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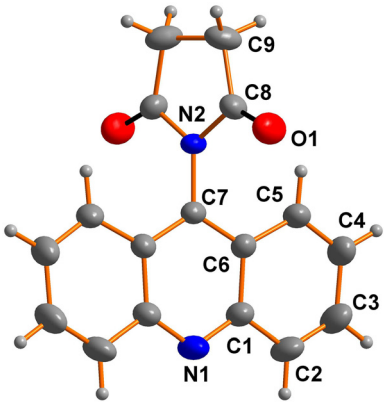
The crystal structure of 1-(acridin-9-yl)pyrrolidine-2,5-dione, $C_{17}H_{22}N_2O_2$



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Abstract

$C_{17}H_{22}N_2O_2$, orthorhombic, *Pbcn*, *a* = 15.4770(15) Å, *b* = 9.7581(9) Å, *c* = 8.8722(10) Å, *V* = 1339.9(2) Å^3, *Z* = 4, $R_{gt}(F)$ = 0.0477, $wR_{ref}(F^2)$ = 0.1057, T = 296(2) K. CCDC no.: 2442467.

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

First, 9-aminoacridine (0.194 g, 1 mmol) was dissolved in 15 mL of N,N-dimethylformamide (DMF). Subsequently, triethylamine (0.7 mL, 5 mmol), 4-dimethylaminopyridine (DMAP, 0.037 g, 0.3 mmol), and succinic anhydride (0.5 g, 5 mmol) were sequentially added to the solution. The reaction mixture was heated to 70 °C under stirring and maintained at this temperature for 5 h. After cooling to room temperature, the mixture was extracted with ethyl acetate and saturated brine, with the ethyl acetate layer being retained. The organic layer was then washed three times with saturated brine to remove residual DMF. The ethyl acetate was removed under reduced pressure to yield a brown solid, which was further purified by washing with ethanol to obtain the target product. The resulting solid was dissolved in 10 mL of diethyl ether, and colorless block-shaped crystals were obtained after allowing the ether to evaporate over 24 h.

Table 1: Data collection and handling.

Crystal:	Colorless Block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	Bruker APEX2, φ and ω scans
θ_{max}, completeness:	25.0°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	10342, 1186, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 814
$N(param)_{refined}$:	97
Programs:	Bruker,[1] SHELX,[2,3] Olex2[4]

2 Experimental details

The structure was solved by Direct Methods and refined with the SHELX[2,3] crystallographic software package. Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. The crystal structure was visualized by OLEX2 software package.

3 Comment

9-Aminoacridine is a compound possessing anti-cholinesterase activity.[3–5] To further enhance its anti-cholinesterase activity, we designed and synthesized a series of compounds using 9-aminoacridine as the core

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structure, among which the title compound represents one derivative. Due to the existence of imine isomers in 9-aminoacridine, the acylation site of the title compound could not be unambiguously determined through nuclear magnetic resonance (NMR) analysis.[6,7] Single-crystal X-ray diffraction serves as an effective and straightforward method for structural elucidation of chemical compounds.[8,9] The title molecule is located on a special position and the asymmetric unit contain one half of a molecule (see the figure).

In the structural characterization of the single crystal, all geometric parameters of the title compound fall within normal ranges. Crystallographic analysis reveals that the dihedral angle formed between the acridine planar skeleton and the C8–C9–N2 plane is 74.9°. Numerous crystal structures of aminoacridine derivatives have been reported,[10–13] among which *N*-(acridin-9-yl)-4-chloro-N-(4-chlorobutanoyl)butanamide (CCDC no. 2325358) exhibits a similar chemical framework to the title compound. The critical distinction lies in their structural motifs: the title compound is a cyclic diimide derivative, whereas the reference compound adopts a linear diamide configuration. Specifically, their crystallographic differences manifest in the bond parameters of the C–N bonds formed by the aminoacridine moiety. The C7–N2 and C8–N2 bond lengths in the title compound measure 1.435 Å and 1.394 Å, respectively, compared to 1.448 Å and 1.415 Å in the reference compound. Furthermore, the C7–N2–N8 bond angle in the title compound is 123.7°, significantly larger than the 114.6° observed in the reference compound.

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Author contributions: The synthesis of compounds and crystal preparation were completed by Dan Zhou, while the data analysis, writing and revision of the paper were completed by Dan Zhou, Hong Ji Li and Yu Qian Kan; supervision, Wen Li. All authors have read and agreed the published version of the manuscript.

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

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