

## SINGLE CRYSTAL X-RAY STRUCTURAL ANALYSIS OF TWO POLYMORPHS OF ETHYL 2-(2-CHLOROPHENYL)-4-HYDROXY-5-OXO-1-PHENYL- 2,5-DIHYDRO-1H-PYRROLE-3-CARBOXYLATE

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A single crystal X-ray structure analysis of two polymorphs of ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate, *i.e.* **I** ( $Z' = 1$ ) and **II** ( $Z' = 4$ ), is performed. It is shown that the molecular geometries are the same in **I** and **II**, and the same bifurcated O–H···O hydrogen bonds (involving hydroxyl and ester C=O groups) exist between the adjacent molecules, giving rise to molecular dimers which, however, are different in **I** and **II**: centrosymmetric (heterochiral) and homochiral, respectively.

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**Keywords:** single crystal, X-ray analysis, pyrrolidinone derivative, polymorphs.

### INTRODUCTION

Pyrrolidinone derivatives are substantial and important compounds due to their broad range of biological and pharmacological activities [1]. They have attracted much interest owing to their HIV-1 integrase inhibitors [2], antibacterial [3], antimicrobial [4], anti-inflammatory [5], anticancer [6], and antitumor [7] activities. The development of simple synthetic routes for widely used organic compounds from readily available reagents is one of the major tasks in organic chemistry. Because of these characteristics and our interest in the synthesis of heterocyclic compounds, we were prompted to synthesize pyrrolidinone derivatives from aniline, diethyl acetylene dicarboxylate, and aldehyde by citric acid monohydrate as a green catalyst under ultrasound irradiation in an excellent yield [8].

### EXPERIMENTAL

Ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate was obtained using the procedure described in [8]. Crystals of **I** and **II** were obtained after slow evaporation of the mother liquor at room

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temperature. Two kinds of colorless crystals grew in the same vessel: a larger amount of big nicely formed blocks of **I** (m.p. = 193-194 °C) and fewer small, poor quality, slightly twinned needle-like plates of **II** (m.p. = 190-191 °C) which turned out to be polymorphs of the same compound ( $Z' = 1$  for **I** and 4 for **II**).

The crystallographic measurements of polymorphs **I** and **II** were performed on a Kuma KM4-CCD and an Agilent Technologies Xcalibur R  $\kappa$ -geometry automated four-circle diffractometers equipped with a CCD camera (Sapphire2 and Ruby, respectively) and graphite-monochromated  $\text{MoK}_\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). The data were collected at 100(2) K and 120(2) K using the Oxford-Cryosystems coolers. Data were corrected for Lorentz and polarization effects. Data collection, cell refinement, data reduction, and analysis were carried out with CrysAlisCCD and CrysAlisRED (for **I**) or CrysAlisPro (for **II**) [9, 10]. The empirical (multi-scan) absorption correction was applied to the data with use of CrysAlisRED or CrysAlisPro. The structures were solved with direct methods using SHELXT-2014 [11] and refined on  $F^2$  by a full-matrix least squares technique with the anisotropic thermal parameters for non-H atoms with SHELXL-2014 [12]. The H atoms in polymorph **I** were found in difference Fourier maps and initially refined isotropically. Those in polymorph **II** were included using geometrical considerations. In the final refinement cycles, the C-bonded H atoms in **I** and all H atoms in **II** were repositioned in their calculated positions and refined using a riding model, with C–H = 0.95-1.00 Å, O–H = 0.84 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for CH and CH<sub>2</sub> and  $1.5U_{\text{eq}}(\text{C})$  for CH<sub>3</sub> and OH. Hydroxyl H atom in polymorph **I** was refined freely. Crystal data, data collection and structure refinement details for **I** and **II** are summarized in Table 1. Figures were made with

**TABLE 1.** Experimental Details for Polymorphs **I** and **II**

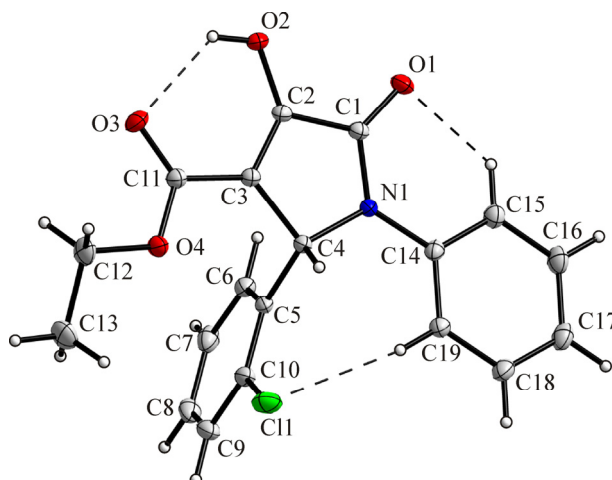
Parameter	Polymorph <b>I</b>	Polymorph <b>II</b>
CCDC No.	1826657	1826658
Chemical formula	$\text{C}_{19}\text{H}_{16}\text{ClNO}_4$	
$M_r$	357.78	
Crystal system, space group	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Temperature, K	100(2)	120(2)
$a, b, c, \text{ \AA}$	8.984(4), 9.707(3), 11.407(4)	14.717(3), 15.984(4), 16.690(4)
$\alpha, \beta, \gamma, \text{ deg.}$	67.25(3), 84.62(3), 63.38(4)	118.37(3), 104.61(3), 92.02(3)
$V, \text{ \AA}^3$	816.3(6)	3286.9(16)
$Z$	2	8
$D_{\text{calc}}, \text{ g/cm}^3$	1.456	1.446
Radiation type	$\text{MoK}_\alpha$	
$\mu, \text{ mm}^{-1}$	0.26	0.26
Crystal size, mm	0.46×0.45×0.22	0.54×0.10×0.03
Diffractometer	Kuma KM4-CCD $\kappa$ -geometry diffractometer with a Sapphire2 CCD camera	Agilent Technologies Xcalibur R $\kappa$ -geometry diffractometer with a Ruby CCD camera
Absorption correction	Multi-scan	
$T_{\text{min}}, T_{\text{max}}$	0.730, 1.000	0.945, 1.000
No. of measured, independent and observed [ $I > 2\sigma(I)$ ] reflections	9980, 4495, 4076	19190, 11940, 5933
$R_{\text{int}}$	0.035	0.071
$(\sin \theta/\lambda)_{\text{max}}, \text{ \AA}^{-1}$	0.703	0.606
$R[F^2 > 2\sigma(F^2)], wR(F^2), S$	0.051, 0.144, 1.10	0.095, 0.250, 1.00
No. of reflections	4495	11940
No. of parameters	230	910
No. of restraints	0	6
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}, \text{ e/\AA}^3$	0.55, -0.44	1.02, -0.51

the DIAMOND program [13]. The crystallographic information files (CIFs) were deposited with the Cambridge Crystallographic Data Centre ([www.ccdc.cam.ac.uk/](http://www.ccdc.cam.ac.uk/); deposition numbers CCDC-1826657, 1826658). C–H $\cdots\pi$  interactions were analysed using the PLATON program [14].

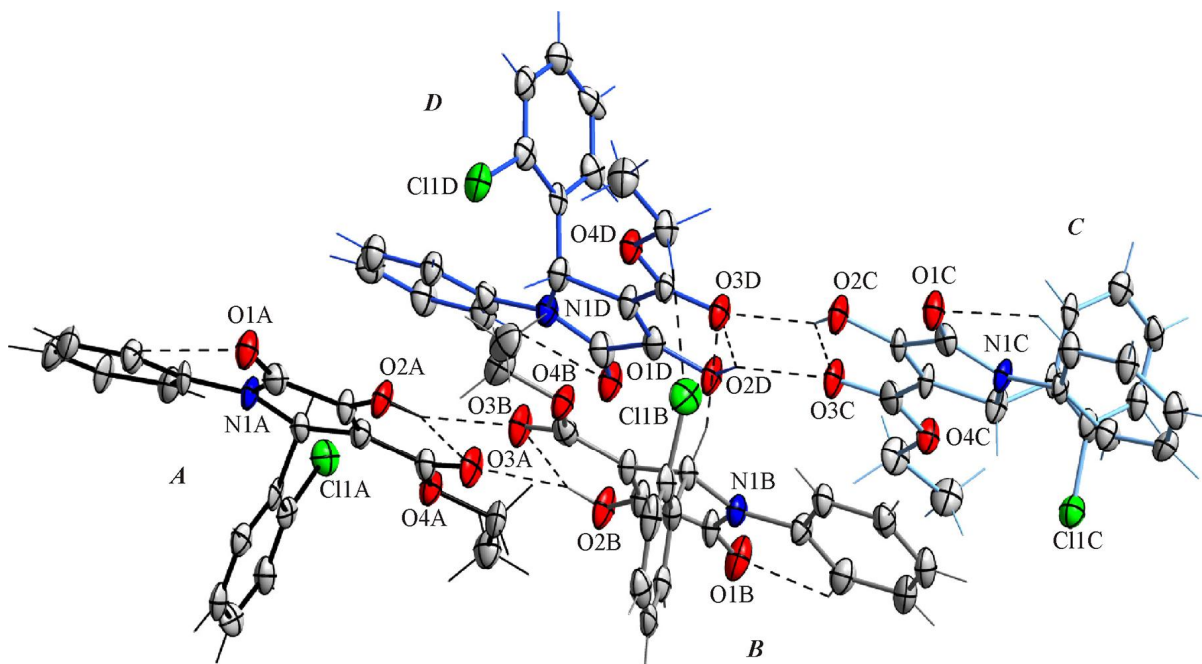
## RESULTS AND DISCUSSION

The synthesis of ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate in the presence of citric acid in a green solvent under ultrasound irradiation was tested. The structure of the product was confirmed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR and FT-IR spectroscopy [8].

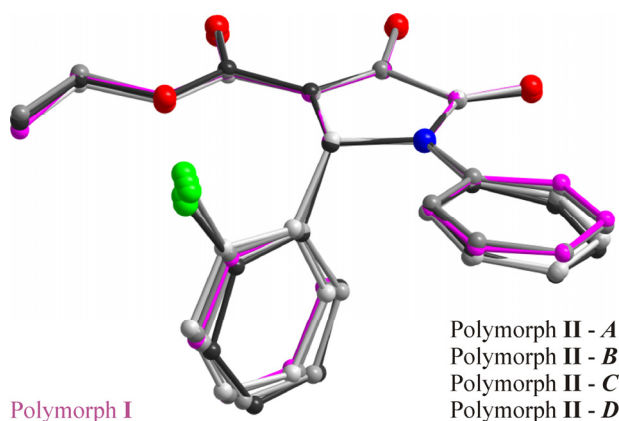
The single crystal X-ray analysis of two polymorphs (**I** and **II**) of this compound was performed. Both crystals are centrosymmetric (space group  $P\bar{1}$ ), thus they contain a racemic compound. The *S*-enantiomers of ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate present in **I** and **II** are shown in Figs. 1 and 2, respectively. As shown in Fig. 2, there are four crystallographically independent molecules of the compound in the crystal of polymorph **II**, however, their overall conformations, as well as the conformation of the molecule in **I**, are almost identical, as shown in Fig. 3. The selected geometrical parameters for all the five molecules present in **I** and **II** are given in Table 2. The overall conformation of the molecules bears many common features with the analogous compounds: the 2-(4-fluorophenyl) derivative reported by us previously [8], and isomorphous to it, the 2-phenyl compound [15] (Cambridge Structural Database (CSD) refcode LIFBEJ) [16]. The C(O)OEt groups in **I** and **II** are planar and almost coplanar with the planes of the respective pyrroline rings (see the C2–C3–C11–O3, C12–O4–C11–C3, and C11–O4–C12–C13 torsion angles in Table 2 which are close to 0° or 180°). This results mainly from the long chain of conjugation, starting from the ester group, through the C3=C2–C1(O) fragment, and ending with N1–Ph. The ester C=O group and the pyrroline hydroxyl substituent are in the *syn* arrangement, which is accompanied by the formation of the intramolecular C–O–H $\cdots$ O=C hydrogen bond stabilizing the overall planar conformation of molecules in **I** and **II** (Figs. 1, 2 and Tables 3, 4). The same was also previously observed in the molecules of 2-(4-fluorophenyl) and 2-phenyl derivatives [8, 15]. In contrast, in the similar compound, *i.e.* methyl 2-phenyl-4-hydroxy-5-oxo-1-(4-chlorophenyl)-2,5-dihydro-1*H*-pyrrole-3-carboxylate [15] (CSD refcode LIFBOT), the C=O<sub>ester</sub> and C–OH groups are in the *anti* orientation to each other. Additional stabilization of the planar conformation of the molecules in both **I** and **II** is provided by weak C15–H15 $\cdots$ O1 hydrogen contacts. As in all mentioned above analogous



**Fig. 1.** Asymmetric unit of polymorph **I** crystal, showing the atom numbering scheme and symmetry-independent intramolecular hydrogen bonds (dashed lines). The *S* enantiomer is shown. Displacement ellipsoids are drawn at the 50% probability level.



**Fig. 2.** Four crystallographically independent molecules present in the asymmetric unit of polymorph **II** crystal, showing the partial atom numbering scheme and symmetry-independent intramolecular hydrogen bonds (dashed lines). The *S* enantiomers are shown. Displacement ellipsoids are drawn at the 50% probability level. (Atom numbering scheme for molecules *A–D* is the same as for polymorph **I**).



**Fig. 3.** Comparison of the geometry of all the molecules of polymorphs **I** and **II** (molecules *A*, *B*, *C*, *D*). The common reference points are pyrroline atoms. H atoms are omitted for clarity.

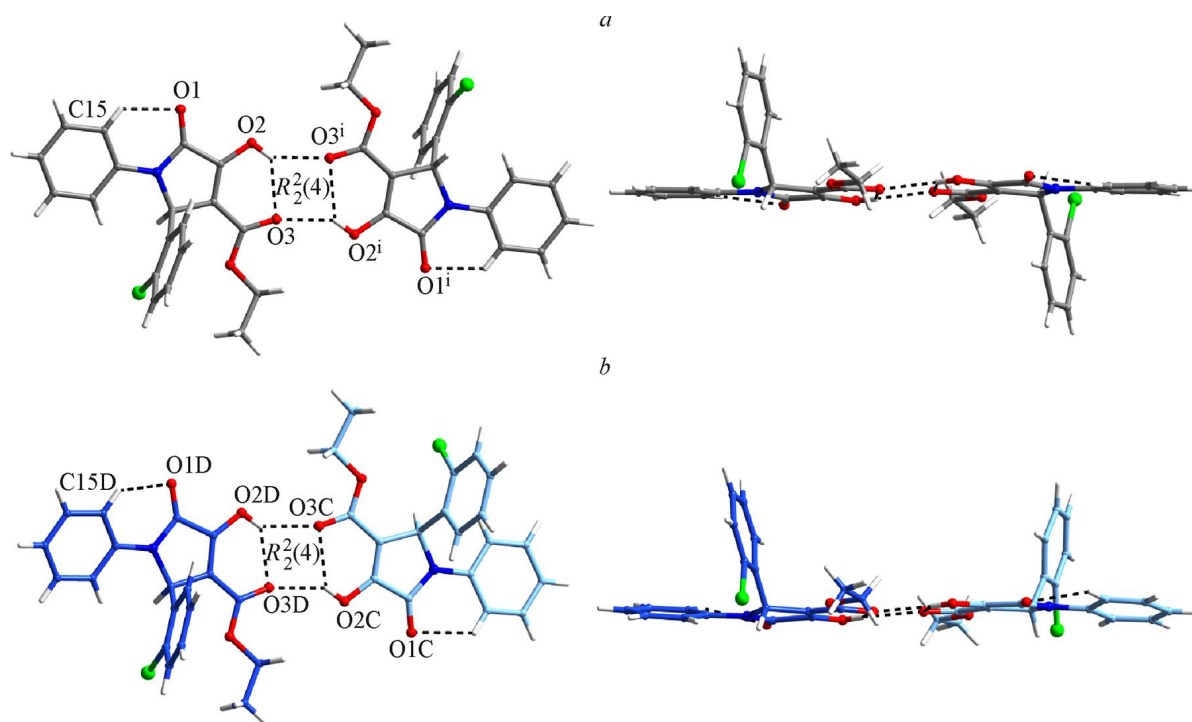
compounds, phenyl substituents at N1 atoms in **I** and **II** are almost coplanar with the pyrroline fragments, while the remaining substituents, *i.e.* 2-chlorophenyl in **I** and **II** and 4-fluorophenyl or phenyl rings in the other similar compounds [8, 15], adopt a synclinal arrangement relative to the pyrroline rings (see the values of C1–N1–C14–C15 and N1–C4–C5–C6 torsion angles in Table 2).

The common structural motif observed in the crystals of both polymorphs **I** and **II** is the molecular dimer built up from two molecules interacting with each other *via* two bifurcated O–H $\cdots$ O hydrogen bonds (*e.g.* O2–H2 $\cdots$ O3/O3<sup>i</sup> in **I**), which gives rise to the intradimeric  $R_2^2(4)$  ring motif. Seemingly, dimers are very similar in **I** and **II** (the molecular

**TABLE 2.** Selected Geometric Parameters (Å, deg.) for Polymorphs **I** and **II**

Parameter	Polymorph <b>I</b>		Polymorph <b>II</b>		
		Molecule <b>A</b>	Molecule <b>B</b>	Molecule <b>C</b>	Molecule <b>D</b>
O1–C1	1.2230(16)	1.215(7)	1.196(7)	1.214(6)	1.225(7)
O2–C2	1.3283(16)	1.328(6)	1.342(6)	1.337(6)	1.314(7)
N1–C1	1.3756(18)	1.387(7)	1.391(7)	1.372(7)	1.379(8)
N1–C4	1.4821(15)	1.471(7)	1.467(7)	1.477(6)	1.489(7)
N1–C14	1.4235(15)	1.422(7)	1.425(7)	1.423(7)	1.430(7)
C1–N1–C14–C15	–8.01(18)	–11.3(10)	–10.2(10)	–14.8(9)	–13.0(10)
C2–C3–C11–O3	3.8(2)	–1.0(10)	–2.3(11)	0.9(10)	–1.0(10)
C12–O4–C11–C3	179.28(11)	178.9(5)	–179.0(5)	178.6(5)	–177.7(5)
C11–O4–C12–C13	163.00(12)	173.4(5)	176.2(5)	178.8(5)	175.1(5)
N1–C4–C5–C6	–49.05(14)	–44.2(7)	–41.3(7)	–46.6(7)	–44.6(7)

geometries as well as the intermolecular interactions within the dimers are the same). However, a closer look reveals a significant difference between them. As shown in Fig. 4, these dimers are centrosymmetric (heterochiral) in **I** and homochiral (formed by two molecules of the same chirality) in **II**. As a result, the chlorophenyl rings are located on the opposite sides (in **I**) or on the same side (in **II**) of the central planar fragment of the dimer. This is reflected in the lower symmetry and a higher number of molecules in the asymmetric unit ( $Z' = 4$  in **II** vs. 1 in **I**) of polymorph **II** compared to **I** (both centrosymmetric). It is to note that in the previously described 2-(4-chlorophenyl) and 2-phenyl derivatives, with the *syn* mutual arrangement of C=O<sub>ester</sub> and C–OH groups [8, 15], homochiral dimers were observed, while in the LIFBOT crystal, with the *anti* mutual arrangement of C=O<sub>ester</sub> and C–OH groups, analogous molecular dimers do not occur.



**Fig. 4.** Heterochiral (centrosymmetric) molecular dimer observed in polymorph **I** (*a*) compared to the homochiral dimer (one of two crystallographically independent) from polymorph **II** (*b*). Top (left) and side (right) views are shown. Bifurcated O–H $\cdots$ O hydrogen bonds and intramolecular C–H $\cdots$ O contacts are shown by dashed lines.

**TABLE 3.** Geometry of Hydrogen Bonds and C–H··· $\pi$  Contacts (Å, deg.) for Polymorph **I**

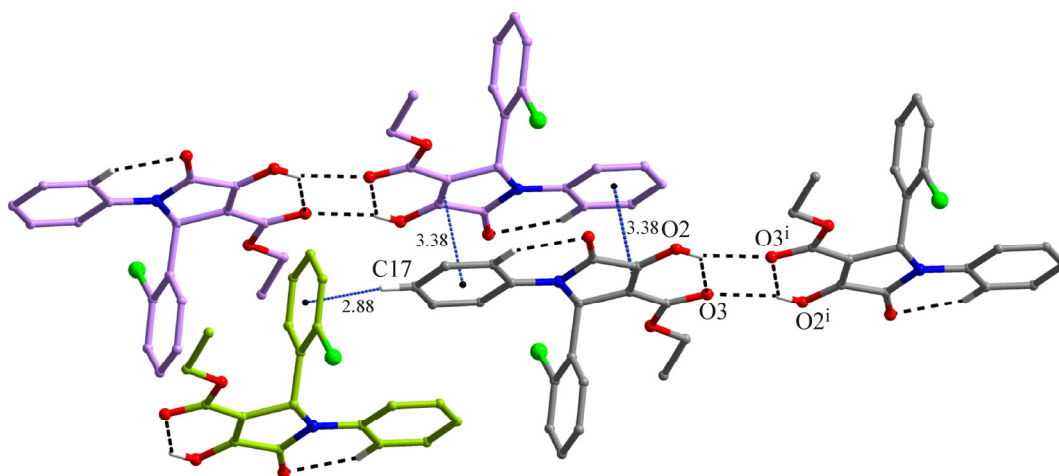
$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$	
O2–H2···O3	0.86(3)	2.21(3)	2.8490(19)	131(2)	
O2–H2···O3 <sup>i</sup>	0.86(3)	2.06(3)	2.7981(17)	144(2)	
C9–H9···O2 <sup>ii</sup>	0.95	2.62	3.384(2)	138	
C12–H12B···O1 <sup>iii</sup>	0.99	2.56	3.392(2)	142	
C15–H15···O1	0.95	2.22	2.866(2)	124	
C19–H19···Cl1	0.95	2.88	3.827(2)	172	
$C-H\cdots\pi^a$	$C-H$	$H\cdots Cg, H\cdots\pi^b$	$C\cdots Cg$	$C-H\cdots Cg$	$C-H\cdots\pi^c$
C17–H17···Cg1 <sup>iv</sup>	0.95	2.88, 2.65	3.750(2)	152	78

Symmetry codes: <sup>i</sup>  $-x+1, -y, -z+1$ ; <sup>ii</sup>  $x-1, y+1, z$ ; <sup>iii</sup>  $x-1, y, z$ ; <sup>iv</sup>  $-x+1, -y+2, -z$ ; <sup>v</sup>  $-x+1, -y+1, -z$ .

<sup>a</sup> Cg1 is the centroids of chlorophenyl C5/C6/C7/C8/C9/C10 ring.

<sup>b</sup> H··· $\pi$  is the perpendicular distance of H atom to ring plane.

<sup>c</sup> C–H··· $\pi$  is the angle of the C–H bond with the  $\pi$ -plane.



**Fig. 5.** Centrosymmetric molecular dimers in **I**, interacting with each other *via* C–H··· $\pi$  contacts and C=C double bond-phenyl  $\pi$ -stacking interactions (dotted lines). H atoms not involved in the shown interactions have been omitted for clarity.

In the crystals of polymorphs **I** and **II**, the adjacent dimers interact with each other *via* the network of C–H···O hydrogen bonds, C–H··· $\pi$  contacts, and C=C double bond-phenyl  $\pi$ -stacking interactions. In **I**, C9 and C12 atoms are donors of interdimeric C–H···O bonds to carbonyl O1 and hydroxyl O2 atoms, while C17 is involved in the C–H··· $\pi$  contact to the chlorophenyl ring (Fig. 5 and Table 3). The adjacent molecules from two different dimers (grey and light grey in Fig. 5), facing each other with their planar sides, interact *via* two centrosymmetric C=C double bond-phenyl  $\pi$ -stacking interactions, with the distance from the centroid of the C14/C15/C16/C17/C18/C19 ring to the midpoint of the C2=C3 double bond being 3.38 Å.

In the crystal of **II**, additional intermolecular C12–H···Cl contacts (between molecules **D** and **B**) are observed (Fig. 6 and Table 4). Differently to that was observed in **I**, here C4, C6, and C16 atoms are involved in the inter-dimeric C–H···O hydrogen bonds, acceptors of which are mainly carbonyl O1 atoms. The greater role is played by the C–H··· $\pi$  interactions: C12 and C17 atoms of each molecule in **II** form C–H··· $\pi$  contacts to phenyl and chlorophenyl rings, respectively. The C=C double bond-phenyl  $\pi$ -stacking is observed between molecules **A** and **D**, as well as between **B** and **C**,

**TABLE 4.** Geometry of Hydrogen Bonds and C–H··· $\pi$  Contacts ( $\text{\AA}$ , deg.) for Polymorph **II**

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2A—H2A···O3A	0.84	2.19	2.834(5)	134
O2A—H2A···O3B	0.84	2.10	2.842(6)	148
C6A—H6A···O1A <sup>i</sup>	0.95	2.58	3.366(7)	141
C15A—H15A···O1A	0.95	2.26	2.891(7)	123
O2B—H2B···O3A	0.84	2.06	2.817(6)	149
O2B—H2B···O3B	0.84	2.24	2.881(6)	133
C4B—H4B···O3D	1.00	2.59	3.379(8)	136
C15B—H15B···O1B	0.95	2.27	2.899(7)	123
O2C—H2C···O3C	0.84	2.20	2.844(5)	134
O2C—H2C···O3D	0.84	2.09	2.820(5)	146
C6C—H6C···O1C <sup>ii</sup>	0.95	2.45	3.270(8)	144
C15C—H15C···O1C	0.95	2.26	2.874(7)	122
C16C—H16C···O1D <sup>iii</sup>	0.95	2.59	3.484(7)	156
O2D—H2D···O3C	0.84	2.14	2.879(5)	146
O2D—H2D···O3D	0.84	2.20	2.847(5)	134
C12D—H12H···C11B	0.99	2.84	3.825(7)	172
C15D—H15D···O1D	0.95	2.30	2.923(7)	123

$C-H\cdots\pi^{(a)}$	$C-H$	$H\cdots Cg, H\cdots\pi^{(b)}$	$C\cdots Cg$	$C-H\cdots Cg$	$C-H\cdots\pi^{(c)}$
C12A—H12A···Cg1B <sup>iv</sup>	0.99	2.97, 2.94	3.776(8)	139	48
C12B—H12C···Cg1A <sup>i</sup>	0.99	2.90, 2.88	3.740(8)	144	50
C12C—H12E···Cg1D <sup>v</sup>	0.99	3.00, 2.98	3.823(8)	142	48
C12D—H12G···Cg1C <sup>ii</sup>	0.99	2.62, 2.61	3.457(8)	142	55
C17A—H17A···Cg2B <sup>i</sup>	0.95	2.67, 2.63	3.587(6)	163	79
C17B—H17B···Cg2A <sup>iv</sup>	0.95	2.72, 2.59	3.614(7)	157	79
C17C—H17C···Cg2D <sup>ii</sup>	0.95	2.74, 2.70	3.624(6)	155	75
C17D—H17D···Cg2C <sup>v</sup>	0.95	2.76, 2.66	3.647(7)	156	76

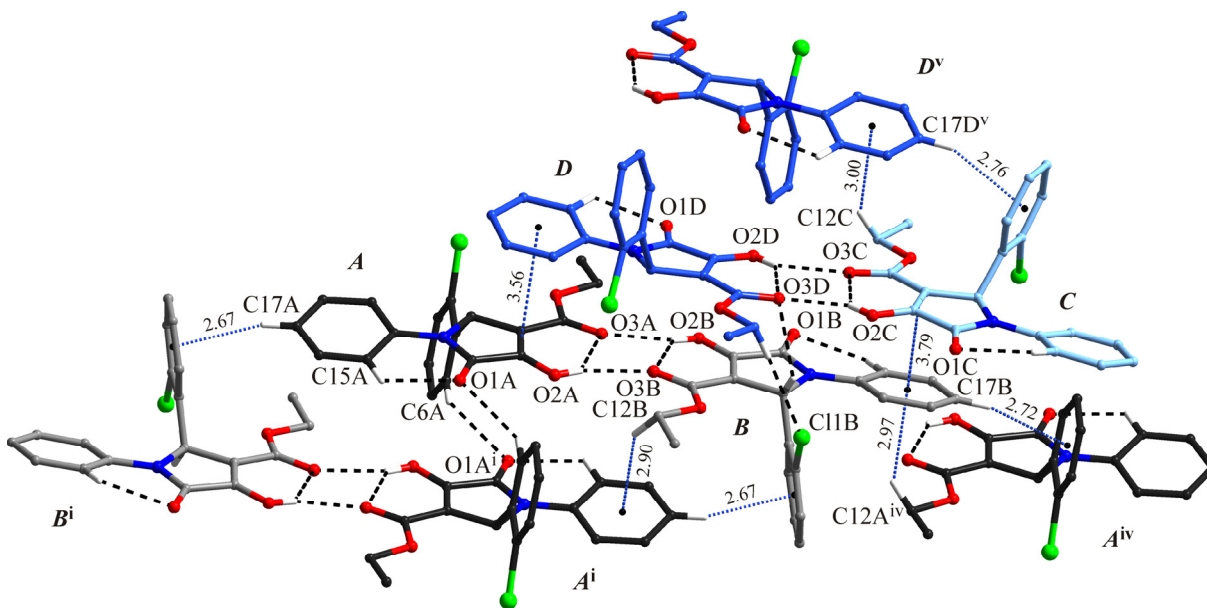
Symmetry codes: <sup>i</sup>  $-x+2, -y+2, -z+2$ ; <sup>ii</sup>  $-x+1, -y+1, -z$ ; <sup>iii</sup>  $x, y, z-1$ ; <sup>iv</sup>  $-x+2, -y+1, -z+1$ ; <sup>v</sup>  $-x+1, -y+1, -z+1$ .

<sup>a</sup> Cg1 and Cg2 are the centroids of phenyl C14/C15/C16/C17/C18/C19 and chlorophenyl C5/C6/C7/C8/C9/C10 rings, respectively.

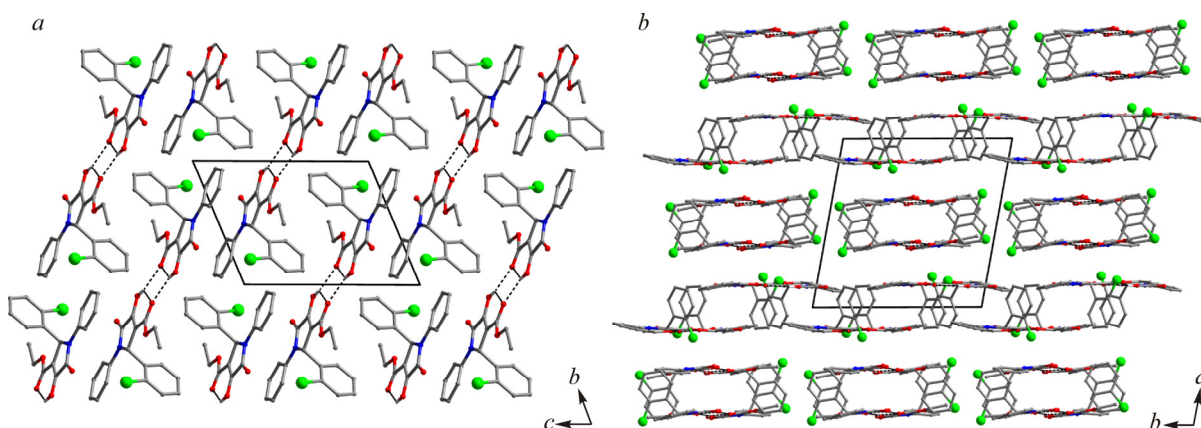
<sup>b</sup> H··· $\pi$  is the perpendicular distance of H atom to ring plane.

<sup>c</sup> C–H··· $\pi$  is the angle of the C–H bond with the  $\pi$  plane.

but the distances from the midpoints of the C2=C3 double bonds to the centroids of the phenyl rings are longer and amount to 3.56  $\text{\AA}$  and 3.79  $\text{\AA}$ , for  $A\cdots D$  and  $B\cdots C$ , respectively. Different architectures of dimers in **I** and **II**, along with different interdimeric interactions give rise to quite different crystal packing diagrams observed in the polymorphs. However, both **I** and **II** reveal the bilayer architecture (Fig. 7), with the main difference lying in the fact that the centrosymmetric double



**Fig. 6.** Homochiral molecular dimers in **II**, interacting with each other *via* C–H···O, C–H···Cl hydrogen bonds (dashed lines), C–H··· $\pi$  contacts, and C=C double bond-phenyl  $\pi$ -stacking interactions (dotted lines). H atoms not involved in the shown interactions have been omitted for clarity.



**Fig. 7.** Comparison of crystal packing diagrams of **I** (a) and **II** (b), showing intradimeric O–H···O hydrogen bonds (dashed lines). C-bound H atoms have been omitted for clarity.

layers in **I** are formed by centrosymmetric dimers, whereas in **II**, two kinds of centrosymmetric double layers (built up from molecules **A** and **B** or **C** and **D**) are formed by homochiral dimers (e.g. **AB**) linked by the centre of symmetry to another homochiral dimer.

## CONCLUSIONS

In this study, the single crystal X-ray analysis of ethyl 2-(2-chlorophenyl)-4-hydroxy-5-oxo-1-phenyl-2,5-dihydro-1*H*-pyrrole-3-carboxylate was performed. Both crystals are centrosymmetric (space group  $P\bar{1}$ ), thus they contain the racemic compound. The common structural motif observed in the crystals of polymorphs **I** and **II** is the molecular dimer built up from two molecules interacting with each other *via* two bifurcated O–H···O hydrogen bonds, which gives rise to the intradimeric  $R_2^2(4)$  ring motif. Dimers are centrosymmetric (heterochiral) in **I** and homochiral (formed by two molecules of the same chirality) in **II**.



## FUNDING

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## CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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