

Synthesis, Crystal Structure Study, and Evaluation of Antimicrobial and Anti-Biofilm Activities of a novel Dihydroisoquinoline-derived

Awatef Selmi ^a, Omar Kammoun ^b, Sami Mnif ^c, Paul Mosset ^d, Majed Kammoun ^{a*}

^a Laboratory of Medicinal and Environment Chemistry, Higher Institute of Biotechnology, University of SFAX, PB 261, 3000 SFAX, Tunisia.

^b Laboratoire Physicochimie de l'Etat Solide, Département de Chimie, Faculté des Sciences de Sfax, Université de Sfax, BP 1171, 3000 Sfax, Tunisia

^c Laboratory of Molecular and Cellular Screening Processes, Centre of Biotechnology of Sfax, University of SFAX, P O Box 1177, Sidi Mansour Road 3018, Sfax, Tunisia.

^d Univ Rennes, CNRS, ISCR (Institut des Sciences Chimiques de Rennes), UMR 6226, F-35000 Rennes, France

*Correspondence e-mail : majed_kammoun@yahoo.fr

1-Data tables of crystal structure of compound 4:

Table 1. Crystallographic data and structure refinement parameters of **4**.

Table 2. Main distances (Å) and bond angles (°) in the **4** crystals.

2-NMR Spectrum:

¹H and ¹³C NMR spectrum of compound **2**.

¹H and ¹³C NMR spectrum of compound **3**.

¹H and ¹³C NMR spectrum of compound **4**.

1-Data tables of crystal structure of compound 4

Table 1. Crystallographic data and structure refinement parameters of **4**.

<i>Crystal data</i>	
Empirical formula	C ₁₇ H ₁₆ Cl NO
Formula weight	285.76 g/mol
Temperature	150 (2) K
Radiation type	Mo-K α
Wavelength	0.71073 Å
Crystal system, space group	triclinic, <i>P</i> ̄ 1
Unit cell dimensions	a = 8.8119(9) Å b = 9.3804(9) Å c = 10.0799(10) Å α = 113.355(3) ° β = 103.999(3) ° γ = 98.895(3) ° 712.25(12) Å ³ 2, 1.332 g.cm ⁻³ 0.263 mm ⁻¹
Volume	300
Z, Calculated density	0.450 x 0.400 x 0.320 mm
Absorption coefficient	colourless
F(000)	prism
Crystal size	
Crystal color	
Crystal description	
<i>Data collection</i>	
Diffractometer	APEXII Kappa-CCD (Bruker-AXS)
θ range for data collection	2.335 to 27.512 °
h _{min} , h _{max}	-11, 10
k _{min} , k _{max}	-9, 12
l _{min} , l _{max}	-13, 13
Reflections collected / unique	11452 / 3237 [R(int) = 0.0357]
Reflections [I > 2 σ]	2676
Completeness to θ_{max}	0.989
Absorption correction type	multi-scan
Max. and min. transmission	0.919 , 0.855
<i>Refinement</i>	
Refinement method	Full-matrix least-squares on F ²
H-atom treatment	H-atom parameters constrained
Data / restraints / parameters	3237 / 0 / 183
Goodness-of-fit	1.059
Final R indices [I > 2 σ]	R ₁ = 0.0384, wR ₂ = 0.1019
R indices (all data)	R ₁ = 0.0478, wR ₂ = 0.1083
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}}$	0.287, -0.274 e.Å ⁻³

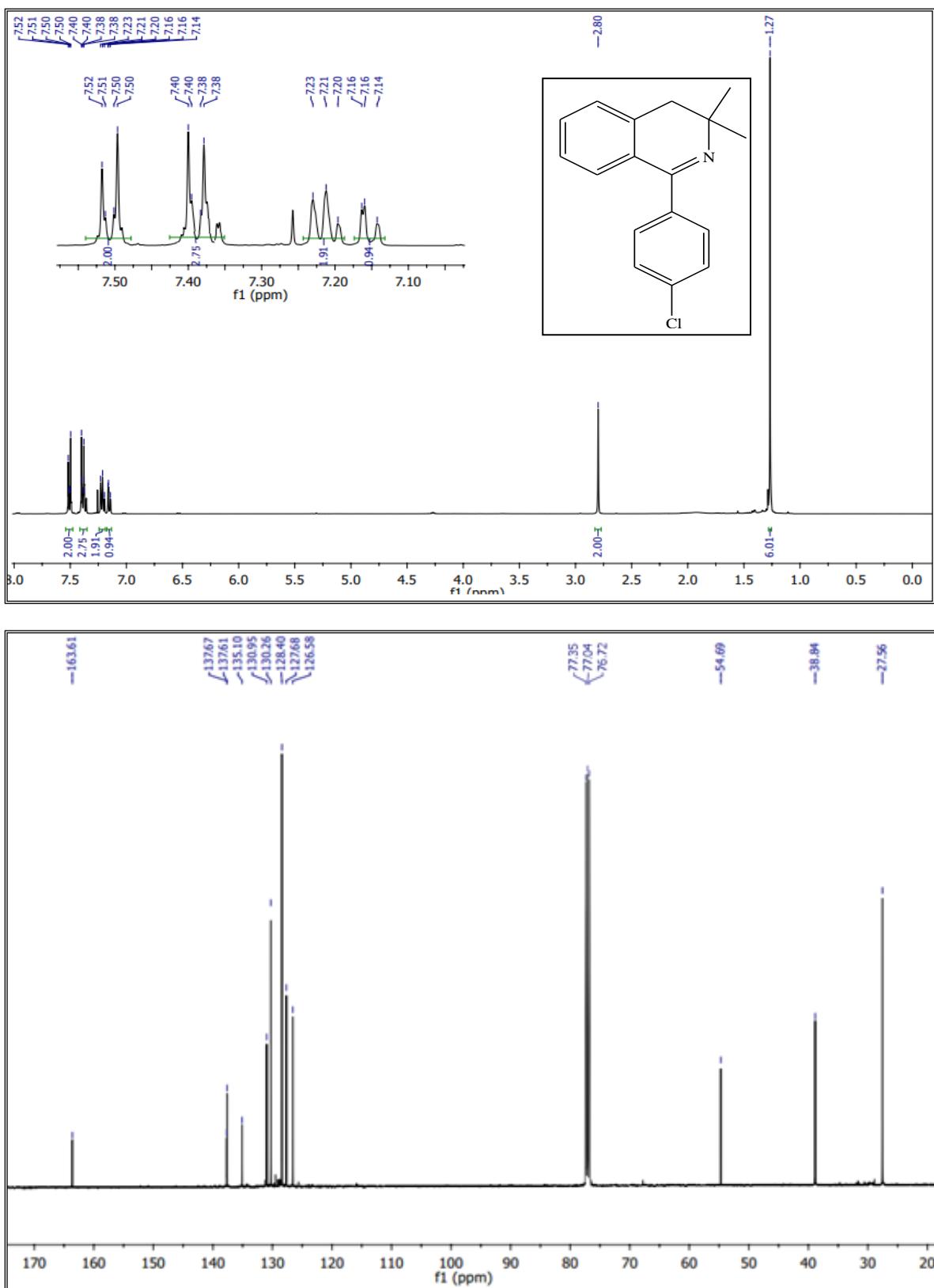
Table 2. Main distances (Å) and bond angles (°) in the **4** crystals.

Cl1 - C1	1.7443(14)	C5 – C6	1.386(2)
C1 – C2	1.381(2)	C5 – H5	0.9500
C1 – C6	1.383(2)	C6 – H6	0.9500
C2 - C3	1.389(2)	C7 – N8	1.3200(18)
C2 – H2	0.9500	C7 - C19	1.4669(19)
C3 – C4	1.3921(19)	N8 – O9	1.2917(15)
C3 – H3	0.9500	N8 - C10	1.5201(18)
C4 – C5	1.394(2)	C10 - C11	1.519(2)
C4 – C7	1.4835(18)	C10 - C12	1.527(2)
C10 - C13	1.530(2)	C14 – C15	1.393(2)
C11 - H11A	0.9800	C14 – C19	1.4060(19)
C11 - H11B	0.9800	C15– C16	1.387(2)
C11 - H11C	0.9800	C15– H15	0.9500
C12 - H12A	0.9800	C16– C17	1.385(2)
C12 - H12B	0.9800	C16– H16	0.9500
C12 - H12C	0.9800	C17– C18	1.387(2)
C13 - C14	1.498(2)	C17– H17	0.9500
C13 – H13A	0.9900	C18– C19	1.402(2)
C13 – H13B	0.9900	C18– H18	0.9500
<hr/>			
C2 - C1 – C6	122.16(13)	C10 – C11 – H11C	109.5
C1 - C1 - Cl1	118.59(11)	H11A – C11 – H11C	109.5
C6 - C1 - Cl1	119.24(12)	H11B – C11 – H11C	109.5
C1 - C2 – C3	118.81(13)	C10 – C12 – H12A	109.5
C1 – C2 – H2	120.6	C10 - C12 – H12B	109.5
C3 – C2 – H2	120.6	H12A - C12 – H12B	109.5
C2 – C3 – C4	120.51(13)	C10 - C12 – H12C	109.5
C2 – C3 – H3	119.7	H12A - C12 – H12C	109.5
C4 – C3 – H3	119.7	H12B - C12 – H12C	109.5
C3 – C4 - C5	119.17(13)	C14 - C13 - C10	111.65(12)
C3 – C4 – C7	121.29(12)	C14 - C13 – H13A	109.3
C5 – C4 – C7	119.44(12)	C10 - C13 – H13A	109.3

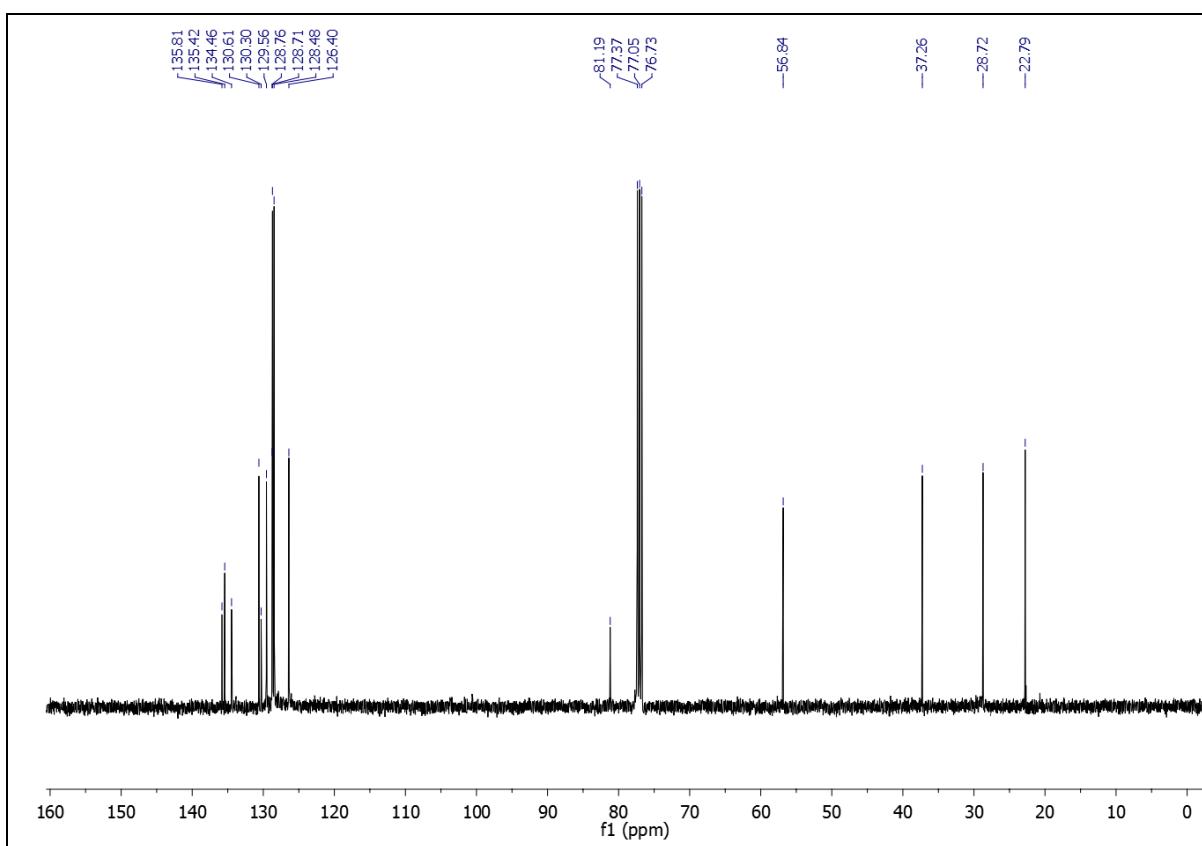
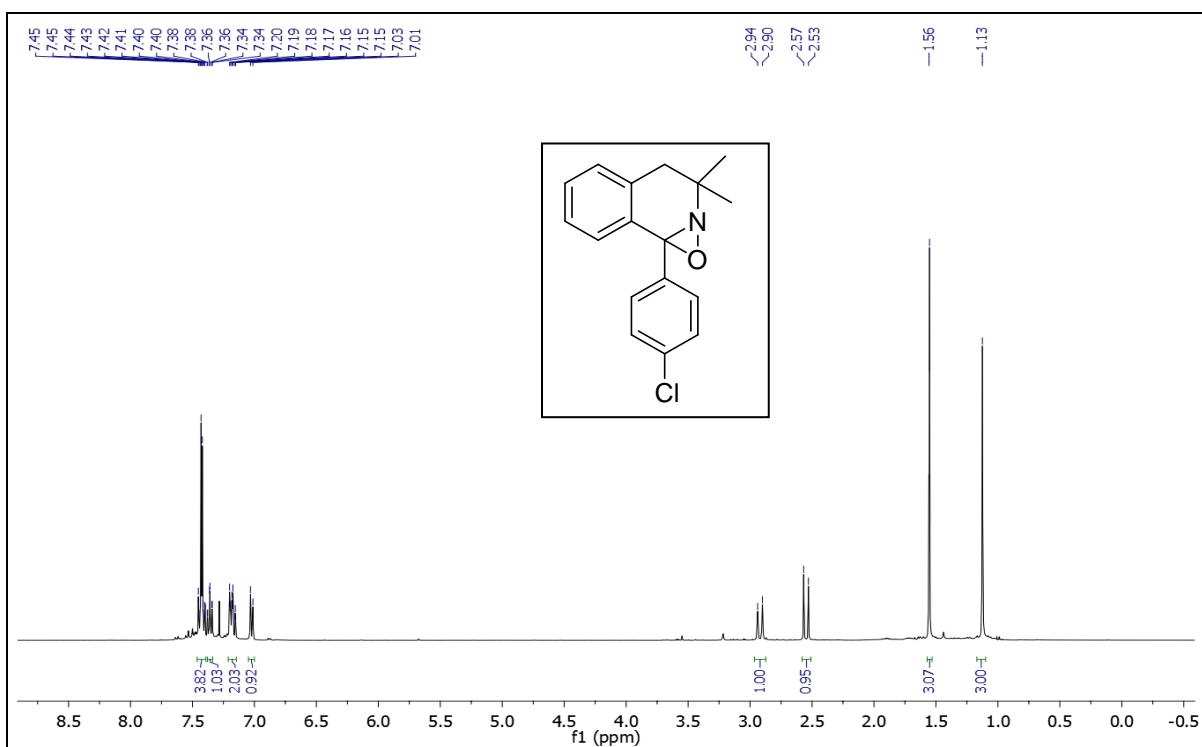
C6 – C5 - C4	120.99(13)	C14 - C13 – H13B	109.3
C6 – C5 – H5	119.5	C10 - C13 – H13B	109.3
C4 – C5 – H5	119.5	H13A - C13 – H13B	108.0
C1 – C6 – C5	118.36(13)	C15 - C14 - C19	119.25(13)
C1 – C6 – H6	120.8	C15 - C14 - C13	123.51(13)
C5 – C6 – H6	120.8	C19 - C14 - C13	117.23(12)
N8 – C7 – C19	120.57(12)	C16 - C15 - C14	120.92(14)
N8 – C7 – C4	118.11(12)	C16 - C15 – H15	119.5
C19 – C7 – C4	121.05(12)	C14- C15 – H15	119.5
O9 – N8 – C7	122.59(12)	C17 – C16 – C15	119.86(14)
O9 – N8 – C10	116.62(11)	C17- C16 – H16	120.1
C7 – N8 – C10	120.46(11)	C15- C16 – H16	120.1
C11 – C10 – N8	109.07(12)	C16 – C17 – C18	120.26(14)
C11 – C10 – C12	111.11(12)	C16- C17 – H17	119.9
N8 – C10 – C12	105.45(11)	C18- C17 – H17	119.9
C11 – C10 – C13	110.83(12)	C17 – C18 – C19	120.33(14)
N8 – C10 – C13	107.87(11)	C17- C18 – H18	119.8
C12 – C10 – C13	112.28(12)	C19- C18 – H18	119.8
C10 – C11 – H11A	109.5	C18- C19 – C14	119.34(13)
C10 – C11 – H11B	109.5	C18- C19 – C7	120.83(13)
H11A – C11 – H11B	109.5	C14- C19 – C7	119.82(13)

2-NMR Spectrum:

¹H and ¹³C NMR spectrum of compound 2.



¹H and ¹³C NMR spectrum of compound 3.



^1H and ^{13}C NMR spectrum of compound 4.

