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Oxidations of pyrrolidines and piperidines to afford CH-functionalized isopropyl-1-carboxylate congeners

Steven Gunawan [a], Nathan Bedard [a], Christopher Foley [a], Christopher Hulme [a,b,*]

[a] Department of Chemistry and Biochemistry, The University of Arizona, Tucson, AZ 85721, United States
[b] Department of Pharmacology and Toxicology, College of Pharmacy, The University of Arizona, Tucson, AZ 85737, United States

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ABSTRACT

This article describes the action of iodine(III) reagents [diacetoxyiodobenzene, $PhI(OAc)_2$, and iodosobenzene, $(PhIO)_n$] in conjunction with TMSBr which act as functional bromine equivalents in unique oxidations of saturated, carbamate protected *N*-heterocycles. Interestingly, during this work, treatment of the same carbamates with molecular bromine alone afforded similar products, which were sequestered by the solvent methanol.

Introduction

Iminiums such as **1** (Fig. 1) are one of the most important electrophiles in synthetic organic transformations for the creation of carbon–carbon and carbon–heteroatom bonds. Covalent attachment of electron withdrawing groups at the nitrogen atom enhances its cationic character making the species a more reactive intermediate (Fig. 1) [1,2]. Amongst these modified cations, much interest has centered around *N*-acyliminium ions **2** and **3**, although ureas **4**, *N*-tosyl derivatives **5**, and hydrazonium ions **6** have also been studied [1]. The importance of *N*-acyliminium ions **2** and **3** has been demonstrated in a multitude of natural product syntheses [3–5] and exploited in secondary reactions on multi-component reaction products leading to the formation of unique small molecules [6–8].

Typically, *N*-acyliminium ions or their precursors are generated *in situ*, being commonly prepared *via* direct oxidative electrochemical methods [9–12], reduction of lactams or imides through hydride addition [1,2,13–17], ring closure of linear amides [18–20], cuprous ion-promoted decomposition of *o*-diazobenzamides [21], and chemical oxidation by means of iodine(III) and TMSX (X = N_3, Cl) reagent combinations [22,23].

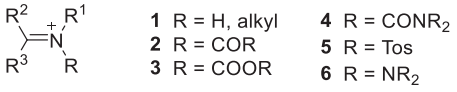

Fig. 1. Iminium ion and its derivatives.

Of particular interest is the use of hypervalent iodine reagents, thus named due to their ability to possess more than eight electrons in the valence shell as required by the octet rule [24]. Two oxidation states dominate the field - iodine(III) and iodine (V) [25–27]. The most widely-used iodine(V) compounds are Dess-Martin periodinane [28] and *o*-iodoxybenzoic acid (IBX) [29]. Conversely, iodine(III) reagents are categorized into five classes: (1) iodosylarenes (ArIO) and their acyclic derivatives ($ArIX_2$) (2) five-membered iodine heterocycles (benziodoxoles, and benziodazoles), (3) iodonium salts ($R_2I^+X^-$), (4) iodonium ylides (ArI = CR_2), and (5) iodonium imides (ArI = NR) [27] where each reagent has different chemical properties and synthetic applications. Iodosylarenes have broad utility as oxidizing reagents [27].

Of particular note is a series of elegant applications of the $(PhIO)_n/TMSN_3$ reagent combination for the β-azidonation of triisopropylsilyl (TIPS) enol ethers [22,30–32]. Further studies demonstrated that $(PhIO)_n$ and $TMSN_3$ enabled the direct α-azidonation of amides, carbamates, and ureas (Scheme 1) [33], representing the first direct chemical oxidation of *N*-protected

* Corresponding author at: Department of Pharmacology and Toxicology, College of Pharmacy, The University of Arizona, Tucson, AZ 85737, United States.
E-mail address: hulme@pharmacy.arizona.edu (C. Hulme).

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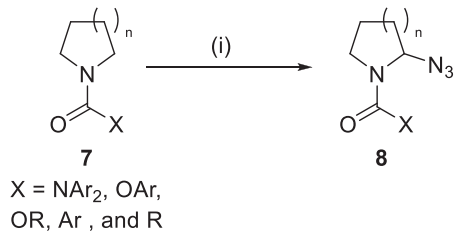
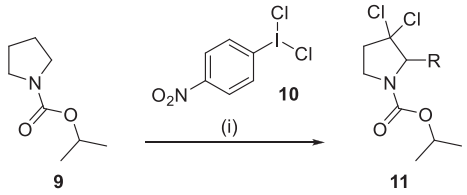
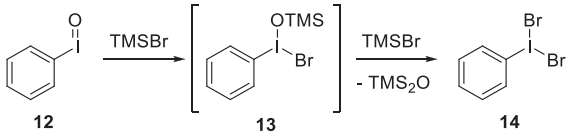


Scheme 1. α-Azidonation of amides, carbamates, and ureas. *Reagents and conditions:* (i) $(PhIO)_n$ (2.4 eq.), $TMSN_3$ (4.8 eq.), CH_2Cl_2, –40 to –25 °C (n = 1 25–82% yield, n = 2 11–41% yield) or IBX (2.4 eq.), $TMSN_3$ (4.8 eq.), CH_2Cl_2, reflux (n = 1, 32–64% yield; n = 2, 15–85% yield).

pyrrolidine and piperidines to products which were readily ionized to *N*-acyliminium ions. Use of two different iodine(III) reagents ($(PhIO)_n$ and IBX) revealed a slower rate of α-azidonation and lower yield of products from the piperidine series **7** (n = 2) than with the pyrrolidine **7** (n = 1) series. Optimal conversions were



Scheme 2. α,β,β-Oxidations of carbamates. *Reagents and conditions:* (i) **10** (4–5 eq.), MeCN, 45 °C (85% yield) (1 h) or MW 150 °C (5 min) (71% yield).



Scheme 3. *In situ* generation of $PhIBr_2$.

observed with the urea derivative (n = 1, X = NPh_2) which afforded α-azido product **8** in high yield [33].

The downside of Magnus' α-azidonation protocol is the instability of the putative reactive intermediate $PhI(N_3)_2$ which decomposes to iodobenzene and 3 mol of N_2(g) with sporadic violent explosions [23]. Interestingly, oxidative applications of Willgerodt's reagent ($PhICl_2$) [23,34] on the same starting materials were subsequently reported. Encouragingly, treatment of carbamate **9** with a modified version of Willgerodt's reagent (dichloro (4-nitrophenyl)iodane, **10**) afforded the surprising α,β,β-oxidation product **11** (R = OH) (Scheme 2). Addition of 5% MeOH to the solvent produced **11** (R = OMe) (80% yield) *via* ionization of **11** to the *N*-acyliminium ion and solvent trapping. A similar oxidation to a methoxy dichloride with *tert*-Butyl hypochlorite in CH_2Cl_2: MeOH has been reported in which an *N*-acyliminium intermediate was proposed [35].

As such, this clearly suggested that the study of alternate $(PhIO)_n$/TMSX combinations was warranted, in particular the reactivity of the short-lived species $PhIBr_2$ **14** [36] (Scheme 3). Especially when photochemical oxidations to ethoxy dibromo species have been demonstrated with pyrrolidine-2-ones [37].

Results and discussion

Pilot studies utilized the acid-stable carbamate **15** to avoid deprotection by HBr, a side product of the expected reaction. Thus,

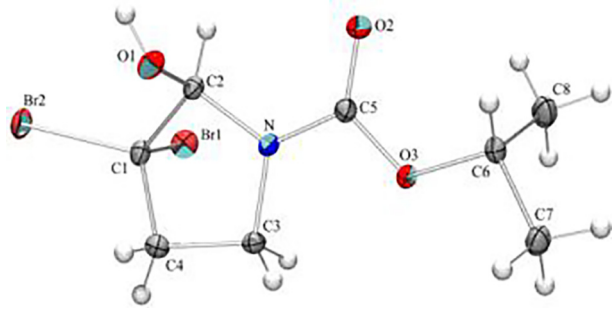
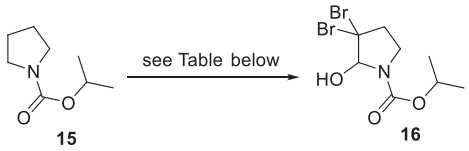


Fig. 2. X-ray crystal structure of **16.**

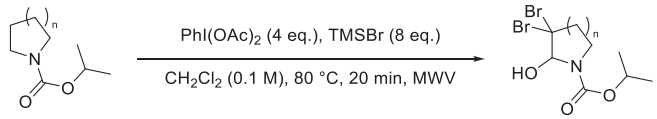
Table 1
Reaction optimization using $(PhIO)_n$ and PIDA hypervalent iodine reagents.



Entry	Solvent	T	t	$Ph(IO)_n$ (eq.)	$PhI(OAc)_2$ (eq.)	TMSBr (eq.)	Yield **16** (%)
1	CH_2Cl_2	0 °C to r.t.	19.5 h	2	–	4	9%
2	MeCN	0 °C to r.t.	17.5 h	2	–	4	3%
3	CH_2Cl_2	–60 °C	3 h	2	–	4	5%
4[a]	CH_2Cl_2	0 °C to r.t.	20 h	10	–	20	6%
5[b]	CH_2Cl_2	80 °C	5 min	2	–	4	22%
6	CH_2Cl_2	0 °C to r.t.	5 d	–	4	8	40%
7	CH_2Cl_2	60 °C	20 min	–	4	8	48%
8	CH_2Cl_2	80 °C	5 min	–	4	8	48%
9	CH_2Cl_2	80 °C	10 min	–	4	8	62%
10	CH_2Cl_2	80 °C	20 min	–	4	8	69%
11	MeCN	80 °C	20 min	–	4	8	39%
12	CH_2Cl_2	120 °C	20 min	–	1	2	14%
13	CH_2Cl_2	120 °C	20 min	–	2	4	35%
14	CH_2Cl_2	120 °C	20 min	–	3	6	56%
15	CH_2Cl_2	120 °C	20 min	–	4	8	65%

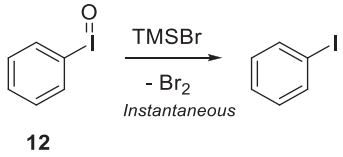
[a] 56% starting material was recovered.
[b] 22% starting material was recovered.

Table 2
Scope of substrates.



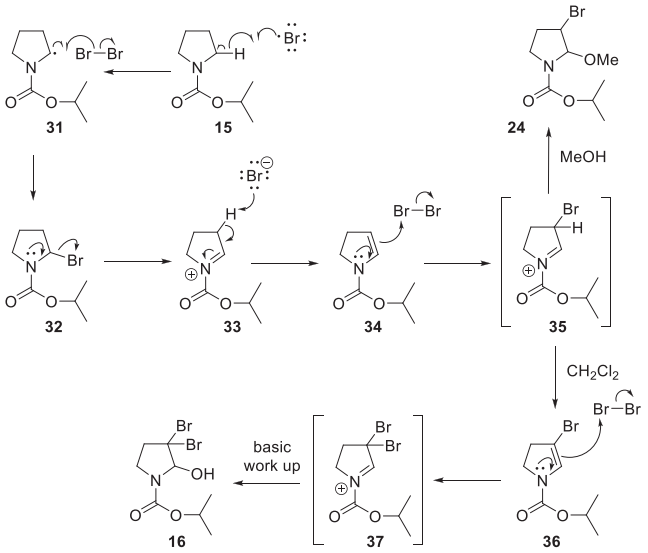
18-20 21-23

Substrate	n	Product	Yield (%)
18	2	**21**	76
19	3	**22**	62
20	4	**23**	56



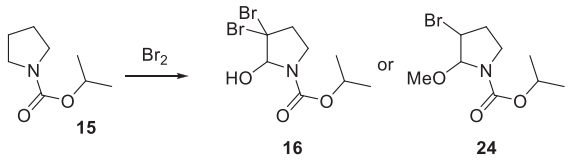
Scheme 4. Decomposition of PIDA/TMSBr to PhI and Br_2.

exposure of **15** to $Ph(IO)_n$ **12** (2 eq.) and TMSBr (4 eq.) afforded the α,β,β-oxidized product **16** albeit in low yield (Table 1, entry 1) (0 °C to rt, o/n). Nonetheless, unequivocal structural confirmation of **16** was provided by X-ray crystallography (Fig. 2) [38]. The yield was not improved upon changing the solvent to MeCN (Table 1, entry 2), lowering the temperature to –60 °C (Table 1, entry 3), or using an excess of reagents (Table 1, entry 4). However, improvement was observed after microwave irradiation at 80 °C for 5 min (Table 1, entry 5), (22% yield, 22% recovered starting material). The use of phenyliodine(III) diacetate (PIDA) **17** with TMSBr [39] afforded a significant improvement in the yield of **16**



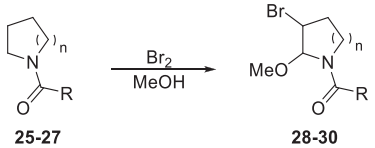
Scheme 5. Proposed radical mechanism for the α,β- and α,β,β-carbamate oxidations.

Table 3
Bromination of carbamate **15**.



Entry	Solvent	T	t	Br_2 (eq.)	Product	Yield (%)
1	CH_2Cl_2	80 °C	1 h	3	**16**	43
2	CH_2Cl_2	120 °C	20 min	3	**16**	41
3	CH_2Cl_2	80 °C	20 min	5	**16**	46
4	MeOH	80 °C	1 h	6	**24**	39

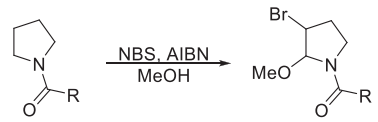
Table 4
Scope of the bromination.



Entry	Substrate	n	R	Br_2 (eq.)	Product	Yield (%)
1	**25**	2	Ph	6	**28**	NR
2	**26**	1	O-*t*Bu	3	**29**	0 [a]
3	**27**	1	O-Bn	3	**30**	11 [b]
4	**27**	1	O-Bn	6	**30**	15 [c]

NR = no reaction.
[a] No recovered product or starting material.
[b] 52% recovered starting material.
[c] 5% recovered starting material.

Table 5
Radical bromination.



15, **26-27** **24**, **29-30**

Entry	Starting material	R	T	t	NBS (eq.)	AIBN (eq.)	Product	Yield (%)
1	**26**	O-*t*Bu	65 °C	reflux, 4 h	1.05	0.05	**29**	0[a]
2	**27**	O-Bn	80 °C	MW, 1 h	10	0.5	**30**	30[b]
3	**15**	O-*i*Pr	80 °C	MW, 1 h	10	0.5	**24**	41

[a] No recovered starting material or desired product.
[b] 32% recovered starting material.

to 40% (5 d, rt) (Table 1, entry 6). Subsequent yields after microwave irradiation continued to improve with prolonged reaction time from 5 to 20 min at 80 °C, ranging from 48 to 69% (Table 1, entry 8–10) with dichloromethane superior to acetonitrile (Table 1, entry 11). Stoichiometry studies at elevated temperature (Table 1, entries 12–15) demonstrated linear yield improvements. The scope of the reaction was also demonstrated by the use of different substrates (Table 2), proving compatible with 6-, 7- and 8-membered rings.

During the reactions, we observed the rapid formation of a dark orange color which was repeated upon mixing $(PhIO)_n$ or PIDA with TMSBr in CH_2Cl_2 and deemed indicative of the generation of molecular bromine. Indeed, prior reports describe the intermediate derived from the PIDA/TMSBr reagent combination decomposing to iodobenzene and bromine after only 5 min (Scheme 4) [40]. This clearly suggested that study of molecular bromine as a potential oxidant was warranted, assuming that $(PhIO)_n$/TMSBr was a functional equivalent. Indeed, when **15** was treated with bromine in CH_2Cl_2 and irradiated at 80 °C for 1 h, product **16** was isolated (43% yield) (Table 3, entry 1) suggesting the occurrence of photobromination [41] in conjunction with or, more likely, in place of the assumed action of $PhIBr_2$ **14**. At an elevated temperature or when excess bromine was employed (Table 3, entry 2 and 3), comparable yields of **16** were attained. However, when the reaction was conducted in a nucleophilic solvent (MeOH), a new α,β-oxidized product **24** was isolated (39% yield) (Table 3, entry 4). It is important to note that when an amide was used as the starting material (*N*-benzoyl-piperidine), no conversion was observed (Table 4, entry 1). Similarly, no product was furnished when employing Boc-protection (Table 4, entry 2), presumably due to the production of HBr (Scheme 5) with subsequent starting material and/or product decomposition. Cbz-protection proved amenable to α,β-oxidation albeit in low yield (Table 4, entry 3 and 4). This was also not surprising as *N*-Cbz deprotection is known to occur with HBr in glacial acetic acid [42,43].

With this data in-hand, it seemed likely the transformation would also occur upon exposure to *N*-bromosuccinimide (NBS) and the radical initiator azoisiisobutyronitrile (AIBN) (Table 5). As such, Boc-protected pyrrolidines afforded no discernible products (Table 5, entry 1). Conversely, *N*-Cbz-pyrrolidines furnished the α,β-product **30** in 30% yield (Table 5, entry 2), higher than the corresponding yield with methanolic bromine (Table 4, entry 3 and 4). Finally, the reaction of *N*-isopropyloxy-pyrrolidine **15** with NBS/AIBN, produced **24**, isolated in comparable yield (41%) to treatment with Br_2 in MeOH (Table 5, entry 3).

A mechanism was proposed to explain formation of the observed products (Scheme 5). Exposure of carbamate **15** to either (i) NBS and AIBN (ii) molecular bromine or (iii) *in situ* generated bromine from $Ph(IO)_n$ and TMSBr, furnishes **31** [10,21]. Subsequent formation of α-bromo-carbamate **32** follows which is readily ionized to **33**. β-proton removal furnishes enamide **34** and evolution of hydrobromic acid. Enamide reaction with bromine readily affords the β-Br *N*-acyliminium ion **35**. The latter is trapped by methanol to give the α,β-product **24**. Conversely, in a non-nucleophilic aprotic solvent (CH_2Cl_2), the reaction proceeds through bromo-enamide **36** to the β,β-dibromo-*N*-acyliminium ion **37**, which upon basic work-up furnishes the final α,β,β-product **16**. Intrigued by the *N*-acyl-α-hydroxy-β,β-dibromo-functionality, sub-structure searching on Scifinder revealed no precedents, although Reaxys revealed apparent reports by Leuchs 100 years ago detailing the action of molecular bromine on a strychnine analog [44]. In this case, an acyl-pyrrolidine ring is formed upon rearrangement of a strychnine derivative, to afford two open α- and β-methylene carbon atoms primed for further reaction with molecular bromine to afford the α-hydroxy-β,β-dibromo- congener. Irrespective of this report, we feel that publication of this non-electrochemical transformation on simple 'deconstructed' saturated nitrogen heterocycles warrants reporting.

Conclusion

Herein, we report new oxidation chemistry mediated by hypervalent iodine(III) reagents in conjunction with TMSBr to furnish α-hydroxy-β,β-dibromine functionalized *N*-isopropyloxy protected pyrrolidines and piperidines. Moreover, additional studies demonstrate that molecular bromine promotes these transformations, with yields improved through use of NBS and the radical initiator AIBN. In addition, the α-methoxy-β-bromine derivative of *N*-isopropyloxy-pyrrolidine was produced when utilizing a methanolic bromine solution.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.tetlet.2021.152978.

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