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Photodecarboxylative additions of phenoxyacetates to *N*-methylphthalimide

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ABSTRACT

Photoaddition of various phenoxyacetates to *N*-methylphthalimide affords the corresponding hydroxyphthalimidines in yields of 21–93%. The diastereoselectivity of the intermolecular addition is studied for a series of 2-substituted phenoxyacetates with low diastereoselectivities being observed. Comparison experiments with anisole and ether-containing phthalimide confirm that the crucial electron-transfer step occurs from the carboxylate functionality.

As is apparent from several review articles, the phthalimide chromophore has attracted significant interest in synthetic organic photochemistry.[1] Among the numerous applications, addition reactions of alkenes, alcohols, ethers, thioethers, alkylbenzenes, and amines to the phthalimide system have been described.[2] We have intensively studied the photodecarboxylative addition of carboxylates, α-keto carboxylates, and heteroatom-substituted carboxylates to phthalimides as a versatile alternative to Grignard-additions.[3] The developed method has been applied to the synthesis of arylmethylene-isoindolin-1-ones (**I**; Fig. 1),[4] and in particular to 'open analogues' of aristolactams (**II**).[5] In continuation of this medicinal chemistry application, we became interested in the synthesis of aryloxymethylene isosteres of arylmethylene-isoindolin-1-ones.

N-Methylphthalimide **1** was chosen as a model compound for this study and was irradiated (λ = 300 ± 25 nm) in aqueous acetone in the presence of three equivalents of phenoxyacetates **2a–j** (Scheme 1).[6] The reaction progress was monitored by TLC analysis or by passing the departing nitrogen stream through a saturated barium hydroxide solution until precipitation of barium carbonate ceased. Following this procedure, the corresponding addition products **3a–j** were isolated in poor to excellent yields of 21–93% (Table 1).

For all compounds **3**, the C–OH group appeared as a characteristic singlet at around 90 ppm in the ^{13}C NMR spectrum. In some cases (**2c**, **g**, **h,** and **j**), larger amounts of the corresponding 'simple' decarboxylation products ($-CO_2H \leftrightarrow -H$ exchange), that is, anisole derivatives, were identified in the crude NMR spectrum, but no attempt was made to isolate these products. The *O*-benzyl-derived carboxylate **2j** gave a somewhat lower yield of the corresponding addition product **3j**. In this case, H-abstraction from the benzylic position may compete with decarboxylation.

The photoreaction involving 2-(4-chloro-2-methylphenoxy) acetate **2e** gave a mixture of the desired compound **3e** and a second, minor product (30%). Both the ^{1}H and ^{13}C NMR gave two com-

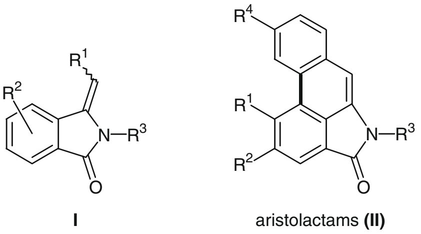
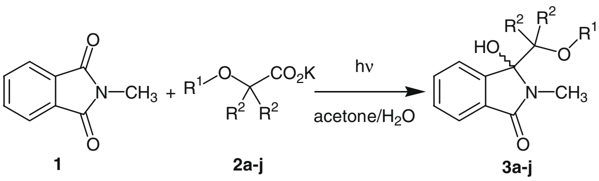


Figure 1. Examples of arylmethylene-isoindolin-1-ones.



Scheme 1. Additions of phenoxyacetates **2** to *N*-methylphthalimide **1**.

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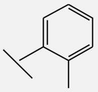
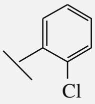
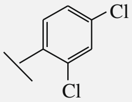
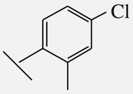
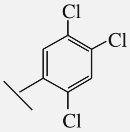
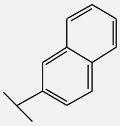
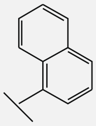
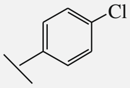
Table 1
Product yields for the photodecarboxylative additions of **2** to **1**

Entry	R^1	R^2	Time (h)	Conversion[a] (%)	Yield (%)
a	Ph	H	1.5	100	85
b		H	1	100	93
c		H	11	61[b]	53 (87[c])
d		H	1	100	71
e		H	1	91	55 (61[c,d])
f		H	3	87	51 (64[c])
g		H	4	65[b]	48 (74[c])
h		H	10	29[b]	21 (72[c])
i		Me	2	73	51 (70[c])
j	Bn	H	5	100[b]	38

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

[b] Larger amounts of 'simple' photodecarboxylation (PDC) products identified in the crude NMR spectrum.

[c] Yield based on conversion.

[d] Contains 30% of **3b**.

plete sets of signals, although overlap within the ^1H NMR made a definite assignment difficult. The purity of the corresponding carboxylic acid of **2e** was confirmed by NMR spectroscopy, and therefore the by-product must have been formed during irradiation. Based on the ^1H NMR spectrum, in particular the aromatic region, it was assumed that partial dehalogenation (–Cl → –H exchange) of **3e** to **3b** had occurred. A mixed NMR was subsequently recorded with independently synthesized **3b**. Following this strategy, all signals from the by-product in **3e** increased in height (Fig. 2 shows the pair of doublets for the methylene bridge $-CH_2Ar$ protons), thus unambiguously proving that it was indeed identical with **3b**. The reason for the partial dehalogenation of **3e** remains unclear at present. However, since this reaction was not observed for the related 2,4-dichlorinated carboxylic acid **2d**, the electronic nature of the *ortho*-substituent may play a crucial role in photodehalogenation. The electron-donating methyl group may thus favor dehalogenation, whereas the chloro-substituent shows no effect. Similar photodehalogenation reactions have been described in the literature.[7]

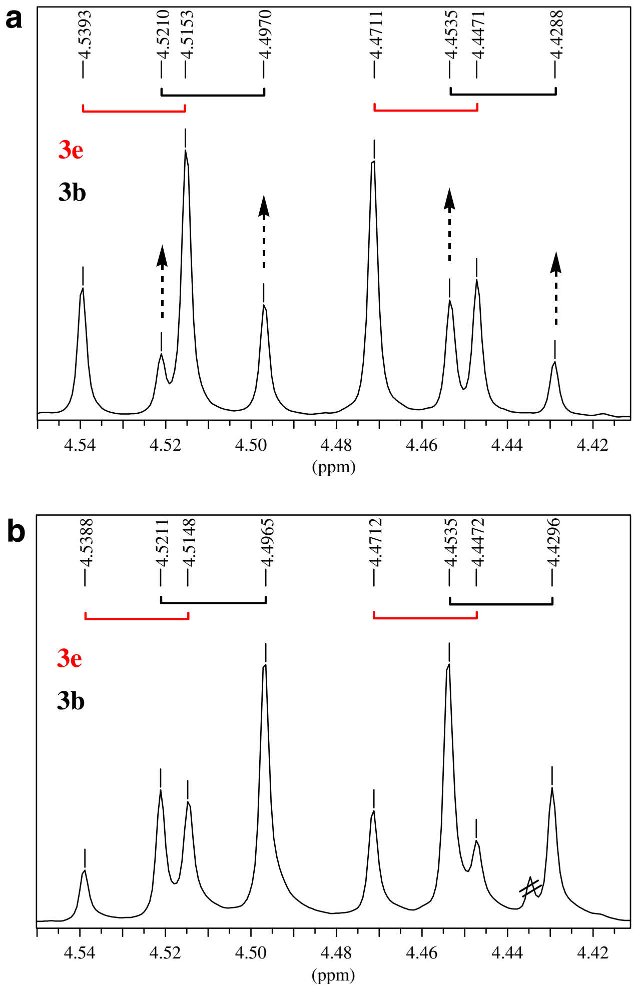
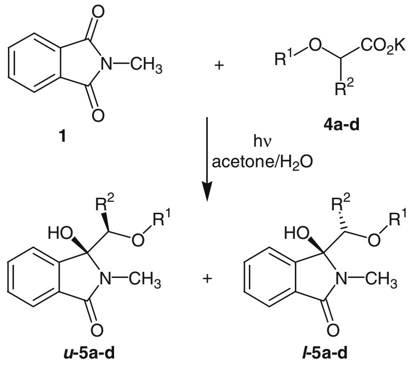


Figure 2. ^1H NMR comparison: (a) original crude mixture (top), and (b) after addition of authentic **3b** (bottom).

The photoreactions involving branched carboxylates **4a–d** furnished diastereoisomeric product mixtures **5a–d** (Scheme 2).[8] In all cases, two sets of signals were observed in the ^1H NMR spectra which were assigned to the two diastereoisomers. The diastereoisomeric ratio (de) was determined by integration of baseline separated signals in the ^1H NMR spectra. Selectivities were low for compounds **5a–c** being 6%; for **5d** a higher de of 28% was achieved



Scheme 2. Additions of branched phenoxyacetates **4** to *N*-methylphthalimide **1**.

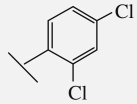
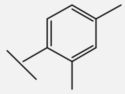
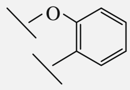
(Table 2). An assignment of the *like*- and *unlike*-diastereoisomers was not made.

Since phthalimides are known to react with ethers via H-abstraction,[9] *N*-methylphthalimide **1** was irradiated in the presence of five equivalents of anisole **6** (Scheme 3). Even after prolonged irradiation of 20 h, no addition product **3a** or photoreduction product was observed. Instead, **1** was recovered in 98% yield.

In order to establish the nature of the crucial photoinduced electron transfer (PET) step,[10], that is, whether it occurs from the carboxylate or the ether functionality, *N*-methoxymethylphthalimide **7** was irradiated in the presence of potassium propionate **8** (Scheme 4). Photodecarboxylative addition occurred smoothly and after 2 h of irradiation, compound **9** was isolated in a moderate yield of 51%.[11] No cyclization or photoreduction products arising from competing *intramolecular* CH-activations were detected.[12]

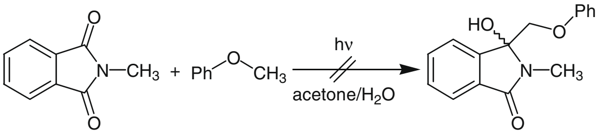
Although product formation for phenoxyacetates **2** and **4** can be explained by two competing electron transfer routes (Scheme 5), the high oxidation potentials of dialkyl ethers (E_{Ox} >2.5 V vs SCE[13]) makes electron transfer from the heteroatom (path **A**) unlikely.[14,15] Instead, electron transfer most likely occurs from the carboxylate functionality (path **B**), generating an unstable carboxy radical that subsequently undergoes decarboxylation.[16] The resulting carbon-centered radical furnishes the observed addition products **3** and **5** (path **C**). The detected 'simple' decarboxylation products are formed through back electron transfer (BET) and subsequent protonation of the corresponding carbanions (path **D**).[17] This pathway was only competitive for carboxylates **2c**, **g**, **h,** and **j**, and consequently, the isolated yields for the desired addition products **3c**, **g**, **h,** and **j** remained low due to complete consumption of the carboxylate. Based on the unsuccessful addition of anisole to **1** it can be concluded that 'simple' decarboxylation products, if formed, do not contribute to the formation of addition products **3** or **5**, respectively.

Table 2
Product yields and de ratios for photodecarboxylative additions of **4** to **1**

Entry	R^1	R^2	Time (h)	Conversion[a] (%)	de[a] (%)	Yield (%)
a	Ph	Me	2	100	6	87
b		Me	11	99	6	61 (62[b])
c		Me	1	100	6	80
d			1	95	28	83 (87[b])

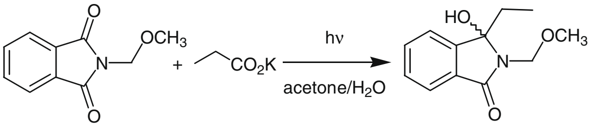
[a] Conversion and de determined by ^{1}H NMR spectroscopy of the crude reaction mixture.

[b] Yield based on conversion.



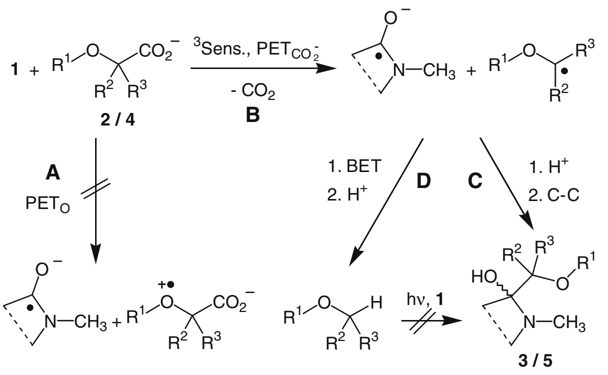
1 6 3a

Scheme 3. Attempted addition of anisole **6** to **1**.



7 8 9

Scheme 4. Addition of propionate **8** to *N*-methoxymethylphthalimide **7**.



Scheme 5. Mechanistic scenario.

The photoreactivity of *N*-methoxymethylphthalimide **7** correlates well with photoaddition reactions involving simple carboxylates.[3b,d] Incorporation of an ether group into the N-side chain has no influence on the ethylation and therefore electron transfer must occur exclusively from the propionate. In contrast, thioether-derived phthalimides are known to completely suppress photodecarboxylative additions.[18]

In conclusion, phenoxyacetates readily undergo photodecarboxylative addition to *N*-methylphthalimide **1**. The simple and general procedure developed offers a versatile access to 3-alkyl- and 3-arylmethylene-isoindolin-1-one isosteres and this potential application is currently being investigated. This application is also currently being transferred to 'micro-photochemistry', that is, photochemical transformations in micro-structured devices.[19]

Acknowledgements

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6. *General procedure for irradiation: N*-methylphthalimide (1.5 mmol) was dissolved in acetone (50 mL). A solution of the potassium carboxylate (4.5 mmol) in water (50 mL) was added, and the mixture was irradiated (Rayonet Photochemical Reactor RPR-200; $\lambda = 300 \pm 20$ nm) at 15–20 °C in a Pyrex tube ($\lambda \geqslant 300$ 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 precipitation of barium carbonate ceased. Most of the acetone was evaporated and the remaining solution was extracted with CH_2Cl_2 (3 × 50 mL). The combined organic layers were washed with 5% $NaHCO_3$ (50 mL) and brine (50 mL), dried over $MgSO_4$, and evaporated. The products were purified by column chromatography (eluent: *n*-hexane/EtOAc = 1:1). In some cases, the pure product precipitated upon evaporation of acetone and was isolated by vacuum filtration and drying in vacuo instead.
   Selected physical and spectral data for *3-hydroxy-2-methyl-3-[(naphthalene-1-yloxy)methyl]isoindolin-1-one* **3h**: yellow solid, mp 177–182 °C. ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 2.91 (s, 3H, NCH_3), 4.17 (s, 1H, OH), 4.47 (d, $^2J = 9.6$ Hz, 1H, CH_2), 4.55 (d, $^2J = 9.6$ Hz, 1H, CH_2), 6.79 (d, $^3J = 7.3$ Hz, 1H, H_{arom}), 7.32 (m, 2H, H_{arom}), 7.42 (m, 2H, H_{arom}), 7.46 (ddd, $^3J = 7.3$, $^3J = 7.3$, $^4J = 1.0$ Hz, 1H, H_{arom}), 7.55 (ddd, $^3J = 7.3$, $^3J = 7.3$, $^4J = 1.0$ Hz, 1H, H_{arom}), 7.69 (m, 4H, H_{arom}). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 24.2, 68.8, 89.2, 105.3, 121.6, 121.8, 122.4, 123.6, 125.5, 125.8, 125.9, 126.9, 127.7, 130.4, 131.9, 132.8, 134.7, 145.6, 153.9, 168.3. IR (KBr): ν = 3295, 2945, 2347, 1676, 1618, 1070, 874 cm^{-1}. MS (EI, 70 eV): *m/z* (%) = 319 (M^+, 6), 301 ($M^+{-}H_2O$, 100), 272 (301-COH, 18), 244 ($M^{+1}{-}C_6H_4$, 7), 162 ($M^+{-}C_{11}H_9O$, 84), 77 (C_6H_5, 23). MS (ESI, positive ions): *m/z* = 320 $(M + H)^+$, 639 $(M_2{+}H)^+$. HR-MS (ESI, positive ions): calcd $[M{+}H]^+$: 320.12812 for $C_{20}H_{17}NO_3{+}H^+$. Found $[M{+}H]^+$: 320.12783. calcd $[M{+}Na]^+$: 342.11006 for $C_{20}H_{17}NO_3{+}Na^+$. Found $[M{+}Na]^+$: 342.10988.
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8. Selected physical and spectral data for *3-[1-(2,4-dichlorophenoxy)ethyl]-3-hydroxy-2-methylisoindolin-1-one* ***u*-/*l*-5b**: yellow solid, mp (mixture) 115–125 °C. Main diastereoisomer: ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 1.01 (d, $^3J = 6.3$ Hz, 3H, CH_3), 2.97 (s, 3H, NCH_3), 4.52 (s, 1H, OH), 4.76 (q, $^3J = 6.3$ Hz, 1H, CH), 6.90 (d, $^3J = 8.8$ Hz, 1H, H_{arom}), 7.13 (dd, $^3J = 8.8$, $^4J = 2.5$ Hz, 1H, H_{arom}), 7.31 (d, $^4J = 2.5$ Hz, 1H, H_{arom}), 7.40 (m, 1H, H_{arom}), 7.47–7.55 (m, 2H, H_{arom}), 7.58 (d, $^3J = 7.9$ Hz, 1H, H_{arom}). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 14.8, 25.8, 80.1, 91.3, 116.8, 123.4, 123.9, 125.1, 127.6, 128.2, 130.7, 130.8, 132.4, 132.6, 144.2, 152.4, 168.4. Minor diastereoisomer: ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 0.82 (d, $^3J = 6.3$ Hz, 3H, CH_3), 2.84 (s, 3H, NCH_3), 3.15 (s, 1H, OH), 4.72 (q, $^3J = 6.3$ Hz, 1H, CH), 6.98 (d, $^3J = 8.9$ Hz, 1H, H_{arom}), 7.14 (dd, $^3J = 8.9$, $^4J = 2.5$ Hz, 1H, H_{arom}), 7.34 (d, $^3J = 2.5$ Hz, 1H, H_{arom}), 7.41 (m, 1H, H_{arom}), 7.47–7.55 (m, 1H, H_{arom}), 7.61 (d, $^3J = 7.9$ Hz, 1H, H_{arom}), 8.02 (d, $^3J = 7.6$ Hz, 1H, H_{arom}). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 14.9, 24.4, 78.7, 91.2, 116.9, 122.4, 125.3, 125.5, 127.3, 128.1, 130.3, 130.6, 132.0, 132.8, 144.1, 152.8, 168.2. IR (KBr, mixture): ν = 3304, 2983, 2346, 1681, 1618, 1064, 750 cm^{-1}. MS (EI, 70 eV, mixture): *m/z* (%) = 351 (M^+, <1), 333 ($M^+{-}H_2O$, <1), 188 ($M^+{-}C_9H_8NO_2^-$, 23), 162 ($M^+{-}C_8H_7Cl_2O^+$, 100), 146 ($M^+{-}C_{11}H_{12}NO_3$, 8), 77 (C_6H_5, 8). HR-MS (ESI, positive ions, mixture): calcd $[M{+}H]^+$: 352.05018 for $C_{17}H_{15}Cl_2NO_3{+}H^+$. Found $[M{+}H]^+$: 352.05007.
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11. Physical and spectral data for *3-ethyl-3-hydroxy-2-(methoxymethyl)-isoindolin-1-one* **9**: yellow oil. ^1H NMR (400 MHz, $CDCl_3$): δ (ppm) = 0.59 (t, $^3J = 7.6$ Hz, 3H, CH_3), 2.24 (m, 2H, CH_2), 3.40 (s, 3H, OCH_3), 4.93 (m, 2H, CH_2), 5.30 (s, 1H, OH), 7.58 (ddd, $^3J = 7.6$, $^3J = 7.6$, $^4J = 1.0$ Hz, 1H, H_{arom}), 7.65 (d, $^3J = 7.6$ Hz, 1H, H_{arom}), 7.71 (ddd, $^3J = 7.6$, $^3J = 7.6$, $^4J = 1.0$ Hz, 1H, H_{arom}), 7.75 (d, $^3J = 7.2$ Hz, 1H, H_{arom}). ^{13}C NMR (100 MHz, $CDCl_3$): δ (ppm) = 8.3, 31.2, 57.0, 70.5, 91.9, 123.1, 123.8, 130.1, 133.5, 144.0, 149.0, 166.0.
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