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Synthesis of 4-Aryl-2-benzazepine-1,5-diones by Photocyclization of *N*-(2-Arylethyl)phthalimides#

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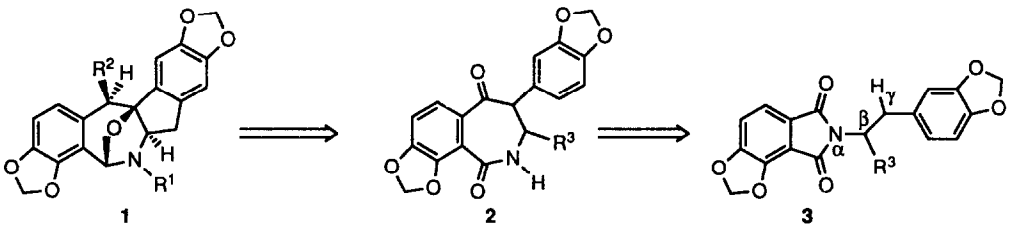
Abstract: The photocyclization of *N*-(2-arylethyl)phthalimides to 4-aryl-2-benzazepine-1,5-diones is described. We found that the presence of electron donating substituents on the aryl ring (as in **10a** and **b**) is necessary for the cyclization process to occur. The procedure also allowed synthesis of 2-benzazepinediones with a carboxylate group at C_3 (**11c** and **d**) which were obtained as 1:1 mixture of diastereoisomers. The results of irradiating phathalimides **12**, which bear an oxygenated substituent at the benzylic position, depended on the nature of the substituent. Attempts to photocyclize *N*-(indan-2-yl)phthalimides **16** and the electron-rich phthalimide **23** failed.

Introduction

Our interest in the total synthesis of ribasine alkaloids (**1**)[1] led us to investigate [2] the photocyclization of phthalimides **3** as a means of obtaining the 4-aryl-2-benzazepine skeleton (**2**) that it is latent in these alkaloids (Scheme 1).



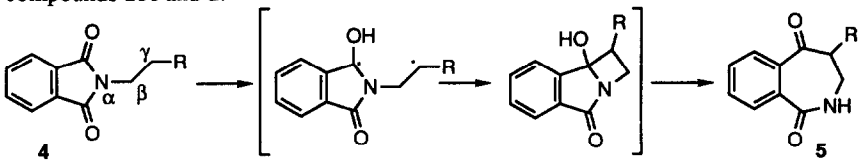
1 **2** **3**

a, R^1= Me, R^2= H Ribasine
b, R^1= R^2= H Norribasine
c, R^1= Me, R^2= OH Ribasidine

Scheme 1

#This work is part of the PhD Thesis by M. R.Paleo, University of Santiago, **1992**

It is known that simple *N*-alkylphthalimides (**4a**) undergo photocyclization in low yield to 2-benzazepinediones (**5**), γ-hydrogen abstraction to a biradical intermediate being followed by cyclization to a highly strained azetidinol which opens in a retrotransannular manner to afford the 2-benzazepinedione system (Scheme 2).[3,4] The corresponding reaction for *N*-(2-arylethyl)phthalimides (**4b**) remains largely unexplored. Until recently, the only reference in the literature has been a brief mention of the formation of a complex mixture of minor products upon irradiation of the simplest member of the class, *N*-(2-phenylethyl)phthalimide (**4**, R= Ph), [5] though while this manuscript was in preparation we became aware of work by Griesbeck and coworkers [6] on the photochemistry of N-phthaloyl derivatives of aromatic aminoacids which included examples closely related to our compounds **10c** and **d**.



4
a, R= Alkyl
b, R= Ar

5

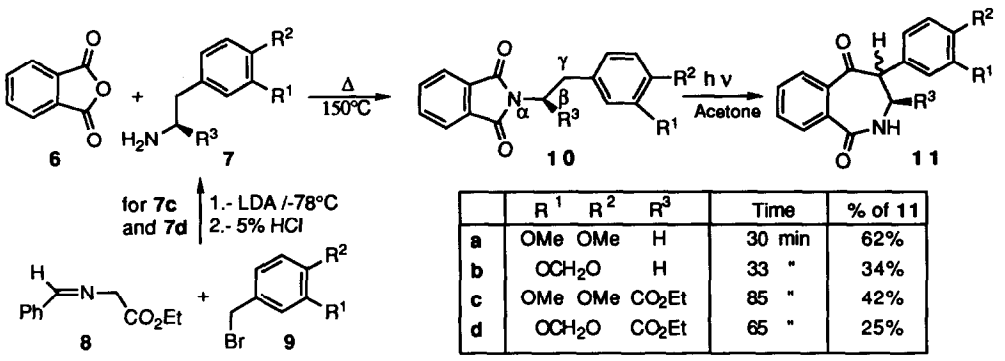
Scheme 2

Results and Discussion

Phthalimides **10a** and **b** were prepared by fusing a mixture of phthalic anhydride (**6**) and the corresponding commercially available 2-arylethylamine **7a** or **b** at 150°C (Scheme 3). Photolysis of **10a** in selected solvents was carried out at room temperature. With methylene chloride as solvent only a 14% yield of **11a** was obtained after 6h of irradiation, while irradiation in acetonitrile for 20h did not produce any 2-benzazepinedione. Best results were obtained with acetone, which led to a 62% yield of **11a** after 30 min, at which time some starting material was still present. Irradiation for longer periods of time was counterproductive the yield dropping to a 30% after 1.5h as a consequence of photochemical decomposition of the product. Under the same conditions the methylenedioxy-substituted derivative **10b** gave, after brief irradiation, a 34% isolated yield of **11b** (Scheme 3). These results contrast with the failure to cyclize of the unsubstituted phthalimide **10** (R^1=R^2=R^3=H)[5] showing the strong influence of the aryl substituents on the photocyclization. Formation of the 2-benzazepinedione is thus favoured by the presence of electron-donating substituents on the aryl ring, probably due to a lowering of its oxidation potential favouring electron transfer from the aryl moiety to the excited phthalimide, which is thought to be the fundamental step in the photochemistry of these systems.[6]

In order to apply this cyclization to the synthesis of ribasine alkaloids (**1**), we wished to prepare a 2-benzazepinedione **11** with a 3-substituent R^3 suitable for construction of the indane ring (Scheme 3). To this end we studied the photocyclization of phthalimides **10c** and **d**, which have a carboxylate group on the β-position.[7] Both compounds were prepared by fusing a mixture of phthalic anhydride (**6**) and the corresponding ethyl 2-amino-3-aryl propanoate (**7c** or **d**), which was prepared by condensing the lithium enolate derived from ethyl benzylidene glycinate **8** with the corresponding benzyl bromide (**9a** or **b**), followed by hydrolysis of the imine with 5% HCl.[8]

Phthalimide **10c** was irradiated in acetone for 85 min, giving approximate equal amounts of two 2-benzazepinediones which were identified, after PTLC purification, as the *cis* (J_{H3-H4}= 3 Hz) and *trans* (J_{H3-H4}= 10.4 Hz)[6] diastereoisomers of **11c** (42% combined yield). This result showed that the carboxylate did not interfere with cyclization.[7,9] A similar result was obtained with the methylenedioxy derivative **10d** which led to 2-benzazepinedione **11d** (again as a 1:1 mixture of diastereoisomers), although in lower yield.

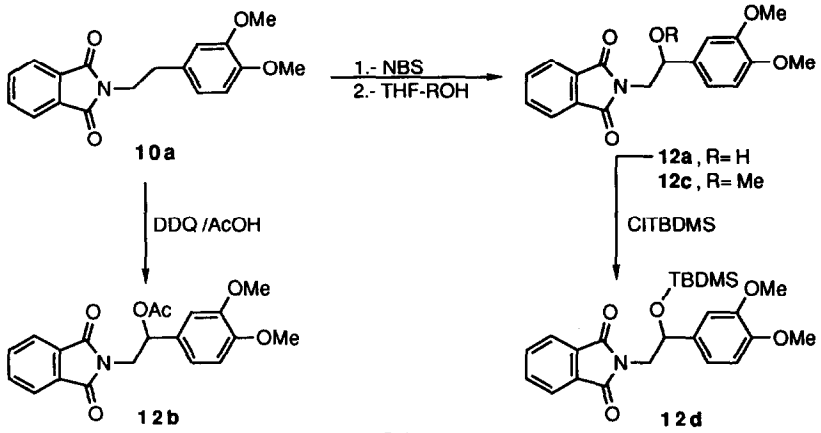


	R¹	R²	R³	Time	% of 11
a	OMe	OMe	H	30 min	62%
b	OCH_2O		H	33 "	34%
c	OMe	OMe	CO_2Et	85 "	42%
d	OCH_2O		CO_2Et	65 "	25%

Scheme 3

Next we investigated the effect of including an oxygenated substituent at the benzylic position (where the hydrogen abstraction takes place), since 2-benzazepinediones oxygenated at C_4 were required to form the target ribasine alkaloids. The hydroxylated phthalimide **12a** was prepared from **10a** by benzylic bromination with NBS followed by solvolysis with a mixture of THF and H_2O (Scheme 4). Irradiation of **12a** in acetone for 50 min gave a complex mixture from which the oxidized phthalimide **14** was isolated in low yield (Scheme 5). This result indicated the need to protect the hydroxyl group, so phthalimides **12b-d** were synthesized.

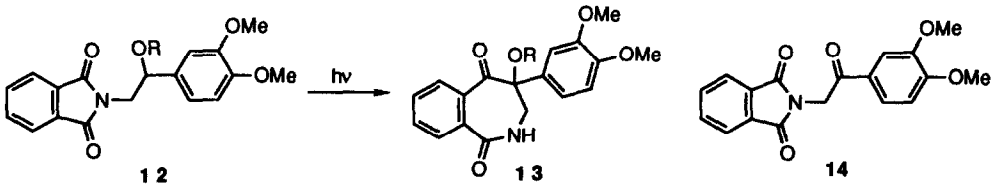
The acetate **12b** was prepared by benzylic oxidation of **10a** with DDQ in acetic acid[10] (90% yield). The γ-methoxy derivative **12c** was obtained similarly to **12a**, by treating the intermediate bromide with THF/MeOH (85% yield). Protection of the hydroxyl group of **12a** with ClTBDMS and imidazole in DMF gave **12d** (93% yield).



Scheme 4

Irradiation of **12b** gave a mixture of products from which the deoxygenated benzazepinedione **11a** was isolated, showing that an acetate group is unstable under the reaction conditions employed. However, irradiation of **12c** and **d** for a short period (15 min) afforded the desired 2-benzazepinediones **13c** and **d** in 27 and 30% yield respectively (Scheme 5).

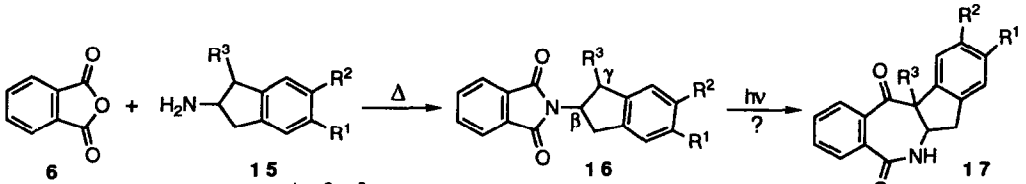
Encouraged by these results, we attempted direct photosynthesis of benzazepinediones **17** (which already incorporate the indane ring of the ribasine alkaloids) from *N*-(indan-2-yl)phthalimides **16**, which can be viewed



		Yield of 13	
a	R= H	-	30% (**14**)
b	R= COMe	-	20% (**11a**)
c	R= Me	27%	
d	R= TBDMS	30%	

Scheme 5

as *N*-(2-arylethyl)phthalimides that are substituted at positions β and γ (Scheme 6). The phthalimides **16** were obtained by heating a mixture of phthalic anhydride and the corresponding 2-aminoindane (**15a-d**) in DMF.

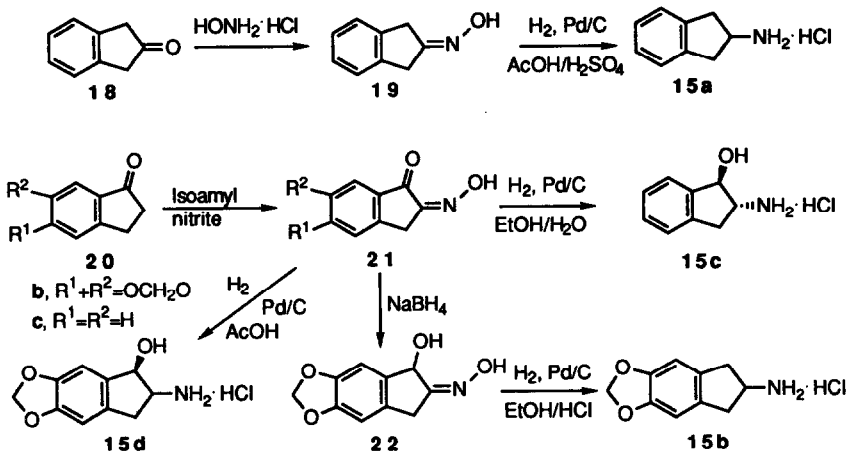


a, $R^1=R^2=R^3=H$
b, $R^1+R^2=OCH_2O$, $R^3=H$
c, $R^1=R^2=H$, $R^3=OH$
d, $R^1+R^2=OCH_2O$, $R^3=OH$
e, $R^1+R^2=OCH_2O$, $R^3=OTBDMS$

Scheme 6

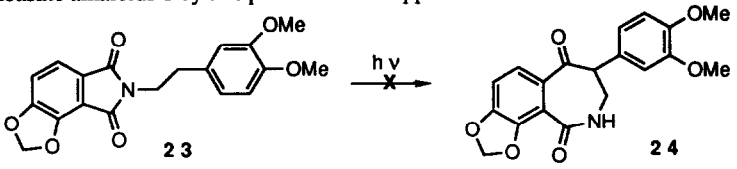
2-Aminoindane (**15a**) was obtained by catalytic hydrogenation of 2-indanone oxime (**19**) [11] (Scheme 7). 2-Amino-5,6-(methylenedioxy)indane (**15b**) was prepared from 5,6-(methylenedioxy)-1-indanone (**20b**) [12] by reaction with isoamyl nitrite to give **21b** [13] (90% yield) followed by $NaBH_4$ reduction to **22** (98%) and hydrogenolysis (90%).[14] Catalytic hydrogenation of **21c** in 3/1 EtOH/H_2O proceeded stereoselectively to give racemic *trans*-**15c** [15] (88%), while under the same conditions **21b** afforded the corresponding 2-amino-1-indanone hydrochloride.[16] By changing the solvent to glacial AcOH the reduction of **21b** gives a diastereomeric mixture of 2-amino-1-indanol **15d** in 81% yield.[17]

When irradiated in acetone solution, the unsubstituted phthalimide **16a** behaved like the acyclic analogue **10** ($R^1= R^2= R^3=$ H). Compound **16b,** which bears electron-donating substituents that favoured the photoprocess in the acyclic phthalimides **10a-d**, again gave a complex mixture, from which we were unable to isolate the desired indanobenzazepinedione **17b**. When a hydroxyl group was introduced in the γ position (**16c** and **16d**) the main product was the corresponding indanone, formed by oxidation of the alcohol. Protection as silyl ether **16e** led to almost instantaneous fragmentation into a complex mixture of products upon irradiation. The non-cyclization of the *N*-(indan-2-yl)phthalimides **16** was apparently a consequence of the higly strained nature of the cyclic azetidinol intermediate, because of which the biradical generated upon hydrogen abstraction underwent fragmentation rather than cyclization. These findings led us to relinquish direct incorporation of the ribasine indane ring and go back to acyclic 2-arylethylamine precursors with a β substituent that would allow construction of the indane ring after photocyclization (as in **10c** and **d**).



Scheme 7

Finally, we investigated the possibility of photocyclization of precursors which already bore the methylendioxy group in the phthalimide portion. To this end we prepared the phthalimide **23** (Scheme 8) and studied its photochemical behaviour. To our surprise this compound did not photocyclize, apparently due to inhibition of the γ-abstraction process by the electron-donating substituents on the phthalimide moiety. For this reason and given the complexity of introducing the 1,3-dioxolane ring at a later stage, we abandoned any further attempt to generate ribasine alkaloids **1** by this photochemical approach.



Scheme 8

Conclusion

We have developed a one step synthesis of 4-aryl-2-benzazepine-1,5-diones by photocyclization of *N*-(2-arylethyl)phthalimides with electron-donating substituents on the aryl ring. The procedure allows the preparation of 4-aryl-2-benzazepinediones with further substituents at positions 3 or 4 of the 2-benzazepine ring. Unfortunately, when the method was applied to *N*-(indan-2-yl)phthalimides none of the desired indane-2-benzazepinediones were isolated. Another limitation is that the photocyclization cannot be applied to compounds bearing electron-donating substituents on the phthalimide moiety.

EXPERIMENTAL SECTION

General Methods. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ at 250 and 62.83 MHz respectively, using TMS as internal reference. Mass spectra were recorded at an ionization voltage of 70 eV. Melting points are uncorrected. All air-sensitive reactions were carried out under dried, deoxygenated Ar using flame-dried glassware and magnetic stirring; reagents were added by syringe through septa. All solvents for air- or moisture-sensitive reactions were dried by standard procedures.[18] The concentration of commercial solutions of *n*-BuLi in hexane (Aldrich) was determined immediately prior to use by titration with diphenylacetic acid.[19]

Preparative irradiation was conducted in a Pyrex immersion well at room temperature using a 450 W water-cooled medium-pressure mercury lamp (Hanovia), with magnetic stirring, under Ar atmosphere

Treatment of ethyl glycinate hydrochloride with benzaldehyde in methylene chloride in the presence of triethylamine and anhydrous $MgSO_4$ afforded the Schiff base methyl ester derivative **8**.[8a]

Preparation of ethyl 2-amino-3-arylpropanoates 7c and 7d

In a flame-dried 25-mL round-bottomed flask equipped with a stirring bar, septum cap and Ar inlet, an LDA solution was prepared by treating diisopropylamine (0.4 mL, 2.9 mmol) in anhydrous THF (10 mL) with 1.6 M *n*-BuLi in hexane (1.8 mL, 2.9 mmol) at -78°C. The cooling bath was removed and the mixture was stirred for 15 min before cooling again to -78°C and addition of the Schiff base **8** (0.5 g, 2.7 mmol) in THF. The yellow solution obtained was stirred for 15 min, a solution of the bromide **9a** or **9b** (2.7 mmol) in anhydrous THF (5 mL) was added and the resulting mixture was stirred at -78°C for 1 h and then slowly brought to rt. An ice-cold saturated aqueous ammonium chloride/ether mixture (10 mL each) was added, the organic layer was removed and the aqueous layer was further extracted with ether (3x5 mL). The organic extract was dried with anhydrous Na_2SO_4 and the solvent was evaporated to leave an oil which was partially hydrolysed to the α-amino ester by treatment with 5% HCl (5 mL) for 2 h at rt. Ether was added and the aqueous layer was separated, cooled in an ice-water bath and treated with solid K_2CO_3 until basic and extracted with CH_2Cl_2 (3x10 mL). The combined organic extracts were dried with anhydrous Na_2SO_4 and evaporation of the solvent gave an oil which was used in the next step without further purification.

General procedure for preparation of *N*-arylethylphthalimides 10.

In a 25 mL flask a mixture of phthalic anhydride (**6**) (3 g, 20.27 mmol) and the corresponding 2-arylethylamine (**7a-d**) (20.27 mmol) was fused at 150°C for 90 min and then allowed to cool to rt. The product was purified by recrystallization.

***N*-[2-(3,4-Dimethoxyphenyl)ethyl]phthalimide (10a):**

5.43 g (86%); m. p. : 168-169°C (EtOH). IR ($CHCl_3$): 1720, 1755 cm^{-1}. UV (MeOH) λ_{max}: 220, 280 nm. ^1H NMR: δ 2.95 (m, 2H), 3.82 (s, 3H), 3.84 (s, 3H), 3.91 (m, 2H), 6.75 (s, 1H), 6.79 (s, 2H), 7.71 (m, 2H), 7.83 (m, 2H). ^{13}C NMR: δ 33.97, 39.24, 55.77, 55.82, 111.47, 112.13, 120.91, 123.17, 130.57, 132.13, 133.88, 147.86, 149.02, 168.20. MS m/z (%): 311 (M^+, 14), 164 (84), 151 (100), 107 (33), 77 (62). Anal. Calc. for $C_{18}H_{17}NO_4$: C, 69.44; H, 5.50; N, 4.50. Found: C, 69.03; H, 5.71; N, 4.52.

***N*-[2-(3,4-Methylenedioxyphenyl)ethyl]phthalimide (10b)**

5.09 g (90%); m.p.: 136-137°C (EtOH). IR ($CHCl_3$): 1710, 1770 cm^{-1}.UV (MeOH) λ_{max}: 218, 288 nm. ^1H NMR: δ 2.90 (t, 2H, J= 7.6 Hz), 3.87 (t, 2H, J= 7.6 Hz), 5.92 (s, 2H), 6.70 (m, 3H), 7.71 (m, 2H), 7.83 (m, 2H). ^{13}C NMR: δ 34.26, 39.41, 100.86, 108.31, 109.27, 121.81, 123.24, 131.81, 132.16, 133.91, 146.35, 147.81, 168.20. MS m/z (%): 295 (M^+, 7), 160 (23), 148 (64), 135 (48), 105 (22), 77 (100). Anal. Calc. for $C_{17}H_{13}NO_4$: C, 69.15; H, 4.44; N, 4.74. Found: C, 68.81; H, 4.24; N, 4.65.

***N*-[1-(Ethoxycarbonyl)-2-(3,4-dimethoxyphenyl)ethyl]phthalimide (10c).**

3.97 g (63%); foam. IR ($CHCl_3$): 1710, 1740, 1775 cm^{-1}. UV (CH_3OH) λ_{max}: 220, 282 nm. ^1H NMR: δ 1.25 (t, 3H, J= 7.1 Hz), 3.50 (m, 2H), 3.68 (s, 3H), 3.75 (s, 3H), 4.23 (q, 2H, J= 7.1 Hz), 5.12 (dd, 1H, J= 6.2, 10.5 Hz), 6.65 (m, 3H), 7.67 (m, 2H), 7.74 (m, 2H). ^{13}C NMR: δ 14.00, 34.11, 53.38, 55.65,

55.73, 61.87, 111.46, 112.08, 121.08, 123.38, 129.33, 131.74, 134.06, 147.94, 148.94, 167.50, 168.86. Anal. Calc. for $C_{21}H_{21}NO_6$: C, 65.79; H, 5.52; N, 3.65. Found: C, 65.41; H, 5.29; N, 3.79.

***N*-[1-(Ethoxycarbonyl)-2-(3,4-methylenedioxyphenyl)ethyl]phthalimide (10d).**

3.31 g (52%); m.p.: 74-75°C (MeOH). IR ($CHCl_3$): 1720, 1740, 1780 cm-1. UV (CH_3OH) λ_{max}: 218, 288 nm. ^1H NMR: δ 1.25 (t, 3H, J= 7.1 Hz), 3.47 (m, 2H), 4.25 (q, 2H, J= 7.1 Hz), 5.06 (dd, 1H, J= 6.0, 10.4 Hz), 5.85 (d, 1H, J= 1.3 Hz), 5.86 (d, 1H, J= 1.3 Hz), 6.59 (s, 2H), 6.67 (s, 1H), 7.70 (m, 2H), 7.79 (m, 2H). ^{13}C NMR: δ 13.99, 34.30, 53.60, 61.92, 100.82, 108.25, 109.19, 121.96, 123.47, 130.56, 131.70, 134.07, 146.43, 147.73, 167.54, 168.77. MS m/z (%): 367 (M^+, 6), 294 (4), 220 (96), 175 (54), 160 (22), 135 (100), 104 (73), 77 (66). Anal. Calc. for $C_{20}H_{17}NO_6$: C, 65.39; H, 4.66; N, 3.81. Found: C, 65.38; H, 4.88; N, 3.99.

***N*-[2-(Hydroxy)-2-(3,4-dimethoxyphenyl)ethyl] phthalimide (12a).**

A suspension of **10a** (2.0 g, 6.43 mmol) and NBS (1.14 g, 6.43 mmol) in anhydrous CCl_4 (60 mL) was heated at reflux and irradiated with a 60 W lamp until the NBS has been transformed into colourless succinimide suspended in the reaction mixture. The suspension was cooled, filtered and the residue was washed with CCl_4. The combined filtrate and washings were evaporated to dryness and the residue was dissolved in CH_2Cl_2 and washed with brine. The organic layer was separated, dried over anh. Na_2SO_4 and evaporated *in vacuo* to give a residue which was dissolved in THF/H_2O (12mL/8mL) and stirred overnight at rt. CH_2Cl_2 was added and the organic layer separated, dried and evaporated to dryness. The product was purified by column chromatography on silica gel, with CH_2Cl_2-MeOH (1%) as eluant, giving 1.5 g (71% yield) of a white solid; m.p.: 173-174°C (MeOH). IR ($CHCl_3$): 1710, 1770, 3460 cm-1. UV (MeOH) λ_{max}: 220, 280 nm. ^1H NMR: δ 3.87 (s, 3H), 3.88 (s, 3H), 3.93 (dd, 1H, J= 3.8, 14.2 Hz), 4.03 (dd, 1H, J= 8.4, 14.2 Hz), 5.04 (dd, 1H, J= 3.8, 8.4 Hz), 6.85 (d, 1H, J= 8.7 Hz), 6.98 (m, 2H), 7.73 (m, 2H), 7.85 (m, 2H). ^{13}C NMR: δ 45.62, 55.90, 72.22, 109.18, 111.32, 118.31, 123.44, 131.97, 133.80, 134.12, 148.95; 149.28, 168.76. MS m/z (%): 327 (M^+, 12), 180 (24), 167 (100), 160 (62), 139 (92), 124 (63), 104 (56), 77 (78). Anal. Calc. for $C_{18}H_{17}NO_5$: C, 66.05; H, 5.23; N, 4.28. Found: C, 66.16; H, 5.26; N, 4.15.

***N*-[2-(Acetoxy)-2-(3,4-dimethoxyphenyl)ethyl] phthalimide (12b).**

A solution of 1.0 mmol of DDQ (230 mg) in 5 mL of anhydrous acetic acid was slowly added at 25°C to a solution of **10a** (300 mg, 0.97 mmol) in 10 mL of anhydrous acetic acid and heated at 70°C overnight. 20 mL of CH_2Cl_2 were added and the organic layer was washed with water (3x10 mL), 10% aqueous NaOH (10 mL) and water (10 mL).The extract was dried (Na_2SO_4) and cocentrated to give an oil which solidified on cooling. Recrystallization from a CH_2Cl_2/Hexane solvent pair afforded 307 mg (90%), m.p.: 139-141°C. IR ($CHCl_3$): 1720, 1730, 1770 cm-1. UV (MeOH) λ_{max}: 220, 280 nm. ^1H NMR: δ 2.02 (s, 3H), 3.87 (s, 3H), 3.89 (s, 3H), 3.93 (m, 1H), 4.15 (dd, 1H, J= 9.0, 14.1 Hz), 6.09 (dd, 1H, J= 4.0, 9.0 Hz), 6.85 (d, 1H, J= 8.2 Hz), 7.01 (m, 2H), 7.73 (m, 2H), 7.84 (m, 2H). ^{13}C NMR: δ 20.77, 42.55, 55.74, 55.82, 72.88, 109.96, 111.22, 119.31, 123.23, 129.57, 131.81, 133.95, 149.11, 149.30, 167.80, 170.05. MS m/z (%) 369 (M^+, 4), 209 (13), 167 (100), 160 (48), 139 (35), 77 (40). Anal. Calc. for $C_{20}H_{19}NO_6$: C, 65.03; H, 5.18; N, 3.79. Found: C, 65.10; H, 5.38; N, 3.73.

***N*-[2-(Methoxy)-2-(3,4-dimethoxyphenyl)ethyl] phthalimide (12c).**

The same procedure as for **12a**, changing the treatment of the intermediate bromide with THF/H_2O for THF/MeOH afforded **12c** in 85% yield; m.p.: 122-123°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1715, 1775 cm-1. UV (MeOH) λ_{max}: 220, 280 nm. ^1H NMR: δ 3.11 (s, 3H), 3.68 (dd, 1H, J= 4.8, 13.8 Hz), 3.81 (s, 3H), 3.82 (s, 3H), 3.96 (dd, 1H, J= 8.9, 13.8 Hz), 4.48 (dd, 1H, J= 4.8, 8.9 Hz), 6.78 (d, 1H, J= 8.6 Hz), 6.86 (m, 2H), 7.65 (m, 2H), 7.77 (m, 2H). ^{13}C NMR: δ 43.87, 55.84, 55.93, 56.78, 80.40, 109.56, 111.08, 119.77, 123.30, 131.19, 132.12, 133.93, 149.11, 149.39, 168.26. MS m/z (%) 341 (M^+, 3), 181 (100), 166 (73), 160 (41), 151 (54), 77 (71). Anal. Calc. for $C_{19}H_{19}NO_5$: C, 66.85; H, 5.61; N, 4.10. Found: C, 66.76; H, 5.79; N, 4.11.

***N*-[2-(*t*-Butyldimethylsilyloxy)-2-(3,4-dimethoxyphenyl)ethyl] phthalimide (12d).**

In a flame-dried 25-mL round-bottomed flask equipped with a stirring bar, septum cap and Ar inlet, a solution of **12a** (0.5 g, 1.5 mmol) in anhydrous DMF (10 mL), ClTBDMS (277 mg, 1.8 mmol) and imidazole (260 mg, 3.8 mmol) were stirred under Ar for 24 h at 25°C, then poured over an ice/water mixture and extracted with CH_2Cl_2. The organic layer was separated, dried (Na_2SO_4) and the solvent evaporated. Crystallization from a CH_2Cl_2/Hexane solvent pair afforded 628 mg (93%), m.p.: 138-140°C. IR ($CHCl_3$): 1710, 1770 cm-1. UV (MeOH) λ_{max}: 218, 226, 280 nm. ^1H NMR: δ -0.22 (s, 3H), -0.20 (s, 3H), 0.75 (s, 9H), 3.68 (dd, 1H, J= 4.2, 13.6 Hz), 3.88 (s, 3H), 3.89 (s, 3H), 3.94 (dd, 1H, J= 9.2, 13.6 Hz), 5.03 (dd, 1H, J= 4.2, 9.2 Hz), 6.82 (d, 1H, J= 8.2 Hz), 6.96 (m, 2H), 7.73 (m, 2H), 7.86 (m, 2H). ^{13}C NMR: δ -5.43, -4.99, 17.77, 25.48, 46.24, 55.86, 71.82, 109.40, 110.99, 118.64, 123.19, 132.23, 133.94, 134.69, 148.72, 149.12, 168.29. MS m/z (%) 441 (M^+, 0,3), 384 (9), 281 (100), 204 (20), 160 (14), 77 (20), 73 (67). Anal. Calc. for $C_{24}H_{31}NO_5Si$: C, 65.28; H, 7.07; N, 3.17. Found: C, 65.01; H, 6.84; N, 2.93.

General procedure for irradiation of phthalimides 10 and 12.

A solution of the phthalimide (0.2 g) in acetone (200 mL) was irradiated, under Ar atmosphere, by using a Hanovia 450 W medium-pressure Hg lamp in a pyrex inmersion well mantained at room temperature. The solution was degassed by bubbling Ar through it for a period of 10 min prior to irradiation. Solvent was removed *in vacuo* and the residue chromatographied by preparative TLC on silica gel with CH_2Cl_2-MeOH (3%) as eluant.

2,3,4,5-Tetrahydro-4-(3,4-dimethoxyphenyl)-1*H*-2-benzazepine-1,5-dione (11a).

Irradiation period: 30 min. Yield: 62%, m.p.: 166-167°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1670, 1690 cm-1. UV (MeOH) λ_{max}: 208, 280 nm. ^1H NMR: δ 3.68 (m, 1H), 3.82 (m, 1H), 3.83 (s, 3H), 3.84 (s, 3H), 4.12 (dd, 1H, J= 4.1, 10.2 Hz), 6.79 (m, 3H), 7.56 (m, 1H), 7.6-7.8 (m, 2H), 7.94 (m, 2H); ($CDCl_3$-D_2O): δ 3.66 (dd, 1H, J= 4.1, 14.9 Hz), 3.82 (dd, 1H, J= 14.9, 10.2 Hz), 3.83 (s, 3H), 3.84 (s, 3H), 4.14 (dd, 1H, J= 4.1, 10.2 Hz), 6.79 (m, 3H), 7.56 (m, 1H), 7.6-7.8 (m, 2H), 7.94 (m, 1H). ^{13}C NMR δ 44.25, 55.94, 61.32, 111.78, 120.08, 128.53, 129.79, 130.01, 131.52, 132.33, 132.60, 137.30, 148.95, 149.51, 170.86, 204.32. Anal. Calc. for $C_{18}H_{17}NO_4$: C, 69.44; H, 5.50; N, 4.50. Found: C, 69.42; H, 5.86; N, 4.60.

2,3,4,5-Tetrahydro-4-(3,4-Methylenedioxyphenyl)-1*H*-2-benzazepine-1,5-dione (11b).

Irradiation period: 33 min. Yield: 34%, m.p. 153-154°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1660, 1690, 3020 cm-1. UV (MeOH) λ_{max}: 208, 238, 286 nm. ^1H NMR: δ 3.64 (m, 1H), 3.78 (m, 1H), 4.07 (dd, 1H, J=

4.1, 10.0 Hz), 5.93 (s, 2H), 6.61 (m, 2H), 6.75 (d, 1H, J= 8.4 Hz), 7.56 (d, 1H, J= 7.6 Hz), 7.67 (m, 2H), 7.95 (d, 1H, J= 7.6 Hz); ($CDCl_3$-D_2O): δ3.62 (dd, 1H, J= 4.1, 14.9 Hz), 3.78 (dd, 1H, J= 10.0, 14.9 Hz), 4.07 (dd, 1H, J= 4.1, 10.0 Hz), 5.93 (s, 2H), 6.61 (m, 2H), 6.75 (d, 1H, J= 8.4 Hz), 7.56 (d, 1H, J= 7.6 Hz), 7.67 (m, 2H), 7.95 (d, 1H, J= 7.6 Hz). ^{13}C NMR: δ 44.13, 61.37, 101.24, 108.41, 108.81, 121.47, 128.61, 129.95, 130.97, 131.45, 132.38, 132.64, 137.15, 147.36, 148.21, 170.90, 204.23. Anal. Calc. for $C_{17}H_{13}NO_4$: C, 69.15; H, 4.44; N, 4.74. Found: C, 68.81; H, 4.37; N, 4.59.

2,3,4,5-Tetrahydro-3-ethoxycarbonyl-4-(3,4-dimethoxyphenyl)-1*H*-2-benzazepine-1,5-dione (11c).

Irradiation time: 85 min. Yield: 42% (1/1 mixture of *cis/trans* isomers).

cis-**11c**: less polar product, m.p.: 68-70°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1670, 1740 cm-1. UV (MeOH) λ_{max}: 214, 236, 282 nm. ^1H NMR: δ 1.25 (t, 3H, J= 7.3 Hz), 3.81 (s, 3H), 3.86 (s, 3H), 4.16 (m, 2H), 4.40 (d, 1H, J= 3.0 Hz), 5.05 (dd, 1H, J= 5.6, 3.0 Hz), 6.6-6.9 (m, 4H), 7.6-7.8 (m, 3H), 7.9-8.1 (m, 1H). ^{13}C NMR: δ 14.05, 55.85, 55.94, 56.03, 62.59, 63.00, 111.50, 113.00, 122.52, 126.71, 129.00, 130.24, 132.45, 133.13, 136.55, 149.19, 149.35, 167.83, 201.81. Anal. Calc. for $C_{21}H_{21}NO_6$: C, 65.79; H, 5.52; N, 3.65. Found: C, 65.41; H, 5.82; N, 3.45.

trans-**11c**: more polar product, m.p.: 59-61°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1670, 1740 cm-1. UV (MeOH) λ_{max}: 214, 236, 282 nm. ^1H NMR: δ 0.89 (t, 3H, J= 7.1 Hz), 3.81 (s, 3H), 3.86 (s, 3H), 3.94 (q, 2H, J= 7.1 Hz), 4.27 (d, 1H, J= 10.4 Hz), 4.68 (dd, 1H, J= 5.9, 10.4 Hz), 6.6-6.8 (m, 4H), 7.4-8.0 (m, 4H). ^{13}C NMR: δ 13.58, 55.97, 57.47, 62.19, 65.10, 77.19, 111.75, 120.63, 123.65, 128.63, 129.89, 131.56, 132.56, 134.34, 137.23, 149.49, 168.72, 169.06, 201.80. Anal. Calcd. for $C_{21}H_{21}NO_6$: C, 65.79; H, 5.52; N, 3.65. Found: C, 65.75; H, 5.64; N, 3.83.

2,3,4,5-Tetrahydro-3-ethoxycarbonyl-4-(3,4-methylenedioxyphenyl)-1*H*-2-benzazepine-1,5-dione (11d).

Irradiation time: 65 min. Yield: 25% (1/1 mixture of *cis/trans* isomers).

cis-**11d**: less polar product, m.p.: 74-75°C (CH_2Cl_2/Hexane). IR ($CHCl_3$): 1670, 1740 cm-1. UV (MeOH) λ_{max}: 212, 236, 288 nm. ^1H NMR: δ 1.26 (t, 3H, J= 7.1 Hz), 4.17 (m, 2H), 4.37 (d, 1H, J= 3.1 Hz), 5.05 (dd, 1H, J= 5.5, 3.1 Hz), 5.96 (s, 2H), 6.61 (m, 2H), 6.78 (m, 2H), 7.6-7.8 (m, 3H), 7.99 (d, 1H, J= 6.3 Hz). ^{13}C NMR: δ 14.04, 56.00, 62.65, 63.08, 101.31, 108.57, 110.07, 123.62, 127.72, 128.95, 130.24, 132.02, 132.47, 133.07, 136.63, 147.88, 148.07, 167.80, 201.68. Anal. Calc. for $C_{20}H_{17}NO_6$: C, 65.39; H, 4.66; N, 3.81. Found: C, 65.18; H, 4.91; N, 3.92.

trans-**11d**: more polar product, m.p.: 70-71°C (CH_2Cl_2/Hexane). IR ($CHCl_3$): 1670, 1740 cm-1. UV (MeOH) λ_{max}: 212, 236, 288 nm. ^1H NMR: δ 0.93 (t, 3H, J= 7.1 Hz), 3.96 (m, 2H), 4.22 (d, 1H, J= 10.5 Hz), 4.64 (dd, 1H, J= 5.9, 10.5 Hz), 5.95 (s, 2H), 6.58 (m, 2H), 6.75 (m, 2H), 7.49 (m, 1H), 7.69 (m, 2H), 7.95 (m, 1H). ^{13}C NMR: δ 13.56, 57.48, 62.21, 65.13, 101.32, 108.41, 108.79, 122.04, 128.73, 129.71, 129.86, 131.48, 132.65, 147.70, 148.22, 168.64, 168.91, 201.93. Anal. Calc. for $C_{20}H_{17}NO_6$: C, 65.39; H, 4.66; N, 3.81. Found: C, 65.26; H, 4.85; N, 3.89.

***N*-(3,4-Dimethoxyacetophenyl)phthalimide (14)**

Irradiation period: 50 min. Yield: 30%, m.p.: 194-195°C (CH_2Cl_2/Hexane). IR ($CHCl_3$): 1690, 1720, 1775 cm-1. UV (MeOH) λ_{max}: 220, 278, 302 nm. ^1H NMR: δ 3.92 (s, 3H), 3.97 (s, 3H), 5.10 (s, 2H), 6.94 (d, 1H, J= 8.2 Hz), 7.52 (s, 1H), 7.65 (d, 1H, J= 8.2 Hz), 7.76 (m, 2H), 7.88 (m, 2H). ^{13}C NMR: δ 43.84,

56.03, 56.13, 110.38, 110.48, 122.78, 123.57, 127.81, 132.41, 134.13, 149.48, 154.23, 168.05, 189.63. Anal. Calc. for $C_{18}H_{15}NO_5$: C, 66.46; H, 4.65; N, 4.31. Found: C, 66.80; H, 4.77; N, 4.51.

2,3,4,5-Tetrahydro-4-methoxy-4-(3,4-dimethoxyphenyl)-1*H*-2-benzazepine-1,5-dione (13c).

Irradiation period: 15 min. Yield: 27%; m.p.: 148-150°C (CH_2Cl_2/Hexane). IR ($CHCl_3$): 1660, 1670 cm-1. UV (MeOH) λ_{max}: 216, 232, 278 nm. ^1H NMR: δ 3.33 (s, 3H), 3.70 (dd, 1H, J= 14.9, 6.5 Hz), 3.83 (dd, 1H, J= 14.9, 6.5 Hz), 3.88 (s, 6H), 6.39 (broad, 1H, NH), 6.80 (m, 2H), 6.90 (d, 1H, J= 1.9 Hz), 7.53 (m, 1H), 7.68 (m, 2H), 7.90 (m, 1H). ^{13}C NMR: δ 49.28, 53.15, 55.93, 56.08, 87.55, 110.57, 111.15, 120.65, 128.62, 129.62, 130.03, 132.18, 132.32, 132.56, 137.47, 149.51, 149.64, 170.27, 201.58. Anal. Calc. for $C_{19}H_{19}NO_5$: C, 66.85; H, 5.61; N, 4.10. Found: C, 66.97; H, 5.27; N, 3.77.

2,3,4,5-Tetrahydro-4-*t*-butyldimethylsilyloxy-4-(3,4-dimethoxyphenyl)-1*H*-2-benzazepine-1,5-dione (13d).

Irradiation period: 15 min. Yield: 30%; m.p.: 153°C (Ether/Hexane). IR ($CHCl_3$): 1660, 1670 cm-1. UV (MeOH) λ_{max}: 220, 280 nm. ^1H NMR: δ 0.03 (s, 3H), 0.21 (s, 3H), 1.00 (s, 9H), 3.46 (dd, 1H, J= 6.9, 15.0 Hz), 3.84 (s, 6H), 3.91 (dd, 1H, J= 5.9, 15.0 Hz), 6.57 (broad, 1H, NH), 6.64-6.77 (m, 2H), 7.03 (m, 1H), 7.5-7.98 (m, 4H). ^{13}C NMR: δ -3.22, -3.16, 18.93, 26.03, 51.89, 55.85, 55.90, 86.71, 109.22, 110.96, 117.93, 128.78, 130.10, 131.26, 132.21, 132.67, 133.15, 137.63, 149.13, 149.23, 170.24, 204.68; Anal. Calc. for $C_{24}H_{31}NO_5Si$: C, 65.28; H, 7.07; N, 3.17. Found: C, 65.42; H, 7.15; N, 3.27.

General procedure for preparation of 16a-d.

In a 25-mL flame-dried round-bottomed flask a suspension of NaH (oil dispersion 80%, 90 mg, 3.0 mmol, washed with THF 2 x 1 mL) in anhydrous DMF was prepared and cooled to 0°C. A solution of the corresponding hydrochloride **15** (2.7 mmol) in DMF (3 mL) was added, stirred for 15 min and allowed to warm to rt. Phthalic anhydride (400 mg, 2.7 mmol) was added and the resulting suspension was heated at 120°C for between 5 and 12h depending on the aminoindane (TLC analysis). After cooling to rt a 1/1 H_2O/CH_2Cl_2 mixture (20 mL) was added. The organic phase was washed with water (5 x 5 mL), dried over Na_2SO_4, and evaporated to dryness. The residue was recrystallyzed from MeOH.

***N*-(Indan-2-yl)phthalimide (16a).**

Yield: 78%; m.p.: 194-195°C (Cl_2CH_2-Hexane). IR ($CHCl_3$): 1715, 1775 cm-1. UV (MeOH) λ_{max}: 220, 242, 290 nm. ^1H NMR: δ 3.18 (dd, 2H, J= 8.8, 15.3 Hz), 3.62 (dd, 2H,J= 9.3, 15.3 Hz), 5.15 (m, 1H), 7.20 (m, 4H), 7.73 (m, 2H), 7.84 (m, 2H). ^{13}C RMN: δ 36.08, 50.08, 123.23, 124.44, 126.74, 132.19, 133.99, 140.96, 168.41. MS m/z 263 (M^+, 13), 148 (73), 130 (70), 116 (100), 104 (68), 89 (52), 77 (76), 76 (87). Anal. Calcd. for $C_{17}H_{13}NO_2$: C, 77.55; H, 4.98; N, 5.32; Found: C, 77.35; H, 5.16; N, 5.40.

***N*-[5,6-(Methylenedioxy)indan-2-yl]phthalimide (16b).**

Yield: 92%; m.p.: 195-196°C (EtOH). IR ($CHCl_3$): 1710, 1770 cm-1. UV (CH_3OH) λ_{max}: 220, 296 nm; ^1H RMN δ 3.07 (dd, 2H, J= 9.0, 14.6 Hz), 3.50 (dd, 2H, J= 9.0, 14.6 Hz), 5.15 (q, 1H, J= 9.0 Hz), 5.92 (d, 1H, J= 1.4 Hz), 5,93 (d, 1H, J= 1,4 Hz), 6.69 (s, 2H), 7.72 (m, 2H), 7.83 (m, 2H); ^{13}C RMN δ 35.98, 50.34, 100.85, 105.06, 123.23, 132.15, 133.40, 133.99, 146.80, 168.37; m/z 307 (M^+, 4), 160 (100), 130

(62), 102 (97), 76 (67); Anal. Calcd. for $C_{18}H_{13}NO_4$: C, 70.35; H, 4.26; N, 4.56. Found: C, 70.05; H, 4.31; N, 4.60.

***trans*-N-(1-Hydroxyindan-2-yl)phthalimide(16c).**

Yield: 70%; m.p.: 217-219°C (MeOH). IR ($CHCl_3$): 1710, 1770 cm-1; UV (MeOH) λ_{max}: 210, 220, 292 nm. ^1H NMR: δ 3.18 (dd, 1H, J=8.6, 15.1 Hz), 3.53 (dd, 1H, J=10.0, 15.1 Hz), 4.76 (m, 1H), 5.85 (d, 1H, J=7.4 Hz), 7.27 (m, 3H), 7.43 (m, 1H), 7.75 (m, 2H), 7.86 (m, 2H). ^{13}C NMR: δ 32.71, 60.42, 77.07, 123.39, 123.95, 124.82, 127.39, 128.54, 132.10, 134.15, 138.80, 142.45, 168.70. Anal. Calcd. for $C_{17}H_{13}NO_3$: C, 73.11; H, 4.69; N, 5.01. Found: C, 72.80; H, 4.65; N, 4.82.

N-[1-Hydroxy-5,6-(methylenedioxy)indan-2-yl]phthalimide (16d).

cis-**16d:** less polar product. Yield: 27% , m.p.: 203-204°C (EtOH). IR ($CHCl_3$): 1710, 1770, 3400-3520 cm-1; UV (MeOH) λ_{max}: 218, 296 nm. ^1H NMR: δ 3.05 (dd, 1H, J= 8.8, 15.8 Hz), 3.91 (dd, 1H, J= 8.0, 15.8 Hz), 5.09 (m, 2H), 5.97 (d, 1H, J= 1.3 Hz), 5.98 (d, 1H, J= 1.3 Hz), 6.72 (s, 1H), 6.93 (s, 1H), 7.74 (m, 2H), 7.85 (m, 2H). ^{13}C NMR: δ 32.47, 53.23, 76.17, 101.28, 104.98, 105.64, 123.46, 132.02, 133.66, 134.20, 135.54, 147.44, 149.13, 169.59.

trans-**16d:** more polar product. Yield: 51%, m.p.: 203-204°C (EtOH). IR ($CHCl_3$): 1710, 1770, 3400-3520 cm-1. UV (MeOH) λ_{max}: 220, 296 nm. ^1H NMR: δ 3.08 (dd, 1H, J= 8.7, 14.9 Hz), 3.38 (dd, 1H, J= 9.7, 14.9 Hz), 4.73 (m, 1H), 5.69 (d, 1H, J= 7.6 Hz), 5.95 (d, 1H, J= 1.1 Hz), 5.97 (d, 1H, J= 1.1 Hz), 6.67 (s, 1H), 6.87 (s, 1H), 7.73 (m, 2H), 7.84 (m, 2H). ^{13}C NMR: δ 32.66, 60.54, 77.16, 101.13, 104.52, 105.17, 123.39, 131.99, 132.07, 134.15, 135.45, 147.43, 148.34, 168.64.

***trans*-N-[1-*t*-Butyldimethylsilyloxy-5,6-(methylenedioxy)indan-2-yl]phthalimide (16e).**

Obtained from *trans*-**16d** by a procedure analogous to the preparation of **12d**. Yield: 95%, m.p.: 67-69°C (CH_2Cl_2-Hexane). IR ($CHCl_3$): 1710, 1770 cm-1. UV (MeOH) λ_{max}: 210, 222, 296 nm. ^1H NMR: δ -0.19 (s, 3H), 0.08 (s, 3H), 0.85 (s, 9H), 2.99 (dd, 1H, J= 8.8, 14.7 Hz), 3.35 (dd, 1H, J= 9.8, 14.7 Hz), 4.84 (m, 1H), 5.79 (d, 1H, J= 7.5 Hz), 5.93 (d, 1H, J= 0.4 Hz), 5.94 (d, 1H, J= 0.4 Hz), 6.64 (s, 1H), 6.73 (s, 1H), 7.75 (m, 2H), 7.86 (m, 2H). ^{13}C NMR: δ -4.75, -4.57, 17.74, 25.59, 32.39, 59.87, 76.59, 101.01, 104.57, 105.13, 123.34, 131.53, 131.99, 134.15, 136.36, 147.21, 147.91, 168.47. Anal. Calcd.for $C_{24}H_{27}NO_5Si$: C, 65.88; H, 6.22; N, 3.20. Found: C, 65.82; H, 6.17; N, 3.16.

***N*-[2-(3,4-dimethoxyphenyl)ethyl]-4,5-(methylenedioxy)phthalimide (23).**

In a 25 mL flask 4,5-methylenedioxyphthalic anhydride (0.5 g, 2.6 mmol) and 3,4-dimethoxyphenethylamine (**7a**) (0.47 g, 2.6 mmol) were fused at 160°C for 80 min and then allowed to cool to rt. The product was purified by recrystallization from EtOH (85% yield), m.p.: 183°C. IR ($CHCl_3$): 1700, 1760 cm^{-1}. ^1H RMN δ 2.92 (m, 2H), 3.84 (s, 3H), 3.85 (s, 3H), 3.88 (m, 2H), 6.23 (s, 2H), 6.77 (m, 3H), 7.01 (d, 1H, J=7.8 Hz), 7.38 (d, 1H, J=7.8 Hz).

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