



J. Am. Chem. Soc., 1996, 118(34), 8006-8014, DOI:[10.1021/ja954005l](https://doi.org/10.1021/ja954005l)

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at <http://pubs.acs.org/page/copyright/permissions.html>



ACS Publications

MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1996 American Chemical Society

Table 3.1:Crystallographic data and refinement parameters of fluorbullvalene (**3**)

Formula: $C_{10}H_8F$
Molecular mass: 147.17
Crystal description: colorless prisms
Crystal size: $0.15 \times 0.20 \times 0.35$ mm
Crystal system: orthorhombic
Space group: <i>Pnam</i> (# 33)
$a = 13.411(3)$, $b = 6.101(1)$, $c = 9.191(3)$ Å
$V = 752.1(5)$ Å ³ , $Z = 4$, $D_{calc} = 1.300$ g·cm ⁻³
$F_{000} = 308$ e, $\mu(\text{Mo-}K\alpha) = 0.860$ cm ⁻¹
Symmetry of molecule in crystal: C_s
Radiation: Mo- $K\alpha$ ($\lambda = 0.7107$ Å), $\omega-2\theta$ scanning technique
Unique reflections measured: 783 up to $(sin\theta/\lambda)_{max} = 0.62$ Å ⁻¹
Structure solution: direct methods (SIR)
Hydrogen atoms: located and refined isotropically
Refinement: full matrix least-squares, function minimized: $\sum w(\Delta F)^2$
Anomalous dispersion: all non-hydrogen atoms ^[1]
Observed reflections included: 538 with $I \geq 3.0 \sigma(I)$
Parameters refined: 79
Agreement factor: $R = 0.043$, (weighted: $R_w = 0.051$) ^[2]
Convergence, largest shift: $(\Delta/\sigma)_{max} = 0.01$
High peak in final diff. map: $(\Delta\rho)_{max} = 0.13(4)$ e·Å ⁻³

^[1] Scattering factors and anomalous dispersion corrections were taken from:
International Tables for X-ray Crystallography, Kynoch Press, Birmingham, England,
1974, vol. IV.

^[2] $R = \frac{\sum |F_{obs}| - |F_{calc}|}{\sum |F_{obs}|}$, $R_w = \sqrt{\frac{\sum w |F_{obs}| - |F_{calc}||^2}{\sum w |F_{obs}|^2}}$, $w = [\sigma(F)^2 + (0.03F_{obs})^2]^{-1}$

Tab. 3.2: Atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms of 3

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq} \cdot \frac{10^4}{\text{\AA}^2}$
C(1A)	0.4833(1)	0.1968(3)	0.1666(2)	597(5)
C(2A)	0.3912(1)	0.2256(3)	0.0831(2)	570(4)
C(3A)	0.3029(1)	0.1496(3)	0.1165(2)	550(4)
C(1C)	0.5057(2)	-0.0123(5)	0.250	626(7)
C(2C)	0.4392(2)	-0.1987(4)	0.250	637(7)
C(3C)	0.3409(2)	-0.1925(4)	0.250	575(7)
C(4)	0.2831(2)	0.0168(5)	0.250	511(6)
F	0.1805(1)	-0.0390(3)	0.250	816(5)

$$U_{eq} = \frac{1}{3} \sum \sum U_{ij} \mathbf{a}_i \cdot \mathbf{a}_j \mathbf{a}_i^* \mathbf{a}_j^*$$

Tab. 3.3: Atomic coordinates and isotropic displacement parameters for hydrogen-atoms of 3

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{iso} \cdot \frac{10^3}{\text{\AA}^2}$
H(1A)	0.542(1)	0.255(3)	0.124(2)	63(4)
H(2A)	0.403(1)	0.292(4)	-0.010(2)	87(6)
H(3A)	0.249(1)	0.173(3)	0.053(2)	68(5)
H(1C)	0.574(2)	-0.066(6)	0.250	97(8)
H(2C)	0.467(2)	-0.347(5)	0.250	83(7)
H(3C)	0.304(2)	-0.302(5)	0.250	64(6)

$$T = \exp[-8\pi^2 U_{iso} (\sin \theta / \lambda)^2]$$

Tab. 3.7: C–H-bond distances (in Å) of 3

C(1A) – H(1A)	0.95(2)	C(1C) – H(1C)	0.98(3)
C(2A) – H(2A)	0.96(2)	C(2C) – H(2C)	0.98(3)
C(3A) – H(3A)	0.94(2)	C(3C) – H(3C)	0.83(3)

In parentheses e.s.d.'s in units of the least significant digits.