

Hongjuan Tong*, Zhoujing Zhu, Juanjuan Wen, Yanjiao Wang and Bin Liu

Crystal structure of *N*-(2-(1-phenylvinyl)phenyl)-*N*-tosylacrylamide, C₂₄H₂₁NO₃S

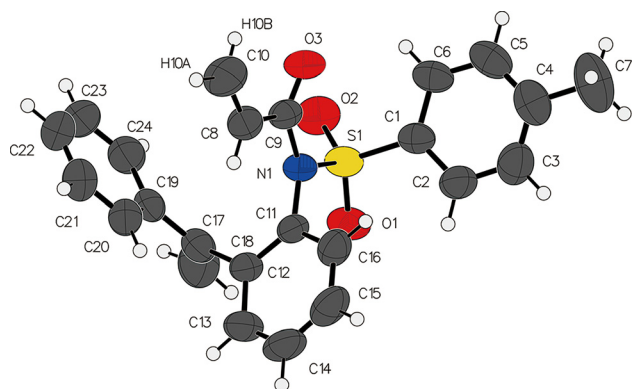


Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.33 × 0.25 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.18 mm ⁻¹
Diffractometer, scan mode:	Rigaku Synergy, ω scans
$\theta_{\max}$, completeness:	28.3°, 97 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	83045, 5160, 0.099
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 4,036
$N(\text{param})_{\text{refined}}$:	279
Programs:	Rigaku, ¹ SHELX, ^{2,3} Olex2 ⁴

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Abstract

C₂₄H₂₁NO₃S, monoclinic, $P2_1/c$ (no. 14), $a = 13.5232(6)$ Å, $b = 8.5187(5)$ Å, $c = 19.3029(10)$ Å, $\beta = 107.174(2)^\circ$, $V = 2124.55(19)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0480$ w $R_{\text{ref}}(F^2) = 0.1348$, $T = 297$ 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.

1 Source of materials

The synthetic route to the title compound, *N*-(2-(1-phenylethenyl)phenyl)-*N*-(tosyl)acrylamide, is outlined herein. Commercial (2-aminophenyl)(phenyl) methanone was converted to 2-(1-phenylethenyl)aniline via a catalytic dehydration reaction. This amine intermediate was then

subjected to *N*-tosylation using *p*-toluenesulfonyl chloride and triethylamine in dichloromethane to yield *N*-(2-(1-phenylethenyl)phenyl)-4-methylbenzenesulfonamide. The final acrylation was achieved by treating the sulfonamide with acryloyl chloride in the presence of sodium hydride in tetrahydrofuran. The crude product was purified by column chromatography. Single crystals of the title compound, which were obtained by slow evaporation from a dichloromethane/*n*-hexane solution, were subjected to X-ray crystallography for definitive structural confirmation.⁵

2 Experimental details

The structure was solved (SHELXT²) and refined (SHELXL³ within Olex2⁴), respectively, with anisotropic displacement parameters for all non-hydrogen atoms.

3 Comment

The construction of nitrogen-containing heterocycles, particularly medium-sized benzazepinones, is of paramount importance in organic and medicinal chemistry due to their prevalence in pharmacologically active compounds.^{6,7} Among modern methodologies, radical-mediated cyclizations of alkenes stand out for their mild conditions and atom economy.⁸ Within this framework, *N*-allyl-*N*-tosyl aniline derivatives serve as privileged substrates for intramolecular sulfonyl radical additions. The title compound, *N*-[2-(1-phenylethenyl)phenyl]-*N*-[(4-methylphenyl)sulfonyl]prop-2-enamide, was designed as an advanced analogue featuring

*Corresponding author: Hongjuan Tong, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China, E-mail: dearthj@126.com. <https://orcid.org/0000-0001-7219-8018>

Zhoujing Zhu, Juanjuan Wen, Yanjiao Wang and Bin Liu, Xianyang Key Laboratory of Molecular Imaging and Drug Synthesis, School of Pharmacy, Shaanxi Institute of International Trade & Commerce, Xianyang, Shaanxi, China. <https://orcid.org/0000-0003-2852-7739> (B. Liu)

two distinct alkenes to probe chemoselective radical cyclizations. Preliminary studies confirm an exceptional regioselectivity favoring cyclization across the acrylamide moiety.^{5,9} To unravel the structural basis for this selectivity, we report herein its unambiguous solid-state molecular structure determined by single-crystal X-ray diffraction, providing crucial conformational insight into its reactivity.

The molecular structure of the title compound was unequivocally confirmed by single-crystal X-ray diffraction. The asymmetric unit comprises one discrete molecule. The molecular conformation is defined by key interplanar angles, revealing a near-orthogonal relationship (86.20°) between the 1-phenylethenyl substituent and the aniline moiety, whereas the p-toluenesulfonyl (tosyl) phenyl group and the aniline are nearly coplanar (dihedral angle = 23.82°). The amide functionality exhibits a C–N bond length of 1.405 Å, indicative of partial double-bond character consistent with electron delocalization within the N–C(=O) fragment. However, a significant twist of 39.55° between the acryloyl group and the aniline plane disrupts extended π -conjugation across the molecule.

In the crystal, molecules are assembled into a one-dimensional supramolecular chain *via* a pivotal C10–H10B...O1 hydrogen bond (H...O = 2.49 Å, D–H...A = 136°). These primary chains are further extended into a cohesive three-dimensional architecture through a combination of van der Waals forces and likely C–H... π interactions, which is commonly observed in similar tosylamide-containing compounds.^{10–13}

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