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Structure of Hexa-2,4-diyne-1,6-diyl Bis(3,5-dimethylbenzenesulfonate)

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Abstract. $C_{22}H_{22}O_6S_2$, $M_r = 446{\cdot}5$, monoclinic, $P2_1/n$, $a = 16{\cdot}556\ (3)$, $b = 5{\cdot}535\ (5)$, $c = 12{\cdot}644\ (3)$ Å, $\beta = 105{\cdot}32\ (2)°$, $U = 1117{\cdot}5$ Å^3, $Z = 2$, $D_x = 1{\cdot}33$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}262$ mm^{-1}, $F(000) = 468$, room temperature, $R = 0{\cdot}035$, $wR = 0{\cdot}037$ for 1103 observed reflections. Molecules of this diacetylene pack along the y axis such that the distance between the potentially reactive C atoms (for 1,4-*trans* polymerization between adjacent molecules) is 5·62 Å. This distance is outside the normally accepted limit of about 4 Å required for the solid-state reaction, and is consistent with the experimentally found inactivity.

Experimental. Title compound prepared by reaction of 3,5-dimethylbenzenesulfonyl chloride with hexa-2,4-diyne-1,6-diol as previously reported for other sulfonate derivatives of diacetylenes (Ando, Bloor, Hubble & Williams, 1980). Amber needles were obtained by slow evaporation of acetone solutions. Crystals did not undergo any noticeable solid-state polymerization when subjected to heat, UV light, or X-radiation. Space group and approximate cell dimensions determined from oscillation and Weissenberg photographs using Cu $K\alpha$ radiation. Accurate cell dimensions and intensity data (h −18 → 18, k 0 → 5, l 0 → 14) using Stoe Stadi-2 diffractometer with graphite-monochromated Mo $K\alpha$ radiation and a crystal of size $1{\cdot}0 \times 0{\cdot}2 \times 0{\cdot}5$ mm mounted along **b** (needle axis). Cell parameters a, c and β refined from 2θ and ω values for 20 $h0l$ reflections, b calculated from $\pm\mu$ scans for two $0k0$ reflections. Data collected for $2 < 2\theta < 50°$, equi-inclination geometry and variable-width ω-step scans, 1597 unique reflections [1103 with $F_o > 6\sigma(F_o)$], two standard reflections for each layer, no significant intensity changes except zero layer which was corrected for a linear fall off in intensity (to a maximum of 5%), Lp corrections, but not absorption.

Table 1. *Atomic fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$U_{eq} = (U_1U_2U_3)^{1/3}$ where U_i are the diagonal elements of the diagonalized orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}(Å^2)
S1	0·36426 (5)	0·52468 (13)	0·12048 (6)	0·0582 (4)
C1	0·47361 (22)	0·0381 (7)	0·45098 (27)	0·0678 (19)
C2	0·42848 (22)	0·1068 (7)	0·36767 (28)	0·0695 (20)
C3	0·37064 (26)	0·1972 (9)	0·26829 (32)	0·0755 (23)
C4	0·32414 (17)	0·3132 (5)	0·01686 (23)	0·0528 (15)
C5	0·37636 (20)	0·2200 (6)	−0·04177 (25)	0·0578 (18)
C6	0·34824 (19)	0·0400 (6)	−0·11781 (24)	0·0635 (18)
C7	0·26630 (22)	−0·0402 (7)	−0·13273 (29)	0·0700 (20)
C8	0·21307 (19)	0·0501 (7)	−0·07499 (27)	0·0663 (19)
C9	0·24238 (19)	0·2304 (6)	0·00067 (28)	0·0618 (19)
C10	0·12474 (30)	−0·0449 (12)	−0·09312 (56)	0·0942 (33)
C11	0·40468 (31)	−0·0725 (11)	−0·17958 (41)	0·0893 (29)
O1	0·41682 (11)	0·3658 (4)	0·21803 (15)	0·0596 (11)
O2	0·29729 (14)	0·6348 (4)	0·15307 (20)	0·0765 (14)
O3	0·42567 (14)	0·6711 (4)	0·09295 (19)	0·0763 (14)

For a monomeric compound with $Z = 2$ the molecules must lie across centres of symmetry. S atoms located from Patterson synthesis and all C, O and H atoms located from subsequent F_o and difference Fourier syntheses. Least-squares refinement, using *SHELX*76 (Sheldrick, 1976), to minimize $\sum w(|F_o| - |F_c|)^2$ with isotropic thermal parameters for H atoms and anisotropic parameters for all others. Final weighting scheme was $w^{-1} = 1 + 0{\cdot}045(9 - F_o)^2$ for $F_o < 9$ and $w^{-1} = 1 + 0{\cdot}0075(F_o - 9)^2$ for $F_o > 9$; no extinction correction. Final $\Delta/\sigma(\max.) = 0{\cdot}2$ (H parameters) and 0·1 (other parameters). A final difference synthesis showed no residual electron density peaks outside the range $\pm 0{\cdot}2$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are listed in Table 1 and bond lengths and angles in Table 2.* Fig. 1 shows the labelling scheme and molecular packing in the y direction. Calculations were performed on a Prime 750 computer of the Napier Polytechnic Computer Unit.

Related literature. The previous paper in this series concerning structures of sulfonate derivatives of di-

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* Lists of structure factors, anisotropic thermal parameters and hydrogen parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52004 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

S1—C4	1·752 (3)	S1—O1	1·576 (2)
S1—O2	1·419 (3)	S1—O3	1·414 (3)
C1—C1^i	1·380 (5)	C1—C2	1·182 (5)
C2—C3	1·453 (5)	C3—O1	1·455 (5)
C3—H31	0·99 (6)	C3—H32	0·96 (4)
C4—C5	1·379 (4)	C4—C9	1·392 (4)
C5—C6	1·378 (5)	C5—H51	0·89 (3)
C6—C7	1·392 (5)	C6—C11	1·502 (6)
C7—C8	1·378 (5)	C7—H71	0·95 (3)
C8—C9	1·379 (5)	C8—C10	1·513 (7)
C9—H91	0·92 (3)	C10—H101	1·05 (6)
C10—H102	0·75 (6)	C10—H103	0·91 (6)
C11—H111	0·98 (6)	C11—H112	0·95 (5)
C11—H113	0·96 (6)		

O1—S1—C4	103·56 (12)	O2—S1—C4	109·48 (14)
O3—S1—C4	110·74 (14)	C5—C4—S1	118·97 (23)
C9—C4—S1	119·46 (23)	O2—S1—O1	108·95 (13)
O3—S1—O1	103·19 (13)	C3—O1—S1	117·03 (21)
O3—S1—O2	119·56 (14)	C2—C1—C1^i	178·99 (40)
C3—C2—C1	177·24 (40)	O1—C3—C2	107·25 (32)
H31—C3—C2	114·9 (30)	H32—C3—C2	112·3 (27)
H31—C3—O1	95·8 (33)	H32—C3—O1	109·2 (26)
H32—C3—H31	115·7 (42)	C9—C4—C5	121·41 (29)
C6—C5—C4	120·10 (30)	H51—C5—C4	117·2 (19)
C8—C9—C4	119·38 (30)	H91—C9—C4	115·5 (20)
H51—C5—C6	122·7 (19)	C7—C6—C5	117·57 (31)
C11—C6—C5	121·39 (34)	C11—C6—C7	121·01 (35)
C8—C7—C6	123·31 (33)	H71—C7—C6	116·5 (20)
H111—C11—C6	109·9 (33)	H112—C11—C6	112·0 (28)
H113—C11—C6	107·5 (32)	H71—C7—C8	120·1 (20)
C9—C8—C7	118·23 (32)	C10—C8—C7	121·42 (38)
C10—C8—C9	120·35 (37)	H91—C9—C8	124·8 (20)
H101—C10—C8	106·9 (35)	H102—C10—C8	118·3 (47)
H103—C10—C8	108·5 (32)	H102—C10—H101	108·9 (57)
H103—C10—H101	100·3 (48)	H103—C10—H102	112·4 (61)
H112—C11—H111	96·3 (40)	H113—C11—H111	126·5 (47)
H113—C11—H112	103·6 (42)		

Symmetry code: (i) 1·0 − *x*, −*y*, 1·0 − *z*.

acetylenes was by Bloor, Ando, Norman, Motevalli, Hursthouse, Milburn, Werninck & Blair (1986). In the present structure the molecules pack together along *y* with a 5·54 Å repeat distance. However, the crucial distance for 1,4-*trans* polymerization is between C2···C2 (at 1 − *x*, 1 − *y*, 1 − *z*), and this is 5·62 Å. The angle of inclination between the diacetylene axis and the stacking direction (the *b* axis) is 71·6°. These parameters are outside the normally accepted limits for facile solid-state polymerization according to the solid-state criterion (Schmidt, 1967) and the least-motion criterion (Baughman, 1974).

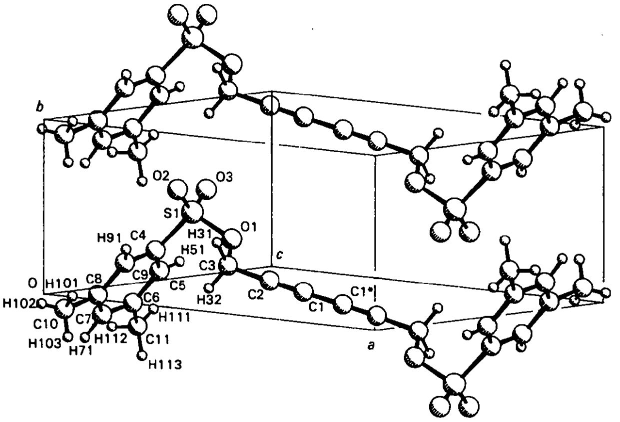

Fig. 1. View to show stacking of molecules in the *y* direction and to indicate the atomic labelling scheme.

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Structure of 7-Amino-2-chloro[1,3]thiazolo[4,5-*d*]pyrimidine, an Adenine Analog

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Abstract. $C_5H_3ClN_4S$, $M_r = 186{\cdot}62$, orthorhombic, *Pccn*, $a = 29{\cdot}874\,(6)$, $b = 7{\cdot}1932\,(9)$, $c = 12{\cdot}9913\,(14)$ Å, $V = 2791{\cdot}7\,(7)$ Å^3, $Z = 16$, $D_x = 1{\cdot}776$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu = 71{\cdot}31$ cm^{-1}, $F(000) = 1504$, $T = 295$ K, $R = 0{\cdot}0389$ for 2101 reflections ($F \geq 4\sigma_F$). The asymmetric unit is

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