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The crystal structure of 1-fluoro-4-(*p*-tolylethynyl) benzene, C₁₅H₁₁F

<https://doi.org/10.1515/ncrs-2021-0190>

Received May 17, 2021; accepted June 6, 2021;

published online July 6, 2021

Abstract

C₁₅H₁₁F, monoclinic, $P2_1/n$ (no. 14), $a = 6.2490(3)$ Å, $b = 7.6343(4)$ Å, $c = 24.2376(9)$ Å, $\beta = 90.914(4)^\circ$, $V = 1156.15(9)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0619$, $wR_{\text{ref}}(F^2) = 0.1829$, $T = 293(2)$ K.

CCDC no.: 2088247

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents were obtained from commercial resources. A reference method was used to synthesise FTEB [5]. Under a dry nitrogen atmosphere, 0.218 g 1-iodo-4-methylbenzene (1 mmol), 0.144 g 4-fluorophenylacetylene (1.2 mmol), CuI (0.002 g, 0.01 mmol), and 0.007 g Pd(PPh₃)₂Cl₂ (0.01 mmol) was added successively to a 100 mL Schlenk flask; then 50 mL triethylamine was added. The mixture was stirred for 3 h, then heated at 333 K for 1 h, cooled, and filtered. The filtrate was dried by rotary evaporation, then extracted by hexane. The colorless crystals were obtained from the hexane solution after 6 h, yield 83% (based on 1-iodo-4-methylbenzene).

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.22 × 0.13 × 0.11 mm
Wavelength:	Cu Kα radiation (1.54184 Å)
μ :	0.64 mm ⁻¹
Diffractometer, scan mode:	SuperNova, ω
θ_{max} , completeness:	66.6°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	3926, 2033, 0.022
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1503
$N(\text{param})_{\text{refined}}$:	146
Programs:	SuperNova [1], Olex2 [2], SHELX [3, 4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	0.9363 (2)	0.3391 (2)	0.10552 (5)	0.1014 (6)
C1	0.5378 (3)	0.3125 (2)	0.23765 (6)	0.0485 (4)
C2	0.1650 (3)	0.3139 (2)	0.37366 (6)	0.0527 (4)
C3	0.4699 (3)	0.3929 (2)	0.18896 (6)	0.0543 (5)
H3	0.333264	0.440751	0.186347	0.065*
C4	−0.0993 (3)	0.3027 (2)	0.46619 (7)	0.0551 (5)
C5	0.1011 (3)	0.2244 (3)	0.46727 (7)	0.0582 (5)
H5	0.148438	0.167894	0.499195	0.070*
C6	0.7412 (3)	0.2389 (2)	0.24075 (7)	0.0571 (5)
H6	0.786833	0.184038	0.273107	0.068*
C7	0.2321 (3)	0.2287 (2)	0.42163 (7)	0.0571 (5)
H7	0.365341	0.174438	0.423096	0.069*
C8	0.6050 (3)	0.4019 (2)	0.14436 (6)	0.0597 (5)
H8	0.561360	0.456061	0.111712	0.072*
C9	−0.1638 (3)	0.3892 (3)	0.41839 (7)	0.0603 (5)
H9	−0.296648	0.444063	0.417003	0.072*
C10	0.2961 (3)	0.3139 (2)	0.32566 (7)	0.0579 (5)
C11	0.4032 (3)	0.3103 (2)	0.28516 (7)	0.0559 (5)
C12	−0.0336 (3)	0.3952 (2)	0.37267 (7)	0.0598 (5)
H12	−0.079666	0.454165	0.341082	0.072*
C13	0.8038 (3)	0.3292 (3)	0.14946 (7)	0.0621 (5)
C14	0.8762 (3)	0.2466 (3)	0.19614 (8)	0.0640 (5)
H14	1.012098	0.197070	0.197866	0.077*
C15	−0.2423 (4)	0.2938 (3)	0.51568 (8)	0.0724 (6)
H15A	−0.288834	0.175268	0.520993	0.109*
H15B	−0.364687	0.367853	0.509661	0.109*
H15C	−0.164470	0.332759	0.547864	0.109*

Experimental details

The structure was solved by direct methods with the SHELXS-2018 program. All H atoms were positioned with idealized geometry ($U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$) using a riding model with C–H = 0.93 Å for benzene and C–H = 0.96 Å for the methyl group.

Comment

Stilbene compounds have a wide range of biological activities. Tolans are known as the most important starting materials to synthesize stilbenes and many F-containing tolans have been reported with their crystal structures [6–18]. Here we report the crystal structure of the title compound, an asymmetric tolan, namely, 1-fluoro-4-(p-tolylolethynyl)benzene (FTEB). The whole molecule is planar except H atoms from the methyl group. There are two kinds of weak intermolecular forces: C–H···F hydrogen bond and C–H··· π , by which a three-dimensional supramolecular structure is created. All the bond lengths of FTEB are similar to the reported results [6–18].

Author contributions: All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.

Research funding: The research was supported by Innovation, Entrepreneurship and Development Capability Enhancement Engineering Scientific Research Project, Luohe Medical College, 2019 (2020–LYZKYZD003).

Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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