DIASTEREOSELECTIVE ALKYLATION OF AROMATIC ENAMIDONE FUNCTIONALITY IN ISOINDOLE SERIES: A CONVENIENT ROUTE TO CHIRAL AZA-ANALOGUES OF SENKYUNOLIDE-E

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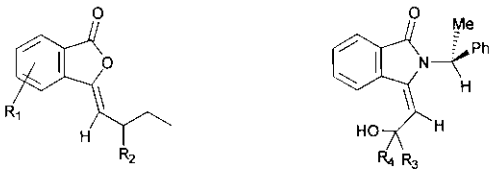
Abstract – Chiral aza-analogues (**2a**) and (**2b**) of Senkyunolide-E were efficiently synthesized in a four-step sequence (72 and 70 % overall yields respectively) from the commercially available phthalic anhydride and (*S*)-(-)-α-phenylethylamine. The key step consisted in the 1,2-addition of Grignard reagents onto enamidone type compounds (**8**) and (**9**). These latters were regioselectively obtained from the parent ω-alkynyl-ω-carbinol lactams (**6**) and (**7**) by a Meyer-Schuster rearrangement.

Monohydroxylated 3-alkylphthalides as compounds (**1**) (Scheme 1) are abundant in a variety of Chinese *Umbelliferous* plants[1,2] and are the major constituents of the dried rhizome of *Lingusticum wallichii* which is commonly known as "Chuan-Xiong" in China,[2] *Cnidium officinale* rhizome "Chuan-Xing" in Korea[3] and "Senkyu" in Chinese traditional medicine used in Japan.[4] Some of these structures possess significant biological properties as antispasmodic,[5] pesticide,[6] fungicide[7] agents and have been used in the treatment of anemia and women's disease.[3] According to this considerable interest in the pharmaceuticals applications, the syntheses of various 3-alkylidene (or 3-arylidene) derivatives (Senkyunolide-E (**1**): R_1=H, R_2=OH) have been recently achieved. [8,9]

Scheme 1

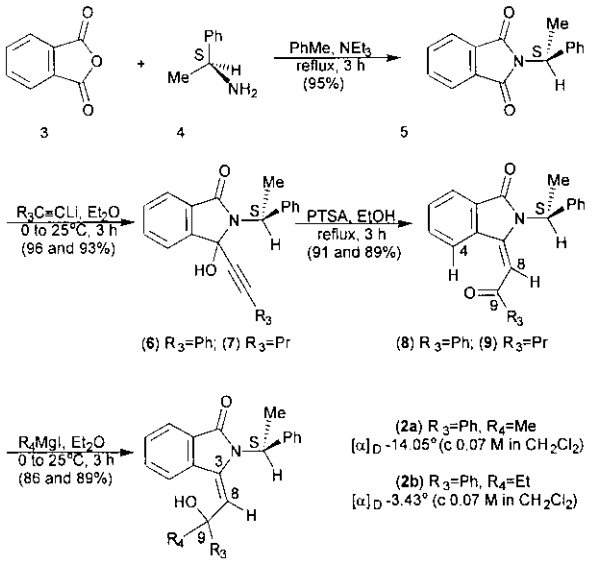


(1) Senkyunolide
B (R_1=4-OH, R_2=H)
C (R_1=5-OH, R_2=H)
E (R_1=H, R_2=OH)

(**2a**) R_3=Ph, R_4=Me
(**2b**) R_3=Ph, R_4=Et

As a part of our continuing investigations on synthetic approach to diversely substituted heterocyclic compounds containing phthalimidine moiety, we wish to describe herein a new approach to alkyl and aryl chiral aza-analogues (**2**) of 3-alkylidene- (or 3-arylidene-) phthalides (Scheme 1). Our strategy focused at the outset on construction of the enamidone functionality followed by transformation of its carbonyl group into hydroxy function. In all cases, phthalic anhydride (**3**) was used as a starting material as depicted in Scheme 2.

Scheme 2



3 4 5

(**6**) R_3=Ph; (**7**) R_3=Pr

(**8**) R_3=Ph; (**9**) R_3=Pr

(**2a**) R_3=Ph, R_4=Me
$[\alpha]_D$ -14.05° (c 0.07 M in CH_2Cl_2)

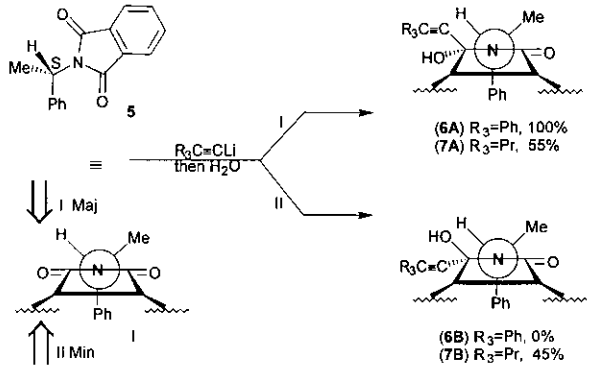
(**2b**) R_3=Ph, R_4=Et
$[\alpha]_D$ -3.43° (c 0.07 M in CH_2Cl_2)

Thus, warming condensation of phthalic anhydride (**3**) with (*S*)-(-)-α-phenylethylamine (**4**) in azeotropic conditions with catalytic amount of triethylamine[10] produced, after a flash chromatography on silica gel column, enantiopure (1*S*)-*N*-(1-phenylethyl)phthalimide (**5**) in high yield (95 %) [$[\alpha]_D$ = +42.57°±0.05 (c 0.2 M, CH_2Cl_2)]. This substrate (**5**) was reacted with phenylethynyl(or pentynyl)lithium (generated *in situ* from n-butyllithium (1.65 M solution in hexane) and phenylacetylene or pent-1-yne) in dry ether yielding the ω-phenylethynyl(or ω-pentynyl)-ω-carbinol lactam (**6**) or (**7**) in 96 and 93 % yields respectively. Surprisingly, the proton NMR spectra of **6** indicated a single hydroxy lactam diastereomer [$[\alpha]_D$ = +10.05°±0.05 (c 0.23 M, CH_2Cl_2)] while the one of **7** showed a 55/45 mixture of diastereomers

(**7A/7B**) unseparable by column chromatography. In the case of (**6**), the high diastereoselectivity observed for the organolithium addition to the prochiral imide carbonyl group center can be rationalized by considering the conformation (**I**) (Figure 1). Indeed, the steric hindrance of the methylbenzyl group induces a diastereofacial discrimination during approach of the incoming organometallic.

In fact, with lithium phenylacetylene the reaction seems to proceed through Felkin-Ahn like model as in conformation (**I**) which involved approach of the nucleophile from the upper side to give adduct (**6A**). The lowest level of diastereoselectivity observed with the pentynyllithium, could due to the fact that this nucleophile is less sterically demanding than the lithium phenylacetylene. Our results are in accordance with those obtained with electrophilic *N*-acyliminium ions[11-14] and imines[15] derivatives bearing chiral auxiliaries (Figure 1).

Figure 1

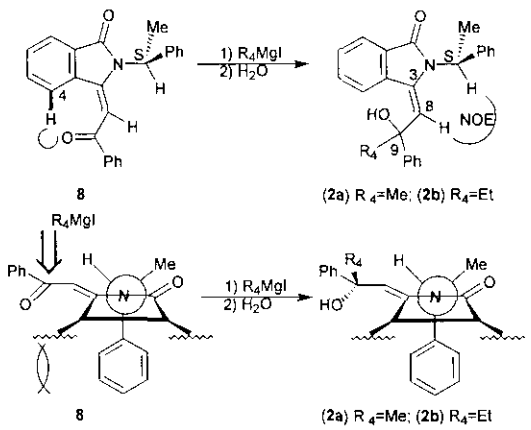


These resulting alcohols (**6**) or (**7**), gave readily the corresponding enamidones (**8**) (91 %) $[[\alpha]_D = -1.35° \pm 0.05$ (c 0.19 M, CH_2Cl_2)] or (**9**) (89 %) $[[\alpha]_D = +42.64° \pm 0.05$ (c 0.16 M, CH_2Cl_2)] in refluxing ethanol for 3 hours with catalytic amount of *p*-toluenesulfonic acid. In a previous paper, a similar Meyer-Schuster rearrangement with related compounds has been put in evidence.[16] At this stage, it is worth of mentioning that proton NMR spectra of enamidones (**8**) and (**9**) indicate a single diastereomer. Moreover, based on the signal of olefinic proton[16-20] and aromatic proton H(4)[18-20] we assess that 2-oxo-2-phenyl-ethylidene side chain in enamidones (**8**) or (**9**) has a *s-cis* conformation and *E* configuration .

The enamidone (**8**) was then reacted with a slight excess of Grignard reagent (1.2 eq) in anhydrous ether with dichloromethane or THF as co-solvent at 0°C to room temperature. Under these conditions, alkylation of the enamidone yields the title compounds (**2a**) (R_3=Ph, R_4=Me) $[[\alpha]_D = -14.05° \pm 0.05$ (c 0.07 M, CH_2Cl_2)] and (**2b**) (R_3=Ph, R_4=Et) $[[\alpha]_D = -3.43° \pm 0.05$ (c 0.07 M, CH_2Cl_2)] in 86 and 89 % yields respectively. The regioselectivity of the addition of these Grignard reagents was in accordance with 1,2-addition onto enone and no trace of adduct resulting from 1,4-addition reaction was detected. Furthermore (Scheme 2), the C(9) carbon of substrate (**8**) (or **9**) constitutes a prostereogenic center, and interestingly the alkylation reaction has led a single diastereomer (**2a**) or (**2b**). Finally, removal of the chiral auxiliary from these alcohols according to classical methods[11,13,21] did not give good results.

The absolute configuration of the newly induced asymmetric center of the enamidols (**2a**) and (**2b**) has not been yet elucidated. So, on the bases of a previous "perpendicular" model proposed by Houk and co-workers during theoretical study of asymetric Diels-Alder reactions,[22] we postulate a transition state (Figure **2**) in which the remote phenyl group would be orientated perpendiculary to the coplanar enamidone system. This model would best explain the observed stereospecificity due to the high steric hindrance of the phenyl group. Moreover, it was backed up by a prediction based on molecular modelling study and energy minimized structures of the *s-cis* conformation and *E* configuration of enamidone (**8**) and *E* configuration of enamidol (**2**) optimized under HyperChem version 5.1 AM1. As a result, the incoming nucleophile would exclusively approach on the opposite less hindered face to give products (**2**).

Figure 2



On the other hand, structure determination of products (**2a**) and (**2b**) are based again on ^{1}H and ^{13}C NMR spectroscopy experiments which were correlated with the model compounds (*Z*)- and (*E*)-3-benzylidene-phthalimidin-2-ylacetic acid derivatives.[20] Indeed, the vinylic proton at C(8) (δ = 5.72 ppm in (**2a**) and δ = 5.67 ppm in (**2b**)) was shifted to higher field (0.68 and 0.73 ppm respectively from the corresponding proton of **8** which was considerably deshielded due to the proximity of the carbonyl group). This shielding effect is similar to the one observed during reduction reaction of *Z*-(2-oxobutylidene)phthalide to corresponding *Z* and *E* senkyunolide-E[8] ($\Delta\delta$ = 0.4 ppm). These values indicate that the 2-hydroxy-2-phenylalkylidene side chain in **2a** and **2b** takes *E*-form as in the precursor (**8**). Moreover, this *E* attribution was confirmed by using NOE DIFFERENCE measurements of products (**2a**) and (**2b**). Furthermore, the C(4) aromatic proton of these enamidols is shielded too (0.81 and 0.83 ppm) compared to the same one in the parent enamidone (**8**) and appears as a doublet of doublet at δ=8.74 ppm with usual benzene coupling constants.

In summary, we reported preliminary results toward chiral isoindolones (**2**), aza-analogues of natural monohydroxylated phthalides (**1**), in four steps by regioselective Meyer-Schuster rearrangement followed by regio- and stereoselective 1,2-addition reaction.

EXPERIMENTAL PART

Melting points are uncorrected. The IR spectra of solids (potassium bromide) were recorded on a Perkin Elmer FTIR paragon 1000 spectrophotometer. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC-200 (200 MHz) instrument in deuteriochloroform and chemical shifts (δ) are expressed in ppm relative to TMS as an internal standard. Ascending TLC was performed on precoated plates of silica gel 60 F 254 (Merck) and the spots visualized using an ultraviolet lamp or iodine vapor. E. Merck silica gel 60 F (70-300 mesh) was used for column chromatography. Optical rotations were measured with a Perkin Elmer 241 polarimeter in a 10 cm cell in dichloromethane at 20°C. The elemental analyses were carried out by the microanalysis laboratory of INSA at Rouen, F 76130 Mont. Saint. Aignan, France. MS spectral measurements were carried out on a AEI MS 902 S spectrometer (70 eV, electron impact).

(1*S*)-*N*-(1-Phenylethyl)phthalimide (5). A mixture of (*S*)-(-)-α-phenylethylamine (**4**) (1.21 g, 10 mmol), phthalic anhydride (1.48 g, 10 mmol) and triethylamine (0.5 mL, 3.6 mmol) in toluene (50 mL) was refluxed with a Dean-stark apparatus for 3 h. The reaction mixture was cooled, then was concentrated under reduced pressure. The residue was dissolved into dichloromethane, washed with 10 % hydrochloric acid solution then with a sodium hydrogen carbonate solution. The organic layer was dried over magnesium sulfate, concentrated under reduced pressure, and recrystallization of the residue from ethanol gave (**5**). This product was obtained in 95% yield (2.38 g); mp 56°C; $[\alpha]_D$ = +42.57°±0.05 (c 0.2 M, CH_2Cl_2); IR (KBr): 3035 (CH), 2950 (CH), 1708 (C=O) cm^{-1}; ^{1}H NMR: δ 1.9 (d, *J* = 7.2 Hz, 3H, $CHC\underline{H}_3$), 5.55 (q, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 7.10-7.35 (m, 3H, $H_{benzene}$), 7.40-7.55 (m, 2H, $H_{benzene}$), 7.58-7.70 (m, 2H, $H_{phthalimide}$), 7.73-8.25 (m, 2H, $H_{phthalimide}$); ^{13}C NMR: δ 18.9 (Me), 51.7 (CH), 123.2 (2CH), 127.8 (2CH), 128.9 (2C), 133.5 (C), 135.1 (2C), 141.1 (C), 169.2 (2C), 177.4 (2CO); ms: (EI) *m/z* 251(M^+). *Anal.* Calcd for $C_{16}H_{13}NO_2$: C, 76.54; H, 5.21; N, 5.57. Found: C, 76.46; H, 5.18; N, 5.51.

General procedure for synthesis of hydroxylactams (6) and (7). To a stirred solution of phenylethynyl(or pentynyl)lithium (11 mmol) (generated *in situ* by reaction of n-butyllithium (11 mmol) (1.65 M solution in hexane) and phenylacetylene (or pent-1-yne) (11 mmol) in 50 mL of dry ether for 15 min) was added in portions (1*S*)-*N*-(1-phenylethyl)phthalimide (**5**) (2.51 g, 10 mmol) over a period of 15 min at 0°C. The mixture was stirred at same temperature for 1 h then at rt for 3 hours again. The reaction mixture was poured into 40 mL of 1 M ammonium chloride solution then was extracted twice with dichloromethane (20 mL). The organic layer was dried over magnesium sulfate and concentrated under reduced pressure to afford the expected hydroxy lactams (**6**) and (**7**) which recrystallized from dry ether.

(1'*S*)-2,3-Dihydro-3-hydroxy-3-phenylethynyl-2-(1'-phenylethyl)-1*H*-isoindol-1-one (6). This compound was obtained as a single diastereomer (**6A**) in 96% yield (3.39 g); mp 195°C; $[\alpha]_D$ = +10.05°±0.05 (c 0.23 M, CH_2Cl_2); IR (KBr): 3368 (br-OH), 3039 (CH), 2986 (CH), 2235 (C≡C), 1689 (C=O) cm^{-1}; ^{1}H NMR: δ 1.92 (d, *J* = 7.3 Hz, 3H, $CHC\underline{H}_3$), 2.82 (s, 1H, OH), 5.11 (q, *J* = 7.3 Hz, 1H,

$C\underline{H}CH_3$), 7.02-7.32 (m, 10H, $H_{benzene}$), 7.35-7.51 (m, 1H, $H_{phthalimide}$), 7.55-7.75 (m, 3H, $H_{phthalimide}$); ^{13}C NMR: δ 19.0 (CH_3), 51.5 (CH), 83.6 (C≡), 85.9 (C≡), 120.8 (C), 122.1 (2CH), 126.1 (CH), 126.5 (C), 126.8 (2CH), 127.4 (2CH), 127.6 (2CH), 128.3 (CH), 129.0 (CH), 130.6 (C), 131.1 (2CH), 131.8 (CH), 141.8 (C), 145.9 (C), 165.6 (CO)); ms: (EI) *m/z* 353 (M^+). *Anal.* Calcd for $C_{24}H_{19}NO_2$: C, 81.56; H, 5.42; N, 3.96. Found: C, 81.38; H, 5.38; N, 3.85.

(1'*S*)-2,3-Dihydro-3-hydroxy-3-(pentynyl)-2-(1'-phenylethyl)-1*H*-isoindol-1-one (7). This compound was obtained as a mixture of the unseparable diastereomers (**7A/7B**) (55/45) in 93% yield (2.97 g); ^1H NMR: major component: δ 0.9 (t, *J* = 7.2 Hz, 3H, $C\underline{H}_3CH_2CH_2$), 1.46 (m, *J* = 7.2 Hz, 2H, $CH_3C\underline{H}_2CH_2$), 1.85 (d, *J* = 7.0 Hz, 3H, $CHC\underline{H}_3$), 2.14 (m, *J* = 7.2 Hz, 2H, $CH_3CH_2C\underline{H}_2$), 4.01 (br, 1H, OH), 5.10 (q, *J* = 7.0 Hz, 1H, $C\underline{H}CH_3$), 6.96-7.23 (m, 3H, $H_{benzene}$), 7.26-7.45 (m, 2H, $H_{benzene}$), 7.48-7.71 (m, 4H, $H_{phthalimide}$); ^1H NMR: minor component: δ 0.79 (t, *J* = 7.2 Hz, 3H, $C\underline{H}_3CH_2CH_2$), 1.33 (m, *J* = 7.2 Hz, 2H, $CH_3\underline{CH}_2CH_2$), 1.83 (d, *J* = 7.2 Hz, 3H, $CHC\underline{H}_3$), 2.05 (m, *J* = 7.2 Hz, 2H, $CH_3CH_2C\underline{H}_2$), 4.01 (br, 1H, OH), 5.05 (q, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 6.96-7.23 (m, 3H, $H_{benzene}$), 7.26-7.45 (m, 2H, $H_{benzene}$), 7.48-7.71 (m, 4H, $H_{phthalimide}$); ms: (EI) *m/z* 319 (M^+). *Anal.* Calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.62; N, 4.38. Found: C, 78.85; H, 6.60; N, 4.32.

General procedure for preparation of enamidones (8) and (9). To a well stirred suspension of hydroxy lactam (**6**) or (**7**) (2.21 mmol) in dry ethanol (15 mL) was added a catalytic quantity of *p*-toluenesulfonic acid (0.05 g, 0.29 mmol) and the mixture was refluxed for 3 h. After cooling at rt, the solution was concentrated *in vacuo* and the residue was dissolved in dichloromethane (30 mL). The organic layer was washed with 2 M sodium bicarbonate solution, brine, water, dried over magnesium sulfate and concentrated under reduced pressure. The resulting solid was recrystallized from dry ether to give the expected enamidone (**8**) or (**9**) in good yields.

(*E*)-(1'*S*)-2,3-Dihydro-3-(2-oxo-2-phenylethylidene)-2-(1'-phenylethyl)-1*H*-isoindol-1-one (8). This compound was obtained as yellow solid as a *S* isomer in 91% yield (0.71 g); mp 113°C (ether); $[\alpha]_D$ = -1.35°±0.05 (c 0.19 M, CH_2Cl_2); IR (KBr): 3025 (CH), 2992 (CH), 1705 (C=O), 1695 (C=O) cm^{-1}; ^1H NMR: δ 1.86 (d, *J* = 7.2 Hz, 3H, $CHC\underline{H}_3$), 6.18 (q, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 6.40 (s, 1H, C=CH), 7.05-7.48 (m, 10H, $H_{benzene}$), 7.51-7.70 (m, 2H, $H_{phthalimide}$), 7.92 (m, 1H, $H_{7phthalimide}$), 8.74 (m, 1H, $H_{4phthalimide}$); ^{13}C NMR: δ 16.6 (CH_3), 48.3 (CH), 107.5 (C=), 123.4 (CH), 126.5 (2CH), 126.9 (CH), 127.3 (CH), 128.2 (2CH), 128.4 (2CH), 129.0 (2CH), 129.5 (C), 131.3 (CH), 132.7 (CH), 133.4 (CH), 134.1 (C), 138.8 (C), 139.7 (C), 144.8 (C), 167.7 (CO), 189.5 (CO); ms: (EI) *m/z* 353 (M^+). *Anal.* Calcd for $C_{24}H_{19}NO_2$: C, 81.56; H, 5.42; N, 3.96. Found: C, 81.44; H, 5.34; N, 3.89.

(*E*)-(1'*S*)-2,3-Dihydro-3-(2-oxo-2-pentylidene)-2-(1'-phenylethyl)-1*H*-isoindol-1-one (9). This compound was obtained as white solid as a *S* isomer in 89% yield (0.63 g); mp 73°C; $[\alpha]_D$ = +42.64±0.05 (c 0.16 M, CH_2Cl_2); IR (KBr): 3035 (CH), 2989 (CH), 1710 (C=O), 1698 (C=O) cm^{-1}; ^1H NMR: δ 0.86 (t, 3H, $C\underline{H}_3CH_2CH_2$), 1.42 (m, 2H, $CH_3C\underline{H}_2CH_2$), 1.84 (d, *J* = 7.2 Hz, 3H, $CHC\underline{H}_3$), 2.28 (m, 2H, $CH_3CH_2C\underline{H}_2$), 5.83 (s, 1H, C=CH), 6.04 (q, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 7.25-7.46 (m, 5H, $H_{benzene}$), 7.51-

7.72 (m, 2H, $H_{phthalimide}$), 7.92 (m, 1H, $H_{phthalimide}$), 8.92 (m, 1H, $H_{4phthalimide}$); ^{13}C NMR: δ 13.6 (CH_3), 16.6 (CH_3), 18.0 (CH_2), 47.1 (CH_2), 48.5 (CH), 108.8 (C=), 123.2 (CH), 126.2 (2CH), 127.3 (2CH), 128.7 (2CH), 129.4 (C), 131.3 (CH), 133.4 (CH), 134.0 (C), 139.5 (C), 144.4 (C), 167.9 (CO), 199.2 (CO); ms: (EI) *m/z* 319 (M^+). *Anal.* Calcd for $C_{21}H_{21}NO_2$: C, 78.97; H, 6.62; N, 4.38. Found: C, 78.89; H, 6.53; N, 4.26.

General procedure for Grignard addition onto enamidone (8). To a well stirred and cold solution of the enamidone (**8**) (10 mmol) under dry nitrogen atmosphere in anhydrous CH_2Cl_2 (20 mL) was added slowly and dropwise a 0.5 M solution of methyl(or ethyl)magnesium iodide (12 mmol) freshly prepared according to the classical procedure in dry ether over a period of 15 min. After 1 h of reaction at 0-5°C, the reaction was allowed to stir for an additional 2 h at rt. After hydrolysis under stirring with water (30 mL) then with 0.5 M NH_4Cl solution (40 mL), the solution was passed through celite. After separation, the organic layer was washed with brine, dried over magnesium sulfate and concentrated under reduced pressure. Recrystallization of the reaction residue from a mixture of ether-hexane gave pure enamidols (**2a**) and (**2b**) in good yields.

(*E*)-(1'*S*)-2,3-Dihydro-3-(2-hydroxy-2-phenylpropylidene)-2-(1'-phenylethyl)-1*H*-isoindol-1-one (2a). This compound was obtained as yellow needles as a single diastereomer in 86% yield (3.18 g); mp 186°C; $[\alpha]_D$ = -14.05°±0.05 (c 0.07 M in CH_2Cl_2); IR (KBr): 3447 (br-OH), 3069 (C-H), 2986-2926 (C-H), 1673 (CO), 1629 (C=C) cm^{-1}; ^1H NMR: δ 1.58 (s, 3H, CH_3), 1.88 (d, *J* = 7.2 Hz, 3H, $CHC\underline{H}_3$), 2.32 (s, 1H, OH), 5.72 (s, 1H, C=CH), 6.13 (q, *J* = 7.2 Hz, 1H, $C\underline{H}CH_3$), 7.03-7.74 (m, 12H, $H_{benzene}$), 7.79 (dd, *J* = 6.5 and 2.2 Hz, 1H, $H_{7phthalimide}$), 7.93 (dd, *J* = 8.0 and 2.0 Hz, 1H, $H_{4phthalimide}$); ^{13}C NMR: δ 16.8 (CH_3), 35.9 (CH_3), 48 (CH), 73.2 (C), 121.2 (CH), 122.8 (CH), 124.9 (2CH), 126.4 (2CH), 126.7 (CH), 127.1 (2CH), 127.9 (2CH), 128.6 (2CH), 128.9 (CH), 129.9 (C), 131.6 (CH), 133.6 (C), 135.7 (C), 140.5 (C), 147.1 (C), 166.9 (CO); ms: (EI) *m/z* 369 (M^+). *Anal.* Calcd for $C_{25}H_{23}NO_2$: C, 81.27; H, 6.27; N, 3.79. Found: C, 81.13; H, 6.11; N, 3.62.

(*E*)-(1'*S*)-2,3-Dihydro-3-(2-hydroxy-2-phenylbutylidene)-2-(1'-phenylethyl)-1*H*-isoindol-1-one (2b). This compound was obtained as a white-yellow solid a single diastereomer in 89% yield (3.41 g); mp 153°C; $[\alpha]_D$ = -3.43°±0.05 (c 0.07 M in CH_2Cl_2); IR (KBr): 3385 (br-OH), 3059 (CH), 2952 (CH), 1690 (C=O), 1636 (C=C) cm^{-1}; ^1H NMR: δ 0.64 (t, *J* = 7.2 Hz, 3H, $C\underline{H}_3CH_2$), 1.71 (q, *J* = 7.2 Hz, 2H, $CH_3C\underline{H}_2$), 1.89 (d, *J* = 7.5 Hz, 3H, $CHC\underline{H}_3$), 2.15 (s, 1H, OH), 5.67 (s, 1H, C=CH), 6.17 (q, *J* = 7.5 Hz, 1H, $C\underline{H}CH_3$), 6.95-7.45 (m, 12 H, $H_{benzene}$), 7.78 (dd, *J* = 6.7 and 1.3 Hz, 1H, $H_{7phthalimide}$), 7.87 (dd, *J* = 7.8 and 1.1 Hz, 1H, $H_{4phthalimide}$); ^{13}C NMR: δ 8.11 (CH_3), 16.9 (CH_3), 40.9 (CH_2), 47.9 (CH), 75.7 (C), 120.2 (CH), 122.7 (CH), 125.6 (2CH), 126.4 (CH), 126.6 (2CH), 126.9 (CH), 127 (CH), 127.1 (CH), 127.5 (C), 127.7 (2CH), 128.6 (2CH), 129.8 (C), 131.5 (CH), 133.6 (C), 140.6 (C), 145.3 (C), 166.9 (CO); ms: (EI) *m/z* 383 (M^+). *Anal.* Calcd for $C_{26}H_{25}NO_2$: C, 81.83; H, 6.57; N, 3.65. Found: C, 81.61; H, 6.44; N, 3.48.

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Received, 12th July, 1999