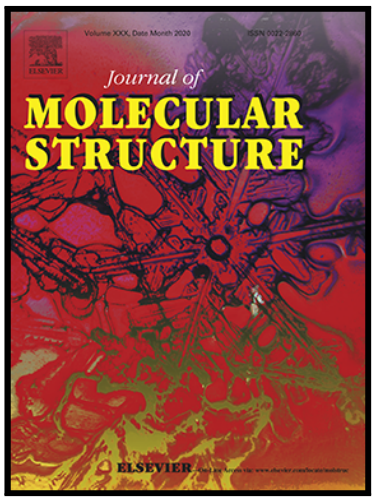

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Synthesis, Crystal structure, DFT studies and Hirshfeld surface analysis of novel isoxazole derivatives

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Highlights

- Novel isoxazole derivatives was synthesized using natural product as a starting material and characterized by ^{1}H and ^{13}C NMR and confirmed by single crystal X-ray diffraction method.
- Interactions in crystal packing were supported by Hirshfeld surface analysis.
- DFT calculation have been investigated and compared with X-ray results.

Abstract

This article describes firstly the synthesis of a new series of isoxazole **5a-e** from p-methoxythymol, extracted from *Tetraclinis Articulata*, as starting material involving 1,3-dipolar cycloaddition reaction and secondly a detailed study of the molecular packing and intermolecular interactions in crystals of five related 5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-phenylisoxazole derivatives. All compounds were synthesized and subjected to solid state characterization by single-crystal X-ray diffraction analysis, and to studies with the use of NMR and Hirshfeld surface analysis. All structures display intermolecular C—H···O hydrogen bonding and C—H···π interactions, forming layers in the crystal lattice. The crystal structures of compounds **5a**; **5c** and **5d** are consolidated by π—π interactions. Hirshfeld surface analysis, the d_{norm} surfaces, electrostatic potential and two-dimensional fingerprint plots were examined to verify the contributions of the different intermolecular contacts within the supramolecular structure. The most important contributions for the crystal packing are from H···H, H···C/C···H and O···H/H···O interactions. Additionally, DFT calculations have been used to analyze the electronic and geometric frontier molecular orbital and Molecular Electrostatic Potential map analyses of the compounds were produced using the optimized structures.

Keywords: Phenolic monoterpenoids, Isoxazole, 1,3-dipolar cycloaddition, Crystal structure, Hydrogen bond, C—H...π interactions, Hirshfeld surface analysis, DFT

1- Introduction

Due to their chemical and biological importance, heterocyclic moieties are attractive targets in medicinal and pharmaceutical chemistry [**1**]. Among heterocyclic compounds, isoxazole has proved to be the one of the most important moieties in organic synthesis. The wide range of biological activities displayed by isoxazole compounds include cytotoxic [**2, 3**], antimicrobial [**4**], analgesic [**5**], antirhinovirus [**6**], anti-inflammatory [**7**], anthelmintic [8], muscle relaxant [**9**], hypoglycemic [**10**], adenosine antagonist [**11**], fungicidal [**12, 13**], herbicidal [**14, 15**], nematocidal [**16**], insecticidal [**17**], antiparasitic [**18**]. The Modification of isoxazole structure has offered a high degree of diversity that has proven useful for the development of new therapeutic agents having improved potency and reduced toxicity. All above biological activities of isoxazole derivatives aroused our attention and inspired us to prepare a new series of isoxazole.

The p-methoxythymol is an important phenolic monoterpenoïd (PM) obtained from the essential oil of many plants such as the genus Origanum, Thymus [**19**] and Satureja [**20**]. It exhibits a large number of pharmacological activities including antimicrobial [**21**], anti-inflammatory [**22**], analgesic, and antioxidant [**23**]. Additionally, phenolic monoterpenoïds are generally regarded as safe (GRAS) food flavoring, which is an indication of low

mammalian toxicity starting materials [**24**]. Due to the aforementioned biological activities, phenolic monoterpenoïd could be highly promising alternatives to synthetic agents for the development of natural and nontoxic therapeutic agents [**25**]. At the same time, it is also suitable as a starting molecule for the synthesis of organic and bio-organic product analog based fine chemicals [**26, 27**].

Encouraged by the aforementioned facts, we envisioned that the introduction of a substituted isoxazole pharmacophore into the parent p-methoxythymol scaffold might produce some new compounds with multiple biological activities. In this study, we describe the design and synthesis of a number of novel isoxazoles derivatives bearing PM.

2- Experimental

All reagents were used as obtained from commercial sources (Aldrich and Acros). Melting points (m.p.) was determined using a Kofler bench apparatus and are uncorrected (They are not taken with a calibrated thermometer). Elemental analyses were taken with an Elementar Vario EL-III Analyzer. Analytical thin-layer chromatography (TLC) was performed on aluminum plates percolated with E. Merck silica gel 60 F254 to a thickness of 0.25 mm. IR spectra were recorded on a Bruker Vertex 70 spectro-photometer using potassium bromide discs in the frequency range 4000–400 cm^{-1}. ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance 300 (75) MHz spectrometer. Chemical shifts (δ ppm) were reported with reference to $SiMe_4$ (^{1}H). All solvents were dried and distilled before use. Phenolic monoterpene **2** used as a starting material for triazole hémisynthesis were isolated by column chromatography from the dichloromethanic extract of the root part of medicinal plant *Tetraclinis articulate*.

2-1 Hemisynthesis

2.1.1 General procedure for the extraction of paramethoxythymol 3 from *Tetraclinis articulata*

Powdered wood of *Tetraclinis articulata* (500 g) was exhaustively extracted by maceration with dichloromethane at room temperature for 24 h under agitation. The solvent was removed in a rotavapor and the residue obtained (26 g) was chromatographed in a silica gel column eluting with a solvent gradient hexane– EtOAc (9:1 v/v) affording 2.9 g of p-methoxythymol **3**, whose identification was confirmed on the basis of its spectroscopic analyses.

2.1.2 General procedure for the synthesis of substituted benzaldoximes 2a-e

To a stirred solution of H_2O/Ethanol (14 ml, 1:1) substituted benzaldehyde **1a-e** (14 mmol), hydroxylamine hydrochloride (1.5 eq, 21 mmol) and NaOH (5.8 ml, 35 mmol, 6.0 M in H_2O) was added. After 5 h, the reaction was quenched with 2 M HCl (10 ml), extracted with Et_2O (3 x 20 ml), and washed with brine (50 ml). The dried (Na_2SO_4) organic layers were concentrated in vacuo to give crude oxime **2a-e** as colorless solids.

2.1.3 General procedure for O-propargylation of paramethoxythymol 3

To a solution of corresponding PM **3** in dry acetone, K_2CO_3 (1 equiv) was added. The reaction mixture was stirred for 30 min and propargyl bromide (1 equiv) was added drop-wise. The mixture was stirred overnight until none of the starting materials were detected using TLC. The solution was concentrated and solubilized in 60ml of CH_2Cl_2. The organic layer was washed with distilled water (2 x 10mL), dried over Na_2SO_4 and concentrated. The resulting crude product was purified by silica gel column chromatography using n-hexane–ethyl acetate (95:5 v/v) as eluent.

1-isopropyl-5-methoxy-4-methyl-2-(prop-2-yn-1-yloxy)benzene, **4.**

Light yellow liquid, Yield 85%; IR (KBr), ν_{max}/cm^{-1}: 3281 (≡C-H), 2960 (C-H), 2120 (C≡C), 1587 (C=C), 1041 (C-O); ^1H NMR (300 MHz, $CDCl_3$) δ(ppm) 6.77 (s, H-6), 6.62 (s, H-3), 5.18 (s, H-10), 3.83 (s, H-11), 3.37 (sept, J= 7.3 Hz, H-9), 2.44 (s, H-13), 2.23 (s,H-11), 1.25 (d, J= 7.3 Hz, H-8); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 158.9 (C_{Ar}), 140.6 (C_{Ar}), 135.6 (C_{Ar}), 130.1 (CH_{Ar}), 128.0 (C_{Ar}), 114.3 (CH_{Ar}), 78.0 (≡CH),76.0 (≡C-), 55.9 (OCH_2), 52.6 (OCH_3), 26.5 (CH), 22.4 (CH_3), 19.3(CH_3). Analysis calculated (%) for $C_{14}H_{18}O_2$: C, 77.03; H, 8.31; found: C, 77.11; H, 8.26.

2.1.4 Typical experimental procedure for the preparation of isoxazoles 5a-e

To a solution of compound alkyne (1 equiv) in dichloromethane (25ml), substituted benzaldoximes (1.1 equiv) and sodium hypochlorite (9–12% in water) (10 ml) were added at 0 °C. Then the reaction mixture was stirred at 0°C for 2-5 h. The progress of the reaction was

monitored by TLC analysis (15% EtOAc/hexane). Then, 50 ml of water was added and the reaction mixture was extracted with dichloromethane (3x20ml). The combined organic layer was washed with water, followed by brine and dried over anhydrous Na_2SO_4 and concentrated in vacuo. The residue was purified by flash chromatography on silica gel using n-hexane–ethyl acetate (92:8 v/v) as eluent.

5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-phenylisoxazole, **5a.**

Colorless crystal, m.p.: 87-89 °C, Yield:70%; IR (KBr), ν_{max}/cm^{-1}: 2954 (C-H), 1609 (C=N), 1511 (C=C), 1209,1056 (C-O); ^{1}H NMR (300 MHz, $CDCl_3$) δ(ppm) 7.88-7.85 (d, J = 6.9 Hz, 2H,Ar-H), 7.49-7.48 (m, 3H,Ar-H), 6.80 (s, 1H), 6.79 (s, 1H), 6.66 (s, $1H_{isoxazole}$), 5.17 (s, 2H,CH_2), 3.85 (s, 3H,OCH_3), 3.44-3.38 (m, 1H), 2.25 (s, 1H), 1.33-1.28 (d, J= 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 169.3(C_{Ar}), 162.5(C_{Ar}), 152.9(C_{Ar}), 148.7(C_{Ar}), 136(C_{Ar}), 130.2(CH_{Ar}), 128.9(CH_{Ar}), 126.9(CH_{Ar}), 124.5(C_{Ar}), 116.1(CH_{Ar}), 109(CH_{Ar}), 101(CH_{Ar}), 63(OCH_2), 55.9(OCH_3), 27.1(CH), 23.1(2×CH_3)., 16.1(CH_3). Analysis calculated (%) for $C_{21}H_{23}NO_3$:C, 74.75; H, 6.87; N, 4.15; found: C, 74.72; H, 6.82; N, 4.06.

5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-(p-tolyl)isoxazole, **5b.**

Colorless crystal, m.p.: 123–125°C, Yield: 74%; IR (KBr), ν_{max}/cm^{-1}: 2958 (C-H), 1612 (C=N), 1509 (C=C), 1203,1032 (C-O); ^{1}H NMR (300 MHz, $CDCl_3$) δ(ppm) 7.78 (d, J = 8.1 Hz, 2H,Ar-H), 7.45 (d, J = 8.1Hz, 2H,Ar-H), 6.83 (s, 1H), 6.82 (s, 1H), 6.66 (s, $1H_{isoxazole}$), 5.16 (s, 2H,CH_2), 3.84 (s, 3H,OCH_3), 3.44 (m, 1H), 2.44 (s,3H, CH_3), 2.29 (m, 1H), 1.33-1.28 (d, J= 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 169.1(C_{Ar}), 162.4(C_{Ar}), 152.9(C_{Ar}),148.7(C_{Ar}), 140.2(C_{Ar}), 136(C_{Ar}), 129.6(CH_{Ar}), 126.7(CH_{Ar}), 126.1(C_{Ar}), 124.5(C_{Ar}), 116(CH_{Ar}), 109(CH_{Ar}), 101(CH_{Ar}), 63(OCH_2), 55.9(OCH_3), 27.1(CH), 23(CH_3), 21.4(CH_3), 16.1(CH_3). Analysis calculated (%) for $C_{22}H_{25}NO_3$: C, 75.19; H, 7.17; N, 3.99; found: C, 75.12; H, 7.12; N, 3.95.

3-(4-chlorophenyl)-5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)isoxazole, **5c.**

Colorless crystal, m.p.: 128–130°C, Yield: 71%; IR (KBr), ν_{max}/cm^{-1}: 2956 (C-H), 1606 (C=N), 1509 (C=C), 1205,1032 (C-O), 832 (C-Cl); ^{1}H NMR (300 MHz, $CDCl_3$) δ(ppm) 7.78 (d, J = 8.4 Hz, 2H,Ar-H), 7.45 (d, J = 8.4Hz, 2H,Ar-H), 6.77 (s, 1H), 6.76 (s, 1H), 6.62 (s, $1H_{isoxazole}$), 5.16 (s, 2H,CH_2), 3.84 (s, 3H,OCH_3), 3.39-3.35 (m, 1H), 2.23 (s,3H, CH_3), 1.27-1.21 (d, J= 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 169.6($\underline{C}_{Ar}$), 161.5($\underline{C}_{Ar}$), 152.9($\underline{C}_{Ar}$), 148.6($\underline{C}_{Ar}$), 136.1($\underline{C}_{Ar}$), 136($\underline{C}H_{Ar}$), 129.2($\underline{C}H_{Ar}$), 128.1($\underline{C}H_{Ar}$), 127.4($\underline{C}_{Ar}$),

124.5($\underline{C}_{Ar}$), 116($\underline{C}H_{Ar}$), 109($\underline{C}H_{Ar}$), 100.8($\underline{C}H_{Ar}$), 63($O\underline{C}H_2$), 55.9($O\underline{C}H_3$), 27($\underline{C}H$), 23($\underline{C}H_3$), 16($\underline{C}H_3$). Analysis calculated (%) for $C_{21}H_{22}ClNO_3$ C, 67.83; H, 5.96; Cl, 9.53; N, 3.77, found: C, 67.77; H, 5.91; N, 3.72.

5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-(4-nitrophenyl)isoxazole, **5d.**
Yellow Color crystals, m.p.:126–128 °C, Yield: 68%; IR (KBr), ν_{max}/cm^{-1}: 2939 (C-H), 1600 (C=N), 1517 (C=C), 1205, 1062 (C-O); ^{1}H NMR (300 MHz, $CDCl_3$) δ(ppm) 7.78 (d, J = 7.2 Hz, 2H,Ar-H), 7.45 (d, J = 7.2 Hz, 2H,Ar-H), 6.77 (s, 1H), 6.76 (s, 1H), 6.73 (s, $1H_{isoxale}$), 5.19 (s, 2H,CH_2), 3.83 (s, 3H,OCH_3), 3.38-3.34 (m, 1H), 2.21 (s,3H, CH_3), 1.33-1.24 (d, J= 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 170.4(C_{Ar}), 160.6(C_{Ar}), 152.9(C_{Ar}), 148.7(C_{Ar}), 148.4(C_{Ar}), 135.9(C_{Ar}), 134.9(C_{Ar}), 127.7(CH_{Ar}), 124.5(C_{Ar}),, 124.2(CH_{Ar}), 115.9(CH_{Ar}), 109(CH_{Ar}), 101.1(CH_{Ar}), 62.9(OCH_2), 55.9(OCH_3), 27(CH_3), 23(CH_3), 16.07(CH_3). Analysis calculated (%) for $C_{21}H_{22}N_2O_5$ C, 65.96; H, 5.80; N, 7.33; found C, 65.92; H, 5.75; N, 7.29.

5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-(2-methoxyphenyl)isoxazole, **5e.**
Colorless crystal, m.p.: 117–119 °C; Yield: 68%; IR (KBr), ν_{max}/cm^{-1}: 2956 (C-H), 1603 (C=N), 1507 (C=C), 1202,1033 (C-O); ^{1}H NMR (300 MHz, $CDCl_3$) δ(ppm) 7.97-7.94 (d, J = 7.8 Hz, 2H,Ar-H), 7.45-7.42 (m, 1H,Ar-H), 7.11-7.06 (m, 2H,Ar-H), 6.89 (s, 1H), 6.82 (s, 1H), 6.79 (s, $1H_{isoxazole}$), 5.16 (s, 2H,CH_2), 3.91 (s,3H,OCH_3), 3.86 (s, 3H,OCH_3), 3.44 (m, 1H), 2.26 (s, 3H,CH_3), 1.31-1.28 (d, J= 7.2 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ(ppm) 167.7($\underline{C}_{Ar}$), 160($\underline{C}_{Ar}$), 157.2($\underline{C}_{Ar}$), 152.7($\underline{C}_{Ar}$), 148.8($\underline{C}_{Ar}$), 136($\underline{C}_{Ar}$), 131.2($\underline{C}H_{Ar}$), 129.4($\underline{C}H_{Ar}$), 124.4($\underline{C}_{Ar}$), 120.9($\underline{C}H_{Ar}$), 117.8($\underline{C}_{Ar}$), 116.2($\underline{C}H_{Ar}$),111.4($\underline{C}H_{Ar}$), 108.9($\underline{C}H_{Ar}$), 104.7($\underline{C}H_{Ar}$), 63.03($O\underline{C}H_2$), 55.8($O\underline{C}H_3$), 55.4($O\underline{C}H_3$), 27($\underline{C}H_3$), 22.9($\underline{C}H_3$), 16($\underline{C}H_3$). Analysis calculated (%) for $C_{22}H_{25}NO_4$ C, 71.91; H, 6.86; N, 3.81; found C, 71.87; H, 6.80; N, 3.77.

2.2 Single crystal X-ray data collection

The X-ray intensity data were collected on a Bruker D8 Venture Super DUO diffractometer with PHOTON100 CMOS area-detector using MoKα radiation (λ= 0.71073Å) monochromated by graphite at room temperature. The following software was used: Frame integration, Bruker SAINT software package [**28**] and data were corrected for Lorentz-Polarization effects and for absorption by the multi-scan semi-empirical method implanted in SADABS [**29].** The structures were solved by direct methods using SHELXT-2014/5 [**30**]

and refined (by weighted full matrix least-square on F^2 techniques) to convergence using the SHELXL-2018/3 program **[31]**. All non hydrogen atoms were refined with anisotropic thermal parameters. The C-bound H atoms were geometrically placed (C—H = 0.93–0.98 Å) and refined as riding with *U*iso(H) = 1.2–1.5*U*eq(C). Crystal data, data collection and structure refinement details for **5a-e** were summarized in Table 1. The molecular graphic were drawn using Diamond programs [**32**].

2.3 Hirshfeld surface and 2D fingerprint plots

The Hirshfeld surfaces calculated for compounds **5a-e** were analyzed to clarify the nature of the intermolecular interactions between different units in the crystal packing motif and visualize them graphically. Thus, a Hirshfeld surface analysis [**33**] and the associated two-dimensional fingerprint plots [**34**] were performed using CrystalExplorer17.5 [**35**] to figure out the normalized contact distance (d_{norm}), which depends on contact distances to the closest atoms outside (d_e) and inside (d_i) the surface. The molecular HS were performed using a standard (high) surface resolution with the three-dimensional surfaces mapped.

2.4 Quantum chemical calculations

Density functional theory (DFT) has proved to be useful for studying the electronic structures of all molecules under investigation. The geometry optimization was performed by means of the Gaussian 09 W program and GaussView molecular visualization software [**36, 37**] on a personal computer, based on density functional theory DFT, using Beck's three parameters hybrid functional exchanges [**38**], with 6-311G (d,p), 6-311++G(d,p) and 6-311++G basis sets and Lee-Yang-Parr correlation functional (B3LYP) [**39, 40**]. Single crystal XRD structural models were taken as a starting geometry. The Molecular Electrostatic Potential (MEP) and Mulliken charges were determined to investigate the charge distribution on the all molecules **5a-e**.

3. Results and discussion

3.1. Synthesis

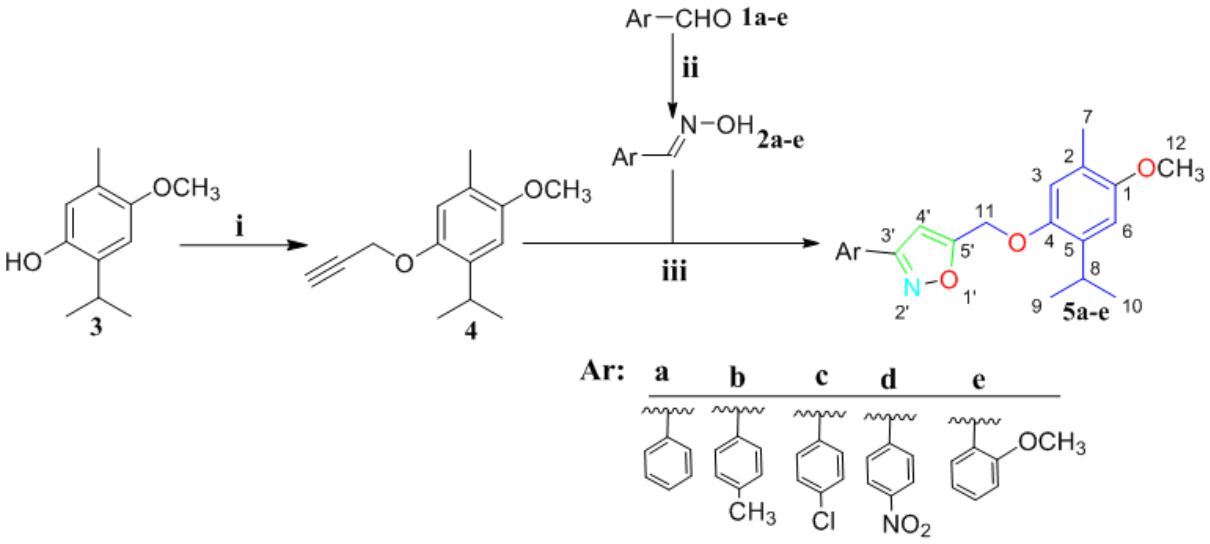
Several methods exist for the synthesis of isoxazole rings. The important and the most used method is the [3+2] cycloaddition reaction [**41**]. The overall synthetic route of isoxazole derived based parathoxythymol is shown in Scheme 1. Our approach to synthesize the

target isoxazoles began with the O-propargylation of paramethoxythymol **3** following previously reported procedures [**42**]. We first treated paramethoxythymol **3** with an excess of propargyl bromide in the presence of potassium bicarbonate in dry acetone under magnetic stirring for 12 hours at room temperature to give compound **4**. Its structure was confirmed according to its spectral data, where the IR spectra indicated the existence of the band at ν_{max} = 3293cm^{-1} that corresponded to the alkyne proton. Its ^{1}H NMR spectrum showed two singlets at 5.18 ppm attributable to the ethylenic proton and at 2.44 ppm attributable to the terminal methylenic protons, respectively. Its ^{13}C NMR spectrum confirmed the above spectral data with the appearance of new signals at δ_C 56.06, 76.04 and 80.38ppm that corresponded to the carbons of the propargyl bromide used.

The benzaldoxime **2a** and its substitutes **2b-e** were prepared via addition-elimination reactions between aromatic aldehyde **1a-e**, hydroxylamine hydrochloride and potassium hydroxide in an ethanol/ water mixture at room temperature. The structures of compounds **2a-e** were identified by their melting points and FT-IR spectroscopy. The physical properties and FT-IR spectroscopy were corresponding to those described in the literature [**43**].

In the next step, the obtained oximes **2a-e** was converted to the related nitrileloxids *in situ* with NaOCl. Then, reaction of nitrileoxides with propargyl compounds **3** by a [3+2] cycloaddition reaction gave the related isoxazoles **5a-e** in good yields (55–74%).

The structures of these compounds were confirmed according to their spectral data, where the IR spectra of compounds **5a-e** indicated the existence of the isoxazole C=N group at ν_{max} =1598–1610 cm^{-1}. The ^{1}H NMR spectra of compounds **5a–e** indicated the existence of the isoxazolic moieties by showing essentially the resonance of the protons introduced by the arylnitrileloxids. The ^{1}H NMR spectrum for **5b** as an example, showed a singlet at $\delta_{\mathbf{H}}$ 5.36 relative to the methylenic protons $H_{\mathbf{11}}$ and another singlet at δ_H 6.66 corresponding to the isoxazole proton $H_{\mathbf{4'}}$. The same spectrum showed two doublets at δ_H 7.78 and 7.45ppm (J=8.1 Hz) attributable to the protons aromatic. The disappearance of the signal for the alkyne proton at 2.44ppm favored the cycloaddition reaction. Further, the ^{13}C NMR spectrum of this cycloadduct **5b** was also a good support for the proposed structure which exhibited characteristic signals at δ_C 63.03ppm corresponding to O–CH_2 carbon, and at δ_C 101.04, 162.4, and 169.1ppm relative to isoxazole carbons. Single crystal structures of **5a** and **5e** were also recorded to further confirm the proposed structures.



Scheme 1. Synthetic route of isoxazole. Reagents and conditions: (i) Propargyl bromide, K_2CO_3, Acetone, rt, 12 h; (ii) $NH_2OH.HCl$, KOH, EtOH:H_2O (1:1), rt, 10 h; (iii) NaOCl, DCM 0°C, 5h.

3.2 Crystal structure description of 5a-e

A suitable single crystals of compounds **5a**-**e** for X-ray diffraction study was obtained by p-methoxythymol extracted from *Tetraclinic Articulata* with 1,3-dipolar cycloaddition. The plot of the crystal structures belonging to the prepared compounds is illustrated in Fig. 1. All compounds crystallize in the triclinic system, space group P$\bar{1}$. Each structure contains three essentially planar aromatic or heterocyclic rings, i.e. benzene (C1—C6), isoxazole (atoms C3'–C5'/N1/ O1), and (C13—C18), of which the latter two are directly connected via C3' and C13 atoms. Selected bond lengths and angles of these compounds are also given in Table 2. Hydrogen bond distances and angles together with details of offset C—H···π and π···π contacts for **5a**-**e** are shown in Table 3. The molecular structures of the five compounds **5a-e** are sufficiently similar to be discussed together as they vary only in the substitution pattern on the benzene ring of the isoxazole system. The phenyl is substituted by a single methyl, Cl and NO_2 group at C16 for **5b**, **5c** and **5d** respectively, while **5e** carries methoxy substituent at C18. A comparison of related distances and angles within the isoxazole ring shows a good agreement between all five structures, with a systematically C3'—N1 and C4'—C5' bonds lengths ranging from 1.304 to 1.316 Å and 1.332 to 1.346Å, corresponding to C=N and C=C double bond, respectively. The N1-O1 single bond present values from 1.405 to 1.415Å. The C5'—O1 and C4—O2 bond lengths are ranging 1.345 to 1.356 Å and 1.378-1.389 Å respectively, indicating the presence of a single bond between oxygen and a sp^2 carbon atom,

whereas the single bond lengths C11—O2 (1.396-1.427 Å) are slightly longer due to oxygen and a sp^3 carbon. Geometrical parameters are in a good agreement with those of the related compounds containing phenyl-isoxazole [**44-46**]. A larger discrepancy is observed for the dihedral angle between the isoxazole ring and the phenyl ring in the p-methoxythymolin: 64.59(8)° (**5c)** and 71.25(11)° (**5e),** this is much larger than the value of 16.04(1)° (**5a**), 8.34(8)° (**5b**), 10.55(12)° (**5d**), 7.19(2) [**43**], 3.63(2) [**43**], 17.1(1)° [**44**] and 36.8(2)° [**46**], while it has close that found in [**45**] (73.04(8)°).

It is noteworthy that the nitrogen N atom does not participation an intermolecular interaction like in all compounds. In addition, a weak C—H···π interaction involving different donor groups and acceptor π-ring systems are present in all crystals. A reasonable separation between aromatic rings to be considered as sensible π···π contacts is < 4.0 Å so that those observed here in compounds **5a**, **5c** and **5d** can be considered to be quite significant.

Table 1: Crystal Data, Summary of Intensity Data Collection, and Structure Refinement

	5a	**5b**	**5c**	**5d**	**5e**
Empirical formula	$C_{21}H_{23}NO_3$	$C_{22}H_{25}NO_3$	$C_{21}H_{22}ClNO_3$	$C_{21}H_{22}N_2O_5$	$C_{22}H_{25}NO_4$
Formula weight	337.40	351.43	371.84	382.40	367.43
Temperature	296(2)	296(2)	296(2)	296(2)	296(2)
Wavelength	0.71073 Å	0.71073 Å	0.71073 Å	0.71073 Å	0.71073 Å
Crystal system, space group	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
a, b, c (Å)	9.4958(10), 9.5333(15), 10.2143(16)	7.8247(3), 10.4383(5), 12.4226(6)	8.792(4), 10.217 (5), 11.316(5)	8.391(2), 10.488(4), 11.224(5)	10.4871(5), 10.5128(5), 10.7032(6)
α, β, γ (°)	96.630(6), 90.474(5), 93.885(6)	78.202(2), 83.069(2), 77.892(2),	102.89(2), 103.51(2), 96.93(2),	97.4752°, 94.48(2), 98.5352°	72.751(2), 84.704(2), 63.683(2)
Volume (A^3)	916.2(2)	967.89(8)	947.3(8)	963.7(6)	1009.08(9)
Z	2	2	2	2	2
Calculated density (g/cm^3)	1.223	1.206	1.304	1.318	1.209
μ (mm^{-1})	0.081	0.080	0.222	0.095	0.083
Crystal shape and color	Plate, colorless	Block, colorless	Block, colorless	Plate, yellow	Block, colorless
Crystal size (mm)	0.38x0.26x0.15	0.41×0.22×0.16	0.33×0.22×0.13	0.40x 0.27x0.13	0.36x0.26x0.11
Theta range for data collection (°)	2.770 – 26.367	2.671- 27.999	2.423- 30.508	2.467– 25.681	2.256 – 24.999
Limiting indices	-11 ≤h ≤11, -11 ≤ k ≤11, -12 ≤ l≤ 12	-10 ≤h ≤10, -13 ≤ k ≤13, -16 ≤ l ≤ 16	-12 ≤h ≤12, -14 ≤ k ≤14, -16 ≤ l ≤ 16	-10≤h ≤10, -12≤ k ≤12, -13 ≤ l≤ 13	-12 ≤h ≤12, -12 ≤ k ≤12, -13 ≤ l≤ 13
Diffractometer	Bruker D8 VENTURE Super DUO	Bruker D8 VENTURE Super DUO	Bruker D8 VENTURE Super DUO	Bruker D8 VENTURE Super DUO	Bruker D8 VENTURE Super DUO
Absorption correction	Multi-scan (*SADABS*, Brucker, 2016)	Multi-scan (*SADABS*, Brucker, 2016)	Multi-scan (*SADABS*, Brucker, 2016)	Multi-scan (*SADABS*, Brucker, 2016)	Multi-scan (*SADABS*, Brucker, 2016)
Tmin. Tmax.	0.6862-0.7462	0.6918-0.7462	0.7041-0.7465	0.6624-0.7462	0.6874-0.7465
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	32299, 3746, 2716 [R(int) = 0.042]	39315, 4684, 3211 [R(int) = 0.039]	34664, 5774, 4096 [R(int) = 0.036]	21479, 3656, 2375 [R(int) = 0.044]	33948, 3828, 2640 [R(int) = 0.05]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	3746/0/231	4684/ 0 / 241	5774/ 0 / 240	3371/0/258	3828/0/250
R[$F^2 > 2\sigma(F^2)$], wR(F^2), S	0.048, 0.133, 1.03	0.049, 0.153, 1.10	0.046, 0.138, 1.03	0.060, 0.174, 1.06	0.049, 0.135, 1.02
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e $Å^{-3}$)	0.16, -0.16	0.25, -0.16	0.29, -0.29	0.17, -0.21	0.15, -0.13

Table 2: Selected experimental and calculated (italic) geometric parameters (Å, °) for compounds **5a-e**

	5a	**5b**	**5c**	**5d**	**5e**
Bond lenghts					
C12—O3	1.411(2)	1.414(2)	1.428(2)	1.405(4)	1.401(3)
	1.418	*1.418*	*1.418*	*1.419*	*1.417*
C1—O3	1.376(2)	1.372(2)	1.381(2)	1.373(3)	1.378(3)
	1.371	*1.371*	*1.371*	*1.370*	*1.372*
C4—O2	1.387(2)	1.383(2)	1.390(2)	1.386(3)	1.378(3)
	1.382	*1.381*	*1.382*	*1.383*	*1.379*
C11—O2	1.410(2)	1.396(2)	1.427(2)	1.400(3)	1.418(3)
	1.413	*1.413*	*1.412*	*1.411*	*1.423*
C11—C5’	1.488(2)	1.482(2)	1.493(2)	1.478(3)	1.480(3)
	1.492	*1.492*	*1.492*	*1.492*	*1.488*
C5’—C4’	1.336(2)	1.331(2)	1.345(2)	1.334(3)	1.334(3)
	1.354	*1.354*	*1.355*	*1.355*	*1.356*
C5’—O1	1.348(2)	1.345(2)	1.355(2)	1.346(3)	1.347(2)
	1.346	*1.346*	*1.346*	*1.347*	*1.349*
N1—O1	1.414(2)	1.415(2)	1.407(2)	1.407(3)	1.406(2)
	1.396	*1.397*	*1.395*	*1.390*	*1.386*
N1—C3’	1.309(2)	1.303(2)	1.316(2)	1.304(3)	1.306(2)
	1.315	*1.316*	*1.316*	*1.316*	*1.320*
C4’—C3’	1.422(2)	1.415(2)	1.428(2)	1.412(3)	1.413(3)
	1.433	*1.433*	*1.432*	*1.431*	*1.431*
C18—O4	-----	-----	-----	-----	1.360(3)
					1.365
C19—O4	-----	-----	-----	-----	1.420(3)
					1.422
O1N—N2	-----	-----	-----	1.228(3)	-----
				1.224	
O2N—N2	-----	------	-----	1.211(4)	-----
				1.223	
Cl—C16	-----	-----	1.746(2)	-----	-----
			1.758		
Angles					
C1—O3—C12	117.8(2)	117.8(2)	117.8(2)	118.1(2)	118.1(2)
	118.7	*118.7*	*118.7*	*118.7*	*118.6*
O3—C1—C2	115.3(2)	115.8(2)	115.6(2)	115.5(2)	115.7(2)
	115.5	*115.6*	*115.4*	*115.4*	*115.5*
O3—C1—C6	124.2(2)	123.8(2)	124.2(2)	123.9(2)	123.6(2)
	124.2	*124.2*	*124.2*	*124.2*	*124.2*
C4—O2—C11	116.8(2)	118.4(2)	117.2(1)	117.5(2)	118.2(2)
	118.4	*118.4*	*118.4*	*118.4*	*118.6*
O2—C4—C3	123.2(2)	123.5(2)	123.6(2)	123.2(2)	123.9(2)
	123.2	*123.3*	*123.3*	*123.3*	*123.6*
C5—C4—O2	116.2(2)	115.4(2)	115.8(2)	115.8(2)	115.1(2)
	116.0	*116.0*	*116.0*	*115.9*	*115.8*
O2—C11—C5’	106.9(2)	106.0(2)	106.9(2)	107.4(2)	106.6(2)
	107.6	*107.8*	*107.7*	*107.5*	*108.3*
N1—O1—C5’	108.3(2)	108.3(2)	108.7(2)	108.7(2)	108.6(2)

	108.9	*108.9*	*109.0*	*109.1*	*109.2*
N1—C3'—C4'	110.7(2)	111.5(2)	110.7(2)	111.52)	110.8(2)
	111.1	*111.1*	*111.1*	*111.2*	*110.5*
C5'—C4'—C3'	105.2(2)	104.9(2)	105.1(2)	105.1(2)	105.5(2)
	103.8	*103.9*	*103.8*	*103.7*	*104.2*
C3'—N1—O1	105.7(2)	105.2(2)	105.9(1)	105.2(2)	105.7(2)
	105.9	*105.9*	*105.9*	*105.9*	*106.4*
N1—C3'—C13	120.5(2)	120.2(2)	120.3(2)	118.7(2)	118.5(2)
	120.4	*120.5*	*120.3*	*120.1*	*119.0*
Cl1—C16—C15	-----	-----	119.1(2)	-----	-----
			119.5		
Cl1—C16—C17	-----	-----	119.6(2)	-----	-----
			119.5		
C18—O4—C19	-----	-----	-----	-----	118.6(2)
					119.0
O1N—N2—O2N	-----	-----	-----	123.8(3)	-----
				124.9	

Table 3
Hydrogen bonds (Å , °), C—H…π and π…π interactions for compounds **5a-e**
Cg1, Cg2 and Cg3 represent the centroids of the N1/O1/C3'-C5', C1–C6 and C13–C18 rings, respectively.

	D—H…A	**D—H**	**H…A**	**D…A or Cg…Cg**	**D—H…A**	**Symmetry codes**
5a	C11—H11A···O1	0.97	2.62	3.432(2)	141	-x, -y+1, -z+1
	C16—H16···O3	0.93	2.59	3.496(2)	165	1+x, -1 +y, -1+z
	C8—H8···O2	0.98	2.35	2.804(2)	107	x, y, z
	C8—H8B···Cg2	0.93	2.66	3.533(2)	150	1-x,1-y,1-z
	Cg3….Cg3			3.992(2)		2-x,-y,-z
5b	C11—H11A···O1	0.97	2.62	3.427(2)	141	2-x, 2-y, -z
	C12—H12C···O3	0.96	2.68	3.555(2)	152	-x, 2-y, 1-z
	C8—H8···O2	0.98	2.32	2.767(2)	107	x, y, z
	C11—H11B···Cg2	0.97	2.91	3.718(2)	142	1 - x, 2 - y, -z
	C10—H10B···Cg3	0.96	2.88	3.740(2)	150	2 - x, 1 - y, - z
5c	C11—H11A···O3	0.97	2.60	3.454(2)	146	-x, 1-y, 1-z
	C18—H18···N1	0.93	2.55	2.856(3)	100	x, y, z
	C8—H8···O2	0.98	2.33	2.783(2)	108	x, y, z
	C12—H12C···Cg2	0.96	2.74	3.577(2)	146	1–x, 1-y, 1–z
	Cg1···Cg3			3.970(2)		-x,-y, 2-z
	Cg3···Cg3			3.811(2)		-1-x,-y, 2-z
5d	C11—H11A···O1N	0.97	2.54	3.488(3)	165	1+x, 1+y, z
	C8—H8···O2	0.98	2.30	2.778(3)	109	x, y, z
	C10—H10B···Cg3	0.96	2.92	3.829(4)	158	1+x, y, z
	Cg1···Cg2			3.960(2)		-1+x, y, z
	Cg3···Cg3			3.836(2)		-1-x, -y, 1-z
5e	C11—H11A···O4	0.97	2.49	3.448(3)	170	1-x,-y, 2-z
	C16—H16···O3	0.93	2.57	3.418(4)	152	-1+x,1+y, 1+z
	C8—H8···O2	0.98	2.34	2.741(3)	103	x, y, z
	C19—H19A···Cg3	0.96	2.99	3.759(4)	138	-x, 1-y, 2-z

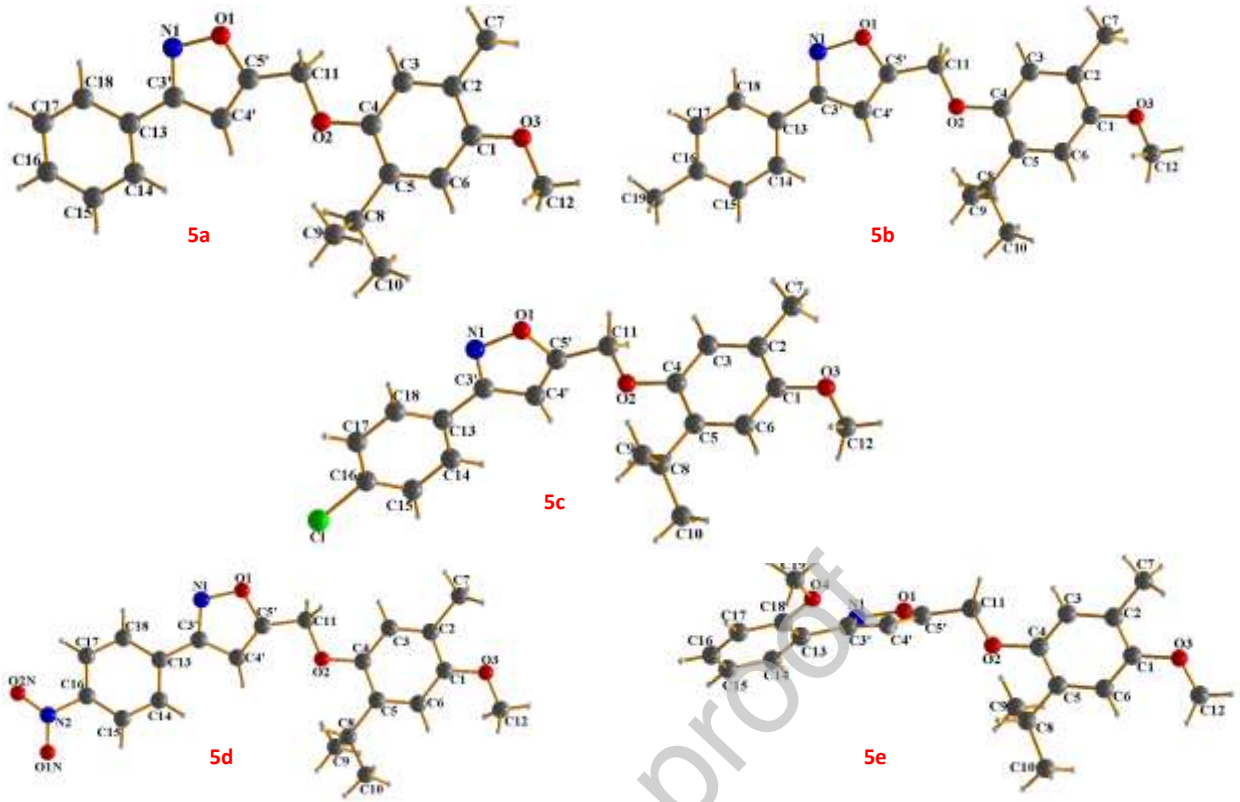


Figure 1: The structures of **5a-e** showing the atom numbering with ellipsoids drawn at the 50% probability level

In the crystal structure of **5a**, the dihedral angle between isoxazole ring and two phenyl (C1–C6) and (C13-C18) are 16.04(1) and 11.69(1)° respectively, while that between the two aromatic six-membered rings is 26.63(8)°. Additional intermolecular C16–H16···O3 and C11–H11A···O1 hydrogen bonds link groups of four molecules (Fig. 2a) and generate sheets parallel to a plane. An intermolecular C–H···π interaction between the H at C11 and the C1-C6 aromatic ring of the neighboring molecule with H···π distance of 2.66 Å interconnects the molecular chains together. These contacts are strongly supported by extensive π···π contacts between the adjacent C13-C18 rings, Fig. 2b, with centroid to centroid distances Cg3···Cg3 = 3.992(2) Å (Table 3). These combinations of dimers formation and π- stacking interactions form layers parallel to (110) (Fig. 2c).

Another significant feature of the packing in **5a** is a series of strong, inversion related C–H···π interactions and π···π contacts to generate sheets of molecules parallel to the bc plane, Fig. 2b. The C11–H11A···O1 contacts serve to connect these sheets (Fig 2c).

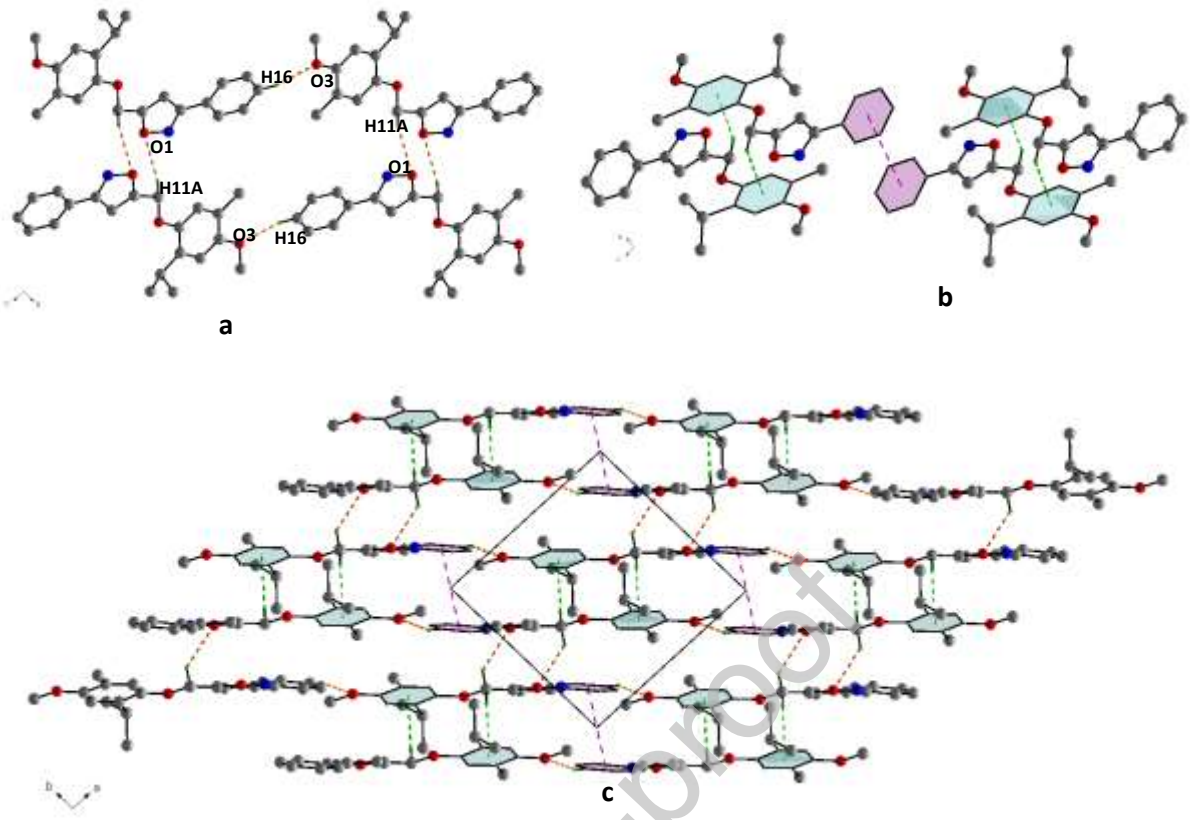


Figure 2: (a) Detail of the intermolecular C–H···O hydrogen bonds (orange dashed lines), **(b)** C–H···π and π···π contacts are shown as green and purple dashed lines, respectively and (c) crystal packing for **5a** viewed along the *c* axis.

In the title compound **5b**, the dihedral angles between the isoxazole ring C3'–C5'/N1/ O1 and two phenyl (C1–C6) and (C13-C18) are 8.34(8) and 26.27(8)° respectively while that between the two aromatic six-membered rings is 34.59(8)°. For **5b** crystal packing is again dominated by C—H···O and C—H···π contacts. A centrosymmetric dimers $R_2^2(8)$ formed by C11—H11A···O1 hydrogen bond are connected to another via C—H···π (C11—H11B···Cg2) interactions, where Cg2 is the centroid of the ring C1—C6 forming chains along a-axis (Fig. 3b). These chains are related by C—H···π (C10—H10B···Cg3) interactions, where Cg3 is the centroid of the ring C13—C18, forming sheets parallel to ab plane (Fig. 3c). Adjacent layers are interconnected by hydrogen bond C12—H12C···O3 (Fig. 3d).

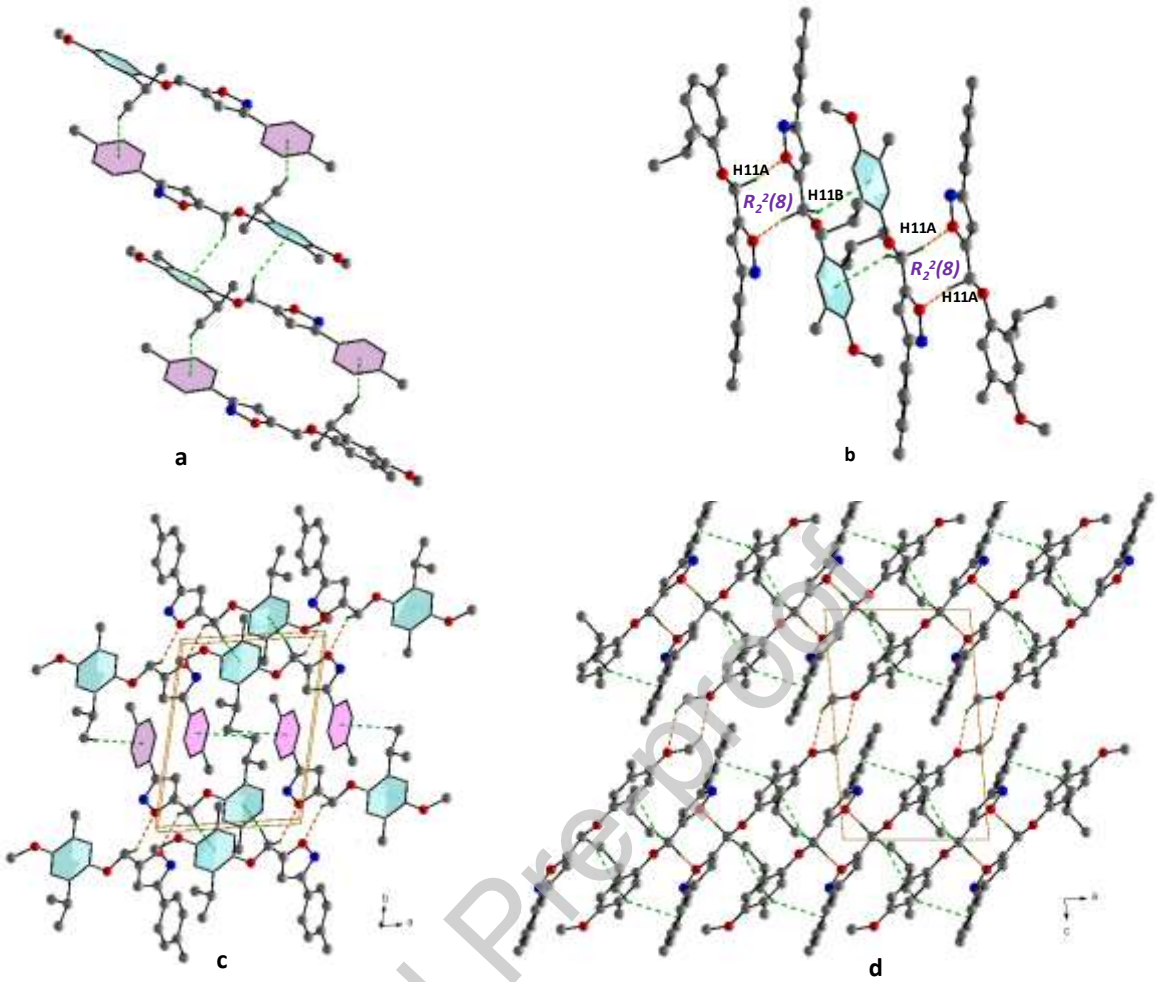


Figure 3: (a) The molecular chain generated by intermolecular C—H···π interactions, (b) C–H···O interconnected by C–H···π interactions, (c) Overall packing for **5b** viewed along the *c* axis showing C–H···O and C–H···π interactions are shown as orange and green dashed lines, respectively and (d) the molecular sheet assembled by intermolecular C—H···O and C—H···π interactions

In crystal **5c**, the dihedral angles between C3'–C5'/N1/ O1 ring and two phenyl (C1–C6) and (C13-C18) are 64.59(8) and 10.31(8)° respectively, while that between the two aromatic six-membered rings is 68.51(7)°. C11–H11A···O3 hydrogen bonds form inversion dimers generating $R_2^2(16)$. An intermolecular C–H···π interaction between the H at C12 and the C1-C6 aromatic ring of the neighboring molecule with H···π distance of 2.74 Å connects the adjacent dimmers forming chains along the a-axis direction. The chains are linked by slipped parallel π···π interactions, between C3'–C5'/N1/ O1 (Cg1) and C13-C18 (Cg3) rings, with centroid to centroid distances Cg1…Cg3 = 3.970(2) Å and Cg3···Cg3 = 3.811(2) Å (Table 3), forming slabs parallel to the ac plane (Fig. 4).

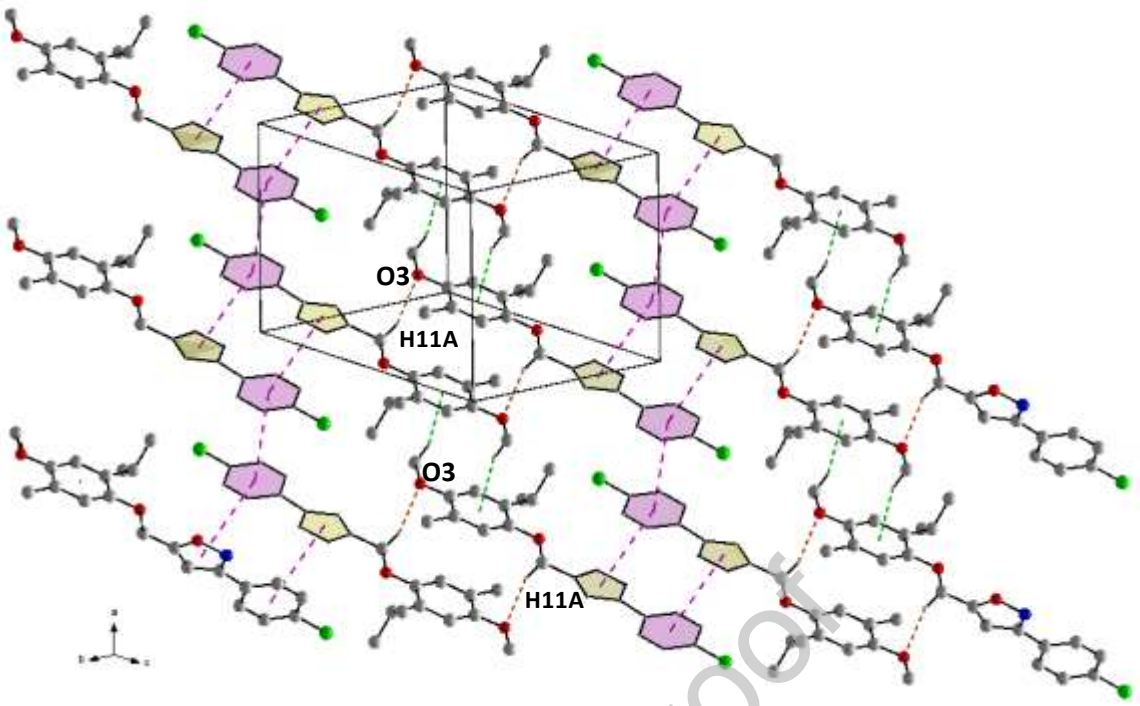


Figure 4: Overall packing for **5c** showing C–H···O, C–H···π and π···π interactions as dashed lines

In the crystal of compound **5d**, The dihedral angles between C3'–C5'/N1/O1 ring and two phenyl (C1–C6) and (C13-C18) are 10.55(12) and 21.01(12)° respectively. In the crystal, the molecules are linked together by a C—H···π -phenyl (C13-C18) interaction (Table 1), with an H…centroid distance of 2.92 Å forming chains propagating along the a direction. The cohesion of the crystal structure is enhanced by two π – π stacking interactions between the rings, with centroid– centroid distances in the range 3.836(2) to 3.960(2) Å (Table 3 and Fig. 5), so the chains are connected via C—H···O hydrogen bond to form of a supramolecular three-dimensional structure (Fig. 5).

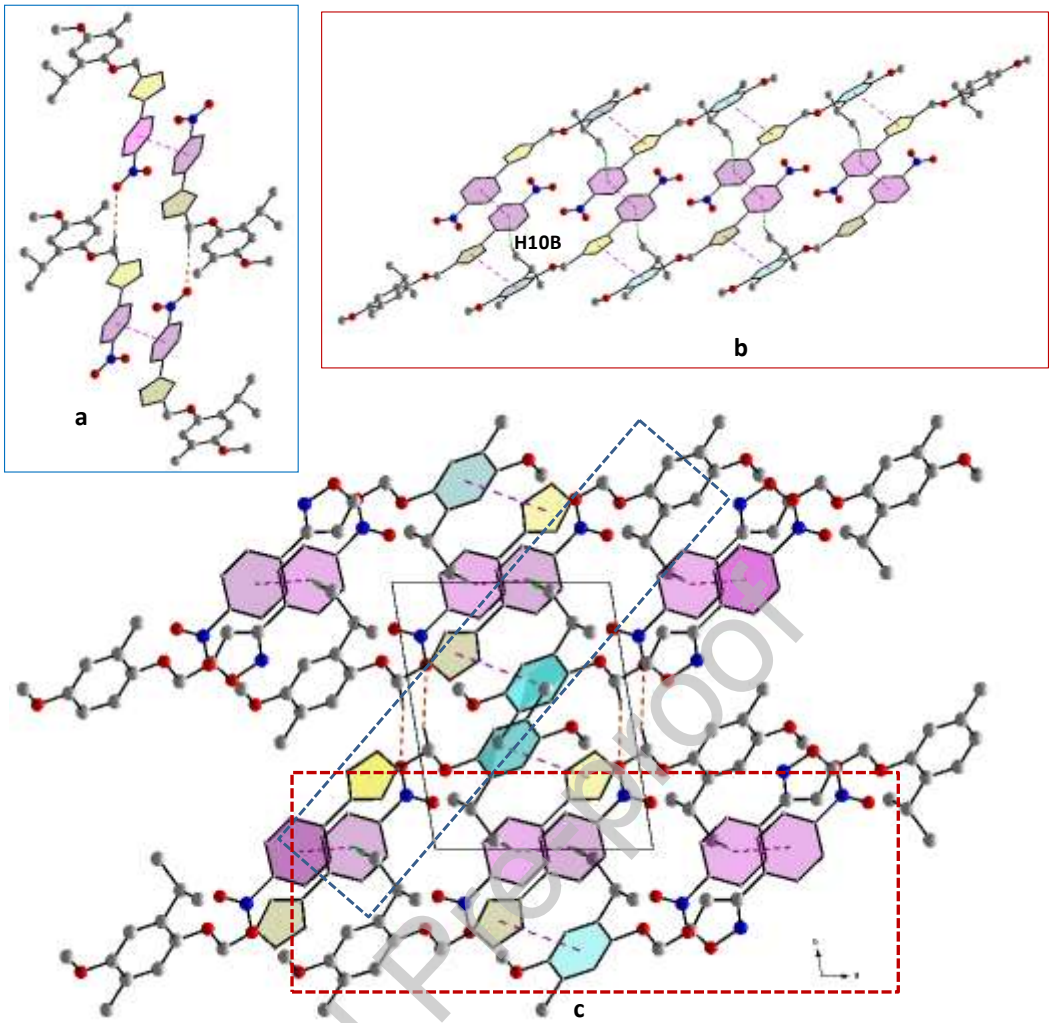


Figure 5: (a) The molecular chain generated by intermolecular C—H···O interactions, (b) C–H···π and π···π contacts are shown as green and purple dashed lines, respectively and (c) crystal packing for **5d** viewed along the *c* axis

Due to the position of the methoxy group in position C18 in the **5e** compound, its structure is different from that of the other compounds, the dihedral angle between the isoxazole and the attached ring is widely different with a value of 34.78(12)°. Moreover, the dihedral angle between the isoxazole and the C1—C6 cycle is 71.25(11)°, while that between the two aromatic six-membered rings is 39.72(11)°. In the crystal structure of **5e**, C11—H11A···O4 hydrogen bonds form inversion dimers with $R_2^2(16)$ ring motifs. Adjacent dimers are linked by C16—H16···O3 contacts that combine to form $R_2^2(26)$ rings generating corrugated chains of molecules running approximately parallel to (011), Fig. 6a. On the other hand, the dimers $R_2^2(16)$ are linked by slipped parallel C—H···π (C19—H19A···Cg3) interactions, where Cg3 is the centroid of the ring C13—C18, forming chains along the *b* direction (Fig. 6b). The packing crystal is shown in Fig. 6c.

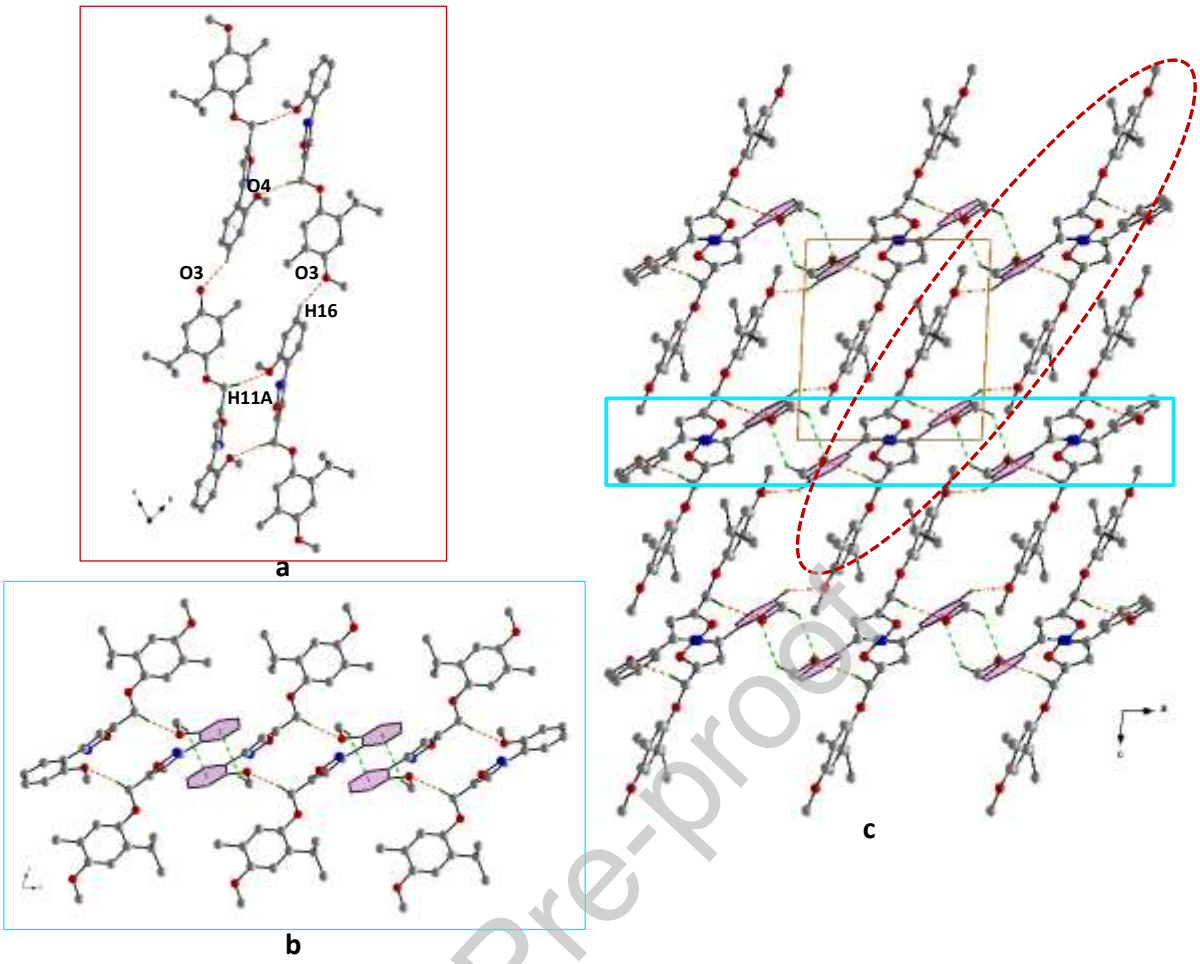


Figure 6: (a) Crystal packing of compound **5e** with indication of the hydrogen-bonding, (b) C—H···O and (c) C—H···O and C—H···π interaction

3.3 Frontier Molecular Orbitals (FMOs)

All molecular calculations were performed in the gas phase using Density Functional Theory (DFT) using the B3LYP/6-311g(d, p) exchange correlation functional. A comparison of bond lengths and bond angles in the crystal to those from the DFT calculation is listed in Table 2, while with B3LYP/6-311++g(d,p)/6-311++g are reported in Table S1. The differences between calculated and experimental bond lengths and angles are within a few Angstroms and degrees, respectively, when compared to the experimental parameters, which indicate that our calculations are acceptable. This sensitive gap is attributed to the difference between the solid phase and gas phase model. The DFT calculations provide some important information on the reactivity and site selectivity of the molecular framework. The energy of the HOMO is directly related to the ionization potential, while the energy of LUMO is related to electron affinity and the energy gap between the HOMO and LUMO characterizes the molecular chemical stability. The energy levels of the HOMO and LUMO for all compounds are

illustrated in Fig. 7. Red and green color distributions represent positive and negative phase in molecular orbital wave function, respectively. The electron density of HOMO in the all compounds is concentrated on p-methoxythymol, the isodensity in the LUMO is localized on the R-phenylisoxazole. The energies of HOMO and LUMO, energy gap, electronegativity (χ), chemical hardness (η), global softness (ξ) and electrophilicity (ψ) index are calculated and presented in Table 4. Furthermore, the high values of the electrophilicity index 6.185 eV compared to the low values of chemical potential (4.284 eV) for **5d** prove its electrophilic character. The dipole moment (D) of compound **5d** is greater than other studied compound, therefore we can say that this compound is more reactive that other compound. On the other side, the chemical potential of **5d** (Table 4) is smaller compared to other compound; therefore **5d** behaves as an acceptor of electrons. The chemical hardness for **5d** is smaller than those of other compounds, indicating that the electrons are attracted from compound **5d.** It is clear that the value of ΔE for the different studied molecules increases in the order **5d** < **5c** < **5e** < **5a** < **5b**; this indicates that compound **5d** exhibit higher chemical reactivity attributed probably to the presence of nitro group related to phenyl group attached to isoxazole ring.

It is noted from Table S2 and Fig. S13, the estimated values of quantum chemical parameters from all compounds with the three basis set are having a similar trend. For example, 6-311g(d,p) basis set shows the minimum dipole moment and the basis set of 6-311++g gives maximum dipole moment value.

Table 4: HOMO–LUMO energies and values of quantum chemical parameters calculated by B3LYP/6-311G (d,p)

property	**Values**				
	5a	**5b**	**5c**	**5d**	**5e**
E_T (eV)	-29776.600	-30846.320	-42278.312	-35340.515	-32892.405
E_{HOMO} (eV)	-5.612	-5.591	-5.671	-5.765	-5.402
E_{LUMO} (eV)	-1.269	-1.171	-1.527	-2.803	-1.213
E_{HOMO-1} (eV)	-6.658	-6.436	-6.710	-6.934	-6.262
E_{LUMO+1} (eV)	-0.469	-0.448	-0.829	-1.303	-0.165
$\Delta E_{(LUMO-HOMO)}$ (eV)	**4.343**	**4.420**	**4.144**	**2.962**	**4.189**
$\Delta E_{(LUMO+1-HOMO-1)}$ (eV)	6.189	5.988	5.881	5.631	6.097
Global hardness (η)	2.172	2.210	2.072	1.481	2.094
Softness (ξ)	0.230	0.226	0.241	0.337	0.239
Chemical potential (μ)	3.441	3.381	3.599	4.284	3.308
Electrophilicity (ψ)	2.723	2.583	3.122	6.185	2.614
Electronegativity (χ)	-3.441	-3.381	-3.599	-4.284	-3.308
Dipole moment (D)	3.405	3.145	4.304	6.888	4.083

$\eta=1/2[E_{LUMO}-E_{HOMO}]$, $\xi=1/2\eta$, $\mu= -[1/2(E_{LUMO}+E_{HOMO})]$, $\psi= \mu^2/2\eta$, $\chi = -\mu$

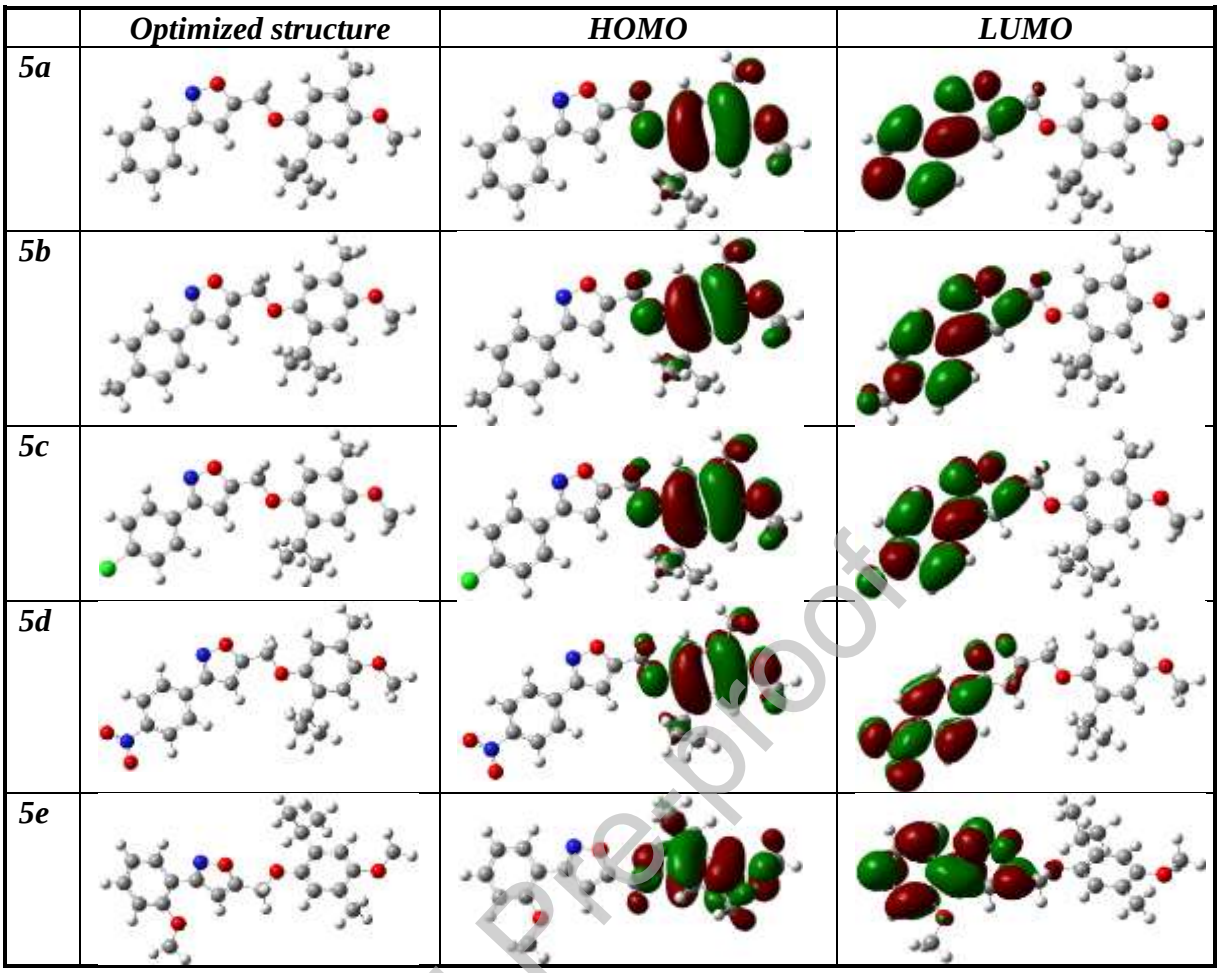


Figure 7: Optimized structure and 3D plots of frontier molecular orbital of **5a-e**

3.4 Molecular Properties

3.4.1 Mulliken population analysis

The Mulliken charge values were calculated using B3LYP functional with 6-311G(d,p), 6-311++G(d,p) and 6-311++G basis set for the all structures. The color range in the scale of positive and negative charge for Milliken atomic charges of **5a-e** with 6-311g(d,p) is shown in Fig. 8. While the Mulliken charge obtained by 311++G(d,p) and 6-311++G basis set are reported in Table S3. The C1/C4/C11/C5'/C4' atoms of the all molecules exhibit positive charge while the other carbon has negative charges, the C16 atom has a positive charge when it is attached to the nitro group. Moreover, all the hydrogen atoms have a net positive charge. Oxygen O2 and O3 have a high negative charge value which imposes a positive charge to C4 and C1 for all compounds, respectively. The Mulliken atomic charge of all compounds shows that the nitrogen atoms N1 has negative charge value which imposes a positive charge to a carbon atom bonded, while the nitrogen atom N2 posses positive charge which was imposed by oxygen atoms O1N and O2N in compound **5d**.

The charge changes with basis set presumably occurs due to polarization. For example, the charges of N1 atom are a range -0.152 to -0.161e- for B3LYP/6-311g(d,p), -0.378 to -0.487e- for B3LYP/6-311++g(d,p) and -0.477 to -0.576e- for B3LYP/6-311++g (Table S3).
The charge of O1 is negative B3LYP with 6-311g(d,p) basis set, however in both B3LYP/6-311++g and B3LYP/6-311++g(d,p) this charge is positive.

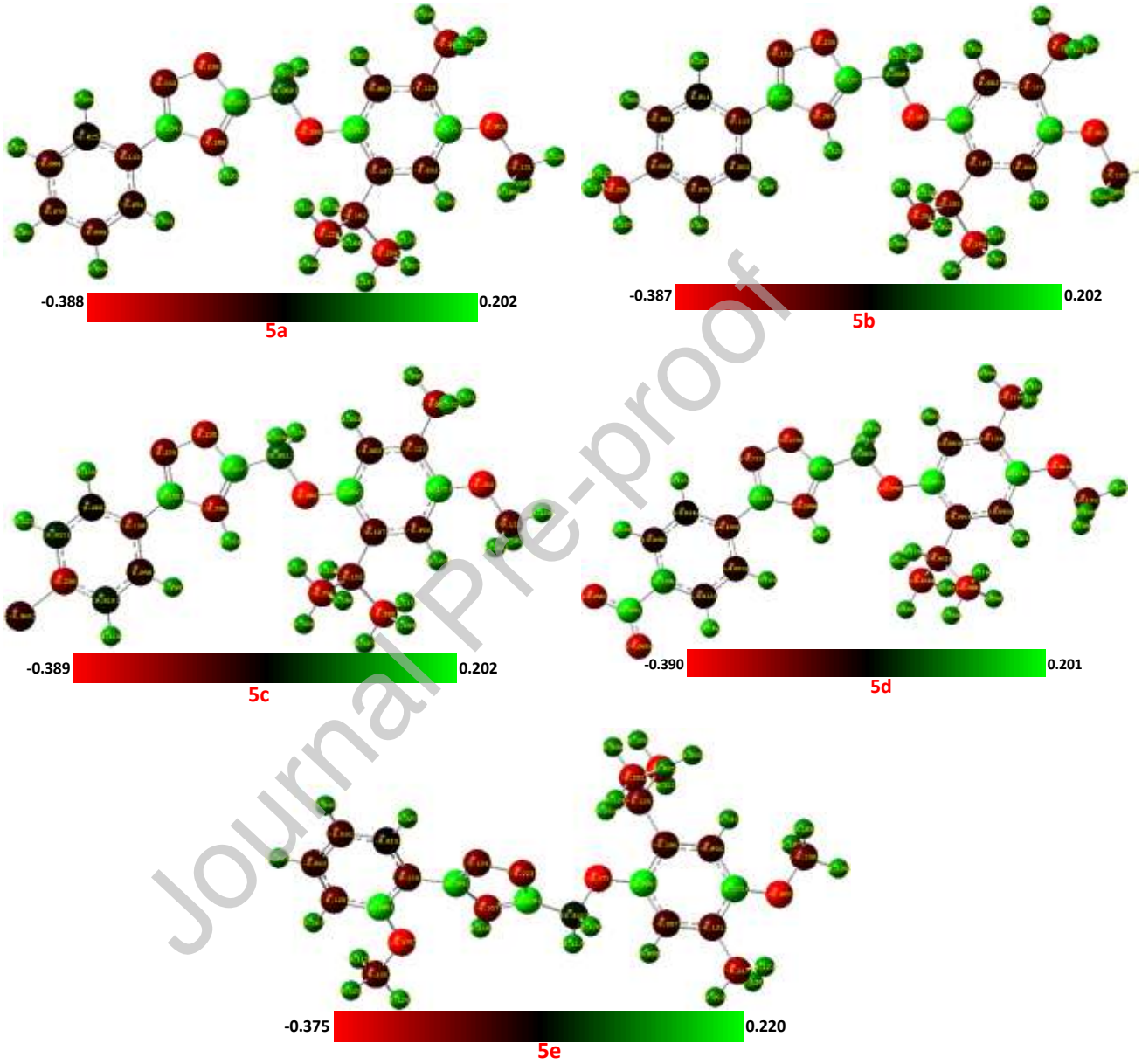


Figure 8: Mulliken atomic charge distribution of **5a-e**

3.4.2 Molecular Electrostatic Potentiel (MEP)

The electrostatic potential shows static distributions of charge on a molecule. In order to study the site of **5a-e** available for electrophilic and nuclephilic attack, the MEP is plot of electrostatic potential mapped onto the constant electron density surface. Molecular

electrostatic potential of **5a-e** using B3LYP/6-311G (d, p) optimized geometry is computed, contour and surface map is shown in Fig. 9b. The negative electrostatic potential is indicated by red regions, the blue region indicates the positive electrostatic potential, the yellow region reveals the slightly rich electron and the green region shows neutral potential.

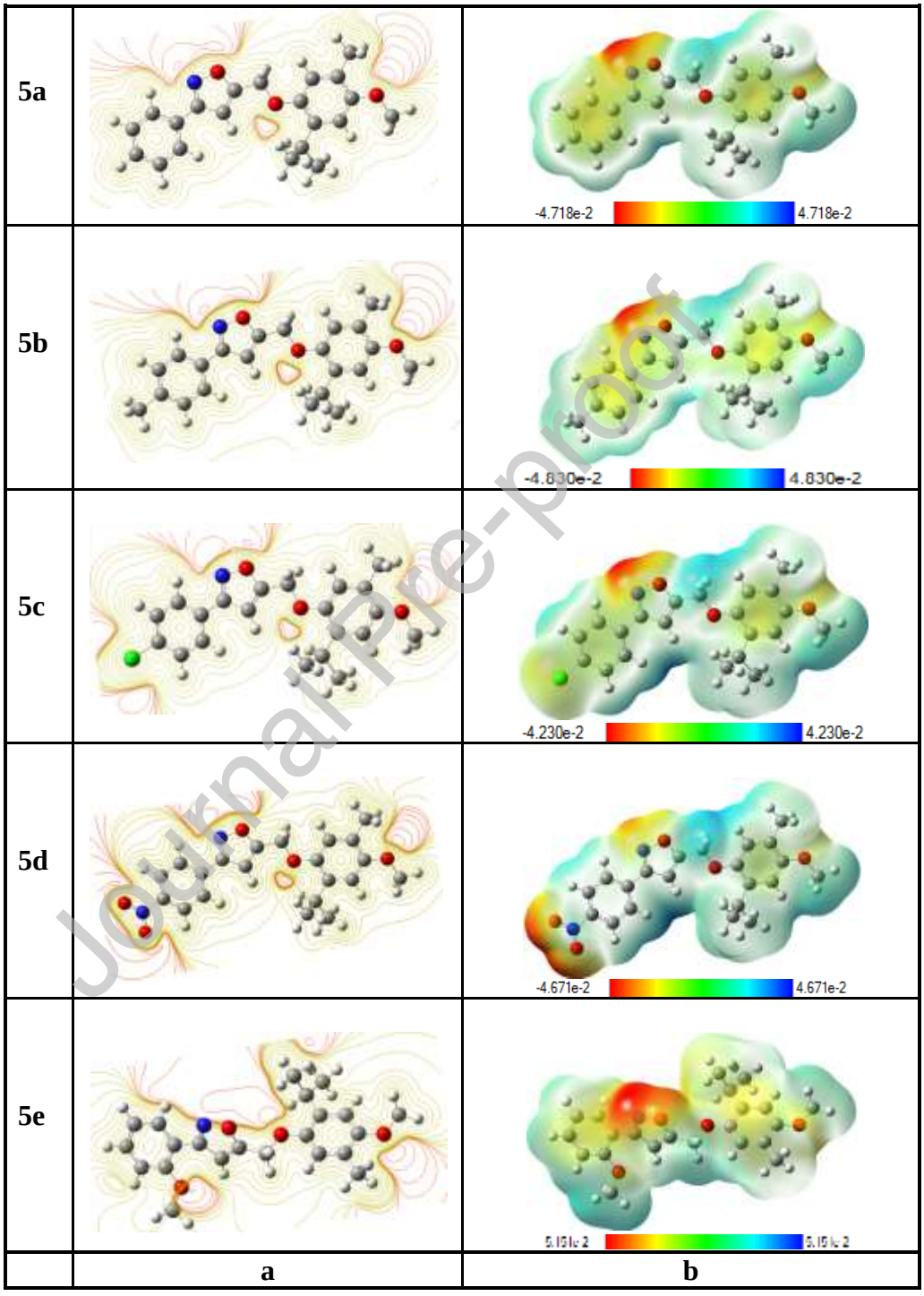


Figure 9: (a) the contour map of electrostatic potential and (b) the total electron density mapped with electrostatic potential of **5a-e** at an isosurface value of 0.020 a.u. and an isodensity of 0.0004 a.u.

For isoxazole compounds, the total electron density surface mapped with electrostatic potential indicates the presence of a high negative charge on N—O units of the isoxazole ring which undergoes electrophilic reactivity. While the positive region of the MEP map over the methoxy hydrogen atoms and all other hydrogen atoms indicates that these sites are susceptible sites for nucleophilic attack. In addition, the region near to Cl in **5c** compound is positive due to the fact that C1 atom is surrounded by the electropositive hydrogen atoms (H15 = 0.119e and H17 = 0.122e). MEP for **5d** compound showed the presence of localized most electronegative potential region (in red color) near the oxygen atoms of the nitro (NO_2) group in the para position of the phenyl ring. The MEP results are supported by the electrostatic potential contour map showing the lines isosurface in where the red lines refer to the strong electron-withdrawing atoms such as in N—O units of the isoxazole ring in all compounds and in NO_2 group for **5d**. Analyses of the MEP surface of these compounds represent the availability of electrons for possible interaction with another group of atoms.

3.5 Hirshfeld surface analysis

The nature of intermolecular interactions in the all compounds **5a-e** has been computed by CrystalExplorer17.5, using Hirshfeld surface analysis mapped over d_{norm}, shape-index and two-dimensional fingerprint plots. The red spots highlight the interatomic contacts included in C—H···O hydrogen bonding in all compounds (Fig. 10a).

Fig. 11a shows the two-dimensional fingerprint plot for the sum of the contacts contributing to the Hirshfeld surface represented in normal mode. The percentage contributions of the various interatomic contacts to the Hirshfeld surfaces are summarized in Table 5. The most important interaction is H···H contributing 47.5-61.2% to the overall all compounds to stabilize the crystal packing, which is reflected in Fig. 11b as widely scattered points of high density due to the large hydrogen content of the molecules with the small split tips at $d_e \approx d_i \approx$ 1 - 1.2 Å.The presence of C—H···π interactions in the all crystals is indicated by the pair of characteristic wings in the fingerprint plot delineated into C···H/H···C (Figs. 10c and 11*c*). The C···H/H···C interactions are represented by the spikes at the bottom right and left ($d_e + d_i \approx$ 2.65 – 2.85Å). The percentage of C···H/H···C increases from the compound **5d** (12.7%) to **5b** (21.9%) is in agreement with the substituted group. The O···H/H···O contacts (8.6 (**5a**)-24.4% (**5d**)) (Fig. 11d) between the oxygen atoms inside the surface and the hydrogen atoms outside the surface and vice versa, $d_e + d_i \approx$ 2.35 – 2.50Å are shown two symmetrical points on the top, bottom left and right, which are characteristic of C—H···O hydrogen bond. Also

worth mentioning is the contribution of C···C contacts ranging from 1.0 to 5.2% in all structures. They are shown on the fingerprint plots as areas on the diagonal at $d_e = d_i \approx 1.7–2.2$ Å (Fig. 11f). These contacts correspond to the presence of the above mentioned $\pi\cdots\pi$ stacking interactions in the crystal structures **5a**, **5c** and **5d**. π—π interaction is indicated by adjacent red and blue triangles in the shape-index map (Fig. 10b).

In **5c**, the structure is further characterized by a significant proportion of H···Cl contacts, comprising about 9.6 % of the molecular surface. The shortest H···Cl contacts are shown on the fingerprint plots as a pair of spikes at $d_e + d_i \approx 3.0$ Å (Fig. 11g).

Table 5: Percentage contributions of interatomic contacts to the Hirshfeld surface for compounds **5a-e**

Contact type	**5a**	**5b**	**5c**	**5d**	**5e**
H···H	59.6	61.2	50.2	47.5	59.1
C···H/H···C	21.5	21.9	16.6	12.7	20.6
O···H/H···O	8.6	9.5	9.9	24.4	12.4
Cl···H/H···Cl	-----	-----	9.6	-----	-----
N···H/H···N	5.3	5.1	4.4	4.1	5.8
C···C	2.1	1.0	3.4	5.2	1.3
C···N/N···C	-----	-----	1.4	2.0	-----
C···O/O···C	1.6	0.6	1.4	2.0	0.7
C···Cl/Cl···C	-----	-----	1.4	-----	-----
O···O	1.1	-----	0.1	1.0	-----
Cl···Cl	-----	-----	0.8	-----	-----
N···N	-----	-----	-----	0.4	-----
N···O/O···N	0.3	0.6	-----	0.7	-----
O···Cl/Cl···O	-----	-----	0.7	-----	-----

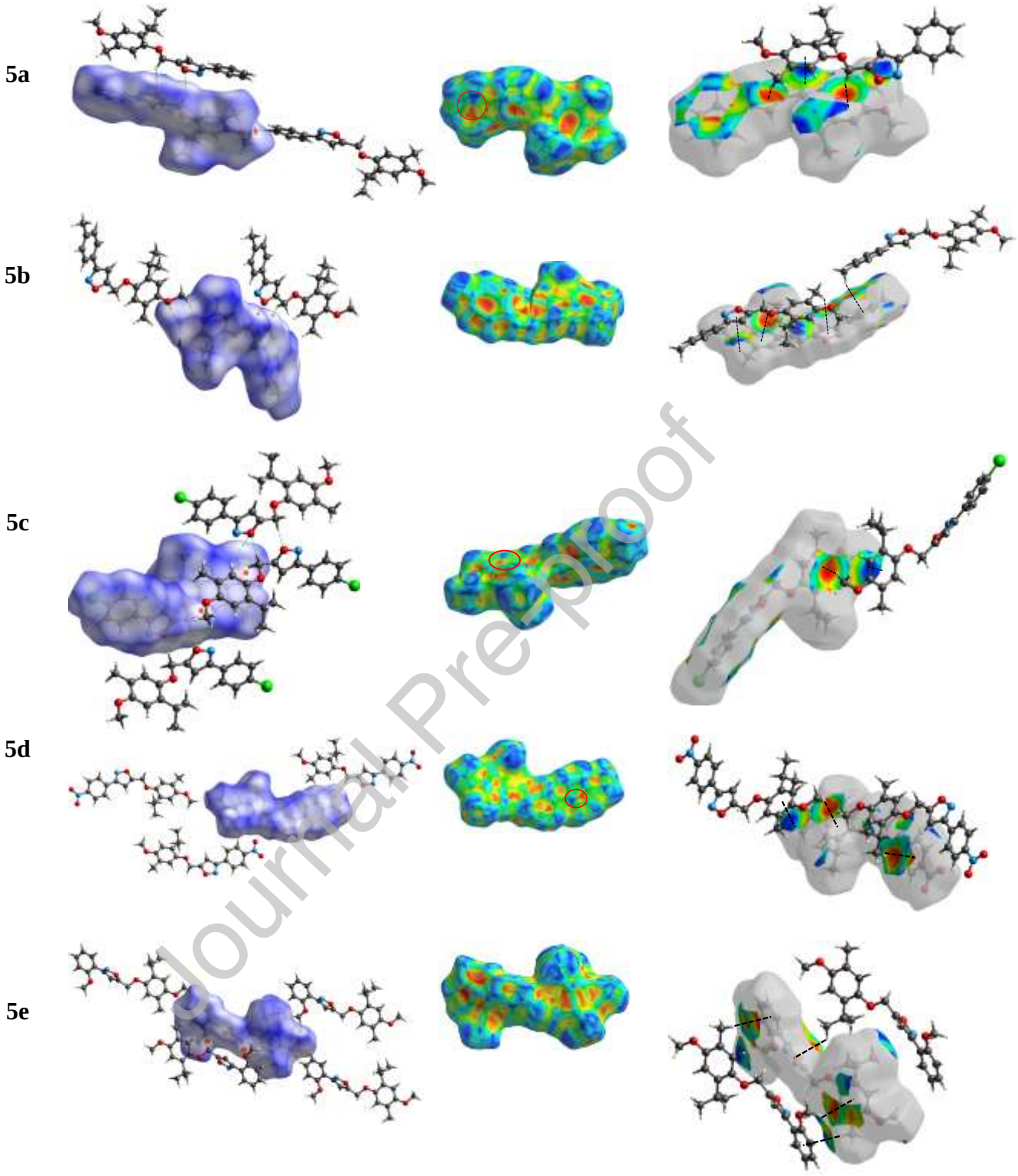


Figure 10: View of the three-dimensional Hirshfeld surface of **5a-e** plotted over (a) d_{norm}**,** (b) shape-index and (c) shape-indexed showing C—H···π interactions through black dashed lines

5a **5b** **5c** **5d** **5e**

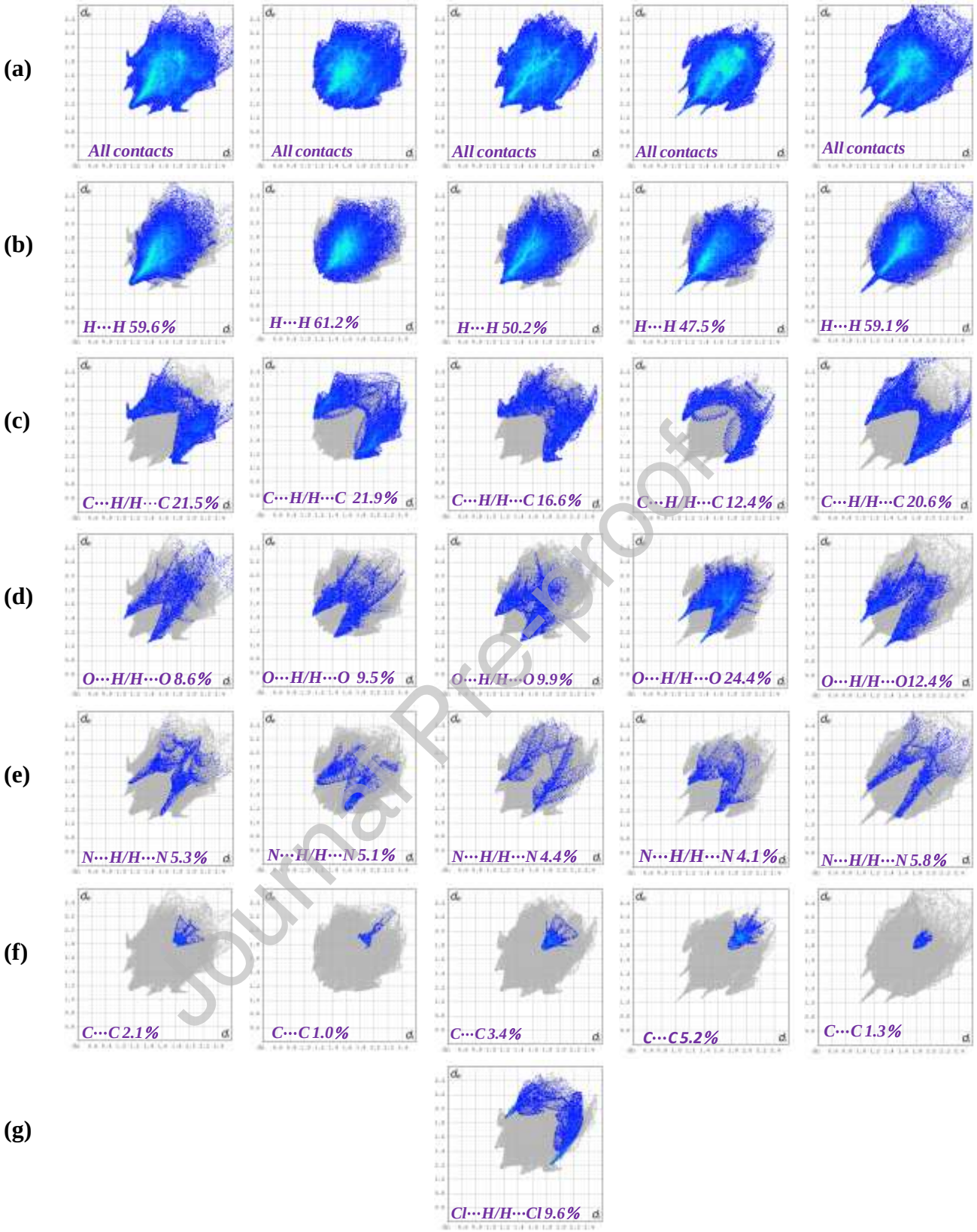


Figure 11: Two-dimensional fingerprint plots and relative contributions of various interactions to the Hirshfeld surface of the compounds **5a-e** corresponding to (a) All interactions, (b) H···H, (c) C···H/H···C, (d) H···O/O···H (e), N···H/H···N, (f) C···C and Cl···H/H···Cl (**5c**)

Conclusion:

We have synthesized new p-methoxythymol derivatives **5a-e** by simple and convenient methodology and characterized them by IR, ^{1}H, ^{13}C NMR and X-ray single crystal diffraction. Overall, we believe that the novel series of substituted isoxazole should be considered as an important advance in medicinal and pharmaceutical chemistry. X-ray crystallographic studies for **5a-e** display intermolecular C—H···O hydrogen bonding and C—H···π interactions, forming layers in the crystal lattice. The results of HS analysis indicate that the major interactions were found for H···H interaction with contributions of 61.2, 59.6, 59.1, 50.2 and 47.5% of the total HS area in **5b**, **5a**, **5e**, **5c** and **5d** products, respectively. Frontier molecular orbital analysis and molecular electrostatic potential map of five isoxazole derivatives has been studied using DFT calculations. The Molecular electrostatic potential analysis gives the information about intermolecular interaction regions. The results indicate that compound **5d**, with smaller LUMO/HOMO gap, more reactive than other compounds. As a result, it is expected that this research will be beneficial for the design, synthesis and various applications of new isoxazole-based compounds.

Supplementary material

These data include NMR spectra for all new compounds **5a**-e. CCDC 2022526, CCDC 2022527, CCDC 2022528, CCDC 2022529, CCDC 2022534 for **5a**, **5b**, **5c**, **5d** and **5e**, respectively contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif, or by emailing data_request@ccdc.cam. ac.uk, or by contacting the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

Credit authorship contribution statement

Yassine Laamari : Performing manipulations

Aziz Auhmani : Conceptualization, Supervision, Writing - review & editing

Mohamed Saadi : Methodology, Software.

Lahcen El Ammari : Methodology, Software, Investigation

Mostafa Khouili: *Sample Tracking*

My Youssef Ait Itto: Conceptualization, Supervision

Abdelwahed Auhmani: Product and material order tracking

El Mostafa Ketatni: Software, Validation, Writing - review

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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References

[1] F. M. Cordero, D. Giomi, and L. Lascialfari. Five-Membered Ring Systems: With O and N Atoms, Progress in Heterocyclic Chemistry, 30 (2018) 279–309.

[2] A. Oubella, M. Y. Ait Itto, A. Auhmani, A. Riahi, A. Robert, J.-C. Daran, H. Morjani, C. A. Parish and M. Esseffar. Diastereoselective synthesis and cytotoxic evaluation of new isoxazoles and pyrazoles with monoterpenic skeleton. Journal of Molecular Structure 1198 (2019) 126924

[3] C. M. Sun, L.-G. Lin, H.-J. Yu, C.-Y. Cheng, Y.-C. Tsai, C.-W. Chu, Y.-H. Din, Y.-P. Chau and M.-J. Don. Synthesis and cytotoxic activities of 4,5-diarylisoxazoles Bioorganic Med. Chem. Lett., 17(4) (2007) 1078–1081.

[4] N. Ganji, A. Rambabu, N. Vamsikrishna, S. Daravath, Shivaraj. Copper(II) complexes with isoxazole Schiff bases: Synthesis, spectroscopic investigation, DNA binding and

nuclease activities, antioxidant and antimicrobial studies. J. Mol. Struct. 1173 (2018) 173–182.

[5] S. Kankala, R. K. Kankala, P. Gundepaka, N. Thota, S. Nerella, M. R. Gangula, H. Guguloth, M. Kagga, R. Vadde, C. S. Vasam. Regioselective synthesis of isoxazole-mercaptobenzimidazole hybrids and their in vivo analgesic and anti-inflammatory activity studies.Bioorganic Med. Chem. Lett. 23 (2013) 1306–1309.

[6] V. A. Makarov, O. B. Riabova, V. G. Granik, P. Wutzler, M. Schmidtke. Novel [(biphenyloxy)propyl]isoxazole derivatives for inhibition of human rhinovirus 2 and coxsackievirus B3 replication. J. Antimicrob. Chemother. 55 (2005) 483–488.

[7] B. V. Kendre, M. G. Landge, S. R. Bhusare. Synthesis and biological evaluation of some novel pyrazole, isoxazole, benzoxazepine, benzothiazepine and benzodiazepine derivatives bearing an aryl sulfonate moiety as antimicrobial and anti-inflammatory agents. Arab. J. Chem. 12 (2019) 2091–2097.

[8] P. Mondal, S. Jana, A. Balaji, R. Ramakrishna, L. Kanthal. Synthesis of some new isoxazoline derivatives of chalconised indoline 2-one as a potential analgesic, antibacterial and anthelmimtic agents. J. Young Pharm. 4 (2012) 38–41.

[9] S. Tu, X. Zhang, Z. Han, X. Cao, S. Wu, S. Yan, W. Hao, G. Zhang, N. Ma. Synthesis of Isoxazolo [5 , 4- b] pyridines by Microwave-Assisted Multi-Component Reactions in Water.. *Journal of combinatorial chemistry* .11(3) (2009): 428-432.

[10] A. Kumar, R. A. Maurya, S. Sharma, P. Ahmad, A. B. Singh, A. K. Tamrakar, A. K. Srivastava. Design and synthesis of 3,5-diarylisoxazole derivatives as novel class of anti-hyperglycemic and lipid lowering agents. Bioorganic Med. Chem. 17 (2009) 5285–5292.

[11] K. Choi, M. Siegel, J. L. Piper, L. Yuan, E. Cho, P. Strnad, B. Omary, K. M. Rich, C. Khosla. Chemistry and biology of dihydroisoxazole derivatives: Selective inhibitors of human transglutaminase 2. Chem. Biol. 12 (2005) 469–475.

[12] C. B. Vicentini, C. Romagnoli, S. Manfredini, D. Rossi, D. Mares. Pyrazolo[3,4-c]isothiazole and isothiazolo[4,3-d]isoxazole derivatives as antifungal agents, Pharm.

Biol. 49 (2011) 545–552.

[13] Z. Li, N. Liu, J. Tu, C. Ji, G. Han, Y. Wang, C. Sheng. Discovery of novel simplified isoxazole derivatives of sampangine as potent anti-cryptococcal agents. Bioorganic Med. Chem. 27 (2019) 832–840.

[14] Y. Zhou, Y. Chen, W. Miao, J. Qu. Synthesis, structures, and herbicidal activity of isoxazole derivatives. J. Heterocycl. Chem. 47 (2010) 1310–1316.

[15] F. Ye, Y. Zhai, T. Kang, S. L. Wu, J. J. Li, S. Gao, L. X. Zhao, Y. Fu. Rational design, synthesis and structure-activity relationship of novel substituted oxazole isoxazole carboxamides as herbicide safener. Pestic. Biochem. Physiol. 157 (2019) 60–68.

[16] A. Srinivas, A. Nagaraj, C. S. Reddy. Synthesis and in vitro study of methylene-bis-tetrahydro[1,3]thiazolo[4,5-c]isoxazoles as potential nematicidal agents. Eur. J. Med. Chem. 45 (2010) 2353–2358.

[17] R. Sun, Y. Li, L. Xiong, Y. Liu, Q. Wang. Design, synthesis, and insecticidal evaluation of new benzoylureas containing isoxazoline and isoxazole group. J. Agric. Food Chem. 59 (2011) 4851–4859.

[18] A. A. N. de Souza, V. F. Xavier, G. S. Coelho, P. A. Sales Junior, A. J. Romanha, S. M. F. Murta, C. M. Carneiro and J. G. Taylor. Synthesis of 3, 5-diarylisoxazole derivatives and evaluation of in vitro trypanocidal activity. Journal of the Brazilian Chemical Society, 29(2) (2018) 269-277.

[19] K. Habbadi, T. Meyer, L. Vial, V. Gaillard, R. Benkirane, A. Benbouazza, I. Kerzaon, E. H. Achbani, C. Lavire. Essential oils of Origanum compactum and Thymus vulgaris exert a protective effect against the phytopathogen Allorhizobium vitis. Environ. Sci. Pollut. Res. 25 (2018) 29943–29952.

[20] M. Yousefzadi, A. Riahi-Madvar, J. Hadian, F. Rezaee, R. Rafiee. In vitro cytotoxic and antimicrobial activity of essential oil from Satureja sahendica. Toxicol. Environ. Chem. 94 (2012) 1735–1745.

[21] P. López, C. Sánchez, R. Batlle, C. Nerín. Vapor-phase activities of cinnamon, thyme, and oregano essential oils and key constituents against foodborne microorganisms. J.

Agric. Food Chem. 55 (2007) 4348–4356.

[22] L. Rubió, M. J. Motilva, M. P. Romero. Recent Advances in Biologically Active Compounds in Herbs and Spices: A Review of the Most Effective Antioxidant and Anti-Inflammatory Active Principles, Crit. Rev. Food Sci. Nutr. 53 (2013) 943–953.

[23] J. Mastelić, I. Jerković, I. Blažević, M. Poljak-Blaži, S. Borović, I. Ivančić-Baće, V. Smrečki, N. Žarković, K. Brčić-Kostic, D. Vikić-Topić, N. Müller. Comparative study on the antioxidant and biological activities of carvacrol, thymol, and eugenol derivatives. J. Agric. Food Chem. 56 (2008) 3989–3996.

[24] Food and Drug Administration. Substances Generally Recognized as Safe Food Additives Permitted for Direct Addition to Food for Human Consumption. C.F.R 2006. Available online:

https://www.accessdata.fda.gov/scripts/cdrh/cfdocs/cfCFR/CFRSearch.cfm?fr=172.515

[25] A. El-Ghaouth. Biologically-based alternatives to synthetic fungicides for the control of postharvest diseases. J. Ind. Microbiol. Biotechnol. 19 (1997) 160–162.

[26] J. D. Rajput, Design. Synthesis and Biological Evaluation of Novel Class Diindolyl Methanes (DIMs) Derived from Naturally Occurring Phenolic Monoterpenoids. Med. Chem. (Los. Angeles). 6 (2016) 123–128.

[27] K. Wang, S. Jiang, Y. Yang, L. Fan, F. Su, M. Ye. Synthesis and antifungal activity of carvacrol and thymol esters with heteroaromatic carboxylic acids. Nat. Prod. Res. 33 (2019) 1924–1930.

[28] Bruker 2016. *APEX3* (Version 5.054), *SAINT+* (Version 6.36A), *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

[29] L. Krause, R. Herbst-Irmer, G. M. Sheldrick, D. Stalke. Comparison of silver and molybdenum microfocus X-ray sources for single-crystal structure determination. J. Appl. Crystallogr. 48 (2015) 3–10.

[30] G. M. Sheldrick. SHELXT - Integrated space-group and crystal-structure determination. Acta Crystallogr. Sect. A Found. Crystallogr. 71 (2015) 3–8.

[31] G. M. Sheldrick. Crystal structure refinement with SHELXL. Acta Crystallogr. Sect. C Struct. Chem. 71 (2015) 3–8.

[32] K. Brandenburg . *DIAMOND*. Crystal Impact GbR, B *Bonn, Germany* (2006)

[33] M. A. Spackman, D. Jayatilaka,.Hirshfeld surface analysis. CrystEngComm. 11 (2009) 19–32.

[34] J. J. McKinnon, D. Jayatilaka, M. A. Spackman. Towards quantitative analysis of intermolecular interactions with Hirshfeld surfaces. Chem. Commun. (2007) 3814–3816.

[35] M. J. Turner, J .J. MacKinnon, S. K. Wolff, D. J. Grimwood, P. R. Spackman, D. Jayatilaka, M. A. Spackman. *Crystal Explorer17.5*. University of Western Australia, Perth, Australia.(2017).

[36] M. Frisch, G. Trucks, H. Schlegel, G. Scuseria, Gaussian 09, revision D. 01, (2009).

[37] A. Frisch, A. Nielson, A. HOLDER,. J. Gaussview user manual. *Gaussian Inc., Pittsburgh, PA*, 556(2000)

[38] A. D. Becke. Density-functional thermochemistry. III. The role of exact exchange. J. Chem. Phys. 98 (1993) 5648–5652.

[39] C. Lee, W. Yang, R. G. Parr. Development of the Colle-Salvetti correlation-energy formula into a functional of the electron density. Phys. Rev B. 37 (1988) 785-789

[40] B. Miehlich, A. Savin, A. Stoll, H. Preuss, Results obtained with the correlation energy density functionals of becke and Lee, Yang and Parr. *Chemical Physics Letters* 157(3) (1989): 200-206

[41] V. V. Vaidya, K. S. Wankhede, M. M. Salunkhe, G. K. Trivedi. Synthesis of isoxazole conjugates of sugars via 1,3-dipolar cycloaddition. Can. J. Chem. 86 (2008) 138–141.

[42] G. jun Fan, W. Mar, M. K. Park, E. Wook Choi, K. Kim, S. Kim. A novel class of inhibitors for steroid 5α-Reductase: Synthesis and evaluation of umbelliferone derivatives. Bioorganic Med. Chem. Lett. 11 (2001) 2361–2363.

[43] M. L. McIntosh, M. R. Naffziger, B. O.Ashburn, L. N. Zakharov, R. G. Carter, Highly regioselective nitrile oxide dipolar cycloadditions with ortho-nitrophenyl alkynes. *Organic & biomolecular chemistry*. *10*(46) (2012) 9204-9213.

[44] C. Niu, L. Yin, L. F. Nie, J. Dou, J. Y. Zhao, G. Li, H. A. Aisa. Synthesis and bioactivity of novel isoxazole chalcone derivatives on tyrosinase and melanin synthesis in murine B16 cells for the treatment of vitiligo. Bioorganic Med. Chem. 24 (2016) 5440–5448.

[45] B. Balakrishnan, C. Praveen, P.R. Seshadri, P.T. Perumal. 5-(3-Methoxyphenyl)-3-phenyl-1,2-oxazole. Acta Crystallogr. Sect. E Struct. Reports Online. 69 (2013) 0–6.

[46] Y. Kandri Rodi, A. Haoudi, F. Capet, C. Rolando, L. El Ammari. 3-benzyl-1-{[3-(4-chlorophenyl)isoxazol-5-yl]methyl}-1H-benzimidazol-2(3H) -one. Acta Crystallogr. Sect. E Struct. Reports Online. 69 (2013) 2663–2670.

[47] C. Fernández-Galleguillos, J. Bórquez, A. Cárdenas, I. Brito. Crystal structure of 3-(2-chloro-6-methoxyquinolin-3-yl)-5-phenylisoxazole ($C_{19}H_{13}ClN_2O_2$). *Zeitschrift für Kristallographie-New Crystal Structures*, 233 (2018) 371–372.

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_audit_update_record
;
2020-05-26 # Formatted by publCIF

;
_publcif_datablock.id '{ed9f8d7d-313e-43c9-a4cb-1b2cebe11232}'

# publcif _publ_body_element loop start
loop_
_publ_body_element
_publ_body_title
_publ_body_contents

 section
;
Chemical context
;
;

;
 section
;
Structural commentary
;
;

;
 section
;
Supramolecular features
;
;

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 section
;
Database survey
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```
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 section
;
Synthesis and crystallization
;
;

;
 section
;
Refinement
;
;
Crystal data, data collection and structure refinement details are
summarized
in Table 1.
;

# publcif _publ_body_element loop end
_audit_creation_method            'SHELXL-2018/3'

_chemical_name_systematic         ?
_chemical_name_common             ?
_chemical_melting_point           ?
_chemical_formula_moiety         'C21 H23 N O3'
_chemical_formula_sum
 'C21 H23 N O3'
_chemical_formula_weight          337.40

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_source
 'C'  'C'   0.0033   0.0016
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'H'  'H'   0.0000   0.0000
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
 'N'  'N'   0.0061   0.0033
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_space_group_crystal_system       triclinic
_space_group_IT_number            2
_space_group_name_H-M_alt         'P -1'
_space_group_name_Hall            '-P 1'

_shelx_space_group_comment
;
The symmetry employed for this shelxl refinement is uniquely defined
by the following loop, which should always be used as a source of
symmetry information in preference to the above space-group names.
They are only intended as comments.
```

```
;

loop_
 _space_group_symop_operation_xyz
 'x, y, z'
 '-x, -y, -z'

_cell_length_a                    9.4958(10)
_cell_length_b                    9.5333(15)
_cell_length_c                    10.2143(16)
_cell_angle_alpha                 96.630(6)
_cell_angle_beta                  90.474(5)
_cell_angle_gamma                 93.885(6)
_cell_volume                      916.2(2)
_cell_formula_units_Z             2
_cell_measurement_temperature     296(2)
_cell_measurement_reflns_used     3746
_cell_measurement_theta_min       2.770
_cell_measurement_theta_max       26.367

_exptl_crystal_description        Plate
_exptl_crystal_colour             colorless
_exptl_crystal_density_meas       ?
_exptl_crystal_density_method     ?
_exptl_crystal_density_diffrn     1.223
_exptl_crystal_F_000              360
_exptl_transmission_factor_min    ?
_exptl_transmission_factor_max    ?
_exptl_crystal_size_max           0.38
_exptl_crystal_size_mid           0.26
_exptl_crystal_size_min           0.15

_exptl_absorpt_coefficient_mu     0.081
_exptl_absorpt_correction_type    multi-scan
_exptl_absorpt_correction_T_min   0.6918
_exptl_absorpt_correction_T_max   0.7462
_exptl_absorpt_process_details    'SADABS(Krause et al., 2015)'

_diffrn_ambient_temperature       296(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_source                    'INCOATEC I\mS micro-focus source'
_diffrn_radiation_monochromator   'HELIOS mirror optics'
_diffrn_measurement_device_type   'Bruker D8 VENTURE Super DUO'
_diffrn_detector                  'PHOTON100 CMOS area-detector'
_diffrn_measurement_method        '\f and \w scans'

_diffrn_reflns_number             32299
_diffrn_reflns_av_unetI/netI      0.0237
_diffrn_reflns_av_R_equivalents   0.0420
_diffrn_reflns_limit_h_min        -11
_diffrn_reflns_limit_h_max        11
_diffrn_reflns_limit_k_min        -11
_diffrn_reflns_limit_k_max        11
_diffrn_reflns_limit_l_min        -12
_diffrn_reflns_limit_l_max        12
```

```
_diffrn_reflns_theta_min          2.770
_diffrn_reflns_theta_max          26.367
_diffrn_reflns_theta_full         25.242
_diffrn_measured_fraction_theta_max   0.999
_diffrn_measured_fraction_theta_full  0.999
_diffrn_reflns_Laue_measured_fraction_max    0.999
_diffrn_reflns_Laue_measured_fraction_full   0.999
_diffrn_reflns_point_group_measured_fraction_max   0.999
_diffrn_reflns_point_group_measured_fraction_full  0.999
_reflns_number_total              3746
_reflns_number_gt                 2716
_reflns_threshold_expression      'I > 2\s(I)'
_reflns_Friedel_coverage          0.000
_reflns_Friedel_fraction_max      .
_reflns_Friedel_fraction_full     .

_reflns_special_details
;
 Reflections were merged by SHELXL according to the crystal
 class for the calculation of statistics and refinement.

 _reflns_Friedel_fraction is defined as the number of unique
 Friedel pairs measured divided by the number that would be
 possible theoretically, ignoring centric projections and
 systematic absences.
;

_computing_data_collection        'APEX3 (Bruker, 2016)'
_computing_cell_refinement        'SAINT (Bruker, 2016)'
_computing_data_reduction          SAINT
_computing_structure_solution     'SHELXT 2014/5 (Sheldrick, 2014)'
_computing_structure_refinement 'SHELXL-2018/3 (Sheldrick, 2018)'
_computing_molecular_graphics    'DIAMOND (Brandenburg et al.,
2012)'

_computing_publication_material   ?
_refine_special_details           ?
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
'w=1/[\s^2^(Fo^2^)+(0.0603P)^2^+0.2440P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      'SHELXL-2018/3 (Sheldrick 2018)'
_refine_ls_extinction_coef        0.018(4)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          3746
_refine_ls_number_parameters      231
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0708
_refine_ls_R_factor_gt            0.0480
_refine_ls_wR_factor_ref          0.1331
```

```
_refine_ls_wR_factor_gt           0.1196
_refine_ls_goodness_of_fit_ref    1.026
_refine_ls_restrained_S_all       1.026
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000

loop_
 _atom_site_label
 _atom_site_type_symbol
 _atom_site_fract_x
 _atom_site_fract_y
 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_site_symmetry_order
 _atom_site_calc_flag
 _atom_site_refinement_flags_posn
 _atom_site_refinement_flags_adp
 _atom_site_refinement_flags_occupancy
 _atom_site_disorder_assembly
 _atom_site_disorder_group
C12 C 0.8165(2) 0.0139(2) 0.5235(2) 0.0745(6) Uani 1 1 d . . . . .
H12A H 0.881825 0.091896 0.555249 0.112 Uiso 1 1 calc R U . . .
H12B H 0.866166 -0.058036 0.473377 0.112 Uiso 1 1 calc R U . . .
H12C H 0.772024 -0.024438 0.596893 0.112 Uiso 1 1 calc R U . . .
C1 C 0.62335(16) 0.15727(17) 0.50029(16) 0.0441(4) Uani 1 1 d . . .
. .
C2 C 0.52111(17) 0.19793(17) 0.41659(15) 0.0426(4) Uani 1 1 d . . .
. .
C3 C 0.42671(16) 0.29290(17) 0.46977(15) 0.0423(4) Uani 1 1 d . . .
. .
H3 H 0.356988 0.320590 0.415652 0.051 Uiso 1 1 calc R U . . .
C4 C 0.43374(15) 0.34793(17) 0.60242(15) 0.0401(4) Uani 1 1 d . . .
. .
C5 C 0.53722(16) 0.30904(17) 0.68605(15) 0.0413(4) Uani 1 1 d . . .
. .
C6 C 0.63091(17) 0.21271(18) 0.63204(16) 0.0464(4) Uani 1 1 d . . .
. .
H6 H 0.700651 0.184715 0.685962 0.056 Uiso 1 1 calc R U . . .
C11 C 0.24090(16) 0.48967(18) 0.57407(16) 0.0448(4) Uani 1 1 d . . .
. .
H11A H 0.184915 0.409551 0.528577 0.054 Uiso 1 1 calc R U . . .
H11B H 0.287854 0.541840 0.508876 0.054 Uiso 1 1 calc R U . . .
C5' C 0.14933(16) 0.58269(17) 0.65769(16) 0.0422(4) Uani 1 1 d . . .
. .
C4' C 0.12880(16) 0.60956(17) 0.78713(16) 0.0451(4) Uani 1 1 d . . .
. .
H4' H 0.175857 0.573595 0.854771 0.054 Uiso 1 1 calc R U . . .
C3' C 0.01954(16) 0.70497(17) 0.79932(16) 0.0430(4) Uani 1 1 d . . .
. .
C13 C -0.04770(16) 0.76744(17) 0.91995(16) 0.0444(4) Uani 1 1 d . .
. . .
C14 C -0.0237(2) 0.7179(2) 1.04001(18) 0.0566(5) Uani 1 1 d . . . .
.
H14 H 0.037071 0.646051 1.044622 0.068 Uiso 1 1 calc R U . . .
```

```
C15 C -0.0890(2) 0.7737(2) 1.1531(2) 0.0699(6) Uani 1 1 d . . . . .
H15 H -0.072021 0.739460 1.233013 0.084 Uiso 1 1 calc R U . . .
C16 C -0.1793(2) 0.8805(2) 1.1471(2) 0.0715(6) Uani 1 1 d . . . . .
H16 H -0.223722 0.918165 1.222635 0.086 Uiso 1 1 calc R U . . .
C17 C -0.2029(2) 0.9304(2) 1.0289(2) 0.0688(6) Uani 1 1 d . . . . .
H17 H -0.263603 1.002426 1.024853 0.083 Uiso 1 1 calc R U . . .
C18 C -0.13828(19) 0.8758(2) 0.9164(2) 0.0569(5) Uani 1 1 d . . . .
.
H18 H -0.155191 0.911453 0.837199 0.068 Uiso 1 1 calc R U . . .
C7 C 0.5152(2) 0.1407(2) 0.27242(17) 0.0589(5) Uani 1 1 d . . . . .
H7A H 0.599474 0.172950 0.230509 0.088 Uiso 1 1 calc R U . . .
H7B H 0.434437 0.173637 0.231147 0.088 Uiso 1 1 calc R U . . .
H7C H 0.507758 0.039115 0.263907 0.088 Uiso 1 1 calc R U . . .
C8 C 0.55189(18) 0.37400(19) 0.82973(15) 0.0496(4) Uani 1 1 d . . .
. .
H8 H 0.462104 0.413570 0.854966 0.060 Uiso 1 1 calc R U . . .
C10 C 0.5815(2) 0.2667(2) 0.92343(19) 0.0681(6) Uani 1 1 d . . . . .
H10A H 0.580190 0.311089 1.012647 0.102 Uiso 1 1 calc R U . . .
H10B H 0.672546 0.231516 0.905456 0.102 Uiso 1 1 calc R U . . .
H10C H 0.510425 0.189547 0.911471 0.102 Uiso 1 1 calc R U . . .
C9 C 0.6656(3) 0.4950(2) 0.8442(2) 0.0796(7) Uani 1 1 d . . . . .
H9A H 0.641000 0.566263 0.790592 0.119 Uiso 1 1 calc R U . . .
H9B H 0.754263 0.459939 0.816294 0.119 Uiso 1 1 calc R U . . .
H9C H 0.673386 0.535106 0.934774 0.119 Uiso 1 1 calc R U . . .
O3 O 0.71274(13) 0.06134(14) 0.44254(13) 0.0607(4) Uani 1 1 d . . .
. .
O2 O 0.34109(12) 0.44297(13) 0.65899(11) 0.0504(3) Uani 1 1 d . . .
. .
O1 O 0.06058(13) 0.65434(14) 0.59095(12) 0.0604(4) Uani 1 1 d . . .
. .
N1 N -0.02261(16) 0.73266(17) 0.68325(15) 0.0609(4) Uani 1 1 d . . .
. .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
C12 0.0520(11) 0.0877(15) 0.0837(15) -0.0090(12) -0.0026(10)
0.0367(11)
C1 0.0400(9) 0.0489(9) 0.0443(9) 0.0030(7) 0.0088(7) 0.0119(7)
C2 0.0450(9) 0.0464(9) 0.0365(8) 0.0031(7) 0.0049(7) 0.0055(7)
C3 0.0419(9) 0.0503(9) 0.0361(8) 0.0062(7) -0.0021(7) 0.0112(7)
C4 0.0377(8) 0.0480(9) 0.0362(8) 0.0055(7) 0.0052(6) 0.0118(7)
C5 0.0384(8) 0.0512(9) 0.0356(8) 0.0068(7) 0.0029(6) 0.0094(7)
C6 0.0409(9) 0.0594(10) 0.0412(9) 0.0077(8) -0.0007(7) 0.0161(8)
C11 0.0395(8) 0.0537(10) 0.0418(9) 0.0023(7) -0.0032(7) 0.0134(7)
C5' 0.0353(8) 0.0466(9) 0.0451(9) 0.0041(7) -0.0041(7) 0.0087(7)
C4' 0.0414(9) 0.0507(10) 0.0447(9) 0.0056(7) -0.0036(7) 0.0141(7)
C3' 0.0360(8) 0.0456(9) 0.0474(9) 0.0039(7) -0.0029(7) 0.0060(7)
C13 0.0363(8) 0.0460(9) 0.0499(10) 0.0001(7) 0.0007(7) 0.0049(7)
C14 0.0622(11) 0.0551(11) 0.0540(11) 0.0048(8) 0.0030(9) 0.0174(9)
```

```
C15 0.0862(15) 0.0748(14) 0.0503(11) 0.0068(10) 0.0088(10)
0.0169(12)
C16 0.0735(14) 0.0780(14) 0.0616(13) -0.0071(11) 0.0186(11)
0.0182(11)
C17 0.0638(13) 0.0709(13) 0.0727(14) -0.0018(11) 0.0090(10)
0.0285(10)
C18 0.0526(10) 0.0609(11) 0.0593(11) 0.0067(9) 0.0019(8) 0.0197(9)
C7 0.0662(12) 0.0682(12) 0.0419(10) -0.0027(8) 0.0030(8) 0.0168(9)
C8 0.0486(9) 0.0679(11) 0.0337(8) 0.0026(8) 0.0007(7) 0.0199(8)
C10 0.0848(14) 0.0779(14) 0.0422(10) 0.0138(9) -0.0059(9) -
0.0014(11)
C9 0.123(2) 0.0626(13) 0.0492(12) 0.0005(10) -0.0052(12) -0.0086(13)
O3 0.0553(7) 0.0723(9) 0.0555(8) -0.0042(6) 0.0054(6) 0.0298(6)
O2 0.0501(7) 0.0663(8) 0.0371(6) 0.0016(5) -0.0006(5) 0.0278(6)
O1 0.0606(8) 0.0794(9) 0.0447(7) 0.0034(6) -0.0038(6) 0.0369(7)
N1 0.0589(9) 0.0764(11) 0.0507(9) 0.0027(8) -0.0004(7) 0.0367(8)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

loop_
 _geom_bond_atom_site_label_1
 _geom_bond_atom_site_label_2
 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
C12 O3 1.411(2) . ?
C12 H12A 0.9600 . ?
C12 H12B 0.9600 . ?
C12 H12C 0.9600 . ?
C1 O3 1.3762(19) . ?
C1 C6 1.387(2) . ?
C1 C2 1.392(2) . ?
C2 C3 1.387(2) . ?
C2 C7 1.509(2) . ?
C3 C4 1.395(2) . ?
C3 H3 0.9300 . ?
C4 O2 1.3872(18) . ?
C4 C5 1.393(2) . ?
C5 C6 1.393(2) . ?
C5 C8 1.527(2) . ?
C6 H6 0.9300 . ?
C11 O2 1.4102(18) . ?
```

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C11 C5' 1.488(2) . ?
C11 H11A 0.9700 . ?
C11 H11B 0.9700 . ?
C5' C4' 1.336(2) . ?
C5' O1 1.3482(18) . ?
C4' C3' 1.422(2) . ?
C4' H4' 0.9300 . ?
C3' N1 1.309(2) . ?
C3' C13 1.475(2) . ?
C13 C14 1.387(2) . ?
C13 C18 1.391(2) . ?
C14 C15 1.385(3) . ?
C14 H14 0.9300 . ?
C15 C16 1.381(3) . ?
C15 H15 0.9300 . ?
C16 C17 1.370(3) . ?
C16 H16 0.9300 . ?
C17 C18 1.373(3) . ?
C17 H17 0.9300 . ?
C18 H18 0.9300 . ?
C7 H7A 0.9600 . ?
C7 H7B 0.9600 . ?
C7 H7C 0.9600 . ?
C8 C10 1.517(2) . ?
C8 C9 1.519(3) . ?
C8 H8 0.9800 . ?
C10 H10A 0.9600 . ?
C10 H10B 0.9600 . ?
C10 H10C 0.9600 . ?
C9 H9A 0.9600 . ?
C9 H9B 0.9600 . ?
C9 H9C 0.9600 . ?
O1 N1 1.4143(18) . ?

loop_
 _geom_angle_atom_site_label_1
 _geom_angle_atom_site_label_2
 _geom_angle_atom_site_label_3
 _geom_angle
 _geom_angle_site_symmetry_1
 _geom_angle_site_symmetry_3
 _geom_angle_publ_flag
O3 C12 H12A 109.5 . . ?
O3 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
O3 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
O3 C1 C6 124.21(15) . . ?
O3 C1 C2 115.32(14) . . ?
C6 C1 C2 120.47(14) . . ?
C3 C2 C1 117.89(14) . . ?
C3 C2 C7 121.56(15) . . ?
C1 C2 C7 120.55(15) . . ?
C2 C3 C4 121.63(15) . . ?
C2 C3 H3 119.2 . . ?
```

```
C4 C3 H3 119.2 . . ?
O2 C4 C5 116.16(14) . . ?
O2 C4 C3 123.21(14) . . ?
C5 C4 C3 120.63(14) . . ?
C6 C5 C4 117.36(14) . . ?
C6 C5 C8 121.08(14) . . ?
C4 C5 C8 121.51(14) . . ?
C1 C6 C5 122.01(15) . . ?
C1 C6 H6 119.0 . . ?
C5 C6 H6 119.0 . . ?
O2 C11 C5' 106.90(13) . . ?
O2 C11 H11A 110.3 . . ?
C5' C11 H11A 110.3 . . ?
O2 C11 H11B 110.3 . . ?
C5' C11 H11B 110.3 . . ?
H11A C11 H11B 108.6 . . ?
C4' C5' O1 109.95(14) . . ?
C4' C5' C11 134.90(15) . . ?
O1 C5' C11 115.09(14) . . ?
C5' C4' C3' 105.22(14) . . ?
C5' C4' H4' 127.4 . . ?
C3' C4' H4' 127.4 . . ?
N1 C3' C4' 110.82(15) . . ?
N1 C3' C13 120.45(14) . . ?
C4' C3' C13 128.71(15) . . ?
C14 C13 C18 118.17(16) . . ?
C14 C13 C3' 120.62(15) . . ?
C18 C13 C3' 121.20(16) . . ?
C15 C14 C13 120.97(17) . . ?
C15 C14 H14 119.5 . . ?
C13 C14 H14 119.5 . . ?
C16 C15 C14 119.8(2) . . ?
C16 C15 H15 120.1 . . ?
C14 C15 H15 120.1 . . ?
C17 C16 C15 119.51(19) . . ?
C17 C16 H16 120.2 . . ?
C15 C16 H16 120.2 . . ?
C16 C17 C18 120.97(19) . . ?
C16 C17 H17 119.5 . . ?
C18 C17 H17 119.5 . . ?
C17 C18 C13 120.54(19) . . ?
C17 C18 H18 119.7 . . ?
C13 C18 H18 119.7 . . ?
C2 C7 H7A 109.5 . . ?
C2 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C2 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C10 C8 C9 110.34(16) . . ?
C10 C8 C5 113.08(15) . . ?
C9 C8 C5 110.33(15) . . ?
C10 C8 H8 107.6 . . ?
C9 C8 H8 107.6 . . ?
C5 C8 H8 107.6 . . ?
C8 C10 H10A 109.5 . . ?
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```
C8 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C8 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
C8 C9 H9A 109.5 . . ?
C8 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C8 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
C1 O3 C12 117.76(14) . . ?
C4 O2 C11 116.84(12) . . ?
C5' O1 N1 108.30(12) . . ?
C3' N1 O1 105.71(12) . . ?

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C6 C1 C2 C3 -1.1(2) . . . . ?
O3 C1 C2 C7 -1.4(2) . . . . ?
C6 C1 C2 C7 178.37(16) . . . . ?
C1 C2 C3 C4 0.7(2) . . . . ?
C7 C2 C3 C4 -178.77(16) . . . . ?
C2 C3 C4 O2 -179.94(14) . . . . ?
C2 C3 C4 C5 0.2(3) . . . . ?
O2 C4 C5 C6 179.44(14) . . . . ?
C3 C4 C5 C6 -0.7(2) . . . . ?
O2 C4 C5 C8 -3.2(2) . . . . ?
C3 C4 C5 C8 176.64(15) . . . . ?
O3 C1 C6 C5 -179.60(15) . . . . ?
C2 C1 C6 C5 0.6(3) . . . . ?
C4 C5 C6 C1 0.3(2) . . . . ?
C8 C5 C6 C1 -177.07(16) . . . . ?
O2 C11 C5' C4' -12.8(3) . . . . ?
O2 C11 C5' O1 170.40(13) . . . . ?
O1 C5' C4' C3' -0.15(19) . . . . ?
C11 C5' C4' C3' -177.02(18) . . . . ?
C5' C4' C3' N1 0.2(2) . . . . ?
C5' C4' C3' C13 178.55(16) . . . . ?
N1 C3' C13 C14 167.28(17) . . . . ?
C4' C3' C13 C14 -10.9(3) . . . . ?
N1 C3' C13 C18 -11.7(3) . . . . ?
C4' C3' C13 C18 170.06(17) . . . . ?
C18 C13 C14 C15 0.5(3) . . . . ?
C3' C13 C14 C15 -178.51(17) . . . . ?
C13 C14 C15 C16 -0.1(3) . . . . ?
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C14 C15 C16 C17 -0.3(3) . . . . ?
C15 C16 C17 C18 0.1(3) . . . . ?
C16 C17 C18 C13 0.4(3) . . . . ?
C14 C13 C18 C17 -0.7(3) . . . . ?
C3' C13 C18 C17 178.34(17) . . . . ?
C6 C5 C8 C10 -44.0(2) . . . . ?
C4 C5 C8 C10 138.77(17) . . . . ?
C6 C5 C8 C9 80.2(2) . . . . ?
C4 C5 C8 C9 -97.1(2) . . . . ?
C6 C1 O3 C12 1.6(3) . . . . ?
C2 C1 O3 C12 -178.59(16) . . . . ?
C5 C4 O2 C11 176.44(14) . . . . ?
C3 C4 O2 C11 -3.4(2) . . . . ?
C5' C11 O2 C4 176.70(13) . . . . ?
C4' C5' O1 N1 0.05(19) . . . . ?
C11 C5' O1 N1 177.60(14) . . . . ?
C4' C3' N1 O1 -0.17(19) . . . . ?
C13 C3' N1 O1 -178.67(14) . . . . ?
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C16 H16 O3  0.93 2.59 3.496(2) 165.4 1_466 yes

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LATT   1
SFAC  C    H    O    N
UNIT  42 46 6 2
MERG   2
OMIT     0   1   0
SHEL 50  0.80
HTAB C11 O1_$1
EQIV $1 -x, -y+1, -z+1
HTAB C16 O3_$2
EQIV $2 -1+x, 1+y, 1+z
FMAP   2
PLAN    6
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```
ACTA
BOND   $H
CONF
L.S.  10
TEMP    23.00
WGHT    0.060300    0.244000
EXTI    0.017548
FVAR       0.69219
MOLE    1
C12   1    0.816483    0.013897    0.523486    11.00000    0.05200
0.08773 =
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AFIX 137
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H12B  2    0.866166   -0.058036    0.473377    11.00000   -1.50000
H12C  2    0.772024   -0.024438    0.596893    11.00000   -1.50000
AFIX   0
C1    1    0.623348    0.157266    0.500295    11.00000    0.03998
0.04890 =
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C2    1    0.521109    0.197926    0.416587    11.00000    0.04498
0.04640 =
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C3    1    0.426708    0.292904    0.469774    11.00000    0.04189
0.05029 =
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H3    2    0.356988    0.320590    0.415652    11.00000   -1.20000
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0.04796 =
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C5    1    0.537223    0.309040    0.686052    11.00000    0.03837
0.05123 =
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C6    1    0.630912    0.212707    0.632045    11.00000    0.04089
0.05940 =
         0.04120    0.00771   -0.00069    0.01609
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H6    2    0.700651    0.184715    0.685962    11.00000   -1.20000
AFIX   0
C11   1    0.240903    0.489671    0.574070    11.00000    0.03946
0.05367 =
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AFIX  23
H11A  2    0.184915    0.409551    0.528577    11.00000   -1.20000
H11B  2    0.287854    0.541840    0.508876    11.00000   -1.20000
AFIX   0
C5'   1    0.149334    0.582692    0.657695    11.00000    0.03530
0.04660 =
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C4'   1    0.128802    0.609561    0.787131    11.00000    0.04141
0.05070 =
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C13   1   -0.047703    0.767442    0.919948    11.00000    0.03627
0.04595 =
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C14   1   -0.023727    0.717873    1.040013    11.00000    0.06223
0.05509 =
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AFIX  43
H14   2    0.037071    0.646051    1.044622    11.00000   -1.20000
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C15   1   -0.088977    0.773743    1.153139    11.00000    0.08623
0.07483 =
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AFIX  43
H15   2   -0.072021    0.739460    1.233013    11.00000   -1.20000
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C16   1   -0.179273    0.880473    1.147070    11.00000    0.07349
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H16   2   -0.223722    0.918165    1.222635    11.00000   -1.20000
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C17   1   -0.202900    0.930429    1.028926    11.00000    0.06379
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0.06094 =
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H18   2   -0.155191    0.911453    0.837199    11.00000   -1.20000
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C7    1    0.515152    0.140700    0.272419    11.00000    0.06616
0.06825 =
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H7A   2    0.599474    0.172950    0.230509    11.00000   -1.50000
H7B   2    0.434437    0.173637    0.231147    11.00000   -1.50000
H7C   2    0.507758    0.039115    0.263907    11.00000   -1.50000
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C8    1    0.551891    0.374003    0.829732    11.00000    0.04857
0.06788 =
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H8    2    0.462104    0.413570    0.854966    11.00000   -1.20000
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C10   1    0.581480    0.266684    0.923431    11.00000    0.08482
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AFIX 137
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```
H10B  2    0.672546    0.231516    0.905456    11.00000   -1.50000
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AFIX   0
C9    1    0.665591    0.494951    0.844177    11.00000    0.12321
0.06264 =
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AFIX   0
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0.07231 =
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O2    3    0.341094    0.442965    0.658992    11.00000    0.05010
0.06633 =
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O1    3    0.060584    0.654341    0.590946    11.00000    0.06058
0.07943 =
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N1    4   -0.022607    0.732664    0.683250    11.00000    0.05886
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HKLF    4



REM  mo_D18_298_Ketateni_LY2_0m_a.res in P-1
REM wR2 = 0.1331, GooF = S = 1.026, Restrained GooF = 1.026 for all
data
REM R1 = 0.0480 for 2716 Fo > 4sig(Fo) and 0.0708 for all 3746 data
REM 231 parameters refined using 0 restraints

END

WGHT      0.0601      0.2452

REM Highest difference peak  0.161,  deepest hole -0.157,  1-sigma
level  0.035
Q1    1   0.4066  0.2920  0.5454  11.00000  0.05    0.16
Q2    1   0.4753  0.2308  0.4575  11.00000  0.05    0.16
Q3    1   0.5912  0.1595  0.5660  11.00000  0.05    0.16
Q4    1   0.1038  0.5449  0.7086  11.00000  0.05    0.15
Q5    1   0.1920  0.5495  0.6254  11.00000  0.05    0.15
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Mat\'eriaux, Faculty of Sciences, Mohammed V University in Rabat,
Avenue Ibn Batouta, B.P. 1014, Rabat, Morocco
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Journal of Molecular Structure.
All required files have been provided.

The manuscript has passed the checkcif tests and generates an acceptable printcif output.

Sincerely yours

Lahcen El Ammari
;

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elm_ketatni@hotmail.fr
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5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-(p-
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#Added by publCIF



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2020-05-28 # Formatted by publCIF

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Introduction
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Experimental
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 subsection
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Synthesis and crystallization
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 subsection
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Refinement
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Crystal data, data collection and structure refinement details are
summarized
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in Table 1.
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Results and discussion
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The symmetry employed for this shelxl refinement is uniquely defined
by the following loop, which should always be used as a source of
symmetry information in preference to the above space-group names.
They are only intended as comments.
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_cell_volume                        967.89(8)
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_reflns_special_details
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 Reflections were merged by SHELXL according to the crystal
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 _reflns_Friedel_fraction is defined as the number of unique
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_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          4684
_refine_ls_number_parameters      241
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0749
_refine_ls_R_factor_gt            0.0491
_refine_ls_wR_factor_ref          0.1533
_refine_ls_wR_factor_gt           0.1407
_refine_ls_goodness_of_fit_ref    1.097
_refine_ls_restrained_S_all       1.097
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000

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 _atom_site_disorder_assembly
 _atom_site_disorder_group
O2 O 0.68668(16) 0.80525(11) 0.06296(10) 0.0688(4) Uani 1 1 d . . .
. .
O1 O 1.02993(15) 0.89811(11) -0.13048(9) 0.0639(3) Uani 1 1 d . . .
. .
O3 O 0.16228(17) 0.95205(14) 0.37681(11) 0.0837(4) Uani 1 1 d . . .
. .
N1 N 1.12346(18) 0.82544(14) -0.21058(11) 0.0629(4) Uani 1 1 d . . .
. .
C12 C 0.0090(2) 0.8968(2) 0.39108(17) 0.0776(5) Uani 1 1 d . . . . .
H12A H 0.037611 0.803134 0.420429 0.116 Uiso 1 1 calc R U . . .
H12B H -0.038884 0.909940 0.321173 0.116 Uiso 1 1 calc R U . . .
H12C H -0.075799 0.939914 0.441309 0.116 Uiso 1 1 calc R U . . .
C1 C 0.2920(2) 0.91198(17) 0.29930(14) 0.0605(4) Uani 1 1 d . . . .
.
C2 C 0.4208(2) 0.98927(16) 0.27011(14) 0.0585(4) Uani 1 1 d . . . .
.
C3 C 0.5524(2) 0.95377(16) 0.19067(14) 0.0589(4) Uani 1 1 d . . . .
.
H3 H 0.638969 1.004860 0.168862 0.071 Uiso 1 1 calc R U . . .
C4 C 0.5582(2) 0.84405(15) 0.14289(13) 0.0524(4) Uani 1 1 d . . . .
.
C5 C 0.4344(2) 0.76360(14) 0.17490(12) 0.0515(4) Uani 1 1 d . . . .
.
C6 C 0.3002(2) 0.80126(16) 0.25257(14) 0.0596(4) Uani 1 1 d . . . .
.
H6 H 0.213132 0.750499 0.273803 0.071 Uiso 1 1 calc R U . . .
C11 C 0.7961(2) 0.89394(16) 0.01237(14) 0.0590(4) Uani 1 1 d . . . .
.
H11A H 0.868089 0.908416 0.065457 0.071 Uiso 1 1 calc R U . . .
H11B H 0.727336 0.979052 -0.019219 0.071 Uiso 1 1 calc R U . . .
C5' C 0.90732(19) 0.83100(15) -0.07524(12) 0.0524(4) Uani 1 1 d . .
. . .
C4' C 0.91606(19) 0.71935(15) -0.11345(13) 0.0537(4) Uani 1 1 d . .
. . .
H4' H 0.847374 0.654721 -0.089097 0.064 Uiso 1 1 calc R U . . .
C3' C 1.05265(19) 0.72051(15) -0.19917(12) 0.0505(4) Uani 1 1 d . .
. . .
C13 C 1.11600(18) 0.62253(15) -0.27180(12) 0.0512(4) Uani 1 1 d . .
. . .
C18 C 1.1960(2) 0.65845(18) -0.37658(13) 0.0640(4) Uani 1 1 d . . .
. .
H18 H 1.211070 0.745936 -0.401701 0.077 Uiso 1 1 calc R U . . .
```

```
C17 C 1.2530(2) 0.56551(19) -0.44322(14) 0.0699(5) Uani 1 1 d . . .
. .
H17 H 1.305882 0.591613 -0.513193 0.084 Uiso 1 1 calc R U . . .
C16 C 1.2340(2) 0.43428(18) -0.40944(15) 0.0647(4) Uani 1 1 d . . .
. .
C15 C 1.1561(2) 0.39943(17) -0.30485(15) 0.0645(4) Uani 1 1 d . . .
. .
H15 H 1.142986 0.311559 -0.279422 0.077 Uiso 1 1 calc R U . . .
C14 C 1.0974(2) 0.49142(16) -0.23718(13) 0.0580(4) Uani 1 1 d . . .
. .
H14 H 1.044579 0.464999 -0.167248 0.070 Uiso 1 1 calc R U . . .
C7 C 0.4163(3) 1.10758(19) 0.32326(17) 0.0799(6) Uani 1 1 d . . . .
.
H7A H 0.507420 1.154287 0.287977 0.120 Uiso 1 1 calc R U . . .
H7B H 0.434003 1.077511 0.400160 0.120 Uiso 1 1 calc R U . . .
H7C H 0.304458 1.166300 0.315480 0.120 Uiso 1 1 calc R U . . .
C8 C 0.4430(2) 0.64075(15) 0.12501(13) 0.0572(4) Uani 1 1 d . . . .
.
H8 H 0.563401 0.616196 0.093073 0.069 Uiso 1 1 calc R U . . .
C9 C 0.3243(4) 0.6722(2) 0.03235(18) 0.1004(8) Uani 1 1 d . . . . .
H9A H 0.364773 0.737272 -0.026049 0.151 Uiso 1 1 calc R U . . .
H9B H 0.206951 0.706859 0.059151 0.151 Uiso 1 1 calc R U . . .
H9C H 0.325674 0.592355 0.004933 0.151 Uiso 1 1 calc R U . . .
C10 C 0.4033(3) 0.52216(17) 0.20992(17) 0.0741(5) Uani 1 1 d . . . .
.
H10A H 0.283437 0.540768 0.239396 0.111 Uiso 1 1 calc R U . . .
H10B H 0.479318 0.505267 0.268473 0.111 Uiso 1 1 calc R U . . .
H10C H 0.422048 0.445217 0.175767 0.111 Uiso 1 1 calc R U . . .
C19 C 1.2969(3) 0.3331(2) -0.48300(19) 0.0928(7) Uani 1 1 d . . . .
.
H19A H 1.398574 0.354685 -0.528899 0.139 Uiso 1 1 calc R U . . .
H19B H 1.205776 0.333960 -0.528550 0.139 Uiso 1 1 calc R U . . .
H19C H 1.326589 0.246079 -0.438529 0.139 Uiso 1 1 calc R U . . .

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 _atom_site_aniso_U_12
O2 0.0766(7) 0.0539(6) 0.0777(7) -0.0299(5) 0.0322(5) -0.0276(5)
O1 0.0652(6) 0.0608(6) 0.0695(6) -0.0220(5) 0.0151(5) -0.0291(5)
O3 0.0722(7) 0.0958(9) 0.0936(9) -0.0554(7) 0.0310(6) -0.0302(6)
N1 0.0623(7) 0.0647(7) 0.0641(7) -0.0228(6) 0.0149(6) -0.0253(6)
C12 0.0649(10) 0.0777(11) 0.0867(12) -0.0276(9) 0.0243(8) -0.0182(8)
C1 0.0562(8) 0.0637(8) 0.0620(8) -0.0261(7) 0.0106(7) -0.0132(7)
C2 0.0599(8) 0.0548(8) 0.0631(8) -0.0262(7) 0.0029(7) -0.0120(6)
C3 0.0575(8) 0.0531(8) 0.0676(9) -0.0220(7) 0.0089(7) -0.0197(6)
C4 0.0531(7) 0.0462(7) 0.0558(8) -0.0169(6) 0.0071(6) -0.0104(6)
C5 0.0533(7) 0.0453(7) 0.0547(7) -0.0158(6) 0.0019(6) -0.0114(6)
C6 0.0566(8) 0.0585(8) 0.0655(9) -0.0218(7) 0.0106(7) -0.0209(6)
C11 0.0589(8) 0.0544(8) 0.0660(9) -0.0214(7) 0.0100(7) -0.0209(6)
C5' 0.0469(7) 0.0551(7) 0.0566(8) -0.0137(6) 0.0039(6) -0.0177(6)
C4' 0.0493(7) 0.0569(8) 0.0583(8) -0.0184(6) 0.0063(6) -0.0224(6)
```

```
C3' 0.0466(7) 0.0551(7) 0.0495(7) -0.0124(6) -0.0006(5) -0.0146(6)
C13 0.0437(6) 0.0588(8) 0.0508(7) -0.0156(6) 0.0010(5) -0.0118(6)
C18 0.0717(9) 0.0643(9) 0.0550(8) -0.0115(7) 0.0060(7) -0.0220(7)
C17 0.0757(10) 0.0816(11) 0.0526(8) -0.0206(8) 0.0137(7) -0.0256(8)
C16 0.0556(8) 0.0758(10) 0.0668(9) -0.0301(8) 0.0080(7) -0.0175(7)
C15 0.0624(9) 0.0615(9) 0.0704(9) -0.0205(7) 0.0079(7) -0.0204(7)
C14 0.0568(8) 0.0625(8) 0.0539(8) -0.0149(6) 0.0087(6) -0.0197(6)
C7 0.0803(11) 0.0760(11) 0.0927(12) -0.0505(10) 0.0111(9) -0.0225(9)
C8 0.0588(8) 0.0480(7) 0.0662(9) -0.0211(6) 0.0040(7) -0.0148(6)
C9 0.150(2) 0.0700(11) 0.0889(13) -0.0299(10) -0.0460(14) -
0.0089(12)
C10 0.0770(10) 0.0543(8) 0.0900(12) -0.0147(8) -0.0005(9) -0.0211(7)
C19 0.0951(13) 0.0966(14) 0.0949(13) -0.0523(11) 0.0321(11) -
0.0345(11)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

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O2 C4 1.3832(18) . ?
O2 C11 1.3965(18) . ?
O1 C5' 1.3449(17) . ?
O1 N1 1.4145(17) . ?
O3 C1 1.3721(19) . ?
O3 C12 1.414(2) . ?
N1 C3' 1.3035(19) . ?
C12 H12A 0.9600 . ?
C12 H12B 0.9600 . ?
C12 H12C 0.9600 . ?
C1 C6 1.382(2) . ?
C1 C2 1.390(2) . ?
C2 C3 1.384(2) . ?
C2 C7 1.507(2) . ?
C3 C4 1.384(2) . ?
C3 H3 0.9300 . ?
C4 C5 1.383(2) . ?
C5 C6 1.388(2) . ?
C5 C8 1.520(2) . ?
```

```
C6 H6 0.9300 . ?
C11 C5' 1.482(2) . ?
C11 H11A 0.9700 . ?
C11 H11B 0.9700 . ?
C5' C4' 1.331(2) . ?
C4' C3' 1.415(2) . ?
C4' H4' 0.9300 . ?
C3' C13 1.471(2) . ?
C13 C14 1.381(2) . ?
C13 C18 1.389(2) . ?
C18 C17 1.372(2) . ?
C18 H18 0.9300 . ?
C17 C16 1.382(3) . ?
C17 H17 0.9300 . ?
C16 C15 1.379(2) . ?
C16 C19 1.504(2) . ?
C15 C14 1.375(2) . ?
C15 H15 0.9300 . ?
C14 H14 0.9300 . ?
C7 H7A 0.9600 . ?
C7 H7B 0.9600 . ?
C7 H7C 0.9600 . ?
C8 C9 1.507(3) . ?
C8 C10 1.513(2) . ?
C8 H8 0.9800 . ?
C9 H9A 0.9600 . ?
C9 H9B 0.9600 . ?
C9 H9C 0.9600 . ?
C10 H10A 0.9600 . ?
C10 H10B 0.9600 . ?
C10 H10C 0.9600 . ?
C19 H19A 0.9600 . ?
C19 H19B 0.9600 . ?
C19 H19C 0.9600 . ?

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C4 O2 C11 118.37(12) . . ?
C5' O1 N1 108.27(11) . . ?
C1 O3 C12 117.80(13) . . ?
C3' N1 O1 105.23(12) . . ?
O3 C12 H12A 109.5 . . ?
O3 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
O3 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
O3 C1 C6 123.77(15) . . ?
O3 C1 C2 115.75(14) . . ?
C6 C1 C2 120.47(14) . . ?
```

```
C1 C2 C3 117.63(14) . . ?
C1 C2 C7 120.88(15) . . ?
C3 C2 C7 121.49(15) . . ?
C4 C3 C2 121.58(15) . . ?
C4 C3 H3 119.2 . . ?
C2 C3 H3 119.2 . . ?
C5 C4 C3 121.03(14) . . ?
C5 C4 O2 115.42(13) . . ?
C3 C4 O2 123.54(14) . . ?
C4 C5 C6 117.26(13) . . ?
C4 C5 C8 121.27(13) . . ?
C6 C5 C8 121.45(14) . . ?
C1 C6 C5 121.95(15) . . ?
C1 C6 H6 119.0 . . ?
C5 C6 H6 119.0 . . ?
O2 C11 C5' 106.04(12) . . ?
O2 C11 H11A 110.5 . . ?
C5' C11 H11A 110.5 . . ?
O2 C11 H11B 110.5 . . ?
C5' C11 H11B 110.5 . . ?
H11A C11 H11B 108.7 . . ?
C4' C5' O1 110.08(13) . . ?
C4' C5' C11 134.52(14) . . ?
O1 C5' C11 115.40(13) . . ?
C5' C4' C3' 104.95(13) . . ?
C5' C4' H4' 127.5 . . ?
C3' C4' H4' 127.5 . . ?
N1 C3' C4' 111.47(13) . . ?
N1 C3' C13 120.19(13) . . ?
C4' C3' C13 128.34(13) . . ?
C14 C13 C18 118.08(15) . . ?
C14 C13 C3' 120.56(13) . . ?
C18 C13 C3' 121.36(14) . . ?
C17 C18 C13 120.33(16) . . ?
C17 C18 H18 119.8 . . ?
C13 C18 H18 119.8 . . ?
C18 C17 C16 121.95(16) . . ?
C18 C17 H17 119.0 . . ?
C16 C17 H17 119.0 . . ?
C15 C16 C17 117.21(16) . . ?
C15 C16 C19 121.20(17) . . ?
C17 C16 C19 121.58(16) . . ?
C16 C15 C14 121.61(16) . . ?
C16 C15 H15 119.2 . . ?
C14 C15 H15 119.2 . . ?
C15 C14 C13 120.81(15) . . ?
C15 C14 H14 119.6 . . ?
C13 C14 H14 119.6 . . ?
C2 C7 H7A 109.5 . . ?
C2 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C2 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C9 C8 C10 111.20(16) . . ?
C9 C8 C5 110.58(14) . . ?
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C10 C8 C5 112.78(14) . . ?
C9 C8 H8 107.3 . . ?
C10 C8 H8 107.3 . . ?
C5 C8 H8 107.3 . . ?
C8 C9 H9A 109.5 . . ?
C8 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C8 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
C8 C10 H10A 109.5 . . ?
C8 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C8 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
C16 C19 H19A 109.5 . . ?
C16 C19 H19B 109.5 . . ?
H19A C19 H19B 109.5 . . ?
C16 C19 H19C 109.5 . . ?
H19A C19 H19C 109.5 . . ?
H19B C19 H19C 109.5 . . ?

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C5' O1 N1 C3' 0.04(15) . . . . ?
C12 O3 C1 C6 -14.8(3) . . . . ?
C12 O3 C1 C2 165.96(16) . . . . ?
O3 C1 C2 C3 -178.74(14) . . . . ?
C6 C1 C2 C3 2.0(2) . . . . ?
O3 C1 C2 C7 1.1(2) . . . . ?
C6 C1 C2 C7 -178.14(15) . . . . ?
C1 C2 C3 C4 -1.0(2) . . . . ?
C7 C2 C3 C4 179.10(15) . . . . ?
C11 O2 C4 C5 168.41(13) . . . . ?
C11 O2 C4 C3 -12.8(2) . . . . ?
C2 C3 C4 C5 -1.6(2) . . . . ?
C2 C3 C4 O2 179.70(13) . . . . ?
C4 O2 C11 C5' -174.92(12) . . . . ?
N1 O1 C5' C4' 0.61(16) . . . . ?
N1 O1 C5' C11 -179.81(12) . . . . ?
O2 C11 C5' C4' 3.0(2) . . . . ?
O2 C11 C5' O1 -176.42(12) . . . . ?
O1 C5' C4' C3' -0.96(16) . . . . ?
C11 C5' C4' C3' 179.58(15) . . . . ?
O1 N1 C3' C4' -0.64(16) . . . . ?
O1 N1 C3' C13 178.72(11) . . . . ?
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C5' C4' C3' N1 1.01(16) . . . . ?
C5' C4' C3' C13 -178.29(13) . . . . ?
N1 C3' C13 C14 154.12(14) . . . . ?
C4' C3' C13 C14 -26.6(2) . . . . ?
N1 C3' C13 C18 -25.7(2) . . . . ?
C4' C3' C13 C18 153.51(15) . . . . ?
C18 C13 C14 C15 -0.2(2) . . . . ?
C3' C13 C14 C15 179.96(13) . . . . ?
C13 C14 C15 C16 -0.5(2) . . . . ?
C14 C15 C16 C17 0.9(2) . . . . ?
C14 C15 C16 C19 -179.51(15) . . . . ?
O3 C1 C6 C5 -179.58(14) . . . . ?
C2 C1 C6 C5 -0.4(2) . . . . ?
O2 C4 C5 C6 -178.03(12) . . . . ?
C3 C4 C5 C6 3.2(2) . . . . ?
O2 C4 C5 C8 0.5(2) . . . . ?
C3 C4 C5 C8 -178.28(13) . . . . ?
C1 C6 C5 C4 -2.2(2) . . . . ?
C1 C6 C5 C8 179.24(14) . . . . ?
C4 C5 C8 C9 -96.65(18) . . . . ?
C6 C5 C8 C9 81.83(19) . . . . ?
C4 C5 C8 C10 138.03(15) . . . . ?
C6 C5 C8 C10 -43.49(19) . . . . ?
C15 C16 C17 C18 -0.6(3) . . . . ?
C19 C16 C17 C18 179.85(16) . . . . ?
C16 C17 C18 C13 -0.1(2) . . . . ?
C14 C13 C18 C17 0.5(2) . . . . ?
C3' C13 C18 C17 -179.62(13) . . . . ?

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C11 H11A O1  0.97 2.62 3.4275(17) 140.7 2_775 yes
C12 H12C O3  0.96 2.68 3.555(2) 151.7 2_576 yes

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_refine_diff_density_rms    0.040

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CELL  0.71073   7.8247  10.4383  12.4226   78.202   83.069   77.892
ZERR     2.00   0.0003   0.0005   0.0006    0.002    0.004    0.003
LATT   1
SFAC  C    H    N    O
UNIT  44 50 2 6
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SHEL 50 0.75
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HTAB C6 O3_$1
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PLAN   20
ACTA
BOND   $H
CONF
LIST   6
L.S.  10
TEMP    23.00
WGHT    0.081300    0.066100
EXTI    0.017086
FVAR       0.59966
O1    4    0.162072    0.952259    0.876735    11.00000    0.07218
0.09576 =
         0.09364   -0.05544    0.03104   -0.03020
O2    4    0.686766    0.805215    0.562925    11.00000    0.07657
0.05391 =
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O3    4    1.029963    0.898031    0.369562    11.00000    0.06521
0.06084 =
         0.06950   -0.02205    0.01509   -0.02912
N2    3    1.123671    0.825472    0.289363    11.00000    0.06222
0.06470 =
         0.06405   -0.02285    0.01491   -0.02527
C1    1    0.008999    0.897036    0.891013    11.00000    0.06483
0.07767 =
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AFIX 137
H1    2   -0.038282    0.909371    0.821009    11.00000   -1.50000
H3    2   -0.076223    0.940688    0.940707    11.00000   -1.50000
H25   2    0.037460    0.803514    0.921029    11.00000   -1.50000
AFIX   0
C2    1    0.291867    0.912031    0.799406    11.00000    0.05614
0.06372 =
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C3    1    0.420867    0.989476    0.770121    11.00000    0.05989
0.05483 =
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C4    1    0.552673    0.953917    0.690602    11.00000    0.05746
0.05311 =
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AFIX  43
H23   2    0.639305    1.004957    0.668794    11.00000   -1.20000
AFIX   0
C5    1    0.558293    0.844009    0.642846    11.00000    0.05309
0.04614 =
         0.05583   -0.01687    0.00716   -0.01035
C6    1    0.796023    0.893987    0.512309    11.00000    0.05894
0.05432 =
         0.06596   -0.02140    0.01001   -0.02092
AFIX  23
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H12   2    0.867905    0.908595    0.565383    11.00000   -1.20000
H11   2    0.727112    0.979037    0.480687    11.00000   -1.20000
AFIX   0
C7    1    0.907426    0.831009    0.424665    11.00000    0.04688
0.05508 =
         0.05655   -0.01368    0.00396   -0.01771
C8    1    0.915942    0.719140    0.386658    11.00000    0.04933
0.05691 =
         0.05830   -0.01844    0.00635   -0.02243
AFIX  43
H13   2    0.847316    0.654477    0.411054    11.00000   -1.20000
AFIX   0
C9    1    1.052670    0.720582    0.300886    11.00000    0.04658
0.05511 =
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C10   1    1.115975    0.622520    0.228111    11.00000    0.04371
0.05877 =
         0.05078   -0.01561    0.00102   -0.01180
C11   1    1.097295    0.491463    0.262922    11.00000    0.05677
0.06249 =
         0.05395   -0.01493    0.00867   -0.01965
AFIX  43
H19   2    1.044403    0.465133    0.332857    11.00000   -1.20000
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C12   1    1.156130    0.399342    0.195212    11.00000    0.06234
0.06153 =
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AFIX  43
H14   2    1.143073    0.311449    0.220613    11.00000   -1.20000
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C13   1    1.234046    0.434425    0.090497    11.00000    0.05559
0.07578 =
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C14   1    1.296606    0.333067    0.016827    11.00000    0.09510
0.09661 =
         0.09488   -0.05229    0.03214   -0.03449
AFIX 137
H15   2    1.325649    0.245974    0.061301    11.00000   -1.50000
H16   2    1.398568    0.354273   -0.028910    11.00000   -1.50000
H2    2    1.205488    0.334330   -0.028881    11.00000   -1.50000
AFIX   0
C15   1    0.299839    0.801307    0.752550    11.00000    0.05654
0.05842 =
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AFIX  43
H24   2    0.212635    0.750684    0.773601    11.00000   -1.20000
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C16   1    0.434519    0.763636    0.674909    11.00000    0.05327
0.04529 =
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C17   1    0.443125    0.640672    0.624983    11.00000    0.05877
0.04800 =
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AFIX  13
H7    2    0.563573    0.616039    0.593187    11.00000   -1.20000
AFIX   0
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0.05433 =
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AFIX 137
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H4    2    0.480370    0.504554    0.767984    11.00000   -1.50000
H5    2    0.420372    0.445508    0.675613    11.00000   -1.50000
AFIX   0
C19   1    0.324882    0.672047    0.532177    11.00000    0.14963
0.06992 =
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AFIX 137
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H8    2    0.322975    0.591644    0.506550    11.00000   -1.50000
H9    2    0.367998    0.734658    0.472709    11.00000   -1.50000
AFIX   0
C20   1    1.253081    0.565505    0.056652    11.00000    0.07563
0.08162 =
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AFIX  43
H18   2    1.306101    0.591518   -0.013308    11.00000   -1.20000
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C21   1    1.196018    0.658608    0.123261    11.00000    0.07170
0.06433 =
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REM  mo_D18_300_Ketateni_LY1_0m_a.res in P-1
REM wR2 = 0.1533, GooF = S = 1.097, Restrained GooF = 1.097 for all
data
REM R1 = 0.0491 for 3211 Fo > 4sig(Fo) and 0.0749 for all 4684 data
REM 241 parameters refined using 0 restraints

END

WGHT      0.0810      0.0660

REM Highest difference peak  0.247,  deepest hole -0.164,  1-sigma
level  0.040
Q1    1   1.0764  0.6741  0.2638  11.00000  0.05    0.25
Q2    1   0.4464  0.7039  0.6543  11.00000  0.05    0.21
Q3    1   1.2326  0.2464  0.0372  11.00000  0.05    0.18
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```
Q4    1   0.4775  0.9634  0.7363  11.00000  0.05    0.18
Q5    1   0.3246  1.0980  0.8923  11.00000  0.05    0.18
Q6    1   0.3532  0.9626  0.7813  11.00000  0.05    0.18
Q7    1   0.2553  0.8986  0.9189  11.00000  0.05    0.17
Q8    1   0.8511  0.8528  0.4598  11.00000  0.05    0.17
Q9    1   0.3338  0.8406  0.8070  11.00000  0.05    0.17
Q10   1   0.3921  0.7639  0.7389  11.00000  0.05    0.17
Q11   1   0.5228  0.9184  0.6447  11.00000  0.05    0.17
Q12   1   0.6022  0.8319  0.5950  11.00000  0.05    0.16
Q13   1   1.0676  0.5713  0.2392  11.00000  0.05    0.16
Q14   1   0.9281  0.7538  0.4322  11.00000  0.05    0.16
Q15   1   1.1917  0.6291  0.1984  11.00000  0.05    0.15
Q16   1   0.0217  0.7854  0.9035  11.00000  0.05    0.15
Q17   1  -0.0184  0.9056  0.9768  11.00000  0.05    0.15
Q18   1   0.8570  0.7990  0.3865  11.00000  0.05    0.15
Q19   1   1.3362  0.3779 -0.0604  11.00000  0.05    0.15
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The manuscript has passed the checkcif tests and generates an
acceptable printcif output.

Sincerely yours

Lahcen El Ammari
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elm_ketatni@hotmail.fr

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3-(4-chlorophenyl)-5-((2-isopropyl-4-methoxy-5-
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#Added by publCIF



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2020-05-28 # Formatted by publCIF

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Introduction
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Experimental
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 subsection
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Synthesis and crystallization
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 subsection
;
Refinement
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Crystal data, data collection and structure refinement details are
summarized
in Table 1.
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 section
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Results and discussion
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The symmetry employed for this shelxl refinement is uniquely defined
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They are only intended as comments.
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C8 C 0.22033(18) 0.17182(13) 0.54368(13) 0.0424(3) Uani 1 1 d . . .
. .
H8 H 0.147396 0.147445 0.592133 0.051 Uiso 1 1 calc R U . . .
C9 C 0.3835(2) 0.15273(19) 0.6110(2) 0.0787(6) Uani 1 1 d . . . . .
H9A H 0.458347 0.173369 0.565113 0.118 Uiso 1 1 calc R U . . .
H9B H 0.378823 0.059861 0.616089 0.118 Uiso 1 1 calc R U . . .
H9C H 0.416612 0.212876 0.694299 0.118 Uiso 1 1 calc R U . . .
C10 C 0.1578(3) 0.07608(18) 0.41171(18) 0.0778(6) Uani 1 1 d . . . .
.
```

```
H10A H 0.058393 0.096425 0.370429 0.117 Uiso 1 1 calc R U . . .
H10B H 0.142182 -0.016748 0.417293 0.117 Uiso 1 1 calc R U . . .
H10C H 0.233417 0.088259 0.364317 0.117 Uiso 1 1 calc R U . . .
C11 C 0.05894(17) 0.42919(13) 0.81478(13) 0.0399(3) Uani 1 1 d . . .
. .
H11A H -0.024489 0.468740 0.770671 0.048 Uiso 1 1 calc R U . . .
H11B H 0.139797 0.502615 0.873914 0.048 Uiso 1 1 calc R U . . .
C12 C 0.36979(19) 0.49180(17) 0.27442(13) 0.0499(4) Uani 1 1 d . . .
. .
H12A H 0.393251 0.544419 0.218302 0.075 Uiso 1 1 calc R U . . .
H12B H 0.288526 0.413797 0.228064 0.075 Uiso 1 1 calc R U . . .
H12C H 0.464147 0.461826 0.312615 0.075 Uiso 1 1 calc R U . . .
C13 C -0.21935(16) 0.06019(13) 0.97159(12) 0.0375(3) Uani 1 1 d . .
. . .
C14 C -0.35269(18) -0.01306(16) 0.87647(14) 0.0465(3) Uani 1 1 d . .
. . .
H14 H -0.384639 0.017378 0.804564 0.056 Uiso 1 1 calc R U . . .
C15 C -0.4393(2) -0.13154(17) 0.88720(16) 0.0519(4) Uani 1 1 d . . .
. .
H15 H -0.527955 -0.180193 0.822803 0.062 Uiso 1 1 calc R U . . .
C16 C -0.39213(19) -0.17550(15) 0.99405(16) 0.0479(3) Uani 1 1 d . .
. . .
C17 C -0.26099(19) -0.10445(17) 1.09089(15) 0.0512(4) Uani 1 1 d . .
. . .
H17 H -0.230784 -0.134966 1.162952 0.061 Uiso 1 1 calc R U . . .
C18 C -0.17483(18) 0.01309(16) 1.07935(14) 0.0470(3) Uani 1 1 d . .
. . .
H18 H -0.086316 0.061128 1.144172 0.056 Uiso 1 1 calc R U . . .
C3' C -0.12167(16) 0.18103(13) 0.95728(12) 0.0364(3) Uani 1 1 d . .
. . .
C4' C -0.13050(17) 0.23110(14) 0.84815(13) 0.0412(3) Uani 1 1 d . .
. . .
H4' H -0.205061 0.198130 0.770221 0.049 Uiso 1 1 calc R U . . .
C5' C -0.00766(16) 0.33628(13) 0.88244(12) 0.0375(3) Uani 1 1 d . .
. . .

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 _atom_site_aniso_U_23
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Cl1 0.0829(3) 0.0519(3) 0.0969(4) 0.0296(2) 0.0388(3) -0.0020(2)
O2 0.0713(7) 0.0371(5) 0.0523(6) 0.0202(4) 0.0400(5) 0.0188(5)
O3 0.0619(6) 0.0442(5) 0.0469(5) 0.0229(4) 0.0244(5) 0.0092(5)
O1 0.0552(6) 0.0460(6) 0.0385(5) 0.0112(4) 0.0104(4) -0.0039(5)
N1 0.0593(8) 0.0462(7) 0.0383(6) 0.0142(5) 0.0125(5) -0.0011(6)
C1 0.0369(6) 0.0368(6) 0.0381(6) 0.0162(5) 0.0113(5) 0.0040(5)
C2 0.0374(6) 0.0310(6) 0.0430(7) 0.0132(5) 0.0105(5) 0.0061(5)
C3 0.0426(7) 0.0311(6) 0.0419(7) 0.0099(5) 0.0169(5) 0.0094(5)
C4 0.0399(6) 0.0321(6) 0.0386(6) 0.0131(5) 0.0171(5) 0.0069(5)
C5 0.0406(6) 0.0299(6) 0.0393(6) 0.0106(5) 0.0156(5) 0.0059(5)
C6 0.0454(7) 0.0336(6) 0.0384(6) 0.0101(5) 0.0188(5) 0.0079(5)
C7 0.0669(10) 0.0328(7) 0.0617(9) 0.0187(6) 0.0217(8) 0.0110(7)
```

```
C8 0.0575(8) 0.0285(6) 0.0482(7) 0.0121(5) 0.0260(6) 0.0069(6)
C9 0.0734(12) 0.0468(10) 0.1221(18) 0.0408(11) 0.0176(12) 0.0165(9)
C10 0.1327(19) 0.0367(8) 0.0601(11) 0.0036(7) 0.0299(12) 0.0092(10)
C11 0.0491(7) 0.0350(6) 0.0403(7) 0.0089(5) 0.0219(6) 0.0073(5)
C12 0.0555(9) 0.0589(9) 0.0395(7) 0.0183(6) 0.0183(6) 0.0054(7)
C13 0.0411(7) 0.0385(6) 0.0397(6) 0.0128(5) 0.0191(5) 0.0114(5)
C14 0.0499(8) 0.0488(8) 0.0430(7) 0.0168(6) 0.0131(6) 0.0069(6)
C15 0.0503(8) 0.0490(8) 0.0535(8) 0.0120(7) 0.0140(7) 0.0001(7)
C16 0.0506(8) 0.0405(7) 0.0629(9) 0.0188(7) 0.0288(7) 0.0102(6)
C17 0.0535(8) 0.0576(9) 0.0554(9) 0.0304(7) 0.0222(7) 0.0146(7)
C18 0.0456(8) 0.0539(8) 0.0455(7) 0.0203(6) 0.0137(6) 0.0076(6)
C3' 0.0404(7) 0.0376(6) 0.0364(6) 0.0107(5) 0.0176(5) 0.0104(5)
C4' 0.0459(7) 0.0435(7) 0.0344(6) 0.0112(5) 0.0126(5) 0.0036(6)
C5' 0.0447(7) 0.0371(6) 0.0347(6) 0.0088(5) 0.0179(5) 0.0098(5)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

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O2 C4 1.3897(15) . ?
O2 C11 1.4270(16) . ?
O3 C1 1.3807(16) . ?
O3 C12 1.4275(19) . ?
O1 C5' 1.3553(18) . ?
O1 N1 1.4073(16) . ?
N1 C3' 1.3161(19) . ?
C1 C6 1.3897(19) . ?
C1 C2 1.4020(19) . ?
C2 C3 1.3941(19) . ?
C2 C7 1.506(2) . ?
C3 C4 1.3951(19) . ?
C3 H3 0.9300 . ?
C4 C5 1.4001(18) . ?
C5 C6 1.4017(18) . ?
C5 C8 1.5229(19) . ?
C6 H6 0.9300 . ?
C7 H7A 0.9600 . ?
```

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C7 H7B 0.9600 . ?
C7 H7C 0.9600 . ?
C8 C9 1.516(3) . ?
C8 C10 1.524(2) . ?
C8 H8 0.9800 . ?
C9 H9A 0.9600 . ?
C9 H9B 0.9600 . ?
C9 H9C 0.9600 . ?
C10 H10A 0.9600 . ?
C10 H10B 0.9600 . ?
C10 H10C 0.9600 . ?
C11 C5' 1.4932(19) . ?
C11 H11A 0.9700 . ?
C11 H11B 0.9700 . ?
C12 H12A 0.9600 . ?
C12 H12B 0.9600 . ?
C12 H12C 0.9600 . ?
C13 C14 1.390(2) . ?
C13 C18 1.399(2) . ?
C13 C3' 1.4786(19) . ?
C14 C15 1.395(2) . ?
C14 H14 0.9300 . ?
C15 C16 1.374(2) . ?
C15 H15 0.9300 . ?
C16 C17 1.382(2) . ?
C17 C18 1.389(2) . ?
C17 H17 0.9300 . ?
C18 H18 0.9300 . ?
C3' C4' 1.4281(19) . ?
C4' C5' 1.345(2) . ?
C4' H4' 0.9300 . ?

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C4 O2 C11 117.16(11) . . ?
C1 O3 C12 117.79(12) . . ?
C5' O1 N1 108.67(11) . . ?
C3' N1 O1 105.92(11) . . ?
O3 C1 C6 124.16(12) . . ?
O3 C1 C2 115.64(12) . . ?
C6 C1 C2 120.19(11) . . ?
C3 C2 C1 118.45(12) . . ?
C3 C2 C7 121.29(12) . . ?
C1 C2 C7 120.26(12) . . ?
C2 C3 C4 121.21(12) . . ?
C2 C3 H3 119.4 . . ?
C4 C3 H3 119.4 . . ?
O2 C4 C3 123.56(11) . . ?
O2 C4 C5 115.77(11) . . ?
C3 C4 C5 120.68(11) . . ?
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C4 C5 C6 117.77(12) . . ?
C4 C5 C8 120.77(11) . . ?
C6 C5 C8 121.44(11) . . ?
C1 C6 C5 121.71(12) . . ?
C1 C6 H6 119.1 . . ?
C5 C6 H6 119.1 . . ?
C2 C7 H7A 109.5 . . ?
C2 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C2 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C9 C8 C5 110.56(12) . . ?
C9 C8 C10 112.36(16) . . ?
C5 C8 C10 113.13(13) . . ?
C9 C8 H8 106.8 . . ?
C5 C8 H8 106.8 . . ?
C10 C8 H8 106.8 . . ?
C8 C9 H9A 109.5 . . ?
C8 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C8 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
C8 C10 H10A 109.5 . . ?
C8 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C8 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
O2 C11 C5' 106.88(11) . . ?
O2 C11 H11A 110.3 . . ?
C5' C11 H11A 110.3 . . ?
O2 C11 H11B 110.3 . . ?
C5' C11 H11B 110.3 . . ?
H11A C11 H11B 108.6 . . ?
O3 C12 H12A 109.5 . . ?
O3 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
O3 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
C14 C13 C18 118.37(14) . . ?
C14 C13 C3' 121.15(12) . . ?
C18 C13 C3' 120.41(13) . . ?
C13 C14 C15 121.02(14) . . ?
C13 C14 H14 119.5 . . ?
C15 C14 H14 119.5 . . ?
C16 C15 C14 119.22(15) . . ?
C16 C15 H15 120.4 . . ?
C14 C15 H15 120.4 . . ?
C15 C16 C17 121.26(14) . . ?
C15 C16 Cl1 119.11(13) . . ?
C17 C16 Cl1 119.63(13) . . ?
C16 C17 C18 119.23(14) . . ?
C16 C17 H17 120.4 . . ?
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C18 C17 H17 120.4 . . ?
C17 C18 C13 120.90(15) . . ?
C17 C18 H18 119.5 . . ?
C13 C18 H18 119.5 . . ?
N1 C3' C4' 110.68(12) . . ?
N1 C3' C13 120.30(12) . . ?
C4' C3' C13 128.93(12) . . ?
C5' C4' C3' 105.10(12) . . ?
C5' C4' H4' 127.5 . . ?
C3' C4' H4' 127.5 . . ?
C4' C5' O1 109.61(12) . . ?
C4' C5' C11 134.07(13) . . ?
O1 C5' C11 116.28(12) . . ?

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C11 H11A O3  0.97 2.60 3.454(2) 146.4 2_566 yes

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_refine_diff_density_rms    0.039

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TITL mo_D19_49_Kettatni_LY7_0m_a.res in P-1
    shelx.res
    created by SHELXL-2018/3 at 23:39:44 on 06-Sep-2020
CELL  0.71073   8.7916  10.2170  11.3158  102.891  103.508   96.933
ZERR     2.00   0.0042   0.0053   0.0052    0.018    0.016    0.019
LATT   1
SFAC  C    H    N    O    CL
UNIT  42 44 2 6 2
MERG   2
OMIT    -1   1   2
SHEL 50 0.70
HTAB C11 O3_$1
eqiv $1  -x, 1-y, 1-z
FMAP   2
PLAN   10
BOND $H
ACTA
L.S.  10
WGHT    0.065700    0.206300
EXTI    0.011481
FVAR       0.78331
MOLE    1
CL1   5   -0.499230   -0.324998    1.006312    11.00000    0.08289
0.05193 =
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```
         0.09686    0.02957    0.03878   -0.00197
O2    4    0.126438    0.348369    0.727572    11.00000    0.07129
0.03713 =
         0.05234    0.02021    0.03996    0.01884
O3    4    0.315434    0.574094    0.369998    11.00000    0.06185
0.04421 =
         0.04691    0.02294    0.02438    0.00920
O1    4    0.071634    0.354108    1.004606    11.00000    0.05523
0.04599 =
         0.03849    0.01120    0.01035   -0.00393
N1    3   -0.002657    0.254894    1.051635    11.00000    0.05934
0.04617 =
         0.03831    0.01415    0.01245   -0.00111
C1    1    0.272086    0.514332    0.458148    11.00000    0.03689
0.03678 =
         0.03807    0.01617    0.01131    0.00403
C2    1    0.227322    0.601079    0.553735    11.00000    0.03740
0.03105 =
         0.04300    0.01319    0.01054    0.00607
C3    1    0.179258    0.546310    0.644312    11.00000    0.04260
0.03109 =
         0.04192    0.00987    0.01691    0.00938
AFIX  43
H3    2    0.149474    0.602682    0.708395    11.00000   -1.20000
AFIX   0
C4    1    0.175020    0.408476    0.640584    11.00000    0.03991
0.03207 =
         0.03864    0.01314    0.01708    0.00686
C5    1    0.220319    0.321379    0.545796    11.00000    0.04062
0.02987 =
         0.03929    0.01064    0.01565    0.00587
C6    1    0.269297    0.377398    0.455471    11.00000    0.04544
0.03357 =
         0.03843    0.01010    0.01878    0.00794
AFIX  43
H6    2    0.300742    0.321479    0.392107    11.00000   -1.20000
AFIX   0
C7    1    0.230195    0.749506    0.556728    11.00000    0.06693
0.03279 =
         0.06174    0.01868    0.02165    0.01098
AFIX 137
H7A   2    0.195768    0.793726    0.627484    11.00000   -1.50000
H7B   2    0.160153    0.756785    0.480422    11.00000   -1.50000
H7C   2    0.336494    0.792603    0.564186    11.00000   -1.50000
AFIX   0
C8    1    0.220325    0.171817    0.543675    11.00000    0.05748
0.02847 =
         0.04824    0.01206    0.02598    0.00693
AFIX  13
H8    2    0.147396    0.147445    0.592133    11.00000   -1.20000
AFIX   0
C9    1    0.383492    0.152730    0.610966    11.00000    0.07336
0.04685 =
         0.12213    0.04081    0.01762    0.01654
AFIX 137
H9A   2    0.458347    0.173369    0.565113    11.00000   -1.50000
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H9B   2    0.378823    0.059861    0.616089    11.00000   -1.50000
H9C   2    0.416612    0.212876    0.694299    11.00000   -1.50000
AFIX   0
C10   1    0.157791    0.076077    0.411714    11.00000    0.13270
0.03667 =
         0.06008    0.00357    0.02990    0.00917
AFIX 137
H10A  2    0.058393    0.096425    0.370429    11.00000   -1.50000
H10B  2    0.142182   -0.016748    0.417293    11.00000   -1.50000
H10C  2    0.233417    0.088259    0.364317    11.00000   -1.50000
AFIX   0
C11   1    0.058938    0.429191    0.814782    11.00000    0.04908
0.03499 =
         0.04033    0.00887    0.02190    0.00733
AFIX  23
H11A  2   -0.024489    0.468740    0.770671    11.00000   -1.20000
H11B  2    0.139797    0.502615    0.873914    11.00000   -1.20000
AFIX   0
C12   1    0.369790    0.491796    0.274419    11.00000    0.05549
0.05892 =
         0.03949    0.01830    0.01832    0.00544
AFIX 137
H12A  2    0.393251    0.544419    0.218302    11.00000   -1.50000
H12B  2    0.288526    0.413797    0.228064    11.00000   -1.50000
H12C  2    0.464147    0.461826    0.312615    11.00000   -1.50000
AFIX   0
C13   1   -0.219355    0.060186    0.971592    11.00000    0.04106
0.03853 =
         0.03966    0.01280    0.01914    0.01141
C14   1   -0.352689   -0.013063    0.876468    11.00000    0.04994
0.04884 =
         0.04296    0.01680    0.01314    0.00691
AFIX  43
H14   2   -0.384639    0.017378    0.804564    11.00000   -1.20000
AFIX   0
C15   1   -0.439318   -0.131537    0.887196    11.00000    0.05027
0.04900 =
         0.05355    0.01204    0.01396    0.00014
AFIX  43
H15   2   -0.527955   -0.180193    0.822803    11.00000   -1.20000
AFIX   0
C16   1   -0.392127   -0.175497    0.994048    11.00000    0.05057
0.04052 =
         0.06289    0.01882    0.02882    0.01018
C17   1   -0.260991   -0.104445    1.090895    11.00000    0.05353
0.05759 =
         0.05541    0.03036    0.02221    0.01458
AFIX  43
H17   2   -0.230784   -0.134966    1.162952    11.00000   -1.20000
AFIX   0
C18   1   -0.174826    0.013094    1.079353    11.00000    0.04560
0.05394 =
         0.04546    0.02026    0.01365    0.00765
AFIX  43
H18   2   -0.086316    0.061128    1.144172    11.00000   -1.20000
AFIX   0
```

```
C3'   1   -0.121674    0.181030    0.957284    11.00000    0.04041
0.03761 =
         0.03638    0.01072    0.01757    0.01042
C4'   1   -0.130503    0.231105    0.848152    11.00000    0.04591
0.04354 =
         0.03443    0.01123    0.01261    0.00362
AFIX  43
H4'   2   -0.205061    0.198130    0.770221    11.00000   -1.20000
AFIX   0
C5'   1   -0.007661    0.336282    0.882445    11.00000    0.04468
0.03707 =
         0.03472    0.00880    0.01788    0.00981
HKLF    4



REM  mo_D19_49_Kettatni_LY7_0m_a.res in P-1
REM wR2 = 0.1380, GooF = S = 1.026, Restrained GooF = 1.026 for all
data
REM R1 = 0.0460 for 4096 Fo > 4sig(Fo) and 0.0706 for all 5774 data
REM 240 parameters refined using 0 restraints

END

WGHT      0.0632      0.2156

REM Highest difference peak  0.291,  deepest hole -0.286,  1-sigma
level  0.039
Q1    1  -0.4813 -0.2739  1.0804  11.00000  0.05    0.29
Q2    1  -0.1323  0.2035  0.8987  11.00000  0.05    0.29
Q3    1   0.2329  0.3538  0.5001  11.00000  0.05    0.27
Q4    1   0.1671  0.4777  0.6385  11.00000  0.05    0.26
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Laboratoire de Chimie Appliqu\'ee des Mat\'riaux, Centre des
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Avenue Ibn Batouta, B.P. 1014, Rabat, Morocco
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Please consider this CIF submission for publication in
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Journal of Molecular Structure.
All required files have been provided.

The manuscript has passed the checkcif tests and generates an acceptable printcif output.

Sincerely yours

Lahcen El Ammari
;

Loop of author details

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;
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.

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#Added by publCIF



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2020-05-28 # Formatted by publCIF

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Introduction
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 section
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Experimental
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;

;
 subsection
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Synthesis and crystallization
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;

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 subsection
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Refinement
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Crystal data, data collection and structure refinement details are
summarized
in Table 1.
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 section
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Results and discussion
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The symmetry employed for this shelxl refinement is uniquely defined
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They are only intended as comments.
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 Reflections were merged by SHELXL according to the crystal
 class for the calculation of statistics and refinement.

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2012)'

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C8 C 1.1983(3) 0.1526(2) 0.0457(2) 0.0592(7) Uani 1 1 d . . . . .
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C11 C 1.0464(3) 0.3709(2) 0.3414(2) 0.0601(7) Uani 1 1 d . . . . .
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C17 C 0.2247(3) 0.0242(3) 0.3889(2) 0.0657(7) Uani 1 1 d . . . . .
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C4' C 0.7922(3) 0.1892(2) 0.3131(2) 0.0557(6) Uani 1 1 d . . . . .
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0.0002(12)
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C9 0.103(3) 0.081(2) 0.122(3) 0.009(2) -0.042(2) -0.0005(19)
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 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
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O2N N2 O1N 123.8(3) . . ?
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O3 C1 C6 123.9(2) . . ?
O3 C1 C2 115.5(2) . . ?
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C4 C3 H3 119.4 . . ?
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C2 C7 H7B 109.5 . . ?
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C10 C8 H8 107.5 . . ?
C5 C8 H8 107.5 . . ?
C9 C8 H8 107.5 . . ?
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C8 C9 H9B 109.5 . . ?
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SFAC  C    H    N    O
UNIT  42 44 4 10
MERG   2
OMIT    -8   4   0
SHEL 50 0.82
HTAB C11 O1N_$1
EQIV $1 1+x,1+y,z
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PLAN   10
ACTA
BOND   $H
L.S.  10
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0.05878 =
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O3    4    1.718405    0.458257    0.123819    11.00000    0.05032
0.08838 =
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O1N   4    0.096763   -0.309551    0.277771    11.00000    0.09308
0.06826 =
         0.14346    0.01742   -0.01637   -0.01825
O2N   4   -0.022970   -0.175373    0.385986    11.00000    0.07629
0.14267 =
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N1    3    0.656563    0.292239    0.450324    11.00000    0.05721
0.05614 =
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N2    3    0.092599   -0.202503    0.335613    11.00000    0.06785
0.08524 =
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C1    1    1.564173    0.415733    0.150479    11.00000    0.04165
0.06139 =
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C2    1    1.514729    0.485697    0.250312    11.00000    0.04747
0.04997 =
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C3    1    1.359785    0.446752    0.281704    11.00000    0.05201
0.05125 =
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C5    1    1.308474    0.268918    0.117225    11.00000    0.04631
0.05138 =
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C6    1    1.463099    0.309601    0.086019    11.00000    0.05200
0.06673 =
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C7    1    1.626123    0.599270    0.322797    11.00000    0.05981
0.05946 =
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H7B   2    1.577777    0.628952    0.393891    11.00000   -1.50000
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0.08061 =
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H9B   2    1.062895    0.255097   -0.053997    11.00000   -1.50000
H9C   2    1.056552    0.111074   -0.115255    11.00000   -1.50000
AFIX   0
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0.06389 =
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H10B  2    1.328506    0.014474    0.093697    11.00000   -1.50000
H10C  2    1.362630    0.052754   -0.033431    11.00000   -1.50000
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C11   1    1.046399    0.370885    0.341351    11.00000    0.05390
0.05210 =
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AFIX  23
H11A  2    1.038692    0.457364    0.321929    11.00000   -1.20000
H11B  2    1.119013    0.379365    0.414550    11.00000   -1.20000
AFIX   0
C12   1    1.769155    0.400604    0.016296    11.00000    0.06985
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AFIX 137
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H12B  2    1.692736    0.406031   -0.050390    11.00000   -1.50000
H12C  2    1.775718    0.310821    0.021327    11.00000   -1.50000
AFIX   0
C13   1    0.505310    0.086832    0.360209    11.00000    0.04841
0.05038 =
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C14   1    0.511573   -0.041275    0.312607    11.00000    0.05569
0.05335 =
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C5'   1    0.884664    0.302373    0.358675    11.00000    0.05192
0.04711 =
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REM  mo_D19_54_Kettatni_LY9_0m_a.res in P-1
REM wR2 = 0.1741, GooF = S = 1.060, Restrained GooF = 1.060 for all
data
REM R1 = 0.0601 for 2375 Fo > 4sig(Fo) and 0.0912 for all 3656 data
REM 258 parameters refined using 0 restraints

END

WGHT      0.0745      0.3657

REM Highest difference peak  0.168,  deepest hole -0.211,  1-sigma
level  0.043
Q1    1   0.2320 -0.0339  0.3402  11.00000  0.05    0.17
Q2    1   0.5056  0.0516  0.3215  11.00000  0.05    0.17
Q3    1   0.5954  0.1427  0.3560  11.00000  0.05    0.16
Q4    1   0.4519  0.1099  0.3672  11.00000  0.05    0.16
Q5    1   0.5040  0.0284  0.3738  11.00000  0.05    0.15
Q6    1   0.0889 -0.3027  0.3960  11.00000  0.05    0.15
Q7    1   1.6978  0.5563  0.4245  11.00000  0.05    0.15
Q8    1   0.9281  0.4686  0.4904  11.00000  0.05    0.15
Q9    1   1.1258  0.2954 -0.0861  11.00000  0.05    0.14
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Journal of Molecular Structure.
All required files have been provided.

The manuscript has passed the checkcif tests and generates an
acceptable printcif output.

Sincerely yours

Lahcen El Ammari
;



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5-((2-isopropyl-4-methoxy-5-methylphenoxy)methyl)-3-(2-
methoxyphenyl)isoxazole
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#Added by publCIF



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2020-05-28 # Formatted by publCIF

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Introduction
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Experimental
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 subsection
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Synthesis and crystallization
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 subsection
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Refinement
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Crystal data, data collection and structure refinement details are
summarized
in Table 1.
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 section
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Results and discussion
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The symmetry employed for this shelxl refinement is uniquely defined
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symmetry information in preference to the above space-group names.
They are only intended as comments.
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_diffrn_measured_fraction_theta_full  0.999
_diffrn_reflns_Laue_measured_fraction_max    0.999
_diffrn_reflns_Laue_measured_fraction_full   0.999
_diffrn_reflns_point_group_measured_fraction_max   0.999
_diffrn_reflns_point_group_measured_fraction_full  0.999
_reflns_number_total              3828
_reflns_number_gt                 2640
_reflns_threshold_expression      'I > 2\s(I)'
_reflns_Friedel_coverage          0.000
_reflns_Friedel_fraction_max      .
_reflns_Friedel_fraction_full     .

_reflns_special_details
;
 Reflections were merged by SHELXL according to the crystal
 class for the calculation of statistics and refinement.

 _reflns_Friedel_fraction is defined as the number of unique
 Friedel pairs measured divided by the number that would be
 possible theoretically, ignoring centric projections and
 systematic absences.
;

_computing_data_collection        'APEX3 (Bruker, 2016)'
_computing_cell_refinement        'SAINT (Bruker, 2016)'
_computing_data_reduction         SAINT
_computing_structure_solution     'SHELXT 2014/5 (Sheldrick, 2014)'
_computing_structure_refinement 'SHELXL-2018/3 (Sheldrick, 2018)'
_computing_molecular_graphics     'DIAMOND (Brandenburg et al.,
2012)'

_computing_publication_material   ?
_refine_special_details           ?
_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
'w=1/[\s^2^(Fo^2^)+(0.0532P)^2^+0.3344P] where P=(Fo^2^+2Fc^2^)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     constr
_refine_ls_extinction_method      'SHELXL-2018/3 (Sheldrick 2018)'
_refine_ls_extinction_coef        0.057(5)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          3828
_refine_ls_number_parameters      250
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0765
_refine_ls_R_factor_gt            0.0489
_refine_ls_wR_factor_ref          0.1352
_refine_ls_wR_factor_gt           0.1177
_refine_ls_goodness_of_fit_ref    1.023
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_refine_ls_restrained_S_all      1.023
_refine_ls_shift/su_max          0.000
_refine_ls_shift/su_mean         0.000

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 _atom_site_fract_z
 _atom_site_U_iso_or_equiv
 _atom_site_adp_type
 _atom_site_occupancy
 _atom_site_site_symmetry_order
 _atom_site_calc_flag
 _atom_site_refinement_flags_posn
 _atom_site_refinement_flags_adp
 _atom_site_refinement_flags_occupancy
 _atom_site_disorder_assembly
 _atom_site_disorder_group
O1 O 0.58317(15) 0.18288(17) 0.91950(15) 0.0678(4) Uani 1 1 d . . .
. .
O2 O 0.59048(18) 0.14058(15) 0.66568(13) 0.0696(5) Uani 1 1 d . . .
. .
O3 O 0.81603(18) -0.10651(16) 0.26229(15) 0.0713(5) Uani 1 1 d . . .
. .
O4 O 0.14742(15) 0.25702(16) 1.10128(15) 0.0646(4) Uani 1 1 d . . .
. .
N1 N 0.51094(18) 0.2747(2) 1.00020(18) 0.0662(5) Uani 1 1 d . . . .
.
C1 C 0.7623(2) -0.0426(2) 0.36246(19) 0.0523(5) Uani 1 1 d . . . . .
C2 C 0.7291(2) -0.1292(2) 0.4732(2) 0.0532(5) Uani 1 1 d . . . . .
C3 C 0.6707(2) -0.0679(2) 0.57469(19) 0.0542(5) Uani 1 1 d . . . . .
H3 H 0.646241 -0.123435 0.649430 0.065 Uiso 1 1 calc R U . . .
C4 C 0.6480(2) 0.0741(2) 0.56753(18) 0.0501(5) Uani 1 1 d . . . . .
C5 C 0.6814(2) 0.1609(2) 0.45727(19) 0.0526(5) Uani 1 1 d . . . . .
C6 C 0.7384(2) 0.1000(2) 0.3550(2) 0.0563(5) Uani 1 1 d . . . . .
H6 H 0.761040 0.156264 0.279528 0.068 Uiso 1 1 calc R U . . .
C7 C 0.7540(3) -0.2848(2) 0.4825(2) 0.0736(7) Uani 1 1 d . . . . .
H7A H 0.717980 -0.324520 0.562761 0.110 Uiso 1 1 calc R U . . .
H7B H 0.854259 -0.345869 0.481132 0.110 Uiso 1 1 calc R U . . .
H7C H 0.705665 -0.283020 0.409653 0.110 Uiso 1 1 calc R U . . .
C8 C 0.6593(3) 0.3150(2) 0.4515(2) 0.0739(7) Uani 1 1 d . . . . .
H8 H 0.579065 0.353910 0.505977 0.089 Uiso 1 1 calc R U . . .
C9 C 0.7897(5) 0.3060(4) 0.5122(4) 0.1394(15) Uani 1 1 d . . . . .
H9A H 0.806647 0.242766 0.600579 0.209 Uiso 1 1 calc R U . . .
H9B H 0.773679 0.403230 0.512134 0.209 Uiso 1 1 calc R U . . .
H9C H 0.870994 0.266122 0.462146 0.209 Uiso 1 1 calc R U . . .
C10 C 0.6229(4) 0.4228(3) 0.3166(3) 0.1112(12) Uani 1 1 d . . . . .
H10A H 0.602818 0.519426 0.321958 0.167 Uiso 1 1 calc R U . . .
H10B H 0.540755 0.426580 0.279862 0.167 Uiso 1 1 calc R U . . .
H10C H 0.701927 0.391260 0.261897 0.167 Uiso 1 1 calc R U . . .
C11 C 0.5460(2) 0.0615(2) 0.77836(18) 0.0539(5) Uani 1 1 d . . . . .
H11A H 0.625915 -0.030482 0.821985 0.065 Uiso 1 1 calc R U . . .
H11B H 0.473437 0.038423 0.754269 0.065 Uiso 1 1 calc R U . . .
C12 C 0.8566(3) -0.0259(3) 0.1501(2) 0.0802(8) Uani 1 1 d . . . . .
```

```
H12A H 0.883896 -0.078881 0.085428 0.120 Uiso 1 1 calc R U . . .
H12B H 0.935688 -0.012751 0.172432 0.120 Uiso 1 1 calc R U . . .
H12C H 0.778091 0.069215 0.115457 0.120 Uiso 1 1 calc R U . . .
C13 C 0.2729(2) 0.3997(2) 1.06321(18) 0.0492(5) Uani 1 1 d . . . . .
C14 C 0.2904(2) 0.5173(2) 1.0795(2) 0.0648(6) Uani 1 1 d . . . . .
H14 H 0.367318 0.534487 1.043195 0.078 Uiso 1 1 calc R U . . .
C15 C 0.1959(3) 0.6088(3) 1.1486(3) 0.0796(7) Uani 1 1 d . . . . .
H15 H 0.209096 0.686871 1.159225 0.095 Uiso 1 1 calc R U . . .
C16 C 0.0822(3) 0.5838(3) 1.2014(2) 0.0793(7) Uani 1 1 d . . . . .
H16 H 0.018382 0.645453 1.248173 0.095 Uiso 1 1 calc R U . . .
C17 C 0.0610(2) 0.4692(3) 1.1863(2) 0.0675(6) Uani 1 1 d . . . . .
H17 H -0.017286 0.454208 1.221897 0.081 Uiso 1 1 calc R U . . .
C18 C 0.1567(2) 0.3760(2) 1.11800(19) 0.0535(5) Uani 1 1 d . . . . .
C19 C 0.0407(3) 0.2170(3) 1.1666(3) 0.0902(8) Uani 1 1 d . . . . .
H19A H -0.051490 0.292300 1.130132 0.135 Uiso 1 1 calc R U . . .
H19B H 0.046830 0.207304 1.258180 0.135 Uiso 1 1 calc R U . . .
H19C H 0.054632 0.124379 1.155681 0.135 Uiso 1 1 calc R U . . .
C3' C 0.37747(19) 0.3016(2) 0.99125(17) 0.0454(4) Uani 1 1 d . . . .
.
C4' C 0.3589(2) 0.2292(2) 0.90718(18) 0.0475(5) Uani 1 1 d . . . . .
H4' H 0.274341 0.230576 0.885464 0.057 Uiso 1 1 calc R U . . .
C5' C 0.4881(2) 0.1585(2) 0.86547(18) 0.0476(5) Uani 1 1 d . . . . .

loop_
 _atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
O1 0.0553(8) 0.0815(11) 0.0782(10) -0.0442(9) 0.0200(7) -0.0297(8)
O2 0.1171(12) 0.0482(8) 0.0510(8) -0.0230(7) 0.0377(8) -0.0435(8)
O3 0.1017(12) 0.0651(9) 0.0657(10) -0.0408(8) 0.0321(9) -0.0440(9)
O4 0.0630(9) 0.0705(10) 0.0739(10) -0.0293(8) 0.0245(7) -0.0398(8)
N1 0.0575(10) 0.0836(13) 0.0746(12) -0.0471(11) 0.0177(9) -
0.0332(10)
C1 0.0623(12) 0.0486(11) 0.0532(12) -0.0265(10) 0.0138(9) -0.0250(9)
C2 0.0671(12) 0.0396(10) 0.0551(12) -0.0184(9) 0.0026(10) -0.0223(9)
C3 0.0745(13) 0.0415(10) 0.0460(11) -0.0100(9) 0.0086(10) -
0.0276(10)
C4 0.0648(12) 0.0421(10) 0.0462(11) -0.0194(9) 0.0159(9) -0.0242(9)
C5 0.0701(13) 0.0432(10) 0.0502(11) -0.0201(9) 0.0186(10) -
0.0286(10)
C6 0.0754(13) 0.0515(11) 0.0509(12) -0.0214(10) 0.0220(10) -
0.0347(10)
C7 0.1104(19) 0.0450(12) 0.0686(15) -0.0213(11) 0.0037(13) -
0.0335(12)
C8 0.122(2) 0.0503(12) 0.0626(14) -0.0285(11) 0.0450(14) -0.0492(13)
C9 0.229(4) 0.113(3) 0.137(3) -0.050(2) -0.003(3) -0.116(3)
C10 0.185(3) 0.0498(14) 0.088(2) -0.0160(14) 0.032(2) -0.0488(18)
C11 0.0712(13) 0.0433(10) 0.0459(11) -0.0125(9) 0.0171(10) -
0.0268(10)
C12 0.114(2) 0.0804(16) 0.0641(15) -0.0405(13) 0.0354(14) -
0.0513(15)
C13 0.0558(11) 0.0495(11) 0.0437(11) -0.0176(9) 0.0071(9) -0.0223(9)
```

```
C14 0.0730(14) 0.0625(13) 0.0701(15) -0.0316(12) 0.0127(11) -
0.0332(11)
C15 0.0963(19) 0.0673(15) 0.0830(17) -0.0420(14) 0.0126(15) -
0.0313(14)
C16 0.0832(17) 0.0722(16) 0.0718(16) -0.0404(13) 0.0159(13) -
0.0147(13)
C17 0.0585(13) 0.0742(15) 0.0598(14) -0.0245(12) 0.0154(11) -
0.0195(11)
C18 0.0533(11) 0.0558(12) 0.0476(11) -0.0167(9) 0.0067(9) -0.0202(9)
C19 0.0814(17) 0.118(2) 0.101(2) -0.0426(18) 0.0351(15) -0.0680(17)
C3' 0.0519(11) 0.0441(10) 0.0430(10) -0.0121(8) 0.0101(8) -0.0251(9)
C4' 0.0551(11) 0.0482(10) 0.0446(10) -0.0158(9) 0.0103(8) -0.0270(9)
C5' 0.0604(12) 0.0439(10) 0.0410(10) -0.0103(8) 0.0103(9) -0.0276(9)

_geom_special_details
;
 All esds (except the esd in the dihedral angle between two l.s.
planes)
 are estimated using the full covariance matrix.  The cell esds are
taken
 into account individually in the estimation of esds in distances,
angles
 and torsion angles; correlations between esds in cell parameters
are only
 used when they are defined by crystal symmetry.  An approximate
(isotropic)
 treatment of cell esds is used for estimating esds involving l.s.
planes.
;

loop_
 _geom_bond_atom_site_label_1
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 _geom_bond_distance
 _geom_bond_site_symmetry_2
 _geom_bond_publ_flag
O1 C5' 1.347(2) . ?
O1 N1 1.406(2) . ?
O2 C4 1.378(2) . ?
O2 C11 1.418(2) . ?
O3 C1 1.378(2) . ?
O3 C12 1.401(3) . ?
O4 C18 1.360(2) . ?
O4 C19 1.420(2) . ?
N1 C3' 1.306(2) . ?
C1 C6 1.383(3) . ?
C1 C2 1.386(3) . ?
C2 C3 1.383(3) . ?
C2 C7 1.511(3) . ?
C3 C4 1.382(3) . ?
C3 H3 0.9300 . ?
C4 C5 1.384(3) . ?
C5 C6 1.386(3) . ?
C5 C8 1.515(3) . ?
C6 H6 0.9300 . ?
C7 H7A 0.9600 . ?
```

```
C7 H7B 0.9600 . ?
C7 H7C 0.9600 . ?
C8 C10 1.504(3) . ?
C8 C9 1.521(4) . ?
C8 H8 0.9800 . ?
C9 H9A 0.9600 . ?
C9 H9B 0.9600 . ?
C9 H9C 0.9600 . ?
C10 H10A 0.9600 . ?
C10 H10B 0.9600 . ?
C10 H10C 0.9600 . ?
C11 C5' 1.480(3) . ?
C11 H11A 0.9700 . ?
C11 H11B 0.9700 . ?
C12 H12A 0.9600 . ?
C12 H12B 0.9600 . ?
C12 H12C 0.9600 . ?
C13 C14 1.388(3) . ?
C13 C18 1.394(3) . ?
C13 C3' 1.474(2) . ?
C14 C15 1.377(3) . ?
C14 H14 0.9300 . ?
C15 C16 1.369(3) . ?
C15 H15 0.9300 . ?
C16 C17 1.374(3) . ?
C16 H16 0.9300 . ?
C17 C18 1.386(3) . ?
C17 H17 0.9300 . ?
C19 H19A 0.9600 . ?
C19 H19B 0.9600 . ?
C19 H19C 0.9600 . ?
C3' C4' 1.413(3) . ?
C4' C5' 1.334(2) . ?
C4' H4' 0.9300 . ?

loop_
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 _geom_angle_site_symmetry_3
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C5' O1 N1 108.55(14) . . ?
C4 O2 C11 118.20(14) . . ?
C1 O3 C12 118.05(16) . . ?
C18 O4 C19 118.57(17) . . ?
C3' N1 O1 105.65(15) . . ?
O3 C1 C6 123.55(18) . . ?
O3 C1 C2 115.72(16) . . ?
C6 C1 C2 120.71(17) . . ?
C3 C2 C1 117.75(17) . . ?
C3 C2 C7 120.94(19) . . ?
C1 C2 C7 121.31(18) . . ?
C4 C3 C2 121.48(18) . . ?
C4 C3 H3 119.3 . . ?
```

```
C2 C3 H3 119.3 . . ?
O2 C4 C3 123.93(16) . . ?
O2 C4 C5 115.11(16) . . ?
C3 C4 C5 120.96(17) . . ?
C4 C5 C6 117.52(17) . . ?
C4 C5 C8 120.60(17) . . ?
C6 C5 C8 121.87(17) . . ?
C1 C6 C5 121.58(18) . . ?
C1 C6 H6 119.2 . . ?
C5 C6 H6 119.2 . . ?
C2 C7 H7A 109.5 . . ?
C2 C7 H7B 109.5 . . ?
H7A C7 H7B 109.5 . . ?
C2 C7 H7C 109.5 . . ?
H7A C7 H7C 109.5 . . ?
H7B C7 H7C 109.5 . . ?
C10 C8 C5 114.4(2) . . ?
C10 C8 C9 110.8(2) . . ?
C5 C8 C9 109.6(2) . . ?
C10 C8 H8 107.3 . . ?
C5 C8 H8 107.3 . . ?
C9 C8 H8 107.3 . . ?
C8 C9 H9A 109.5 . . ?
C8 C9 H9B 109.5 . . ?
H9A C9 H9B 109.5 . . ?
C8 C9 H9C 109.5 . . ?
H9A C9 H9C 109.5 . . ?
H9B C9 H9C 109.5 . . ?
C8 C10 H10A 109.5 . . ?
C8 C10 H10B 109.5 . . ?
H10A C10 H10B 109.5 . . ?
C8 C10 H10C 109.5 . . ?
H10A C10 H10C 109.5 . . ?
H10B C10 H10C 109.5 . . ?
O2 C11 C5' 106.60(15) . . ?
O2 C11 H11A 110.4 . . ?
C5' C11 H11A 110.4 . . ?
O2 C11 H11B 110.4 . . ?
C5' C11 H11B 110.4 . . ?
H11A C11 H11B 108.6 . . ?
O3 C12 H12A 109.5 . . ?
O3 C12 H12B 109.5 . . ?
H12A C12 H12B 109.5 . . ?
O3 C12 H12C 109.5 . . ?
H12A C12 H12C 109.5 . . ?
H12B C12 H12C 109.5 . . ?
C14 C13 C18 118.57(18) . . ?
C14 C13 C3' 119.99(18) . . ?
C18 C13 C3' 121.43(17) . . ?
C15 C14 C13 121.2(2) . . ?
C15 C14 H14 119.4 . . ?
C13 C14 H14 119.4 . . ?
C16 C15 C14 119.3(2) . . ?
C16 C15 H15 120.3 . . ?
C14 C15 H15 120.3 . . ?
C15 C16 C17 121.1(2) . . ?
```

```
C15 C16 H16 119.5 . . ?
C17 C16 H16 119.5 . . ?
C16 C17 C18 119.7(2) . . ?
C16 C17 H17 120.1 . . ?
C18 C17 H17 120.1 . . ?
O4 C18 C17 124.16(19) . . ?
O4 C18 C13 115.71(17) . . ?
C17 C18 C13 120.1(2) . . ?
O4 C19 H19A 109.5 . . ?
O4 C19 H19B 109.5 . . ?
H19A C19 H19B 109.5 . . ?
O4 C19 H19C 109.5 . . ?
H19A C19 H19C 109.5 . . ?
H19B C19 H19C 109.5 . . ?
N1 C3' C4' 110.84(16) . . ?
N1 C3' C13 118.54(17) . . ?
C4' C3' C13 130.60(17) . . ?
C5' C4' C3' 105.53(17) . . ?
C5' C4' H4' 127.2 . . ?
C3' C4' H4' 127.2 . . ?
C4' C5' O1 109.42(16) . . ?
C4' C5' C11 134.48(19) . . ?
O1 C5' C11 116.08(16) . . ?

loop_
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 _geom_hbond_atom_site_label_H
 _geom_hbond_atom_site_label_A
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 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
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C11 H11A O4  0.97 2.49 3.445(3) 170.1 2_657 yes
C16 H16 O3  0.93 2.57 3.418(3) 152.1 1_466 yes

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_refine_diff_density_min   -0.135
_refine_diff_density_rms    0.032

_shelx_res_file
;
TITL mo_D19_50_Kettatni_LY8_0m_a.res in P-1
    shelx.res
    created by SHELXL-2018/3 at 22:42:14 on 06-Sep-2020
CELL  0.71073  10.4871  10.5128  10.7032   72.751   84.704   63.683
ZERR     2.00   0.0005   0.0005   0.0006    0.003    0.004    0.002
LATT   1
SFAC  C    H    N    O
UNIT  44 50 2 8
MERG   2
OMIT     1   0   2
OMIT     0   1   0
SHEL 50 0.82
HTAB C11 O4_$1
```

```
EQIV $1 1-x,-y,2-z
HTAB C16 O3_$2
EQIV $2 -1+x,1+y,1+z
FMAP   2
PLAN    5
ACTA
BOND   $H
L.S.  10
WGHT    0.053200    0.334400
EXTI    0.057305
FVAR       1.14435
MOLE    1
O1    4    0.583171    0.182880    0.919502    11.00000    0.05525
0.08153 =
         0.07817   -0.04415    0.02000   -0.02969
O2    4    0.590475    0.140582    0.665676    11.00000    0.11708
0.04824 =
         0.05103   -0.02295    0.03773   -0.04351
O3    4    0.816035   -0.106508    0.262291    11.00000    0.10170
0.06508 =
         0.06565   -0.04084    0.03206   -0.04397
O4    4    0.147423    0.257018    1.101283    11.00000    0.06302
0.07052 =
         0.07393   -0.02926    0.02453   -0.03981
N1    3    0.510944    0.274728    1.000205    11.00000    0.05754
0.08364 =
         0.07463   -0.04707    0.01767   -0.03322
C1    1    0.762307   -0.042560    0.362458    11.00000    0.06225
0.04858 =
         0.05315   -0.02650    0.01379   -0.02500
C2    1    0.729103   -0.129212    0.473190    11.00000    0.06707
0.03964 =
         0.05510   -0.01840    0.00259   -0.02231
C3    1    0.670733   -0.067891    0.574690    11.00000    0.07449
0.04148 =
         0.04603   -0.00998    0.00863   -0.02756
AFIX  43
H3    2    0.646241   -0.123435    0.649430    11.00000   -1.20000
AFIX   0
C4    1    0.647986    0.074116    0.567527    11.00000    0.06483
0.04212 =
         0.04623   -0.01937    0.01593   -0.02415
C5    1    0.681425    0.160885    0.457272    11.00000    0.07012
0.04322 =
         0.05020   -0.02005    0.01863   -0.02857
C6    1    0.738375    0.099967    0.354988    11.00000    0.07537
0.05146 =
         0.05086   -0.02144    0.02200   -0.03467
AFIX  43
H6    2    0.761040    0.156264    0.279528    11.00000   -1.20000
AFIX   0
C7    1    0.754024   -0.284849    0.482536    11.00000    0.11043
0.04499 =
         0.06860   -0.02130    0.00368   -0.03349
AFIX 137
H7A   2    0.717980   -0.324520    0.562761    11.00000   -1.50000
```

```
H7B   2    0.854259   -0.345869    0.481132    11.00000   -1.50000
H7C   2    0.705665   -0.283020    0.409653    11.00000   -1.50000
AFIX   0
C8    1    0.659271    0.315044    0.451504    11.00000    0.12219
0.05033 =
         0.06264   -0.02845    0.04496   -0.04923
AFIX  13
H8    2    0.579065    0.353910    0.505977    11.00000   -1.20000
AFIX   0
C9    1    0.789666    0.305953    0.512183    11.00000    0.22877
0.11336 =
         0.13673   -0.04989   -0.00320   -0.11633
AFIX 137
H9A   2    0.806647    0.242766    0.600579    11.00000   -1.50000
H9B   2    0.773679    0.403230    0.512134    11.00000   -1.50000
H9C   2    0.870994    0.266122    0.462146    11.00000   -1.50000
AFIX   0
C10   1    0.622904    0.422825    0.316606    11.00000    0.18495
0.04981 =
         0.08770   -0.01601    0.03203   -0.04881
AFIX 137
H10A  2    0.602818    0.519426    0.321958    11.00000   -1.50000
H10B  2    0.540755    0.426580    0.279862    11.00000   -1.50000
H10C  2    0.701927    0.391260    0.261897    11.00000   -1.50000
AFIX   0
C11   1    0.546002    0.061456    0.778363    11.00000    0.07119
0.04331 =
         0.04593   -0.01247    0.01711   -0.02683
AFIX  23
H11A  2    0.625915   -0.030482    0.821985    11.00000   -1.20000
H11B  2    0.473437    0.038423    0.754269    11.00000   -1.20000
AFIX   0
C12   1    0.856624   -0.025881    0.150064    11.00000    0.11379
0.08042 =
         0.06414   -0.04051    0.03540   -0.05128
AFIX 137
H12A  2    0.883896   -0.078881    0.085428    11.00000   -1.50000
H12B  2    0.935688   -0.012751    0.172432    11.00000   -1.50000
H12C  2    0.778091    0.069215    0.115457    11.00000   -1.50000
AFIX   0
C13   1    0.272889    0.399708    1.063207    11.00000    0.05580
0.04946 =
         0.04368   -0.01757    0.00708   -0.02231
C14   1    0.290383    0.517260    1.079534    11.00000    0.07299
0.06253 =
         0.07007   -0.03158    0.01268   -0.03319
AFIX  43
H14   2    0.367318    0.534487    1.043195    11.00000   -1.20000
AFIX   0
C15   1    0.195866    0.608819    1.148571    11.00000    0.09628
0.06725 =
         0.08300   -0.04198    0.01262   -0.03134
AFIX  43
H15   2    0.209096    0.686871    1.159225    11.00000   -1.20000
AFIX   0
```

```
C16   1    0.082238    0.583833    1.201371    11.00000    0.08324
0.07221 =
         0.07177   -0.04038    0.01595   -0.01467
AFIX  43
H16   2    0.018382    0.645453    1.248173    11.00000   -1.20000
AFIX   0
C17   1    0.060991    0.469189    1.186344    11.00000    0.05851
0.07424 =
         0.05982   -0.02452    0.01542   -0.01946
AFIX  43
H17   2   -0.017286    0.454208    1.221897    11.00000   -1.20000
AFIX   0
C18   1    0.156677    0.375991    1.118000    11.00000    0.05329
0.05576 =
         0.04761   -0.01668    0.00666   -0.02023
C19   1    0.040704    0.217009    1.166611    11.00000    0.08142
0.11764 =
         0.10143   -0.04265    0.03507   -0.06803
AFIX 137
H19A  2   -0.051490    0.292300    1.130132    11.00000   -1.50000
H19B  2    0.046830    0.207304    1.258180    11.00000   -1.50000
H19C  2    0.054632    0.124379    1.155681    11.00000   -1.50000
AFIX   0
C3'   1    0.377465    0.301554    0.991248    11.00000    0.05193
0.04409 =
         0.04301   -0.01207    0.01008   -0.02513
C4'   1    0.358879    0.229206    0.907175    11.00000    0.05505
0.04818 =
         0.04456   -0.01576    0.01027   -0.02702
AFIX  43
H4'   2    0.274341    0.230576    0.885464    11.00000   -1.20000
AFIX   0
C5'   1    0.488103    0.158499    0.865471    11.00000    0.06041
0.04393 =
         0.04098   -0.01033    0.01032   -0.02757
HKLF    4




REM  mo_D19_50_Kettatni_LY8_0m_a.res in P-1
REM wR2 = 0.1352, GooF = S = 1.023, Restrained GooF = 1.023 for all
data
REM R1 = 0.0489 for 2640 Fo > 4sig(Fo) and 0.0765 for all 3828 data
REM 250 parameters refined using 0 restraints

END

WGHT      0.0525      0.3356

REM Highest difference peak  0.151,  deepest hole -0.135,  1-sigma
level  0.032
Q1    1   0.2044  0.7299  1.1978  11.00000  0.05    0.15
Q2    1   0.6958 -0.0755  0.4084  11.00000  0.05    0.15
Q3    1   0.3220  0.3388  1.0363  11.00000  0.05    0.15
Q4    1   0.3062  0.4191  1.1043  11.00000  0.05    0.14
```

```
Q5    1   0.6205  0.1349  0.4949  11.00000  0.05    0.14
;
_shelx_res_checksum   42559
```