A BF_3-Mediated Nitrogen-to-Carbon Rearrangement of N-Protected 2,3-Dihydro-3-hydroxy-1*H*-benzisoindol-1-ones, and Its Interception for a Facile Preparation of 3-Substituted Benzisoindolones

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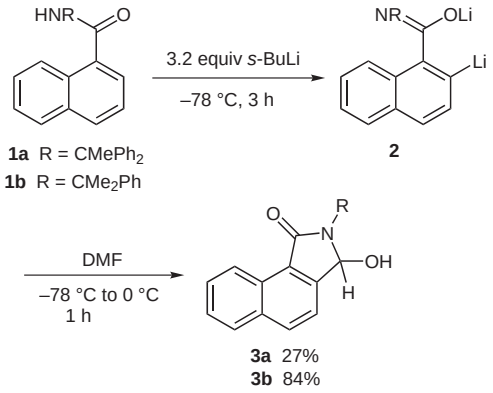
Received 25 May 2006

Abstract: A BF_3-mediated release of the hydroxy and the nitrogen-protecting group of *N*-(cumyl or 1,1-diphenylethyl)-2,3-dihydro-3-hydroxy-1*H*-benzisoindol-1-ones is accompanied by recombination of the nitrogen-protecting unit to the 3-position of the ring system. The addition of sulfur or carbon nucleophiles affords products of preferential capture of the rearrangement intermediate offering a convenient and rapid synthetic route to N-unprotected 2,3-dihydro-3-substituted-1*H*-benzisoindol-1-ones.

Key words: rearrangements, iminium ions, nucleophile, aromatic metalation, PARP inhibitors

The naphthyl amide organic fragment is prevalent in a number of bioactive compounds[1–6] including those targeting the NK1 receptor,[1] the inhibition of aldosterone synthase,[3] the leukocyte proteases cathepsin G[4] and chymase,[5] and a malarial receptor.[2] Aromatic amides including some naphthyl amides are also central to the inhibition of a group of poly(ADP-ribose) polymerase enzymes (PARPs)[7–9] that bind NAD^+ and have been implicated in the cellular response to DNA injury.[10] The catalytic domain of the PARP enzymes has been shown to be structurally similar to that of bacterial ribosyltransferases, including *Pseudomonas aeruginosa* exotoxin A, which we are currently studying. PARP inhibitors often contain aromatic amide or lactam functionalities and operate by preferentially binding to the nicotinamide portion of the binding pocket normally occupied by NAD^+.[9,11,12] As such, we have targeted novel amides and imides possessing the naphthalene[11] backbone as part of a study to find inhibitors of exotoxin A.

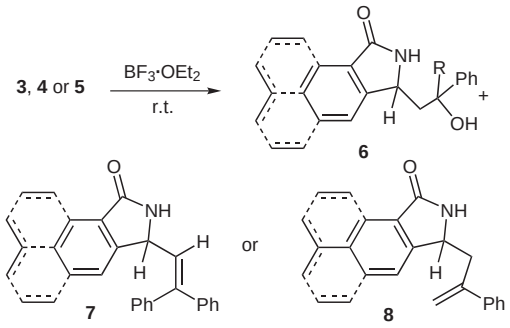
Specifically, we have been probing the functionalization of the 2-position of N-protected 1-naphthamides. We applied the Dai protocol for the *ortho*-metalation of N,N-disubstituted amides (3.2 equiv s-BuLi, THF, –78 °C, 3 h)[13] to monosubstituted amides **1** (**a**: R = 1,1-diphenylethyl, **b**: R = cumyl) as a means to introduce additional functionality. Consistent with the literature,[14] the metalation proceeds at the 2-position rather than the 8-position giving dianions **2**. Quenching with DMF affords two naphthalimidines (2,3-dihydro-3-hydroxy-1*H*-benz[*e*]isoindol-1-ones, **3**), which differ only by their protection on the nitrogen (Scheme 1). At this point, nitrogen deprotection was attempted with $BF_3{\cdot}OEt_2$[15] and a surprising yet parallel manifold of products was obtained for both compounds **3a** and **3b**. Scheme 2 and the first two entries of Table 1 indicate the assigned structures of the unexpected products. In each product, the nitrogen-protecting group is apparently lost and its carbon skeleton is reattached at the adjacent carbon atom.[16]



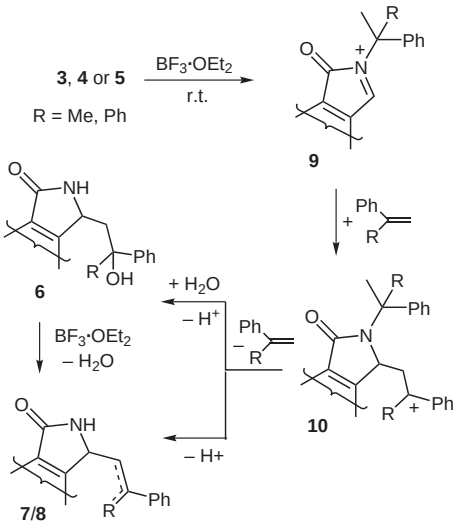
Scheme 1

Three more substrates were prepared in order to establish the generality of the rearrangement. Application of the Dai metalation conditions to the appropriate *N*-1,1-diphenylethyl and *N*-cumyl amides gave phthalimidines (2,3-dihydro-3-hydroxy-1*H*-isoindol-1-ones) **4a** (79%) and **4b**[15] (67%), respectively, and 3-hydroxy-2,3-dihydro-1*H*-benz[*f*]isoindol-1-one (**5b**, 50%, Table 1). As an aside, we are aware of only three reports of metalation of 1,3-unsubstituted-2-naphthylamides. Using only N,N-dialkylated amides, yields are low and unselective[17,18] or the reaction favors lithiation at the 1-position possibly due to 6-methoxy substitution.[19] In our lithiation using an N-monosubstituted amide, the 1-substituted product is isolated in 22% yield. Even though this chemistry has not been optimized, a 50% yield of 3-substituted product is noteworthy[20] and furthermore, the total yield of products is quite high compared to the other examples in this relatively unexplored series of substrates.

SYNLETT 2006, No. 18, pp 3115–3119
Advanced online publication: 25.10.2006
DOI: 10.1055/s-2006-951495; Art ID: S11606ST



Scheme 2



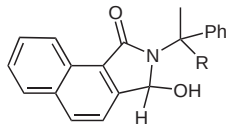
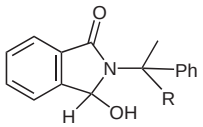
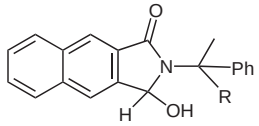
Scheme 3

Treatment of phthalimidines **4** and **5b** with $BF_3{\cdot}OEt_2$ provided the same rearrangement products demonstrating that the benzo fusion on the aromatic is not a requirement and that the placement of the benzo group has minimal effect (Scheme 2, Table 1).

A mechanism for the rearrangement is suggested in Scheme 3. The products are consistent with Lewis acid mediated iminium ion (**9**) formation, which can bring about the loss of the nitrogen-protecting group from some of the substrates, with the lost residue taking the form of alkene (α-methylstyrene or 1,1-diphenylethene). This material then recombines to the proximal carbon of another iminium ion **9** offering carbocation **10**. Loss of proton and BF_3-induced loss of the nitrogen-protecting group via complexation with the carbonyl can explain products **7** and **8**. Alcohols **6** form by reintroduction of nucleophilic oxygen to cation **10** either before or after loss of the nitrogen substituent. A control experiment confirmed that alkenes **7** and **8** may also arise from the action of BF_3 on alcohols **6**.[21] Accordingly, longer reaction times give more of the dehydrated products **7** and **8**. A crossover experiment was supportive of the intermolecularity of the rearrangement. The clean release and reattachment sequence of events distinguishes this rearrangement from related N-to-C transformations entailing pericyclic azonia-[22] or aza-Cope[23] rearrangements of *N*-acyliminium ions.

The above mechanism featuring common *N*-acyliminium ion chemistry[24,25] can fully and adequately account for the rearrangement products. However, as a means of continuing the pursuit of aromatic amides, it was felt that this reaction could be readily adapted to introduce alternative groups at the 3-position and achieve loss of the nitrogen-protecting unit in a single reaction mixture.[26] Hence interception of the electrophilic intermediate **9** by an added nucleophile should permit the introduction of diverse groups at the 3-position while precluding re-attack of α-methylstyrene or 1,1-diphenylethylene. Given the large number of known 3-substituted isoindolones[27] and our interest in naphthyl-derived targets, we focused primarily on substitution of compounds **3b** and **5b**.

Table 1 Products from the BF_3-Induced Rearrangement of N-Substituted Phthalimidines (Scheme 2)

Starting material		R	Products (%)		
			6	**7**	**8**
	**3a**	Ph	**ac**, 26	**c**, 47	–
	3b	Me	**bc**, 50	–	**c**, 38
	**4a**	Ph	**ad**, 8	**d**, 75	–
	4b	Me	**bd**, 42	–	**d**, 28
	**5b**	Me	**e**, 52	–	**e**, 38

A wide variety of common neutral carbon nucleophiles proved amenable for this chemistry. Thiols are also viable, whether bearing hydrophobic or hydrophilic substituents. The products namely 2,3-dihydro-3-substituted 1*H*-benzisoindol-1-ones,[28] could be obtained in 58–80% yields after chromatographic purification (Table 2).[29]

In this transformation, the already-incorporated formyl unit, originally from DMF, and the nucleophile combine to represent the doubly electrophilic synthetic equivalents $HC^{+2}SR$ or $HC^{+2}CR_3$. Moreover, the method is superior to other iminium substitutions in that the hydroxyl group does not require conversion to a methoxy group prior to its displacement.[30] Also, the substitution at the 3-position and the deprotection conveniently occur as a one-pot protocol.

In summary, we report what is believed to be a new rearrangement of *N*-cumyl (and *N*-diphenylethyl)-3-hydroxyisoindolones to 3-substituted isoindolones by way of Lewis acid mediated release and recombination of the N-protecting group. The addition of carbon- or sulfur-based nucleophiles permits capture of the transient iminium and formation of a series of N-unprotected-2,3-dihydro-3-substituted 1*H*-benzisoindol-1-ones. The preparation of these potential PARP inhibitors is a facile and appealing 2-step procedure from simple N-protected aromatic amides. We also suggest the results of past ortholithiation/quenching chemistry of *N*,*N*-dialkyl-2-naphthyl amides may not be fully indicative of the synthetic potential available and such a reaction with N-monosubstituted analogues may prove more useful.

Acknowledgment

The authors thank the Natural Sciences and Engineering Research Council of Canada (NSERC) and the Canadian Cystic Fibrosis Foundation for funding this research. P.A.D. thanks NSERC and the Ontario Government for graduate scholarships. M.M.P. thanks NSERC for a USRA scholarship.

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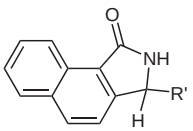
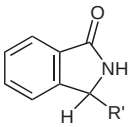
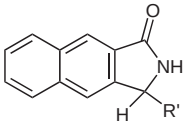
Table 2 The Formation of 2,3-Dihydro-3-substituted 1*H*-(Benz)isoindol-1-ones by Capture of Transient Iminium Ions

Starting material	Added nucleophile	Product		Product R′ group	Yield (%)
3b	$C_{16}H_{33}SH$		**11**	$-SC_{16}H_{33}$	78
	$HO(CH_2)_2SH$		**12**	$-S(CH_2)_2OH$	70
	CH_2=$CHCH_2TMS$		**13**	$-CH_2CH=CH_2$	80
	1-(TMSO)cyclopentene		**14**	2-Cyclopentan-1-onyl	58
4a	$C_{16}H_{33}SH$		**15**	$-SC_{16}H_{33}$	84
5b	$HOC(O)CH_2SH$		**16**	$-SCH_2CO_2H$	67
	Furan		**17**	2-Furyl	64
	$HC{\equiv}CCH_2TMS$		**18**	Allenyl	80

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(16) Rearrangement products **7** and **8** could be identified through spectral analysis including the observation of vinylic resonances in the ^{1}H NMR spectra. Tertiary alcohols **6**, obtained as mixtures of diastereomers, were identified with the assistance of IR spectroscopy and mass spectrometry where applicable. Strong M^+ peaks were seen under CI conditions. Full characterization data including elemental analysis or HRMS was obtained for all new compounds. Sample procedure for rearrangement: Isoindolone **3a** (154 mg, 0.406 mmol, 1.0 equiv) was dissolved in 2.5 mL dry CH_2Cl_2 in a flame-dried flask under argon and cooled to 0 °C. Then, $BF_3{\cdot}OEt_2$ (0.06 mL, 0.570 mmol, 1.4 equiv) was dissolved in 2.5 mL of dry CH_2Cl_2 in a flame-dried flask under argon and transferred to the solution of **3a** via cannula, followed by a 2.5 mL CH_2Cl_2 rinse. The resulting dark brown solution quickly became clear and colorless, and was stirred overnight at r.t. The reaction was quenched with H_2O, extracted with CH_2Cl_2, dried with brine and $MgSO_4$ and concentrated. Flash chromatography with 15% EtOAc in hexane gave 69.0 mg (47% yield) of **7c** and 39.6 mg (26% yield) of **6ac**.
3-(2,2-Diphenylethenyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (**7c**): mp: 199-200 °C. ^{1}H NMR (400 MHz, DMSO-d_6): δ = 9.08 (d, *J* = 8.3 Hz, 1 H), 8.14 (d, *J* = 8.4 Hz, 1 H), 8.04 (d, *J* = 8.2 Hz, 1 H), 7.68–7.65 (m, 1 H), 7.61–7.41 (m, 9 H), 7.29–7.19 (m, 3 H), 5.81 (d, *J* = 9.9 Hz, 1 H), 5.04 (d, *J* = 9.9 Hz, 1 H). ^{13}C NMR (100 MHz, DMSO-d_6): δ = 170.6, 170.5, 147.1, 145.3, 140.6, 138.5, 132.9, 132.7, 129.7, 128.83, 128.77, 128.4, 127.9, 127.8, 127.0, 126.5, 125.5, 125.4, 122.9, 120.8, 55.4. IR (neat) 3447, 1690 cm^{-1}. MS (EI): *m/z* (%) = 362 (29), 361 (100) [M^+], 284 (37). Anal. Calcd (%): C, 86.40; H, 5.30; N, 3.88. Found: C, 86.20; H, 5.54; N, 3.95.
3-(2,2-Diphenyl-2-hydroxyethyl)-2,3-dihydro-1*H*-benzo[*e*]isoindol-1-one (**6ac**): ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.88 (d, *J* = 8.3 Hz, 1 H), 7.84 (d, *J* = 8.3 Hz, 1 H), 7.74 (d, *J* = 8.1 Hz, 1 H), 7.47–7.36 (m, 4 H), 7.25–7.20 (m, 5 H), 7.15–7.03 (m, 4 H), 4.32 (d, *J* = 10.4 Hz, 1 H), 2.96 (d, *J* = 13.8 Hz, 1 H), 2.17 (dd, *J* = 13.8, 10.4 Hz, 1 H). ^{13}C NMR (75.5 MHz, acetone-d_6): δ = 170.8, 151.8, 149.9, 148.9, 148.8, 147.3, 134.1, 133.2, 130.4, 129.1, 129.0, 128.4, 127.9, 127.6, 127.1, 127.0, 124.3, 121.2, 78.6, 54.2, 47.4. IR (nujol): 3426, 3353, 1668 cm^{-1}. MS (EI): *m/z* (%) = 379 (8) [M^+], 361 (32), 284 (16), 196 (42), 184 (27), 183 (32), 182 (59), 105 (27). MS (CI): *m/z* (%) = 380 (90) [M + H]$^+$, 362 (44), 213 (53), 200(100), 196 (55), 183 (39). HRMS (EI): *m/z* calcd for $C_{26}H_{19}NO$ [M – 18]$^+$: 361.1468; found: 361.1470.
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(29) **Representative Procedure.**
Isoindolone **3b** (125 mg, 0.395 mmol, 1.0 equiv) and allyltrimethylsilane (0.25 mL, 1.57 mmol, 4.0 equiv) were slurried in 2.5 mL of dry CH_2Cl_2 in a flame-dried flask under argon and cooled to 0 °C. Then, $BF_3{\cdot}OEt_2$ was dissolved in 2.5 mL of dry CH_2Cl_2 in a flame-dried flask under argon and transferred to the solution of **3b** and thiol via cannula, followed by a 2.5 mL CH_2Cl_2 rinse. The clear colorless reaction mixture was stirred at r.t. overnight before quenching with H_2O, extracting with CH_2Cl_2, drying with brine and $MgSO_4$ and concentrating. Flash chromatography with 20% EtOAc in hexane gave 71 mg (80% yield) of **13**.
3-Allyl-2,3-dihydro-1*H*-benz[*e*]isoindol-1-one (**13**): ^{1}H NMR (400 MHz, $CDCl_3$): δ = 9.22 (d, *J* = 8.4 Hz, 1 H), 8.02 (d, *J* = 8.4 Hz, 1 H), 7.92 (d, *J* = 8.4 Hz, 1 H), 7.67 (t, *J* = 7.2 Hz, 1 H), 7.57 (t, *J* = 7.2 Hz, 1 H), 7.52 (d, *J* = 8.4 Hz, 1 H), 5.89–5.78 (m, 1 H), 5.19 (d, *J* = 16.4 Hz, 1 H), 5.16 (d, *J* = 10.0 Hz, 1 H), 4.72 (dd, *J* = 8.0, 4.5 Hz, 1 H), 2.85–2.79 (m, 1 H), 2.42–2.35 (m, 1 H). ^{13}C NMR (100.6 MHz,

$CDCl_3$): δ = 171.9, 147.5, 133.1, 133.0, 132.7, 129.5, 128.1, 127.9, 126.5, 125.9, 123.9, 119.6, 119.1, 55.7, 38.7. IR: 3448, 3215, 1690 cm^{-1}. MS (EI): *m*/*z* (%) = 223 (8) [M^+], 183 (14), 182 (100), 127 (13). Anal. Calcd: C, 80.69; H, 5.87. Found: C, 80.90; H, 5.85.

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