

## Hypervalent iodine compounds: reagents of the future

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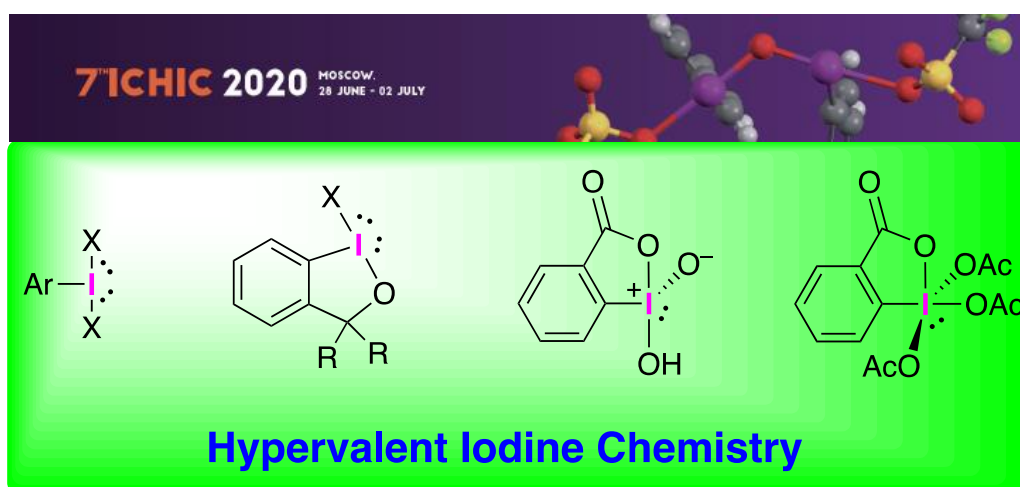
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### Abstract

This short introductory review provides a brief summary of history and recent developments in the field of hypervalent iodine chemistry in connection with the 7th International Conference on Hypervalent Iodine Chemistry (ICHIC-2021, Moscow, 27<sup>th</sup> June to 1<sup>st</sup> July, 2021). Hypervalent iodine reagents and catalysts are intensively used in modern organic chemistry as mild, environmentally safe, and economical alternative to heavy metal reagents. General features and applications of hypervalent iodine compounds are overviewed.



**Keywords:** Iodine, hypervalent iodine, iodonium, oxidation

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## 1. Introduction

Iodine is an essential element closely linked to our daily life.<sup>1</sup> In the human body, iodine is present in the thyroid gland in the form of thyroxine, a metabolism-regulating hormone. In natural organic compounds, iodine occurs exclusively in the monovalent state. However, it can form a myriad of polycoordinated compounds in different oxidation states. In modern literature, polyvalent compounds of iodine are commonly named as “hypervalent” iodine compounds, which reflects the special [3c-4e] hypervalent bonding present in these molecules.<sup>2-5</sup> Structural features and reactivity pattern of hypervalent iodine compounds in many aspects are similar to the derivatives of heavy transition metals. Reactions of iodine compounds are commonly discussed in terms of oxidative addition, ligand exchange, reductive elimination, and ligand coupling, which are typical of the transition metal chemistry. In contrast to the heavy metals, iodine is an environmentally friendly and a relatively inexpensive element. Current average bulk price of iodine varies within the range of \$10–20 per kg, which is orders of magnitude cheaper than platinum, palladium, or osmium. Organic compounds of polyvalent iodine have emerged as versatile, efficient and environmentally friendly synthetic reagents with numerous applications in academic and industrial research.<sup>6-9</sup> Hypervalent iodine compounds are widely used as mild and selective oxidants and electrophilic group transfer reagents in organic synthesis. Diaryliodonium salts have found widespread industrial application as initiators of polymerization.<sup>9</sup> This introductory review is published in connection with the 7th International Conference on Hypervalent Iodine Chemistry (ICHIC-2021, Moscow, 27<sup>th</sup> June to 1<sup>st</sup> July 2021). A brief summary of general features and applications of hypervalent iodine compounds is provided and the history and recent developments in the field of hypervalent iodine chemistry are overviewed.

## 2. Brief History of Organohypervalent Iodine Chemistry

The first polycoordinated organoiodine compound, (dichloroiodo)benzene, was reported by the German chemist C. Willgerodt in 1886.<sup>10</sup> This was followed by the preparation of (diacetoxyiodo)benzene<sup>11</sup> and iodosylbenzene<sup>12</sup> in 1892, 2-iodoxybenzoic acid (IBX) in 1893,<sup>13</sup> and diaryliodonium salts reported by Hartmann and Meyer in 1894.<sup>14</sup> In 1914 Willgerodt published a comprehensive book describing almost 500 organic compounds of polyvalent iodine known at that time.<sup>15</sup> Research activity in the area of polyvalent organoiodine compounds during the period between 1914 and 1970s was relatively low and represented mainly by valuable contributions from the laboratories of I. Masson, R. B. Sandin, F. M. Beringer, K. H.

Pausacker, A. N. Nesmeyanov, and O. Neilands. Two significant reviews were published during this period: the reviews by Sandin<sup>16</sup> and Banks<sup>17</sup> published in Chemical Reviews in 1943 and 1966.

Since the end of 1970s – beginning of 1980s the interest in polyvalent organoiodine compounds has experienced a renaissance. This resurgence of interest in polyvalent organic iodine has been initiated by the discovery of several new classes of polyvalent organoiodine compounds and, most notably, by the development of useful synthetic applications of some of these compounds, which are now regarded as valuable organic reagents known under general name of hypervalent iodine reagents. The foundation of modern hypervalent iodine chemistry was laid out in the 1980s by the groundbreaking works of G. F. Koser, J. C. Martin, R. M. Moriarty, P. J. Stang, A. Varvoglis, and N. S. Zefirov.

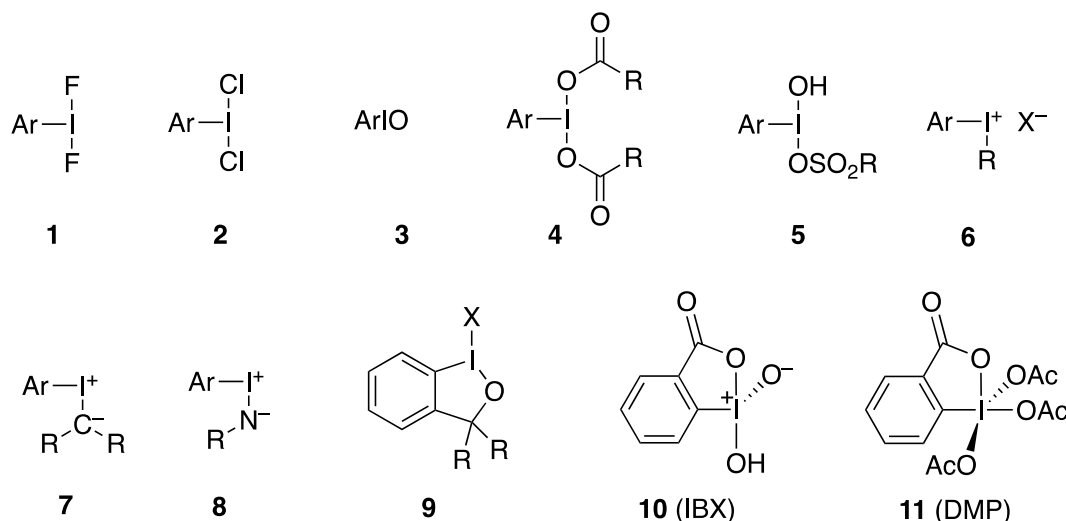
Important contributions to the development of hypervalent iodine chemistry in the 1990s had been made by the research groups of A. Varvoglis, N. S. Zefirov, L. M. Yagupolskii, A. R. Katritzky, R. A. Moss, J. C. Martin, D. H. R. Barton, R. M. Moriarty, G. F. Koser, P. J. Stang, H.-J. Frohn, T. Umemoto, M. Yokoyama, Y. Kita, M. Ochiai, T. Okuyama, T. Kitamura, H. Togo, E. Dominguez, I. Tellitu, J. D. Protasiewicz, A. Kirschning, K. S. Feldman, T. Wirth, S. Quideau, S. Hara, N. Yoneda, L. Skulski, S. Spyroudis, V. V. Grushin, V. W. Pike, D. A. Widdowson, and many others. During the 1980s – 1990s, hypervalent iodine research had been summarized in several reviews and books. Most notable were the two books published in 1992 and 1997 by A. Varvoglis: a comprehensive monograph “The Organic Chemistry of Polycoordinated Iodine”<sup>18</sup> and a book on the application of hypervalent iodine compounds in organic synthesis.<sup>19</sup> Several general reviews and numerous book chapters and specialized reviews were also published during 1980s and 1990s.

Since the beginning of the 21st century, the chemistry of organohypervalent iodine compounds has experienced an explosive development. Five books<sup>1-5</sup> and several hundred reviews summarizing various aspects of hypervalent iodine chemistry have been published since the year of 2000, and hundreds (if not thousands) of research works utilizing hypervalent iodine reagents are published every year. Since 2001, the International Conference on Hypervalent Iodine Chemistry (ICHIC) has been convened on a regular basis. Previous ICHIC conferences were held in Kallithea, Greece (12-14 September 2001), Thessaloniki, Greece (1-2 June 2006), Bordeaux, France (4-7 July 2010), Chiba, Japan (2-5 July 2014), Les Diablerets, Switzerland (3-6 July 2016), and Cardiff, UK (1-4 July 2018). The 7th International Conference on Hypervalent Iodine Chemistry (ICHIC-2021) will be held in Moscow State University, Russia, from 27<sup>th</sup> June to 1<sup>st</sup> July 2021. ICHIC-2021 will be dedicated to the memory of Prof. Nikolay S. Zefirov (1935-2017) who made a significant contribution to the field of hypervalent iodine chemistry. Similar to previous ICHIC conferences, the scientific program of ICHIC-2021 covers all key topics of hypervalent iodine chemistry including: (1) Synthesis, structure and bonding, (2) Applications in organic synthesis, (3) Mechanistic studies, (4) Coordination chemistry, (5) Applications in materials science, (6) Theoretical and spectroscopic studies.

### 3. General classification of hypervalent iodine compounds

Organoiodine(III) compounds are commonly classified by the type of ligands attached to the iodine atom. The following general classes of polyvalent iodine compounds have found broad application as reagents in organic synthesis: (difluoroiodo)arenes **1**, (dichloroiodo)arenes **2**, iodosylarenes **3**, [bis(acyloxy)iodo]arenes **4**, aryl iodine(III) organosulfonates **5**, iodonium salts **6**, iodonium ylides **7**, iodonium imides **8**, and benziodoxole derivatives **9-11** (Figure 1). The most important and commercially available representatives of aryl iodine(III) carboxylates **4** are represented by (diacetoxyiodo)benzene  $\text{PhI}(\text{OAc})_2$ , which is usually abbreviated as PIDA (phenyliodine diacetate), and by [bis(trifluoroacetoxy)iodo]benzene  $\text{PhI}(\text{OCOCF}_3)_2$ , abbreviated as PIFA

[(phenyliodine bis(trifluoroacetate))]. The most important representative of aryl iodine(III) organosulfonates **5**, the commercially available [hydroxy(tosyloxy)iodo]benzene  $\text{PhI}(\text{OH})\text{OTs}$ , is abbreviated as HTIB and is also known as Koser's reagent.



**Figure 1.** Common classes of hypervalent iodine compounds.

Polyvalent iodine compounds **1-11** have found wide application in organic synthesis as efficient reagents for various oxidative transformations. (Difluoroiodo)arenes **1** and (dichloroiodo)arenes **2** are effective fluorinating and chlorinating reagents, respectively. Iodosylarenes **3**, aryl iodine(III) carboxylates **4** and organosulfonates **5** are strong oxidizing agents and have found widespread application as reagents for oxygenation and oxidative functionalization of organic substrates. Iodonium salts **6** in general do not possess significant oxidizing properties, but have a diverse reactivity pattern mainly due to the exceptional leaving group ability of the iodoarene fragment. Iodonium ylides **7** and imides **8** are excellent carbene and nitrene precursors, respectively. Benziodoxoles **9** have a considerably higher stability compared to their acyclic analogs, which made possible the development of several valuable group transfer reagents. The most important organoiodine(V) compounds are represented by IBX (**10**) and DMP (**11**) which have found broad application as efficient oxidizing reagents.

#### 4. Recent developments and future perspectives

Current surging interest in hypervalent iodine chemistry is driven mainly by the very useful oxidizing properties of iodine(III) and iodine(V) reagents, combined with their benign environmental character and commercial availability. The development of numerous new synthetic methodologies has been inspired by exploration and practical utilization of the similarities between hypervalent iodine and derivatives of heavy transition metals. In particular, hypervalent iodine reagents and catalysts can effectively promote coupling reactions leading to the formation of new C–C, C–N and other carbon-element bonds. The utilization of hypervalent iodine reagents for the construction of carbon-carbon bonds was surveyed in two reviews.<sup>20,21</sup> Numerous recent reviews have summarized reactions of hypervalent iodine reagents leading to the formation of new C–N bonds.<sup>22-26</sup> Fluorinations and other oxidative halogenations triggered by hypervalent iodine(III)

compounds have been demonstrated to be synthetically useful reactions with excellent selectivities.<sup>27,28</sup> The use of iodine(III) species allowing facile formation of various heterocyclic system via oxidative formation of C–C, C–N, C–O, C–S, N–N, N–O, or N–S bonds has recently been reviewed.<sup>29-34</sup> Hypervalent iodine mediated oxidative methodologies have found wide application in total synthesis of natural products.<sup>35,36</sup>

The discovery of catalytic activity of iodine compounds in numerous oxidative transformations is one of the most impressive recent achievements. The development of highly efficient, enantioselective molecular catalysts based on the unique redox chemistry of iodine has added a new dimension to the field of hypervalent iodine chemistry and initiated a major surge of research activity.<sup>37-40</sup>

Numerous recent reviews have been dedicated to synthetic applications of several specific classes of hypervalent iodine compounds. Aryliodonium salts have attracted significant interest as electrophilic arylating reagents<sup>41-47</sup> and precursors for Positron Emission Tomography (PET).<sup>48,49</sup> Iodonium ylides have also been recently utilized as PET precursors for nucleophilic radiofluorination.<sup>50,51</sup>

The development of the group-transfer benziiodoxole-based reagents is one of the most significant recent achievements in the field of hypervalent iodine chemistry.<sup>52-55</sup> Trifluoromethylbenziiodoxoles have received the status of common trifluoromethylating reagents.<sup>52-57</sup> Ethynylbenziiodoxoles are useful alkynylating reagents.<sup>58-60</sup> Azidobenziiodoxoles are the most effective reagents for radical azidation.<sup>52-55,61,62</sup> In general, radical and photochemical reactions of hypervalent iodine compounds belong now to one of the hottest areas of modern hypervalent iodine chemistry.<sup>63,64</sup>

Numerous new, useful hypervalent iodine reagents have recently been developed, such as: the most powerful iodine(V) oxidants,<sup>65</sup> pseudocyclic hypervalent iodine reagents with improved physical properties and reactivity pattern,<sup>66,67</sup> benzyne precursors that are triggered by water at room temperature,<sup>45</sup> and water-soluble IBX derivatives.<sup>68</sup>

The development of new synthetic methodologies and the use of hypervalent iodine reagents in specific classes of reactions have attracted significant research activity. Examples of recent reviews dedicated to specific reactions include the following works: applications of hypervalent iodine(III) reagents in direct C–H bond functionalization,<sup>69</sup> iodosobenzene-mediated construction of heterocyclic scaffolds,<sup>70</sup> functionalization of alkenes using hypervalent iodine reagents,<sup>71,72</sup> hypervalent iodine-mediated synthesis of spiroheterocycles,<sup>73</sup> organocatalytic approaches used to promote group transfer from hypervalent iodine species,<sup>74</sup> phenol dearomatization reactions using hypervalent iodine reagents,<sup>75,76</sup> rearrangements induced by hypervalent iodine,<sup>77</sup> applications of hypervalent iodine(III) reagents in organophosphorus chemistry,<sup>78</sup> hypervalent iodine reagents in transition metal chemistry,<sup>79</sup> and many other works. Theoretical studies of the mechanisms of catalytic and stoichiometric iodine-mediated reactions has been discussed in numerous publications.<sup>80-87</sup>

New methods for generation of hypervalent iodine compounds using green chemistry approaches have recently been developed. In particular, the anodic oxidation of iodoarenes can serve as efficient method for the synthesis of hypervalent iodine reagents, eliminating the necessity to use hazardous chemical oxidants. The hypervalent iodine species generated at the anode can be utilized as either in-cell or ex-cell mediators for various valuable oxidative transformations such as fluorinations and oxidative cyclizations.<sup>88</sup> The aerobic synthesis of iodine(III) and iodine(V) reagents represents another environmentally sustainable procedure. This method is based on chemical interception of reactive intermediates generated during aldehyde autooxidation accomplishing the oxidation of aryl iodides. The aerobically generated hypervalent iodine intermediates can be utilized in an array of oxidative functionalizations of organic substrates.<sup>89</sup>

The industrial applications of hypervalent iodine chemistry at present are limited mainly to the use of hypervalent iodine oxidants in kilogram-scale syntheses of various pharmaceuticals and utilization of

diaryliodonium salts in polymer science and industry.<sup>2,4,8</sup> In particular, iodonium salts have found broad industrial application as initiators of polymerization.<sup>9,90,91</sup> It is expected that the interest in practical applications of hypervalent iodine compounds will significantly grow in the future.

## 5. Conclusions

In conclusion, this brief survey of recently published books and reviews demonstrates increasing research activity in different areas of hypervalent iodine chemistry. Hypervalent iodine reagents and synthetic methodologies involving hypervalent iodine species have become essential tools of modern organic synthesis. We anticipate that the inspiring chemistry of hypervalent iodine compounds will continue to attract significant interest and research activity in the future.

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## Author's Biography



**Viktor V. Zhdankin** was born in Ekaterinburg, Russian Federation. His M.S. (1978), Ph.D. (1981), and Doctor of Chemical Sciences (1986) degrees were earned at Moscow State University. 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 Professor Peter J. Stang. In 1993, he joined the faculty of the University of Minnesota Duluth,

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