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Liquid Crystals

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Novel mesogenic asymmetric diacylhydrazines: synthesis, mesomorphic behaviour and X-ray study

M. Parra^a; P. Hidalgo^a; J. Barberá^b; E. Carrasco^a; C. Saavedra^a

^a Facultad de Ciencias Químicas, Departamento de Química Orgánica, Universidad de Concepción, Concepción, Chile ^b Facultad de Ciencias-Instituto de Ciencia de Materiales de Aragón, Química Orgánica, Universidad de Zaragoza-C.S.I.C., 50009-Zaragoza, España

To cite this Article Parra, M. , Hidalgo, P. , Barberá, J. , Carrasco, E. and Saavedra, C.(2006) 'Novel mesogenic asymmetric diacylhydrazines: synthesis, mesomorphic behaviour and X-ray study', *Liquid Crystals*, 33: 4, 391 – 397

To link to this Article: DOI: 10.1080/02678290500483288

URL: <http://dx.doi.org/10.1080/02678290500483288>

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Novel mesogenic asymmetric diacylhydrazines: synthesis, mesomorphic behaviour and X-ray study

M. PARRA*[†], P. HIDALGO[†], J. BARBERÁ[‡], E. CARRASCO[†] and C. SAAVEDRA[†]

[†]Facultad de Ciencias Químicas, Departamento de Química Orgánica, Universidad de Concepción, Casilla 160-C, Concepción, Chile

[‡]Facultad de Ciencias-Instituto de Ciencia de Materiales de Aragón, Química Orgánica, Universidad de Zaragoza-C.S.I.C., 50009-Zaragoza, España

(Received 26 July 2005; accepted 18 October 2005)

The synthesis and liquid crystalline properties of a new series of diacylhydrazine derivatives (**2a–g**) are reported. All compounds of series **2** exhibit a smectic C (SmC) phase. The first homologues (**2a**, **2b**) display a monotropic SmC mesophase, whereas the highest homologues (**2c–g**) exhibit an enantiotropic SmC phase. The liquid crystalline properties were investigated by differential scanning calorimetry, polarizing optical microscopy and X-ray measurements.

1. Introduction

It is well known that liquid crystallinity can be affected by molecular shape and conformation as well as by intermolecular interaction. The correlation between chemical structure and mesomorphic properties is one of the most important problems in liquid crystals science. An understanding of the influence of different structural elements of the molecules on physico-chemical characteristics of mesomorphic organic compounds helps the chemist to synthesize liquid crystals with required properties [1–4].

In the well established classical concept of calamitic liquid crystals a molecular geometry as close to linearity as possible is required, which is generally obtained by using aromatic, heteroaromatic or aliphatic rings as mesogenic units [5]. Five-membered heterocyclic compounds, such as thiadiazoles and oxadiazoles, have also proved highly efficient in promoting mesomorphic properties [6–26]. The diacylhydrazines are the most common precursors in the synthesis of the 1,3,4-oxadiazole and 1,3,4-thiadiazole-based liquid crystalline (LC) derivatives; they possess the heterocyclic ring in the central position of the mesogenic core [21, 23]. However, mesomorphic diacylhydrazines are rare, and there are few publications on diacylhydrazine derivatives showing liquid crystals properties. Early examples of mesomorphic diacylhydrazines that exhibit relatively broad smectic LC phases, were synthesized by Schubert *et al.* [27] and K. Dimitrowa *et al.* [28]. In these cases, it was suggested that the formation of hydrogen bonds between

the diacylhydrazine moieties would yield supramolecular structures of parallel molecular layers, thus explaining the appearance of smectic LC phases [29].

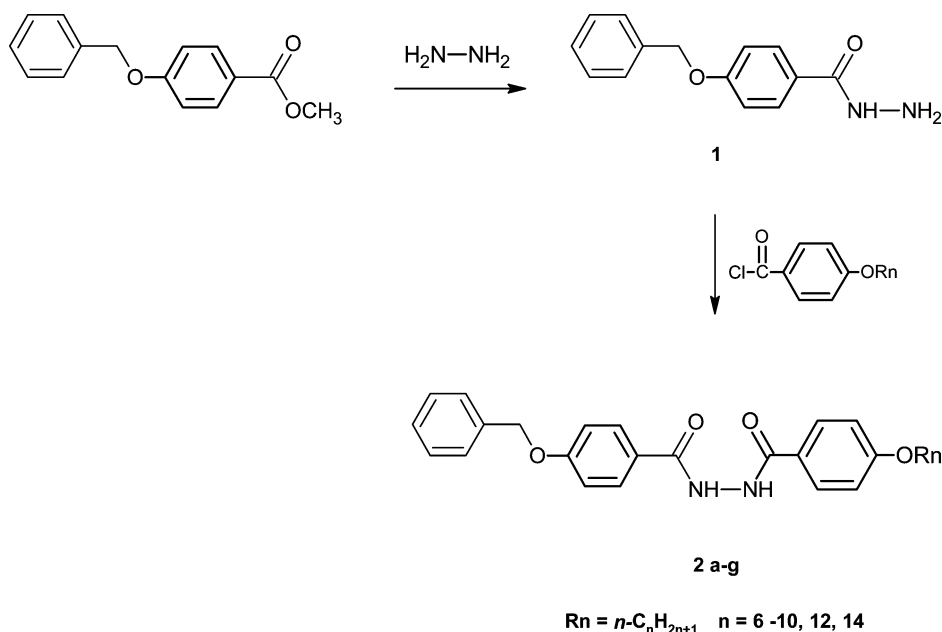
In this paper, we report the synthesis and characterization of new mesomorphic asymmetric diacylhydrazines, specifically 4-*n*-alkoxybenzoic acid N'-(4-benzyloxyphenyl-2-carbonyl)hydrazides (series **2a–g**). These compounds display liquid crystalline properties, in each case a smectic C (SmC) mesophase is observed. The first members of this series (**2a**, **2b**) display a monotropic SmC phase, whereas the other homologues (**2c–g**) exhibit an enantiotropic SmC phase. Our motivation for the synthesis of these materials is to prepare novel chiral liquid crystalline diacylhydrazine, chiral 1,3,4-oxadiazole and chiral 1,3,4-thiadiazole derivatives possessing ferroelectric properties. Two points must be noted. First, the occurrence of a tilted SmC mesophase in series **2** compounds opens an interesting possibility for the design of ferroelectric liquid crystals (FLCs), because the benzyl group can be replaced by chiral chains in order to obtain a chiral SmC phase (SmC*) necessary for ferroelectric properties. Second, chiral liquid crystalline 1,3,4-oxadiazole and 1,3,4-thiadiazole derivatives can be prepared from compounds of series **2**. These heterocycles might exhibit interesting ferroelectric and/or cholesteric behaviour, and are being investigated in current research.

2. Results and discussion

2.1. Synthesis

The synthetic route to the diacylhydrazine-based LC materials of this study is shown in scheme 1. The

*Corresponding author. Email: mparra@udec.cl

Scheme 1. Synthetic route for diacylhydrazines of series **2a-g**.

diacylhydrazine derivatives **2a-g** were synthesized starting with the condensation of methyl 4-benzyloxybenzoate and hydrazine yielding the corresponding hydrazide **1**. This was reacted with 4-*n*-alkoxybenzoyl chloride ($n=6-10, 12, 14$) leading to the formation of the homologous series **2a-g** [23].

2.2. Mesomorphic properties

The optical, thermal and thermodynamic data for compounds **2a-g** are given in table 1. A plot of transition temperature against the number of carbon atoms in the alkoxy chain is given in figure 1, in which transition temperatures obtained both on heating and on cooling are represented.

All the compounds in series **2a-g** have similar melting points and exhibit mesomorphic properties. In each case a SmC mesophase is observed. The first members of the series ($n=6, 7$) show a monotropic SmC mesophase. The other homologues ($n=8-10, 12, 14$) show an enantiotropic SmC mesophase. The SmC-I transition temperature increases with increase in chain length, leading to a broadening of the SmC mesophase range. On slow cooling from the isotropic melt, these homologues clearly show the existence of a SmC phase. On the other hand, as the length of alkoxy chain increases, the thermal stability of the SmC phase increases. These results indicate that the diacylhydrazines of the series **2a-g** also follow the common rule that the SmC phase is more favoured in molecules with longer flexible chains [12, 30].

The mesomorphic properties of the series **2a-g** compounds can be explained by taking into account the formation of H-bonding between molecules of the

Table 1. Transition temperatures and associated enthalpy changes for compounds of series **2a-g**. Cr=crystal, SmC=smectic C, I=isotropic phase.

Compound	Transition	Temperature/ $^{\circ}$ C	$\Delta H/J g^{-1}$
2a ($n=6$)	Cr-I	168.1	109.5
	I-SmC	158.7	-11.8
	SmC-Cr	144.9	-73.1
2b ($n=7$)	Cr-I	165.7	95.9
	I-SmC	164.0	-11.2
	SmC-Cr	142.9	-70.9
2c ($n=8$)	Cr-SmC	167.7	73.8
	SmC-I	170.9	10.9
	I-SmC	169.5	-12.8
2d ($n=9$)	SmC-Cr	140.6	-74.2
	Cr-SmC	166.3	92.6
	SmC-I	174.8	17.1
2e ($n=10$)	I-SmC	173.5	-16.6
	SmC-Cr	145.7	-95.0
	Cr-SmC	166.2	107.2
2f ($n=12$)	SmC-I	178.6	20.9
	I-SmC	177.0	-20.5
	SmC-Cr	145.8	-106.9
2g ($n=14$)	Cr-SmC	164.1	87.7
	SmC-I	182.9	18.5
	I-SmC	181.8	-18.9
2g ($n=14$)	SmC-Cr	142.8	-95.8
	Cr-SmC	163.2	75.2
	SmC-I	185.5	17.4
2g ($n=14$)	I-SmC	184.8	-17.1
	SmC-Cr	140.9	-74.5

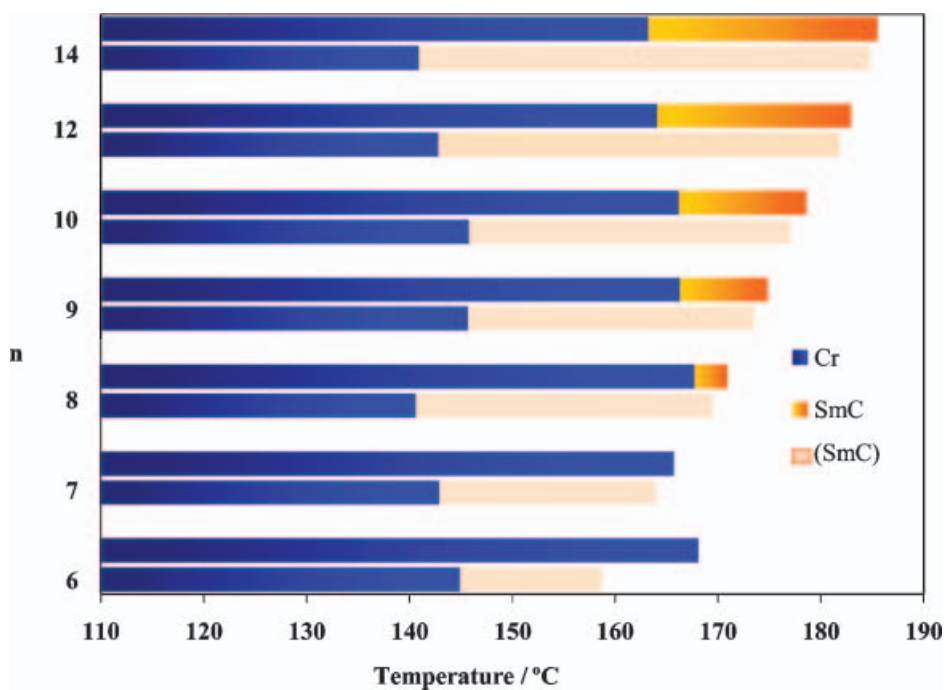


Figure 1. Plot of transition temperature versus the number of carbon atoms in the alkoxy chain for series **2a–g** compounds. SmC=smectic C obtained on heating; (SmC)=smectic C obtained on cooling.

diacylhydrazines. This parallel molecular arrangement would encourage smectic mesomorphism by providing additional lateral intermolecular attraction and by lining up molecules in a layered order [27–29].

2.3. Textures observed by polarizing optical microscopy

The mesophase exhibited by series **2a–g** compounds was identified from the optical texture observed by polarizing optical microscopy (POM), using the classification systems reported by Sackmann and Demus [31], and Gray and Goodby [32]. The SmC phase was determined from textural observations by thermal POM during heating and cooling cycles. Phase transition temperatures were found to be in reasonable agreement with those from DSC thermograms.

The SmC mesophase exhibited by compounds of series **2** was identified by the appearance of a broken focal-conic fan texture coexisting with the fine four-brush schlieren texture (see figure 2).

2.4. X-ray diffraction study

The mesophases of this series of compounds were studied by variable temperature X-ray diffraction. The aim of this study was to confirm the SmC nature of the mesophase as well as to determine the layer spacing and its evolution with changing chain length. The patterns

were recorded at high temperatures during cooling when the mesophase was monotropic, and during either heating or cooling when the mesophase was enantiotropic.

In all cases, the diffractograms supported the SmC nature of the mesophase assigned on the basis of the microscope textures. The patterns contain a single sharp maximum at small angles, due to the reflection of X-rays on the smectic layers, and some diffuse scattering at large angles, related to the liquid-like packing inside the layers and to the conformationally disordered chains. From Bragg's law, the small angle ring gives the layer spacing d in the smectic structure, and the measured values of this parameter are gathered in table 2. Table 2 also includes the length L of each mesogenic molecule in its fully extended conformation, measured using Dreiding stereomodels. For all the compounds the measured, layer thickness d is significantly smaller than the calculated molecular length L , which confirms that the smectic phase has a structure of tilted molecules. As expected, the layer spacing increases as the alkoxy chain becomes longer.

The maximum observed at large angles arises from short range order within the smectic layers and, by using Bragg's law, provides an estimation of the mean intermolecular distance. This distance is about 4.45 Å, a value typical for calamitic mesophases.

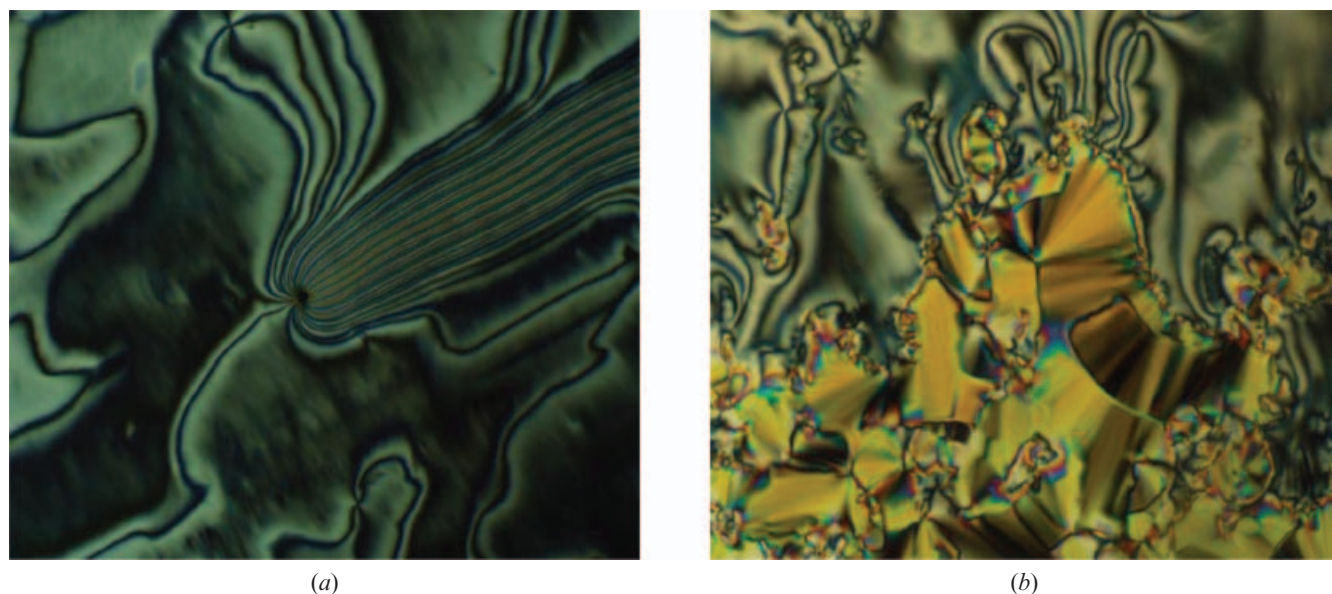


Figure 2. Mesophase textures obtained on cooling. (a) Schlieren texture of compound **2f** at 146°C; (b) broken focal-conic fan texture of compound **2f** coexisting with schlieren at 170°C.

Table 2. Results of the high temperature X-ray diffraction study of compounds **2a–g** in their SmC mesophase. Parameter d represents the smectic layer thickness, L is the molecular length estimated from Dreiding stereomodels for a fully extended conformation.

Compound	Temperature/°C	Phase	$d/\text{Å}$	$L/\text{Å}$
2a ($n=6$)	158	SmC	23.7	30.5
2b ($n=7$)	161	SmC	24.9	31.75
2c ($n=8$)	164	SmC	26.7	33
2d ($n=9$)	168	SmC	27.4	34.25
2e ($n=10$)	175	SmC	28.7	35.5
2f ($n=12$)	170	SmC	30.8	38
2g ($n=14$)	172	SmC	33.1	40.5

No other features were detected in the X-ray patterns that could directly support the existence of intermolecular interactions through H-bonds. However, two factors suggest that there are intermolecular associations. (i) It is unusual for the benzyl group to act as the terminal group in a mesogenic molecule, (ii) The overall increase in layer thickness d from the shorter ($n=6$) to the longer ($n=14$) homologue is 9.4 Å, i.e. 1.2 Å per methylene unit. If the layers contained non-associated molecules, this would mean that the hydrocarbon chains are in their all-*trans* conformation; furthermore, an increase of 1.2 Å per methylene group would imply that the chains are not tilted but oriented practically along the normal to the layers. As this is not possible in a smectic C mesophase, the molecules must be associated in a head-to-tail fashion, in such a way that

the layers consist of an internal sublayer containing the rigid moieties connected through H-bonds, and the hydrocarbon chains statistically extend upwards and downwards (see figure 3).

3. Experimental

The structures of the compounds were confirmed by ^1H NMR, ^{13}C NMR (Bruker AC-250P) and FTIR (Nicolet 550) spectra. Transition temperatures and textures of mesophases were determined by optical microscopy using an Ortholux Pol BK-11 polarizing microscope equipped with a Mettler FP 800 hot stage. The transition temperatures and enthalpies were investigated by differential scanning calorimetry using a Rheometric DSC-V calorimeter. Samples were encapsulated in aluminium pans and studied at a scanning rate of 5°Cmin^{-1} during heating and cooling. The instrument was calibrated using an indium standard (156.6°C , 28.44Jg^{-1}). The purity of the final products was evaluated by thin layer chromatography.

X-ray diffraction patterns were obtained with a pinhole camera (Antoon-Paar) operating with a point-focused Ni-filtered Cu- K_α beam. The samples were held in Lindemann glass capillaries (1 mm diam.) and heated with a variable temperature oven. The patterns were collected on flat photographic films, with the capillary axis and film perpendicular to the X-ray beam; spacings were obtained via Bragg's law.

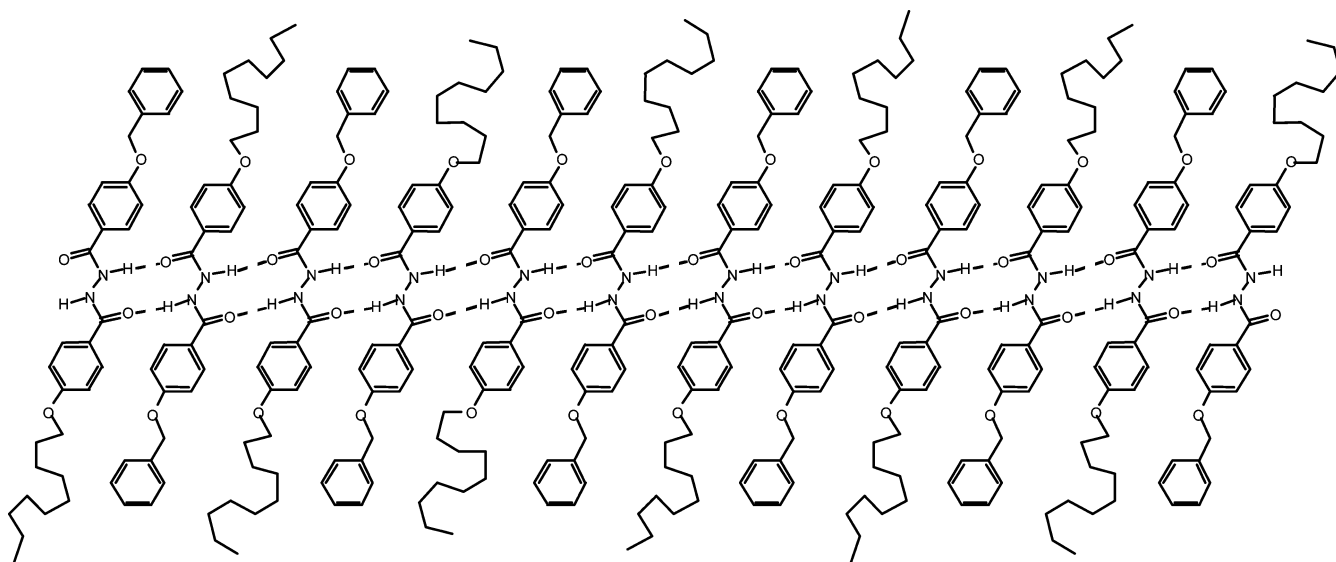


Figure 3. Schematic representation of the supramolecular structure of compounds **2**.

3.1. 4-Benzyloxyphenyl-2-carboxylic acid hydrazide (**1**)

This compound was synthesized according to the procedure described in [23]; yield 84%, m.p. 139°C.

3.2. 4-*n*-Alkoxybenzoic acid *N'*-(4-benzyloxyphenyl-2-carbonyl)hydrazides (**2a–g**)

In a general method [23], 4-*n*-alkoxybenzoyl chloride (5.20 g, 16 mmol) was added to a solution containing 4.00 g (16 mmol) of compound **1** and 1.26 g (16 mmol) of pyridine in 50 ml of *N*-methyl-2-pyrrolidone (NMP). The reaction mixture was stirred for 12 h at room temperature and then poured into water. The solid product was filtered off and crystallized from ethanol. Yield (%): **2a** 74, **2b** 76, **2c** 80, **2d** 77, **2e** 83, **2f** 88, **2g** 75.

2a ($n=6$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.97$ (t, $J=6.63$ Hz, 3H, CH_3), 1.38–1.84 (m, 8H, 4 CH_2), 4.11 (t, $J=6.35$ Hz, 2H, OCH_2 of the alkoxy chain), 5.28 (s, 2H, OCH_2 of the benzyl group), 7.14 (d, $J=8.75$ Hz, 2H, 2 arom. H), 7.25 (d, $J=8.78$ Hz, 2H, 2 arom. H), 7.53 (m, 5H, 5 arom. H), 8.00 (d, $J=2.35$ Hz, 2H, 2 arom. H), 8.01 (d, $J=2.30$ Hz, 2H, 2 arom. H), 10.43 (s, 2H, N–H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=13.8$, 22.0, 25.0, 28.4, 30.9 (aliph. C), 67.6 (OCH_2 of the alkoxy chain), 69.3 (OCH_2 of the benzyl group), 114.0, 114.4, 127.7, 127.9, 128.4, 129.3 (arom. C), 124.7, 125.1, 136.7, 161.2, 161.6 (quaternary arom. C), 165.6 (C=O). IR (KBr disk): $\text{cm}^{-1}=3240$ (N–H), 3058 ($\text{Csp}^2\text{--H}$), 2930 ($\text{Csp}^3\text{--H}$), 1642 (C=O), 1610 (C=C).

2b ($n=7$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.95$ (t, $J=6.58$ Hz, 3H, CH_3), 1.36–1.84 (m, 10H, 5

CH_2), 4.11 (t, $J=6.35$ Hz, 2H, OCH_2 of the alkoxy chain), 5.28 (s, 2H, OCH_2 of the benzyl group), 7.11 (d, $J=8.80$ Hz, 2H, 2 arom. H), 7.21 (d, $J=8.83$ Hz, 2H, 2 arom. H), 7.47 (m, 5H, 5 arom. H), 8.01 (d, $J=2.75$ Hz, 2H, 2 arom. H), 8.04 (d, $J=2.75$ Hz, 2H, 2 arom. H), 10.44 (s, 2H, N–H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=14.1$, 22.2, 25.6, 28.6, 30.7, 31.4 (aliph. C), 67.8 (OCH_2 of the alkoxy chain), 69.5 (OCH_2 of the benzyl group), 114.2, 114.6, 127.9, 128.1, 128.6, 129.5 (arom. C), 124.7, 125.1, 136.7, 161.2, 161.6 (quaternary arom. C), 165.6 (C=O). IR (KBr disk): $\text{cm}^{-1}=3239$ (N–H), 3057 ($\text{Csp}^2\text{--H}$), 2928 ($\text{Csp}^3\text{--H}$), 1644 (C=O), 1608 (C=C).

2c ($n=8$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.98$ (t, $J=6.55$ Hz, 3H, CH_3), 1.40–1.85 (m, 12H, 6 CH_2), 4.12 (t, $J=6.40$ Hz, 2H, OCH_2 of the alkoxy chain), 5.30 (s, 2H, OCH_2 of the benzyl group), 7.15 (d, $J=8.71$ Hz, 2H, 2 arom. H), 7.27 (d, $J=8.73$ Hz, 2H, 2 arom. H), 7.55 (m, 5H, 5 arom. H), 8.01 (d, $J=2.40$ Hz, 2H, 2 arom. H), 8.03 (d, $J=2.39$ Hz, 2H, 2 arom. H), 10.45 (s, 2H, N–H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=14.0$, 22.1, 25.5, 28.5, 28.6, 28.7, 31.3 (aliph. C), 67.8 (OCH_2 of the alkoxy chain), 69.5 (OCH_2 of the benzyl group), 114.2, 114.6, 127.8, 128.0, 128.5, 129.4 (arom. C), 124.7, 125.1, 136.7, 161.1, 161.4 (quaternary arom. C), 165.5 (C=O). IR (KBr disk): $\text{cm}^{-1}=3235$ (N–H), 3056 ($\text{Csp}^2\text{--H}$), 2925 ($\text{Csp}^3\text{--H}$), 1645 (C=O), 1610 (C=C).

2d ($n=9$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.95$ (t, $J=6.50$ Hz, 3H, CH_3), 1.35–1.84 (m, 14H, 7 CH_2), 4.14 (t, $J=6.38$ Hz, 2H, OCH_2 of the alkoxy chain), 5.29 (s, 2H, OCH_2 of the benzyl group), 7.14 (d, $J=8.69$ Hz, 2H, 2 arom. H), 7.25 (d, $J=8.70$ Hz, 2H,

2 arom. H), 7.54 (m, 5H, 5 arom. H), 8.02 (d, $J=2.38$ Hz, 2H, 2 arom. H), 8.05 (d, $J=2.40$ Hz, 2H, 2 arom. H), 10.46 (s, 2H, N-H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=13.9, 22.0, 25.4, 28.6, 28.7, 28.9, 31.2$ (aliph. C), 67.7 (OCH₂ of the alkoxy chain), 69.4 (OCH₂ of the benzyl group), 114.1, 114.5, 127.7, 127.9, 128.4, 129.3 (arom. C), 124.7, 125.1, 136.6, 161.0, 161.4 (quaternary arom. C), 165.4 (C=O). IR (KBr disk): $\text{cm}^{-1}=3237$ (N-H), 3056 (Csp²-H), 2926 (Csp³-H), 1643 (C=O), 1615 (C=C).

2e ($n=10$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.94$ (t, $J=6.60$ Hz, 3H, CH₃), 1.34–1.82 (m, 16H, 8 CH₂), 4.12 (t, $J=6.40$ Hz, 2H, OCH₂ of the alkoxy chain), 5.28 (s, 2H, OCH₂ of the benzyl group), 7.12 (d, $J=8.90$ Hz, 2H, 2 arom. H), 7.22 (d, $J=8.91$ Hz, 2H, 2 arom. H), 7.52 (m, 5H, 5 arom. H), 7.97 (d, $J=3.45$ Hz, 2H, 2 arom. H), 8.00 (d, $J=3.48$ Hz, 2H, 2 arom. H), 10.39 (s, 2H, N-H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=14.1, 22.2, 25.6, 28.7, 28.8, 29.1, 31.4$ (aliph. C), 67.8 (OCH₂ of the alkoxy chain), 69.5 (OCH₂ of the benzyl group), 114.3, 114.7, 127.9, 128.1, 128.6, 129.5 (arom. C), 124.7, 125.1, 136.7, 161.2, 161.6 (quaternary arom. C), 165.6 (C=O). IR (KBr disk): $\text{cm}^{-1}=3237$ (N-H), 3057 (Csp²-H), 2924 (Csp³-H), 1644 (C=O), 1612 (C=C).

2f ($n=12$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.95$ (t, $J=6.56$ Hz, 3H, CH₃), 1.34–1.82 (m, 20H, 10 CH₂), 4.13 (t, $J=6.41$ Hz, 2H, OCH₂ of the alkoxy chain), 5.29 (s, 2H, OCH₂ of the benzyl group), 7.12 (d, $J=8.88$ Hz, 2H, 2 arom. H), 7.23 (d, $J=8.90$ Hz, 2H, 2 arom. H), 7.53 (m, 5H, 5 arom. H), 7.98 (d, $J=3.75$ Hz, 2H, 2 arom. H), 8.01 (d, $J=3.78$ Hz, 2H, 2 arom. H), 10.40 (s, 2H, N-H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=14.0, 22.1, 25.5, 28.7, 28.8, 29.1, 31.3$ (aliph. C), 67.8 (OCH₂ of the alkoxy chain), 69.5 (OCH₂ of the benzyl group), 114.2, 114.6, 127.9, 128.0, 128.5, 129.4 (arom. C), 124.6, 125.0, 136.7, 161.1, 161.5 (quaternary arom. C), 165.5 (C=O). IR (KBr disk): $\text{cm}^{-1}=3240$ (N-H), 3058 (Csp²-H), 2923 (Csp³-H), 1645 (C=O), 1610 (C=C).

2g ($n=14$). ^1H NMR (DMSO- d_6 , TMS, 250 MHz): $\delta=0.90$ (t, $J=6.60$ Hz, 3H, CH₃), 1.30–1.85 (m, 24H, 12 CH₂), 4.10 (t, $J=6.46$ Hz, 2H, OCH₂ of the alkoxy chain), 5.25 (s, 2H, OCH₂ of the benzyl group), 7.10 (d, $J=8.83$ Hz, 2H, 2 arom. H), 7.21 (d, $J=8.85$ Hz, 2H, 2 arom. H), 7.49 (m, 5H, 5 arom. H), 8.02 (d, $J=3.94$ Hz, 2H, 2 arom. H), 8.01 (d, $J=3.98$ Hz, 2H, 2 arom. H), 10.40 (s, 2H, N-H). ^{13}C NMR (DMSO- d_6 , TMS, 62.9 MHz): $\delta=13.9, 21.9, 22.3, 25.6, 28.7, 28.8, 29.1, 30.8, 31.4$ (aliph. C), 67.8 (OCH₂ of the alkoxy chain), 69.5 (OCH₂ of the benzyl group), 114.2, 114.6, 127.9, 128.6, 128.8, 129.4 (arom. C), 124.7, 125.1, 136.7, 161.2, 161.6 (quaternary arom. C), 165.6 (C=O). IR

(KBr disk): $\text{cm}^{-1}=3241$ (N-H), 3056 (Csp²-H), 2922 (Csp³-H), 1644 (C=O), 1612 (C=C).

Acknowledgement

This work was supported by FONDECYT (grants 1030696 and 7040041) and 'Dirección de Investigación, Universidad de Concepción'.

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