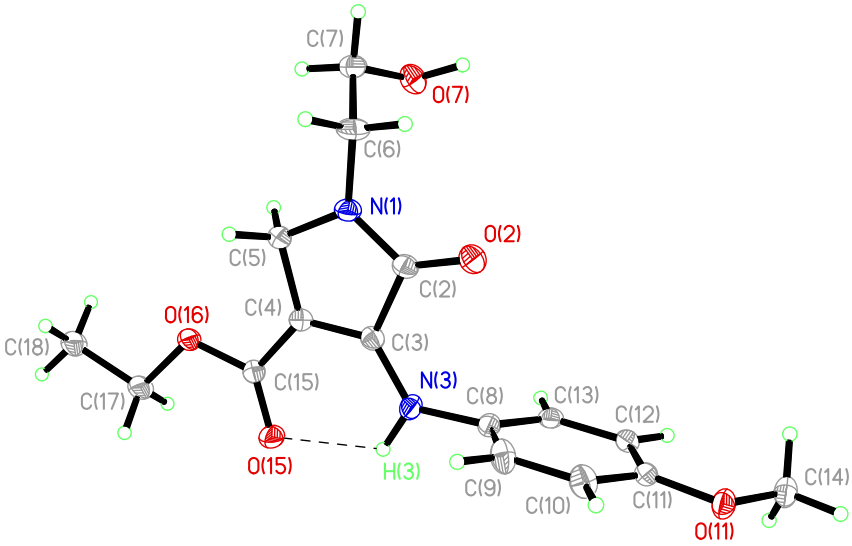
Fatin Nur Ain Abdul Rashid, Muhamad Zulfaqar Bacho, Alexandra M. Z. Slawin, Mohd Abdul Fatah Abdul Manan, Saiful Azmi Johari and Mohd Fazli Mohammat*

Crystal structure of ethyl 1-(2-hydroxyethyl)-4-((4-methoxyphenyl)amino)-5-oxo-2,5-dihydro-1*H*-pyrrole-3-carboxylate, $C_{16}H_{20}N_2O_5$



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Abstract

$C_{16}H_{20}N_2O_5$, triclinic, $P\bar{1}$ (no. 2), a = 7.9179(7) Å, b = 10.1982(9) Å, c = 10.4144(8) Å, α = 76.184(7)°, β = 87.090(7)°, γ = 73.500(8)°, V = 782.86(12) Å^3, Z = 2, $R_{gt}(F)$ = 0.0458, w$R_{ref}(F^2)$ = 0.1333, T = *93 K*.

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***Corresponding author: Mohd Fazli Mohammat**, Organic Synthesis Research Laboratory, Institute of Science, Universiti Teknologi MARA, 42300 Bandar Puncak Alam, Selangor, Malaysia,
E-mail: mohdfazli@uitm.edu.my
Fatin Nur Ain Abdul Rashid and Muhamad Zulfaqar Bacho, Organic Synthesis Research Laboratory, Institute of Science, Universiti Teknologi MARA, 42300 Bandar Puncak Alam, Selangor, Malaysia. https://orcid.org/0000-0002-2851-5503 (F.N.A. Abdul Rashid)
Alexandra M. Z. Slawin, EaStCHEM School of Chemistry, University of St Andrews, North Haugh, St Andrews, KY16 9ST, UK
Mohd Abdul Fatah Abdul Manan, Faculty of Applied Sciences, Universiti Teknologi MARA, 40450 Shah Alam, Selangor, Malaysia
Saiful Azmi Johari, Antimicrobial Laboratory, Anti-Infective Branch, Bioactivity Programme, Natural Products Division, Forest Research Institute Malaysia (FRIM), 52109 Kepong, Selangor, Malaysia. https://orcid.org/0000-0003-3898-2524

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 prism
Size:	0.20 × 0.10 × 0.10 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.10 mm^{-1}
Diffractometer, scan mode:	Rigaku XtaLAB P200, ω
θ_{max}, completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7943, 3178, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 2979
$N(param)_{refined}$:	218
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Mercury [4]

Source of materials

A mixture of 2,3-dioxopyrrolidine (2.00 g, 9.29 mmol), *p*-anisidine (1.37 g, 11.15 mmol) and formic acid (0.56 mL, 14.87 mmol) in ethanol was refluxed for 24 h. The solution was concentrated by distillation. The organic mixture was

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O2	0.11051 (12)	0.16554 (10)	0.41846 (9)	0.0256 (2)
O7	−0.05650 (12)	−0.00798 (9)	0.74929 (9)	0.0252 (2)
O11	0.62501 (12)	0.08398 (9)	0.02766 (8)	0.0218 (2)
O15	0.37685 (11)	0.49239 (9)	0.62943 (9)	0.0227 (2)
O16	0.15173 (11)	0.51537 (9)	0.77205 (8)	0.0210 (2)
N1	−0.03381 (13)	0.26014 (11)	0.58643 (10)	0.0192 (2)
N3	0.36381 (13)	0.32308 (11)	0.44944 (10)	0.0210 (2)
C2	0.09672 (15)	0.23665 (12)	0.49985 (11)	0.0180 (3)
C3	0.22057 (15)	0.31601 (12)	0.52269 (11)	0.0168 (3)
C4	0.15872 (15)	0.37848 (12)	0.62344 (11)	0.0171 (3)
C5	−0.00996 (15)	0.34710 (12)	0.67152 (11)	0.0182 (3)
H5A	0.000488	0.295627	0.765591	0.022*
H5B	−0.108557	0.434339	0.660656	0.022*
C6	−0.18845 (16)	0.21055 (14)	0.58967 (12)	0.0235 (3)
H6A	−0.183048	0.162756	0.516621	0.028*
H6B	−0.294674	0.292329	0.574819	0.028*
C7	−0.20545 (16)	0.10962 (13)	0.71960 (12)	0.0228 (3)
H7A	−0.222266	0.159903	0.791731	0.027*
H7B	−0.311069	0.077179	0.714958	0.027*
C8	0.42331 (15)	0.25494 (13)	0.34363 (12)	0.0183 (3)
C9	0.36126 (16)	0.32034 (13)	0.21533 (13)	0.0237 (3)
H9	0.271743	0.407427	0.198590	0.028*
C10	0.42897 (16)	0.25948 (13)	0.11218 (12)	0.0228 (3)
H10	0.385147	0.304037	0.024651	0.027*
C11	0.56138 (15)	0.13291 (12)	0.13611 (11)	0.0175 (3)
C12	0.62112 (15)	0.06565 (12)	0.26465 (12)	0.0176 (3)
H12	0.708970	−0.022341	0.281932	0.021*
C13	0.55152 (15)	0.12793 (12)	0.36767 (11)	0.0175 (3)
H13	0.592751	0.082492	0.455646	0.021*
C14	0.75390 (17)	−0.05001 (13)	0.05126 (12)	0.0240 (3)
H14A	0.858124	−0.044962	0.094595	0.029*
H14B	0.787361	−0.076083	−0.033088	0.029*
H14C	0.704607	−0.120876	0.108448	0.029*
C15	0.24119 (15)	0.46570 (12)	0.67301 (11)	0.0178 (3)
C17	0.22268 (18)	0.60931 (14)	0.82359 (12)	0.0246 (3)
H17A	0.228085	0.691792	0.752255	0.029*
H17B	0.343285	0.559982	0.859765	0.029*
C18	0.10359 (18)	0.65577 (14)	0.93055 (13)	0.0257 (3)
H18A	0.100625	0.573518	1.001217	0.031*
H18B	−0.015559	0.703724	0.893858	0.031*
H18C	0.147814	0.720445	0.966503	0.031*
H7	−0.058 (3)	−0.0669 (17)	0.6883 (15)	0.047 (5)*
H3	0.431 (2)	0.3830 (15)	0.4679 (17)	0.037 (4)*

extracted with dichloromethane, washed with water and dried over $MgSO_4$. The solvent was evaporated under reduced pressure and the crude was purified by column chromatography on silica gel (ethyl acetate:petroleum ether, 50:50) to afford **1** as brown solid product (2.34 g, 79%).

Experimental details

The data were indexed and processed using CrysAlis^{PRO} [1]. A multi-scan absorption correction was performed. The structure was solved with SHELXT Version 2018/2 [2] solution program. The model was refined with SHELXL Version 2018/3 [3].

Comment

Pyrrolinones play an important role in organic synthesis and find vast applications in pharmaceutical fields due to their broad range of biological properties such as anti-inflammatory, antifungal, and antibacterial activities [5, 6]. Owing to their notable pharmaceutical effects, they have received tremendous attention over the past few years [7]. Thus, in view of diversifying the structural motifs of these classes of compounds, we decided to prepare the analogue of *N*-substituted pyrrolinone *via* amination of 2,3-dioxopyrrolidine with *p*-anisidine [8]. The starting material, 2,3-dioxopyrrolidine was initially prepared according to the reported procedure [9–11]. This paper describes the crystal structure of ethyl 1-(2-hydroxyethyl)-4-((4-methoxyphenyl)amino)-5-oxo-2,5-dihydro-1*H*-pyrrole-3-carboxylate (**1**).

The crystal structure of **1** is similar to previously reported structure of ethyl 1-(4-fluorophenyl)-4-phenyl-1*H*-pyrrole-3-carboxylate [12]. All bond angles and lengths are within the normal ranges and comparable to the related structures [12–15]. The 4-methoxyaniline (N3/C8–C13/O11/C–14) and hydroxyethyl (C6/C7/O7) moieties are observed slightly twisted at N3–C8 and C6–C7 bonds, respectively. The twisted angle of C3–N3–C8–C9 is found to be 87.97° while the twisted angle for N1–C6–C7–O7 is −57.11. The ethyl acetate (O15/O16/C15/C17/C18) and 4-methoxyaniline (N3/C8–C13/O11/C14) moieties are almost perpendicular to each other, subtending a dihedral angle of 86.00°. Meanwhile, the pyrrole ring (N1/C2–C5) forms a dihedral angle of 83.17° with the phenyl ring (C8–C13). In the molecule, an intramolecular N3–H3···O15 hydrogen bond is observed, forming a pseudo six-membered ring, which further stabilizes and lock its atoms in a nearly planar arrangement. A similar feature was observed in the reported structure of ethyl 3-(4-methoxyphenyl)-5-methylcarbamoyl-1*H*-pyrazole-4-carboxylate [16].

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

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