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Recent progress in hypervalent iodine chemistry

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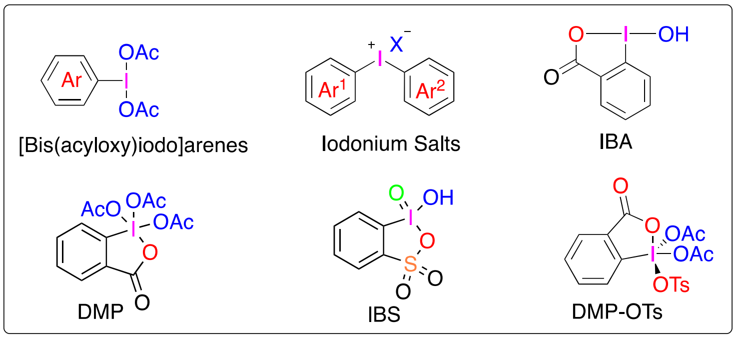
Abstract

Hypervalent iodine compounds are associated with unique and environmentally friendly properties and have been intensively utilized as verstaile reagents and catalysts in modern organic synthesis. In many cases, the hypervalent iodine promoted reactions are quite distinctive and cannot be performed by using any other, non-iodine-based reagent. The importance of hypervalent iodine reagents in modern chemistry is exemplified by the ever-increasing number of research articles and reviews in this area. This short overview presents a brief introduction to Part 4 of the Arkivoc series on hypervalent iodine chemistry. This introductory article provides a summary of major review papers on hypervalent iodine that were published in 2023-2024 following the publication of Part 3 of this series.



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1. Introduction: Hypervalency and Hypervalent Iodine

A main group element compound that possesses a number of formally assignable electrons exceeding the octet in the valence shell around its central atom is generally termed as ‘hypervalent’.[1–4] This expansion of the electronic valence shell beyond the classical Lewis–Langmuir octet, particularly in the non-metallic elements, gave birth to a new and important era in the chemistry of these elements. Jeremy I. Musher,[3] in 1969, primarily defined hypervalent molecules as those formed by the non-metals of groups 15-18 in any of their stable valence states higher than 3, 2, 1, and 0, respectively. However, in the recent times, this terminology has also been extended to the group 13 and 14 elements.[5] Hypervalent molecules display certain special structural features and reactivity pattern that can be attributed to the hypervalent bonding, which involves a 3c-4e bond.[5–7] Organohypervalent iodine compounds are amongst the most widely utilized hypervalent molecules. Rundel[8] and Pimentel[9] developed the molecular description of the 3c-4e bond, that involves formation of three molecular orbitals by the combination of p-orbital on the central atom and one atomic orbital from each of the two ligands. The three molecular orbitals obtained through the combination of these atomic orbitals are bonding (BMOs), non-bonding (NBMOs), and anti-bonding molecular orbitals (ABMOs) in which bonding and non-bonding molecular orbitals each contain one pair of electrons. A representative example of such presentation from ‘10-I-3’ is given in Figure 1. The ‘5p’ orbital of the iodine interacts with the half-filled orbitals of the two ligands ‘L’ thus forming three molecular orbitals (MOs). Further, the node present in the non-bonding molecular orbitals (NBMOs) results in the charge distribution of almost +1.0 on the iodine atom and -0.5 on each ligand. A normal covalent bond exists between the carbon substituent and the iodine atom. Both ligands are attached by the hypervalent bond and occupy the apical positions leading to distorted trigonal bipyramidal geometry, while the carbon substituent occupies the equatorial position. Besides, the ‘12-I-5’ species, has overall either a square bipyramidal or pseudooctahedral geometry.

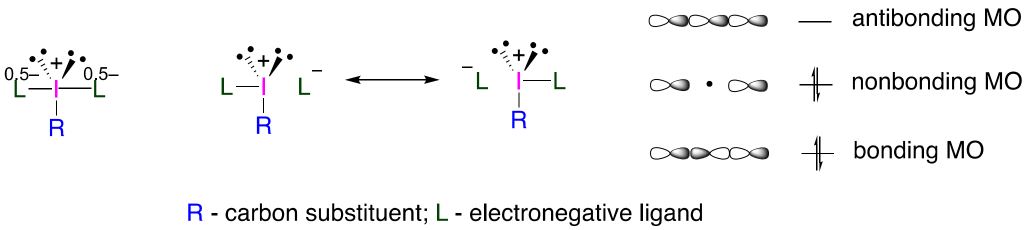

Figure 1. Description of the ‘3c-4e’ in hypervalent iodine ‘10-I-3’ species.

Hypervalent iodine compounds have been intensively utilized as reagents/catalysts in organic synthesis due to their unique and environmentally friendly properties. Certain features of hypervalent iodine compounds are similar to that of transition metals, such as Ag(I), Hg(II) and Tl(III), and further investigation of these similarities augmented into the invention of new reagents and important synthetic methodologies. The low toxicity, easy handling and high-bench stability associated with these compounds make them valuable alternatives to these heavy metals to access diverse organic transformations. These compounds have shown paramount significance in organic synthesis for the development of organic transformations that include couplings,[10] oxidative rearrangements,[11] oxidations,[12] cyclizations,[13] atom-transfer reactions,[14] alkene functionalization,[15] C–H bond functionalization,[16] photochemical transformations,[17] α-functionalization of carbonyl compounds,[18] and organocatalysis.[19] A range of hypervalent iodine(III) (λ^3-iodanes) and hypervalent(V) (λ^5-iodanes) compounds was reported in the literature, and some representative examples are shown in Figure 2. It is pertinent to highlight that in many cases the hypervalent iodine-based transformations are quite unique and cannot be performed by using any other, non-iodine-based reagent.

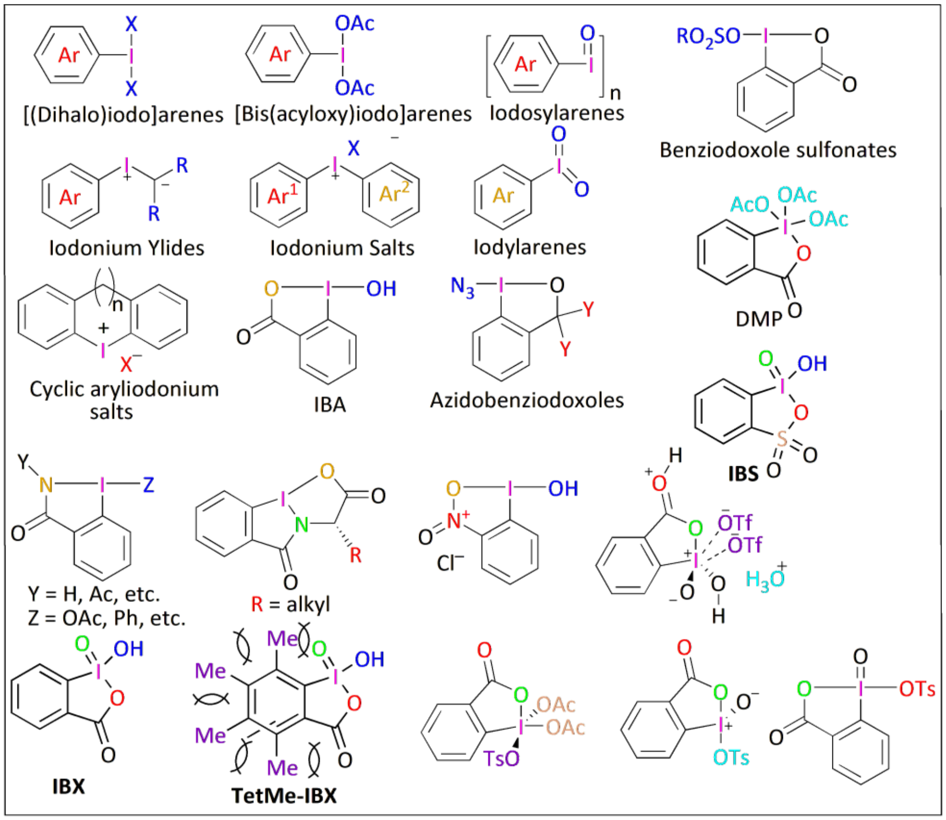

Figure 2. Representative examples of acyclic and cyclic hypervalent iodine(III) (λ^3-iodanes) and hypervalent(V) (λ^5-iodanes) compounds.

2. Recent Developments and Future Perspectives

Polyvalent iodine plays a pivotal role in the modern organic synthesis that can be realized by a vast number of recently published books and reviews covering several hot areas of hypervalent iodine chemistry. Since the beginning of the 21st century, six books[20-25] and several hundred review articles have appeared in the literature highlighting the extensive utilization of these compounds. Till date, seven international conferences dedicated to hypervalent iodine chemistry have been organized through the globe and this series is still continuing with the 8th conference to be held in 2025 in China. Current surging interest in the hypervalent iodine chemistry and development of new synthetic methodologies based on hypervalent iodine compounds inspired us for the publication of Part 4 of the hypervalent iodine (HI) series. Previously published parts 1-3 of the HI series were phenomenal that cover numerous synthetic applications of hypervalent iodine compounds. Parts 1-3 of the series contain total twenty seven papers covering wide range of diverse applications of hypervalent iodine compounds. Part 3 of the series, specifically includes selective aryl transfer from (mesityl)iodonium(III) salts,[26] computational study on the iodobenzene-catalyzed oxidative cyclization,[27] nucleophilic fluorination of 1-arylbenziodoxolones,[28] synthesis and properties of liquid phenyliodine dicarboxylates,[29] etc. Numerous recent reviews have been dedicated to the hypervalent iodine compounds and their applications in organic synthesis. Since 2022, after the publication of Part 3 of the HI series, more than forty major review articles have appeared in the literature covering different aspects of hypervalent iodine compounds.[30-66,68-72] Recently, hypervalent iodine (III) based chemical transformations published mainly in the last 7−8 years, between 2016 and spring 2024 have been comprehensively presented by Yoshimura and Zhdankin.[30] Collection on recent synthetic applications of hypervalent iodine reagents has been covered in three installments by Aneja *et al*.[31-33]

In addition to the acyclic iodanes, diverse range of cyclic hypervalent iodine compounds have been explored in modern organic synthesis.[39] The enhanced stability of the cyclic derivatives is particularly significant for hypervalent iodine, allowing the preparation and broad synthetic application of numerous valuable reagents. Numerous benziodoxole derivatives have been utilized as reagents for transfer of the substituent on hypervalent iodine to organic substrate. Transformations of these reagents with organic substrates can be performed under metal-free conditions, in the presence of transition metal catalysts, or using photocatalysts under photoirradiation conditions. Cyclic hypervalent iodine (III) reagents with the ligands, N_3, NHR, CN, CF_3, SCF_3, OR, OAc, ONO_2, and $C(=N_2)CO_2R$ can be easily utilized for the transfer of these ligands to organic substrates.[40] As well, aryl-, alkenyl-, and alkynylbenziodoxoles have recently received wide synthetic applications as efficient reagents for direct arylation, alkenylation, and alkynylation under mild reaction conditions that include metal-free conditions as well as photoredox and transition metal catalysis.[41] Synthesis of wide range of valuable and structurally diverse complex molecules can be achieved using these reagents. Ethynylbenziodoxolone (EBX) reagents are among the most verstaile electrophilic alkynylation reagents[42] An extended structural analysis of EBX reagents and their analogues based on X-Rays, MEP and ^{1}H- and ^{13}C-NMR has been recently revealed.[43] The merger of pseudocyclic hypervalent iodine reagents with carbenes or carbenoids enables disconnections that are almost impossible using classical reactivity.[44] Hypervalent iodine compounds have been exceptionally useful as partners in reactions with metal carbenes and as reagents combining the reactivity of iodine(III) and carbenes in a single molecule giving carbyne equivalents.[44] Cyclic hypervalent fluoro-iodane, has been known for various alkene functionalizations enabling the synthesis of fluoro-benzoxazepines, indoles, and ketones, wherein the unexpected non-covalent interaction between the nucleophile, substrate, and iodane played a key role in deciding the product formation.[45]

Cyclic diaryliodonium salts represent another important class of hypervalent iodine reagents that have been widely explored for the synthesis of numereous axially chiral biaryls and biaryl compounds. Mono- and difunctionalizations of cyclic diaryliodoniums and their applications for the synthesis of fused ring systems and as organocatalysts is well documented by Cheng *et al*.[46] Besides, one-pot double functionalization of carbon–iodine(III) and *ortho* carbon–hydrogen bonds using diaryliodonium(III) salts involving arylation/intramolecular rearrangement, arylation followed by electrophilic aromatic substitution, three-component [2+2+2] cascade annulation, sequential metal-catalyzed arylations, and double functionalization *via* aryne formation has emerged out as verstaile method in modern organic synthesis.[47]

Numereous specific reports highlighting the role of hypervalent iodine reagents towards various valuable transformations, such as, azidation,[48-49] vicinal diamination[50] and trifluoromethylthiolation,[51] have been judiciously documented by different researchers. Waser reported initially some selected examples of azidations through *in-situ* generated unstable non-cyclic iodanes and then discussed some stable cyclic iodanes for azidation in detail.[48] Another seminal report dealing with the azidation using unstable azidoiodinanes generated *in-situ* from commonly used hypervalent iodine reagents (such as diacetoxy-iodobenzene or iodosylbenzene) and azide anion source ($TMSN_3$ or NaN_3) as well as application of stable azidobenziodoxoles for selective direct azidation of C−H bonds or double carbon-carbon bonds was recently presented.[49] Oxidative amination of alkenes or related molecules has emerged out as a sustainable method that can be utilized for the construction of two C–N bonds in an efficient manner. The major breakthroughs in this area, involving iodine-based reagents/catalysts, especially the inter/intra-molecular vicinal diamination of alkenes with electron-rich or deficient diverse nitrogen sources reported in 2015-2022 has been reviewed recently.[50] Proposed mechanistic pathways have been given emphasis in this report to get insights into the key factors governing the issues of regioselectivity, enantioselectivity, and diastereoselectivity ratios. Electrophilic sources of nitrogen-based groups have been known for many decades, but with limited examples only. However, hypervalent iodine reagents bearing transferable N-based groups gave a boost to this area with increasing opportunity in this area.[52]

Certain other recent reviews have been dedicated to other synthetic applications of specific classes of hypervalent iodine compounds. Vittal *et al* have systematically presented the various applications of (diacetoxyiodo)benzene ($PhI(OAc)_2$, PIDA) in organic synthesis involving C−C, C-hetero, hetero-hetero and multiple bond forms from the summer of 2015 to the present.[53] Certain representative developed procedures using different iodine(III) reagents for the functionalization of aryls and heteroaryls by introducing halogens have been summarized.[54] Another important aspect is the utilization of iodanes for the rapid formation of natural products or related complex architectures. A plethora of synthetic strategies based on several key steps mediated by iodanes to produce complex natural products has been presented recently.[55]

The use of hypervalent iodine compounds in combination with transition metals or metal-catalysis has been another area of interest in organic synthesis.[56-57] One elobrative approach of metal-catalyzed hypervalent iodine mediated reactions is trifluoromethylation that allows access to wide range of valuable trifluoromethylated products.[56] Combination of hypervalent iodine reagents and transition metals such as palladium, nickel, iridium, gold, rhodium, copper, iron, ruthenium, platinum, silver, zinc, rhenium and cobalt has been successfully executed for many synthetically useful organic reactions viz. oxidation, rearrangement, amination, halogenation, amidation, ring-opening, cyclization and C−H and C−C functionalization reactions.[57] Furthermore, the development of NH transfer reactions utilizing hypervalent iodine in combination with simple sources of ammonia has expedited the synthesis of sulfoximines and sulfonimidamides.[58] Iodonitrenes (ArI=NR), are powerful reactive species, broadly used for single-nitrogen-atom insertion reaction, and skeletal editing for the construction of N-heterocycles.[59] These compounds have been considerably used to produce biologically relevant heterocycles as well as functionalized molecular architectures.

Iodonium ylides are important class of hypervalent iodine (HVI) reagents, first reported in 1957 by Neiland. In addition to traditional cycloaddition or metallocarbene-based transformations, scope of the iodonium ylides has been expanded to single electron transfer (SET) based X–H insertion reactions and radiofluorinations. Mechanistic proposal and recent reports/theories of reactions between iodonium ylides an dLewis basic nucleophiles explicitly invoke halogen bonding.[60]

Diazo compounds have well justified their position as indispensable reagents in the synthetic toolbox due to the broad spectrum of chemical transformations that they can promote. α-Diazo-λ^3-iodanes combine on the same carbon atom two exceptional functionalities; namely, an excellent leaving group and a masked carbene moiety.[61] These compounds with electron-withdrawing groups coupled to diazo methyl center have similar reactivity pattern as that of ordinary diazo compounds.[62] In the presence of rhodium catalyst, photocatalyst, or nucleophiles, these compounds can be converted into corresponding rhodium-carbenes, diazomethyl radicals, ester radicals or nucleophilic intermediates, which can be utilized as key intermediates in organic synthesis.

Asymmetric oxidative transfromations have led to a magnificient development in the last years in which establishment of chiral hypervalent iodines(III/V) have played an important role.[63-64] Many different catalytic versions have provided a fundamental advance in this area with the findings of new chiral hypervalent iodine catalysts and their applications in developing stereoselective reactions with high enantiomeric excess.[63] Besides, chiral hypoiodite species derived from onium iodites, such as, ammonium and guanidinium salts have emerged as excellent catalysts in diverse enantioselective oxidative bond-forming reactions.[65]

Photochemical transfromations of HIRs that can be achieved with or without the use of photo/metal catalysts have been another important area in the synthetic community, one such example involves, photocatalytic decarboxylation of carboxylic acids.[66] Further, hypervalent iodine reagents do absorb in the apparently non-absorbing region at higher wavelengths. This low absorbtion arises from direct spin-forbidden electronic transition from the ground state to the triplet excited state.[67] The main advantages of direct excitation approach of HIRs may involve further control over selectivity as compared to purely outer sphere radical species formation and operational simplicity.[68]

Besides, the utilization of hypervalent iodine compounds in the functionalization of carbohydrates including C–H or N–H insertion reactions, *O*-arylations, glycal functionalization, C-2 deoxy-2-iodoglyco-conjugates, iminosugars, and C-3-oxo-glycals, have been widely explored.[69]

Hypervalent iodine reagents can be regenerated using various external chemical oxidants, such as, mCPBA[70], while electrochemical regeneration[71-72] has emerged out as a sustainable approach as the requirement of chemical oxidants is eliminated with limited byproduct waste. Recently, a large number of oxidative transformations involving electrochemical regeneration of catalytic hypervalent iodine eliminating any stochiometric redox reagents for the I(I)/I(III) or (I(I)/I(V) redox cycle has been developed.[71-72]

3. Conclusions

In conclusion, this overview of recently published review articles witnesses intensifying research interest in the hypervalent iodine chemistry and synthetic applications of these compounds as reagents or as catalysts. We anticipate that utilization of these compounds as versatile synthetic tools not only in simple oxidative transformations instead in asymmetric synthesis/catalysis will show immense growth in coming years. It is also expected that the industrial/practical use of HIRs will also attract noteworthy interest in future.

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Viktor V. Zhdankin was born in Ekaterinburg, Russian Federation. His MS (1978), PhD (1981), and Doctor of Chemical Sciences (1986) degrees were earned at Moscow State University in the laboratory of Nikolay S. Zefirov. He moved to the University of Utah in 1990, where he worked for three years as Instructor of organic chemistry and Senior Research Associate with Peter J. Stang. In 1993, he joined the faculty of the University of Minnesota Duluth, where he is currently a Professor of Chemistry. Dr. Zhdankin has published more than 300 research papers, gave over a hundred research presentations in many countries, edited several books, co-authored the *Handbook of Heterocyclic Chemistry* (3rd Edition, 2010) with Professors A. R. Katritzky, C. A. Ramsden, and J. A. Joule, and authored a book on *Hypervalent Iodine Chemistry* (Wiley, 2013). His main research interests are in the areas of synthetic and mechanistic organic chemistry of hypervalent main-group elements and organofluorine chemistry. In 2011, he received the National Award of the American Chemical Society for *Creative Research & Applications of Iodine Chemistry*.