Data collection

A crystal (approximate dimensions 0.28 x 0.24 x 0.08 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Siemens SMART Platform CCD diffractometer for a data collection at 173(2) K. A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 26 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 60 seconds and a detector distance of 4.86 cm. A randomly oriented region of reciprocal space was surveyed to the extent of one sphere and to a resolution of 0.84 Å. Four major sections of frames were collected with 0.30° steps in ω at four different ϕ settings and a detector position of -28° in 2θ. The intensity data were corrected for absorption and decay (SADABS).[1] Final cell constants were calculated from the xyz centroids of 1614 strong reflections from the actual data collection after integration (SAINT).[2] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-97[3] and refined using SHELXL-97.[3] The space group *P*-1 was determined based on the lack of systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters. All hydrogen atoms were refined positionally and with relative isotropic displacement parameters. The final full matrix least squares refinement converged to $R1 = 0.0414$ and $wR2 = 0.0972$ (F^2, all data).

Structure description

The structure is the one suggested.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Department of Chemistry, University of Minnesota. All calculations were performed using Pentium computers using the current SHELXTL suite of programs. All publications arising from this report MUST either 1)include William W. Brennessel as a coauthor or 2)acknowledge William W. Brennessel, Victor G. Young, Jr., and the X-Ray Crystallographic Laboratory.

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[1] An empirical correction for absorption anisotropy, R. Blessing, *Acta Cryst.* **A51**, 33-38 (1995).

[2] SAINT V6.2, Bruker Analytical X-Ray Systems, Madison, WI (2001).

[3] SHELXTL V6.10, Bruker Analytical X-Ray Systems, Madison, WI (2000).

Some equations of interest:

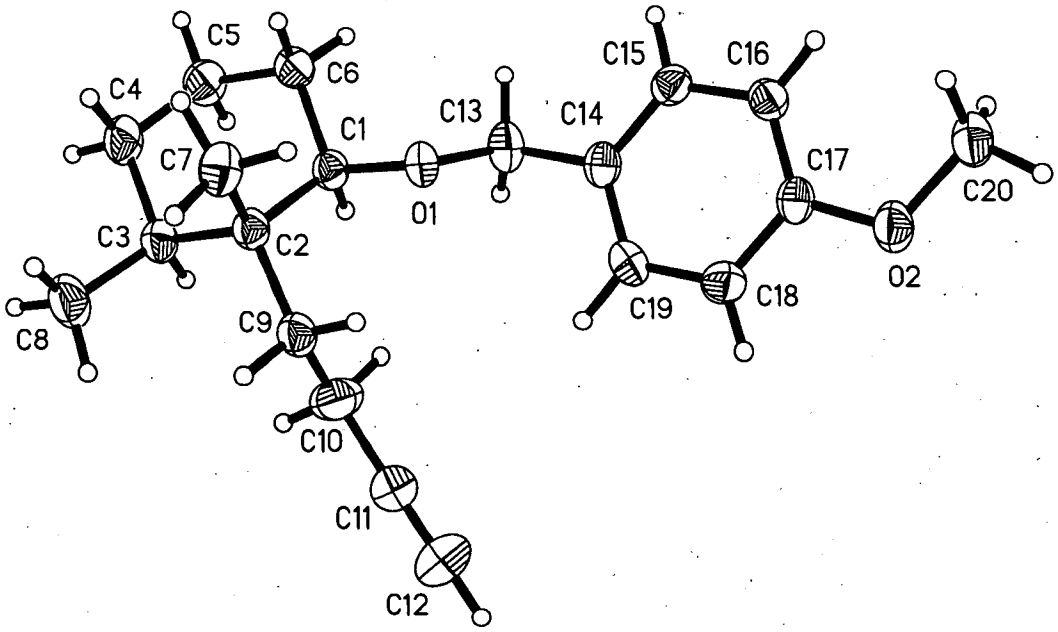
$$R_{int} = \Sigma \left| F_o^2 - \langle F_o^2 \rangle \right| / \Sigma |F_o^2|$$

$$R_1 = \Sigma \left| |F_o| - |F_c| \right| / \Sigma |F_o|$$

$$wR2 = [\Sigma[w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]]^{1/2}$$

$$\text{where } w = q / [\sigma^2 (F_o^2) + (a*P)^2 + b*P + d + e*\sin(\theta)]$$

$$\text{GooF} = S = [\Sigma[w(F_o^2 - F_c^2)^2] / (n-p)]^{1/2}$$


C5
C6
C4
C15
C16
C13
C14
C1
C7
C17
C20
O1
C3
C2
O2
C8
C9
C19
C18
C10
C11
C12

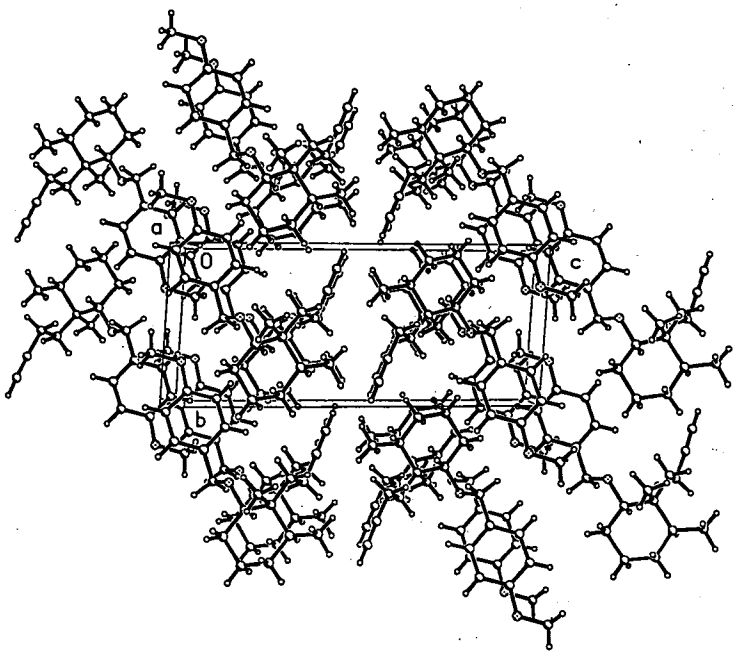

a
0
c
b

Table 1. Crystal data and structure refinement for 03070.

Identification code	03070	
Empirical formula	C20 H28 O2	
Formula weight	300.42	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	*P*-1	
Unit cell dimensions	a = 7.1495(8) Å	α = 93.773(2)°
	b = 7.2623(8) Å	β = 97.577(2)°
	c = 17.207(2) Å	γ = 92.186(2)°
Volume	882.7(2) Å^3	
Z	2	
Density (calculated)	1.130 Mg/m^3	
Absorption coefficient	0.071 mm^{-1}	
F(000)	328	
Crystal color, morphology	colorless, plate	
Crystal size	0.28 x 0.24 x 0.08 mm^3	
Theta range for data collection	1.20 to 25.06°	
Index ranges	$-8 \le h \le 8$, $-8 \le k \le 8$, $-20 \le l \le 20$	
Reflections collected	8486	
Independent reflections	3116 [R(int) = 0.0212]	
Observed reflections	2471	
Completeness to theta = 25.06°	99.3%	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.954804	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3116 / 0 / 283	
Goodness-of-fit on F^2	1.010	
Final R indices [I>2sigma(I)]	$R1$ = 0.0414, $wR2$ = 0.0910	
R indices (all data)	$R1$ = 0.0566, $wR2$ = 0.0972	
Largest diff. peak and hole	0.142 and -0.172 e.Å^{-3}	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 03070. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O1	1058(1)	4498(1)	1948(1)	29(1)
O2	-3437(1)	-2680(1)	340(1)	37(1)
C1	1663(2)	6186(2)	2409(1)	28(1)
C2	3123(2)	5716(2)	3100(1)	29(1)
C3	3631(2)	7541(2)	3633(1)	33(1)
C4	4318(3)	9107(2)	3165(1)	40(1)
C5	2931(3)	9440(2)	2446(1)	44(1)
C6	2384(2)	7670(2)	1922(1)	36(1)
C7	4876(2)	4922(2)	2795(1)	39(1)
C8	5054(3)	7301(3)	4358(1)	48(1)
C9	2265(2)	4178(2)	3547(1)	35(1)
C10	411(3)	4567(3)	3864(1)	49(1)
C11	-423(3)	2900(3)	4146(1)	54(1)
C12	-1088(3)	1545(4)	4347(1)	76(1)
C13	-724(2)	4578(2)	1473(1)	36(1)
C14	-1412(2)	2656(2)	1160(1)	30(1)
C15	-1669(2)	2162(2)	362(1)	31(1)
C16	-2349(2)	400(2)	58(1)	30(1)
C17	-2772(2)	-902(2)	569(1)	28(1)
C18	-2532(2)	-426(2)	1375(1)	34(1)
C19	-1861(2)	1322(2)	1666(1)	34(1)
C20	-3665(2)	-3209(2)	-486(1)	37(1)

Table 3. Bond lengths [Å] and angles [°] for 03070.

O(1)-C(13)	1.4257(17)	C(12)-H(12A)	0.92(2)
O(1)-C(1)	1.4348(16)	C(13)-C(14)	1.503(2)
O(2)-C(17)	1.3737(16)	C(13)-H(13A)	1.034(16)
O(2)-C(20)	1.4340(18)	C(13)-H(13B)	1.003(17)
C(1)-C(6)	1.524(2)	C(14)-C(15)	1.384(2)
C(1)-C(2)	1.541(2)	C(14)-C(19)	1.401(2)
C(1)-H(1A)	1.004(15)	C(15)-C(16)	1.394(2)
C(2)-C(7)	1.539(2)	C(15)-H(15A)	0.997(15)
C(2)-C(9)	1.549(2)	C(16)-C(17)	1.384(2)
C(2)-C(3)	1.5634(19)	C(16)-H(16A)	0.945(15)
C(3)-C(8)	1.527(2)	C(17)-C(18)	1.394(2)
C(3)-C(4)	1.537(2)	C(18)-C(19)	1.378(2)
C(3)-H(3A)	1.010(16)	C(18)-H(18A)	0.954(16)
C(4)-C(5)	1.520(2)	C(19)-H(19A)	0.975(16)
C(4)-H(4A)	1.010(18)	C(20)-H(20A)	0.988(19)
C(4)-H(4B)	0.972(17)	C(20)-H(20B)	1.007(19)
C(5)-C(6)	1.527(2)	C(20)-H(20C)	0.982(19)
C(5)-H(5A)	0.985(18)	C(13)-O(1)-C(1)	113.86(10)
C(5)-H(5B)	1.017(18)	C(17)-O(2)-C(20)	116.87(12)
C(6)-H(6A)	0.999(17)	O(1)-C(1)-C(6)	112.69(12)
C(6)-H(6B)	0.991(17)	O(1)-C(1)-C(2)	107.88(11)
C(7)-H(7A)	0.991(19)	C(6)-C(1)-C(2)	113.36(12)
C(7)-H(7B)	1.008(19)	O(1)-C(1)-H(1A)	107.6(8)
C(7)-H(7C)	0.98(2)	C(6)-C(1)-H(1A)	107.4(8)
C(8)-H(8A)	1.01(2)	C(2)-C(1)-H(1A)	107.6(8)
C(8)-H(8B)	0.98(2)	C(7)-C(2)-C(1)	110.46(12)
C(8)-H(8C)	1.03(2)	C(7)-C(2)-C(9)	106.25(12)
C(9)-C(10)	1.527(2)	C(1)-C(2)-C(9)	109.20(12)
C(9)-H(9A)	0.982(16)	C(7)-C(2)-C(3)	111.76(12)
C(9)-H(9B)	0.993(17)	C(1)-C(2)-C(3)	106.96(11)
C(10)-C(11)	1.469(2)	C(9)-C(2)-C(3)	112.22(12)
C(10)-H(10A)	0.968(19)	C(8)-C(3)-C(4)	110.44(13)
C(10)-H(10B)	1.026(19)	C(8)-C(3)-C(2)	113.49(13)
C(11)-C(12)	1.169(3)	C(4)-C(3)-C(2)	111.68(12)

C(8)-C(3)-H(3A)	107.4(9)	C(10)-C(9)-H(9B)	109.5(9)
C(4)-C(3)-H(3A)	107.7(9)	C(2)-C(9)-H(9B)	108.7(9)
C(2)-C(3)-H(3A)	105.8(9)	H(9A)-C(9)-H(9B)	106.8(13)
C(5)-C(4)-C(3)	112.74(13)	C(11)-C(10)-C(9)	111.45(16)
C(5)-C(4)-H(4A)	110.1(9)	C(11)-C(10)-H(10A)	107.3(11)
C(3)-C(4)-H(4A)	108.2(9)	C(9)-C(10)-H(10A)	111.5(11)
C(5)-C(4)-H(4B)	110.7(10)	C(11)-C(10)-H(10B)	108.5(10)
C(3)-C(4)-H(4B)	107.9(10)	C(9)-C(10)-H(10B)	110.4(10)
H(4A)-C(4)-H(4B)	107.0(13)	H(10A)-C(10)-H(10B)	107.5(15)
C(4)-C(5)-C(6)	112.15(14)	C(12)-C(11)-C(10)	177.8(2)
C(4)-C(5)-H(5A)	111.0(10)	C(11)-C(12)-H(12A)	179.0(16)
C(6)-C(5)-H(5A)	110.7(10)	O(1)-C(13)-C(14)	109.13(12)
C(4)-C(5)-H(5B)	108.2(10)	O(1)-C(13)-H(13A)	110.5(9)
C(6)-C(5)-H(5B)	108.5(10)	C(14)-C(13)-H(13A)	110.1(9)
H(5A)-C(5)-H(5B)	106.1(14)	O(1)-C(13)-H(13B)	107.3(9)
C(1)-C(6)-C(5)	109.57(13)	C(14)-C(13)-H(13B)	111.1(9)
C(1)-C(6)-H(6A)	109.5(9)	H(13A)-C(13)-H(13B)	108.7(12)
C(5)-C(6)-H(6A)	110.0(9)	C(15)-C(14)-C(19)	117.84(13)
C(1)-C(6)-H(6B)	110.3(9)	C(15)-C(14)-C(13)	121.06(14)
C(5)-C(6)-H(6B)	109.5(9)	C(19)-C(14)-C(13)	121.09(13)
H(6A)-C(6)-H(6B)	107.9(13)	C(14)-C(15)-C(16)	121.98(14)
C(2)-C(7)-H(7A)	111.8(11)	C(14)-C(15)-H(15A)	120.5(9)
C(2)-C(7)-H(7B)	111.4(10)	C(16)-C(15)-H(15A)	117.5(9)
H(7A)-C(7)-H(7B)	108.5(15)	C(17)-C(16)-C(15)	119.23(14)
C(2)-C(7)-H(7C)	109.3(11)	C(17)-C(16)-H(16A)	121.5(9)
H(7A)-C(7)-H(7C)	108.8(15)	C(15)-C(16)-H(16A)	119.3(9)
H(7B)-C(7)-H(7C)	106.9(14)	O(2)-C(17)-C(16)	124.47(13)
C(3)-C(8)-H(8A)	110.4(11)	O(2)-C(17)-C(18)	115.93(13)
C(3)-C(8)-H(8B)	112.9(12)	C(16)-C(17)-C(18)	119.60(13)
H(8A)-C(8)-H(8B)	105.6(16)	C(19)-C(18)-C(17)	120.51(14)
C(3)-C(8)-H(8C)	112.3(11)	C(19)-C(18)-H(18A)	121.5(9)
H(8A)-C(8)-H(8C)	107.3(16)	C(17)-C(18)-H(18A)	117.9(9)
H(8B)-C(8)-H(8C)	108.0(16)	C(18)-C(19)-C(14)	120.84(14)
C(10)-C(9)-C(2)	116.89(13)	C(18)-C(19)-H(19A)	118.8(9)
C(10)-C(9)-H(9A)	107.3(9)	C(14)-C(19)-H(19A)	120.4(9)
C(2)-C(9)-H(9A)	107.3(9)	O(2)-C(20)-H(20A)	105.5(10)

O(2)-C(20)-H(20B)	111.8(10)	H(20A)-C(20)-H(20C)	109.2(14)
H(20A)-C(20)-H(20B)	111.7(14)	H(20B)-C(20)-H(20C)	108.5(14)
O(2)-C(20)-H(20C)	110.1(10)		

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 03070. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U_{11} + ... + 2\ h\ k\ a^*\ b^*\ U_{12}\ ]$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	27(1)	27(1)	31(1)	-4(1)	-2(1)	-1(1)
O2	43(1)	28(1)	36(1)	-1(1)	-2(1)	-4(1)
C1	31(1)	25(1)	29(1)	-3(1)	3(1)	-1(1)
C2	31(1)	26(1)	29(1)	-1(1)	1(1)	-1(1)
C3	36(1)	30(1)	31(1)	-2(1)	1(1)	-4(1)
C4	46(1)	29(1)	41(1)	-4(1)	-1(1)	-9(1)
C5	58(1)	29(1)	43(1)	6(1)	0(1)	-9(1)
C6	43(1)	34(1)	30(1)	4(1)	1(1)	-6(1)
C7	32(1)	37(1)	47(1)	-3(1)	2(1)	2(1)
C8	59(1)	40(1)	39(1)	-1(1)	-12(1)	-11(1)
C9	41(1)	29(1)	31(1)	3(1)	-3(1)	-4(1)
C10	57(1)	48(1)	46(1)	8(1)	20(1)	-6(1)
C11	56(1)	62(1)	41(1)	6(1)	5(1)	-18(1)
C12	83(2)	77(2)	69(2)	16(1)	11(1)	-38(1)
C13	30(1)	33(1)	40(1)	-3(1)	-5(1)	1(1)
C14	22(1)	30(1)	36(1)	0(1)	-2(1)	3(1)
C15	26(1)	33(1)	34(1)	4(1)	3(1)	-1(1)
C16	28(1)	36(1)	26(1)	0(1)	3(1)	0(1)
C17	22(1)	29(1)	33(1)	0(1)	-1(1)	2(1)
C18	37(1)	34(1)	32(1)	8(1)	3(1)	-1(1)
C19	34(1)	38(1)	26(1)	-1(1)	-2(1)	2(1)
C20	34(1)	35(1)	39(1)	-8(1)	3(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 03070.

	x	y	z	U(eq)
H1A	540(20)	6670(20)	2635(9)	34
H3A	2410(20)	7920(20)	3823(9)	40
H4A	5580(30)	8780(20)	3003(10)	48
H4B	4540(20)	10220(20)	3518(10)	48
H5A	3430(20)	10410(20)	2143(10)	53
H5B	1740(30)	9940(20)	2632(10)	53
H6A	3500(20)	7230(20)	1686(9)	43
H6B	1400(20)	7930(20)	1487(10)	43
H7A	5610(30)	5870(30)	2561(11)	59
H7B	5730(30)	4390(20)	3229(11)	59
H7C	4480(30)	3910(30)	2395(11)	59
H8A	5310(30)	8510(30)	4686(12)	73
H8B	4600(30)	6420(30)	4708(12)	73
H8C	6330(30)	6870(30)	4215(12)	73
H9A	2030(20)	3070(20)	3182(9)	41
H9B	3220(20)	3870(20)	3984(10)	41
H10A	590(30)	5500(30)	4299(11)	59
H10B	-540(30)	5040(20)	3433(11)	59
H12A	-1590(30)	470(30)	4509(13)	92
H13A	-610(20)	5400(20)	1013(10)	43
H13B	-1610(20)	5150(20)	1814(9)	43
H15A	-1360(20)	3070(20)	-19(9)	37
H16A	-2480(20)	110(20)	-491(9)	36
H18A	-2820(20)	-1350(20)	1715(9)	41
H19A	-1730(20)	1620(20)	2232(10)	40
H20A	-4120(30)	-4520(30)	-537(10)	55
H20B	-4580(30)	-2410(20)	-793(10)	55
H20C	-2440(30)	-3100(20)	-686(10)	55

Table 6. Torsion angles [°] for 03070.

C13-O1-C1-C6	-75.54(16)	C7-C2-C9-C10	-175.89(14)
C13-O1-C1-C2	158.56(12)	C1-C2-C9-C10	-56.74(18)
O1-C1-C2-C7	62.95(15)	C3-C2-C9-C10	61.69(18)
C6-C1-C2-C7	-62.56(16)	C2-C9-C10-C11	169.84(14)
O1-C1-C2-C9	-53.56(14)	C9-C10-C11-C12	-58(6)
C6-C1-C2-C9	-179.06(12)	C1-O1-C13-C14	-167.57(12)
O1-C1-C2-C3	-175.22(11)	O1-C13-C14-C15	-116.89(15)
C6-C1-C2-C3	59.28(15)	O1-C13-C14-C19	64.79(18)
C7-C2-C3-C8	-59.89(18)	C19-C14-C15-C16	-0.1(2)
C1-C2-C3-C8	179.10(14)	C13-C14-C15-C16	-178.44(13)
C9-C2-C3-C8	59.36(18)	C14-C15-C16-C17	-0.3(2)
C7-C2-C3-C4	65.73(17)	C20-O2-C17-C16	1.23(19)
C1-C2-C3-C4	-55.28(16)	C20-O2-C17-C18	-178.96(13)
C9-C2-C3-C4	-175.02(13)	C15-C16-C17-O2	-179.55(12)
C8-C3-C4-C5	-178.60(16)	C15-C16-C17-C18	0.6(2)
C2-C3-C4-C5	54.10(19)	O2-C17-C18-C19	179.63(13)
C3-C4-C5-C6	-52.8(2)	C16-C17-C18-C19	-0.5(2)
O1-C1-C6-C5	177.90(13)	C17-C18-C19-C14	0.1(2)
C2-C1-C6-C5	-59.21(17)	C15-C14-C19-C18	0.2(2)
C4-C5-C6-C1	53.9(2)	C13-C14-C19-C18	178.54(14)

Symmetry transformations used to generate equivalent atoms: