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Journal of Photochemistry and Photobiology A: Chemistry 171 (2005) 237–242

Journal of Photochemistry and Photobiology A:Chemistry

www.elsevier.com/locate/jphotochem

Sunlight-induced reactions of some heterocyclic bases with ethers in the presence of TiO_2
A green route for the synthesis of heterocyclic aldehydes

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Received 28 July 2004; received in revised form 20 October 2004; accepted 26 October 2004
Available online 8 December 2004

Abstract

The reactions between various heterocyclic bases and ethers induced by sunlight are reported. In several cases the photoreaction occurred with higher yields in liquid–solid heterogeneous system in the presence of polycrystalline TiO_2 than in homogeneous system. The derivatives obtained with trioxane may give an easy entry to heterocyclic aldehydes.

Keywords: Photocatalysis; TiO_2; Sunlight; Ethers; Heterocycles; Aldehydes

In pursuing our research on sunlight-induced processes, we focused our attention on the reactions between the heterocyclic bases and ethers in the presence of TiO_2. The use of the solar energy as one of the most renewable and less pollutant reagent is achieving larger interest as the concern regarding our environment is taking place. Recently, we published the sunlight-induced reaction between some protonated heterocyclic bases and amides (formamide, *N*,*N*-dimethylformamide, *N*,*N*-dimethylacetamide) in the presence or in the absence of TiO_2 (anatase). We found that the exposition to the sunlight allows the introduction of the amide moiety on almost all the bases used, and the presence of TiO_2 increases the yields of the final products [1]. As we pointed out, some aspects of the mechanism of the reaction are rather unclear, but if the kind of products and the selectivity of the attack to the protonated bases are considered, a radical mechanism must be operating. In the attempt to enlarge the feasibility of the reaction to other classes of molecules and to search evidences to elucidate the operating mechanism, we draw our attention to the reaction of protonated quinoline (**1**) with some ethers, like tetrahydrofurane (**2**), tetrahydropyrane (**3**), dioxane (**4**), diethylether (**5**) and dioxolane (**6**).

The possibility to generate a radical on the carbon atom α to the oxygen is widely reported in the literature [2–4].

These radicals efficiently attack the protonated heterocyclic bases on the positions α or γ to the heterocyclic nitrogen. As far we know, this is the first time that data regarding this kind of free radical functionalisation of heterocyclic bases induced by sunlight are presented. In most cases, the presence of TiO_2 is necessary to initiate the reaction and generally is beneficial to obtain higher yields on respect to the reactions without TiO_2 whereas it does not affect the regioselectivity of the product(s) formed, that is the reactivity of the radicals implied.

In Scheme 1 and Table 1, the products obtained by irradiating with sunlight different ethers in the presence of protonated quinoline (**1**) are reported.

To extend the applicability of the reaction, quinoline (**1**) and other heterocyclic bases, namely quinaldine (**19**), lepi-

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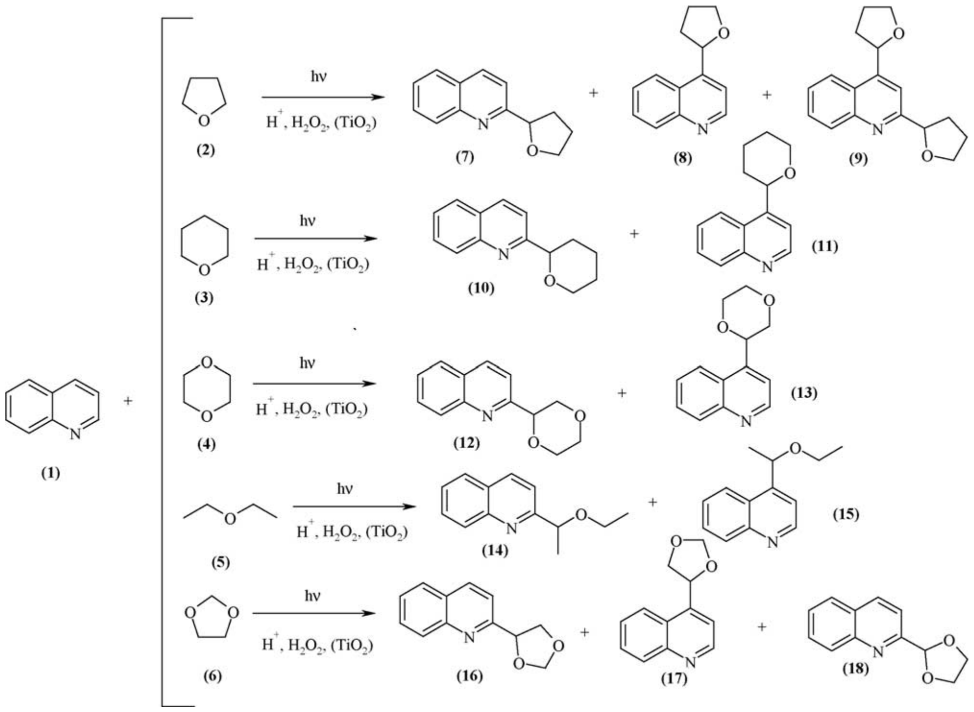
doi:10.1016/j.jphotochem.2004.10.017



Scheme 1.

dine (**20**), quinoxaline (**21**), isoquinoline (**22**) and 4-cyanopyridine (**23**), were irradiated with and without TiO_2, in the presence of trioxane (**24**), in order to obtain the target masked aldehyde. The reaction showed to be general as reported in Scheme 2 and Table 2. The presence of TiO_2 appeared to be beneficial or essential for obtaining the final products, although an exhaustive explanation is not straightforward.

No reactions occurred in the dark either at room temperature or refluxing the mixture, demonstrating that light activation is essential. The data show that the reaction is fairly general and may be used for the functionalisation of the heterocyclic bases with each of the used ethers. Particularly interesting are the derivatives obtained with trioxane considering that their acidic hydrolysis affords excellent yields (88–95%) to the heterocycle bearing a formyl group in the α or γ position, as reported in the literature [5]. We like to point out that similar products were obtained by Minisci and coworkers [2] using for example iron(II) and H_2O_2 to create OH radicals that are able to abstract an hydrogen atom from suitable donors. Our system is simpler and more environment friendly for the conditions used and the amount of chemical employed, while the yields depend on irradiation time and irradiance condition (product (**27**), for instance, was obtained in 44% yield after 35 h of sunlight irradiation). It is worth noting that the reaction conditions have not been optimised. Another point to stress is the fact that for most of the ethers we were obliged to use a mixture of solvents; it was possible to run the reaction between tetrahydrofurane (**2**) and quinoline (**1**) having the ether as the only solvent and obtaining 100% conversion after 10 h, with products distribution of (**7**) 13%, (**8**) 3% and (**9**) 84%. The finding that the product (**9**) is the most abundant one, suggests that a great amount of radical is formed. By considering that for the kind of products obtained it is possible to recognize a

Table 1
Products and yields obtained irradiating quinoline and ethers with and without TiO_2 after 15 h of irradiation

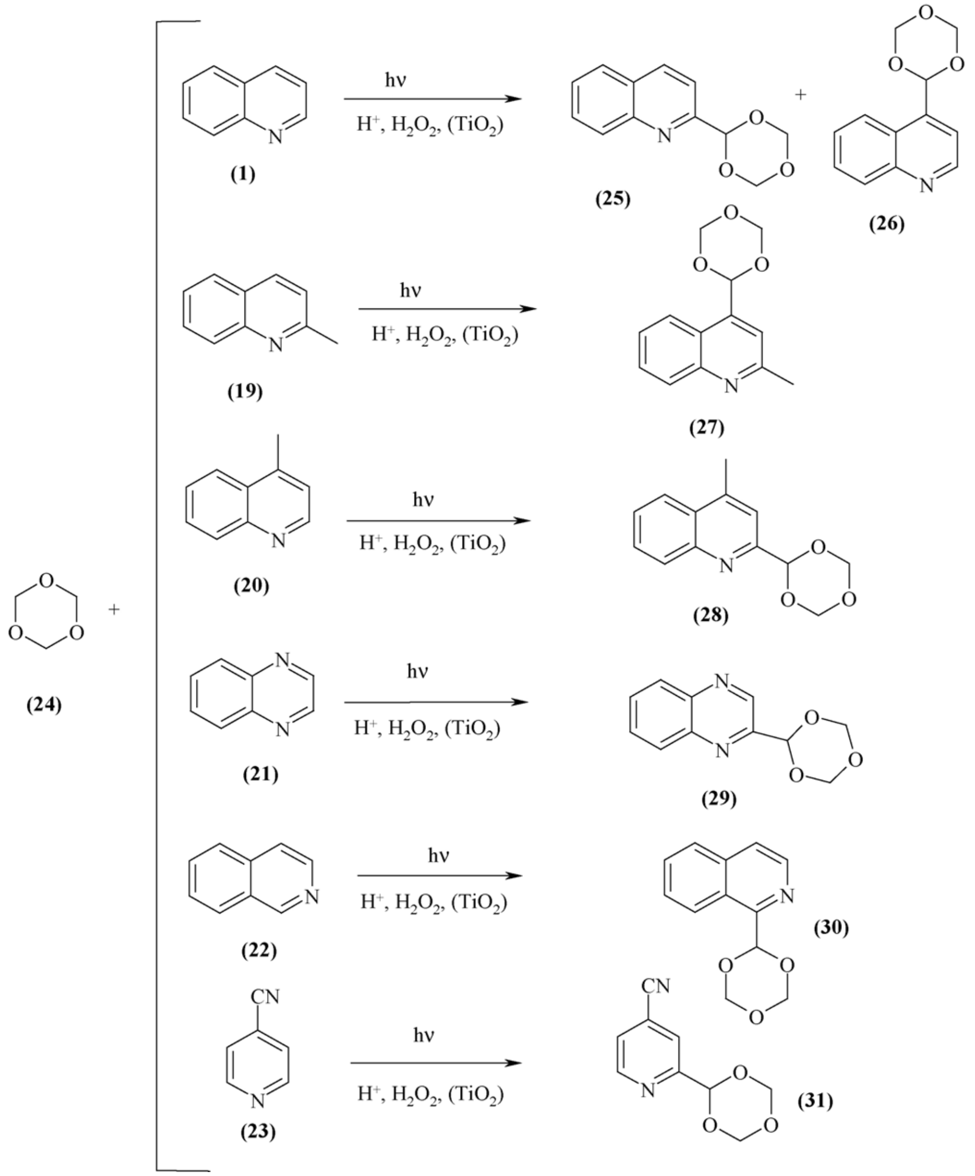
Ether	With 10 mg of TiO_2 anatase		Without TiO_2	
	(Products) ratio	Yield (%)	(Products) ratio	Yield (%)
(**2**)	(**7**) 83, (**8**) 12, (**9**) 5	75	(**7**) 88, (**8**) 12	34
(**3**)	(**10**) 31, (**11**) 69	25	(**10**) 33, (**11**) 67	18
(**4**)	(**12**) 39, (**13**) 61	26	(**12**) 36, (**13**) 64	12
(**5**)	(**14**) 27, (**15**) 73	45	–	0
(**6**)	(**16**) 31, (**17**) 42, (**18**) 27	33	–	0

Note: The reactions were run irradiating 1 mmol of base, 1 mmol of trifluoroacetic acid and 4 mmol of H_2O_2 in 10 mL of ether in H_2O or H_2O and CH_3CN (see experimental). Yields on converted bases are quantitative.

Table 2
Products and yields for the reactions between trioxane (**24**) and the different heterocyclic bases after 15 h of irradiation

Base	Products	Without TiO_2	Anatase (10 mg)	Rutile (10 mg)
(**1**)	(**25**) + (**26**)	3 + 5	8 + 17	9 + 19
(**19**)	(**27**)	–	15	18
(**20**)	(**28**)	4	15	40
(**21**)	(**29**)	25	31	29
(**22**)	(**30**)	44	24	26
(**23**)	(**31**)	–	20	7

Note: The reactions were run irradiating 1 mmol of base, 1 mmol of trifluoroacetic acid and 4 mmol of H_2O_2 in 40 mL of H_2O and 5.6 mmol of trioxane. Yields on converted bases are quantitative.

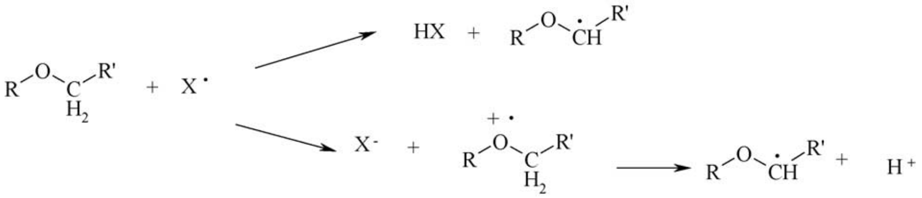


Scheme 2.

free radical reaction, the steps necessary for their formation are: the production of the α-oxyalkyl radical and the attack of this radical to the protonated heterocyclic base followed by the rearomatisation yielding to the final product. Minisci and coworkers published a large number of papers regarding the attack of the nucleophilic radicals to the heterocyclic bases and the subsequent rearomatisation, while the formation of the α-oxyalkyl radical here reported without TiO_2 (as well as the radicals formed in the case of the amides, in the previous paper) remains an open question. Regarding the reaction in the absence of TiO_2, it is possible to make two hypotheses:

(1) It could be considered that few H_2O_2 molecules decompose and initiate the reaction, although most of the UV radiation of the lamp with $\lambda < 290$ nm is cut by the walls of the Pyrex photoreactor and only a very small fraction can reach the solution and excite H_2O_2 whose extinction coefficient becomes negligible for radiation with $\lambda > 300$ nm. Nevertheless the same bases do not react at all without TiO_2 (see Tables 1 and 2), and this indicates, in our opinion, that OH is not formed by direct irradiation in the system used.

(2) Some light could be absorbed by the base that is able to initiate the reaction in its excited state (for example via electron transfer from the ether), but this explanation is not completely satisfying. Indeed quinoline gives certain amount of products with some ethers but not with others, while quinaldine and lepidine (with practically the same UV spectrum) give rise to a different behaviour with the same ether: the first forms some products, the second one does not react at all.



Scheme 3.

The data reported in the tables show that the presence of TiO_2 is essential in several cases, showing its involvement in a key reaction step. To better understand its involvement, another form of TiO_2, Rutile, was employed, and the results are reported in Table 2. From these data it is possible to assign to the presence of TiO_2 a crucial step in the formation of the α-oxyalkyl radicals. Two ways may operate in the creation of these radicals: either the hydrogen abstraction from the carbon atom α to the oxygen, or an electron transfer from the oxygen followed by deprotonation from the carbon α to the oxygen radical cation, as depicted in Scheme 3.

As reported in the literature [6–8], a possible explanation may lay in the fact that hydrogen peroxide could react with TiO_2 forming OH radicals either directly or via hydroperoxide species (TiOOH), that can decompose under irradiation. These radicals would abstract the hydrogen from the ether.

Another hypothesis may involve a reaction of the ether with photoproduced holes on the surface of excited TiO_2.

The finding that the yield increases by increasing the amount of H_2O_2 (see Table 3) strongly suggests that H_2O_2 adsorbs and reacts onto TiO_2 surface sites producing an increasing amount of OH radicals. The existence of a plateau for the highest concentrations of H_2O_2 could be due to a surface saturation effect. A possible hydrogen abstraction of H_2O_2 by hydroxyl radicals, more likely if a high production of OH radicals occurred by interaction light-H_2O_2, cannot be excluded, although this appears negligible under the experimental conditions used.

The significant photoactivity of rutile TiO_2 and the higher yields generally found with anatase TiO_2 (see Table 2), with the exception of 4-cyanopyridine (**23**) and quinoxaline (**21**), can not be simply explained by taking into account differences in the specific surface areas or in the intrinsic electronic factors of the photocatalysts. Indeed the specific surface areas of the two samples used throughout this work are very similar and the band gap and the conduction band values of TiO_2 (rutile) and TiO_2 (anatase), by considering the energy of the used radiation, are suitable for the occurrence of the (e^-–h^+) separation and for the electron transfer both to O_2 and H_2O_2, respectively.

Table 3
Effect of the amount of H_2O_2

H_2O_2 mmol	(**30**) Yield (%)
0.25	5
0.5	9
1.0	24
5.0	24
10.0	24

Note: Isoquinoline 1 mmole, CF_3COOH 1 mmol, trioxane 5.6 mmol and TiO_2 Anatase 10 mg in 40 mL of H_2O.

A tentative explanation would take into account the different physico-chemical surface properties of the two polymorphs influencing H_2O_2 adsorption [9], but in addition the different extent of adsorption–desorption of the heterocyclic bases, ethers and their intermediates and final products, due to different polarity and different acid–base properties of the surface hydroxyl groups, cannot be excluded.

It is worth noting that the photoactivity of rutile phase in this kind of photoreactions could be due to the fact that adsorption–desorption characteristics of H_2O_2 and O_2 would favour adsorption of the first [10–11]. It has been found that TiO_2 (rutile) works similarly to TiO_2 (anatase) for phenol photo-oxidation in aqueous medium when H_2O_2 or Ag^+ are used as electron scavengers kinetically more efficient than O_2 [12].

Although a conclusive explanation of the behaviour of 4-cyanopyridine (**23**), and at a minor extent of quinoxaline (**21**), is not simple, it is worth noting that the pK_a values of these heterocyclic bases are the lowest ones and this suggests a possible different acid–base interaction on the surface of the two TiO_2 samples used (pK_a values: quinoline 4.85, isoquinoline 5.14, quinoxaline 0.6, quinaldine 5.42, lepidine 5.20 and 4-cyanopyridine 1.90).

Experimental part. Mass spectra were performed on a GC–MS instrument, using a gas chromatograph equipped with SBP-1 fused silica column (30 m × 0.2 mm i.d., 0.2 mm film thickness) and helium as carrier gas. NMR spectra were carried out on a Bruker Avance spectrometer operating at 250 MHz (^1H). ^1H chemical shifts are referred to internal TMS.

General procedure. The experimental conditions are slightly different in dependence of the ether used. A Pyrex tube containing a solution of the ether (10 mL for the liquid tetrahydrofurane (**2**), tetrahydropyrane (**3**), 1,4-dioxane (**4**), diethylether (**5**) or 1,3-dioxolane (**6**), 5 g for the solid trioxane (**24**)) in water (30 mL for (**2**), (**4**) or (**6**), 40 mL for (**24**), 5 mL + 10 mL of CH_3CN for (**3**) or (**5**)) and 10 mg of polycrystalline Anatase TiO_2 (Merck, BET specific surface area: 10 $m^2 g^{-1}$) or Rutile TiO_2 (Tioxide, BET specific surface area: 8 $m^2 g^{-1}$) was sonicated for 1 min to obtain a homogeneous suspension. The heterocyclic base (1 mmol), CF_3COOH (1 mmol) and H_2O_2 (4 mmol) were added to the suspension and the resulting mixture was exposed to the sunlight, (mean irradiance in Milan in the period December–February is 200 W m^{-2}) with a mechanical stirring (1000 rpm) for 15 h. At the end of the irradiation, the solution was alkalinised with NaOH 10%, extracted with CH_2Cl_2 and dried on Na_2SO_4. The solvent is evacuated under vacuum, and the resulting mixture was either analysed via GC-mass or separated via flash-chromatography (silica–gel, solvent ethyl acetate:hexane 7:3, except for quinoline and (**6**) where the eluent was ethyl acetate:hexane 1:1). For compounds already reported in literature, their identification was made by comparison with the authentical samples.

(**7**) Oil; mass (*m/z*) 199 M^+, 156, 143 and 128; NMR ($CDCl_3$): 8.36 (d, 8 Hz, 1H, CH), 8.12 (d, 8 Hz, 1H, CH), 7.90–7.60 (m, 4H, 4 CH), 5.38 (m, 1H, CH), 4.20 (m, 1H, CH), 4.02 (m, 1H, CH) and 2.10 (m, 4H, 2 CH_2); Anal. Calcd. $C_{13}H_{13}NO$: C 78.36, H 6.58 and N 7.03; found C 78.06, H 6.55 and N 7.06.

(**8**) Oil; mass (*m/z*) 199 M^+, 198, 156, 143 and 129; NMR ($CDCl_3$): 8.83 (d, $J = 4$ Hz, 1H, CH), 8.12 (d, $J = 8$ Hz, 1H, CH), 8.13 (d, $J = 7$ Hz, 1H, CH), 7.70 (dd, $J = 7$ Hz and 1 Hz, 1H, CH), 7.57 (m, 2H, 2 CH), 5.60 (t, $J = 7$ Hz, 1H, CH), 4.22 (m, 1H, CH), 4.03 (m, 1H, CH), 2.60 (m, 1H, CH), 2.04 and 2.00 (ms, 4H, 2CH_2); Anal. Calcd. $C_{13}H_{13}NO$: C 78.36, H 6.58 and N 7.03; found C 78.68, H 6.60 and N 7.00.

(**9**) Oil; mass (*m/z*) 269 M^+, 226 and 156; NMR ($CDCl_3$): 8.13 (m, 1H, CH), 7.90, (m, 1H, CH), 7.76 (m, 2H, 2 CH), 7.51 (m, 1H, CH), 7.58 (m, 1H, CH), 5.18 (m, 1H, CH), 4.19 (m, 2H, CH_2), 4.04 (m, 2H, CH_2), 2.09, 2.05 and 1.90 (ms, 8H, 4 CH_2); Anal. Calcd. $C_{17}H_{19}NO_2$: C 75.81, H 7.11 and N 5.20; found C 75.61, H 7.08 and N 5.18.

(**10**) Oil; mass (*m/z*) 213 M^+, 185, 156 and 129; NMR ($CDCl_3$): 8.18 (d, 9 Hz, 1H, CH), 8.08 (d, 9 Hz, 1H, CH), 7.80 (d, 8 Hz, 1H, CH), 7.69 (ms, 1H, CH), 7.62 (d, 9 Hz, 1H, CH), 7.51 (ms, 1H, CH), 4.66 (dd, 11 Hz and 2 Hz, 1H, CH), 4.22 (m, 1H, CH), 3.70 (m, 1H, CH), 2.10, 1.95 and 1.69 (ms, 6H, 3 CH_2); Anal. Calcd. $C_{14}H_{15}NO$: C 78.84, H 7.09 and N 6.57; found C 78.56, H 7.11 and N 6.54.

(**11**) Oil; mass (*m/z*) 213 M^+, 185, 156, 130 and 129; NMR ($CDCl_3$): 8.91 (d, 4 Hz, 1H, CH), 8.19 (d, 8 Hz, 1H, CH), 8.01 (d, 8 Hz, 1H, CH), 7.72 (m, 1H, CH), 7.59 (ms, 2H, 2CH), 5.06 (d, 9 Hz, 1H, CH), 4.27 (dd, 11 Hz and 2 Hz, 1H, CH), 3.75 (m, 1H, CH), 2.04 and 1.76 (ms, 6H, 3 CH_2); Anal. Calcd. $C_{14}H_{15}NO$: C 78.84, H 7.09 and N 6.57; found C 79.16, H 7.06 and N 6.60.

(**12**) m.p. 63–65 °C (lit. 63–65) [13].

(**13**) m.p. 58–60 °C (lit. 60–61) [13].

(**14**) Oil; mass (*m/z*) 200 M^+-1, 158, 157, 129 and 128; NMR ($CDCl_3$): 8.20 (d, 9 Hz, 1H, CH), 8.09 (d, 9 Hz, 1H, CH), 7.82 (d, 4 Hz, 1H, CH), 7.72 (m, 1H, CH), 7.62 (d, 11 Hz, 1H, CH), 7.53 (m, 1H, CH), 4.73 (q, 6 Hz, 1H, CH), 3.41 (m, 2H, CH_2), 1.54 (d, 6 Hz, 3H, CH_3) and 1.22 (t, 7 Hz, 3H, CH_3); Anal. Calcd. $C_{13}H_{15}NO$: C 7.58, H 7.51 and N 6.96; found C 77.31, H 7.53 and N 6.97.

(**15**) Oil; mass (*m/z*) 201 M^+, 158, 157, 156, 154 and 130; NMR ($CDCl_3$): 8.90 (d, 4 Hz, 1H, CH), 8.12, (m, 2H, 2 CH), 7.71 (t, 8 Hz, 1H, CH), 7.56 (d, 8 Hz, 1H, CH), 7.50 (d, 4 Hz, 1H, CH), 5.14 (q, 6 Hz, 1H, CH), 3.44 (m, 2H, CH_2), 1.57 (d, 7 Hz, 3H CH_3) and 1.24 (t, 8 Hz, 3H, CH_3); Anal. Calcd. $C_{13}H_{15}NO$: C 77.58, H 7.51 and N 6.96; found C 77.34, H 7.48 and N 6.95.

(**16**) Oil; mass (*m/z*) 201 M^+, 200, 171, 170, 156, 143, 142 and 128; NMR ($CDCl_3$): 8.21 (d, 9 Hz, 1H, CH), 8.05 (d, 9 Hz, 1H, CH), 7.83 (d, 8 Hz, 1H, CH), 7.73 (m, 1H, CH), 7.64 (d, 7 Hz, 1H, CH), 7.54 (m, 1H, CH), 5.33 (s and t, 8 Hz) and 5.16 (t, 7 Hz and s, 3H, CH and CH_2), 4.43 (t, 8 Hz, 1H, CH) and 4.09 (dd 7 and 8 Hz, 1H, CH); Anal. Calcd. $C_{12}H_{11}NO_2$: C 71.63, H 5.51 and N 6.96; found C 71.83, H 5.50 and N 7.00.

(**17**) Oil; mass (*m/z*) 201 M^+, 171, 170, 155, 154, 143, 142 and 115; NMR ($CDCl_3$): 8.93 (d, 4 Hz, 1H, CH), 8.19 (d, 8 Hz, 1H, CH), 7.76 and 7.60 (2 ms, 4H, 4 CH), 5.72 (t, 7 Hz, 1H, CH), 5.35 (s) and 5.20 (s, 2H, CH_2), 4.56 (t, 8 Hz, 1H, CH) and 3.76 (dd, 7 and 8 Hz, 1H, CH); Anal. Calcd. $C_{12}H_{11}NO_2$: C 71.63, H 5.51 and N 6.96; found C 71.44, H 5.52 and N 6.94.

(**18**) Oil; mass (*m/z*) 201 M^+, 200, 129, 128 and 73; NMR ($CDCl_3$): 8.17 (d, 8 Hz, 1H, CH), 7.66, 7.60 (2 m, 5H, 5 CH), 6.46 (s, 1H, CH) and 4.15 (s, 4H, 2 CH_2); Anal. Calcd. $C_{12}H_{11}NO_2$: C 71.63, H 5.51 and N 6.96; found C 71.71, H 5.52 and N 6.96.

(**25**) m.p. 95–96 °C; mass (*m/z*) 217 M^+, 157, 130, 129 and 128; NMR ($CDCl_3$): 8.33 (d, 8 Hz, 1H, CH), 8.22 (d, 8 Hz, 1H, CH), 7.86, 7.78 and 7.62 (ms, 4H, aromatics), 6.22 (s, 1H, CH) and 5.44 (s, 4H, 2 CH_2); Anal. Calcd. $C_{12}H_{11}NO_3$: C 66.35, H 5.10 and N 6.45; found C 66.44, H 5.11 and N 6.47.

(**26**) m.p. 142–143 ° C; mass (*m/z*) 217 M^+, 157, 143, 130, 129 and 128; NMR ($CDCl_3$): 8.99 (d, 4 Hz, 1H, CH), 8.21 (t, 10 Hz, 2H, CH), 7.78, 7.73 and 7.63 (ms, 3 H, aromatics), 4.46 (s, 1H, CH) and 5.47 (s, 4H, 2 CH_2); Anal. Calcd. $C_{12}H_{11}NO_3$: C 66.35, H 5.10 and N 6.45; found C 66.60, H 5.08 and N 6.42.

(**27**) m.p. 160–161 °C (lit. 162) [5].

(**28**) m.p. 99–100 °C (lit. 100) [5].

(**29**) m.p. 142–145 °C (lit. 143–145) [5].

(**30**) m.p. 98–100° C; mass (*m/z*) 217 M^+, 158, 129 and 128; NMR ($CDCl_3$): 8.88 (d, 6 Hz, 1H, CH), 8.5 (d, 6 Hz, 1H, CH), 7.84 (dd, 7 Hz and 2 Hz, 1H, CH), 7.71 (ms, 3H, aromatics), 6.43 (s, 1H, CH), 5.49 and 5.43 (ds, 7 Hz, 4H, 2 CH_2); Anal. Calcd. $C_{12}H_{11}NO_3$: C 66.35, H 5.10 and N 6.45; found C 66.52, H 5.13 and N 6.46.

(**31**) m.p. 108–110 °C; mass (*m/z*) 191 M^+–H, 133, 104, 103 and 89; NMR ($CDCl_3$): 8.8 (d, 5 Hz, 1H, CH), 7.92 (d, 1.5 Hz, 1H, CH), 7.56 (dd, 5 and 1.5 Hz, 1H, CH), 5.98 (s, 1H, CH) and 5.38 (ds, 7 Hz, 4 H, 2 CH_2); Anal. Calcd. $C_9H_8N_2O_3$: C 56.25, H 4.20 and N 14.54; found C 56.44, H 4.18 and N 14.53.

Acknowledgment

We are indebted with Ministero dell’ Istruzione Universitaria e della Ricerca (MIUR).

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