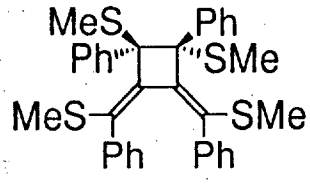
X-Ray Structure

of

1,2-Bis(methylthio)-3,4-bis(methylthiobenzylidene)-1,2-diphenylcyclobutane



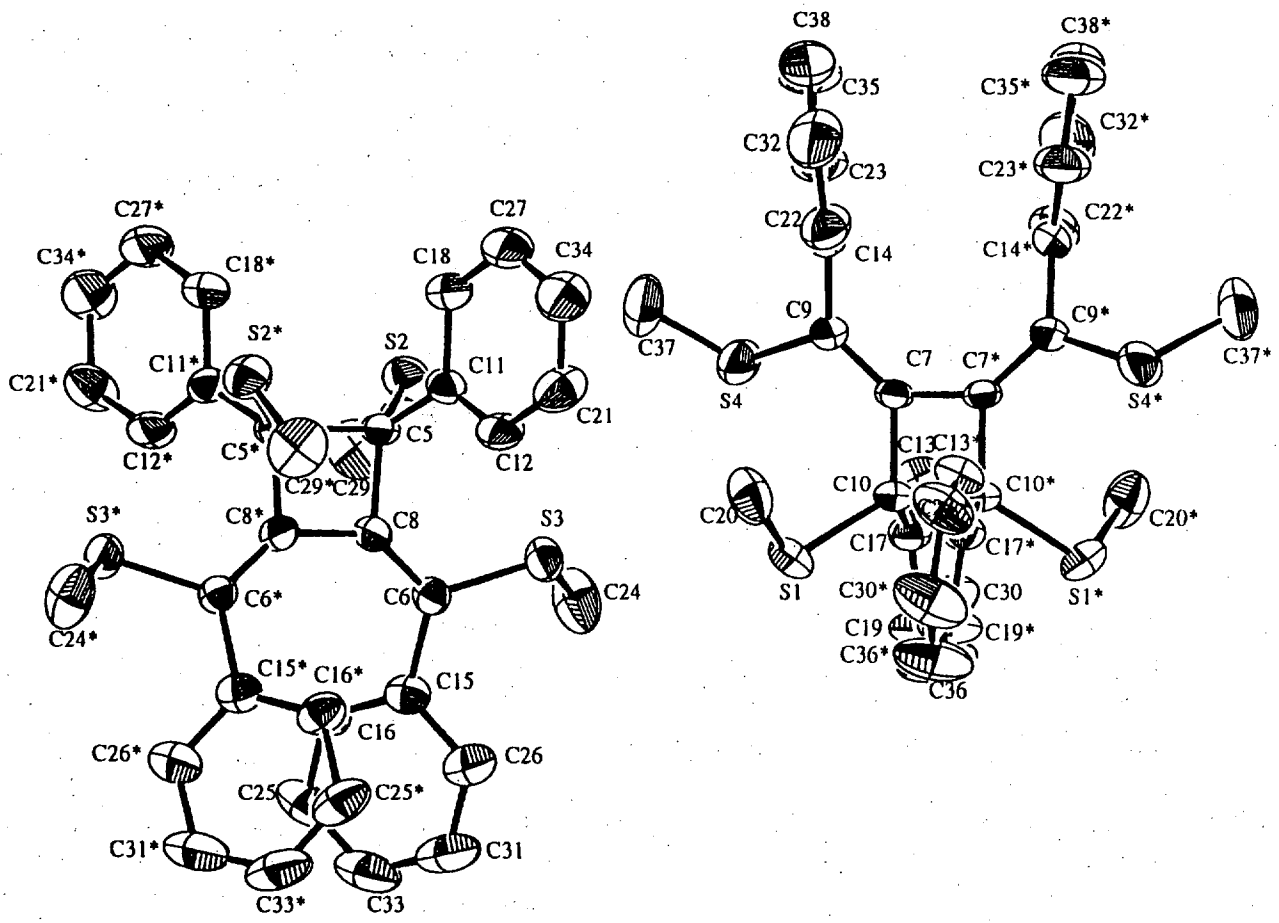


Table of Contents

I. Experimental Procedures

A. Data Collection

A yellow cube of $C_{34}H_{32}S_4$ having approximate dimension of 0.50 x 0.40 x 0.40 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with MoKα radiation (λ = 0.71073 Å) on a Mac Science MXC18 diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 22 reflection in measured by the computer controlled diagonal slit method of centering. The monoclinic cell parameters and calculated volume are: a = 18.064 (5), b = 11.274 (2), c = 16.359 (3) Å, β = 116.62 (2)°, V = 2978 (1) $Å^3$. For Z = 4, F.W. = 568.87, and the calculated density is 1.266 g/cm^3. From the systematic absences; the space group was determined to be P2/a (#13).

The data were collected at a temperature of 25 ± 1 °C using the ω–2θ scan technique. The scan rate varied from 0 to 5 °/min (in omega). Data were collected to a maximum 2θ of 26.47°. The scan range (in deg.) was determined as a function of θ to correct for the separation of the Kα doublet ; the scan width was calculated as follows:

$$\omega \text{ scan width} = 1.35 + 0.350 \tan\theta$$

B. Data Reduction

A total of 7683 reflections were collected. As a check on crystal and electronic stability 3 representative reflections were measured every 100 reflections. The slope of the least-squares line through a plot of intensity versus time was -9 ± 1 counts/hour which corresponds to a total loss in intensity of 1.0%.

Lorentz and polarization corrections were applied to the data. No absorption correction was made. An extinction correction was not necessary.

C. Structure Solution and Refinement

The structure was solved by direct methods. A total of 38 atoms were located from an E-map prepared from the phase set with probability statistics. The remaining atoms were located in succeeding difference Fourire syntheses and refined anisotropically. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|-|Fc|)^2$ and the weight w is defined as 1.0 for all observed reflections.

Scattering factors were taken from Cromer and Waber (ref 1). Anomalous dispersion effects were included in Fc (ref 2); the values for $\Delta f'$ and $\Delta f''$ were those of Cromer (ref 3).

$$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.052$$

$$Rw = SQRT(\Sigma w(Fo-Fc)^2/\Sigma wFo^2) = 0.053$$

The standard deviation of an observation of unit weight was 1.0. The highest peak in the final difference Fourier had a height of 0.49 e/Å^3 ; the minimum negative peak had a height of -0.55 e/Å^3 (ref 4). Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ, and various classes of indices showed no unusual trends. All calculations were performed on a SUN computer.

References

(1) D. T. Cromer and J. T. Waber, "International Tables for X-Ray Crystallography", vol. IV, The Kynoch Press, Birmingham, England, 1974, Table 2.2B.

(2) J. A. Ibers and W. C. Hamilton, Acta Crystallogr., 17, 781 (1964).

(3) D. T. Cromer, "International Tables for X-Ray Crystallography," Vol. IV, The Knoch Press, Birmingham, England, 1974, Table 2.3.1.

(4) D. W. J. Cruickshank, Acta Crystallogr., 2, 154 (1949).

II. Tables

A. Crystal Data

Empirical Formula	$C_{34}H_{32}S_4$
Formula Weight	568.87
Crystal Color, Habit	yellow, cube
Crystal Dimension	0.50 X 0.40 X 0.40 mm
Crystal System	monoclinic
Lattice Parameters	a = 18.064 (5) Å b = 11.274 (2) Å c = 16.359 (3) Å β = 116.62 (2)°
Volume of unit cell	V = 2978 (1) Å^3
Space Group	P2/a (#13)
Z value	4
D_{calc}	1.266 g/cm^3

B. Data Collection

Diffractometer	Mac Science MXC18
Radiation	MoKα (λ = 0.71073 Å)
Total Reflections Measured	7683
Reflection (hkl) limits	-23 < h < 20 -14 < k < 0 0 < l < 21
Unique Reflections	6278

C. Structure Solution and Refinement

Structure Solution	Direct methods
Least squares refinement method	Full matrix
Absorption correction	none
Function minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
F(000)	471
Linear absorption coefficient	3.267/cm
Reflections used in L.S.	4624
Data reduction cut-off	3.00
Maximum $\sin(\theta)/\lambda$	0.627
L.S. parameters	471
Eta coefficient	1.0000
Residuals, R	0.052
Residuals, Rw	0.053
Max shift/e.s.d.	1.6561

D.

FRACTIONAL ATOMIC COORDINATES & U(iso)

Atom	x/a	y/b	z/c	U(iso)
S(1)	0.63516(6)	0.11746(8)	0.37737(7)	0.0499(5)
S(2)	0.37132(6)	0.35004(9)	0.00005(7)	0.0524(5)
S(3)	0.45248(5)	0.11337(9)	0.15149(6)	0.0446(5)
S(4)	0.64754(7)	0.37457(9)	0.26996(7)	0.0566(5)
C(5)	0.29841(19)	0.28035(27)	0.03692(22)	0.035(2)
C(6)	0.35747(19)	0.06494(28)	0.06303(22)	0.035(2)
C(7)	0.72648(19)	0.33345(26)	0.45052(21)	0.033(1)
C(8)	0.29460(18)	0.14461(26)	0.03050(21)	0.033(1)
C(9)	0.6835(2)	0.4140(3)	0.3853(2)	0.037(2)
C(10)	0.7321(2)	0.1978(3)	0.4444(2)	0.035(2)
C(11)	0.3181(2)	0.3394(3)	0.1279(2)	0.040(2)
C(12)	0.3349(3)	0.2789(3)	0.2042(3)	0.050(2)
C(13)	0.8294(2)	0.2255(3)	0.3732(3)	0.048(2)
C(14)	0.6637(2)	0.5363(3)	0.4045(2)	0.042(2)
C(15)	0.3520(2)	-0.0597(3)	0.0305(3)	0.042(2)
C(16)	0.3085(2)	-0.0861(3)	-0.0613(3)	0.050(2)
C(17)	0.7901(2)	0.1512(3)	0.4089(2)	0.040(2)
C(18)	0.3176(3)	0.4641(3)	0.1323(3)	0.054(2)
C(19)	0.8071(3)	0.0302(3)	0.4113(3)	0.068(3)
C(20)	0.5563(3)	0.1964(6)	0.3934(4)	0.064(3)
C(21)	0.3538(3)	0.3357(4)	0.2871(3)	0.067(3)
C(22)	0.6180(3)	0.5521(4)	0.4516(3)	0.059(2)
C(23)	0.6835(3)	0.6350(3)	0.3661(4)	0.067(2)
C(24)	0.5306(3)	0.0610(7)	0.1214(4)	0.071(3)
C(25)	0.3064(3)	-0.2015(4)	-0.0940(4)	0.069(3)
C(26)	0.3943(3)	-0.1506(3)	0.0916(3)	0.058(2)
C(27)	0.3362(3)	0.5215(4)	0.2128(3)	0.069(3)
C(28)	0.8826(3)	0.1815(4)	0.3409(3)	0.071(3)
C(29)	0.3731(4)	0.2558(6)	-0.0875(4)	0.076(3)
C(30)	0.8983(4)	0.0624(5)	0.3435(4)	0.093(4)
C(31)	0.3913(3)	-0.2660(4)	0.0578(5)	0.080(3)
C(32)	0.5906(3)	0.6649(5)	0.4619(4)	0.081(3)
C(33)	0.3477(3)	-0.2901(4)	-0.0331(5)	0.082(3)
C(34)	0.3547(3)	0.4580(4)	0.2906(3)	0.076(3)
C(35)	0.6564(4)	0.7471(4)	0.3788(5)	0.092(3)
C(36)	0.8605(4)	-0.0120(4)	0.3794(5)	0.099(4)
C(37)	0.5501(4)	0.4475(8)	0.2118(5)	0.084(4)
C(38)	0.6109(4)	0.7612(5)	0.4242(4)	0.087(3)

Temperature factor of the form: exp[-2pi^2U], U=U(iso)
or 1/3 SUM(i)SUM(j){U(ij)*astar(i).astar(j).a(i).a(j).cos(ij)}

E. ANISOTROPIC THERMAL PARAMETERS

Atom	U11	U22	U33	U12	U13	U23
S(1)	0.0491(5)	0.0378(5)	0.0491(5)	-.0152(4)	0.0195(4)	-.0126(4)
S(2)	0.0437(5)	0.0444(5)	0.0579(6)	-.0099(4)	0.0253(5)	0.0006(5)
S(3)	0.0333(4)	0.0457(5)	0.0423(5)	0.0045(4)	0.0118(4)	-.0107(4)
S(4)	0.0688(7)	0.0465(6)	0.0351(5)	0.0127(5)	0.0121(5)	0.0014(4)
C(5)	0.032(2)	0.024(1)	0.040(2)	-0.001(1)	0.015(1)	-0.001(1)
C(6)	0.032(2)	0.031(2)	0.035(2)	-0.001(1)	0.015(1)	-0.003(1)
C(7)	0.030(2)	0.020(1)	0.040(2)	-0.001(1)	0.015(1)	-0.003(1)
C(8)	0.032(2)	0.026(2)	0.035(2)	-0.003(1)	0.015(1)	-0.003(1)
C(9)	0.034(2)	0.028(2)	0.039(2)	0.003(1)	0.013(1)	-0.001(1)
C(10)	0.036(2)	0.021(1)	0.037(2)	-0.001(1)	0.015(1)	0.000(1)
C(11)	0.032(2)	0.029(2)	0.046(2)	0.002(1)	0.014(1)	-0.008(1)
C(12)	0.055(2)	0.032(2)	0.051(2)	0.010(2)	0.026(2)	-0.007(2)
C(13)	0.051(2)	0.033(2)	0.052(2)	0.011(2)	0.028(2)	0.010(2)
C(14)	0.037(2)	0.029(2)	0.044(2)	0.007(1)	0.007(2)	0.001(1)
C(15)	0.036(2)	0.032(2)	0.050(2)	0.001(1)	0.026(2)	-0.003(2)
C(16)	0.041(2)	0.040(2)	0.058(2)	-0.002(2)	0.026(2)	-0.013(2)
C(17)	0.045(2)	0.025(2)	0.040(2)	0.003(1)	0.020(2)	-0.002(1)
C(18)	0.063(3)	0.029(2)	0.051(2)	0.003(2)	0.017(2)	0.001(2)
C(19)	0.087(3)	0.025(2)	0.080(3)	0.004(2)	0.054(3)	0.000(2)
C(20)	0.039(2)	0.076(3)	0.062(3)	-0.009(2)	0.017(2)	-0.017(3)
C(21)	0.087(3)	0.053(3)	0.046(2)	0.023(2)	0.032(2)	0.004(2)
C(22)	0.064(3)	0.046(2)	0.053(3)	0.015(2)	0.024(2)	0.000(2)
C(23)	0.060(3)	0.032(2)	0.089(3)	-0.001(2)	0.030(3)	0.007(2)
C(24)	0.039(2)	0.094(4)	0.069(3)	-0.004(3)	0.026(2)	-0.023(3)
C(25)	0.058(3)	0.053(3)	0.082(3)	-0.013(2)	0.039(3)	-0.031(2)
C(26)	0.057(2)	0.038(2)	0.067(3)	0.008(2)	0.032(2)	0.007(2)
C(27)	0.085(3)	0.039(2)	0.060(3)	0.012(2)	0.022(3)	-0.013(2)
C(28)	0.070(3)	0.060(3)	0.073(3)	0.019(2)	0.048(3)	0.024(2)
C(29)	0.067(3)	0.082(4)	0.071(4)	-0.003(3)	0.046(3)	-0.009(3)
C(30)	0.107(4)	0.066(3)	0.098(4)	0.040(3)	0.077(4)	0.018(3)
C(31)	0.074(3)	0.033(2)	0.115(5)	0.010(2)	0.051(4)	0.014(3)
C(32)	0.079(4)	0.072(4)	0.070(3)	0.031(3)	0.025(3)	-0.015(3)
C(33)	0.069(3)	0.038(2)	0.122(5)	-0.009(2)	0.053(3)	-0.026(3)
C(34)	0.095(4)	0.055(3)	0.053(3)	0.022(3)	0.026(3)	-0.017(2)
C(35)	0.082(4)	0.031(2)	0.127(5)	0.001(2)	0.028(4)	0.007(3)
C(36)	0.133(5)	0.038(3)	0.114(5)	0.030(3)	0.088(4)	0.005(3)
C(37)	0.070(4)	0.089(5)	0.056(4)	0.019(3)	-0.010(3)	-0.002(3)
C(38)	0.082(4)	0.043(3)	0.097(4)	0.019(3)	0.012(3)	-0.017(3)

T=exp[-2pi**2(U11. h**2. astar**2+U22. k**2. bstar**2+U33. l**2. cstar**2 +2U12. h. k. astar. bstar+2U13. h. l. astar. cstar+2U23. k. l. bstar. cstar)]

F. Table 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
S(1)	C(10)	1.835(3)	S(1)	C(20)	1.796(7)
S(2)	C(5)	1.851(4)	S(2)	C(29)	1.795(7)
S(3)	C(6)	1.764(3)	S(3)	C(24)	1.790(7)
S(4)	C(9)	1.755(3)	S(4)	C(37)	1.783(7)
C(5)	C(8)	1.533(4)	C(5)	C(11)	1.520(5)
C(5)	C(5*)	1.620(4)	C(6)	C(8)	1.356(4)
C(6)	C(15)	1.490(5)	C(7)	C(9)	1.350(4)
C(7)	C(10)	1.539(4)	C(7)	C(7*)	1.453(4)
C(8)	C(8*)	1.467(4)	C(9)	C(14)	1.493(5)
C(10)	C(17)	1.502(6)	C(10)	C(10*)	1.635(4)
C(11)	C(12)	1.333(6)	C(11)	C(18)	1.408(5)
C(12)	C(21)	1.397(7)	C(13)	C(17)	1.385(6)
C(13)	C(28)	1.380(8)	C(14)	C(22)	1.370(8)
C(14)	C(23)	1.401(6)	C(15)	C(16)	1.380(6)
C(15)	C(26)	1.396(5)	C(16)	C(25)	1.401(6)
C(17)	C(19)	1.395(5)	C(18)	C(27)	1.367(7)
C(19)	C(36)	1.37(1)	C(21)	C(34)	1.380(6)
C(22)	C(32)	1.402(8)	C(23)	C(35)	1.404(7)
C(25)	C(33)	1.371(7)	C(26)	C(31)	1.405(6)
C(27)	C(34)	1.365(7)	C(28)	C(30)	1.369(7)
C(30)	C(36)	1.37(1)	C(31)	C(33)	1.362(10)
C(32)	C(38)	1.377(9)	C(35)	C(38)	1.34(1)
C(5*)	C(8*)	1.533(4)	C(5*)	S(2*)	1.851(4)
C(5*)	C(11*)	1.520(5)	C(7*)	C(10*)	1.539(4)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(7*)	C(9*)	1.350(4)	C(8*)	C(6*)	1.356(4)
C(10*)	S(1*)	1.835(3)	C(10*)	C(17*)	1.502(6)
S(2*)	C(29*)	1.795(7)	C(11*)	C(12*)	1.333(6)
C(11*)	C(18*)	1.408(5)	C(9*)	S(4*)	1.755(3)
C(9*)	C(14*)	1.493(5)	C(6*)	S(3*)	1.764(3)
C(6*)	C(15*)	1.490(5)	S(1*)	C(20*)	1.796(7)
C(17*)	C(13*)	1.385(6)	C(17*)	C(19*)	1.395(5)
C(12*)	C(21*)	1.397(7)	C(18*)	C(27*)	1.367(7)
S(4*)	C(37*)	1.783(7)	C(14*)	C(22*)	1.370(8)
C(14*)	C(23*)	1.401(6)	S(3*)	C(24*)	1.790(7)
C(15*)	C(16*)	1.380(6)	C(15*)	C(26*)	1.396(5)
C(13*)	C(28*)	1.380(8)	C(19*)	C(36*)	1.37(1)
C(21*)	C(34*)	1.380(6)	C(27*)	C(34*)	1.365(7)
C(22*)	C(32*)	1.402(8)	C(23*)	C(35*)	1.404(7)
C(16*)	C(25*)	1.401(6)	C(26*)	C(31*)	1.405(6)
C(28*)	C(30*)	1.369(7)	C(36*)	C(30*)	1.37(1)
C(32*)	C(38*)	1.377(9)	C(35*)	C(38*)	1.34(1)
C(25*)	C(33*)	1.371(7)	C(31*)	C(33*)	1.362(10)

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(10)	S(1)	C(20)	106.0(2)	C(5)	S(2)	C(29)	106.2(3)
C(6)	S(3)	C(24)	105.6(2)	C(9)	S(4)	C(37)	105.0(3)
S(2)	C(5)	C(8)	114.7(3)	S(2)	C(5)	C(11)	104.2(2)
S(2)	C(5)	C(5*)	116.4(2)	C(8)	C(5)	C(11)	119.2(3)
C(8)	C(5)	C(5*)	87.1(2)	C(11)	C(5)	C(5*)	115.5(3)
S(3)	C(6)	C(8)	116.9(2)	S(3)	C(6)	C(15)	118.0(2)
C(8)	C(6)	C(15)	125.1(3)	C(9)	C(7)	C(10)	130.4(3)
C(9)	C(7)	C(7*)	136.3(3)	C(10)	C(7)	C(7*)	92.9(2)
C(5)	C(8)	C(6)	129.1(3)	C(5)	C(8)	C(8*)	92.8(2)
C(6)	C(8)	C(8*)	137.6(3)	S(4)	C(9)	C(7)	118.9(3)
S(4)	C(9)	C(14)	116.9(2)	C(7)	C(9)	C(14)	124.2(3)
S(1)	C(10)	C(7)	117.1(2)	S(1)	C(10)	C(17)	103.9(2)
S(1)	C(10)	C(10*)	117.4(3)	C(7)	C(10)	C(17)	116.8(3)
C(7)	C(10)	C(10*)	86.1(2)	C(17)	C(10)	C(10*)	115.7(2)
C(5)	C(11)	C(12)	123.2(3)	C(5)	C(11)	C(18)	118.9(3)
C(12)	C(11)	C(18)	117.9(4)	C(11)	C(12)	C(21)	121.9(3)
C(17)	C(13)	C(28)	121.4(4)	C(9)	C(14)	C(22)	120.0(4)
C(9)	C(14)	C(23)	120.4(4)	C(22)	C(14)	C(23)	119.2(4)
C(6)	C(15)	C(16)	120.4(3)	C(6)	C(15)	C(26)	120.6(3)
C(16)	C(15)	C(26)	118.9(3)	C(15)	C(16)	C(25)	121.4(4)
C(10)	C(17)	C(13)	122.0(3)	C(10)	C(17)	C(19)	120.9(4)
C(13)	C(17)	C(19)	117.1(4)	C(11)	C(18)	C(27)	121.2(4)
C(17)	C(19)	C(36)	120.8(5)	C(12)	C(21)	C(34)	119.4(4)
C(14)	C(22)	C(32)	121.3(5)	C(14)	C(23)	C(35)	118.2(6)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(16)	C(25)	C(33)	119.1(5)	C(15)	C(26)	C(31)	119.1(4)
C(18)	C(27)	C(34)	120.1(4)	C(13)	C(28)	C(30)	120.7(6)
C(28)	C(30)	C(36)	118.6(7)	C(26)	C(31)	C(33)	121.0(4)
C(22)	C(32)	C(38)	118.8(6)	C(25)	C(33)	C(31)	120.5(5)
C(21)	C(34)	C(27)	119.5(4)	C(23)	C(35)	C(38)	122.0(6)
C(19)	C(36)	C(30)	121.5(5)	C(32)	C(38)	C(35)	120.4(6)
C(5)	C(5*)	C(8*)	87.1(2)	C(5)	C(5*)	S(2*)	116.4(2)
C(5)	C(5*)	C(11*)	115.5(3)	C(8*)	C(5*)	S(2*)	114.7(3)
C(8*)	C(5*)	C(11*)	119.2(3)	S(2*)	C(5*)	C(11*)	104.2(2)
C(7)	C(7*)	C(10*)	92.9(2)	C(7)	C(7*)	C(9*)	136.3(3)
C(10*)	C(7*)	C(9*)	130.4(3)	C(8)	C(8*)	C(5*)	92.8(2)
C(8)	C(8*)	C(6*)	137.6(3)	C(5*)	C(8*)	C(6*)	129.1(3)
C(10)	C(10*)	C(7*)	86.1(2)	C(10)	C(10*)	S(1*)	117.4(3)
C(10)	C(10*)	C(17*)	115.7(2)	C(7*)	C(10*)	S(1*)	117.1(2)
C(7*)	C(10*)	C(17*)	116.8(3)	S(1*)	C(10*)	C(17*)	103.9(2)
C(5*)	S(2*)	C(29*)	106.2(3)	C(5*)	C(11*)	C(12*)	123.2(3)
C(5*)	C(11*)	C(18*)	118.9(3)	C(12*)	C(11*)	C(18*)	117.9(4)
C(7*)	C(9*)	S(4*)	118.9(3)	C(7*)	C(9*)	C(14*)	124.2(3)
S(4*)	C(9*)	C(14*)	116.9(2)	C(8*)	C(6*)	S(3*)	116.9(2)
C(8*)	C(6*)	C(15*)	125.1(3)	S(3*)	C(6*)	C(15*)	118.0(2)
C(10*)	S(1*)	C(20*)	106.0(2)	C(10*)	C(17*)	C(13*)	122.0(3)
C(10*)	C(17*)	C(19*)	120.9(4)	C(13*)	C(17*)	C(19*)	117.1(4)
C(11*)	C(12*)	C(21*)	121.9(3)	C(11*)	C(18*)	C(27*)	121.2(4)
C(9*)	S(4*)	C(37*)	105.0(3)	C(9*)	C(14*)	C(22*)	120.0(4)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(9*)	C(14*)	C(23*)	120.4(4)	C(22*)	C(14*)	C(23*)	119.2(4)
C(6*)	S(3*)	C(24*)	105.6(2)	C(6*)	C(15*)	C(16*)	120.4(3)
C(6*)	C(15*)	C(26*)	120.6(3)	C(16*)	C(15*)	C(26*)	118.9(3)
C(17*)	C(13*)	C(28*)	121.4(4)	C(17*)	C(19*)	C(36*)	120.8(5)
C(12*)	C(21*)	C(34*)	119.4(4)	C(18*)	C(27*)	C(34*)	120.1(4)
C(14*)	C(22*)	C(32*)	121.3(5)	C(14*)	C(23*)	C(35*)	118.2(6)
C(15*)	C(16*)	C(25*)	121.4(4)	C(15*)	C(26*)	C(31*)	119.1(4)
C(13*)	C(28*)	C(30*)	120.7(6)	C(19*)	C(36*)	C(30*)	121.5(5)
C(21*)	C(34*)	C(27*)	119.5(4)	C(22*)	C(32*)	C(38*)	118.8(6)
C(23*)	C(35*)	C(38*)	122.0(6)	C(16*)	C(25*)	C(33*)	119.1(5)
C(26*)	C(31*)	C(33*)	121.0(4)	C(28*)	C(30*)	C(36*)	118.6(7)
C(32*)	C(38*)	C(35*)	120.4(6)	C(25*)	C(33*)	C(31*)	120.5(5)

H. Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
S(1)	C(10)	C(7)	C(9)	-43.3(6)	S(1)	C(10)	C(7)	C(7*)	129.7(3)
S(1)	C(10)	C(17)	C(13)	123.2(3)	S(1)	C(10)	C(17)	C(19)	-57.3(3)
S(1)	C(10)	C(10*)	C(7*)	-128.9(3)	S(1)	C(10)	C(10*)	S(1*)	112.4(3)
S(1)	C(10)	C(10*)	C(17*)	-10.9(4)	S(2)	C(5)	C(8)	C(6)	50.6(5)
S(2)	C(5)	C(8)	C(8*)	-122.2(3)	S(2)	C(5)	C(11)	C(12)	-127.3(4)
S(2)	C(5)	C(11)	C(18)	53.6(4)	S(2)	C(5)	C(5*)	C(8*)	120.4(3)
S(2)	C(5)	C(5*)	S(2*)	-123.4(2)	S(2)	C(5)	C(5*)	C(11*)	-0.7(4)
S(3)	C(6)	C(8)	C(5)	20.6(6)	S(3)	C(6)	C(8)	C(8*)	-170.1(4)
S(3)	C(6)	C(15)	C(16)	-141.1(4)	S(3)	C(6)	C(15)	C(26)	35.6(6)
S(4)	C(9)	C(7)	C(10)	-18.5(6)	S(4)	C(9)	C(7)	C(7*)	171.5(4)
S(4)	C(9)	C(14)	C(22)	119.9(3)	S(4)	C(9)	C(14)	C(23)	-52.9(4)
C(5)	C(8)	C(6)	C(15)	-160.0(4)	C(5)	C(8)	C(8*)	C(5*)	4.6(3)
C(5)	C(8)	C(8*)	C(6*)	-167.1(5)	C(5)	C(11)	C(12)	C(21)	179.2(4)
C(5)	C(11)	C(18)	C(27)	-178.9(4)	C(5)	C(5*)	C(8*)	C(8)	-4.3(3)
C(5)	C(5*)	C(8*)	C(6*)	168.5(4)	C(5)	C(5*)	S(2*)	C(29*)	-68.2(3)
C(5)	C(5*)	C(11*)	C(12*)	103.7(4)	C(5)	C(5*)	C(11*)	C(18*)	-75.4(5)
C(6)	C(8)	C(5)	C(11)	-73.8(6)	C(6)	C(8)	C(5)	C(5*)	168.5(4)
C(6)	C(8)	C(8*)	C(5*)	-167.1(5)	C(6)	C(8)	C(8*)	C(6*)	21.2(9)
C(6)	C(15)	C(16)	C(25)	176.4(5)	C(6)	C(15)	C(26)	C(31)	-176.7(5)
C(7)	C(9)	S(4)	C(37)	144.4(4)	C(7)	C(9)	C(14)	C(22)	-59.8(5)
C(7)	C(9)	C(14)	C(23)	127.3(4)	C(7)	C(10)	S(1)	C(20)	-34.2(4)
C(7)	C(10)	C(17)	C(13)	-7.4(4)	C(7)	C(10)	C(17)	C(19)	172.1(3)
C(7)	C(10)	C(10*)	C(7*)	-10.2(3)	C(7)	C(10)	C(10*)	S(1*)	-128.9(3)
C(7)	C(10)	C(10*)	C(17*)	107.7(3)	C(7)	C(7*)	C(10*)	C(10)	10.8(3)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle
C(7)	C(7*)	C(10*)	S(1*)	129.7(3)
C(7)	C(7*)	C(9*)	S(4*)	171.5(4)
C(8)	C(5)	S(2)	C(29)	31.3(3)
C(8)	C(5)	C(11)	C(18)	-177.0(4)
C(8)	C(5)	C(5*)	S(2*)	120.4(3)
C(8)	C(6)	S(3)	C(24)	-135.6(4)
C(8)	C(6)	C(15)	C(26)	-143.8(5)
C(8)	C(8*)	C(5*)	C(11*)	113.4(3)
C(8)	C(8*)	C(6*)	C(15*)	9.3(8)
C(9)	C(7)	C(10)	C(10*)	-162.3(4)
C(9)	C(7)	C(7*)	C(9*)	-26.7(8)
C(9)	C(14)	C(23)	C(35)	173.8(4)
C(10)	C(7)	C(7*)	C(10*)	-11.5(3)
C(10)	C(17)	C(13)	C(28)	179.9(3)
C(10)	C(10*)	C(7*)	C(9*)	-162.3(4)
C(10)	C(10*)	C(17*)	C(13*)	-106.5(3)
C(11)	C(5)	S(2)	C(29)	163.4(3)
C(11)	C(5)	C(5*)	C(8*)	-117.0(3)
C(11)	C(5)	C(5*)	C(11*)	121.9(3)
C(11)	C(18)	C(27)	C(34)	-0.8(8)
C(12)	C(11)	C(18)	C(27)	1.9(7)
C(13)	C(17)	C(10)	C(10*)	-106.5(3)
C(13)	C(28)	C(30)	C(36)	-0.4(7)
C(14)	C(9)	C(7)	C(7*)	-8.8(7)

atom	atom	atom	atom	angle
C(7)	C(7*)	C(10*)	C(17*)	-106.1(3)
C(7)	C(7*)	C(9*)	C(14*)	-8.8(7)
C(8)	C(5)	C(11)	C(12)	2.1(6)
C(8)	C(5)	C(5*)	C(8*)	4.2(3)
C(8)	C(5)	C(5*)	C(11*)	-117.0(3)
C(8)	C(6)	C(15)	C(16)	39.6(7)
C(8)	C(8*)	C(5*)	S(2*)	-122.2(3)
C(8)	C(8*)	C(6*)	S(3*)	-170.1(4)
C(9)	C(7)	C(10)	C(17)	80.8(5)
C(9)	C(7)	C(7*)	C(10*)	160.9(5)
C(9)	C(14)	C(22)	C(32)	-172.8(3)
C(10)	C(7)	C(9)	C(14)	161.2(4)
C(10)	C(7)	C(7*)	C(9*)	160.9(5)
C(10)	C(17)	C(19)	C(36)	-179.6(4)
C(10)	C(10*)	S(1*)	C(20*)	66.2(3)
C(10)	C(10*)	C(17*)	C(19*)	73.0(4)
C(11)	C(5)	C(8)	C(8*)	113.4(3)
C(11)	C(5)	C(5*)	S(2*)	-0.7(4)
C(11)	C(12)	C(21)	C(34)	0.4(8)
C(12)	C(11)	C(5)	C(5*)	103.7(4)
C(12)	C(21)	C(34)	C(27)	0.7(8)
C(13)	C(17)	C(19)	C(36)	0.0(5)
C(14)	C(9)	S(4)	C(37)	-35.4(4)
C(14)	C(22)	C(32)	C(38)	-0.5(6)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(14)	C(23)	C(35)	C(38)	-1.7(8)	C(15)	C(6)	S(3)	C(24)	45.0(4)
C(15)	C(6)	C(8)	C(8*)	9.3(8)	C(15)	C(16)	C(25)	C(33)	0.8(8)
C(15)	C(26)	C(31)	C(33)	-0.2(10)	C(16)	C(15)	C(26)	C(31)	0.0(8)
C(16)	C(25)	C(33)	C(31)	-1.0(10)	C(17)	C(10)	S(1)	C(20)	-164.7(3)
C(17)	C(10)	C(7)	C(7*)	-106.1(3)	C(17)	C(10)	C(10*)	C(7*)	107.7(3)
C(17)	C(10)	C(10*)	S(1*)	-10.9(4)	C(17)	C(10)	C(10*)	C(17*)	-134.3(3)
C(17)	C(13)	C(28)	C(30)	-0.2(6)	C(17)	C(19)	C(36)	C(30)	-0.6(8)
C(18)	C(11)	C(5)	C(5*)	-75.4(5)	C(18)	C(11)	C(12)	C(21)	-1.7(7)
C(18)	C(27)	C(34)	C(21)	-0.5(8)	C(19)	C(17)	C(10)	C(10*)	73.0(4)
C(19)	C(17)	C(13)	C(28)	0.4(5)	C(19)	C(36)	C(30)	C(28)	0.8(8)
C(20)	S(1)	C(10)	C(10*)	66.2(3)	C(22)	C(14)	C(23)	C(35)	0.9(6)
C(22)	C(32)	C(38)	C(35)	-0.3(7)	C(23)	C(14)	C(22)	C(32)	0.1(5)
C(23)	C(35)	C(38)	C(32)	1.4(8)	C(25)	C(16)	C(15)	C(26)	-0.3(7)
C(25)	C(33)	C(31)	C(26)	0(1)	C(29)	S(2)	C(5)	C(5*)	-68.2(3)
C(5*)	C(5)	C(8)	C(8*)	-4.3(3)	C(5*)	C(8*)	C(6*)	S(3*)	20.6(6)
C(5*)	C(8*)	C(6*)	C(15*)	-160.0(4)	C(5*)	C(11*)	C(12*)	C(21*)	179.2(4)
C(5*)	C(11*)	C(18*)	C(27*)	-178.9(4)	C(7*)	C(7)	C(10)	C(10*)	10.8(3)
C(7*)	C(10*)	S(1*)	C(20*)	-34.2(4)	C(7*)	C(10*)	C(17*)	C(13*)	-7.4(4)
C(7*)	C(10*)	C(17*)	C(19*)	172.1(3)	C(7*)	C(9*)	S(4*)	C(37*)	144.4(4)
C(7*)	C(9*)	C(14*)	C(22*)	-59.8(5)	C(7*)	C(9*)	C(14*)	C(23*)	127.3(4)
C(8*)	C(5*)	S(2*)	C(29*)	31.3(3)	C(8*)	C(5*)	C(11*)	C(12*)	2.1(6)
C(8*)	C(5*)	C(11*)	C(18*)	-177.0(4)	C(8*)	C(6*)	S(3*)	C(24*)	-135.6(4)
C(8*)	C(6*)	C(15*)	C(16*)	39.6(7)	C(8*)	C(6*)	C(15*)	C(26*)	-143.8(5)
C(10*)	C(7*)	C(9*)	S(4*)	-18.5(6)	C(10*)	C(7*)	C(9*)	C(14*)	161.2(4)

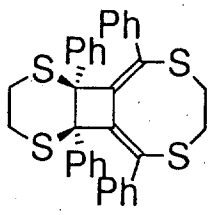
Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(10*)	C(17*)	C(13*)	C(28*)	179.9(3)	C(10*)	C(17*)	C(19*)	C(36*)	-179.6(4)
S(2*)	C(5*)	C(8*)	C(6*)	50.6(5)	S(2*)	C(5*)	C(11*)	C(12*)	-127.3(4)
S(2*)	C(5*)	C(11*)	C(18*)	53.6(4)	C(11*)	C(5*)	C(8*)	C(6*)	-73.8(6)
C(11*)	C(5*)	S(2*)	C(29*)	163.4(3)	C(11*)	C(12*)	C(21*)	C(34*)	0.4(8)
C(11*)	C(18*)	C(27*)	C(34*)	-0.8(8)	C(9*)	C(7*)	C(10*)	S(1*)	-43.3(6)
C(9*)	C(7*)	C(10*)	C(17*)	80.8(5)	C(9*)	C(14*)	C(22*)	C(32*)	-172.8(3)
C(9*)	C(14*)	C(23*)	C(35*)	173.8(4)	C(6*)	C(15*)	C(16*)	C(25*)	176.4(5)
C(6*)	C(15*)	C(26*)	C(31*)	-176.7(5)	S(1*)	C(10*)	C(17*)	C(13*)	123.2(3)
S(1*)	C(10*)	C(17*)	C(19*)	-57.3(3)	C(17*)	C(10*)	S(1*)	C(20*)	-164.7(3)
C(17*)	C(13*)	C(28*)	C(30*)	-0.2(6)	C(17*)	C(19*)	C(36*)	C(30*)	-0.6(8)
C(12*)	C(11*)	C(18*)	C(27*)	1.9(7)	C(12*)	C(21*)	C(34*)	C(27*)	0.7(8)
C(18*)	C(11*)	C(12*)	C(21*)	-1.7(7)	C(18*)	C(27*)	C(34*)	C(21*)	-0.5(8)
S(4*)	C(9*)	C(14*)	C(22*)	119.9(3)	S(4*)	C(9*)	C(14*)	C(23*)	-52.9(4)
C(14*)	C(9*)	S(4*)	C(37*)	-35.4(4)	C(14*)	C(22*)	C(32*)	C(38*)	-0.5(6)
C(14*)	C(23*)	C(35*)	C(38*)	-1.7(8)	S(3*)	C(6*)	C(15*)	C(16*)	-141.1(4)
S(3*)	C(6*)	C(15*)	C(26*)	35.6(6)	C(15*)	C(6*)	S(3*)	C(24*)	45.0(4)
C(15*)	C(16*)	C(25*)	C(33*)	0.8(8)	C(15*)	C(26*)	C(31*)	C(33*)	-0.2(10)
C(13*)	C(17*)	C(19*)	C(36*)	0.0(5)	C(13*)	C(28*)	C(30*)	C(36*)	-0.4(7)
C(19*)	C(17*)	C(13*)	C(28*)	0.4(5)	C(19*)	C(36*)	C(30*)	C(28*)	0.8(8)
C(22*)	C(14*)	C(23*)	C(35*)	0.9(6)	C(22*)	C(32*)	C(38*)	C(35*)	-0.3(7)
C(23*)	C(14*)	C(22*)	C(32*)	0.1(5)	C(23*)	C(35*)	C(38*)	C(32*)	1.4(8)
C(16*)	C(15*)	C(26*)	C(31*)	0.0(8)	C(16*)	C(25*)	C(33*)	C(31*)	-1.0(10)
C(26*)	C(15*)	C(16*)	C(25*)	-0.3(7)	C(26*)	C(31*)	C(33*)	C(25*)	0(1)

X-Ray Structure

of

2,7,9,14-Tetraphenyl-3,6,10,13-tetrathiatricyclo-[6,6,0,$0^{9,14}$]tetradeca-1,7-diene



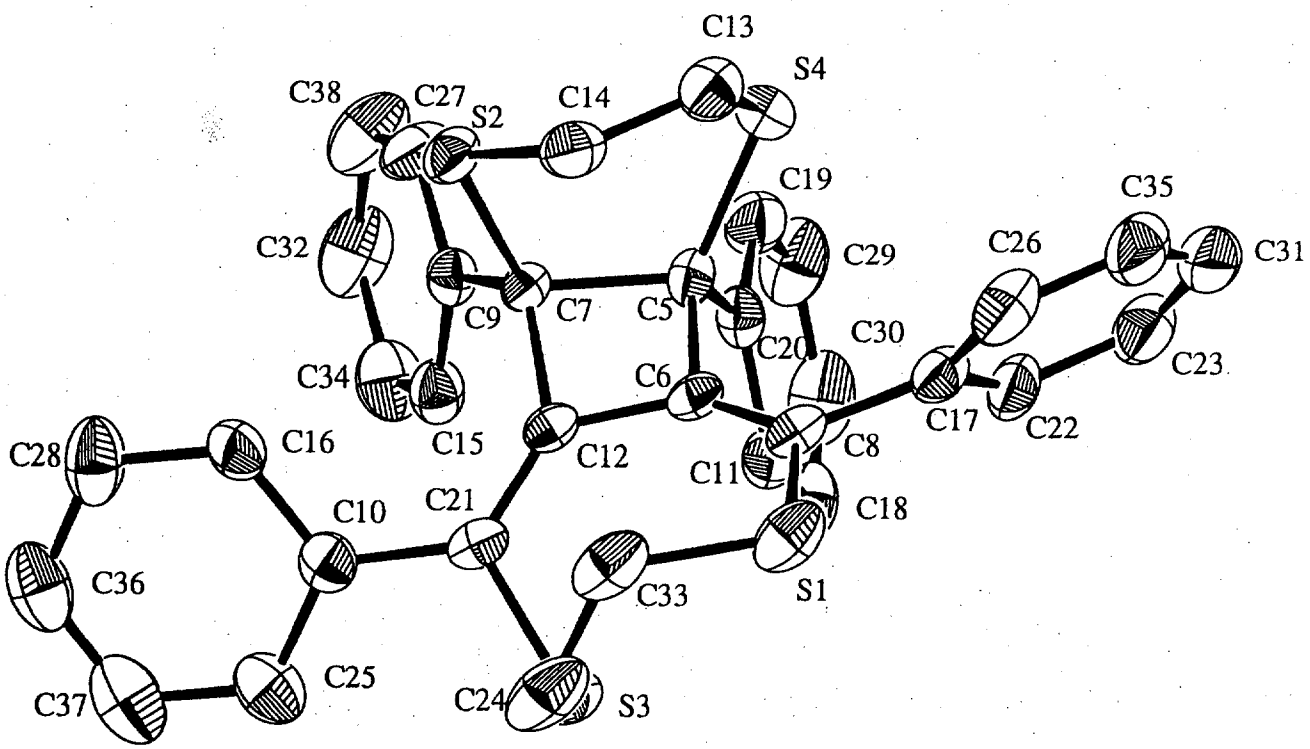


Table of Contents

I. Experimental Procedures

A. Data Collection

A colorless prism of $C_{34}H_{28}S_4$ having approximate dimension of 0.40 x 0.30 x 0.15 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with MoKα radiation (λ = 0.71069 Å) on a Mac Science MXC18 diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 22 reflection in measured by the computer controlled diagonal slit method of centering. The orthorhombic cell parameters and calculated volume are: a = 9.803 (2), b = 32.311 (8), c = 8.859 (2) Å, V = 2806.0 (10) $Å^3$. For Z = 4, F.W. = 564.84, and the calculated density is 1.336 g/cm^3. From the systematic absences; the space group was determined to be $P2_12_12_1$ (#19).

The data were collected at a temperature of 25 ± 1 °C using the ω–2θ scan technique. The scan rate varied from 0 to 5 °/min (in omega). Data were collected to a maximum 2θ of 26.43°. The scan range (in deg.) was determined as a function of θ to correct for the separation of the Kα doublet ; the scan width was calculated as follows:

$$\omega \text{ scan width} = 1.08 + 0.350 \tan\theta$$

B. Data Reduction

A total of 3512 reflections were collected. As a check on crystal and electronic stability 3 representative reflections were measured every 100 reflections. The slope of the least-squares line through a plot of intensity versus time was -9 ± 1 counts/hour which corresponds to a total loss in intensity of 7.0%.

Lorentz and polarization corrections were applied to the data. No absorption correction was made. An extinction correction was not necessary.

C. Structure Solution and Refinement

The structure was solved by direct methods. A total of 38 atoms were located from an E-map prepared from the phase set with probability statistics. The remaining atoms were located in succeeding difference Fourire syntheses and refined anisotropically. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|-|Fc|)^2$ and the weight w is defined as 1.0 for all observed reflections.

Scattering factors were taken from Cromer and Waber (ref 1). Anomalous dispersion effects were included in Fc (ref 2); the values for $\Delta f'$ and $\Delta f''$ were those of Cromer (ref 3).

$$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.057$$
$$Rw = SQRT(\Sigma w(Fo-Fc)^2/\Sigma wFo^2) = 0.063$$

The standard deviation of an observation of unit weight was 1.0. The highest peak in the final difference Fourier had a height of 1.17 e/Å^3 ; the minimum negative peak had a height of -1.40 e/Å^3 (ref 4). Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ, and various classes of indices showed no unusual trends. All calculations were performed on a SUN computer.

References

(1) D. T. Cromer and J. T. Waber, "International Tables for X-Ray Crystallography", vol. IV, The Kynoch Press, Birmingham, England, 1974, Table 2.2B.

(2) J. A. Ibers and W. C. Hamilton, Acta Crystallogr., 17, 781 (1964).

(3) D. T. Cromer, "International Tables for X-Ray Crystallography," Vol. IV, The Knoch Press, Birmingham, England, 1974, Table 2.3.1.

(4) D. W. J. Cruickshank, Acta Crystallogr., 2, 154 (1949).

II. Tables

A. Crystal Data

Empirical Formula	$C_{34}H_{28}S_4$
Formula Weight	564.84
Crystal Color, Habit	pale yellow, plate
Crystal Dimension	0.40 X 0.30 X 0.15 mm
Crystal System	orthorhombic
Lattice Parameters	a = 9.803 (2) Å b = 32.311 (8) Å c = 8.859 (2) Å
Volume of unit cell	V = 2806.0 (10) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.336 g/cm^3

B. Data Collection

Diffractometer	Mac Science MXC18
Radiation	MoKα (λ = 0.71069 Å)
Total Reflections Measured	3805

Reflection (hkl) limits	$0 < h < 12$ $-41 < k < 0$ $0 < l < 11$
Unique Reflections	3351

C. Structure Solution and Refinement

Structure Solution	Direct methods
Least squares refinement method	Full matrix
Absorption correction	none
Function minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
Linear absorption coefficient	3.464/cm
Reflections used in L.S.	2412
Data reduction cut-off	3.00
Maximum $\sin(\theta)/\lambda$	0.650
L.S. parameters	407
Eta coefficient	1.0000
Residuals, R	0.057
Residuals, Rw	0.063
Max shift/e.s.d.	0.1018
Average shift/e.s.d.	0.0057

D. FRACTIONAL ATOMIC COORDINATES & U(iso)

Atom	x/a	y/b	z/c	U(iso)
S(1)	0.5691(3)	0.0297(1)	0.4316(3)	0.055(1)
S(2)	0.7206(2)	0.1317(1)	-0.0156(2)	0.043(1)
S(3)	0.9091(3)	0.0303(1)	0.4169(3)	0.054(1)
S(4)	0.5544(2)	0.1757(1)	0.2488(3)	0.046(1)
C(5)	0.7096(8)	0.1494(2)	0.3049(8)	0.036(4)
C(6)	0.7012(8)	0.1030(2)	0.3454(8)	0.033(4)
C(7)	0.8103(7)	0.1355(2)	0.1678(8)	0.033(4)
C(8)	0.6037(8)	0.0833(2)	0.4226(9)	0.039(4)
C(9)	0.9339(9)	0.1633(2)	0.1453(9)	0.038(4)
C(10)	1.0338(9)	0.0586(2)	0.1529(9)	0.043(4)
C(11)	0.8569(9)	0.1568(3)	0.5402(9)	0.043(5)
C(12)	0.8203(7)	0.0940(2)	0.2513(8)	0.033(4)
C(13)	0.4693(9)	0.1384(3)	0.1296(11)	0.050(5)
C(14)	0.5588(10)	0.1096(3)	0.0367(10)	0.050(5)
C(15)	1.0497(9)	0.1581(3)	0.2317(10)	0.045(5)
C(16)	1.0237(10)	0.0717(3)	0.0019(12)	0.047(5)
C(17)	0.4975(8)	0.1057(3)	0.5126(9)	0.041(4)
C(18)	0.9299(9)	0.1790(3)	0.6450(10)	0.052(5)
C(19)	0.7952(10)	0.2184(3)	0.4089(11)	0.053(5)
C(20)	0.7852(8)	0.1754(2)	0.4187(9)	0.039(4)
C(21)	0.9150(8)	0.0646(2)	0.2579(9)	0.039(4)
C(22)	0.5321(9)	0.1337(3)	0.6224(9)	0.051(5)
C(23)	0.4353(13)	0.1562(4)	0.6999(11)	0.063(7)
C(24)	0.8069(15)	-0.0113(3)	0.3325(11)	0.071(7)
C(25)	1.1552(9)	0.0391(3)	0.1970(12)	0.057(5)
C(26)	0.3583(11)	0.0984(4)	0.4892(11)	0.062(6)
C(27)	0.9273(11)	0.1978(3)	0.0550(10)	0.056(6)
C(28)	1.1301(11)	0.0671(3)	-0.0988(13)	0.066(6)
C(29)	0.8711(12)	0.2420(3)	0.5100(13)	0.069(7)
C(30)	0.9374(11)	0.2226(3)	0.6263(12)	0.068(7)
C(31)	0.2953(11)	0.1500(4)	0.6747(13)	0.074(7)
C(32)	1.1432(12)	0.2214(4)	0.1402(13)	0.073(8)
C(33)	0.6710(14)	0.0057(3)	0.2870(11)	0.061(6)
C(34)	1.1538(11)	0.1865(3)	0.2298(14)	0.062(6)
C(35)	0.2588(10)	0.1208(4)	0.5673(12)	0.079(7)
C(36)	1.2506(11)	0.0487(3)	-0.0522(14)	0.067(7)
C(37)	1.2614(11)	0.0345(3)	0.0958(15)	0.074(7)
C(38)	1.0296(12)	0.2279(3)	0.0501(12)	0.072(7)

Temperature factor of the form: exp[-2pi^2U], U=U(iso)
or 1/3 SUM(i)SUM(j){U(ij)*astar(i).astar(j).a(i).a(j).cos(ij)}

E. ANISOTROPIC THERMAL PARAMETERS

Atom	U11	U22	U33	U12	U13	U23
S(1)	0.075(2)	0.046(1)	0.046(1)	-0.024(1)	0.001(1)	0.008(1)
S(2)	0.043(1)	0.055(1)	0.030(1)	-0.002(1)	-0.003(1)	0.004(1)
S(3)	0.074(2)	0.047(1)	0.041(1)	0.010(1)	-0.001(1)	0.012(1)
S(4)	0.043(1)	0.048(1)	0.048(1)	0.010(1)	0.000(1)	0.003(1)
C(5)	0.034(4)	0.043(4)	0.030(3)	-0.005(3)	0.005(3)	-0.001(3)
C(6)	0.042(4)	0.035(4)	0.023(3)	-0.002(3)	-0.009(3)	0.002(3)
C(7)	0.039(4)	0.035(4)	0.026(3)	0.003(3)	0.000(3)	0.003(3)
C(8)	0.047(5)	0.041(4)	0.029(4)	-0.007(4)	-0.006(4)	0.007(4)
C(9)	0.041(4)	0.037(4)	0.035(4)	0.000(4)	0.005(4)	-0.004(3)
C(10)	0.053(5)	0.031(4)	0.044(4)	0.001(4)	0.003(4)	-0.006(4)
C(11)	0.043(4)	0.048(5)	0.038(4)	0.001(4)	0.003(4)	-0.002(4)
C(12)	0.038(4)	0.036(4)	0.025(3)	-0.005(3)	-0.007(3)	0.002(3)
C(13)	0.041(5)	0.064(6)	0.047(5)	-0.007(5)	-0.005(4)	0.005(5)
C(14)	0.043(5)	0.057(6)	0.051(5)	-0.002(5)	-0.004(5)	0.015(5)
C(15)	0.044(5)	0.042(5)	0.047(5)	-0.004(4)	0.010(4)	0.001(4)
C(16)	0.054(6)	0.035(5)	0.053(5)	0.010(4)	0.009(5)	0.000(4)
C(17)	0.040(4)	0.050(5)	0.034(4)	-0.006(4)	0.000(4)	0.011(4)
C(18)	0.041(5)	0.071(6)	0.042(4)	-0.013(5)	0.005(4)	-0.016(5)
C(19)	0.070(6)	0.040(5)	0.051(5)	0.003(5)	0.012(5)	0.008(4)
C(20)	0.041(4)	0.035(4)	0.041(4)	-0.003(3)	0.012(4)	-0.003(4)
C(21)	0.050(5)	0.036(4)	0.033(4)	-0.002(4)	-0.006(4)	0.006(3)
C(22)	0.039(5)	0.081(7)	0.033(4)	-0.002(5)	0.006(4)	0.009(5)
C(23)	0.079(8)	0.070(7)	0.039(5)	0.003(7)	0.005(6)	0.001(5)
C(24)	0.13(1)	0.04(1)	0.05(1)	-0.02(1)	0.00(1)	0.01(0)
C(25)	0.042(5)	0.056(6)	0.072(6)	-0.001(4)	-0.005(5)	-0.010(5)
C(26)	0.056(6)	0.092(8)	0.039(5)	-0.022(6)	-0.002(5)	0.008(6)
C(27)	0.057(6)	0.066(6)	0.046(5)	-0.006(5)	-0.006(5)	0.022(5)
C(28)	0.070(7)	0.056(6)	0.071(7)	-0.017(5)	0.031(6)	-0.009(5)
C(29)	0.095(8)	0.055(6)	0.058(6)	-0.020(6)	0.012(7)	-0.017(5)
C(30)	0.072(7)	0.070(7)	0.063(6)	-0.029(6)	0.013(6)	-0.021(5)
C(31)	0.058(7)	0.100(9)	0.063(7)	0.015(6)	0.017(6)	0.028(6)
C(32)	0.077(8)	0.072(7)	0.071(7)	-0.039(7)	0.015(7)	0.001(6)
C(33)	0.108(9)	0.032(5)	0.042(5)	-0.014(6)	-0.004(6)	0.000(4)
C(34)	0.045(5)	0.069(7)	0.072(7)	-0.012(5)	0.004(6)	-0.007(6)
C(35)	0.043(5)	0.140(10)	0.054(6)	0.011(7)	0.007(5)	0.025(7)
C(36)	0.056(6)	0.056(6)	0.090(8)	-0.009(5)	0.018(6)	-0.023(6)
C(37)	0.054(6)	0.069(7)	0.100(9)	-0.002(6)	0.005(7)	-0.025(7)
C(38)	0.089(8)	0.069(7)	0.058(6)	-0.035(6)	-0.001(6)	0.009(5)

T=exp[-2pi**2(U11.h**2.astar**2+U22.k**2.bstar**2+U33.l**2.cstar**2 +2U12.h.k.astar.bstar+2U13.h.l.astar.cstar+2U23.k.l.bstar.cstar)]

F. Table 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
S(1)	C(8)	1.767(7)	S(1)	C(33)	1.80(1)
S(2)	C(7)	1.852(7)	S(2)	C(14)	1.80(1)
S(3)	C(21)	1.793(8)	S(3)	C(24)	1.84(1)
S(4)	C(5)	1.812(8)	S(4)	C(13)	1.806(10)
C(5)	C(6)	1.544(9)	C(5)	C(7)	1.63(1)
C(5)	C(20)	1.51(1)	C(6)	C(8)	1.34(1)
C(6)	C(12)	1.46(1)	C(7)	C(9)	1.52(1)
C(7)	C(12)	1.535(9)	C(8)	C(17)	1.50(1)
C(9)	C(15)	1.38(1)	C(9)	C(27)	1.37(1)
C(10)	C(16)	1.41(1)	C(10)	C(21)	1.50(1)
C(10)	C(25)	1.40(1)	C(11)	C(18)	1.37(1)
C(11)	C(20)	1.42(1)	C(12)	C(21)	1.329(10)
C(13)	C(14)	1.52(1)	C(15)	C(34)	1.37(1)
C(16)	C(28)	1.38(2)	C(17)	C(22)	1.37(1)
C(17)	C(26)	1.40(1)	C(18)	C(30)	1.42(1)
C(19)	C(20)	1.40(1)	C(19)	C(29)	1.39(1)
C(22)	C(23)	1.38(2)	C(23)	C(31)	1.40(2)
C(24)	C(33)	1.50(2)	C(25)	C(37)	1.38(2)
C(26)	C(35)	1.40(2)	C(27)	C(38)	1.40(1)
C(28)	C(36)	1.39(2)	C(29)	C(30)	1.37(2)
C(31)	C(35)	1.39(2)	C(32)	C(34)	1.38(2)
C(32)	C(38)	1.39(2)	C(36)	C(37)	1.39(2)

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(8)	S(1)	C(33)	106.5(4)	C(7)	S(2)	C(14)	102.6(4)
C(21)	S(3)	C(24)	98.6(4)	C(5)	S(4)	C(13)	103.6(4)
S(4)	C(5)	C(6)	118.3(5)	S(4)	C(5)	C(7)	115.7(5)
S(4)	C(5)	C(20)	109.6(5)	C(6)	C(5)	C(7)	86.4(5)
C(6)	C(5)	C(20)	114.3(6)	C(7)	C(5)	C(20)	110.8(6)
C(5)	C(6)	C(8)	128.3(7)	C(5)	C(6)	C(12)	91.0(5)
C(8)	C(6)	C(12)	140.1(6)	S(2)	C(7)	C(5)	112.6(5)
S(2)	C(7)	C(9)	107.6(5)	S(2)	C(7)	C(12)	113.3(5)
C(5)	C(7)	C(9)	114.7(6)	C(5)	C(7)	C(12)	85.4(5)
C(9)	C(7)	C(12)	121.9(6)	S(1)	C(8)	C(6)	128.8(6)
S(1)	C(8)	C(17)	108.4(6)	C(6)	C(8)	C(17)	122.6(6)
C(7)	C(9)	C(15)	120.7(7)	C(7)	C(9)	C(27)	121.2(8)
C(15)	C(9)	C(27)	117.4(8)	C(16)	C(10)	C(21)	119.7(8)
C(16)	C(10)	C(25)	117.4(8)	C(21)	C(10)	C(25)	122.9(7)
C(18)	C(11)	C(20)	123.3(8)	C(6)	C(12)	C(7)	92.9(5)
C(6)	C(12)	C(21)	132.4(7)	C(7)	C(12)	C(21)	133.6(7)
S(4)	C(13)	C(14)	117.3(6)	S(2)	C(14)	C(13)	113.9(7)
C(9)	C(15)	C(34)	121.6(9)	C(10)	C(16)	C(28)	121.9(9)
C(8)	C(17)	C(22)	121.6(7)	C(8)	C(17)	C(26)	121.1(8)
C(22)	C(17)	C(26)	117.2(8)	C(11)	C(18)	C(30)	117.8(9)
C(20)	C(19)	C(29)	122.9(9)	C(5)	C(20)	C(11)	121.0(6)
C(5)	C(20)	C(19)	123.2(7)	C(11)	C(20)	C(19)	115.7(8)
S(3)	C(21)	C(10)	115.6(5)	S(3)	C(21)	C(12)	117.0(6)
C(10)	C(21)	C(12)	127.3(7)	C(17)	C(22)	C(23)	122.1(9)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(22)	C(23)	C(31)	121(1)	S(3)	C(24)	C(33)	109.1(7)
C(10)	C(25)	C(37)	120.5(10)	C(17)	C(26)	C(35)	121(1)
C(9)	C(27)	C(38)	123.3(9)	C(16)	C(28)	C(36)	119(1)
C(19)	C(29)	C(30)	119.1(9)	C(18)	C(30)	C(29)	121.1(9)
C(23)	C(31)	C(35)	117(1)	C(34)	C(32)	C(38)	120(1)
S(1)	C(33)	C(24)	117.4(7)	C(15)	C(34)	C(32)	119(1)
C(26)	C(35)	C(31)	120.8(10)	C(28)	C(36)	C(37)	119(1)
C(25)	C(37)	C(36)	121.2(10)	C(27)	C(38)	C(32)	116.9(10)

H. Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
S(1)	C(8)	C(6)	C(5)	162.9(6)	S(1)	C(8)	C(6)	C(12)	-5(1)
S(1)	C(8)	C(17)	C(22)	127.9(8)	S(1)	C(8)	C(17)	C(26)	-51(1)
S(1)	C(33)	C(24)	S(3)	-57.3(9)	S(2)	C(7)	C(5)	S(4)	-21.2(6)
S(2)	C(7)	C(5)	C(6)	98.6(5)	S(2)	C(7)	C(5)	C(20)	-146.7(5)
S(2)	C(7)	C(9)	C(15)	-150.8(7)	S(2)	C(7)	C(9)	C(27)	38.7(9)
S(2)	C(7)	C(12)	C(6)	-97.2(5)	S(2)	C(7)	C(12)	C(21)	94.3(9)
S(2)	C(14)	C(13)	S(4)	-29.6(10)	S(3)	C(21)	C(10)	C(16)	156.2(6)
S(3)	C(21)	C(10)	C(25)	-21.8(10)	S(3)	C(21)	C(12)	C(6)	-5(1)
S(3)	C(21)	C(12)	C(7)	159.4(7)	S(4)	C(5)	C(6)	C(8)	-39(1)
S(4)	C(5)	C(6)	C(12)	132.8(5)	S(4)	C(5)	C(7)	C(9)	102.3(6)
S(4)	C(5)	C(7)	C(12)	-134.6(5)	S(4)	C(5)	C(20)	C(11)	143.6(7)
S(4)	C(5)	C(20)	C(19)	-39.5(10)	C(5)	S(4)	C(13)	C(14)	-32.3(8)
C(5)	C(6)	C(8)	C(17)	-13(1)	C(5)	C(6)	C(12)	C(7)	-16.4(6)
C(5)	C(6)	C(12)	C(21)	152.4(8)	C(5)	C(7)	S(2)	C(14)	-38.9(6)
C(5)	C(7)	C(9)	C(15)	83.0(9)	C(5)	C(7)	C(9)	C(27)	-87.5(9)
C(5)	C(7)	C(12)	C(6)	15.5(5)	C(5)	C(7)	C(12)	C(21)	-153.0(9)
C(5)	C(20)	C(11)	C(18)	178.5(8)	C(5)	C(20)	C(19)	C(29)	-176.6(9)
C(6)	C(5)	S(4)	C(13)	-40.7(6)	C(6)	C(5)	C(7)	C(9)	-137.8(6)
C(6)	C(5)	C(7)	C(12)	-14.7(5)	C(6)	C(5)	C(20)	C(11)	8(1)
C(6)	C(5)	C(20)	C(19)	-175.0(8)	C(6)	C(8)	S(1)	C(33)	-10.7(9)
C(6)	C(8)	C(17)	C(22)	-55(1)	C(6)	C(8)	C(17)	C(26)	125(1)
C(6)	C(12)	C(7)	C(9)	131.9(7)	C(6)	C(12)	C(21)	C(10)	179.9(7)
C(7)	S(2)	C(14)	C(13)	68.3(7)	C(7)	C(5)	S(4)	C(13)	59.7(6)
C(7)	C(5)	C(6)	C(8)	-157.2(8)	C(7)	C(5)	C(6)	C(12)	15.4(5)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(7)	C(5)	C(20)	C(11)	-87.6(8)	C(7)	C(5)	C(20)	C(19)	89.4(9)
C(7)	C(9)	C(15)	C(34)	-169.9(8)	C(7)	C(9)	C(27)	C(38)	169.1(8)
C(7)	C(12)	C(6)	C(8)	154(1)	C(7)	C(12)	C(21)	C(10)	-15(1)
C(8)	S(1)	C(33)	C(24)	94.7(8)	C(8)	C(6)	C(5)	C(20)	91.6(10)
C(8)	C(6)	C(12)	C(21)	-36(1)	C(8)	C(17)	C(22)	C(23)	176.2(9)
C(8)	C(17)	C(26)	C(35)	-176.6(9)	C(9)	C(7)	S(2)	C(14)	-166.3(5)
C(9)	C(7)	C(5)	C(20)	-23.1(8)	C(9)	C(7)	C(12)	C(21)	-36(1)
C(9)	C(15)	C(34)	C(32)	0(1)	C(9)	C(27)	C(38)	C(32)	1(1)
C(10)	C(16)	C(28)	C(36)	0(1)	C(10)	C(21)	S(3)	C(24)	-90.7(7)
C(10)	C(25)	C(37)	C(36)	0(1)	C(11)	C(18)	C(30)	C(29)	1(1)
C(11)	C(20)	C(19)	C(29)	0(1)	C(12)	C(6)	C(5)	C(20)	-95.8(7)
C(12)	C(6)	C(8)	C(17)	178.0(9)	C(12)	C(7)	S(2)	C(14)	56.0(6)
C(12)	C(7)	C(5)	C(20)	100.0(6)	C(12)	C(7)	C(9)	C(15)	-17(1)
C(12)	C(7)	C(9)	C(27)	172.0(7)	C(12)	C(21)	S(3)	C(24)	93.6(7)
C(12)	C(21)	C(10)	C(16)	-28(1)	C(12)	C(21)	C(10)	C(25)	153.3(8)
C(13)	S(4)	C(5)	C(20)	-174.2(5)	C(15)	C(9)	C(27)	C(38)	-1(1)
C(15)	C(34)	C(32)	C(38)	0(1)	C(16)	C(10)	C(25)	C(37)	2(1)
C(16)	C(28)	C(36)	C(37)	1(1)	C(17)	C(8)	S(1)	C(33)	166.1(6)
C(17)	C(22)	C(23)	C(31)	3(1)	C(17)	C(26)	C(35)	C(31)	-2(1)
C(18)	C(11)	C(20)	C(19)	1(1)	C(18)	C(30)	C(29)	C(19)	0(1)
C(20)	C(11)	C(18)	C(30)	-2(1)	C(20)	C(19)	C(29)	C(30)	-1(1)
C(21)	S(3)	C(24)	C(33)	-60.6(8)	C(21)	C(10)	C(16)	C(28)	179.6(8)
C(21)	C(10)	C(25)	C(37)	-179.8(8)	C(22)	C(17)	C(26)	C(35)	4(1)
C(22)	C(23)	C(31)	C(35)	0(1)	C(23)	C(22)	C(17)	C(26)	-4(1)

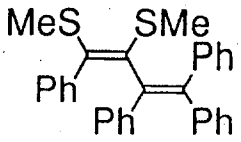
Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(23)	C(31)	C(35)	C(26)	0(1)	C(25)	C(10)	C(16)	C(28)	-2(1)
C(25)	C(37)	C(36)	C(28)	-1(1)	C(27)	C(9)	C(15)	C(34)	0(1)
C(27)	C(38)	C(32)	C(34)	0(1)					

X-Ray Structure

of

cis-1,2-Bis(methylthio)-1,3,4,4-tetraphenyl-1,3-butadiene



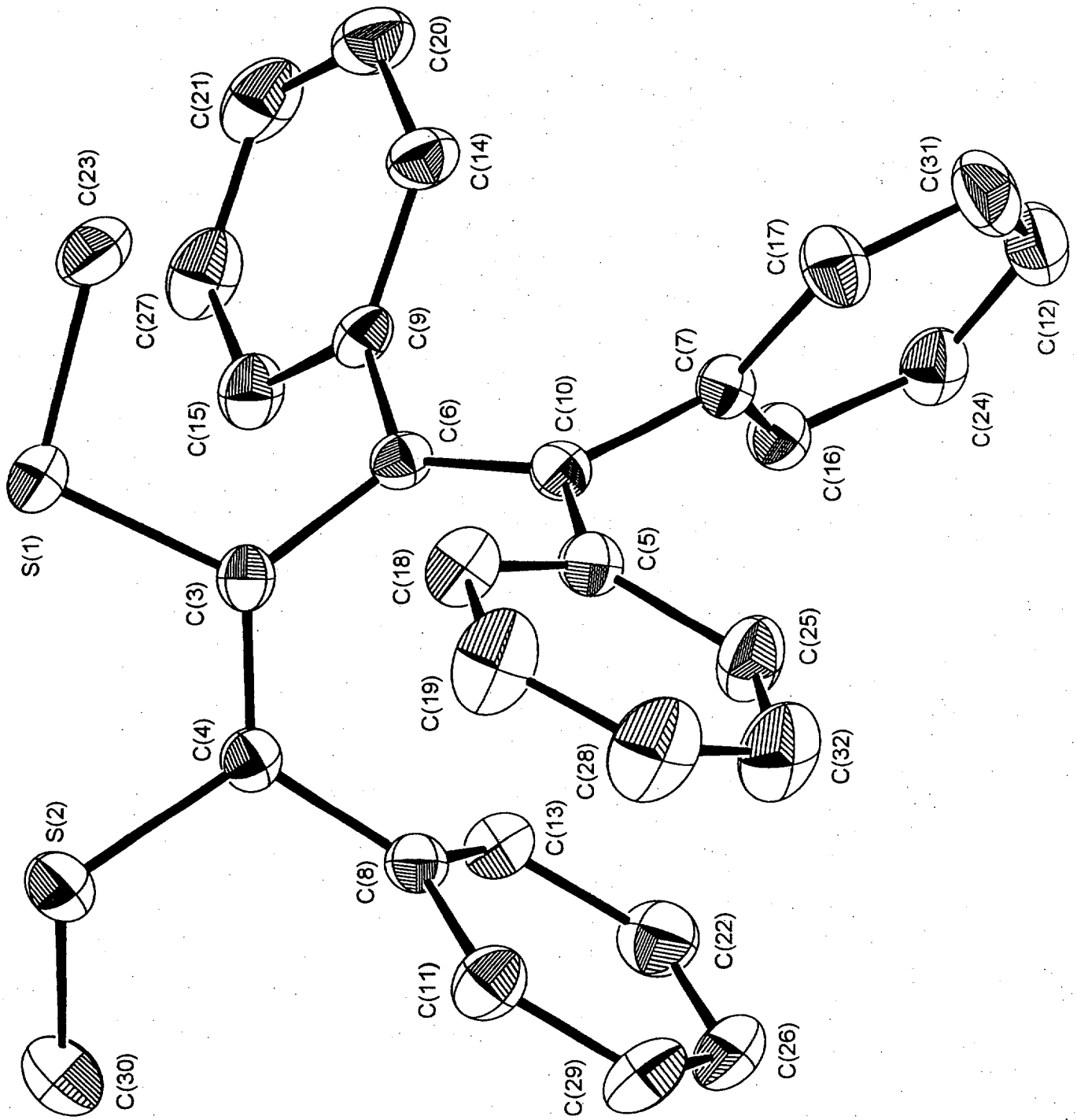

C(20)
C(21)
C(14)
C(23)
C(27)
C(9)
C(15)
C(6)
C(10)
C(7)
C(17)
C(31)
C(12)
C(16)
C(24)
S(1)
C(3)
C(18)
C(5)
C(19)
C(25)
C(4)
C(28)
C(32)
S(2)
C(13)
C(8)
C(22)
C(11)
C(26)
C(30)
C(29)

Table of Contents

I. Experimental Procedures

A. Data Collection

A colorless prism of $C_{30}H_{26}S_2$ having approximate dimension of 0.60 x 0.30 x 0.25 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with MoKα radiation (λ = 0.71073 Å) on a Mac Science MXC18 diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 22 reflection in measured by the computer controlled diagonal slit method of centering. The monoclinic cell parameters and calculated volume are: a = 12.994 (3), b = 18.214 (3), c = 10.907 (2) Å, β = 107.68 (1)°, V = 2459.5 (7) $Å^3$. For Z = 4, F.W. = 450.67, and the calculated density is 1.217 g/cm^3. From the systematic absences; the space group was determined to be $P2_1/n$ (#14).

The data were collected at a temperature of 25 ± 1 °C using the ω–2θ scan technique. The scan rate varied from 0 to 5 °/min (in omega). Data were collected to a maximum 2θ of 52.86°. The scan range (in deg.) was determined as a function of θ to correct for the separation of the Kα doublet ; the scan width was calculated as follows:

$$\omega \text{ scan width} = 1.03 + 0.350 \tan\theta$$

B. Data Reduction

A total of 6309 reflections were collected. As a check on crystal and electronic stability 3 representative reflections were measured every 100 reflections. The slope of the least-squares line through a plot of intensity versus time was -9 ± 1 counts/hour which corresponds to a total loss in intensity of 1.0%.

Lorentz and polarization corrections were applied to the data. No absorption correction was made. An extinction correction was not necessary.

C. Structure Solution and Refinement

The structure was solved by direct methods. A total of 32 atoms were located from an E-map prepared from the phase set with probability statistics. The remaining atoms were located in succeeding difference Fourire syntheses and refined anisotropically. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|-|Fc|)^2$ and the weight w is defined as 1.0 for all observed reflections.

Scattering factors were taken from Cromer and Waber (ref 1). Anomalous dispersion effects were included in Fc (ref 2); the values for $\Delta f'$ and $\Delta f''$ were those of Cromer (ref 3).

$$R = \Sigma||Fo|-|Fc||/\Sigma|Fo| = 0.060$$
$$Rw = SQRT(\Sigma w(Fo-Fc)^2/\Sigma wFo^2) = 0.108$$

The standard deviation of an observation of unit weight was 1.0. The highest peak in the final difference Fourier had a height of 0.87 e/Å^3 ; the minimum negative peak had a height of -0.67 e/Å^3 (ref 4). Plots of $\Sigma w(|Fo|-|Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ, and various classes of indices showed no unusual trends. All calculations were performed on a SUN computer.

References

(1) D. T. Cromer and J. T. Waber, "International Tables for X-Ray Crystallography", vol. IV, The Kynoch Press, Birmingham, England, 1974, Table 2.2B.
(2) J. A. Ibers and W. C. Hamilton, Acta Crystallogr., 17, 781 (1964).
(3) D. T. Cromer, "International Tables for X-Ray Crystallography," Vol. IV, The Knoch Press, Birmingham, England, 1974, Table 2.3.1.
(4) D. W. J. Cruickshank, Acta Crystallogr., 2, 154 (1949).

II. Tables

A. Crystal Data

Empirical Formula	$C_{30}H_{26}S_2$
Formula Weight	450.67
Crystal Color, Habit	yellow, prism
Crystal Dimension	0.60 X 0.30 X 0.25 mm
Crystal System	monoclinic
Lattice Parameters	a = 12.994 (3) Å b = 18.214 (3) Å c = 10.907 (2) Å β = 107.68 (1) °
Volume of unit cell	V = 2459.5 (7) Å^3
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.217 g/cm^3

B. Data Collection

Diffractometer	Mac Science MXC18
Radiation	MoKα (λ = 0.71073 Å)
Total Reflections Measured	6309
Reflection (hkl) limits	-16 < h < 16 0 < k < 23 0 < l < 14
Unique Reflections	2679

C. Structure Solution and Refinement

Structure Solution	Direct methods
Least squares refinement method	Full matrix
Absorption correction	none
Function minimized	$\Sigma w(\|Fo\|-\|Fc\|)^2$
Linear absorption coefficient	4.2796/cm
Reflections used in L.S.	2481
Data reduction cut-off	3.00
Maximum $\sin(\theta)/\lambda$	0.650
L.S. parameters	315
Eta coefficient	1.0000
Residuals, R	0.060
Residuals, Rw	0.108
Max shift/e.s.d.	0.1018
Average shift/e.s.d.	0.0057