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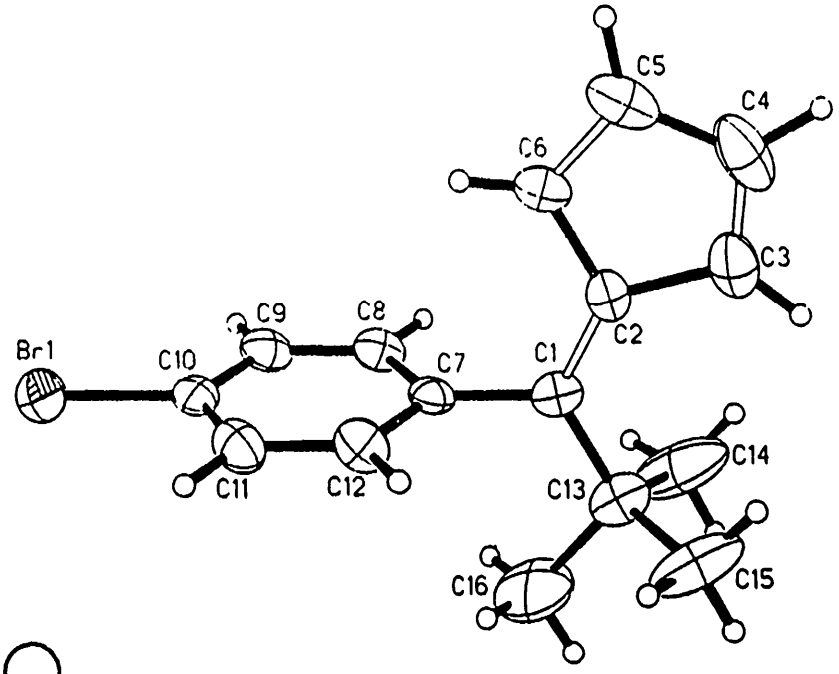
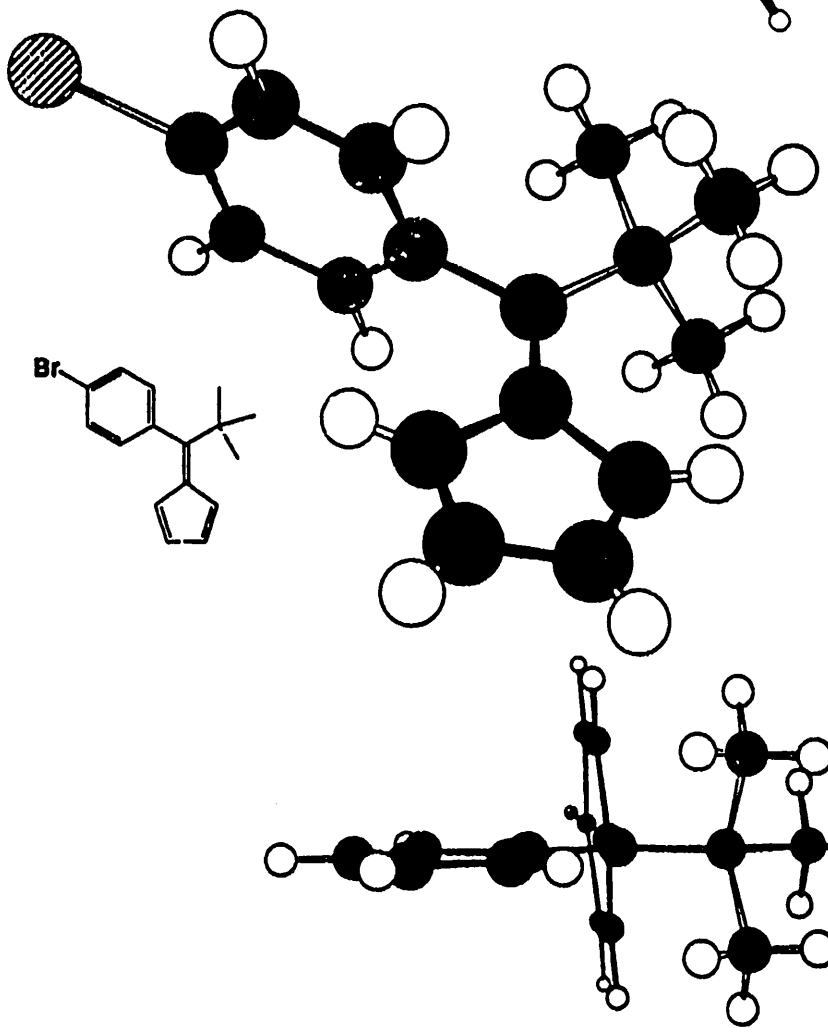
SILVERMAN 13273-13283

H-13283-M1

6-(*p*-Bromophenyl)-6-*t*-butylfulvene (9)





Torsional angles:

2-1-7-12	-93.5 °
2-1-7-8	87.0°
7-1-2-3	179.4 °
7-1-2-6	0.0°
13-1-2-3	-1.1°
13-1-2-6	180.0°

Atom distances:

C1-C11	1.491Å

H-13283-M2

Collection of X-ray Diffraction Data. A yellow crystal of approximate dimensions 0.10 x 0.20 x 0.36 mm was oil-mounted on a glass fiber and transferred to the Syntex $P2_1$ automated four-circle diffractometer which is equipped with a modified LT-1 low temperature system. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described methods similar to those of Churchill[1]. Intensity data were collected at 168 K using a θ-2θ scan technique with Mo Kα radiation under the conditions described in Table 1. All 2079 data were corrected for absorption and for Lorentz and polarization effects and were placed on an approximately absolute scale. The diffraction symmetry was 2/m with systematic absences $0k0$ for k = 2n+1 and $h0\ell$ for ℓ = 2n+1. The centrosymmetric monoclinic space group $P2_1/c$ [C_{2h}^5; No. 14] is therefore uniquely defined.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using either our locally 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[4b] were included. The quantity minimized during least-squares analysis was $\sum w(|F_o|-|F_c|)^2$ where $w^{-1} = \sigma^2(|F_o|) + 0.0005(|F_o|)^2$.

The structure was solved by direct methods (SHELXTL PLUS) and refined by full-matrix least-squares techniques. Hydrogen atoms were included using a riding model with d(C-H) = 0.96Å and U(iso) = 0.08Å^2. Refinement of positional and thermal parameters led to convergence with R_F = 4.8%; R_{wF} = 5.0% and GOF = 1.40 for 155 variables refined against those 1437 data with $|F_o| > 3.0\sigma(|F_o|)$. A final difference-Fourier synthesis showed no significant features, ρ(max) = 0.52eÅ^{-3}.

H-13283-M3

Table 1. Experimental Data for the X-ray Diffraction Study

Formula: $C_{16}H_{17}Br$

Fw: 289.2

Temperature (K): 168

Crystal System: Monoclinic

Space Group: $P2_1/c$ [C_{2h}^5; No. 14]

a = 9.3008(7) Å

b = 16.2059(11) Å

c = 9.6876(8) Å

β = 105.155(6)°

V = 1409.4(2) Å^3

Z = 4

D_{calcd}, Mg/m^3 = 1.363

Diffractometer: Syntex $P2_1$ (Siemens R3m/V System)

Radiation: Mo Kα ($\bar{\lambda}$ = 0.710730 Å)

Monochromator: Highly oriented graphite

Data Collected: $+h,+k,\pm\ell$

Scan Type: θ-2θ

Scan Width: 1.2° plus Kα-separation

Scan Speed: 3.0 deg min^{-1} (in ω)

2θ Range, deg: 4.0 to 45.0

μ(Mo Kα), mm^{-1} = 2.86

Absorption Correction: Semi-empirical (ψ-scan method)

Reflections Collected: 2079

Reflections with $|F_o| > 3.0\sigma(|F_o|)$: 1437

No. of Variables: 155

R_F = 4.8%, R_{wF} = 5.0%

Goodness of Fit: 1.14

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

H-13283-M4

	x	y	z	U(eq)
C(1)	9536(5)	10334(3)	2358(5)	258(19)
C(2)	11007(6)	10128(3)	2738(5)	270(19)
C(3)	12344(7)	10643(4)	3020(6)	433(24)
C(4)	13520(7)	10159(5)	3338(6)	568(28)
C(5)	13052(6)	9300(4)	3285(6)	493(25)
C(6)	11562(6)	9272(3)	2929(5)	340(20)
C(7)	8435(6)	9646(3)	2131(5)	271(18)
C(8)	7971(6)	9309(3)	3275(5)	342(20)
C(9)	6971(5)	8665(3)	3082(5)	337(20)
C(10)	6414(5)	8348(3)	1715(6)	309(20)
C(11)	6842(6)	8673(3)	553(6)	376(21)
C(12)	7848(6)	9316(3)	773(5)	368(21)
C(13)	8916(7)	11218(3)	2152(6)	402(22)
C(14)	9291(10)	11686(4)	3523(7)	905(38)
C(15)	9352(9)	11662(4)	966(7)	776(34)
C(16)	7161(8)	11200(4)	1665(9)	890(39)
Br(1)	5044(1)	7461(1)	1433(1)	481(3)

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

Table 3. Interatomic Distances (Å) with Esd's

H-13283-M5

C(1)-C(2)	1.362(7)	C(1)-C(7)	1.491(7)
C(1)-C(13)	1.536(7)	C(2)-C(3)	1.463(8)
C(2)-C(6)	1.474(7)	C(3)-C(4)	1.316(9)
C(4)-C(5)	1.456(10)	C(5)-C(6)	1.338(8)
C(7)-C(8)	1.401(8)	C(7)-C(12)	1.394(7)
C(8)-C(9)	1.376(7)	C(9)-C(10)	1.388(7)
C(10)-C(11)	1.391(8)	C(10)-Br(1)	1.893(5)
C(11)-C(12)	1.379(7)	C(13)-C(14)	1.490(8)
C(13)-C(15)	1.499(9)	C(13)-C(16)	1.577(10)

Table 4. Interatomic Angles (Deg.) with Esd's

C(2)-C(1)-C(7)	117.3(4)	C(2)-C(1)-C(13)	125.4(5)
C(7)-C(1)-C(13)	117.2(4)	C(1)-C(2)-C(3)	130.9(5)
C(1)-C(2)-C(6)	124.0(5)	C(3)-C(2)-C(6)	105.1(5)
C(2)-C(3)-C(4)	108.5(6)	C(3)-C(4)-C(5)	109.9(6)
C(4)-C(5)-C(6)	108.7(5)	C(2)-C(6)-C(5)	107.9(5)
C(1)-C(7)-C(8)	121.2(4)	C(1)-C(7)-C(12)	120.7(5)
C(8)-C(7)-C(12)	118.1(5)	C(7)-C(8)-C(9)	121.7(5)
C(8)-C(9)-C(10)	118.7(5)	C(9)-C(10)-C(11)	121.1(5)
C(9)-C(10)-Br(1)	119.2(4)	C(11)-C(10)-Br(1)	119.6(4)
C(10)-C(11)-C(12)	119.1(5)	C(7)-C(12)-C(11)	121.2(5)
C(1)-C(13)-C(14)	111.5(4)	C(1)-C(13)-C(15)	112.3(5)
C(14)-C(13)-C(15)	113.0(5)	C(1)-C(13)-C(16)	110.2(5)
C(14)-C(13)-C(16)	105.0(6)	C(15)-C(13)-C(16)	104.2(5)

H-13283-M6

Table 5. Anisotropic displacement coefficients ($Å^2 x 10^4$)

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	340(34)	324(29)	150(27)	61(25)	136(25)	2(21)
C(2)	305(35)	364(31)	160(28)	-74(26)	92(23)	-14(22)
C(3)	452(42)	559(37)	265(34)	-180(34)	54(29)	5(27)
C(4)	368(42)	1006(57)	302(35)	-239(41)	40(29)	90(33)
C(5)	310(38)	760(47)	392(37)	114(34)	63(28)	56(31)
C(6)	290(34)	429(33)	298(31)	82(27)	71(25)	7(25)
C(7)	190(28)	306(28)	308(32)	63(25)	52(24)	46(24)
C(8)	311(33)	414(32)	288(32)	43(28)	53(25)	33(24)
C(9)	240(31)	444(33)	340(33)	38(27)	102(25)	87(26)
C(10)	159(29)	298(30)	472(35)	57(24)	84(25)	51(25)
C(11)	326(33)	417(35)	362(34)	13(28)	51(26)	-76(26)
C(12)	342(35)	457(33)	308(34)	-3(29)	89(26)	-22(26)
C(13)	561(41)	360(32)	318(33)	98(29)	174(29)	-6(26)
C(14)	1655(82)	483(44)	532(46)	489(48)	203(49)	27(33)
C(15)	1231(67)	571(45)	634(48)	373(45)	433(45)	321(35)
C(16)	775(61)	548(45)	1322(76)	341(44)	231(53)	139(44)
Br(1)	354(4)	405(4)	646(5)	-42(3)	61(3)	46(3)

The anisotropic displacement exponent takes the form:

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

H-13283-MT

Table 6. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^4$)

	x	y	z	U
H(3A)	12373	11234	2972	800
H(4A)	14537	10342	3581	800
H(5A)	13706	8831	3463	800
H(6A)	10956	8785	2818	800
H(8A)	8389	9524	4218	800
H(9A)	6636	8450	3867	800
H(11A)	6408	8458	-387	800
H(12A)	8166	9534	-21	800
H(14A)	10355	11715	3875	800
H(14B)	8889	12234	3368	800
H(14C)	8884	11409	4212	800
H(15A)	10419	11692	1182	800
H(15B)	8979	11364	89	800
H(15C)	8944	12210	866	800
H(16A)	6795	11756	1535	800
H(16B)	6829	10902	782	800
H(16C)	6790	10935	2389	800

H-13283-m8

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{16}H_{17}Br$
Color; Habit	Yellow prism
Crystal Size (mm)	0.10 x 0.20 x 0.36
Crystal System	Monoclinic
Space Group	$P2_1/c$
Unit Cell Dimensions	a = 9.3008(7) Å
	b = 16.2059(11) Å
	c = 9.6876(8) Å
	β = 105.155(6)°
Volume	1409.4(2) $Å^3$
Z	4
Formula weight	289.2
Density(calc.)	1.363 Mg/m^3
Absorption Coefficient	2.864 mm^{-1}
F(000)	592

H-13283-m9

Data Collection

Diffractometer System	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	168
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 45.0°
Scan Type	θ-2θ
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	$0 \le h \le 10$, $0 \le k \le 17$ $-10 \le \ell \le 10$
Reflections Collected	2079
Independent Reflections	1696 (R_{int} = 1.5%); ($\|F_o\| > 0$)
Observed Reflections	1437 ($\|F_o\| > 3.0\sigma(\|F_o\|)$)
Absorption Correction	Semi-empirical (ψ-scan method)
Min./Max. Transmission	0.3883 / 0.4917

H-13283-m10

Solution and Refinement

System Used	Siemens SHELXTL (MicroVAX & PC Versions)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(\|F_o\|-\|F_c\|)^2$
Extinction Correction	$\chi = 0.0004(2)$, where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Riding model, fixed isotropic U
Weighting Scheme	$w^{-1} = \sigma^2(\|F_o\|) + 0.0005(\|F_o\|)^2$
Final R Indices (obs. data)	$R_F = 4.8\%$, $R_{wF} = 5.0\%$
Goodness-of-Fit	1.40
Number of Variables	155
Data-to-Parameter Ratio	9.3:1
Largest and Mean Δ/σ	0.001, < 0.001
Largest Difference Peak	0.52 eÅ^{-3}
Largest Difference Hole	-0.30 eÅ^{-3}

References.

1. Churchill, M. R.; Lashewycz, R. A.; Rotella, F. J. Inorg. Chem. 1977, 16, 265-271.

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

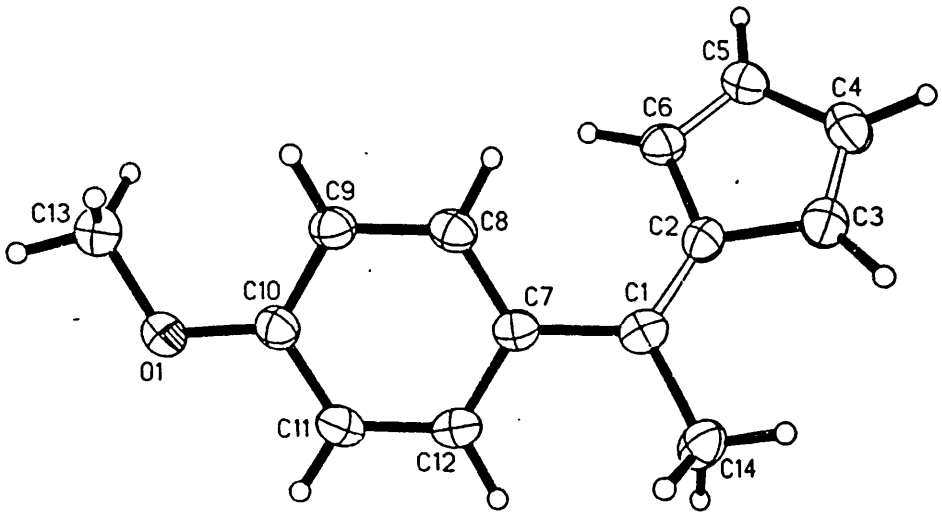
3. Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990.

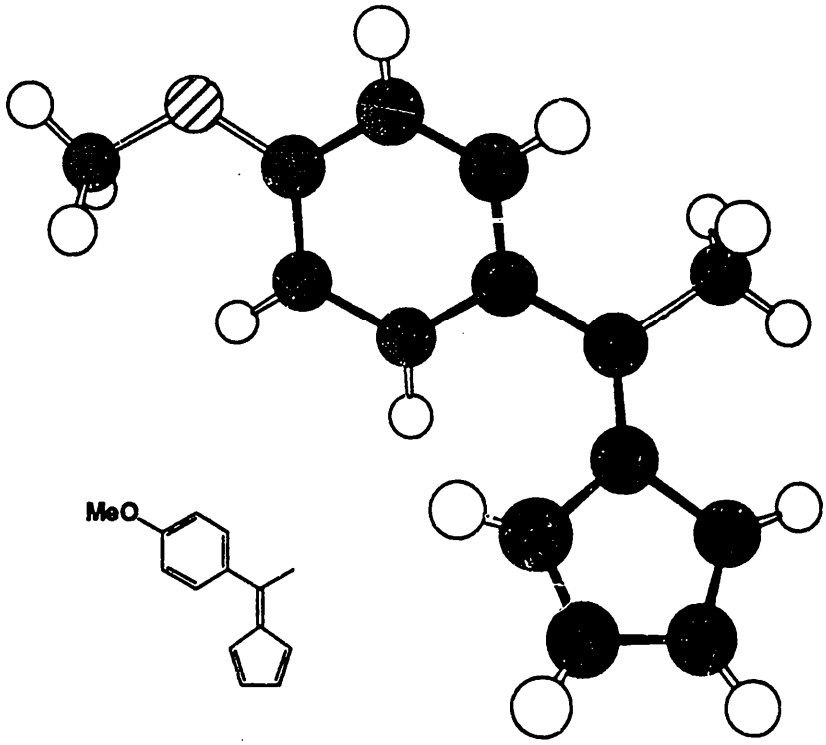
4. International Tables for X-Ray Crystallography; Kynoch Press: Birmingham, England, 1974; (a) pp 99-101; (b) pp 149-150.

* The thermal ellipsoid plot is shown at the 50% probability level.

H-13283-M12

6-(*p*-Methoxyphenyl)-6-methylfulvene (8)



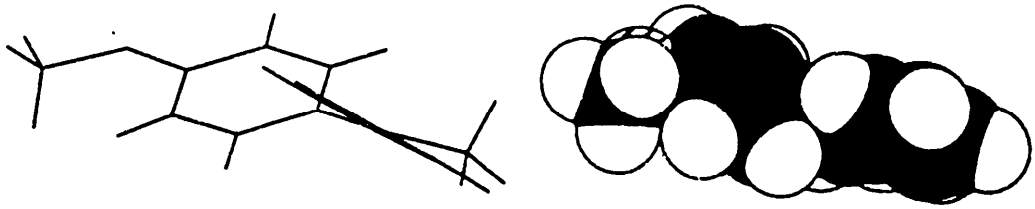


Torsional angles:

2-1-7-8	-38.7°
2-1-7-12	144.0°
7-1-2-3	172.0°
7-1-2-6	-13.3°
14-1-2-3	-9.8°
14-1-2-6	165.0°

Atom distances:

C1-C7	1.479 Å
H6-H8	2.366 Å



H-13283-M13

Collection of X-ray Diffraction Data. An orange crystal of approximate dimensions 0.23 x 0.30 x 0.52 mm was oil-mounted on a glass fiber and transferred to the Syntex $P2_1$ automated four-circle diffractometer which is equipped with a modified LT-1 low temperature system. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out by previously described methods similar to those of Churchill[1]. Intensity data were collected at 168 K using a θ-2θ scan technique with Mo Kα radiation under the conditions described in Table 1. All 2107 data were corrected for Lorentz and polarization effects and were placed on an approximately absolute scale. The diffraction symmetry was 2/m with systematic absences $0k0$ for k = 2n+1 and $h0\ell$ for ℓ = 2n+1. The centrosymmetric monoclinic space group $P2_1/c$ [C_{2h}^5; No. 14] is therefore uniquely defined.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using either our locally 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[4b] were included. The quantity minimized during least-squares analysis was $\sum w(|F_o|-|F_c|)^2$ where $w^{-1} = \sigma^2(|F_o|) + 0.0005(|F_o|)^2$.

The structure was solved by direct methods (SHELXTL PLUS) and refined by full-matrix least-squares techniques. Hydrogen atoms were located from a difference-Fourier map and included with isotropic temperature parameters. Refinement of positional and thermal parameters led to convergence with R_F = 3.6%; R_{wF} = 4.5% and GOF = 1.51 for 193 variables refined against those 1630 data with $|F_o| > 3.0\sigma(|F_o|)$. A final difference-Fourier synthesis showed no significant features, ρ(max) = 0.19eÅ^{-3}.

H-13283-M14

Table 1. Experimental Data for the X-ray Diffraction Study

Formula: $C_{14}H_{14}O$

Fw: 198.3

Temperature (K): 168

Crystal System: Monoclinic

Space Group: $P2_1/c$ [C_{2h}^5; No. 14]

a = 10.7334(9) Å

b = 11.1897(9) Å

c = 9.5808(9) Å

β = 112.265(7)°

V = 1064.89(16) Å^3

Z = 4

D_{calcd}, Mg/m^3 = 1.237

Diffractometer: Syntex $P2_1$ (Siemens R3m/V System)

Radiation: Mo Kα ($\bar{\lambda}$ = 0.710730 Å)

Monochromator: Highly oriented graphite

Data Collected: $+h,+k,\pm\ell$

Scan Type: θ-2θ

Scan Width: 1.2° plus Kα-separation

Scan Speed: 3.0 deg min^{-1} (in ω)

2θ Range, deg: 4.0 to 50.0

μ(Mo Kα), mm^{-1} = 0.071

Reflections Collected: 2107

Reflections with $|F_o| > 3.0\sigma(|F_o|)$: 1630

No. of Variables: 193

R_F = 3.6%, R_{wF} = 4.5%

Goodness of Fit: 1.51

H-13283-M15

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

	x	y	z	U(eq)
C(1)	8740(1)	12552(1)	873(1)	269(5)
C(2)	8239(1)	13645(1)	1015(1)	269(5)
C(3)	8905(1)	14805(1)	1140(2)	322(5)
C(4)	8041(1)	15669(1)	1157(2)	345(5)
C(5)	6772(1)	15123(1)	1024(2)	325(5)
C(6)	6883(1)	13926(1)	956(1)	282(5)
C(7)	8045(1)	11428(1)	952(1)	257(4)
C(8)	7408(1)	11294(1)	1972(1)	271(5)
C(9)	6766(1)	10245(1)	2083(2)	284(5)
C(10)	6746(1)	9285(1)	1146(2)	286(5)
C(11)	7396(1)	9391(1)	137(2)	318(5)
C(12)	8046(1)	10434(1)	53(2)	295(5)
C(13)	5414(2)	8117(1)	2133(2)	367(6)
C(14)	10016(2)	12438(2)	582(2)	352(6)
O(1)	6147(1)	8211(1)	1164(1)	369(4)

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

H-13283-M16 (1)

Table 3. Interatomic Distances (Å) with Esd's

C(1)-C(2)	1.363(2)	C(1)-C(7)	1.479(2)
C(1)-C(14)	1.503(2)	C(2)-C(3)	1.464(2)
C(2)-C(6)	1.470(2)	C(3)-C(4)	1.345(2)
C(4)-C(5)	1.454(2)	C(5)-C(6)	1.348(2)
C(7)-C(8)	1.398(2)	C(7)-C(12)	1.407(2)
C(8)-C(9)	1.385(2)	C(9)-C(10)	1.395(2)
C(10)-C(11)	1.395(2)	C(10)-O(1)	1.366(2)
C(11)-C(12)	1.378(2)	C(13)-O(1)	1.429(2)

Table 4. Interatomic Angles (Deg.) with Esd's

C(2)-C(1)-C(7)	122.2(1)	C(2)-C(1)-C(14)	121.1(1)
C(7)-C(1)-C(14)	116.7(1)	C(1)-C(2)-C(3)	127.2(1)
C(1)-C(2)-C(6)	127.6(1)	C(3)-C(2)-C(6)	105.0(1)
C(2)-C(3)-C(4)	108.7(1)	C(3)-C(4)-C(5)	108.9(1)
C(4)-C(5)-C(6)	108.9(1)	C(2)-C(6)-C(5)	108.4(1)
C(1)-C(7)-C(8)	121.1(1)	C(1)-C(7)-C(12)	121.6(1)
C(8)-C(7)-C(12)	117.2(1)	C(7)-C(8)-C(9)	122.3(1)
C(8)-C(9)-C(10)	119.4(1)	C(9)-C(10)-C(11)	119.3(1)
C(9)-C(10)-O(1)	124.2(1)	C(11)-C(10)-O(1)	116.4(1)
C(10)-C(11)-C(12)	120.7(1)	C(7)-C(12)-C(11)	121.1(1)
C(10)-O(1)-C(13)	116.7(1)		

Table 5. Anisotropic displacement coefficients ($Å^2 x 10^4$) H-13283-M17

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	248(7)	343(8)	201(6)	26(6)	67(5)	31(5)
C(2)	280(7)	318(7)	206(6)	-13(5)	89(5)	9(5)
C(3)	330(8)	346(8)	281(7)	-46(6)	106(6)	-4(6)
C(4)	430(8)	278(8)	303(7)	-46(6)	110(6)	-31(6)
C(5)	373(8)	324(8)	280(7)	46(6)	126(6)	-15(6)
C(6)	289(7)	315(8)	249(7)	2(6)	111(5)	-10(5)
C(7)	230(6)	291(7)	237(6)	50(5)	75(5)	23(5)
C(8)	306(7)	269(7)	228(7)	35(6)	91(6)	-6(5)
C(9)	323(7)	293(7)	259(7)	40(6)	137(6)	15(5)
C(10)	283(7)	258(7)	317(7)	33(6)	111(6)	13(5)
C(11)	349(8)	285(8)	341(8)	36(6)	155(6)	-53(6)
C(12)	285(7)	348(8)	282(7)	55(6)	142(6)	6(6)
C(13)	405(8)	316(9)	430(9)	-39(7)	215(7)	-12(7)
C(14)	293(8)	388(9)	407(8)	46(7)	168(7)	69(7)
O(1)	437(6)	263(5)	481(6)	-26(4)	258(5)	-54(4)

The anisotropic displacement exponent takes the form:

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

Table 6. H-Atom coordinates ($x10^4$) and isotropic displacement coefficients ($Å^2 x10^4$)

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	x	y	z	U
H(3)	9851(15)	14895(13)	1236(16)	365(39)
H(4)	8227(16)	16536(15)	1272(19)	477(45)
H(5)	5953(15)	15554(15)	997(17)	453(44)
H(6)	6174(14)	13344(12)	826(15)	288(36)
H(8)	7438(14)	11943(13)	2671(16)	333(38)
H(9)	6373(13)	10197(12)	2850(15)	277(34)
H(11)	7361(14)	8713(14)	-510(18)	400(40)
H(12)	8514(13)	10488(12)	-661(16)	288(34)
H(13A)	5992(15)	8229(13)	3185(19)	387(41)
H(13B)	4625(16)	8714(14)	1814(17)	416(41)
H(13C)	5044(17)	7294(17)	1982(18)	518(47)
H(14A)	10565(17)	11761(16)	1092(21)	548(49)
H(14B)	10563(17)	13187(17)	831(20)	584(51)
H(14C)	9802(17)	12288(15)	-516(21)	533(48)

Table 7. Hydrogen Atom Distances and Angles.

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C(3)-H(3)	0.989(16)	C(4)-H(4)	0.987(17)
C(5)-H(5)	0.994(17)	C(6)-H(6)	0.973(15)
C(8)-H(8)	0.980(15)	C(9)-H(9)	0.977(17)
C(11)-H(11)	0.972(17)	C(12)-H(12)	0.991(17)
C(13)-H(13A)	0.971(15)	C(13)-H(13B)	1.031(16)
C(13)-H(13C)	0.991(18)	C(14)-H(14A)	0.970(17)
C(14)-H(14B)	0.999(18)	C(14)-H(14C)	1.002(20)

C(2)-C(3)-H(3)	123.3(9)	C(4)-C(3)-H(3)	127.9(9)
C(3)-C(4)-H(4)	126.6(10)	C(5)-C(4)-H(4)	124.4(10)
C(4)-C(5)-H(5)	126.0(10)	C(6)-C(5)-H(5)	125.1(10)
C(2)-C(6)-H(6)	125.3(9)	C(5)-C(6)-H(6)	126.3(9)
C(7)-C(8)-H(8)	120.0(10)	C(9)-C(8)-H(8)	117.7(10)
C(8)-C(9)-H(9)	118.2(8)	C(10)-C(9)-H(9)	122.4(8)
C(10)-C(11)-H(11)	117.9(11)	C(12)-C(11)-H(11)	121.4(11)
C(7)-C(12)-H(12)	119.4(8)	C(11)-C(12)-H(12)	119.5(8)
O(1)-C(13)-H(13A)	111.9(12)	O(1)-C(13)-H(13B)	110.7(10)
H(13A)-C(13)-H(13B)	110.7(13)	O(1)-C(13)-H(13C)	105.2(12)
H(13A)-C(13)-H(13C)	109.5(13)	H(13B)-C(13)-H(13C)	108.7(13)
C(1)-C(14)-H(14A)	113.3(13)	C(1)-C(14)-H(14B)	112.3(12)
H(14A)-C(14)-H(14B)	110.2(14)	C(1)-C(14)-H(14C)	110.2(11)
H(14A)-C(14)-H(14C)	104.6(16)	H(14B)-C(14)-H(14C)	105.7(16)

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STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	$C_{14}H_{14}O$
Color; Habit	Orange prism
Crystal Size (mm)	0.23 x 0.30 x 0.52
Crystal System	Monoclinic
Space Group	$P2_1/c$
Unit Cell Dimensions	$\underline{a}$ = 10.7334(9) Å
	$\underline{b}$ = 11.1897(9) Å
	$\underline{c}$ = 9.5808(9) Å
	β = 112.265(7)°
Volume	1064.89(16) $Å^3$
Z	4
Formula weight	198.3
Density(calc.)	1.237 Mg/m^3
Absorption Coefficient	0.071 mm^{-1}
F(000)	424

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Data Collection

Diffractometer System	Siemens R3m/V
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	168
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 50.0°
Scan Type	θ–2θ
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	$-6 \leq h \leq 12$, $-1 \leq k \leq 13$ $-11 \leq \ell \leq 11$
Reflections Collected	2107
Independent Reflections	1791 (R_{int} = 1.7%); ($\|F_o\| > 0$)
Observed Reflections	1630 ($\|F_o\| > 3.0\sigma(\|F_o\|)$)

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Solution and Refinement

System Used	Siemens SHELXTL (MicroVAX & PC Versions)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(\|F_o\|-\|F_c\|)^2$
Extinction Correction	$\chi = 0.0117(10)$, where $F^* = F\,[\,1 + 0.002\chi F^2/\sin(2\theta)\,]^{-1/4}$
Hydrogen Atoms	Refined (x,y,z) and U(iso)
Weighting Scheme	$w^{-1} = \sigma^2(\|F_o\|) + 0.0005(\|F_o\|)^2$
Final R Indices (obs. data)	$R_F = 3.6\%$, $R_{wF} = 4.5\%$
Goodness-of-Fit	1.51
Number of Variables	193
Data-to-Parameter Ratio	8.4:1
Largest and Mean Δ/σ	0.001, < 0.001
Largest Difference Peak	0.19 eÅ^{-3}
Largest Difference Hole	-0.16 eÅ^{-3}

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

* The thermal ellipsoid plot is shown at the 50% probability level.