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MULTINUCLEAR NMR STRUCTURAL STUDY OF NOVEL γ-IMINOPHOSPHONATE AND PHOSPHINE OXIDE DERIVATIVES

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We report for the first time the synthesis of γ-iminophosphonates and phosphine oxides from the acid-catalysed reaction of primary amines with γ-phosphonylketones. The full characterization of these compounds through their ^{1}H, ^{31}P, and ^{13}C NMR spectra indicates that they are obtained as a mixture of *Z* and *E* isomers. An unambiguous method for the assignment of these configurations, based on the ^{13}C chemical shifts of C2 carbon atoms in the α position with respect to the C=N double bond is used. The ^{31}P chemical shifts are also of diagnostic importance in assigning the *Z* and *E* configurations. Indeed, the phosphorus atom is found to resonate at a slightly higher field in *Z* isomers.

K e y w o r d s: ^{13}C NMR, ^{31}P NMR, ^{1}H NMR, γ-iminophosphonates, phosphine oxides, *E*/*Z* isomerism.

INTRODUCTION

Imines, also known as Schiff bases or azomethines, are very important compounds due to their broad spectrum of biological properties, including antimicrobial [1—4], antifungal [5, 6], anticancer [7—10], anti-HIV [11], and herbicidal [12] activities. Certain imine derivatives have also found applications as ligands in coordination chemistry [13—15]. On the other hand, imines and their derivatives are recognized as key intermediates for the synthesis of biologically active nitrogen heterocycles such as those involved in alkaloid synthesis [16—19].

The introduction of a phosphonate functionality on imines may be very interesting for the enhancement of the biological and complexing properties of these molecules, and for synthetic transformations leading to aminophosphonate derivatives and azaheterocyclic phosphonates which are known to exhibit a variety of pharmacological properties [20, 21].

The configuration determination of the C=N double bond in imines is routinely accomplished using various experimental techniques, especially spectroscopic ones [22—24]. The majority of these molecular systems are known to exist in the solution as single *Z* and *E* forms, or their *Z*/*E* mixtures.

In view of the above, and in the continuation of our studies on the preparation and potential synthetic applications of imines [25—27], we report here for the first time the synthesis of γ-iminophosphonates and phosphine oxides from the acid-catalysed reaction of primary amines with γ-phosphonylketones. The full characterization of these compounds through their ^{1}H, ^{31}P, and ^{13}C NMR spectra indicated that they were obtained as a mixture of *Z* and *E* isomers. Unambiguous methods for the assignment of these configurations, based on ^{13}C and ^{31}P NMR, have been used.

Table 1

Physical data, IR *and* HRMS *for the* γ*-phosphonylimines* **2**

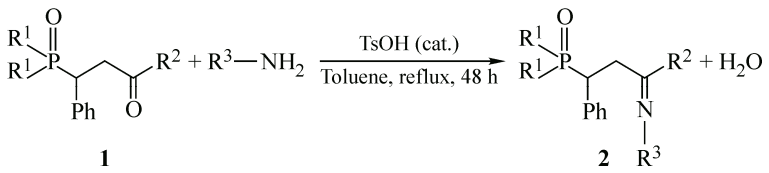
Compound	R^1	R^2	R^3	Physical aspect	Melting point, °C	Yield, % [a]	IR data, cm^{-1}	Molecular formula	HRMS data, calc. / found
2a	EtO	Me	CH_2—Ph	Yellow oil		95	$\nu_{P=O}$ = 1229 $\nu_{C=N}$ = 1650	$C_{21}H_{28}NO_3P$	373.1807 / 373.1809
2b	EtO	Ph	CH_2—Ph	Yellow oil		84	$\nu_{P=O}$ = 1242 $\nu_{C=N}$ = 1651	$C_{26}H_{30}NO_3P$	435.1963 / 435.1968
2c	EtO	Me	3-Me—C_6H_4—CH_2	Yellow oil		80	$\nu_{P=O}$ = 1243 $\nu_{C=N}$ = 1659	$C_{22}H_{30}NO_3P$	387.1963 / 387.1969
2d	EtO	Me	*i*Pr	Yellow oil		86	$\nu_{P=O}$ = 1247 $\nu_{C=N}$ = 1646	$C_{17}H_{28}NO_3P$	325.1807 / 325.1808
2e	EtO	Ph	*i*Pr	Yellow oil		91	$\nu_{P=O}$ = 1242 $\nu_{C=N}$ = 1655	$C_{22}H_{30}NO_3P$	387.1963 / 387.1961
2f	Ph	Me	CH_2—Ph	Brown solid	96—98	80	$\nu_{P=O}$ = 1201 $\nu_{C=N}$ = 1659	$C_{29}H_{28}NOP$	437.1908 / 437.1911
2g	Ph	Ph	CH_2—Ph	White solid	93—94	90	$\nu_{P=O}$ = 1241 $\nu_{C=N}$ = 1640	$C_{34}H_{30}NOP$	499.2065 / 499.2069
2h	Ph	Me	3-Me—C_6H_4—CH_2	Brown solid	101—102	82	$\nu_{P=O}$ = 1269 $\nu_{C=N}$ = 1656	$C_{30}H_{30}NOP$	451.2065 / 451.2066
2i	Ph	Me	Ph	Red solid	107—109	85	$\nu_{P=O}$ = 1290 $\nu_{C=N}$ = 1633	$C_{28}H_{26}NOP$	423.1752 / 423.1758
2j	Ph	Ph	*i*Pr	White solid	89—91	83	$\nu_{P=O}$ = 1200 $\nu_{C=N}$ = 1634	$C_{30}H_{30}NOP$	451.2065 / 451.2069

[a] Isolated yield.

EXPERIMENTAL

Compounds. The starting γ-ketophosphonates and phosphine oxides **1** were easily prepared according to the reported procedures [28, 29]. It was found that the reaction of these compounds with primary amines performed in refluxing toluene for 48 h, in the presence of a catalytic amount of p-toluenesulfonic acid, led to the formation of γ-iminophosphonates and phosphine oxides **2** (Scheme 1). The scope of the reaction was assessed with a range of γ-phosphonylketones and primary amines. All the substrates reacted in good to high yields (Table 1).

General procedure for the synthesis of γ-iminophosphonates and phosphine oxides 2. A mixture of γ-ketophosphonate or phosphine oxide **1** (0.005 mol), primary amine (0.01 mol), and paratoluene sulfonic acid (0.1 g) in dry toluene (40 ml) was heated at reflux, with Dean-Stark separation of water, for 48 h. The reaction mixture was then concentrated under vacuum. The residue obtained was washed with petroleum ether.

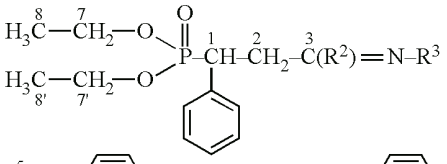


1 **2**

Scheme 1. Synthesis of γ-iminophosphonates and phosphine oxides **2**

Table 2

^{13}C NMR *chemical shifts* (ppm) *and* J(C,P) (Hz) *for* γ*-phosphonylimines* (**2a**—**2e**)



2a : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH_2$–C$_6$H$_5$ **2b** : R^2 = C$_6$H$_5$– ; $R^3 = {}^{5}CH_2$–C$_6$H$_5$

2c : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH_2$–C$_6$H$_4$–$^{6}CH_3$ **2d** : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH(^{6}CH_3)_2$

2e : R^2 = C$_6$H$_5$– ; $R^3 = {}^{5}CH(^{6}CH_3)_2$

Parameter	**2a**		**2b**		**2c**		**2d**		**2e**	
	Z	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*
C_1	38.8	39.8	39.2	41.0	38.2	39.8	39.0	41.1	38.0	41.3
^1J(C,P)	142.6	138.1	141.9	138.1	140.4	138.1	140.4	138.1	140.4	139.6
C_2	43.0	43.5	43.4	44.8	41.7	43.9	41.5	43.7	39.9	42.8
C_3	165.7	165.7	166.2	169.0	165.4	165.4	162.0	163.1	161.6	163.5
^3J(C,P)	16.6	16.6	14.3	17.4	16.6	16.6	16.6	16.6	15.9	18.1
C_4	20.2	17.5			20.4	20.2	23.4	23.2		
C_5	51.3	53.7	55.6	56.6	51.5	53.7	50.2	51.5	50.3	50.8
C_6					17.5	19.3	17.4	20.7	19.6	20.2
C_7	60.8	60.8	62.0	62.0	60.8	60.8	61.8	62.4	60.9	63.1
^2J(C,P)	7.6	7.6	6.8	6.8	6.8	6.8	7.6	6.8	7.6	6.0
$C_{7'}$	61.5	61.5	62.6	62.6	61.4	61.4	62.0	62.7	61.8	63.3
^2J(C,P)	6.8	6.8	6.8	6.8	6.8	6.8	6.8	6.8	7.6	6.0
C_8	15.2	15.2	16.1	16.1	15.2	15.2	16.1	16.4	14.9	15.2
^3J(C,P)	6.0	6.0	6.8	6.8	6.0	6.0	6.8	6.0	6.8	7.5
$C_{8'}$	15.3	15.3	16.3	16.3	15.3	15.3	16.2	16.4	15.0	15.4
^3J(C,P)	6.0	6.0	6.0	6.0	6.0	6.0	5.3	4.5	4.5	5.3
C-arom	125.4		125.4	130.8	125.1		125.3		126.5	
	125.6		125.7	133.2	125.4		125.9		126.9	
	125.9		126.3	133.3	125.8		128.7		127.0	
	126.4		126.5	137.7	125.9		128.9		127.1	
	126.7		126.8	137.8	126.3		129.1		127.2	
	127.3		126.9	139.1	126.5		129.2		128.2	
	127.6		127.4	139.5	128.0		136.1		128.9	
	128.0		127.6		128.1		136.3		129.4	
	128.1		128.3		128.5				129.7	
	128.5		128.4		128.6				131.7	
	128.6		128.5		135.2				132.2	
	135.2		128.7		135.3				135.1	
	135.3		128.8		136.5				135.5	
	136.7		129.1		136.6				139.1	
	137.7		129.3		139.2				141.4	
			130.0		141.6					

N o t e. Assignment are interchangeable for C_7 and $C_{7'}$, C_8 and $C_{8'}$.

Spectra. All NMR spectra were recorded using a Bruker AC-300 spectrometer operating at 300.132 MHz for ^{1}H, 121.495 MHz for ^{31}P and 75.476 MHz for ^{13}C.

The ^{1}H NMR parameters were as follows: spectral width 6188.1 Hz; acquisition time 2.65 s; digital resolution 0.19 Hz; number of scans 16. For the ^{31}P NMR: spectral width 73529.4 Hz; acquisition time 0.89 s; digital resolution 0.56 Hz; number of scans 16. As for ^{13}C NMR: spectral width 19531.2 Hz; acquisition time 1.68 s; digital resolution 0.30 Hz; number of scans 700.

Samples were prepared in a 5 mm NMR tube in $CDCl_3$ at a concentration of 50 mg/ml and at 298 K, no zero filling.

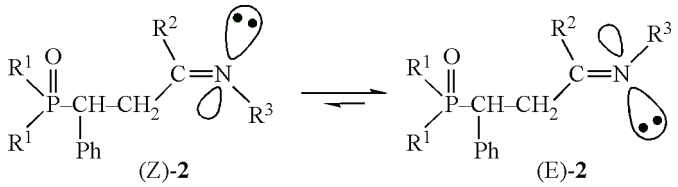
The chemical shifts are reported in ppm relative to tetramethylsilane (internal reference) for ^{1}H and ^{13}C NMR and relative to 85 % H_3PO_4 (external reference) for ^{31}P NMR. The coupling constants are reported in Hertz (Hz). For ^{1}H NMR, the multiplicities of signals are indicated by the following abbreviations: s — singlet, d — doublet, t — triplet, m — multiplet.

Mass spectra were determined on an Agilent 5975B spectrometer under electronic impact (EI) conditions.

IR spectra were recorded on a Nicolet IR200 spectrometer.

RESULTS AND DISCUSSION

The ^{13}C, ^{31}P, and ^{1}H NMR data from compounds **2** are summarized in Tables 2 to 6. All NMR spectra of compounds **2** showed a signal doubling indicating that they are obtained as a mixture of *Z* and *E* isomers (Scheme 2).



Scheme 2. *Z* and *E* isomers in compounds **2**

The *Z* and *E* configurations were attributed on the basis of the ^{13}C chemical shifts of C2 carbon atoms in the α position with respect to the C=N double bond (Tables 2 and 3). Indeed, according to some literature data [25, 30—34] concerning the stereochemistry of imines, hydrazones, and oximes, the carbon atom adjacent to the C=N double bond resonates at higher fields when it is in syn position to the group on the nitrogen atom (R^3 in our case).

The C2 carbon atom is characterized by two singlets in the region included between 39.0 ppm and 45.6 ppm. The first one, which appears at higher fields, is assigned to C2 when it is *syn* with respect to the R^3 group (*Z* isomer). However, the second singlet, which appears at lower fields, is assigned to C2 when it is *anti* to the R^3 group. This assignment corresponds to the *E* isomer. It is important to note here that the ^{13}C chemical shifts of the C4 carbon atom, which is in the α′ position with respect to the C=N double bond, follow the same correlations as the C2 carbon atom (Tables 2 and 3).

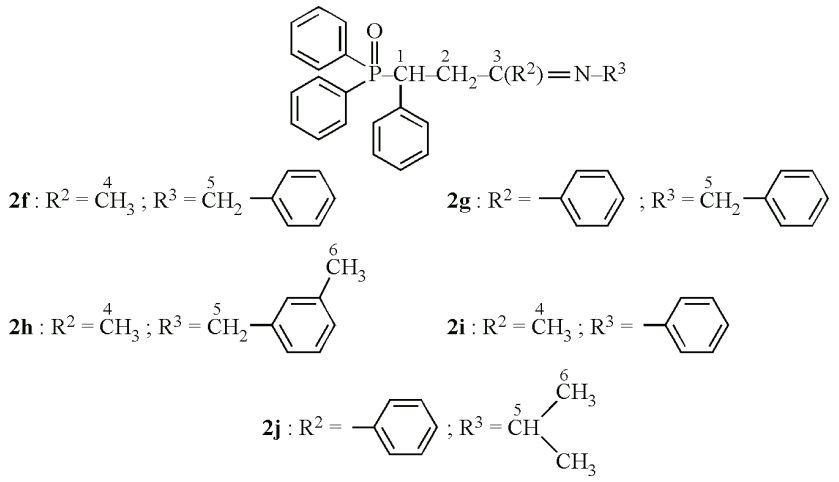
The relative proportions of the *Z* and *E* isomers were estimated from the inverse-gated $^{31}P\{^{1}H\}$ NMR spectra where a singlet for each isomer is present (Table 4). In all cases, *E* isomers were the predominant forms probably due to the steric hindrance between the 2-phenyl-2-phosphonoethyl and R^3 groups, which destabilizes the *Z* isomers. This observation was confirmed by theoretical RHF/6-31G calculations, performed with the Gaussian 03 program, which showed a stabilization of 1.0—2.8 Kcal/mol in favour of the *E* isomers (Table 4).

The ^{31}P chemical shifts are also of diagnostic importance in assigning the *Z* and *E* configurations. Indeed, the phosphorus atom was found to resonate at a slightly higher fields in *Z* isomers (Table 4).

The ^{1}H NMR spectra display the characteristic signals of all the protons (Tables 5 and 6), particularly those corresponding to the CH_2—CH—P motif which resonate as two multiplets between 2.5 ppm and 4.5 ppm, probably consisting in an ABMX spin system. Such a multiplicity can be ra-

Table 3

^{13}C NMR *chemical shifts* (ppm) *and* J(C,P) (Hz) *for* γ*-phosphonylimines* (**2f—2j**)



Parameter	**2f**		**2g**		**2h**		**2i**		**2j**	
	Z	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*
C_1	41.1	42.2	41.8	43.4	41.1	42.2	39.9	41.4	41.7	43.3
^1J(C,P)	67.9	67.9	67.2	67.2	68.7	68.7	68.7	56.6	68.7	67.2
C_2	41.1	45.6	39.9	44.7	43.4	45.6	39.0	42.1	39.7	43.2
C_3	166.6	166.6	165.1	167.9	166.4	166.4	167.2	167.2	162.0	163.6
^3J(C,P)	12.8	12.8	13.6	13.6	12.8	12.8	12.8	12.8	14.3	14.3
C_4	21.4	21.2			21.4	21.2	20.3	20.1		
C_5	54.5	54.7	54.4	55.4	54.2	54.6			50.4	50.9
C_6					19.0	19.7			19.5	20.2
C-arom	126.3		126.3	130.4	125.1	132.0	114.5		126.0	
	126.5		126.6	131.1	125.2	132.6	114.9		126.3	
	126.7		126.8	131.2	126.1	132.8	121.0		127.4	
	126.9		126.9	131.8	126.6	135.9	122.0		127.5	
	127.4		127.1	136.5	127.7	136.5	127.6		127.6	
	127.8		127.2	136.6	128.3	137.6	127.7		127.8	
	128.0		127.4	138.5	128.5	137.9	127.9		127.9	
	128.1		128.4	139.1	128.7	139.3	128.1		129.5	
	128.2		128.5	139.2	128.9	140.3	128.4		129.7	
	128.4		128.7		129.6		129.1		130.1	
	128.6		128.9		129.7		129.6		130.3	
	129.0		129.3		130.1		129.8		130.4	
	129.7		129.6		130.2		129.9		130.9	
	130.0		129.7		130.9		130.0		131.1	
	130.2		129.8		131.0		130.1		131.8	
	130.9		130.0		131.1		130.2		134.1	
	131.0		130.1		131.2		130.3		135.3	
	131.1		130.2		131.4		130.4		139.0	
	131.2		130.3		131.5		130.8		139.3	
	131.3						135.1			
	132.6						136.6			
	136.4						148.1			
	136.5						149.9			
	137.6									

Table 4

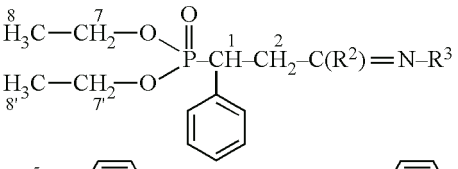
^{31}P NMR *chemical shifts* (ppm), % *Z*/*E*, *and calculated* ($E_Z - E_E$) *for the* γ*-phosphonylimines* **2**

Compound	δ ^{31}P (*Z*)	δ ^{31}P (*E*)	*Z*[a], %	*E*[a], %	Δ*E* ($E_Z - E_E$)[b], Kcal/mol	Compound	δ ^{31}P (*Z*)	δ ^{31}P (*E*)	*Z*[a], %	*E*[a], %	Δ*E* ($E_Z - E_E$)[b], Kcal/mol
2a	28.1	29.1	6	94	1.41	**2f**	33.4	34.3	15	85	2.78
2b	27.8	28.6	30	70	1.00	**2g**	32.4	33.9	39	61	1.10
2c	27.4	29.2	7	93	1.42	**2h**	33.5	34.4	23	77	2.80
2d	27.8	28.8	29	71	1.36	**2i**	33.5	34.0	26	74	1.74
2e	27.8	28.4	22	78	1.12	**2j**	33.5	34.5	16	84	2.73

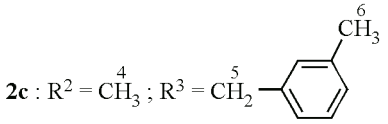
[a] Determined from the inverse-gated ^{31}P{^{1}H} NMR spectra.
[b] RHF/6-31G calculations with the Gaussian 03 program.

Table 5

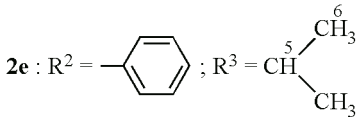
^{1}H NMR *chemical shifts* (ppm), *coupling constants* (Hz), *and multiplicities for* γ*-phosphonylimines* (**2a—2e**)



2a : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH_2$–C$_6$H$_5$ **2b** : R^2 = C$_6$H$_5$– ; $R^3 = {}^{5}CH_2$–C$_6$H$_5$



2c : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH_2$–C$_6$H$_4$–^{6}CH$_3$ **2d** : $R^2 = {}^{4}CH_3$; $R^3 = {}^{5}CH$(CH$_3$)$_2$

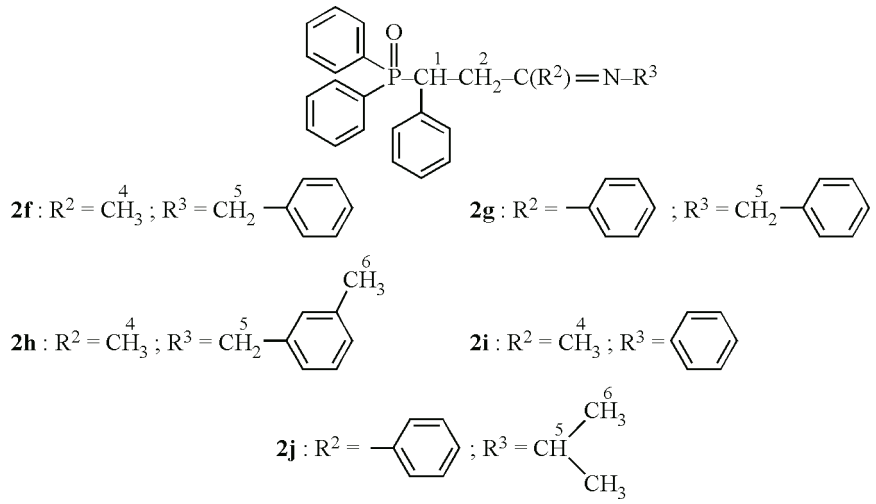


2e : R^2 = C$_6$H$_5$– ; $R^3 = {}^{5}CH$(CH$_3$)$_2$

Parameter	**2a**		**2b**		**2c**		**2d**		**2e**	
	Z	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*
H_1	3.53—3.59 m		3.58—3.63 m		3.59—3.64 m		3.71—3.90 m		3.66—3.85 m	
H_2	2.47—2.99 m		3.05—3.50 m		2.87— 2.97 m		2.82—2.95 m		3.08—3.22 m	
H_4	1.90 s	1.62 s			1.76 s	1.61 s	1.62 s	2.02 s		
H_5	3.65 s	3.62 s	4.20 s	3.60 s	4.18 s	3.62 s	3.30—3.46 m		3.24—3.40 m	
H_6					2.15 s	2.12 s	1.29 d		0.78 d	0.58 d
							($^{3}J_{H,H}$ = 6.0 Hz)			
H_7	3.67—3.94 m		3.74—3.95 m		3.71—3.91 m		3.98—4.16 m		3.47—4.02 m	
$H_{7'}$	3.67—3.94 m		3.74—3.95 m		3.71—3.91 m		3.98—4.16 m		3.47—4.02 m	
H_8	0.91 t	0.96 t	0.55 t	0.95 t	0.97 t	0.97 t	0.90 t	0.90 t	0. 92 t	0.85 t
	($^{3}J_{H,H}$ = 6.0 Hz)									
$H_{8'}$	1.09 t	1.12 t	1.16 t	1.07 t	1.11 t	1.11 t	1.05 t	1.05 t	1.11 t	1.19 t
	($^{3}J_{H,H}$ = 6.0 Hz)									
H_{ar}	6.87—7.50 m		6.69— 8.18 m		6.67—7.18 m		6.17—7.99 m		6.96—7.89 m	

Table 6

^{1}H NMR *chemical shifts* (ppm), *coupling constants* (Hz), *and multiplicities for γ-phosphonylimines* (**2f—2j**)



Parameter	2f		2g		2h		2i		2j	
	Z	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*	*Z*	*E*
H_1	4.45—4.60 m		4.23—4.31 m		4.53—4.57 m		4.38—4.41 m		3.86—3.97 m	
H_2	2.97—3.26 m		3.17—3.44 m		2.97—3.09 m		3.00—3.39 m		3.00—3.40 m	
H_4	1.83 s	1.59 s			1.82 s	1.59 s	2.28 s	2.38 s		
H_5	4.32 s	3.79 s	4.20 s	3.64 s	3.78 s	4.30 s			3.40—3.60 m	
H_6					2.27 s	2.24 s			0.74 d	1.04 d
									($^3J_{H,H}$ = 6.0 Hz)	
H_{ar}	7.03—7.93 m		6.61—8.18 m		6.85—7.93 m		6.17—7.99 m		6.13—7.72 m	

tionalized taking into account that the methylene protons are diastereotopic due to the neighbouring asymmetric carbon.

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