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PII: S0040-4020(16)30817-1

DOI: 10.1016/j.tet.2016.08.041

Reference: TET 28022

To appear in: *Tetrahedron*

Received Date: 7 July 2016

Revised Date: 4 August 2016

Accepted Date: 12 August 2016

Please cite this article as: Enright RN, Grinde JL, Wurtz LI, Paeth MS, Wittman TR, Cliff ER, Sankari YT, Henningsen LT, Tan C, Scanlon JD, Willoughby PH, Synthesis of *N*,*O*-acetals by net amide C-N bond insertion of aldehydes into *N*-acyl phthalimides and *N*-acyl azoles, *Tetrahedron* (2016), doi: 10.1016/j.tet.2016.08.041.

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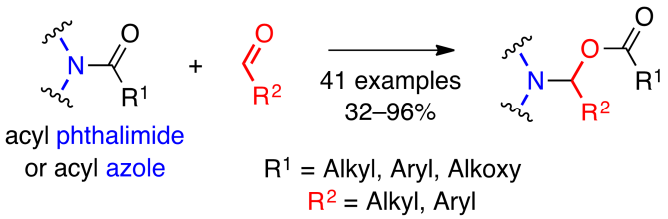

Tetrahedron
journal homepage: www.elsevier.com

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ARTICLE INFO

Article history:
Received
Received in revised form
Accepted
Available online

Keywords:
N,O-Acetal Synthesis
C–N Insertion
Acyl Phthalimide
Acyl Azole

ABSTRACT

We found that *N*-acyl phthalimides and several *N*-acylated azoles are capable of reacting with aldehydes to form *O*-acyl-*N,O*-acetals in an apparent amide C–N bond insertion. In the context of *N*-acyl phthalimides, the reaction is mediated by substoichiometric amounts of sodium iodide and potassium phthalimide. DFT computations supported a proposed mechanism and provided insights into the effect of the alkali metal additive. This strategy could be used to prepare a myriad of *N,O*-acetals from a range of aldehydes. A one-pot procedure was also developed in which *N*-acyl phthalimide was generated *in situ* prior to forming the *N,O*-acetal product. The one-pot strategy was used to demonstrate that activated amides derived from imidazole, pyrazole, (benzo)triazole, and tetrazole are also amenable substrates. Collectively, these studies provide an approach to the synthesis of a variety of *N,O*-acetals under mild conditions from inexpensive starting materials.

1. Introduction

N,O-Acetals are a privileged functionality because of their (i) role in the bioactivity of medicinally-relevant molecules and (ii) usefulness as a synthetic building block for the preparation of complex nitrogen-containing compounds. There are numerous[1] bioactive natural products with an *N,O*-acetal moiety including, for example, cytotoxic agents psymberin (i.e., irciniastatin A, **1**, Figure 1a)[2] and pederin (**2**).[3] Recently, structure-activity studies by Floreancig and co-workers demonstrated that the *N,O*-acetal moiety of **1** and **2** was necessary for potent bioactivity.[4] In the context of organic synthesis, *N,O*-acetals have proven to be useful surrogates of imines because the oxygen functionality is readily activated under acidic conditions allowing for substitution by a variety of nucleophiles (cf. Figure 1b).[5] *N*-Acylated variants of *N,O*-acetals have proven particularly useful because they are generally bench stable unlike their *N*-acyl imine counterparts, which are highly susceptible to hydrolysis by adventitious water. Recently, Doyle[6] expanded the utility of *N,O*-acetals in organic synthesis by reporting a Ni-catalyzed cross coupling of *N*-acylated *N,O*-acetals (e.g., **3**) with arylboroxines to give 2-aryl quinolines (e.g., **4**). A related procedure reported by Lautens[7] describes a Pd-catalyzed arylation of trifluoromethyl-containing *N,O*-acetals. Collectively, these points demonstrate that the medicinal chemistry and organic synthesis communities would continue to benefit from additional approaches to prepare *N,O*-acetals.

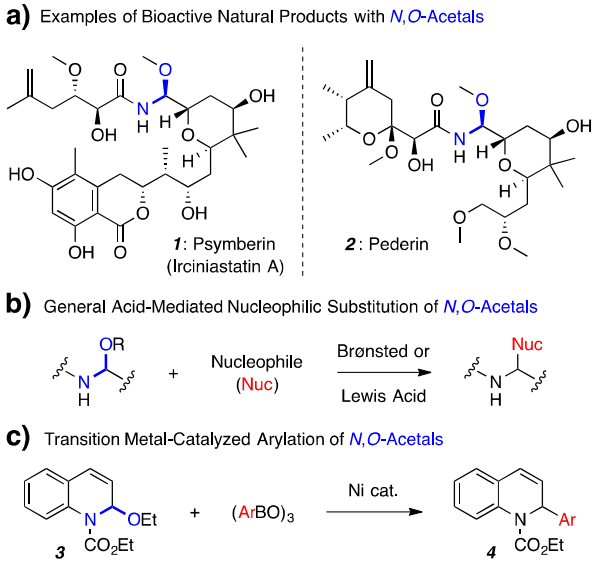


Figure 1: *N,O*-Acetals are (a) a critical functional group to bioactive natural products, (b) useful electrophiles in organic synthesis, and (c) participants in transition metal-catalyzed cross coupling reactions.

Numerous reports have described methods for preparing a variety of *N*-acylated *N*,*O*-acetals.[8] Herein we report conditions that effect the synthesis of *N*,*O*-acetals from *N*-acyl phthalimides (cf. **5**, Figure 2) and acyl azoles. The resulting products appear to arise via insertion of the aldehyde carbonyl into the amido C–N bond, providing a novel approach to making *O*-acyl-*N*-imido-*N*,*O*-acetals (e.g., **6**). Known strategies for making this family of phthalimide-containing *N*,*O*-acetals involve decarboxylative acetoxylation of α-phthalimdo acids with stoichiometric lead[8c] or radical intermediates.[8g] The conditions described in this report are mild (i.e., substoichiometric NaI and potassium phthalimide) and complement previous methods by using aldehyde starting materials. This report also includes a detailed mechanistic analysis, optimization studies for general reaction conditions, a one-pot procedure that uses commercially available starting materials, and a thorough evaluation of substrate scope.

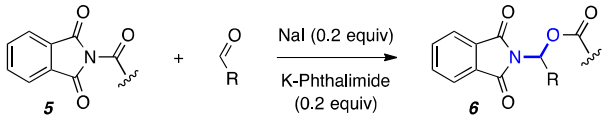
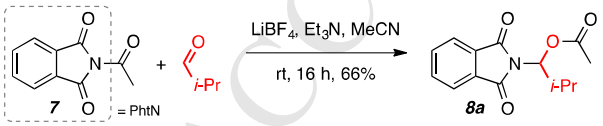


Figure 2: Methodology described in this report for the synthesis of *N*-phthalimido-*O*-acyl-*N*,*O*-acetals by reaction between *N*-acyl phthalimides and aldehydes.

2. Results and Discussion

As part of a study into the reactivity of *N*-acyl phthalimides with aldehydes, we found that treating a mixture of *N*-acetyl phthalimide (**7**, Scheme 1) and isobutyraldehyde with Et_3N and $LiBF_4$ led to the unexpected formation of *N*,*O*-acetal **8a**. We initially speculated that we had simply expanded the scope of conditions previously reported Katritzky in which *N*,*O*-acetals were formed by treating acyl benzotriazoles with an aldehyde and K_2CO_3.[9] However, product was not observed when we applied Katritzky's conditions (i.e., 0.25 equiv of K_2CO_3 in MeCN) to the synthesis of **8a**. When the K_2CO_3 loading was increased to 1 equiv, the yield of **8a** improved to only 8%.[10] It was not immediately clear why use of *N*-acyl phthalimide **7** led to diminished reaction efficiency under Katritzky's conditions. However, it was encouraging that the combination of $LiBF_4$ and Et_3N gave product in moderate yield, suggesting that similar conditions could provide a general approach to making *N*,*O*-acetals from amides.

Scheme 1. Synthesis of *N*,*O*-acetal **8a** by net C–N bond insertion of isobutyraldehyde into *N*-acyl phthalimide **7**



Prompted by the unusual nature of the transformation, we initially turned our attention to the mechanism. We performed a double label crossover experiment in which a mixture of isobutyraldehyde and *N*-acyl phthalimides **9** and **10** was treated with $LiBF_4$ and triethylamine (cf. Figure 3a). ^1H NMR and LC/MS analysis clearly showed that the reaction gave rise to a mixture of the four possible *N*,*O*-acetal products (i.e., **11-14**). The positive crossover experiment indicated that phthalimide dissociates during the course of the reaction, and led us to propose the mechanism depicted in Figure 3b. Specifically, the liberated phthalimide anion (i.e., **15**, shown with metal counterion M^+) adds to the aldehyde to form alkoxide adduct **16**. Acylation of **16** by *N*-acyl phthalimide **7** gives rise to the observed product, **8a**, and regenerates phthalimide anion. The proposed mechanism suggests that **15** is serving as a catalyst because the liberated phthalimide anion can subsequently engage another molecule of aldehyde to form *N*,*O*-acetal product and regenerate additional **15**. To account for the initial concentration of the phthalimide anion catalyst, we also propose that the reaction is initiated by hydrolyzing a sacrificial amount of *N*-acyl phthalimide with an OH donor (cf. Figure 3c). For example, hydrolysis of *N*-acyl phthalimide **7** in the presence of adventitious water would release **15** after proton transfer to triethylamine. Alternatively, initiation could occur by acyl transfer to i) the enol content of isobutyraldehyde and/or ii) a carboxylate impurity from aldehyde autoxidation. Under the proposed mechanism, Et_3N discourages formation of neutral phthalimide, which would likely not engage aldehyde in the proposed mechanism.

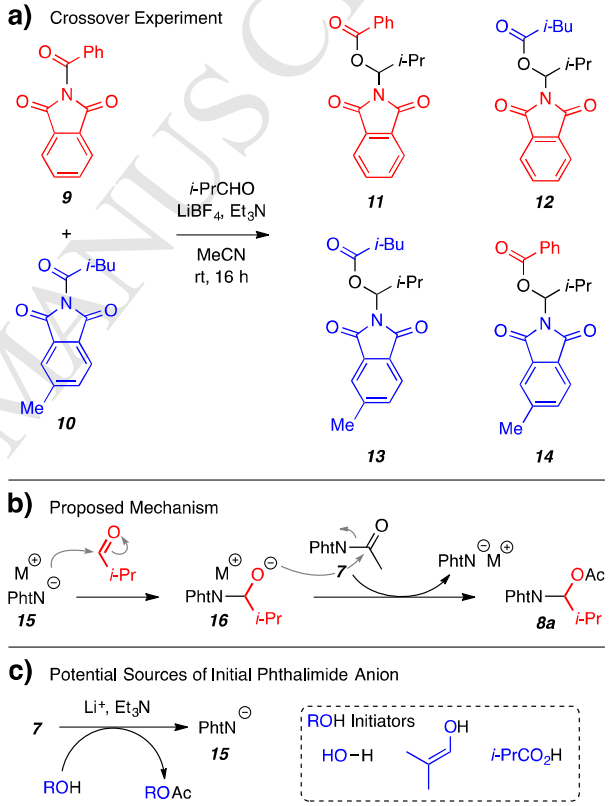
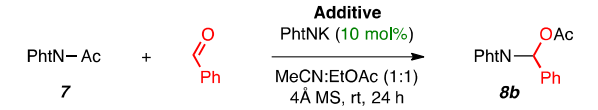


Figure 3: Mechanistic studies and resulting hypotheses: (a) a double label crossover experiment (b) a proposed mechanism for the conversion of *N*-acyl phthalimides into *N*,*O*-acetals (c) an initiation pathway in which an OH donor converts a sacrificial amount of *N*-acyl phthalimide into phthalimide anion.

Guided by the proposed mechanism, we turned to optimizing the reaction in the presence of catalytic metal phthalimide (cf. Table 1). To avoid competitive initiation by an enol, we performed the optimization studies with non-enolizable benzaldehyde, which gave *N*,*O*-acetal product **8b**. Additionally, 4Å molecular sieves were used to remove water, and benzaldehyde was freshly distilled to remove autoxidation impurities. Early optimization studies indicated that a 1:1 cosolvent mixture of acetonitrile and ethyl acetate gave improved yields. Substituting $LiBF_4$ and Et_3N for 10 mol% potassium phthalimide (PhtNK) led to a reduced yield of 39% (cf. entry 1, Table 1). This result was encouraging because it demonstrated

that a substoichiometric amount of metal phthalimide anion was capable of promoting reaction. No reaction was observed when PhtNK was omitted or phthalimide (N–H) was used, the later result supported the notion that Et_3N was initially (cf. Scheme 1) required to ensure the presence of phthalimide anion.[11]

Table 1. Reaction Optimization



Entry	Additive (*1 equiv*)	Yield[a]	Entry	Additive (*10 mol%*)	Yield[a]
1	none	39%	14	NaI	68%
2	$LiBF_4$	26%	15	NaCl	4%
3	LiCl	16%	16	NaBr	23%
4	LiI	55%	17	NaOAc	51%
5	$MgBr_2$•OEt_2	1%	18	Na_2CO_3	14%
6	NaI	80%	19	KBr	1%
7	CuI	1%	20	KI	38%
8	$Cu(OTf)_2$	2%	21	KOAc	23%
9	$Zn(OTf)_2$	15%	22	K_2CO_3	7%
10	$CoCl_2$	0%	23	KPF_6	10%
11	BF_3•OEt_2	1%	24	NaI (20 mol%)[b]	81%
12	TBAB	46%	25	PhtNNa[c]	53%
13	18-crown-6	14%	26	PhtNLi[c]	12%

[a]Yields were determined by ^{1}H NMR. [b]The loading of both NaI and PhtNK was increased to 20 mol%. [c]PhtNK was replaced by 10 mol% of the indicated additive.[12]

Screening a number of stoichiometric additives (cf. entries 2–11) and phase transfer reagents (cf. entries 12 and 13) led to a wide range of yields. Use of NaI gave the highest yield of 80%. This led us to study the ability of NaI to perform as a substoichiometric additive, and use of 10 mol% led to a slightly reduced yield of 68% (cf. entry 14). Use of 10 mol% of other alkali metal salts gave lower yields, ranging between 1–51% (cf. entries 15–23). We settled on the use of 20 mol% of both PhtNK and NaI, which gave 81% (cf. entry 24) yield and was similar to that obtained when using stoichiometric NaI in combination with 10 mol% of PhtNK (cf. entry 6).

The additive screen revealed an interesting trend with respect to the effect of the metal cation on reaction efficiency. Namely, use of substoichiometric amounts of soluble sodium salts (e.g., NaI and NaOAc) gave better yields than the corresponding potassium salts. Additionally, 10 mol% NaI gave higher yields than stoichiometric amounts of either LiI or $LiBF_4$. We suspected that NaI was providing an alternative counterion for the phthalimide catalyst, which would mean that sodium phthalimide could also be an efficient catalyst. This was confirmed by substituting potassium phthalimide with 10 mol% sodium phthalimide, which gave product in 52% yield (cf. entry 25). Additionally, lithium phthalimide was also used directly, which gave product in much lower yield (cf. 11%) than when either the potassium or sodium salts were used.[13] While it is not unexpected that the reaction yield be highly dependent on the metal counterion, it is interesting that use of lithium additives led to a much less efficient reaction.

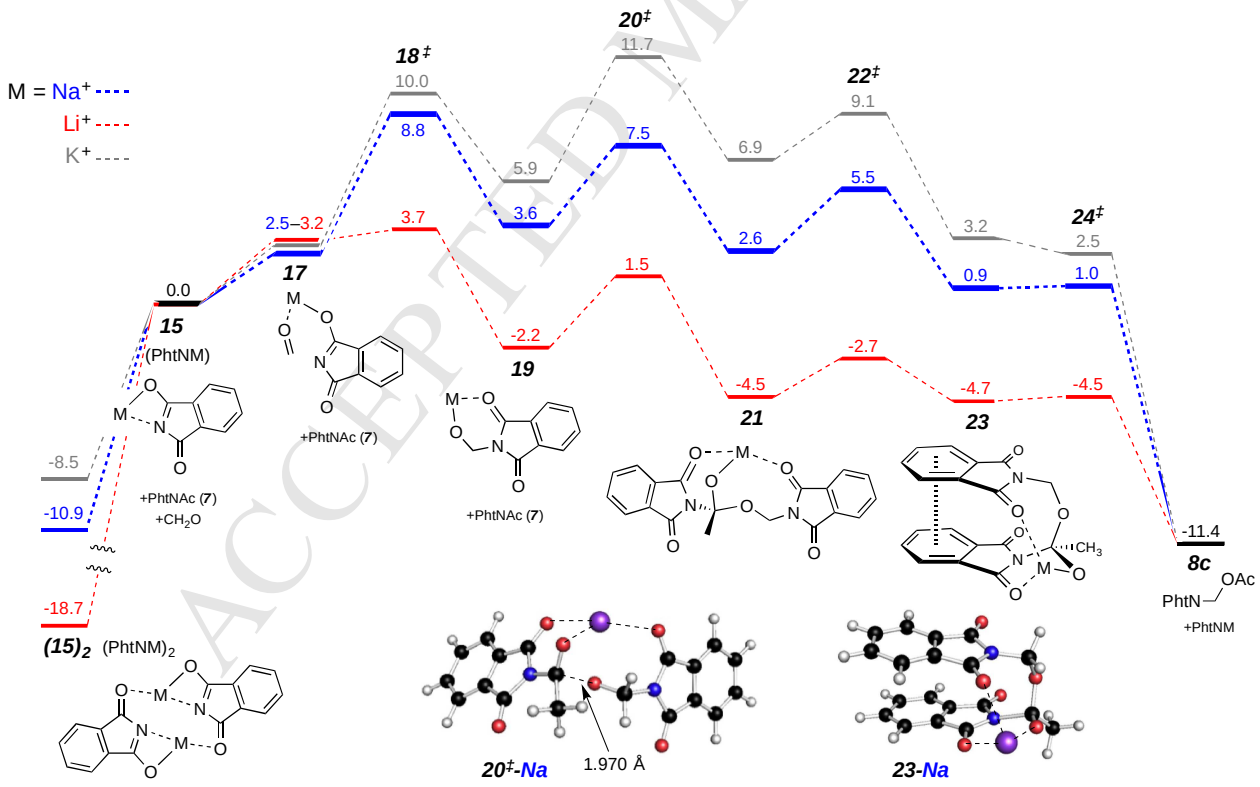


Figure 4. Relative free energies for the metal phthalimide-mediated reaction between *N*-acyl phthalimide **7** and formaldehyde to give *N,O*-acetal **8c**. Pathways involving Li^+, Na^+, and K^+ are indicated with different colors. Representative 3D structures of **20**‡ and **23** with sodium counterion are included to depict sodium coordination (cf. hashed lines to sodium) and π-stacking in **23**. DFT calculations were performed using M06/6-31+G(d,p)[14] with an automatically generated density fitting basis set and the SMD solvation model[15] (MeCN). All free energies values are in kcal/mol.

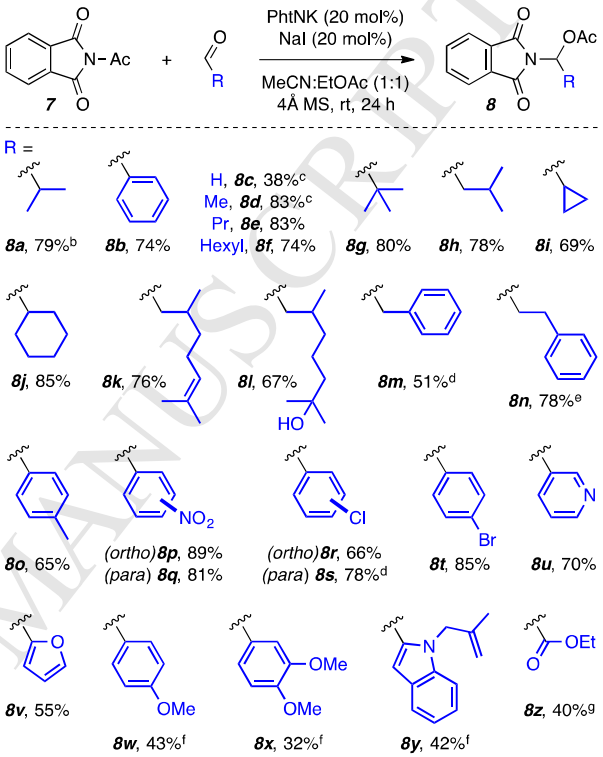
To provide support for the proposed reaction mechanism and gain insight into the alkali metal counterion effect, we turned to DFT computations. Based on the mechanism proposed in Figure 3b, we computed the lowest energy paths for reaction between **7** and formaldehyde to give *N*,*O*-acetal **8c** involving sodium, and potassium counterions (cf. Figure 4). The lowest energy path began with coordination (cf. **17**) and adduct formation (via **18**‡) between formaldehyde and the phthalimide salt (**15**) to give alkoxide **19**. Acylation of **19** then proceeds through tetrahedral intermediate **21** (via **20**‡). We were unable to locate a transition state that directly gave product from **21**. Instead, we located π-stacked tetrahedral intermediate **23** (via **22**‡), which gave product **8c** through a nearly barrierless transition state (cf. **24**‡). The rate-determining step for the pathway involving sodium counterion (shown in blue) was formation of alkoxide adduct **19** with a barrier of 6.3 kcal/mol from intermediate **17**. In the case of the potassium counterion (shown in grey), formation of intermediate **19** had a larger barrier of 7.1 kcal/mol from **17**. It is noteworthy that the barrier for formation of intermediate **21** increased from 3.9 to 5.8 kcal/mol when comparing the sodium and potassium pathways.

Using the same approach, we computed the energetics of the lithium phthalimide-mediated pathway (cf. red). Interestingly, nearly all intermediates and transition states were substantially lower in free energy than when calculated with sodium and potassium counterions. This is in contrast to the experimental results from our optimization studies in which use of lithium phthalimide or lithium-containing additives gave sluggish reactions and reduced yields. This led us to compute the energy of a variety of aggregation states for lithium phthalimide. A dimeric form (cf. $\mathbf{15}_2$) was found to be 18.7 kcal/mol lower in energy than the lithium phthalimide monomer. Additionally, metal phthalimide dimerization was found to be energetically favorable in the sodium (i.e., -10.9 kcal/mol) and potassium (i.e., -8.5 kcal/mol) pathways. These results suggest that each pathway involves uphill deaggregation of $\mathbf{15}_2$ prior to forming the *N*,*O*-acetal product, albeit to a much greater extent for the lithium pathway. Collectively, these results suggest why soluble lithium salts are not as effective metal additives for this transformation.

The scope of compatible aldehydes was evaluated using the optimized conditions (cf. Scheme 2). In general, we found that aliphatic (cf. **8a**, **8c**–**h**), cyclic (cf. **8i** and **8j**), and alkene-containing (cf. **8k**) aldehydes of varying substitution and steric hindrance readily reacted with **7** to give *N*,*O*-acetals with yields ranging from 38–85%. Successful use of hydroxycitronellal (cf. **8l**) demonstrates that aldehydes with hindered alcohols are capable of forming product.[16] The formation of products **8b** and **8m**–**8y** indicate that aryl- and heteroaryl-containing aldehydes also readily participate under the reaction conditions. Use of electron rich aldehydes to give products **8w**–**8y** resulted in poor yields, which is likely due to an unfavorable equilibrium for addition with phthalimide anion. This was overcome by adding a stoichiometric amount of both NaI and potassium phthalimide, which gave adequate yields ranging from 32–43%. To prepare **8z**, ethyl glyoxalate, which readily polymerizes at room temperature, was generated *in situ* by oxidative cleavage[17] of diethyl tartrate. The glyoxalate-containing mixture was then treated with NaI and potassium phthalimide to give *N*,*O*-acetal **8z** in a net one-pot procedure.

To gain insight into the scope of amenable *N*-acyl phthalimides and enhance the operational simplicity of the reported methodology, the optimized conditions were modified so *N*,*O*-acetals could be obtained in a one-pot procedure (cf. Scheme 3). Specifically, adding an acid chloride to a mixture containing aldehyde, potassium phthalimide, and NaI gave the corresponding *N*,*O*-acetal. It was crucial that the acid chloride was the limiting reagent; otherwise, only *N*-acyl phthalimide and trace yields of product were obtained. Additionally, it is necessary to use NaI in excess because much of the soluble sodium cation is likely sequestered as insoluble NaCl. Monitoring the reaction by ^{1}H NMR indicated that *N*-acyl phthalimide (cf. **25**) is rapidly formed and accumulates in the reaction mixture before being converted into *N*,*O*-acetal.

Scheme 2. Aldehyde Scope[a]

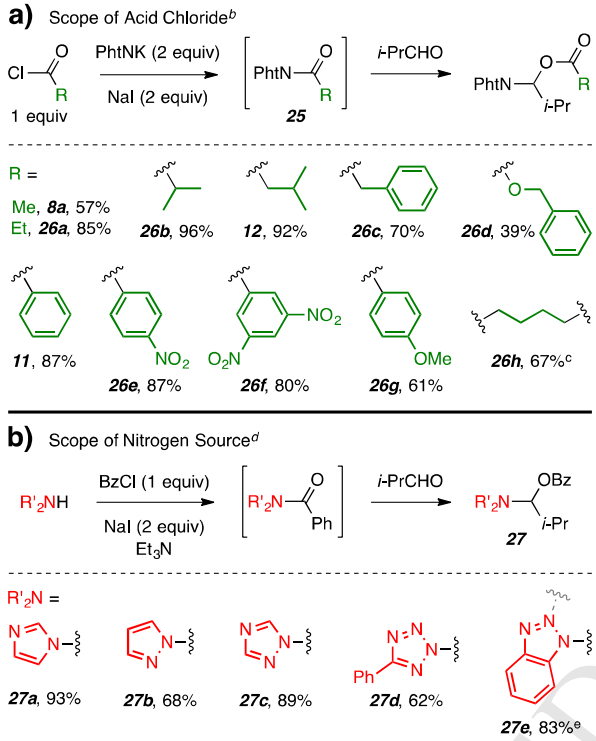


[a]Yields refer to isolated yields. [b]Product was prepared on a decagram scale. [c]Reaction was performed with ca. 4–4.5 equiv of aldehyde reactant. [d]Reaction was performed with 1 equiv of Et_3N. [e]Product was prepared on a gram scale. [f]Reaction was performed with 1 equiv of NaI and PhtNK. [g]The product was prepared by a two-step, one-pot procedure involving oxidative cleavage of ethyl glyoxalate prior to addition of **7**, NaI, and PhtNK (cf. Experimental Section).

The one-pot strategy was applied to a number of acid chlorides to give products **8a**, **11**, **12**, and **26a**–**h** in good yields (cf. Scheme 3a). Acid chlorides with a variety of steric hindrance gave high yields (cf. **8a, 12, 26a**–**c**). Use of benzyl chloroformate gave **26d**, indicating that *O*-acyloxy-*N*,*O*-acetals can also be formed under the one-pot protocol. Benzoyl chlorides attached to both electron withdrawing and donating groups readily gave products **11** and **26e**–**26g**. Additionally, bis-*N*,*O*-acetal **26h** was prepared using 0.5 equiv of adipoyl chloride. Collectively, these results demonstrate that the one-pot procedure is a convenient approach to preparing *N*,*O*-acetals without having to isolate the *N*-acyl phthalimide precursor. However, it should be noted that use of acetyl chloride under these conditions reproducibly gave a lower yield (i.e., 57%) compared with the protocol involving *N*-acyl phthalimide **7** (i.e., 79%). For this reason and because it is both bench stable[18] and readily prepared on a large scale (cf. Experimental Section), **7** is generally our substrate of choice for preparation of *O*-acetyl-*N*,*O*-acetals (cf. those in Scheme 2).

By replacing potassium phthalimide with a heteroaromatic amine and Et_3N, we were also able to demonstrate that other activated amides form *N,O*-acetals under one-pot conditions (cf. Scheme 3b). Specifically, *N,O*-acetals **27a–e** were prepared in one-pot from the corresponding amines. It is noteworthy that **27a** and **27c** were not stable to chromatographic purification under a number of attempted conditions. However, the one-pot procedure delivered the crude products in >95% purity.

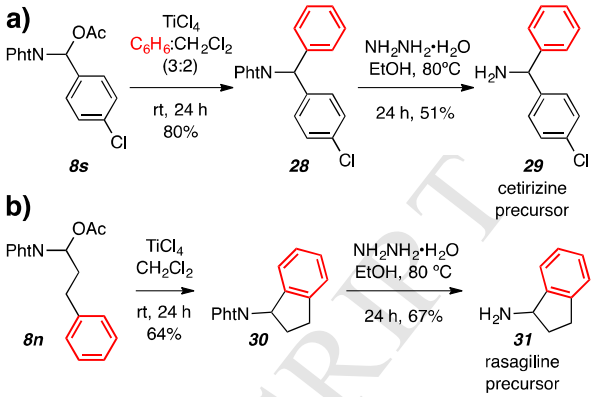
Scheme 3. Scope of Acid Chloride and Nitrogen Source in the One-Pot Synthesis of *N,O*-Acetals[a]



[a]Yields refer to isolated yields. [b]Reaction was performed by adding acid chloride (1 equiv) to a co-solvent mixture of MeCN and EtOAc (1:1) containing *i*-PrCHO, NaI, PhtNK, and 4Å MS at room temperature. [c]Reaction was performed with ca. 0.5 equiv of adipoyl chloride. [d]Reaction was performed by adding benzoyl chloride (1 equiv) to a co-solvent mixture of MeCN and EtOAc (1:1) containing *i*-PrCHO, NaI, amine (R'_2NH), Et_3N, and 4Å MS at room temperature prepared. [e]The product was isolated as a 3:2 mixture of N1 (black attachment) and N2 (grey attachment) regioisomers.

The *N*-phthalimido-*O*-acyl-*N,O*-acetals are themselves useful because they can be readily converted into pharmaceutical building blocks. For example, we found that treating *N,O*-acetal **8s** with benzene and $TiCl_4$ gave α-branched benzyl phthalimide **28** (cf. Scheme 4a). The reaction is Friedel–Crafts-like and presumably proceeds through a highly electrophilic phthalimidoyl iminium ion.[5c,8c] Subsequent phthalimide removal in the presence of hydrazine affords α-branched benzylic amine **29**, which is a known[19] precursor to cetirizine, the active ingredient in Zyrtec. Similarly, *N,O*-acetal **8n** was treated with $TiCl_4$ to induce an intramolecular Friedel–Crafts reaction to give **30**. In this case, subsequent phthalimide removal would form 1-aminoindan (**31**), a precursor to rasagiline, the active ingredient in Azilect.[20] These results demonstrate the potential utility of the *N,O*-acetals prepared under the reported conditions. Namely, using this approach, a number of α-branched aryl amines, a privileged functionality in medicinal chemistry, are readily prepared from inexpensive starting materials.

Scheme 4: Representative Synthetic Utility



3. Conclusion

Aldehydes are capable of reacting with *N*-acyl phthalimides and *N*-acyl azoles to give *N,O*-acetals by a net C–N bond insertion. Optimization studies indicated that the highest yields are achieved when sodium cation is included, which was superior to use of a number of other metal additives, including lithium and potassium salts. DFT computational analysis supported the proposed mechanistic hypothesis and was consistent with a double label crossover experiment. Additionally, the computations supported the optimization studies by indicating that the most favorable energy path to products occurs with a sodium counterion. A broad scope of aldehydes is capable of participating in the reaction, and a one-pot procedure was developed to further improve the operational simplicity of the procedure. The one-pot procedure was also used to expand the scope of products to include those derived from a variety of *N*-acyl phthalimides and nitrogen-containing heterocycles. The synthetic utility of the methodology was demonstrated by showing that *N,O*-acetals prepared under the reported conditions can be converted into known synthesis precursors to cetirizine and rasagiline.

4. Experimental Section

General Comments: Solvents were dried prior to use by distillation and/or storing over activated 3Å or 4Å molecular sieves. Reactions performed with less than 5 mmole of starting material were carried out in screw cap vials or culture tubes fitted with a Teflon®-lined cap. Reactions requiring 4Å molecular sieves (4Å MS) were performed using powdered 4Å MS that had been "activated" by heating in a muffle furnace at 300 ºC for at least 24 hours and stored in a desiccator.

General Procedure A: Synthesis of *N,O*-Acetals by Reaction of *N*-Acyl Phthalimides with Aldehydes. Aldehyde (2 equiv) was added to a mixture of *N*-acyl phthalimide (1 equiv), potassium phthalimide (20 mg, 0.11 mmol, 0.2 equiv), NaI (16 mg, 0.11 mmol, 0.2 equiv), 4Å MS (100 mg), MeCN (0.53 mL), and EtOAc (0.53 mL) at room temperature with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x), 1 M aq. NaOH (3x), and brine (1x). The organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by normal phase flash chromatography using a mixture of hexanes/EtOAc eluent.

General Procedure B: Synthesis of *N,O*-Acetals by Reaction of *N*-Acyl Phthalimides with Electron Rich Aldehydes. Aldehyde (2 equiv) was added to a mixture of *N*-acyl phthalimide

(1 equiv), potassium phthalimide (100 mg, 0.54 mmol, 1 equiv), NaI (80 mg, 0.53, 1 equiv), 4Å MS (100 mg), MeCN (0.53 mL), and EtOAc (0.53 mL) at room temperature with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x), 1 M aq. NaOH (3x), and brine (1x). The organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by normal phase gradient flash chromatography using a mixture of hexanes/EtOAc eluent.

General Procedure C: One-Pot Synthesis of *N,O*-Acetals from Potassium Phthalimide and an Acid Chloride. Acid chloride (1 equiv) was added to a mixture of isobutyraldehyde (110 µL, 1.2 mmol, 2.2 equiv), potassium phthalimide (200 mg, 1.1 mmol, 2 equiv), NaI (160 mg, 1.1 mmol, 2 equiv), 4Å MS (100 mg), MeCN (0.53 mL), and EtOAc (0.53 mL) at room temperature with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x), 1 M aq. NaOH (3x), and brine (1x). The organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by normal phase gradient flash chromatography using a mixture of hexanes/EtOAc eluent.

General Procedure D: One-Pot Synthesis of *N,O*-Acetals from an Amine and an Acid Chloride. Benzoyl chloride (1 equiv) was added to a mixture of isobutyraldehyde (100 µL, 1.1 mmol, 2 equiv), amine (1.5–2 equiv), NaI (160 mg, 1.1 mmol, 2 equiv), triethylamine (150 µL, 1.1 mmol, 1 equiv), 4Å MS (100 mg), MeCN (0.53 mL, 1 M), and EtOAc (0.53 mL, 1 M) at room temperature with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed 1 M aq. NaOH (3x) and brine (1x). The organic phase was dried ($MgSO_4$) and concentrated.

*2-Acetylisoindoline-1,3-dione (**7**).* Acetic anhydride (52.2 mL, 0.553 mol) was added to a mixture of potassium phthalimide (51.2 g, 0.276 mol) and acetonitrile (350 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in EtOAc and washed with sat. aq. $NaHCO_3$ and brine. The organic phase was dried ($MgSO_4$) and concentrated. The resulting solid was recrystallized from EtOAc to give known **7** as an off-white solid (45.6 g, 0.241 mol, 87%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.99 (nfom, 2H), 7.86 (nfom, 2H), and 2.69 (s, 3H). ^{1}H NMR data for **7** was in good agreement with those described previously.[21]

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl Acetate (**8a**).* Isobutyraldehyde (9.5 mL, 104 mmol) was added to a mixture of **7** (10.74 g, 56.8 mmol), potassium phthalimide (1.97 g, 10.6 mmol), NaI (1.64 g, 10.9 mmol), 4Å MS (1.12 g), acetonitrile (50 mL), and ethyl acetate (50 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The resulting organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by flash chromatography (SiO_2, hexanes:EtOAc 4:1) to give **8a** (11.73 g, 44.9 mmol, 79%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (nfom, 2H), 7.76 (nfom, 2H), 6.22 (d, *J* = 10.5 Hz, 1H), 2.82–2.99 (nfom, 1H), 2.10 (s, 3H), 1.08 (d, *J* = 6.7 Hz, 3H), and 0.89 (d, *J* = 6.8 Hz, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*One Pot Synthesis of 1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl Acetate (**8a**). N,O*-Acetal **8a** was prepared according to General Procedure C with acetyl chloride (38 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8a** (79 mg, 0.30 mmol, 57%). The ^{1}H NMR data matched that obtained using the aforementioned procedure (see above), and was in good agreement with previously reported characterization results.[8g]

*(1,3-Dioxoisoindolin-2-yl)(phenyl)methyl Acetate (**8b**). N,O*-Acetal **8b** was prepared according to General Procedure A from **7** (102 mg, 0.54 mmol) and benzaldehyde (108 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, hexanes:EtOAc 9:1 to 4:1) gave **8b** (119 mg, 0.40 mmol, 74%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85 (nfom, 2H), 7.73 (nfom, 2H), 7.69 (s, 1H), 7.53–7.59 (m, 2H), 7.33–7.43 (m, 3H), and 2.21 (s, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*(1,3-Dioxoisoindolin-2-yl)methyl Acetate (**8c**). N,O*-Acetal **7** was prepared according to General Procedure A from **8c** (91 mg, 0.48 mmol) and paraformaldehyde (72 mg). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8c** (40 mg, 0.18 mmol, 38%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (nfom, 2H), 7.79 (nfom, 2H), 5.72 (s, 2H), and 2.21 (s, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*1-(1,3-Dioxoisoindolin-2-yl)ethyl Acetate (**8d**). N,O*-Acetal **8d** was prepared according to General Procedure A from **7** (98 mg, 0.52 mmol) and 4 equivalents of acetaldehyde (0.1 mL, 2 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8d** (101 mg, 0.43 mmol, 83%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (nfom, 2H), 7.77 (nfom, 2H), 6.75 (q, *J* = 6.5 Hz, 1H), 2.08 (s, 3H), and 1.86 (d, *J* = 6.4 Hz, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*1-(1,3-Dioxoisoindolin-2-yl)butyl Acetate (**8e**). N,O*-Acetal **8e** was prepared according to General Procedure A from **7** (101 mg, 0.53 mmol) and butyraldehyde (96 µL, 1.1 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8e** (116 mg, 0.44 mmol, 83%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (nfom, 2H), 7.75 (nfom, 2H), 6.62 (t, *J* = 7.5 Hz, 1H), 2.11–2.37 (m, 2H), 2.08 (s, 3H), 1.30–1.49 (m, 2H), and 0.97 (t, *J* = 7.3 Hz, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*1-(1,3-Dioxoisoindolin-2-yl)heptyl Acetate (**8f**). N,O*-Acetal **8f** was prepared according to General Procedure A from **7** (101 mg, 0.53 mmol) and heptanal (151 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1) gave **8f** (118 mg, 0.39 mmol, 74%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (nfom, 2H), 7.77 (nfom, 2H), 6.61 (dd, *J* = 7.0, 8.1 Hz, 1H), 2.12–2.40 (m, 2H), 2.09 (s, 3H), 1.14–1.44 (m, 8H), and 0.86 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.6, 166.9, 134.5, 131.5, 123.8, 74.9, 31.6, 31.2, 28.6, 24.9, 22.5, 20.9, and 14.1. IR (neat): 2957, 2930, 2860, 1781, 1752, 1723, 1469, 1376, 1223, 1022, and 722 cm^{-1}. HRMS: Calcd for $C_{17}H_{21}NNaO_4^+$ [M+Na^+] requires 326.1363; found 326.1363.

*1-(1,3-Dioxoisoindolin-2-yl)-2,2-dimethylpropyl Acetate (**8g**). N,O*-Acetal **8g** was prepared according to General Procedure A from **7** (103 mg, 0.54 mmol) and trimethylacetaldehyde (116 µL, 1.1 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8g** (117 mg, 0.43 mmol, 80%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85 (nfom, 2H), 7.74 (nfom, 2H), 6.23 (s, 1H), 2.12 (s, 3H), and 1.10 (s, 9H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.0, 167.3, 134.3, 131.6, 123.6, 82.3, 36.7, 26.0, and 20.5. IR (neat): 2967, 2912, 2874, 1777, 1746, 1720, 1612, 1370, 1354, 1234, 1217, 1122, 1052, 1038, and 1024 cm^{-1}. HRMS: Calcd for $C_{15}H_{17}NNaO_4^+$ [M+Na^+] requires 298.1050; found 298.1053. MP: 87–89 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-3-methylbutyl Acetate (**8h**).* *N,O*-Acetal **8h** was prepared according to General Procedure A from **7** (103 mg, 0.54 mmol) and isovaleraldehyde (117 µL, 1.1 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8h** (115 mg, 0.42 mmol, 78%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (nfom, 2H), 7.77 (nfom, 2H), 6.71 (dd, *J* = 6.9, 8.1 Hz, 1H), 2.21–2.33 (nfom, 1H), 2.08 (s, 3H), 2.00–2.14 (nfom, 1H), 1.69–1.53 (nfom, 1H), 0.99 (d, *J* = 6.8 Hz, 3H), and 0.96 (d, *J* = 6.7 Hz, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*Cyclopropyl(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8i**).* *N,O*-Acetal **8i** was prepared according to General Procedure A from **7** (93 mg, 0.49 mmol) and cyclopropanecarboxaldehyde (73 µL, 0.98 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **8i** (87 mg, 0.34 mmol, 69%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.90 (nfom, 2H), 7.77 (nfom, 2H), 5.85 (d, *J* = 9.7 Hz, 1H), 2.11 (s, 3H), 2.08–2.22 (m, 1H), 0.53–0.82 (m, 3H), and 0.42 (nfom, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 166.7, 134.4, 131.6, 123.8, 78.5, 20.9, 13.0, 4.8, and 2.5. IR (neat): 3007, 1780, 1761, 1742, 1720, 1468, 1377, 1226, 1103, 1015, 975, and 717 cm^{-1}. HRMS: Calcd for $C_{14}H_{13}NNaO_4^+$ [M+Na$^+$] requires 282.0737; found 282.0738. MP: 126–131 ºC.

*Cyclohexyl(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8j**).* *N,O*-Acetal **8j** was prepared according to General Procedure A from **7** (102 mg, 0.54 mmol) and cyclohexanecarboxaldehyde (129 µL, 1.1 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **8j** (139 mg, 0.46 mmol, 85%) as white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (nfom, 2H), 7.76 (nfom, 2H), 6.32 (d, *J* = 10.5 Hz, 1H), 2.59 (qt, *J* = 11, 3.7 Hz, 1H), 2.10 (s, 3H), 1.87–2.02 (nfom, 1H), 1.59–1.85 (m, 3H), 1.40–1.54 (nfom, 1H), and 0.94–1.39 (m, 5H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.8, 166.9, 134.4, 131.4, 123.7, 78.1, 37.9, 29.2, 27.9, 25.9, 25.2, 25.1, and 20.7. IR (neat): 2930, 2854, 1780, 1758, 1721, 1363, 1222, 1022, 870, and 721 cm^{-1}. HRMS: Calcd for $C_{17}H_{19}NNaO_4^+$ [M+Na$^+$] requires 324.1206; found 324.1209. MP: 110–112 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-3,7-dimethyloct-6-en-1-yl Acetate (**8k**).* *N,O*-Acetal **8k** was prepared according to General Procedure A from **7** (103 mg, 0.54 mmol) and (±)-citronellal (194 µL, 1.1 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **8k** (141 mg, 0.41 mmol, 76%) as a mixture of two diastereomers that formed a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$, ~1:1 mixture of two diastereomers): δ 7.83–7.91 (m, 2H), 7.71–7.78 (m, 2H), 6.66–6.75 (m, 1H), 4.95–5.05 (m, 1H), 2.49 (ddd, *J* = 4.2, 9.2, 13.5 Hz, 1H, one diastereomer), 2.31 (nfom, 1H, one diastereomer), 2.07 (s, 3H), 1.83–2.1 (m, 4H), 1.59 (s, 3H, one diastereomer), 1.573 (s, 3H, one diastereomer), 1.568 (s, 3H, one diastereomer), 1.54 (s, 3H, one diastereomer), 1.14–1.70 (m, 2H), 0.97 (d, *J* = 6.4 Hz, 3H, one diastereomer), and 0.93 (d, *J* = 6.5 Hz, 3H, one diastereomer). ^{13}C NMR (75 MHz, $CDCl_3$, mixture of two diastereomers): δ 169.70, 169.66, 166.96, 166.92, 134.5, 131.69, 131.63, 124.36, 124.33, 123.8, 73.9, 73.5, 38.3, 37.8, 37.1, 36.3, 29.2, 28.9, 25.7, 25.3, 25.1, 21.01, 20.97, 19.4, and 17.7. IR (neat): 2963, 2929, 2876, 1781, 1753, 1723, 1468, 1376, 1224, 1020, 976, and 872 cm^{-1}. HRMS: Calcd for $C_{20}H_{25}NNaO_4^+$ [M+Na$^+$] requires 366.1676; found 366.1674.

*1-(1,3-Dioxoisoindolin-2-yl)-7-hydroxy-3,7-dimethyloctyl Acetate (**8l**).* *N,O*-Acetal **8l** was prepared according to General Procedure A from **7** (102 mg, 0.54 mmol) and (±)-7-hydroxycitronellal (190 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, hexanes:EtOAc 6:1 to 4:1 to 1:1) gave **8l** (129 mg, 0.36 mmol, 67%) colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$, ~1:1 mixture of diastereomers): δ 7.85–7.93 (m, 2H), 7.74–7.82 (m, 2H), 6.68–6.77 (m, 1H), 2.48–2.61 (m, 1H, one diastereomer), 2.25–2.38 (m, 1H, one diastereomer), 2.079 (s, 3H, one diastereomer), 2.075 (s, 3H, one diastereomer), 1.76–2.14 (m, 3H), 1.07–1.61 (m, 5H), 1.21 (s, 3H), 1.189 (s, 3H, one diastereomer), 1.182 (s, 3H, one diastereomer), 0.98 (d, *J* = 6.5 Hz, 3H, one diastereomer), and 0.96 (d, *J* = 6.6 Hz, 3H, one diastereomer). ^{13}C NMR (75 MHz, $CDCl_3$, mixture of diastereomers): δ 169.7, 169.6, 166.94, 166.86, 134.51, 134.48, 131.5, 123.78, 123.75, 73.8, 73.5, 70.93, 70.89, 44.0, 43.8, 38.2, 37.6, 37.4, 36.8, 29.33, 29.31, 29.2, 29.1, 21.34, 21.26, 20.94, 20.89, 19.38, and 19.32. IR (neat): 3487 (br), 2967, 2938, 1781, 1751, 1723, 1468, 1375, 1225, 1020, and 723 cm^{-1}. HRMS: Calcd for $C_{20}H_{27}NNaO_5^+$ [M+Na$^+$] requires 384.1781; found 384.1781.

*1-(1,3-Dioxoisoindolin-2-yl)-2-phenylethyl Acetate (**8m**).* Phenylacetaldehyde (125 µL, 1.1 mmol) was added to a mixture of **7** (101 mg, 0.53 mmol), triethylamine (74 µL, 0.53 mmol), potassium phthalimide (20 mg, 0.11 mmol), NaI (16 mg, 0.11 mmol), 4Å MS (100 mg), MeCN (0.53 mL), and EtOAc (0.53 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The resulting organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by gradient flash chromatography (SiO_2, hexanes:EtOAc 9:1 to 4:1) to give **8m** (83 mg, 0.27 mmol, 51%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.81(nfom, 2H), 7.69 (nfom, 2H), 7.14–7.29 (m, 5H), 6.83 (dd, *J* = 6.7, 8.7 Hz, 1H), 3.66 (dd, *J* = 8.7, 13.7 Hz, 1H), 3.52 (dd, *J* = 6.6, 13.8 Hz, 1H), and 2.05 (s, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*1-(1,3-Dioxoisoindolin-2-yl)-3-phenylpropyl Acetate (**8n**).* Hydrocinnamaldehyde (1.41 mL, 10.7 mmol) was added to a mixture of *N*-acetyl phthalimide (**7**, 1.10 g, 5.82 mmol), potassium phthalimide (0.21 g, 1.1 mmol), NaI (0.16 g, 1.1 mmol), 4Å MS (0.20 g), acetonitrile (5.3 mL), and ethyl acetate (5.3 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The resulting organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by gradient flash chromatography (pure hexanes to 6:1 hexanes:EtOAc to 4:1 hexanes:EtOAc) to give **8n** (1.47 g, 4.55 mmol, 78%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83 (nfom, 2H), 7.72 (nfom, 2H), 7.11–7.23 (m, 4H), 7.07 (nfom, 1H), 6.65 (br t, *J* = 6.4 Hz, 1H), 2.48–2.84 (m, 4H), and 2.06 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.5, 166.8, 139.9, 134.4, 131.4, 128.4, 128.3, 126.1, 123.6, 74.5, 32.4, 31.4, and 20.8. IR (neat): 3088, 3063, 3028, 2936, 2865, 1781, 1753, 1722, 1604, 1496, 1468, 1454, 1376, 1359, 1222, 1024, 721, and 700 cm^{-1}. HRMS: Calcd for $C_{19}H_{17}NNaO_4^+$ [M+Na$^+$] requires 346.1050; found 346.1053. MP: 80–82 ºC.

*(1,3-Dioxoisoindolin-2-yl)(p-tolyl)methyl Acetate (**8o**).* *N,O*-Acetal **8o** was prepared according to General Procedure A from **7** (99 mg, 0.52 mmol) and *p*-tolualdehyde (126 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, hexanes:EtOAc 9:1 to 4:1) gave **8o** (106 mg, 0.34 mmol, 65%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85 (nfom, 2H), 7.73 (nfom, 2H), 7.65 (s, 1H), 7.45 (d, *J* = 8.1 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 2.34 (s, 3H), and 2.20 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.4, 166.4, 138.9, 134.5, 132.2, 131.7, 129.2, 126.4, 123.8, 74.3, 21.3, and 20.9. IR (neat): 3031, 2953, 2924, 2865, 1780, 1760, 1747, 1727, 1613, 1517, 1468, 1376, 1356, 1332, 1223, 1031, 1020, 738, and 720 cm^{-1}. HRMS: Calcd for

$C_{18}H_{15}NNaO_4^+$ [M+Na$^+$] requires 332.0893; found 332.0869. MP: 132–137 ºC.

*(1,3-Dioxoisoindolin-2-yl)(2-nitrophenyl)methyl Acetate (**8p**).* *N,O*-Acetal **8p** was prepared according to General Procedure A from **7** (101 mg, 0.53 mmol) and 2-nitrobenzaldehyde (165 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8p** (161 mg, 0.47 mmol, 89%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.21 (s, 1H), 8.06 (dd, *J* = 1.1, 8.1 Hz, 1H), 7.98 (d, *J* = 7.9 Hz, 1H), 7.86 (nfom, 2H), 7.77 (nfom, 2H), 7.74 (dt, *J* = 1.5, 7.8 Hz, 1H), 7.52 (dt, *J* = 1.6, 7.9 Hz, 1H), and 2.20 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.6, 166.1, 147.2, 134.7, 133.2, 131.2, 130.1, 129.7, 129.6, 125.2, 123.9, 71.0, and 20.6. IR (neat): 1784, 1761, 1729, 1530, 1380, 1352, 1218, 1023, and 721 cm^{-1}. HRMS: Calcd for $C_{17}H_{12}N_2NaO_6^+$ [M+Na$^+$] requires 363.0588; found 363.0610. MP: 150–153 ºC.

*(1,3-Dioxoisoindolin-2-yl)(4-nitrophenyl)methyl Acetate (**8q**).* *N,O*-Acetal **8q** was prepared according to General Procedure A from **7** (99 mg, 0.52 mmol) and 4-nitrobenzaldehyde (161 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8q** (144 mg, 0.42 mmol, 81%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.25 (d, *J* = 8.9 Hz, 2H), 7.88 (nfom, 2H), 7.78 (nfom, 2H), 7.731 (d, *J* = 8.6 Hz, 2H), 7.729 (s, 1H), and 2.25 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.0, 166.1, 148.2, 142.0, 134.9, 131.4, 127.6, 124.1, 123.8, 72.9, and 20.8. IR (neat): 3108, 3080, 2849, 2777, 2732, 1781, 1765, 1709, 1680, 1526, 1382, 1347, 1324, 1199, 1109, 850, 815, 740, and 722 cm^{-1}. HRMS: Calcd for $C_{17}H_{12}N_2NaO_6^+$ [M+Na$^+$] requires 363.0588; found 363.0601. MP: 104–107 ºC.

*(2-Chlorophenyl)(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8r**).* *N,O*-Acetal **8r** was prepared according to General Procedure A from **7** (100 mg, 0.53 mmol) and 2-chlorobenzaldehyde (120 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8r** (114 mg, 0.35 mmol, 66%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.91 (br d, *J* = 8.1 Hz, 1H), 7.87 (s, 1H), 7.85 (nfom, 2H), 7.74 (nfom, 2H), 7.25–7.42 (m, 3H), and 2.22 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 168.7, 166.1, 134.6, 132.2, 131.4, 130.3, 129.7, 129.3, 126.5, 123.9, 71.7, and 20.8. IR (neat): 3072, 2962, 1781, 1766, 1749, 1728, 1459, 1445, 1379, 1351, 1333, 1217, 1023, and 722 cm^{-1}. HRMS: Calcd for $C_{17}H_{12}ClNNaO_4^+$ [M+Na$^+$] requires 352.0347; found 352.0350. MP: 137–142 ºC.

*(4-Chlorophenyl)(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8s**).* 4-Chlorobenzaldehyde (1.53 g, 11 mmol) was added to a mixture of *N*-acetyl phthalimide (**7**, 1.01 g, 5.34 mmol), potassium phthalimide (0.20 g, 1.1 mmol), NaI (0.16 g, 1.1 mmol), 4Å MS (0.20 g), acetonitrile (5.3 mL), and ethyl acetate (5.3 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The resulting organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) to give **8s** (1.37 g, 4.15 mmol, 78%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.87 (nfom, 2H), 7.76 (nfom, 2H), 7.64 (s, 1H), 7.51 (br d, *J* = 8.5 Hz, 2H), 7.36 (br d, *J* = 8.5 Hz, 2H), and 2.21 (s, 3H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[8g]

*(4-Bromophenyl)(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8t**).* *N,O*-Acetal **8t** was prepared according to General Procedure A from **7** (104 mg, 0.55 mmol) and 4-bromobenzaldehyde (198 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8t** (175 mg, 0.47 mmol, 85%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.87 (nfom, 2H), 7.75 (nfom, 2H), 7.62 (s, 1H), 7.52 (d, *J* = 8.6 Hz, 2H), 7.44 (d, *J* = 8.6 Hz, 2H), and 2.21 (s, 3H) as a white solid. ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.2, 166.3, 134.7, 134.2, 131.8, 131.6, 128.3, 124.0, 123.2, 73.7, and 20.9. IR (neat): 3062, 1781, 1763, 1746, 1727, 1377, 1356, 1332, 1219, 1030, 1012, 740, and 722 cm^{-1}. HRMS: Calcd for $C_{17}H_{12}BrNNaO_4^+$ [M(^{79}Br)+Na$^+$] requires 395.9842; found 395.9847. MP: 172–174 ºC.

*(1,3-Dioxoisoindolin-2-yl)(pyridin-3-yl)methyl Acetate (**8u**).* *N,O*-Acetal **8u** was prepared according to General Procedure A from **7** (102 mg, 0.54 mmol) and 3-pyridinecarboxaldehyde (100 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, CH_2Cl_2:EtOH 98:2 for ca. six column volumes then hexanes:EtOAc:Et_3N 50:45:5 for ca. 10 column volumes) gave **8u** (113 mg, 0.38 mmol, 70%) as an oily solid. ^{1}H NMR (300 MHz, $CDCl_3$, referenced to TMS): δ 8.79 (d, *J* = 1.8 Hz, 1H), 8.61 (dd, *J* = 1.4, 4.8 Hz, 1H), 7.97 (br d, *J* = 8.0 Hz, 1H), 7.88 (nfom, 2H), 7.77 (nfom, 2H), 7.72 (s, 1H), 7.36 (dd, *J* = 4.6, 8.0 Hz, 1H), and 2.23 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.0, 166.1, 150.1, 147.9, 134.7, 134.5, 131.3, 131.0, 123.9, 123.2, 72.3, and 20.7. IR (neat): 3188, 3060, 2960, 2920, 2850, 1774, 1732, 1730, 1606, 1469, 1309, and 1054 cm^{-1}. HRMS: Calcd for $C_{16}H_{13}N_2O_4^+$ [M+H$^+$] requires 297.0870; found 297.0869.

*(1,3-Dioxoisoindolin-2-yl)(furan-2-yl)methyl Acetate (**8v**).* *N,O*-Acetal **8v** was prepared according to General Procedure A from **7** (104 mg, 0.55 mmol) and 2-furaldehyde (88 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8v** (86 mg, 0.30 mmol, 55%) as a golden yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.91 (nfom, 2H), 7.77 (nfom, 2H), 7.70 (s, 1H), 7.40 (dd, *J* = 0.9, 2.0 Hz, 1H), 6.58 (br d, *J* = 3.4 Hz, 1H), 6.41 (dd, *J* = 1.9, 3.5 Hz, 1H), and 2.18 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.1, 166.1, 147.4, 143.1, 134.7, 131.7, 124.1, 110.8, 109.6, 68.8, and 20.8. IR (neat): 3150, 3129, 2944, 1783, 1760, 1729, 1611, 1504, 1469, 1376, 1357, 1335, 1216, 1031, 1015, and 717 cm^{-1}. HRMS: Calcd for $C_{15}H_{11}NNaO_5^+$ [M+Na$^+$] requires 308.0529; found 308.0533. MP: 116–120 ºC.

*(1,3-Dioxoisoindolin-2-yl)(4-methoxyphenyl)methyl Acetate (**8w**).* *N,O*-Acetal **8w** was prepared according to General Procedure B from **7** (93 mg, 0.49 mmol) and 4-methoxybenzaldehyde (131 µL, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8w** (69 mg, 0.21 mmol, 43%) a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85 (nfom, 2H), 7.73 (nfom, 2H), 7.62 (s, 1H), 7.52 (d, *J* = 8.8 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 3.79 (s, 3H), and 2.20 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.3, 166.4, 160.0, 134.5, 131.6, 128.0, 127.2, 123.8, 113.8, 74.3, 55.4, and 20.9. IR (neat): 2961, 2937, 1749, 1726, 1614, 1516, 1376, 1362, 1354, 1253, 1222, 1031, and 734 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{18}H_{15}NNaO_5^+$ [M+Na$^+$] requires 348.0842; found 348.0859. MP: 84–89 ºC.

*(3,4-Dimethoxyphenyl)(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8x**).* *N,O*-Acetal **8x** was prepared according to General Procedure B from **7** (100 mg, 0.53 mmol) and 3,4-dimethoxybenzaldehyde (176 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to 4:1) gave **8x** (62 mg, 0.17 mmol, 32%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86 (nfom, 2H), 7.74 (nfom, 2H), 7.59 (s, 1H), 7.14–7.20 (m 2H), 6.86 (d, *J* = 8.0 Hz, 1H), 3.91 (s, 3H), 3.87 (s, 3H), and 2.20 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.4, 166.5, 149.6, 149.0, 134.6, 131.7, 127.6, 123.9, 119.5, 110.8, 110.1, 74.7, 56.1, 56.0, and 21.0. IR (neat): 3005, 2960, 2939, 2839, 1780, 1756, 1723, 1519, 1375, 1356, 1331, 1266,

1220 1161, 1144, 1119, 1027, 916, 737, and 718 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{19}H_{17}NNaO_6^+$ [M+Na^+] requires 378.0948; found 378.0947.

*(1,3-Dioxoisoindolin-2-yl)(1-(2-methylallyl)-1H-indol-2-yl)methyl Acetate (**8y**).* *N*,*O*-Acetal **7** was prepared according to General Procedure B from **7** (100 mg, 0.53 mmol) and 1-(2-methylallyl)-1*H*-indole-2-carbaldehyde[22] (213 mg, 1.1 mmol). Purification by gradient flash chromatography (SiO_2, hexanes:EtOAc 6:1 to 4:1 to 2:1) gave **8y** (84 mg, 0.22 mmol, 42%) as a pale yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86 (s, 1H), 7.84 (nfom, 2H), 7.73 (nfom, 2H), 7.64 (d, *J* = 7.8 Hz, 1H), 7.15–7.21 (m, *J* = 4.0 Hz, 2H), 7.07–7.15 (m, 1H), 6.97 (s, 1H), 4.65 (s, 2H), 4.44 (s, 1H), 4.02 (s, 1H), 2.21 (s, 3H), and 1.64 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.1, 166.1, 140.6, 137.7, 134.6, 132.3, 131.6, 127.0, 123.8, 122.7, 121.4, 120.2, 110.8, 109.7, 103.5, 68.6, 49.1, 20.9, and 19.9. IR (neat): 3060, 2940, 1780, 1759, 1725, 1462, 1373, 1217, 1034, and 715 cm^{-1}. HRMS: Calcd for $C_{23}H_{20}N_2NaO_4^+$ [M+Na^+] requires 411.1315; found 411.1320. MP: 57–61 ºC.

*(3,4-Dimethoxyphenyl)(1,3-dioxoisoindolin-2-yl)methyl Acetate (**8z**).* Phenyliodonium diacetate (PIDA, 240 mg, 0.75 mmol) was added to a mixture of (+)-diethyl l-tartate (110 µL, 0.63 mmol) and CH_2Cl_2 (1.1 mL) at room temperature and with stirring. After 1 h, *N*-acyl phthalimide **7** (100 mg, 0.53 mmol), NaI (16 mg, 0.11 mmol), potassium phthalimide (20 mg, 0.11), and triethylamine (150 µL, 1.1 mmol) were sequentially added. After stirring overnight, the mixture was diluted in EtOAc and sequentially washed with sat. aq. NH_4Cl (1x), 1 M aq. NaOH (3x), and brine. The organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by gradient flash chromatography (SiO_2, pure hexanes to hexanes:EtOAc 6:1 to hexanes:EtOAc 4:1) to give **8z** (62 mg, 0.21 mmol, 40%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.95 (nfom, 2H), 7.73 (nfom, 2H), 6.98 (s, 1H), 4.31 (q, *J* = 7.1 Hz, 2H), 2.19 (s, 3H), and 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 169.2, 166.0, 164.7, 134.7, 131.6, 124.3, 68.6, 63.2, 20.7, and 14.1. IR (neat): 2986, 1788, 1757, 1733, 1387, 1374, 1216, 1057, and 718 cm^{-1}. HRMS: Calcd for $C_{14}H_{13}NNaO_6^+$ [M+Na^+] requires 314.0635; found 314.0638. MP: 115–121 ºC.

*2-Benzoylisoindoline-1,3-dione (N-Benzoyl Phthalimide, **9**).* Benzoyl chloride (7.8 mL, 67 mmol) was added to a mixture of potassium phthalimide (5.0 g, 27 mmol), 4-dimethylaminopyridine (3.7 g, 30 mmol), and acetonitrile (34 mL) at room temperature and with stirring. After 24 h, the mixture was gravity filtered and the solid residue was recrystallized from a mixture of hexanes and EtOAc to give known **9** as a white solid (4.51 g, 18.0 mmol, 67%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.00 (nfom, 2H), 7.84–7.91 (m, 4H), 7.64–7.71 (m, 1H), and 7.48–7.55 (m, 2H). ^{1}H NMR data for **9** was in good agreement with those described previously.[23]

*5-Methyl-2-(3-methylbutanoyl)isoindoline-1,3-dione (**10**).* Isovaleryl chloride (0.50 mL, 4.1 mmol) was added to a mixture of 4-methylphthalimide (0.78 g, 4.8 mmol), triethylamine (0.86 mL, 6.2 mmol), and acetonitrile (8.2 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in EtOAc and washed with sat. aq. $NaHCO_3$ and brine. The organic phase was dried ($MgSO_4$) and concentrated. The crude material was purified by flash chromatography (SiO_2, hexanes:EtOAc 4:1) to give **10** (713 mg, 2.91 mmol, 71%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.83 (d, *J* = 7.7 Hz, 1H), 7.74 (s, 1H), 7.65 (d, *J* = 7.6 Hz, 1H), 2.90 (d, *J* = 6.9 Hz, 2H), 2.57 (s, 3H), 2.25 (septet, *J* = 6.7 Hz, 1H), and 1.03 (d, *J* = 6.7 Hz, 6H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.6, 165.6, 165.5, 147.1, 136.2, 131.4, 128.5, 124.6, 124.3, 47.4, 25.0, 22.4, and 22.1. IR (neat): 2956, 1750, 1711, 1612, 1364, 1309, 1109, 1047, and 738 cm^{-1}. HRMS: Calcd for $C_{14}H_{15}NNaO_3^+$ [M+Na^+] requires 268.0944; found 268.0945. MP: 180–186 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl Benzoate (**11**).* *N*,*O*-Acetal **11** was prepared according to General Procedure C with benzoyl chloride (62 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **11** (149 mg, 0.46 mmol, 87%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.1 (br d, *J* = 7.5 Hz, 2H), 7.88 (nfom, 2H), 7.74 (nfom, 2H), 7.57 (br t, *J* = 7.5 Hz, 1H), 7.43 (br t, *J* = 7.8 Hz, 2H), 6.48 (d, *J* = 10.4 Hz, 1H), 3.01–3.19 (m, 1H), 1.16 (d, *J* = 6.6 Hz, 3H), and 0.97 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.9, 165.3, 134.4, 133.5, 131.5, 130.0, 129.3, 128.5, 123.8, 79.8, 29.7, 19.1, and 18.0. IR (neat): 3064, 2970, 2877, 1781, 1733, 1601, 1469, 1452, 1371, 1252, 1090, 1064, 994, and 710 cm^{-1}. HRMS: Calcd for $C_{19}H_{17}NNaO_4^+$ [M+Na^+] requires 346.1050; found 346.1051. MP: 95–103 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl 3-Methylbutanoate (**12**).* *N*,*O*-Acetal **12** was prepared according to General Procedure C with isovaleryl chloride (65 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **12** (147 mg, 0.49 mmol, 92%) as a clear oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (nfom, 2H), 7.75 (nfom, 2H), 6.20 (d, *J* = 10.4 Hz, 1H), 2.80–2.99 (m, 1H), 2.23 (br d, *J* = 6.5 Hz, 2H), 2.00–2.17 (m, 1H), 1.08 (d, *J* = 6.6 Hz, 3H), 0.912 (d, *J* = 6.6 Hz, 3H), 0.907 (d, *J* = 6.6 Hz, 3H), and 0.89 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 172.0, 167.0, 134.5, 131.5, 123.8, 79.3, 43.1, 29.5, 25.7, 22.4 (2xC), 19.1, and 18.0. IR (neat): 2964, 2931, 2875, 1783, 1749, 1724, 1469, 1372, 1164, 1087, 1015, and 721 cm^{-1}. HRMS: Calcd for $C_{17}H_{21}NaNO_4^+$ [M+Na^+] requires 326.1363; found 326.1366.

*2-Methyl-1-(5-methyl-1,3-dioxoisoindolin-2-yl)propyl 3-Methylbutanoate (**13**).* Isolation and analytic data for **13**, observed in the crossover study, was obtained by independent preparation using the following procedure. Isovaleryl chloride (65 µL, 0.53 mmol) was added to a mixture of 4-methylphthalimide (200 mg, 1.2 mmol), isobutyraldehyde (108 µL, 1.19 mmol), NaI (170 mg, 1.1 mmol), 4Å MS (100 mg), triethylamine (150 µL, 1.1 mmol), ethyl acetate (1.1 mL), and acetonitrile (1.1 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in EtOAc and washed with 1M NaOH (3x) and brine. The organic phase was dried ($MgSO_4$) and concentrated. Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **13** (105 mg, 0.33 mmol, 62%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.6 Hz, 1H), 7.68 (s, 1H), 7.55 (d, *J* = 7.6 Hz, 1H), 6.19 (d, *J* = 10.4 Hz, 1H), 2.90 (nfom, 1H), 2.52 (s, 3H), 2.23 (d, *J* = 7.3 Hz, 2H), 2.09 (septet, *J* = 6.7 Hz, 1H), 1.08 (d, *J* = 6.6 Hz, 3H), 0.91 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 7 Hz, 3H), and 0.88 (d, *J* = 7.6 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 171.9, 167.1, 166.9, 145.8, 135.0, 131.8, 128.9, 124.2, 123.6, 79.1, 43.1, 29.5, 25.6, 22.3, 22.1, 19.0, and 17.9. IR (neat): 2963, 2875, 1779, 1748, 1721, 1618, 1468, 1430, 1363, 1101, and 1006 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{18}H_{23}NNaO_4^+$ [M+Na^+] requires 340.1519; found 340.1522. MP: 53–55 ºC.

*2-Methyl-1-(5-methyl-1,3-dioxoisoindolin-2-yl)propyl Benzoate (**14**).* Isolation and analytic data for **14**, observed in the crossover study, was obtained by independent preparation using the following procedure. Benzoyl chloride (62 µL, 0.54 mmol) was added to a mixture of 4-methylphthalimide (200 mg, 1.2 mmol), isobutyraldehyde (108 µL, 1.19 mmol), NaI (160 mg, 1.1 mmol), 4Å MS (100 mg), triethylamine (150 µL, 1.1 mmol), ethyl acetate (1.1 mL), and acetonitrile (1.1 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in EtOAc

and washed with 1M NaOH (3x) and brine. The organic phase was dried ($MgSO_4$) and concentrated. Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **14** (145 mg, 0.43 mmol, 80%) as a yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.09 (d, *J* = 7.7 Hz, 2H), 7.75 (d, *J* = 7.6 Hz, 1H), 7.67 (s, 1H), 7.50–7.60 (m, 2H), 7.43 (dd, *J* = 7.6, 7.8 Hz, 2H), 6.46 (d, *J* = 10.4 Hz, 1H), 3.09 (nfom, 1H), 2.50 (s, 3H), 1.15 (d, *J* = 6.7 Hz, 3H), and 0.96 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.1, 167.0, 165.3, 145.8, 135.0, 133.5, 131.9, 130.0, 129.4, 128.9, 128.5, 124.2, 123.7, 79.7, 29.8, 22.1, 19.1, and 18.0. IR (neat): 2965, 2938, 2874, 1780, 1748, 1720, 1469, 1373, 1359, 1223, and 720 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{20}H_{19}NNaO_4^+$ [M+Na$^+$] requires 360.1206; found 360.1223.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl Propionate (**26a**).* *N,O*-Acetal **26a** was prepared according to General Procedure C with propionyl chloride (48 µL, 0.55 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **26a** (129 mg, 0.47 mmol, 85%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (nfom, 2H), 7.76 (nfom, 2H), 6.23 (d, *J* = 10.4 Hz, 1H), 2.82–3.00 (m, 1H), 2.28–2.49 (m, 2H), 1.12 (t, *J* = 7.5 Hz, 3H), 1.09 (d, *J* = 6.7 Hz, 3H), and 0.89 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 173.3, 169.9, 134.4, 131.5, 123.8, 79.3, 29.6, 27.3, 19.0, 17.9, and 8.9. IR (neat): 2981, 2970, 2939, 2880, 1776, 1755, 1719, 1472, 1391, 1373, 1356, 1333, 1199, 1168, 1083, 874, and 638 cm^{-1}. HRMS: Calcd for $C_{15}H_{17}NNaO_4^+$ [M+Na$^+$] requires 298.1050; found 298.1056. MP: 122–125 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl Isobutyrate (**26b**).* *N,O*-Acetal **26b** was prepared according to General Procedure C with isobutyryl chloride (56 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **26b** (148 mg, 0.51 mmol, 96%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.89 (nfom, 2H), 7.77 (nfom, 2H), 6.19 (d, *J* = 10.4 Hz, 1H), 2.83–3.02 (m, 1H), 2.61 (septet, *J* = 7.0 Hz, 1H), 1.19 (d, *J* = 7.0 Hz, 3H), 1.14 (d, *J* = 7.0 Hz, 3H), 1.10 (d, *J* = 6.7 Hz, 3H), and 0.90 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 175.9, 166.9, 134.4, 131.5, 123.7, 79.3, 33.9, 29.6, 18.94, 18.89, 18.76, and 17.9. IR (neat): 2973, 2940, 2878, 1780, 1749, 1722, 1469, 1387, 1372, 1332, 1146, and 720 cm^{-1}. HRMS: Calcd for $C_{16}H_{19}NNaO_4^+$ [M+Na$^+$] requires 312.1206; found 312.1203. MP: 87–90 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl 2-Phenylacetate (**26c**).* *N,O*-Acetal **26c** was prepared according to General Procedure C with phenylacetyl chloride (71 µL, 0.54 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **26c** (127 mg, 0.38 mmol, 70%) as a clear oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.86 (nfom, 2H), 7.74 (nfom, 2H), 7.18–7.31 (m, 5H), 6.19 (d, *J* = 10.4 Hz, 1H), 3.68 (d, *J* = 15.1 Hz, 1H), 3.62 (d, *J* = 15.2 Hz, 1H), 2.81–2.99 (m, 1H), 1.00 (d, *J* = 6.6 Hz, 3H), and 0.87 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 170.6, 166.9, 134.5, 133.4, 131.5, 129.5, 128.6, 127.3, 123.8, 79.8, 41.0, 29.6, 18.9, and 17.9. IR (neat): 2968, 1781, 1745, 1721, 1372, 1241, 1123, 1003, and 719 cm^{-1}. HRMS: Calcd for $C_{20}H_{19}NNaO_4^+$ [M+Na$^+$] requires 360.1206; found 360.1207.

*Benzyl (1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl) Carbonate (**26d**).* *N,O*-Acetal **26d** was prepared according to General Procedure C with benzyl chloroformate (77 µL, 0.54 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **26d** (73 mg, 0.21 mmol, 39%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.88 (nfom, 2H), 7.76 (nfom, 2H), 7.28–7.37 (m, 5H), 6.12 (d, *J* = 10.5 Hz, 1H), 5.19 (d, *J* = 12.1 Hz, 1H), 5.08 (d, *J* = 12.1 Hz, 1H), 2.89–3.07 (m, 1H), 1.12 (d, *J* = 6.6 Hz, 3H), and 0.88 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.8, 154.2, 134.8, 134.6, 131.5, 128.7, 128.6, 128.5, 123.9, 83.0, 70.1, 29.5, 19.0, and 17.9. IR (neat): 2969, 1784, 1757, 1723, 1469, 1457, 1387, 1368, 1243, and 950 cm^{-1}. HRMS: Calcd for $C_{20}H_{19}NNaO_5^+$ [M+Na$^+$] requires 376.1155; found 376.1156.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl 4-Nitrobenzoate (**26e**).* *N,O*-Acetal **26e** was prepared according to General Procedure C with 4-nitrobenzoyl chloride (100 mg, 0.54 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **26e** (172 mg, 0.47 mmol, 87%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.23–8.32 (m, 4H), 7.90 (nfom, 2H), 7.81 (nfom, 2H), 6.50 (d, *J* = 10.4 Hz, 1H), 3.03–3.21 (m, 1H), 1.17 (d, *J* = 6.6 Hz, 3H), and 0.99 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.8, 163.5, 150.8, 134.72, 134.66, 131.4, 131.2, 123.9, 123.7, 80.5, 29.7, 19.1, and 17.9. IR (neat): 3110, 3080, 3060, 2971, 2940, 2880, 1780, 1738, 1721, 1609, 1529, 1469, 1387, 1371, 1350, 1263, 1095, 938, and 718 cm^{-1}. HRMS: Calcd for $C_{19}H_{16}N_2NaO_6^+$ [M+Na$^+$] requires 391.0901; found 391.0898. MP: 137–139 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl 3,5-Dinitrobenzoate (**26f**).* 3,5-Dinitrobenzoyl chloride (127 mg, 0.55 mmol) was added to a mixture of isobutyraldehyde (110 µL, 1.2 mmol), potassium phthalimide (200 mg, 1.1 mmol), NaI (160 mg, 1.1 mmol), 4Å MS (100 mg), MeCN (1 mL), and EtOAc (1 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The organic phase was dried ($MgSO_4$) and concentrated. Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **26f** (181 mg, 0.44 mmol, 80%) as a colorless solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 9.24 (t, *J* = 2.2 Hz, 1H), 9.18 (d, *J* = 2.2 Hz, 2H), 7.91 (nfom, 2H), 7.80 (nfom, 2H), 6.52 (d, *J* = 10.4 Hz, 1H), 3.09–3.27 (m, 1H), 1.20 (d, *J* = 6.6 Hz, 3H), and 1.00 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 166.7, 161.6, 148.8, 134.8, 133.2, 131.3, 129.8, 124.1, 123,0, 81.5, 29.7, 19.2, and 17.9. IR (neat): 3098, 2970, 2880, 1738, 1743, 1724, 1545, 1371, 1345, 1265, 1157, 1080, and 718 cm^{-1}. HRMS: Calcd for $C_{19}H_{15}N_3NaO_8^+$ [M+Na$^+$] requires 436.0751; found 436.0758. MP: 154–156 ºC.

*1-(1,3-Dioxoisoindolin-2-yl)-2-methylpropyl 4-Methoxybenzoate (**26g**).* *N,O*-Acetal **26g** was prepared according to General Procedure C with 4-methoxybenzoyl chloride (98 mg, 0.57 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 4:1) gave **26g** (123 mg, 0.35 mmol, 61%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.04 (d, *J* = 8.8 Hz, 2H), 7.87 (nfom, 2H), 7.73 (nfom, 2H), 6.91 (d, *J* = 8.8 Hz, 2H), 6.44 (d, *J* = 10.3 Hz, 1H), 3.85 (s, 3H), 2.97–3.16 (m, 1H), 1.15 (d, *J* = 6.6 Hz, 3H), and 0.96 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.1, 165.1, 163.8, 134.4, 132.2, 131.6, 123.8, 121.7, 113.8, 79.6, 55.6, 29.8, 19.1, and 18.0. IR (neat): 2968, 2940, 2880, 2840, 1780, 1731, 1606, 1512, 1372, 1257, 1168, 1088, and 720 cm^{-1}. HRMS: Calcd for $C_{20}H_{19}NNaO_5^+$ [M+Na$^+$] requires 376.1155; found 376.1163. MP: 101–103 ºC.

*Bis(1-(1,3-dioxoisoindolin-2-yl)-2-methylpropyl) Adipate (**26h**).* Adipoyl chloride (39 µL, 0.27 mmol, 1 equiv) was added to a mixture of isobutyraldehyde (110 µL, 1.2 mmol, 4.4 equiv), potassium phthalimide (200 mg, 1.1 mmol, 4 equiv), NaI (160 mg, 1.1 mmol, 4 equiv), 4Å MS (100 mg), MeCN (1 mL), and EtOAc (1 mL) at room temperature and with stirring. After 24 h, the mixture was diluted in Et_2O, and sequentially washed with sat. aq. NH_4Cl (1x) and 1 M aq. NaOH (3x). The organic phase was dried ($MgSO_4$) and concentrated. Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **26h** (99 mg, 0.18 mmol, 67%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$, ~1:1 mixture of two diastereomers): δ 7.83–7.91 (m, 4H), 7.72–7.79 (m, 4H), 6.181 (d, *J* = 10.5 Hz, 2H, one diastereomer), 6.178 (d, *J* = 10.5 Hz, 2H, one diastereomer), 2.77–2.96 (m, 2H), 2.27–

2.41 (m, 4H), 1.52–1.66 (m, 4H), 1.056 (d, *J* = 6.7 Hz, 3H), 1.051 (d, *J* = 6.6 Hz, 3H), 0.875 (d, *J* = 6.8 Hz, 3H), and 0.869 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR [75 MHz, $CDCl_3$, ~1:1 mixture of two diastereomers (diastereomeric carbons shown with *)]: δ 172.1, 167.0, 134.5, 131.5, 123.8, 79.3, 33.46*, 33.44*, 29.5, 24.00*, 23.98*, 19.0, and 17.9. IR (neat): 2967, 2940, 2880, 1780, 1748, 1721, 1469, 1380, 1372, 1125, 1015, and 720 cm^{-1}. HRMS: Calcd for $C_{30}H_{32}N_2NaO_8^+$ [M+Na$^+$] requires 571.2051; found 571.2050. MP: 156–159 ºC.

*1-(1H-Imidazol-1-yl)-2-methylpropyl Benzoate (**27a**).* *N,O*-Acetal **27a** was prepared according to General Procedure D with imidazole (66 mg, 0.97 mmol) and benzoyl chloride (62 µL, 0.53 mmol). The product was found to not be stable under standard flash chromatography conditions, however the crude product was obtained in high purity and gave **27a** (120 mg, 0.49 mmol, 93%) as a clear oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.03 (br d, *J* = 7.7 Hz, 2H), 7.78 (s, 1H), 7.59 (br t, *J* = 7.5 Hz, 1H), 7.45 (br t, *J* = 7.7 Hz, 2H), 7.15 (s, 1H), 7.09 (s, 1H), 6.40 (d, *J* = 9.4 Hz, 1H), 2.43–2.62 (m, 1H), 1.15 (d, *J* = 6.6 Hz, 3H), and 0.88 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.1, 136.9, 133.7, 129.83, 129.78, 128.9, 128.6, 116.7, 83.8, 33.0, 18.5, and 17.7. IR (neat): 3113, 3068, 3035, 2970, 2934, 2879, 1728, 1601, 1584, 1493, 1263, 1091, 1068, 1025, 989, 712, and 663 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{14}H_{17}N_2O_2^+$ [M+H$^+$] requires 245.1285; found 245.1288.

*2-Methyl-1-(1H-pyrazol-1-yl)propyl Benzoate (**27b**).* *N,O*-Acetal **27b** was prepared according to General Procedure D with pyrazole (69 mg, 1.0 mmol) and benzoyl chloride (62 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **27b** (118 mg, 0.36 mmol, 68%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.07 (br d, *J* = 8.2 Hz, 2H), 7.70 (br d, *J* = 2.3 Hz, 1H), 7.60 (br s, 1H), 7.56 (br t, *J* = 7.8 Hz, 1H), 7.42 (br t, *J* = 8.2 Hz, 2H), 6.49 (d, *J* = 9.5 Hz, 1H), 6.24–6.28 (m, 1H), 2.81–2.99 (m, 1H), 1.15 (d, *J* = 7.3 Hz, 3H), and 0.83 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.6, 140.8, 133.6, 130.6, 130.0, 129.2, 128.5, 105.7, 87.2, 32.1, 18.5, and 17.8. IR (neat): 3107, 3064, 3035, 2971, 2935, 2878, 1725, 1452, 1399, 1299, 1091, 987, and 711 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{14}H_{17}N_2O_2^+$ [M+H$^+$] requires 245.1285; found 245.1297.

*2-Methyl-1-(1H-1,2,4-triazol-1-yl)propyl Benzoate (**27c**).* *N,O*-Acetal **22c** was prepared according to General Procedure D with 1,2,4-triazole (71 mg, 1.0 mmol) and benzoyl chloride (62 µL, 0.53 mmol). The product was found to not be stable under standard flash chromatography conditions, however the crude product was obtained in high purity and gave **27c** (115 mg, 0.47 mmol, 89%) as a colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.42 (s, 1H), 8.07 (br d, *J* = 8.1 Hz, 2H), 8.01 (s, 1H), 7.59 (br t, *J* = 7.7 Hz, 1H), 7.45 (br t, *J* = 7.6 Hz, 2H), 6.57 (d, *J* = 9.2 Hz, 1H), 2.82–2.99 (m, 1H), 1.18 (d, *J* = 6.7 Hz, 3H), and 0.87 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.2, 152.4, 133.7, 129.9 (3xC), 128.54, 128.50, 84.7, 31.4, 18.1, and 17.7. IR (neat): 3123, 3066, 2972, 2936, 2879, 1728, 1508, 1261, 1091, and 711 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{13}H_{16}N_3O_2^+$ [M+H$^+$] requires 246.1237; found 246.1252.

*2-Methyl-1-(5-phenyl-2H-tetrazol-2-yl)propyl Benzoate (**27d**).* *N,O*-Acetal **27d** was prepared according to General Procedure D with 5-phenyl-1*H*-tetrazole (121 mg, 0.83 mmol) and benzoyl chloride (62 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **27d** (105 mg, 0.33 mmol, 62%) as a colorless oil. The product structure is inferred from a related reaction where ethyl chloroformate was used in place of benzoyl chloride to give known[8i] *N,O*-acetal **32** [cf. synthesis of ethyl (2-methyl-1-(5-phenyl-2*H*-tetrazol-2-yl)propyl) carbonate (**32**) described below]. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.16–8.22 (m, 2H), 8.11 (br d, *J* = 8.2 Hz, 2H), 7.60 (br t, *J* = 7.1 Hz, 1H), 7.41–7.53 (m, 5H), 7.20 (d, *J* = 8.7 Hz, 1H), 2.85–3.02 (m, 1H), 1.22 (d, *J* = 6.7 Hz, 3H), and 0.96 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.3, 164.7, 134.1, 130.6, 130.2, 129.0, 128.7, 128.6, 128.4, 127.1, 87.4, 32.6, 17.9, and 17.6. IR (neat): 3094, 3068, 2972, 2888, 2828, 1737, 1451, 1257, 1087, 1068, 1022, and 999 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{18}H_{18}N_4NaO_2^+$ [M+Na$^+$] requires 345.1322; found 345.1322.

*1-(1H-Benzo[d][1,2,3]triazol-1-yl)-2-methylpropyl Benzoate (**27e** N1 regioisomer) and 1-(2H-Benzo[d][1,2,3]triazol-2-yl)-2-methylpropyl Benzoate (**27e** N2 regioisomer).* A mixture of regioisomers of *N,O*-Acetal **27e** was prepared according to General Procedure D with benzotriazole (121 mg, 1.02 mmol) and benzoyl chloride (62 µL, 0.53 mmol). Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave a 3:2 mixture of regioisomers of **27e** (130 mg, 0.44 mmol, 83%). Analytical data for each isolated regioisomer was acquired by partially separating the mixture of regioisomers using flash chromatography (SiO_2, hexanes:EtOAc 6:1) as a colorless oil. Analytical Data for **27e** N1 regioisomer: ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.04–8.10 (m, 3H), 7.87 (d, *J* = 8.4 Hz, 1H), 7.51–7.62 (m, 2H), 7.44 (br t, *J* = 7.7 Hz, 2H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.13 (d, *J* = 9.7 Hz, 1H), 3.14–3.28 (m, 1H), 1.28 (d, *J* = 6.7 Hz, 3H), and 0.86 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.4, 145.9, 134.0, 133.0, 130.1, 128.7, 128.2, 124.5, 120.2, 110.4, 84.7, 32.2, 18.8, and 18.1. IR (neat): 3065, 2971, 2928, 2876, 1731, 1452, 1257, 1091, 1025, 748, and 711 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{17}H_{18}N_3O_2^+$ [M+H$^+$] requires 296.1394; found 296.1417. Analytical Data for 22e N2 regioisomer: ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.12 (br d, *J* = 7.8 Hz, 2H), 7.90 (nfom, 2H), 7.58 (br t, *J* = 7.7 Hz, 1H), 7.44 (br t, *J* = 7.7 Hz, 2H), 7.39 (nfom, 2H), 7.17 (d, *J* = 9.0 Hz, 1H), 2.93–3.11 (m, 1H), 1.24 (d, *J* = 6.7 Hz, 3H), and 0.86 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 165.0, 144.3, 133.9, 128.8, 128.6, 127.14, 127.09, 118.8, 90.5, 33.1, 18.2, and 17.7. IR (neat): 3068, 2972, 2937, 2878, 1737, 1601, 1564, 1452, 1247, 1089, 748, and 709 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{17}H_{18}N_3O_2^+$ [M+H$^+$] requires 296.1394; found 296.1394.

*2-((4-Chlorophenyl)(phenyl)methyl)isoindoline-1,3-dione (**28**).* $TiCl_4$ (100 µL, 0.91 mmol) was added to a mixture of *N,O*-acetal **8s** (100 mg, 0.30 mmol) in benzene (0.6 mL) CH_2Cl_2 (0.4 mL) at room temperature with stirring. After 24 h, the reaction mixture was diluted in EtOAc, washed with sat. aq. NH_4Cl, dried ($MgSO_4$), and concentrated. Purification by gradient flash chromatography (SiO_2, hexanes:EtOAc 9:1 to 6:1) gave **28** (84 mg, 0.24 mmol, 80%) as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.85 (nfom, 2H), 7.73 (nfom, 2H), 7.28–7.4 (m, 9H), and 6.68 (s, 1H). ^{13}C NMR (75 MHz, $CDCl_3$): δ 167.9, 137.8, 136.8, 134.3, 133.7, 131.8, 130.3, 128.65, 128.62 (2xC), 128.0, 123.6, and 57.2. IR (neat): 3060, 3030, 2900, 1770, 1714, 1492, 1382, 1355, 1328, 1090, and 720 cm^{-1}. HRMS (ESI-TOF): Calcd for $C_{21}H_{14}{}^{35}ClNNaO_2^+$ [M+H$^+$] requires 370.0605; found 370.0590. MP: 156–157 ºC.

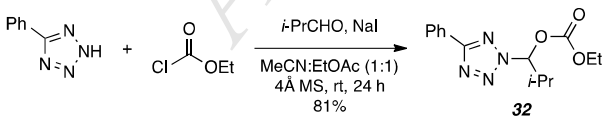
*(4-Chlorophenyl)(phenyl)methanamine (**29**).* Phthalimide **28** (59 mg, 0.17 mmol), ethanol (680 µL), and dichloromethane (230 µL) were added to a culture tube, capped with a Teflon®-lined screw cap, and heated to 40 ºC. After the mixture became homogenous, hydrazine hydrate (33 µL, 0.68 mmol) was added, the Teflon®-lined screw cap was replaced, and the mixture was heated at 80 ºC with stirring. After 24 h, the mixture was cooled to rt, filtered through celite [ethanol:dichloromethane (3:1) eluent], and acidified with 3M aq. HCl. The organic phase was

removed and the organic impurities were removed by washing the aqueous phase with EtOAc (3x). The resulting aqueous mixture was basified to pH>10 with 1M aq. NaOH and extracted with dichloromethane (3x). The dichloromethane extracts were combined, dried ($MgSO_4$), and concentrated to give **29** (19 mg, 0.087 mmol, 51%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.16–7.48 (m, 9H), 5.18 (s, 1H), and 1.93 (br s, 2H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[24]

*2-(2,3-Dihydro-1H-inden-1-yl)isoindoline-1,3-dione (**30**).* $TiCl_4$ (100 µL, 0.91 mmol) was added to a mixture of *N,O*-acetal **8n** (105 mg, 0.33 mmol) in CH_2Cl_2 (3.1 mL) at room temperature with stirring. After 24 h, the reaction mixture was diluted in EtOAc, washed with sat. aq. NH_4Cl, dried ($MgSO_4$), and concentrated. Purification by flash chromatography (SiO_2, hexanes:EtOAc 6:1) gave **30** (57 mg, 0.21 mmol, 64%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.82 (nfom, 2H), 7.71 (nfom, 2H), 7.07–7.33 (m, 4H), 5.88 (dd, *J* = 6.7, 8.9 Hz, 1H), 3.37 (ddd, *J* = 5.1, 8.9, 16.1 Hz, 1H), 3.00 (ddd, *J* = 7.7, 7.7, 15.7 Hz, 1H), and 2.41–2.62 (m, 2H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[25]

*2,3-Dihydro-1H-inden-1-amine (**31**).* Phthalimide **30** (110 mg, 0.42 mmol), ethanol (1.7 mL), and dichloromethane (0.57 mL) were added to a culture tube, capped with a Teflon®-lined screw cap, and heated to 40 ºC. After the mixture became homogenous, hydrazine hydrate (82 µL, 1.7 mmol) was added, the Teflon®-lined screw cap was replaced, and the mixture was heated at 80 ºC with stirring. After 24 h, the mixture was cooled to rt, filtered through celite [ethanol:dichloromethane (3:1) eluent], and acidified with 3M aq. HCl. The organic phase was removed and the organic impurities were removed by washing the aqueous phase with EtOAc (3x). The resulting aqueous mixture was basified to pH>10 with 1M aq. NaOH and extracted with dichloromethane (3x). The dichloromethane extracts were combined, dried ($MgSO_4$), and concentrated to give **31** (37 mg, 0.28 mmol, 67%). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.09–7.42 (m, 4H), 4.37 (dd, *J* = 7.4, 7.5 Hz, 1H), 2.97 (ddd, *J* = 3.5, 8.7, 15.9 Hz, 1H), 2.81 (ddd, *J* = 8.2, 8.2, 16.4 Hz, 1H), 2.51 (dddd, *J* = 3.5, 7.5, 7.5, 12.4 Hz, 1H), 2.1 (br s, NH_2), and 1.69 (dddd, *J* = 8.3, 8.3, 8.3, 12.5, 1H). The ^{1}H NMR data is in good agreement with previously reported characterization results.[26]

*Ethyl (2-Methyl-1-(5-phenyl-2H-tetrazol-2-yl)propyl) Carbonate (**32**).* To aid with structure assignment confirmation of **27d**, *N,O*-Acetal **32** was prepared according to General Procedure D with 5-phenyl-1*H*-tetrazole (121 mg, 0.83 mmol) and ethyl chloroformate (50 µL, 0.53 mmol) (cf. equation shown below). ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.16–8.24 (m, 2H), 7.43–7.54 (m, 3H), 6.78 (d, *J* = 8.8 Hz, 1H), 4.15–4.32 (m, 2H), 2.80 (nfom, 1H), 1.31 (t, *J* = 7.15 Hz, 3H), 1.19 (d, *J* = 6.7 Hz, 3H), and 0.87 (d, *J* = 6.8 Hz, 3H). The crude product (125 mg, 0.43 mmol, 81%) was sufficiently pure for routine ^{1}H NMR analysis, which was in good agreement with reported data.[8i]



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Acknowledgments

This research was supported by the American Chemical Society Petroleum Research Fund Undergraduate New Investigator Grant Program. Hi-Res mass spectrometry data was acquired from instrumentation purchased with funds provided by the NSF Major Research Instrumentation (NSF-MRI) Grant Program (CHE-1429616). Calculations were performed using the Midwest Undergraduate Computational Chemistry Consortium (MU3C) facility at Hope College (funding via NSF-MRI: CHE-1039925).

References and notes

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