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Crystal structure of diethyl 2-(4-methoxyphenyl)-1-phenyl-1,2-dihydropyridine-3,5-dicarboxylate, $C_{24}H_{25}NO_5$

Table 1: Data collection and handling.

Crystal:	Colourless Prism
Size:	$0.35 \times 0.30 \times 0.25$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	26.0° , >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	7397, 4069, 0.020
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3395
$N(\text{param})_{\text{refined}}$:	274
Programs:	OLEX2 [1], SHELX [2], CrysAlis ^{PRO} [3]

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Abstract

$C_{24}H_{25}NO_5$, triclinic, $P\bar{1}$, $a = 9.2625(8)$ Å, $b = 10.5974(10)$ Å, $c = 11.7431(11)$ Å, $\alpha = 77.836(8)^\circ$, $\beta = 87.564(8)^\circ$, $\gamma = 66.951(9)^\circ$, $V = 1035.83(17)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0404$, $wR_{\text{ref}}(F^2) = 0.0985$, $T = 107.5$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

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Source of material

The solution of 4-methoxybenzaldehyde (272 mg, 2 mmol), aniline (186 mg, 2 mmol), ethyl propiolate (392 mg, 4 mmol) and 0.2 mL acetic acid in ethanol (30 mL) was heated in a steam bath for 2 h. The reaction mixture was evaporated to dryness in vacuo, washed with water and brine, and extracted with ethyl acetate (EA). The residue was purified by flash column chromatography (silica gel) using a mixed solvent of EA/hexane (5–10%) to afford the yellowish solid. Yield, 245 mg, 30%. The title compound (81 mg, 0.2 mmol) was dissolved in EA/hexane, and slowly evaporated at room temperature. After four days, colorless crystals were obtained.

Experimental details

All hydrogen atoms were placed in the calculated positions and all the non-hydrogen atoms were refined anisotropically.

Comment

Dihydropyridines (DHPs) are important nitrogen-containing heterocyclic compounds, which possess a π -conjugated system, as well as desirable the classic electron aromaticity and hydrogen-transfer ability [4]. These characteristics result in the application in synthetic chemistry [5–7] and medicinal chemistry [8, 9].

The NMR results are consistent with the proposed structure. The bond lengths and angles in crystallographically molecule are in the normal ranges [10]. In conclusion, we have developed a facile three-component reaction for the

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

Atom	x	y	z	U _{iso} ^a /U _{eq}
O1	−0.49221 (12)	0.71665 (11)	−0.02649 (9)	0.0293 (3)
O2	0.04274 (11)	0.05943 (11)	0.14012 (8)	0.0213 (2)
O3	−0.09874 (11)	−0.05960 (10)	0.23478 (8)	0.0191 (2)
O4	−0.29641 (12)	0.22339 (11)	0.67686 (8)	0.0232 (2)
O5	−0.31878 (11)	0.04811 (10)	0.60735 (8)	0.0204 (2)
N1	−0.02005 (13)	0.28587 (12)	0.39950 (9)	0.0156 (3)
C1	−0.04499 (16)	0.25486 (14)	0.28569 (11)	0.0162 (3)
H1	0.0550	0.2303	0.2467	0.019*
C2	−0.08748 (15)	0.12705 (14)	0.30905 (12)	0.0160 (3)
C3	−0.16182 (15)	0.09722 (14)	0.40554 (12)	0.0166 (3)
H3	−0.1978	0.0253	0.4139	0.020*
C4	−0.18615 (15)	0.17570 (14)	0.49589 (12)	0.0162 (3)
C5	−0.10389 (15)	0.25907 (14)	0.49189 (12)	0.0162 (3)
H5	−0.1056	0.2991	0.5555	0.019*
C6	−0.04017 (15)	0.04085 (14)	0.21979 (11)	0.0162 (3)
C7	−0.27122 (16)	0.15448 (15)	0.60154 (12)	0.0176 (3)
C8	−0.16712 (16)	0.38022 (14)	0.20529 (12)	0.0163 (3)
C9	−0.30892 (16)	0.46327 (15)	0.24524 (12)	0.0202 (3)
H9	−0.3292	0.4426	0.3237	0.024*
C10	−0.42117 (16)	0.57629 (15)	0.17133 (12)	0.0206 (3)
H10	−0.5147	0.6307	0.2002	0.025*
C11	−0.39211 (17)	0.60703 (15)	0.05399 (12)	0.0205 (3)
C12	−0.25110 (18)	0.52459 (17)	0.01223 (13)	0.0252 (3)
H12	−0.2314	0.5445	−0.0664	0.030*
C13	−0.14008 (17)	0.41313 (16)	0.08746 (12)	0.0224 (3)
H13	−0.0460	0.3595	0.0587	0.027*
C14	−0.64232 (18)	0.79714 (18)	0.01131 (15)	0.0331 (4)
H14A	−0.7022	0.8692	−0.0529	0.050*
H14B	−0.6288	0.8393	0.0727	0.050*
H14C	−0.6970	0.7370	0.0396	0.050*
C15	0.09745 (15)	0.33909 (14)	0.41003 (12)	0.0168 (3)
C16	0.12971 (16)	0.42634 (15)	0.31480 (12)	0.0207 (3)
H16	0.0716	0.4535	0.2448	0.025*
C17	0.24879 (17)	0.47261 (16)	0.32487 (13)	0.0231 (3)
H17	0.2711	0.5297	0.2610	0.028*
C18	0.33466 (17)	0.43430 (16)	0.42935 (13)	0.0235 (3)
H18	0.4142	0.4656	0.4357	0.028*
C19	0.30109 (17)	0.34899 (16)	0.52415 (13)	0.0230 (3)
H19	0.3571	0.3246	0.5948	0.028*
C20	0.18463 (16)	0.29958 (15)	0.51471 (12)	0.0196 (3)
H20	0.1648	0.2402	0.5781	0.023*
C21	−0.05236 (18)	−0.15066 (16)	0.15069 (13)	0.0226 (3)
H21A	0.0515	−0.2233	0.1721	0.027*
H21B	−0.0499	−0.0966	0.0735	0.027*
C22	−0.1708 (2)	−0.21540 (18)	0.15127 (14)	0.0319 (4)
H22A	−0.1725	−0.2684	0.2281	0.048*
H22B	−0.1426	−0.2764	0.0969	0.048*
H22C	−0.2729	−0.1427	0.1292	0.048*
C23	−0.40231 (17)	0.01698 (16)	0.71033 (12)	0.0220 (3)
H23A	−0.4815	0.1029	0.7265	0.026*
H23B	−0.3298	−0.0298	0.7776	0.026*
C24	−0.47784 (19)	−0.07666 (18)	0.68498 (14)	0.0299 (4)
H24A	−0.5488	−0.0294	0.6180	0.045*
H24B	−0.5348	−0.0990	0.7510	0.045*
H24C	−0.3983	−0.1615	0.6697	0.045*

synthesis of 1,2-dihydropyridines (1,2-DHPs) using a one-pot method. The method has several advantages, including easy experimental workup, the use of simple and available starting materials, high atom economy, and eco-friendly. This facile procedure may be used to synthesize a diverse range of 1,2-dihydropyridine derivatives for high throughput screening in medicinal chemistry.

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