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The crystal structure of dimethyl 8-(3-methoxy-2-(methoxycarbonyl)-3-oxoprop-1-en-1-yl)-4-methyl-1-(p-tolyl)-1,3a,4,8b-tetrahydro-3H-furo[3,4-b]indole-3,3-dicarboxylate. C_{28}H_{29}NO_{9}

Abstract

C_{28}H_{29}NO_{9}, triclinic, P\overline{1} (no. 2), a = 9.1736(7) Å, b = 12.4005(9) Å, c = 13.0593(9) Å, \alpha = 67.098(7)^\circ, \beta = 72.187(6)^\circ, \gamma = 88.103(6)^\circ, V = 1296.61(19) Å³, Z = 2, R_{gt} (F) = 0.0444, wR_{ref} (F^2) = 0.1119, T = 200 K.

CCDC no.: 2277072

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.

Table 1: Data collection and handling.

| Crystal: | Colourless block |
| Size: | 0.14 × 0.12 × 0.11 mm |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| μ: | 0.10 mm⁻¹ |
| Diffractometer, scan mode: | SuperNova, ω |
| \( \theta_{\text{max}} \), completeness: | 25.0°, >99% |
| N(hk|l)|measured, N(hk|l)|unique, R_{int}: | 8519, 4565, 0.024 |
| Criterion for \( I_{\text{obs}} \), N(hk|l)|φ|: | \( I_{\text{obs}} > 2\sigma(I_{\text{obs}}) \), 3684 |
| N(param|refined): | 349 |
| Programs: | Bruker\(^{1}\), SHELX\(^{2,5}\) |

1 Source of material

A flame-dried schlenk tube (25 mL) was evacuated and recharged with N\(_2\) for 3 times. Under N\(_2\) atmosphere, the tube was charged with 10 mol% Zn(NTf\(_2\))\(_2\), 4 Å M.S. (60 mg), dimethyl 2-(1-methyl-1H-indol-4-yl)methylene)malonate (0.3 mmol, 90 mg, synthesized by the reported procedure earlier\(^3\)), dimethyl 3-(p-tolyl)oxirane-2,2-dicarboxylate (0.45 mmol, 123 mg, synthesized by the reported procedure earlier\(^4\))
 Among them, 1H-furo[3,4-b]indole is a predicted as an important potential molecule in drug discovery and with a distance restraint of N-iso(H) set to 1.2 Å and re-duced with a distance restraint of N-iso(H) set to 1.2 Å and reduced with a distance restraint of N-iso(H) = 1.2 Å and reduced with a distance restraint of N-iso(H) = 1.2 Å and reduced with a distance restraint of N-iso(H) = 1.2 Å and reduced with a distance restraint of N-iso(H) = 1.2 Å.

**2 Experimental details**

The C-bound H atoms were geometrically placed (C–H = 0.95–0.98 Å) and refined as riding with $U_{	ext{iso}}(H) = 1.2–1.5 U_{	ext{eq}}(C)$. The N-bound H atoms were located in a difference Fourier map but were refined with a distance restraint of N–H = 0.88 – 0.91 Å, and with $U_{	ext{iso}}(H)$ set to 1.2 $U_{	ext{eq}}(N)^{3–5}$.

**3 Comment**

Fused indolines are present in a large number of biologically active alkaloids. Among them, 1H-furo[3,4-b]indole is a heterocyclic compound that consists of a fused indole ring system. 1,3-Dipolar cycloaddition of carbonyl ylide and indole has been successfully applied to synthesize the key intermediate 1H-furo[3,4-b]indole. It has also been identified as an important potential molecule in drug discovery due to its ability to modulate various biological targets. The unique chemical structure and diverse biological activities of 1H-furo[3,4-b]indole make it an important target for drug discovery and development. Therefore, a 1H-furo...
[3,4-b]indole analogue, for instance, the title compound, was synthesized.

As shown in the single crystal structure figure, there is one crystallographically independent molecule in the asymmetric unit. The compound contains two six membered rings, one aza five-membered ring and one tetrahydrofuran ring. Moreover, there are four methyl carboxylate groups. The C–O bond lengths in the carbonyl of four methyl carboxylate groups are similar. The torsion angles of C(3)–C(2)–C(7)–C(8) and C(8)–N(1)–C(3)–C(2) are 8.14(18)° and −11.88(19)° respectively, while the torsion angles of N(1)–C(8)–C(9)–O(3) and C(2)–C(7)–C(10)–O(3) are −123.58(15)° and 143.82(14)° respectively, which indicates that three fused rings are non-coplanar. In addition, the torsion angle of C(6)–C(1)–C(22)–C(23) is 42.3(3)°.

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

**Research funding:** Undergraduate Training Program for Innovative and Entrepreneurship in University (Grant No. 202310417006) and Research Start-Up Funding in University.

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

**References**