JOC The Journal of Organic Chemistry

J. Org. Chem., 1997, 62(22), 7575-7584, DOI:10.1021/jo970614v

SUPPORTING INFORMATION FOR: JO970614V

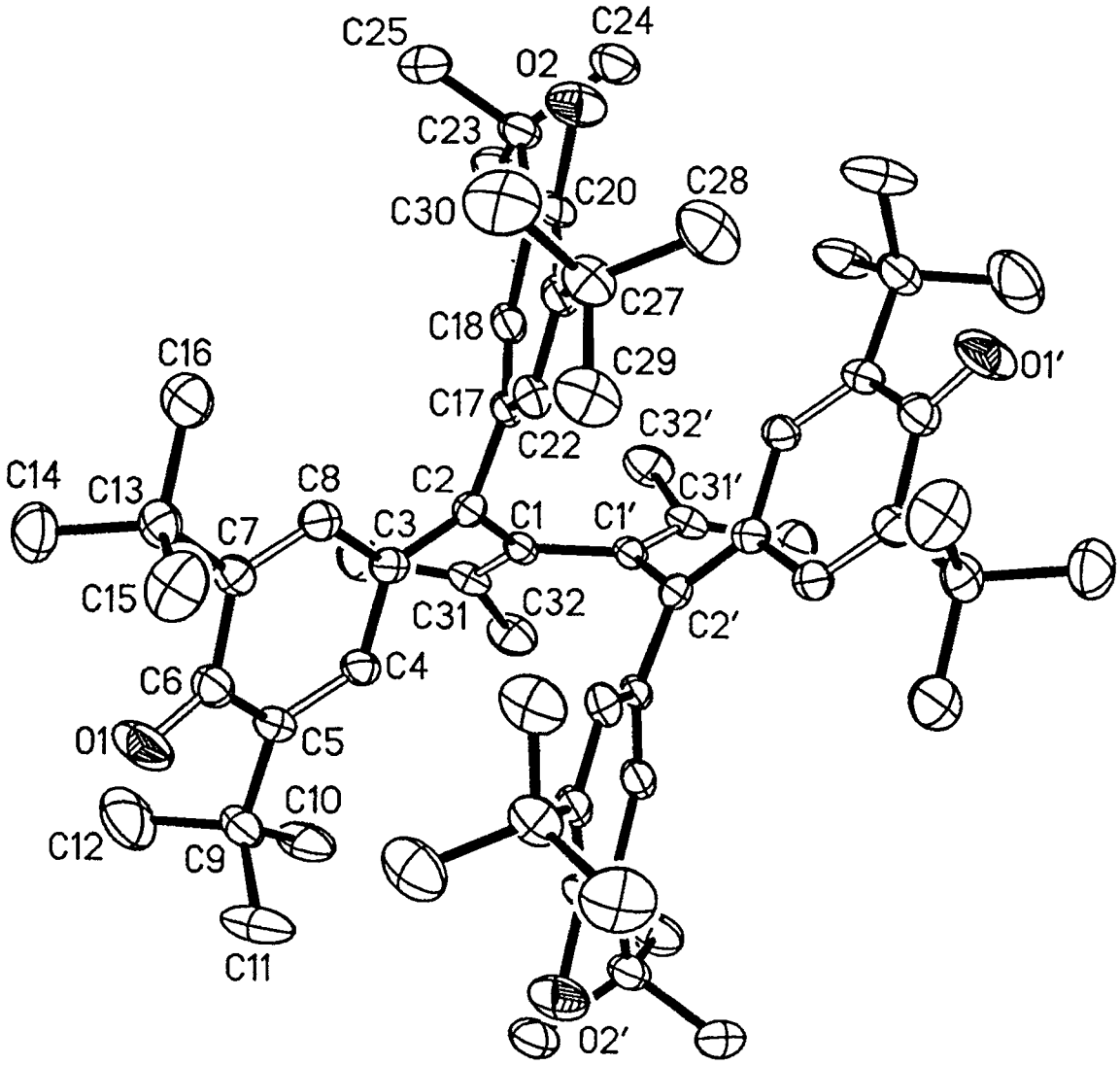

Figure S1: ORTEP plot of Bisgalvinol **7.** * The plot is shown at the 50% probability level.

X-ray Diffraction Study of Bisgalvinol 7

Crystals of bisgalvinol 7 were grown from a saturated methanol solution. The grown crystals were submitted to the X-ray Diffraction Center at the Department of Chemistry, University of California-Irvine, run by Dr. Joseph Ziller. Experimental details and data follow.

Collection of X-ray Diffraction Data

An orange crystal of approximate dimensions 0.27 X 0.33 X 0.40 m m was oil-mounted on a glass fiber and transferred to the Siemens P3 diffractometer (which is equipped with a modified LT-2 low temperature system). Determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described techniques similar to those of Churchill.[1] Low temperature (158 K) intensity data were collected via a θ–2θ scan technique with MoKα radiation under the conditions given in Table B.1.

All 4280 data were corrected for Lorentz and polarization effects and placed on an approximately absolute scale. Any reflection with I(net) < O was assigned the value $|F_o| = 0$. The systematic extinctions observed were *hkl* for $h+k$ = 2n+1 and *h0l* for l = 2n+1; the diffraction symmetry was 2/m. The two possible monoclinic space groups are Cc [C^4_s ; No. 9] or C2/c [C^6_{2h}; No. 15]. The centrosymmetric space group C2/c was later determined to be the correct choice.

Solution and Refinement of the Crystal Structure

All crystallographic calculations were carried out using either the UCI modified version of the UCLA Crystallographic Computing Package[2] or the SHELXTL PLUS program set.[3] The analytical scattering factors for neutral atoms were used throughout the analysis;[4a] both the real ($\Delta f'$) and imaginary ($i\Delta f''$) components of anomalous dispersion were included. The quantity

minimized during least-squares analysis was $Sw(|F_o|-|F_c|)^2$ where $w^{-1} = \sigma^2(|F_o|) + 0.0007(|F_o|)^2$.

The structure was solved by direct methods (SHELXTL PLUS), and refined by full-matrix least-squares techniques. The molecule is located about a two-fold rotation axis (0, y, 1/4). Hydrogen atoms were included using a riding model with d(C-H) = 0.96 Å, d(O-H) 0.85 Å and U(iso) = 0.08 Å^2 . Refinement of positional and anisotropic thermal parameters led to convergence with R_F = 7.1%, R_{wF} = 7.2% and GOF = 1.50 for 317 variables refined against the 2773 data with $|F_o| > 3.0\sigma(|F_o|)$ A final difference-Fourier map was "clean," ρ(max) = 0.28 eÅ^{-3}.

References

1. Churchill, M. R.; Lashewycz, R. A.; Rotella, F. *J. Inorg. Chem.* **1977**, *16*, 265.

2. UCLA Crystallographic Computing Package, University of California Los Angeles, 1981, C. Strouse; personal communication.

3. Siemens Analytical X-Ray Instruments, Inc.,; Madison, WI 1990.

4. *International Tables for X-Ray Crystallography*; Kynoch Press: Birmingham, England, 1974; (a) pp 99-101; (b) pp 149-150.

Acknowledgement. Funds for the purchase of the Siemens R3m/V diffractometer system were made available to UCI from the National Science Foundation under Grant CHE-85-14495.

Table S1: Experimental Data for the X-ray Diffraction Study.

Formula: $C_{66}H_{94}O_4$

Fw: 951.4

Temperature (K): 158

Crystal System: Monoclinic

Space Group: C2/c

a = 17.650(4) Å

b = 18.434(5) Å

c = 19.199(4) Å

β = 104.88(2)°

V = 6037(2) A3

Z = 4

D_{calcd} Mg/m^3 = 1.05

Diffractometer: Siemens P3 (R3m/V System)

Radiation: MoKα (λ = 0.710730 Å)

Monochromator: Highly oriented graphite

Data Collected: $+h, +k, +l$

Scan Type: θ-2θ

Scan Range: 1.20° plus Kα-separation

Scan Speed: 3.0 deg min^{-1} (in ω)

2θ Range: 4.0 to 45.0°

μ(MoKα), mm^{-1} = 0.059

Reflections Collected: 4280

Reflections with $|F_o| > 3.0\sigma(|F_o|$: 2773

No. of Variables: 317

R_F = 7.11%, R_{wF} = 7.2%

Goodness of Fit: 1.50

Table S2: Atomic coordinates ($X10^4$) and equivalent isotropic displacement coefficients ($Å^2$ $X10^4$).

	x	y	z	U(eq)
C(1)	-393(2)	6051(2)	2234(2)	216(13)
C(2)	-600(2)	6739(2)	1818(2)	221(14)
C(3)	-1288(2)	7109(2)	1798(2)	231(14)
C(4)	-1813(2)	6882(2)	2227(2)	244(14)
C(5)	-2520(2)	7198(2)	2183(2)	272(15)
C(6)	-2726(2)	7863(2)	1748(2)	308(16)
C(7)	-2223(2)	8087(2)	1273(2)	258(14)
C(8)	-1551(2)	7713(2)	1311(2)	253(14)
C(9)	-3101(2)	6894(2)	2586(2)	333(16)
C(10)	-2819(3)	6163(2)	2942(2)	468(19)
C(11)	-3200(3)	7411(3)	3181(3)	555(21)
C(12)	-3896(3)	6755(3)	2038(3)	664(23)
C(1)	-2470(2)	8731(2)	760(2)	296(15)
C(14)	-3309(3)	8622(3)	290(2)	467(18)
C(15)	-2434(3)	9431(2)	1201(3)	533(20)
C(16)	-1937(3)	8822(2)	250(2)	440(18)
C(17)	-25(2)	7041(2)	1443(2)	203(13)
C(18)	241(2)	6620(2)	949(2)	266(14)
C(19)	701(2)	6914(2)	521(2)	266(14)
C(20)	910(2)	7655(2)	636(2)	277(15)
C(21)	691(2)	8086(2)	1146(2)	257(14)
C(22)	208(2)	7766(2)	1533(2)	256(15)
C(23)	906(2)	6462(2)	-83(2)	313(15)
C(24)	1799(2)	6442(2)	-25(2)	411(17)
C(25)	456(3)	6774(3)	-822(2)	439(18)
C(26)	637(3)	5675(2)	-67(2)	407(18)
C(27)	924(2)	8892(2)	1261(2)	352(17)
C(28)	1814(3)	8978(3)	1502(3)	535(20)
C(29)	596(3)	9238(2)	1850(3)	475(19)
C(30)	586(3)	9327(2)	566(3)	592(22)
C(31)	-897(2)	5482(2)	2157(2)	273(15)
C(32)	-801(3)	4851(2)	2673(2)	396(17)
C(33)	-1626(2)	5421(2)	1540(2)	363(16)
O(1)	-3297(2)	8236(2)	1784(2)	530(13)
O(2)	1353(2)	7960(2)	212(2)	441(12)

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

Table S3: Interatomic Distances (A) with Esd's.

C(1)-C(2)	1.493(5)	C(1)-C(31)	1.359(5)
C(1)-C(1')	1.498(6)	C(2)-C(3)	1.386(5)
C(2)-C(17)	1.495(6)	C(3)-C(4)	1.449(6)
C(3)-C(8)	1.451(5)	C(4)-C(S)	1.361(6)
C(S)-C(6)	1.476(5)	C(5)-C(9)	1.540(6)
C(6)-C(7)	1.485(6)	C(6)-O(1)	1.236(5)
C(7)-C(8)	1.359(6)	C(7)-C(13)	1.532(5)
C(9)-C(10)	1.534(6)	C(9)-C(11)	1.532(7)
C(9)-C(12)	1.544(6)	C(13)-C(14)	1.538(5)
C(13)-C(15)	1.535(6)	C(13)-C(16)	1.530(7)
C(17)-C(18)	1.396(6)	C(17)-C(22)	1.395(5)
C(18)-C(19)	1.403(6)	C(19)-C(20)	1.418(6)
C(19)-C(23)	1.545(6)	C(20)-C(21)	1.389(6)
C(20)-O(2)	1.384(5)	C(21)-C(22)	1.397(6)
C(21)-C(27)	1.542(5)	C(23)-C(24)	1.552(6)
C(23)-C(25)	1.549(5)	C(23)-C(26)	1.529(6)
C(27)-C(28)	1.527(6)	C(27)-C(29)	1.536(7)
C(27)-C(30)	1.539(6)	C(31)-C(32)	1.508(6)
C(31)-C(33)	1.513(5)		

Table S4: Interatomic Angles (Deg.) with Esd's.

C(2)-C(1)-C(31)	122.3(3)	C(2)-C(1)-C(1')	114.9(2)
C(31)-C(1)-C(1')	122.7(3)	C(1)-C(2)-C(3)	121.7(4)
C(1)-C(2)-C(17)	118.2(3)	C(3)-C(2)-C(17)	120.1(3)
C(2)-C(3)-C(4)	121.7(3)	C(2)-C(3)-C(8)	121.9(4)
C(4)-C(3)-C(8)	116.3(3)	C(3)-C(4)-C(5)	123.4(3)
C(4)-C(5)-C(6)	118.7(4)	C(4)-C(5)-C(9)	122.1(3)
C(6)-C(S)-C(9)	119.2(3)	C(S)-C(6)-C(7)	118.6(4)
C(S)-C(6)-O(1)	121.1(4)	C(7)-C(6)-O(1)	120.3(4)
C(6)-C(7)-C(8)	118.6(3)	C(6)-C(7)-C(13)	119.6(3)
C(8)-C(7)-C(13)	121.8(4)	C(3)-C(8)-C(7)	123.6(4)
C(S)-C(9)-C(10)	111.2(3)	C(S)-C(9)-C(11)	111.5(3)
C(10)-C(9)-C(11)	107.5(4)	C(S)-C(9)-C(12)	108.9(4)
C(10)-C(9)-C(12)	107.0(4)	C(11)-C(9)-C(12)	110.7(4)
C(7)-C(13)-C(14)	110.3(3)	C(7)-C(13)-C(15)	109.4(3)
C(14)-C(13)-C(15)	109.3(3)	C(7)-C(13)-C(16)	112.1(3)
C(14)-C(13)-C(16)	107.2(3)	C(1S)-C(13)-C(16)	108.4(3)
C(2)-C(17)-C(18)	120.5(3)	C(2)-C(17)-C(22)	120.8(3)
C(18)-C(17)-C(22)	118.5(4)	C(17)-C(18)-C(19)	121.9(4)
C(18)-C(19)-C(20)	116.6(4)	C(18)-C(19)-C(23)	120.9(3)
C(20)-C(19)-C(23)	122.4(4)	C(19)-C(20)-C(21)	123.5(4)
C(19)-C(20)-O(2)	117.6(4)	C(21)-C(20)-O(2)	118.9(3)
C(20)-C(21)-C(22)	116.8(3)	C(20)-C(21)-C(27)	122.6(4)
C(22)-C(21)-C(27)	120.5(4)	C(17)-C(22)-C(21)	122.6(4)
C(19)-C(23)-C(24)	112.9(3)	C(19)-C(23)-C(25)	108.9(3)
C(24)-C(23)-C(25)	110.3(4)	C(19)-C(23)-C(26)	111.3(4)
C(24)-C(23)-C(26)	106.9(3)	C(25)-C(23)-C(26)	106.4(3)
C(21)-C(27)-C(28)	111.0(3)	C(21)-C(27)-C(29)	111.6(4)
C(28)-C(27)-C(29)	106.6(3)	C(21)-C(27)-C(30)	110.4(3)
C(28)-C(27)-C(30)	110.4(4)	C(29)-C(27)-C(30)	106.6(4)
C(1)-C(31)-C(32)	124.3(3)	C(1)-C(31)-C(33)	122.9(3)
C(32)-C(31)-C(33)	112.8(3)		

Table S5: Anisotropic displacement coefficients ($Å^2$ X10^4).

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	183(20)	240(21)	242(22)	-5(18)	84(17)	-35(18)
C(2)	213(22)	251(22)	191(21)	-23(18)	38(18) -	45(18)
C(3)	242(23)	232(22)	226(22)	-51(19)	73(19)	-41(19)
C(4)	222(23)	276(22)	243(22)	-53(19)	76(19) -	42(18)
C(5)	255(24)	319(25)	273(24)	10(19)	125(20)	-1(20)
C(6)	264(25)	343(26)	323(25)	15(21)	90(21) -	44(21)
C(7)	256(23)	233(22)	293(23)	7(19)	86(20) -	33(19)
C(8)	253(23)	253(22)	251(23)	-50(19)	65(19) -	25(19)
C(9)	250(24)	372(26)	416(26)	44(21)	153(21)	81(22)
C(10)	467(30)	524(31)	527(31)	-10(25)	333(26)	91(26)
C(11)	722(37)	500(31)	640(34)	116(28)	531(31)	92(27)
C(12)	352(31)	841(42)	830(40)	-82(29)	209(30)	216(33)
C(13)	277(24)	253(23)	350(25)	56(19)	66(21)	38(20)
C(14)	373(28)	520(30)	461(29)	79(24)	19(24)	113(24)
C(15)	670(36)	333(27)	548(33)	78(25)	65(28)	28(25)
C(16)	406(28)	425(28)	499(30)	119(23)	136(24)	213(24)
C(17)	139(21)	238(23)	231(22)	12(18)	49(18)	27(18)
C(18)	188(22)	296(23)	308(24)	-9(19)	54(19)	44(20)
C(19)	215(22)	330(25)	261(23)	-15(19)	77(19)	15(19)
C(20)	218(23)	324(24)	323(24)	-73(20)	130(20)	66(21)
C(21)	189(22)	287(23)	293(23)	0(19)	61(20)	25(20)
C(22)	200(22)	244(23)	315(24)	14(18)	52(20)	19(19)
C(23)	274(24)	386(25)	318(25)	-38(20)	147(21)	-44(21)
C(24)	361(26)	512(29)	422(27)	-1(24)	211(22)	-53(24)
C(25)	431(28)	602(32)	323(27)	-68(25)	167(23)	38(24)
C(26)	427(28)	374(27)	485(30)	-60(22)	237(24)	-112(23)
C(27)	376(27)	230(23)	478(29)	-72(20)	158(24)	45(21)
C(28)	465(32)	434(29)	752(37)	-172(25)	237(28)	-145(27)
C(29)	543(32)	279(25)	662(34)	-127(23)	263(28)	-79(24)
C(30)	799(40)	304(27)	735(38)	-7(27)	307(32)	166(26)
C(31)	259(24)	255(23)	354(25)	12(19)	171(21)	2(19)
C(32)	406(28)	325(25)	512(29)	-54(21)	219(24)	88(23)
C(33)	304(26)	374(26)	409(27)	-110(21)	88(22)	-124(22)
O(1)	444(20)	589(22)	662(23)	280(17)	333(18)	211(18)
O(2)	504(20)	365(18)	532(20)	-153(15)	272(17)	37(15)

The anisotropic displacement exponent takes the form:

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

Table S6: H-Atom coordinates ($X10^4$) and isotropic displacement coefficients ($Å^2$ $X10^4$).

	x	y	z	U
H(4A)-	1653	6489	2561	800
H(8A)-	1231	7855	998	800
H(10A)	-2755	5826	2579	800
H(10B)	-2325	6227	3293	800
H(10C)	-3199	5978	3175	800
H(11A)	-2698	7489	3515	800
H(11B)	-3406	7865	2969	800
H(11C)	-3554	7205	3432	800
H(12A)	-3819	6428	1673	800
H(12B)	-4256	6544	2281	800
H(12C)	-4107	7204	1818	800
H(14A)	-3339	8184	13	800
H(14B)	-3667	8591	591	800
H(14C)	-3447	9028	-31	800
H(1SA)	-1909	9493	1498	800
H(1SB)	-2572	9837	880	800
H(1SC)	-2792	9400	1502	800
H(16A)	-1952	8392	-35	800
H(16B)	-2115	9228	-62	800
H(16C)	-1409	8907	530	800
H(18A)	109	6114	905	800
H(22A)	36	8054	1880	800
H(24A)	1990	6928	-36	800
H(24B)	2064	6215	422	800
H(24C)	1897	6171	-421	800
H(25A)	-97	6782	-856	800
H(25B)	635	7258	-872	800
H(25C)	553	6475	-1200	800
H(26A)	83	5657	-107	800
H(26B)	749	5414	-462	800
H(26C)	916	5458	381	800
H(28A)	1951	9482	1571	800
H(28B)	2009	8722	1949	800
H(28C)	2042	8778	1141	800
H(29A)	746	9740	1908	800
H(29B)	33	9202	1717	800
H(29C)	804	8986	2296	800
H(30A)	734	9827	648	800
H(30B)	791	9137	186	800
H(30C)	24	9288	431	800

H(32A)	-331	4915	3055	800
H(32B)	-1246	4829	2874	800
H(32C)	-765	4409	2419	800
H(33A)	-1659	5833	1228	800
H(33B)	-1600	4986	1272	800
H(33C)	-2081	5405	1727	800
H(2A)	1434	8402	336	800

Table S7: Fractional Coordinates for all atoms (including symmetry equivalents).

ATOM	X	Y	Z
C1	-0.03931	0.60512	0.22335
C2	-0.05997	0.67388	0.18176
C3	-0.12883	0.71093	0.17981
C4	-0.18127	0.68819	0.22266
C5	-0.25202	0.71982	0.21828
C6	-0.27265	0.78633	0.17478
C7	-0.22233	0.80871	0.12733
C8	-0.15506	0.77130	0.13113
C9	-0.31007	0.68939	0.25861
C10	-0.28186	0.61631	0.29418
C11	-0.31998	0.74109	0.31813
C12	-0.38959	0.67549	0.20382
C13	-0.24696	0.87309	0.07604
C14	-0.33093	0.86216	0.02901
C15	-0.24337	0.94308	0.12009
C16	-0.19373	0.88215	0.02504
C17	0.00249	0.70414	0.14426
C18	0.02412	0.66199	0.09493
C19	0.07006	0.69140	0.05211
C20	0.09098	0.76553	0.06362
C21	0.06913	0.80860	0.11459
C22	0.02082	0.77659	0.15326
C23	0.09058	0.64616	-0.00827
C24	0.17987	0.64418	-0.00250
C25	0.04558	0.67742	-0.08224
C26	0.06366	0.56749	-0.00672
C27	0.09238	0.88920	0.12606
C28	0.18135	0.89781	0.15020
C29	0.05956	0.92384	0.18498
C30	0.05857	0.93268	0.05663
C31	-0.08973	0.54822	0.21573
C32	-0.08008	0.48515	0.26730
C33	-0.16256	0.54208	0.15399

O1	-0.32972	0.82363	0.17844
O2	0.13532	0.79600	0.02125
C1A	0.03930	0.60512	0.27665
C2A	0.05996	0.67388	0.31824
C3A	0.12883	0.71093	0.32019
C4A	0.18126	0.68819	0.27734
C5A	0.25202	0.71982	0.28172
C6A	0.27265	0.78633	0.32522
C7A	0.22232	0.80871	0.37267
C8A	0.15506	0.77130	0.36887
C9A	0.31007	0.68939	0.24139
C10A	0.28186	0.61631	0.20582
C11A	0.31997	0.74109	0.18187
C12A	0.38958	0.67549	0.29618
C13A	0.24696	0.87310	0.42396
C14A	0.33093	0.86216	0.47099
C1SA	0.24337	0.94308	0.37991
C16A	0.19373	0.88215	0.47496
C17A	0.00248	0.70414	0.35574
C18A	-0.02412	0.66199	0.40507
C19A	-0.07006	0.69140	0.44789
C20A	-0.09098	0.76554	0.43638
C21A	-0.06913	0.80860	0.38541
C22A	-0.02082	0.77659	0.34674
C23A	-0.09058	0.64617	0.50827
C24A	-0.17987	0.64418	0.50250
C25A	-0.04558	0.67742	0.58224
C26A	-0.06366	0.56749	0.50672
C27A	-0.09238	0.88920	0.37394
C28A	-0.18135	0.89781	0.34980
C29A	-0.05957	0.92385	0.31502
C30A	-0.05857	0.93268	0.44337
C31A	0.08973	0.54822	0.28427
C32A	0.08007	0.48516	0.23270
C33A	0.16256	0.54209	0.34601
O1A	0.32972	0.82363	0.32156
O2A	-0.13533	0.79600	0.47875
H4A	-0.16527	0.64888	0.25606
H8B	-0.12310	0.78548	0.09977
H10A	-0.27553	0.58261	0.25791
H10B	-0.23250	0.62273	0.32930
H10C	-0.31992	0.59783	0.31754
H11A	-0.26978	0.74894	0.35154
H11B	-0.34057	0.78649	0.29695
H11C	-0.35543	0.72046	0.34322
H12A	-0.38186	0.64279	0.16728

H12B	-0.42556	0.65436	0.22805
H12C	-0.41070	0.72039	0.18178
H14A	-0.33392	0.81842	0.00127
H14B	-0.36668	0.85913	0.05914
H14C	-0.34471	0.90281	-0.00306
H15A	-0.19090	0.94935	0.14983
H15B	-0.25722	0.98367	0.08795
H15C	-0.27919	0.93999	0.15016
H16A	-0.19522	0.83915	-0.00351
H16B	-0.21149	0.92279	-0.00621
H16C	-0.14091	0.89068	0.05301
H18A	0.01090	0.61141	0.09054
H22A	0.00358	0.80542	0.18797
H24A	0.19896	0.69281	-0.00355
H24B	0.20639	0.62149	0.04224
H24C	0.18967	0.61713	-0.04209
H25A	-0.00969	0.67822	-0.08562
H25B	0.06351	0.72583	-0.08718
H25C	0.05528	0.64749	-0.12002
H26A	0.00829	0.56569	-0.01067
H26B	0.07491	0.54142	-0.04625
H26C	0.09163	0.54578	0.03808
H28A	0.19505	0.94816	0.15706
H28B	0.20089	0.87224	0.19488
H28C	0.20418	0.87779	0.11406
H29A	0.07456	0.97396	0.19080
H29B	0.00334	0.92017	0.17167
H29C	0.08037	0.89858	0.22964
H30A	0.07336	0.98268	0.06475
H30B	0.07914	0.91369	0.01859
H30C	0.00237	0.92882	0.04307
H32A	-0.03311	0.49154	0.30546
H32B	-0.12457	0.48287	0.28741
H32C	-0.07650	0.44092	0.24193
H33A	-0.16590	0.58334	0.12279
H33B	-0.16001	0.49858	0.12719
H33C	-0.20808	0.54053	0.17269
H32D	0.03311	0.49154	0.19454
H32E	0.12457	0.48287	0.21259
H32F	0.07650	0.44092	0.25807
H33D	0.16590	0.58335	0.37721
H33E	0.16001	0.49858	0.37281
H33F	0.20807	0.54053	0.32731
H4AA	0.16526	0.64888	0.24394
H8AA	0.12310	0.78548	0.40023
H18B	-0.01091	0.61141	0.40946

H22B	-0.00358	0.80542	0.31203
H10D	0.27552	0.58261	0.24209
H10E	0.23249	0.62274	0.17070
H10F	0.31992	0.59783	0.18246
H11D	0.26978	0.74894	0.14846
H11E	0.34057	0.78649	0.20305
H11F	0.35543	0.72046	0.15678
H12D	0.38186	0.64279	0.33272
H12E	0.42556	0.65436	0.27195
H12F	0.41069	0.72039	0.31822
H14D	0.33392	0.81842	0.49873
H14E	0.36668	0.85913	0.44086
H14F	0.34471	0.90281	0.50306
H15D	0.19090	0.94935	0.35017
H15E	0.25721	0.98368	0.41205
H15F	0.27919	0.93999	0.34984
H16D	0.19522	0.83915	0.50351
H16E	0.21148	0.92279	0.50621
H16F	0.14090	0.89068	0.44699
H24D	-0.19896	0.69281	0.50355
H24E	-0.20639	0.62149	0.45776
H24F	-0.18967	0.61713	0.54209
H25D	0.00969	0.67823	0.58562
H25E	-0.06351	0.72583	0.58718
H25F	-0.05529	0.64749	0.62002
H26D	-0.00830	0.56569	0.51067
H26E	-0.07492	0.54142	0.54625
H26F	-0.09163	0.54579	0.46192
H28D	-0.19506	0.94816	0.34294
H28E	-0.20089	0.87224	0.30512
H28F	-0.20418	0.87779	0.38594
H29D	-0.07457	0.97396	0.30920
H29E	-0.00335	0.92018	0.32833
H29F	-0.08037	0.89858	0.27036
H30D	-0.07336	0.98268	0.43525
H30E	-0.07914	0.91369	0.48141
H30F	-0.00238	0.92882	0.45693
H2A	0.14345	0.84016	0.03363
H2AA	-0.14345	0.84016	0.46637

STRUCTURE DETERMINATION SUMMARY

Table S8: Crystal Data.

Empirical Formula	$C_{66}H_{94}O_4$
Color; Habit	Orange prism
Crystal Size (mm)	0.27 X 0.33 X 0.40
Crystal System	Monoclinic
Space Group	C2/c
Unit Cell Dimensiona	a = 17.650(4) A
	b = 18.434(5) A
	c = 19.199(4) A
	β = 104.88(2)°
Volume	6037(2) Å^3
Z	4
Formula weight	951.4
Density(calc.)	1.047 Mg/m^3
Absorption Coefficient	0.059 mm^{-1}
F(000)	2088

Table S9: Data Collection.

Diffractometer System	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	158
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 45.0°
Scan Type	9-2
Scan Speed	Fixed; 3.00° /min. in ω
Scan Range (ω)	1.20° plus Kα-separation
Background Measurement	Estimated from 96 step profile
Standard Reflections	2 measured every 98 reflections
Index Ranges	$0 \le h \le 19$, $0 \le k \le 19$
	$-20 \le l \le 20$
Reflections Collected	4280
Independent Reflections	3492 (R_{int} = 1.3 %); ($\|F_o\| > 0$)
Observed Reflections	2773 ($\|F_o\| > 3.0\sigma(\|F_o\|)$)

Table S10: Solution and Refinement.

System Used	Siemens SHELXTL PLUS (MicroVAX II)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\Sigma w(\lvert F_o \rvert - \lvert F_c \rvert)^2$
Extinction Correction	$\chi = 0.00009(4)$, where $F^* = F [1 + 0.002\chi F^2/\sin(2\theta)]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(\lvert F_o \rvert) + 0.0007(\lvert F_o \rvert)^2$
Final R Indices (obs. data)	$R_F = 7.1\%$, $R_{wF} = 7.2$
Goodness-of-Fit	1.50
Number of Variables	317
Data-to-Parameter Ratio	8.7:1
Largest and Mean Δ/σ	0.001, < 0.001
Largest Difference Peak	0.28 eÅ^{-3}
Largest Difference Hole	-0.31 eÅ^{-3}