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STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{40}H_{60}Sb_4$
Color; Habit	red orange block
Crystal size (mm)	.2 x .3 x .4
Crystal System	Tetragonal
Space Group	$I\bar{4}$
Unit Cell Dimensions	a = 15.895(3) Å
	c = 8.207(3) Å
Volume	2073.5(9) $Å^3$
Z	2
Formula weight	1027.8
Density(calc.)	1.646 Mg/m^3
Absorption Coefficient	2.602 mm^{-1}
F(000)	1008

Data Collection

Diffractometer Used	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	296
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 60.0°
Scan Type	Wyckoff
Scan Speed	Variable; 4.00 to 10.00°/min. in ω
Scan Range (ω)	1.00°
Background Measurement	Stationary crystal and stationary counter at beginning and end of scan, each for 50.0% of total scan time
Standard Reflections	3 measured every 197 reflections
Index Ranges	$0 \leq h \leq 22$, $0 \leq k \leq 22$ $0 \leq l \leq 11$
Reflections Collected	1723
Independent Reflections	1712 (R_{int} = 1.77%)
Observed Reflections	1553 ($F > 5.0\sigma(F)$)
Absorption Correction	Semi-empirical
Min./Max. Transmission	0.3985 / 0.4536

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Absolute Structure	η = 1.16(10)
Extinction Correction	χ = 0.00202(9), where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(F) + 0.0003F^2$
Number of Parameters Refined	102
Final R Indices (obs. data)	R = 2.04 %, wR = 2.71 %
R Indices (all data)	R = 2.42 %, wR = 2.82 %
Goodness-of-Fit	1.00
Largest and Mean Δ/σ	0.002, 0.000
Data-to-Parameter Ratio	15.2:1
Largest Difference Peak	0.34 $e\text{Å}^{-3}$
Largest Difference Hole	-0.36 $e\text{Å}^{-3}$

Table 1. Atomic coordinates ($x10^4$) and equivalent isotropic displacement coefficients ($Å^2 x10^3$)

	x	y	z	U(eq)
Sb(1)	9039(1)	4452(1)	1670(1)	33(1)
C(1)	8000(3)	4349(3)	4891(6)	41(1)
C(2)	7382(3)	4941(3)	4932(7)	42(1)
C(3)	6922(2)	4940(3)	3419(8)	40(1)
C(4)	7245(3)	4354(3)	2418(6)	39(1)
C(5)	7985(2)	3951(2)	3243(7)	36(1)
C(6)	8583(4)	4116(5)	6208(7)	72(2)
C(7)	7180(4)	5505(4)	6347(9)	73(2)
C(8)	6196(3)	5517(3)	3058(11)	70(2)
C(9)	6939(4)	4102(5)	760(7)	71(2)
C(10)	8049(3)	2992(3)	3177(11)	65(2)

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

Table 2. Bond lengths (Å)

Sb(1)-C(5)	2.260 (4)	Sb(1)-Sb(1A)	2.836 (1)
Sb(1)-Sb(1B)	2.836 (1)	C(1)-C(2)	1.361 (6)
C(1)-C(5)	1.492 (7)	C(1)-C(6)	1.471 (8)
C(2)-C(3)	1.441 (8)	C(2)-C(7)	1.501 (9)
C(3)-C(4)	1.343 (7)	C(3)-C(8)	1.504 (7)
C(4)-C(5)	1.500 (6)	C(4)-C(9)	1.500 (8)
C(5)-C(10)	1.528 (6)		

Table 3. Bond angles (°)

C(5)-Sb(1)-Sb(1A)	101.2(1)	C(5)-Sb(1)-Sb(1B)	105.8(1)
Sb(1A)-Sb(1)-Sb(1B)	76.7(1)	C(2)-C(1)-C(5)	107.7(4)
C(2)-C(1)-C(6)	127.6(5)	C(5)-C(1)-C(6)	124.7(4)
C(1)-C(2)-C(3)	110.1(4)	C(1)-C(2)-C(7)	125.9(5)
C(3)-C(2)-C(7)	124.0(4)	C(2)-C(3)-C(4)	109.5(4)
C(2)-C(3)-C(8)	123.9(5)	C(4)-C(3)-C(8)	126.6(6)
C(3)-C(4)-C(5)	108.6(4)	C(3)-C(4)-C(9)	128.0(5)
C(5)-C(4)-C(9)	123.4(5)	Sb(1)-C(5)-C(1)	110.9(3)
Sb(1)-C(5)-C(4)	99.9(3)	C(1)-C(5)-C(4)	104.0(3)
Sb(1)-C(5)-C(10)	106.3(3)	C(1)-C(5)-C(10)	117.0(5)
C(4)-C(5)-C(10)	117.5(4)		

Table 4. Anisotropic displacement coefficients ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sb(1)	29(1)	35(1)	35(1)	-3(1)	2(1)	0(1)
C(1)	35(2)	54(2)	33(2)	-3(2)	4(2)	8(2)
C(2)	38(2)	41(2)	47(2)	-6(2)	11(2)	-5(2)
C(3)	27(2)	37(2)	55(2)	-1(1)	3(2)	7(2)
C(4)	30(2)	47(2)	39(2)	-10(2)	0(2)	3(2)
C(5)	31(2)	31(1)	46(2)	1(1)	7(2)	-1(2)
C(6)	60(3)	110(5)	45(3)	7(3)	-5(2)	28(3)
C(7)	64(3)	87(4)	68(4)	-7(3)	17(3)	-35(4)
C(8)	40(2)	67(3)	103(6)	18(2)	16(3)	17(4)
C(9)	48(3)	117(6)	49(3)	-19(3)	-4(2)	-20(4)
C(10)	58(3)	31(2)	105(5)	1(2)	28(4)	10(3)

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + \ldots + 2hka^*b^*U_{12})$

Table 5. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x 10^3$)

	x	y	z	U
H(6A)	8486	4455	7157	80
H(6B)	8486	3536	6467	80
H(6C)	9154	4187	5852	80
H(7A)	7556	5400	7240	80
H(7B)	7248	6074	5981	80
H(7C)	6611	5418	6696	80
H(8A)	5995	5401	1978	80
H(8B)	5750	5424	3827	80
H(8C)	6379	6091	3122	80
H(9A)	6466	4446	471	80
H(9B)	7379	4177	-28	80
H(9C)	6771	3522	779	80
H(10A)	7610	2758	3841	80
H(10B)	7976	2812	2070	80
H(10C)	8586	2804	3571	80