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

Formula: $C_{10}H_8F$
Molecular mass: 147.17
Crystal description: coulorless prisms
Crystal size: $0.15 \times 0.20 \times 0.35$ mm
Crystal system: orthorhombic
Space group: *Pnam* (# 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, $\mu_{(Mo-K_\alpha)} = 0.860$ cm^{-1}
Symmetry of molecule in crystal: C_s
Radiation: Mo$-K_\alpha(\lambda = 0.7107$ Å), ω–2θ 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] $R = \frac{\sum\left||F_{obs}|-|F_{calc}|\right|}{\sum|F_{obs}|}$, $R_w = \sqrt{\frac{\sum w\left||F_{obs}|-|F_{calc}|\right|^2}{\sum w|F_{obs}|^2}}$, $w = \left[\,\sigma(F)^2 + (0.03F_{obs})^2\,\right]^{-1}$

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

Atom	x	y	z	$U_{eq} \cdot \frac{10^4}{\text{Å}^2}$
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)

$U_{eq} = \frac{1}{3} \sum \sum U_{ij}\, \boldsymbol{a}_i \cdot \boldsymbol{a}_j\, a_i^*\, a_j^*$

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

Atom	x	y	z	$U_{iso} \cdot \frac{10^3}{\text{Å}^2}$
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)

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

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.