Collection of X-ray Diffraction Data. A colorless crystal of approximate dimensions 0.33 x 0.33 x 0.37 mm was oil-mounted[1] on a glass fiber and transferred to the Siemens P4 rotating-anode diffractometer. The determination of Laue symmetry, crystal class, unit cell parameters and the crystal's orientation matrix were carried out according to standard procedures[2]. Intensity data were collected at 158 K using a θ-2θ scan technique with MoKα radiation under the conditions described in Table 1. All 1137 data were corrected for Lorentz and polarization effects and were placed on an approximately absolute scale. The diffraction symmetry was *mmm* with systematic absences $hk0$ for h = 2n+1, $0k\ell$ for k = 2n+1 and $h0\ell$ for ℓ = 2n+1. The centrosymmetric orthorhombic space group Pbca is therefore uniquely defined.

Solution and Refinement of the Crystal Structure. All crystallographic calculations were carried out using the UCI-modified version of the UCLA Crystallographic Computing Package[3] and the SHELXTL PLUS program set[4]. The analytical scattering factors for neutral atoms were used throughout the analysis[5]; both the real ($\Delta f'$) and imaginary ($i\Delta f''$) components of anomalous dispersion were included. The quantity minimized during least-squares analysis was $\sum w(F_o-F_c)^2$ where $w^{-1} = \sigma^2(F) + 0.0003F^2$.

The structure was solved by direct methods and refined by full-matrix least-squares techniques. Hydrogen atoms were located from a difference-Fourier synthesis and included with isotropic thermal parameters. At convergence, R_F = 3.1%; R_{wF} = 3.6% and GOF = 1.47 for 128 variables refined against those 848 data with $F > 2.0\sigma(F)$. A final difference-Fourier synthesis yielded ρ(max) = 0.13eÅ^{-3}.

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

Formula: C_8H_9FO

Fw: 140.2

Temperature (K): 158

Crystal System: Orthorhombic

Space Group: Pbca

a = 6.6799(9) Å

b = 12.256(3) Å

c = 17.611(4) Å

V = 1441.8(5) $Å^3$

Z = 8

D_{calcd}, Mg/m^3 = 1.291

Diffractometer: Siemens P4 Rotating-anode

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

Monochromator: Highly oriented graphite

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

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} = 0.101

Reflections Collected: 1137

Independent Reflections: 888

Reflections with $F > 2.0\sigma(F)$: 848

No. of Variables: 128

R_F = 3.1%, R_{wF} = 3.6%

Goodness of Fit: 1.28

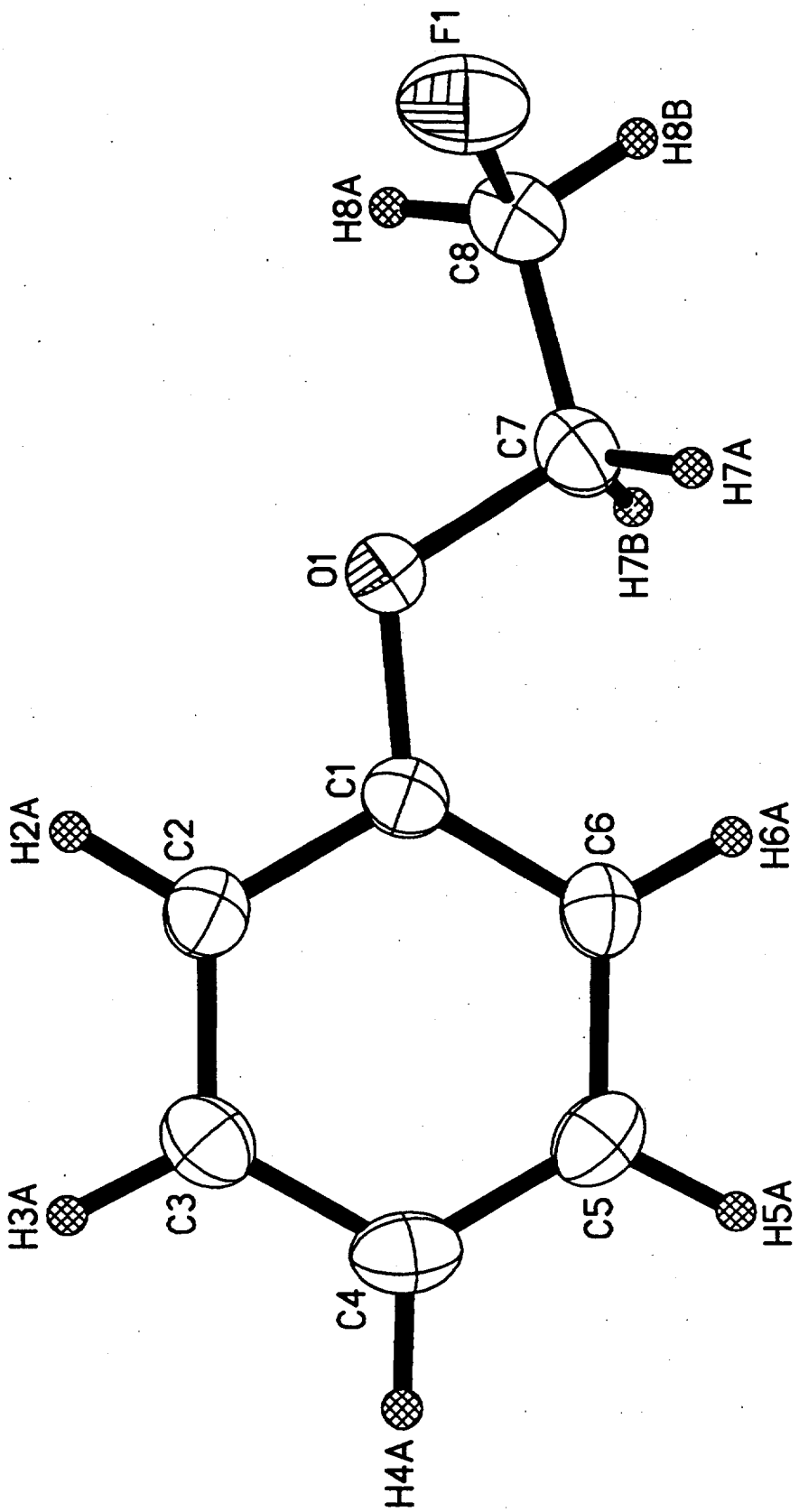
F1
H8B
H8A
C8
C7
H7A
H7B
O1
C1
H2A
C2
C6
H6A
H3A
C3
C5
H5A
C4
H4A

Table 2. Atomic coordinates ($x10^5$) and equivalent isotropic displacement coefficients ($Å^2 x10^4$)

	x	y	z	U(eq)
C(1)	22846(22)	87757(12)	54169(9)	255(5)
C(2)	36731(25)	92434(13)	59066(9)	292(6)
C(3)	38551(26)	88602(14)	66390(9)	326(6)
C(4)	26723(26)	80074(14)	68907(9)	349(6)
C(5)	13117(25)	75384(14)	64023(9)	334(6)
C(6)	11056(24)	79169(12)	56623(9)	295(6)
C(7)	7029(27)	88283(15)	41962(9)	313(6)
C(8)	8127(27)	94725(15)	34813(9)	324(6)
O(1)	22017(16)	92251(8)	47028(6)	314(4)
F(1)	25496(15)	92016(8)	30793(5)	434(4)

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

Table 3. Interatomic Distances (Å)

C(1)-C(2)	1.390(2)	C(1)-C(6)	1.384(2)
C(1)-O(1)	1.374(2)	C(2)-C(3)	1.378(2)
C(3)-C(4)	1.383(2)	C(4)-C(5)	1.377(2)
C(5)-C(6)	1.390(2)	C(7)-C(8)	1.488(2)
C(7)-O(1)	1.427(2)	C(8)-F(1)	1.399(2)

Table 4. Interatomic Angles (°)

C(2)-C(1)-C(6)	120.0(1)	C(2)-C(1)-O(1)	115.4(1)
C(6)-C(1)-O(1)	124.6(1)	C(1)-C(2)-C(3)	119.9(1)
C(2)-C(3)-C(4)	120.5(2)	C(3)-C(4)-C(5)	119.5(2)
C(4)-C(5)-C(6)	120.8(2)	C(1)-C(6)-C(5)	119.4(1)
C(8)-C(7)-O(1)	108.3(1)	C(7)-C(8)-F(1)	110.1(1)
C(1)-O(1)-C(7)	117.7(1)		

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

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	265(9)	272(9)	228(9)	21(7)	25(7)	-26(7)
C(2)	299(10)	278(10)	298(10)	-21(8)	31(8)	-16(8)
C(3)	329(10)	358(10)	291(10)	11(9)	-13(8)	-55(8)
C(4)	427(11)	366(10)	254(9)	84(9)	46(9)	32(8)
C(5)	349(10)	290(10)	364(10)	-9(9)	92(8)	30(8)
C(6)	266(10)	297(10)	323(10)	-36(8)	19(8)	-37(8)
C(7)	298(10)	343(11)	299(10)	-20(8)	-40(9)	-27(8)
C(8)	297(10)	374(11)	301(10)	52(9)	-3(9)	-31(8)
O(1)	342(7)	355(7)	246(6)	-110(5)	-32(5)	24(5)
F(1)	470(7)	498(7)	333(6)	122(5)	81(5)	-6(5)

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 ($Å^2 x 10^3$)

	x	y	z	U
H(2A)	4472(24)	9828(12)	5729(8)	28(4)
H(3A)	4827(28)	9195(12)	6959(9)	41(5)
H(4A)	2822(24)	7762(11)	7405(10)	36(4)
H(5A)	474(24)	6936(13)	6558(8)	34(4)
H(6A)	167(24)	7594(12)	5339(8)	32(4)
H(7A)	943(24)	8066(14)	4086(9)	37(5)
H(7B)	-621(27)	8916(12)	4435(9)	36(5)
H(8A)	903(25)	10262(14)	3582(9)	39(5)
H(8B)	-324(30)	9296(13)	3152(9)	46(5)

STRUCTURE DETERMINATION SUMMARY

Crystal Data

Empirical Formula	C_8H_9FO
Color; Habit	Colorless cube
Crystal size (mm)	0.33 x 0.33 x 0.37
Crystal System	Orthorhombic
Space Group	Pbca
Unit Cell Dimensions	a = 6.6799(9) Å
	b = 12.256(3) Å
	c = 17.611(4) Å
Volume	1441.8(5) $Å^3$
Z	8
Formula weight	140.2
Density(calc.)	1.291 Mg/m^3
Absorption Coefficient	0.101 mm^{-1}
F(000)	592

Data Collection

Diffractometer Used	Siemens P4RA
Radiation	MoKα (λ = 0.71073 Å)
Temperature (K)	158
Monochromator	Highly oriented graphite crystal
2θ Range	4.0 to 45.0°
Scan Type	θ-2θ
Scan Speed	Constant; 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
Reflections Collected	1137
Independent Reflections	888
Observed Reflections	848 ($F > 2.0\sigma(F)$)

Solution and Refinement

System Used	Siemens SHELXTL PLUS (PC Version)
Solution	Direct Methods
Refinement Method	Full-Matrix Least-Squares
Quantity Minimized	$\sum w(F_o - F_c)^2$
Extinction Correction	$\chi = 0.0033(4)$, 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	$w^{-1} = \sigma^2(F) + 0.0003F^2$
Number of Parameters Refined	128
Final R Indices (obs. data)	$R_F = 3.1\%$, $R_{wF} = 3.6\%$
R Indices (all data)	$R_F = 3.3\%$, $R_{wF} = 3.7\%$
Goodness-of-Fit	1.47
Largest and Mean Δ/σ	< 0.001, < 0.001
Data-to-Parameter Ratio	6.6:1
Largest Difference Peak	0.13 $e\text{Å}^{-3}$
Largest Difference Hole	-0.12 $e\text{Å}^{-3}$

References.

1. The crystal was immersed in a lube-oil additive which allows for manipulation on the bench-top and prevents decomposition due to air or moisture. The crystal was secured to a glass fiber (the oil acts as the adhesive) which is attached to an elongated brass mounting-pin. Further details appear in Hope, H.; Experimental Organometallic Chemistry: A Practicum in Synthesis and Characterization, ACS Symposium Series No. 357, Wayda, A. L. and Darensbourg, M. Y., Eds., 1987.

2. XSCANS Software Users Guide, Version 2.1, Siemens Industrial Automation, Inc.; Madison, WI 1994.

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

4. Sheldrick, G.; SHELXTL PLUS program set; Siemens Analytical X-Ray Instruments, Inc.; Madison, WI 1990.

5. International Tables for X-Ray Crystallography 1992, Vol. C., Dordrecht: Kluwer Academic Publishers.

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