	0.3263 (5)	−0.0452 (3)	0.5422 (9)	0.110
C7′	0.4036 (5)	−0.0610 (2)	0.5716 (7)	0.088
C8′	0.4736 (4)	−0.0329 (2)	0.5761 (5)	0.062
C9′	0.5572 (4)	−0.0518 (2)	0.6217 (5)	0.062
C10′	0.5701 (3)	−0.0429 (2)	0.7640 (5)	0.056
C11′	0.5537 (3)	0.0051 (2)	0.7918 (5)	0.047
C12′	0.4787 (3)	0.0184 (2)	0.8357 (5)	0.057
C13′	0.4518 (4)	0.0648 (2)	0.8168 (6)	0.070
C14′	0.4970 (4)	0.0924 (2)	0.7231 (6)	0.063
C15′	0.6178 (3)	0.0405 (2)	0.7497 (5)	0.053
C16′	0.2934 (5)	0.0884 (3)	0.467 (1)	0.132
C17′	0.5942 (8)	−0.1134 (2)	0.4964 (9)	0.130
C18′	0.6604 (5)	−0.0636 (3)	0.9305 (7)	0.101
C19′	0.4118 (4)	−0.0113 (2)	0.8916 (7)	0.085
C20′	0.6434 (4)	0.0681 (2)	0.8694 (6)	0.068
C21′	0.7023 (3)	0.0245 (2)	0.6948 (6)	0.068
C22′	0.6391 (6)	0.0907 (3)	0.2288 (8)	0.119
C23′	0.547 (1)	0.0066 (3)	0.241 (1)	0.196
C24′	0.4516 (9)	0.0944 (7)	0.208 (1)	0.321

Data collection and cell refinement: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994). Molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1985). Software used to prepare material for publication: *SHELXL*93.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1821–1823

anti,anti-N-(2,6-Dimethyl-1,3,5-trithian-1-ylidene)-4-methylbenzenesulfonamide

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(*Received* 24 *May* 1994; *accepted* 21 *June* 1994)

Abstract

The major product of the dimethylation of the tosylimide of 1,3,5-trithiane is shown to be the title compound, $C_{12}H_{17}NO_2S_4$, which has all the substituents in equatorial positions with respect to the 1,3,5-trithiane ring.

Comment

We are currently investigating the potential of the tosylimides of 1,3-dithiolane (I), 1,3-dithiane (II) and 1,3,5-trithiane (III) as chiral acyl-anion equivalents in

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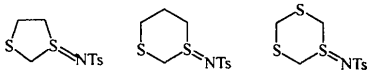
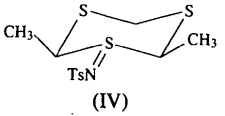
asymmetric synthesis. Alkylation of these compounds proceeds in a highly diastereoselective manner and our solution NMR studies indicate that the preferred product is *anti* with respect to the S═N bond and the alkyl substituent. It was considered desirable to obtain some support for the NMR structural assignments by investigating the crystal structure of at least one of these products.



(I) (II) (III)

The molecular structure of the title compound (IV) is shown in Fig. 1 and confirms that the major product of the dimethylation of the tosylimide of (III) has an all-*anti* arrangement. Furthermore, the conformational preference of the S═N bond is of interest. A recent study (Errington, Sparey & Taylor, 1994) of compounds (I), (II) and (III) showed that the 'normal' axial preference of the S═N bond of tosylsulfimides is reversed when repulsive *gauche* effects are important. The equatorial preference of the S═N bond in the title compound (IV) is in accord with these observations.



(IV)

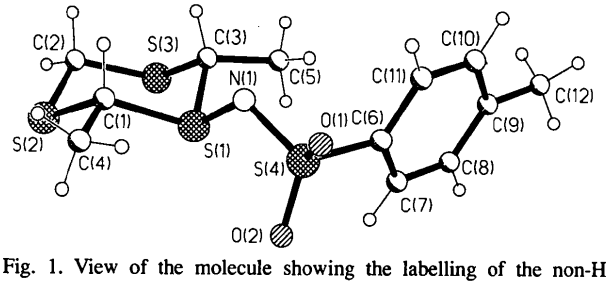


Fig. 1. View of the molecule showing the labelling of the non-H atoms.

Experimental

Reaction of the tosylimide (III) with 2 eq. of NaH in dmf followed by reaction with 2 eq. of iodomethane gave the title compound as the major product.

Crystal data

$C_{12}H_{17}NO_2S_4$
M_r = 335.51
Triclinic
$P\bar{1}$
a = 7.486 (6) Å
b = 8.965 (6) Å
c = 11.323 (8) Å
α = 98.91 (5)°
β = 92.71 (6)°
γ = 98.82 (6)°
V = 739.9 (9) Å^3
Z = 2
D_x = 1.506 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 24 reflections
θ = 10–11°
μ = 0.638 mm^{-1}
T = 220 (2) K
Block
0.27 × 0.18 × 0.16 mm
Colourless

Data collection

Siemens *R3m* diffractometer
ω–2θ scans
Absorption correction: none
2836 measured reflections
2614 independent reflections
2240 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0324
θ_{max} = 25.05°
$h = 0 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$
3 standard reflections monitored every 200 reflections
intensity variation: insignificant

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0382
$wR(F^2)$ = 0.1076
S = 1.066
2614 reflections
172 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0763P)^2 + 0.3206P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
S1	0.02914 (8)	0.23127 (7)	0.44695 (5)	0.0225 (2)
S2	0.09021 (9)	0.18226 (7)	0.69943 (6)	0.0295 (2)
S3	−0.28043 (9)	0.09508 (7)	0.56689 (6)	0.0294 (2)
S4	0.16447 (9)	0.33092 (7)	0.24818 (6)	0.0271 (2)
N1	0.0817 (3)	0.3743 (2)	0.3752 (2)	0.0263 (5)
O1	0.2771 (3)	0.4683 (2)	0.2293 (2)	0.0397 (5)
O2	0.2422 (3)	0.1927 (2)	0.2391 (2)	0.0364 (5)
C1	0.1361 (3)	0.3150 (3)	0.5965 (2)	0.0247 (5)
C2	−0.1496 (4)	0.1727 (3)	0.7060 (2)	0.0299 (6)
C3	−0.2067 (3)	0.2397 (3)	0.4783 (2)	0.0265 (6)
C4	0.3382 (4)	0.3524 (3)	0.5906 (3)	0.0366 (7)
C5	−0.3217 (4)	0.2075 (4)	0.3606 (3)	0.0389 (7)
C6	−0.0223 (3)	0.2971 (3)	0.1406 (2)	0.0262 (6)
C7	−0.0950 (4)	0.1494 (3)	0.0875 (2)	0.0319 (6)
C8	−0.2483 (4)	0.1257 (3)	0.0096 (2)	0.0355 (7)
C9	−0.3314 (4)	0.2458 (3)	−0.0157 (2)	0.0325 (6)
C10	−0.2544 (4)	0.3927 (3)	0.0380 (3)	0.0366 (7)
C11	−0.1009 (4)	0.4186 (3)	0.1149 (2)	0.0327 (6)
C12	−0.4990 (4)	0.2206 (4)	−0.0983 (3)	0.0492 (8)

Table 2. *Selected geometric parameters* (Å, °)

S1—N1	1.630 (2)	S3—C3	1.797 (3)
S1—C3	1.828 (3)	S3—C2	1.797 (3)
S1—C1	1.835 (3)	S4—O1	1.433 (2)
S2—C2	1.790 (3)	S4—N1	1.609 (2)
S2—C1	1.798 (3)		
N1—S1—C3	103.18 (13)	S2—C1—S1	110.41 (14)
N1—S1—C1	100.48 (13)	S2—C2—S3	114.9 (2)
C3—S1—C1	98.48 (14)	C5—C3—S3	109.3 (2)
C2—S2—C1	100.78 (14)	C5—C3—S1	108.8 (2)
C3—S3—C2	100.59 (14)	S3—C3—S1	108.46 (14)
C2—S2—C1—S1	−66.3 (2)	C2—S3—C3—S1	69.5 (2)
C1—S2—C2—S3	63.7 (2)	C1—S1—C3—S3	−73.3 (2)
C3—S3—C2—S2	−65.8 (2)	C3—S1—C1—S2	72.1 (2)

All H atoms were inserted using a riding model and given isotropic displacement parameters equal to $1.2U_{eq}$ (or $1.5U_{eq}$ for methyl groups) of the parent atom.

Data collection and cell refinement: Siemens *R3m* system. Data reduction: *SHELXTL-Plus* (Sheldrick, 1991). Program(s) used to solve structure: *SHELXTL-Plus*. Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1994).

We thank the SERC for financial support (to TJS).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: HR1035). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1823–1825

Dimethyl 1-(4-Acetoxy-3,5-dimethoxyphenyl)-4,7,9-trimethoxy-8-oxospiro[4.5]-deca-6,9-diene-*trans*-2,3-dicarboxylate Acetone Solvate

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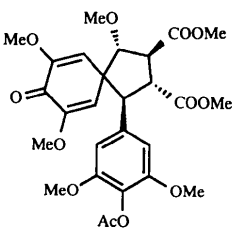
(*Received* 20 *April* 1994; *accepted* 17 *June* 1994)

Abstract

In this isomer of the title compound, $C_{27}H_{32}O_{12}.C_3H_6O$, the five-membered ring adopts an envelope conformation and the ester groups occupy *trans* positions with respect to the ring.

Comment

The structure of a product of peroxidatic oxidation of methyl sinapate was determined as a part of a study on the stereoselectivity of the oxidative coupling of phenols. The title compound (I) was the main product and the *trans* configuration of the ester groups demonstrates a degree of diastereoselectivity in the coupling reaction. The details of the synthetic work and the crystal structure of the *cis* diastereomer have been published elsewhere (Setälä, Pajunen, Kilpeläinen & Brunow, 1994). The five-membered ring is in an envelope conformation with C10 the out-of-plane atom. The angle between the least-squares plane through atoms C6, C7, C8 and C9 and the plane of C6, C9 and C10 is 39.5 (5)°. Disorder of one of the ester groups was resolved. The ester group O5, O6, C21 has two orientations with 0.43 (2) and 0.57 (2) occupancy. The structure contains a poorly defined acetone molecule.



(I)

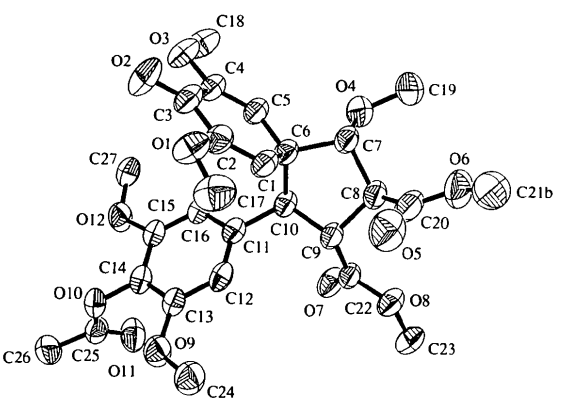


Fig. 1. A drawing of the molecule with the numbering of the non-H atoms. The displacement ellipsoids represent 30% probability levels. Only one orientation of the disordered ester group is shown.

Experimental

Crystal data

$C_{27}H_{32}O_{12}.C_3H_6O$
M_r = 606.60
Triclinic
$P\bar{1}$
a = 9.278 (4) Å
b = 11.635 (8) Å
c = 15.812 (4) Å
α = 76.46 (5)°
β = 78.46 (4)°
γ = 72.77 (5)°
V = 1569.2 (13) Å^3
Z = 2
D_x = 1.284 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 20 reflections
θ = 7–14°
μ = 0.101 mm^{-1}
T = 297 (2) K
Transparent block
0.40 × 0.30 × 0.30 mm
Colourless

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