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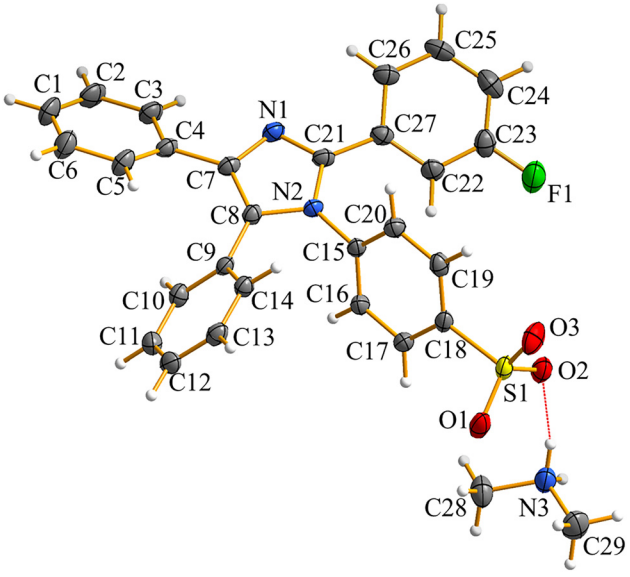
The crystal structure of dimethylammonium 4-[2-(4-fluorophenyl)-4, 5-diphenyl-1*H*-imidazol-1-yl] benzenesulfonate, $C_{29}H_{26}FN_3O_3S$



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Abstract

$C_{29}H_{26}FN_3O_3S$, monoclinic, $P2_1/c$ (no. 14), a = 15.6550(2) Å, b = 7.9540(1) Å, c = 21.5331(3) Å, β = 108.033(2)°, V = 2549.59(6) Å^3, Z = 4, $R_{gt}(F)$ = 0.0334, $wR_{ref}(F^2)$ = 0.0875, T = 150 K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data and the list of the atoms including atomic coordinates and displacement parameters can be found in the cif-file attached to this article.

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Table 1: Data collection and handling.

Crystal:	Block
Size:	0.15 × 0.13 × 0.12 mm
Wavelength:	Cu*K*α radiation (1.54184 Å)
μ:	1.49 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω scan
θ_{max}, completeness:	66.6°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	31034, 4508, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4147
$N(param)_{refined}$:	346
Programs:	Rigaku,[1,2] SHELX,[3] Olex2[4,5]

1 Source of materials

Synthesis of 4-[2-(4-fluorophenyl)-4,5-diphenyl-1H-imidazol-1-yl] benzenesulfonic (TPI–SO3H–2H). A mixture of sulfanilic acid (4.27 mmol, 0.741 g) and 3-fluorobenzaldehyde (3.57 mmol, 0.375 mL) in acetic acid (15 mL) was stirred at 120 °C for 2 h under the protection of nitrogen gas. After cooling to room temperature, benzil (3.57 mmol, 0.750 g) and ammonium acetate (35.7 mmol, 2.75 g) were added, and the mixture was further stirred under heating for 10 h. The crude product was further purified by column chromatography using dichloromethane-methanol (10:1, v/v) to afford the final product as a white solid with a yield of 80 %.[6] Synthesis of dimethylammonium 4-[2-(4-fluorophenyl)-4, 5-diphenyl-1*H*-imidazol-1-yl]benzenesulfonate: A mixture of TPI–SO3H–2H (0.04 g, 0.08 mmol), *N*,*N*′-dimethylformamide (DMF, 1 mL) and H_2O (3 mL) was added to a 15 mL Teflon-lined reactor. The reaction was heated at 120 °C for 34 h. The resulting crystals were washed several times with water and dried under vacuum at room temperature for 4 h, yielding the product with a 70 % yield.

2 Experimental details

Diffraction data were collected with a Rigaku XtaLAB single crystal diffractometer using Cu–Kα radiation (λ = 1.54184 Å).[1,2] The structure was solved by Direct

Methods and refined by the full-matrix least squares technique on F^2 using the SHELXTL package.[3] Hydrogen atoms were generated geometrically.[4,5]

3 Comment

1,2,4,5-Tetraphenylimidazole (TPI), a prototypical tetraarylimidazole derivative, adopts a highly twisted propeller-like conformation. There are four phenyl rings connected to a central imidazole core via substantial torsion angles, resulting in a non-planar geometry. These properties can effectively mitigate π-π stacking interactions in the aggregated state. The distortion is pivotal to the aggregation-induced emission (AIE) behavior, where in fluorescence intensity increases dramatically upon molecular aggregation. Recent research reports suggested that modifications of aryl rotors can significantly influence the AIE properties of tetraarylimidazole derivatives.[7–10] The title structure crystallizes in the monoclinic system with the $P2_1/c$ space group, containing a TPI–SO3H–2H anion and one $[Me_2NH_2]^+$ cation in an asymmetric unit. Specifically, $[Me_2NH_2]^+$ and the SO_3 group ligand form strong N–H···O (the distance range from 1.953 to 2.003 Å, and the angle range from 148.7° to 165.7°) hydrogen bonds, constructing a nonporous two-dimensional planar structure. These values were close to those reported for related compounds. All C–C and C–N bond lengths and angles are listed in the expected range.[10]

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