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Photodecarboxylative benzylations of phthalimide in pH 7 buffer: a simple access to 3-arylmethyleneisoindolin-1-ones

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ABSTRACT

Photoadditions of phenylacetates to phthalimide in pH 7 buffer solution give the corresponding benzylated-hydroxyphthalimidines in moderate to high yields of up to 94%. In a micro-structured reactor, higher conversions and purities are achieved. With branched phenylacetates, photoaddition affords diastereoisomeric mixtures with low to moderate de values. Subsequent acid-catalyzed dehydration furnishes the corresponding 3-arylmethyleneisoindolin-1-ones in good to excellent yields and with high E-selectivities. Irradiation of the parent 3-phenylmethyleneisoindolin-1-one under oxidative conditions only leads to cis/trans-isomerization.

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Arylmethyleneisoindolin-1-ones (I; Fig. 1) and the structurally related aristolactams (II) incorporating the free NH-group represent important classes of pharmaceutically active compounds.^{1,2} Consequently, a large number of synthetic pathways to these target compounds have been reported.^{3–9} An alternative approach incorporating a photochemical key-step to N-alkylated derivatives of I has been developed by Griesbeck and co-workers.¹⁰

The key-step in the latter approach is the photodecarboxylative (PDC) addition of carboxylates to phthalimides, which represents a versatile alkylation method.^{11,12} Multi-gram scale photoreactions have also been realized using a 308 nm excimer light source.¹³ A major drawback of the original PDC procedure is the necessity of N-substituents due to an increase in pH during irradiation (final pH ca. 9-10). With 'free' phthalimide the initially formed benzylated-hydroxyphthalimidines are therefore obtained as their potassium salts and cannot be isolated easily. Early attempts to convert directly these salts to arylmethyleneisoindolin-1-ones (I) by an acidic work-up and subsequent extraction furnished complex mixtures with unreacted starting materials. Since the acidity of phthalimide $(pK_a = 8.3)$ differs significantly from that of common carboxylic acids ($pK_a = 5-6$), we therefore applied a mixture of acetone and pH 7 buffer solution for the irradiations instead



Figure 1. General structure of arylmethyleneisoindolin-1-ones and aristolactams.



Scheme 1. Additions of phenylacetates 2a-p to phthalimide (1).

(Scheme 1).¹⁴ When phthalimide (1) was irradiated for 2–5 h $(\lambda = 300 \pm 25 \text{ nm})$ using this mixture in the presence of three equivalents of phenylacetates **2a**–**p**, the desired addition products **3a**–**p** were isolated in yields of up to 94% (Table 1).¹⁵





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Table 1
Product yields for photodecarboxylative benzylations of 1

Product	Ar	Conversion ^a (%)	Time (h)	Yield (%)
3a	Ph	100	3	92
3b	,C	100	3	92
3c	OMe	100	3	85
3d	, OAc	100	2	92
3e	× F	100	3	80
3f	, Cl	100	3	86
3g	Br	100	3	76
3h		100	3	92
3i	OAc	100	3	93
3j	X,	100	3	91
3k	AcO	15 ^b	3	10 (67) ^c
31	I X	79 ^b	5	36 (46) ^c
3m		100	3	90
3n	oMe رواند کې د کې	100 ^b	3	93
30		100	3	54
3p	OMe OMe OMe	100 ^b	2	94

^a Conversion determined by ¹H NMR spectroscopy of the crude reaction mixture.

^b Larger amounts of 'simple' PDC product detected.

^c Yield based on conversion.

The progress of the reaction was easily monitored by TLC analysis or by passing the departing nitrogen stream through a saturated barium hydroxide solution until the precipitation of barium carbonate stopped. In most cases, the photoproduct **3** simply precipitated upon concentration of acetone, and could be isolated by filtration. All compounds **3** showed characteristic singlets for the C–OH group at around 90 ppm in their ¹³C NMR spectra. In isolated cases the corresponding 'simple' decarboxylation products (–CO₂H↔–H exchange), that is, toluene derivatives, or dibenzyls (Ar-CH₂CH₂-Ar) were detected by NMR in the crude products, but no attempt was made to isolate these compounds. Total consumption of phenylacetate due to these conversion rates for products **3k** and **3l**.

The easy reaction protocol was also applied to 'micro-photochemistry', that is, photochemical reactions in micro-reactors.¹⁶ The syntheses of **3b** and **3e** were used as the model reactions. A commercially available micro-reactor (dwell device, mikroglas), which was placed under a UV panel (Luzchem) fitted with five UVB lamps, was chosen (Fig. 2).¹⁷ Using a residence time of 2 h, improved yields of 97% (**3b**) and 98% (**3e**) were achieved. The thin layer within the micro-channel plate (0.5 mm) obviously favors a better penetration of the solution by light.

The PDC reactions involving branched carboxylates **4a** and **4b** resulted in *like-* and *unlike-*mixtures of **5a** and **5b** in high yields



Figure 2. Micro-reactor (dwell device, mikroglas) under a UV exposure panel (Luzchem). A 5 Euro-cent coin is used to illustrate the size of the reactor.

(Scheme 2). Complete conversions were achieved after 2 h and 4 h of irradiation. In both cases, two complete sets of signals were observed in the NMR spectra which were assigned to the individual diastereoisomers. The diastereoisomeric ratios (de) were determined by integration of baseline separated signals in the ¹H NMR spectra. Stereoselectivities were low with 13% for **5a** and moderate with 33% for **5b** (Table 2), respectively. An assignment of the diastereoisomers was not possible.

The arylmethyleneisoindolin-1-ones **6a–p** were easily available in moderate to high yields of 41–97% via acid-catalyzed dehydration in dichloromethane (Scheme 3; Table 3).¹⁸ In almost all cases, high *E*-selectivities were obtained as confirmed by ¹H NMR spectroscopy. The olefinic proton of the *Z*-isomer is strongly influenced by the shielding effect of the aromatic ring, whereas no such effect was observed in the case of the *E*-isomer.^{3a} Under the standard hydrolysis conditions, the acetoxy-substituted derivatives **6d**, **i**, and **k** underwent partial hydrolysis of the ester group (ca. 30%). Thus, complete hydrolysis to the corresponding phenolic compounds was enforced by heating the irradiation products **3d**, **i**,



Scheme 2. Additions of branched phenylacetates 4 to phthalimide (1).

Table 2 Product yields and de ratios for photodecarboxylative additions of ${\bf 4}$ to ${\bf 1}$

Product	R	Time (h)	de ^a (%)	Yield (%)
5a	Me	2	13	97
5b	Et	4	33	94

 $^{\rm a}$ de ratios were determined by $^1{\rm H}$ NMR spectroscopy of the crude reaction mixture.



Scheme 3. Acid-catalyzed dehydration of 3a-p.

Table 3				
Product yields and	E:Z ratios	for dehy	/drations	of 6a-p

Product	Yield (%)	E:Z ^a
6a	88	>10:1
6b	80	>10:1
6c	91	>10:1
6d	70 ^b	>10:1
6e	90	>10:1
6f	90	>10:1
6g	87	>10:1
6h	82	>10:1
6i	60 ^b	>10:1
6j	63	>10:1
6k	23 ^b	>10:1
61	41	>10:1
6m	97	3:1
6n	90	>10:1
60	70	>10:1
6p	94	7:1

^a Determined by ¹H NMR spectroscopy.

^b After dehydration and hydrolysis (see text).

and ${\bf k}$ in a mixture of acetone, water, and concd HCl (40:28:12) instead. 19

Following the initial dehydration procedure,¹⁸ the branched photoproducts **5a** and **5b** were likewise converted into the corresponding arylmethyleneisoindolin-1-ones **7a** and **7b** in 88% and 89%, respectively (Scheme 4). In both cases, high *E*-selectivities of >10:1 were again observed.^{3a}

An attempt was made to convert **6a** into the parent aristolactam **8** via an oxidative electrocyclization, as proposed by Griesbeck and co-workers (Scheme 5, path **A**).^{10a} This photochemical reaction is widely observed for stilbenes, and proceeds easily in the presence of traces of a mild oxidant.²⁰ In contrast, irradiations of air-saturated benzene solutions of **6a** at 254 or 300 nm for 4 h only gave *cis/trans*-isomerizations (Scheme 5, path **B**). In both cases, ¹H NMR spectroscopic analysis of the crude product showed an isomeric ratio of approximately 1:1. Obviously, the geometry of the isoindolinone-ring prevents the necessary close proximity of the 'inner' carbon atoms for successful electrocyclization.²¹

The mechanism of the photodecarboxylative addition is illustrated in Scheme 6.²² The reaction is initiated by triplet-sensitization of acetone. Subsequent electron transfer from the carboxylate to the triplet-excited phthalimide yields an unstable carboxy radical which undergoes rapid decarboxylation to its corresponding benzyl radical (path **A**). Protonation by water and C–C bond formation furnishes the observed photoaddition products.²³ The mecha-



Scheme 4. Acid-catalyzed dehydration of 5a and 5b.



Scheme 5. Attempted electrocyclic ring-closure of 6a.



Scheme 6. Mechanistic scenario.

nistic scenario differs slightly for the di- and tri-alkoxyphenyl acetates **2n**–**p**, respectively. For these compounds electron transfer from the electron-rich aryl moiety has been proposed instead (path **B**).¹⁰ Decarboxylation, protonation, and C–C bond formation again give the desired addition products.

The developed reaction cascade is remarkably efficient and gives the desired 3-arylmethyleneisoindolin-1-ones in a simple two-step procedure in good to high yields and excellent purities. In addition, all the starting materials are stable and readily available in large quantities. In contrast to most thermal syntheses,^{3–9} the reaction protocols also completely avoid transition metal catalysts or other sensitive reagents. An alternative photochemical access to compounds **3a** and **3c**, starting from the phthalimide anion and toluene derivatives, has been reported by Suau and co-workers.²⁴ However, this photoaddition suffers from low selectivity if other potent hydrogen donor sites are present as, for example, in 4-methoxytoluene.

In conclusion, phthalimide readily undergoes photobenzylations with a range of phenylacetates at pH 7. Using a commercially available micro-reactor, higher yields could be obtained, thus unambiguously proving the concept of 'micro-photochemistry'. Subsequent acid-catalyzed dehydration opens a short pathway to important 3-arylmethyleneisoindolin-1-ones with high *E*-selectivity. The free NH-group allows easy incorporation of a variety of substituents and this application is currently being investigated.

Acknowledgments

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- 15. General irradiation procedure: Phthalimide (1.5 mmol) was dissolved in acetone (50 mL). A solution of the potassium phenylacetate (4.5 mmol) in pH 7 buffer (Fixanal[®], 50 mL) was added, and the mixture was irradiated (Rayonet Photochemical Reactor RPR-200; λ = 300 ± 20 nm) at 15-20 °C in a Pyrex Schlenk tube (λ ≥ 300 nm) while purging with a slow stream of nitrogen. The progress of the reaction was monitored by TLC analysis or by passing the departing gas stream through a saturated barium hydroxide solution until

the precipitation of barium carbonate had ceased. Most of the acetone was evaporated and the remaining solution was extracted with CH_2Cl_2 (3 \times 50 mL). The combined organic layer was washed with saturated NH₄Cl (1×50 mL) and brine (1 \times 50 mL), dried over MgSO₄, and evaporated. The crude products were purified by column chromatography (eluent: *n*-hexane/EtOAc = 1:1). In most cases, the pure product precipitated during evaporation of acetone and was isolated by vacuum filtration and dryied in vacuo instead. Selected physical and spectral data for 3-(3,4-dimethoxybenzyl)-3-hydroxy-isoindolin-1-one 3n: colorless solid, mp 170 °C. R_f (SiO₂, n-hexane/EtOAc = 1:1): 0.13. ¹H NMR (300 MHz, acetone- d_6): δ (ppm) = 3.28 (d, ²J = 13.5 Hz, 1H, CH₂), 3.36 (d, ²J = 13.5 Hz, 1H, CH₂), 3.58 (s, 3H, OCH₃), 3.69 (s, 3H, OCH₃), 5.32 (s, 1H, OH), 6.64 (m, 3H, Harom), 7.43 (m, 2H, Harom), 7.60 (m, 2H, Harom), 7.81 (s, 1H, NH). ¹³C NMR (75 MHz, acetone- d_6): δ (ppm) = 45.3, 55.9, 56.0, 88.9, 112.1, 115.6, 123.4, 123.8, 123.9, 129.2, 129.8, 132.6, 133.2, 149.2, 149.6, 149.8, 168.5. IR (KBr): v = 3329, 2948, 2833, 1686, 1514, 1467, 1421, 1263, 1236, 1140, 1023, 767, 701 cm⁻¹. HR-MS (ESI, negative mode): Calcd $[M-H]^*$: 298.1085 for C₁₇H₁₇NO₄ – H^{*}. Found $[M-H]^*$: 298.1088. HR-MS (ESI, positive mode): Calcd [M+Na]⁺: 322.1050 for C₁₇H₁₇NO₄ + Na⁺. Found [M+Na]⁺: 322.1053.

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- 17. The dwell-reactor is made out of FoturanTM glass ($\lambda \ge 300 \text{ nm}$) and has a total path length of 1.15 m (20 turns) on a 118 mm × 73 mm aperture. The reactor consisted of a (bottom) serpentine reaction channel 0.5 × 2 mm (D × W), with a second (top), heat-exchanging channel through which water is passed in order to control the reactor temperature. The degassed reaction mixture (10 mL)¹⁵ was pumped through the reaction channel via a syringe pump and collected in a test tube.
- 18. General dehydration procedure: Compound **3** (1–2 mmol) was dissolved in CH_2Cl_2 (30 mL). The reaction mixture was cooled in an ice-bath (0 °C) and 3–5 drops of concd H_2SO_4 were added. The reaction mixture turned yellow instantly and precipitation occurred. After stirring overnight at room temperature, H_2O (ca. 30 mL) was added, and most of the CH_2Cl_2 was evaporated. In most cases, the pure product precipitated completely during evaporation and was isolated by vacuum filtration, washing with H_2O (ca. 50 mL), and drying in vacuo. Alternatively, the remaining solution was extracted with CH_2Cl_2 (3 × 30 mL). The combined organic layer was washed with brine (1 × 50 mL), dried over MgSO₄ and evaporated.

Selected physical and spectral data for (*E*)-3-(4-methoxybenzylidene)isoindolin-1-one *E*-**6**c: yellow solid, mp 196 °C. $R_{\rm f}$ (SiO₂, *n*-hexane/EtOAc = 1:1): 0.55. ¹H NMR (300 MHz, CDCl₃): δ (ppm) = 3.86 (s, 3H, OCH₃), 6.52 (s, 1H, CH_{olef}), 6.76 (m, 2H, H_{arom}), 7.38 (m, 2H, H_{arom}), 7.50 (ddd, ³J = 7.6, ⁴J = 1.0 Hz, 1H, H_{arom}), 7.63 (ddd, ³J = 7.6, ⁴J = 1.2 Hz, 1H, H_{arom}), 7.77 (dd, ³J = 7.6, ⁴J = 1.2 Hz, 1H, H_{arom}), 7.77 (dd, ³J = 7.6, ⁴J = 1.0 Hz, 1H, H_{arom}), 7.88 (dd, ³J = 7.6, ⁴J = 1.2 Hz, 1H, H_{arom}), 7.79 (s, 1H, NH). ¹³C NMR (75 MHz, CDCl₃): δ (ppm) = 31.1, 106.0, 115.0, 119.8, 123.7, 127.7, 129.1, 129.9, 131.9, 132.3, 138.5, 159.4, 165.2, 169.0. IR (KBr): *v* = 3416, 1702, 1602, 1513, 1300, 1252, 1179, 1031, 849, 820 cm⁻¹. HR-MS (ESI, negative mode): Calcd [M-H]⁺: 250.0874 for C₁₆H₁₃NO₂-H⁺. Found [M-H]⁺: 250.0866. HR-MS (ESI, positive mode): Calcd [M+H]⁺: 252.1019 for C₁₆H₁₃NO₂ + H⁺. Found [M+H]⁺: 274.0830.

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