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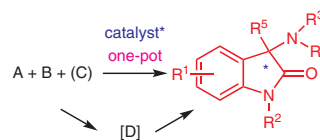
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Synthesis of Chiral 3-Substituted 3-Amino-2-oxindoles through Enantioselective Catalytic Domino and Tandem Reactions

Hélène Pellissier* 



1 Introduction

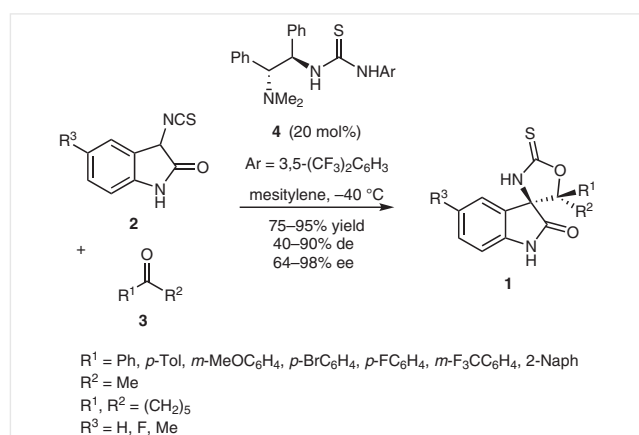
Chiral oxindoles are widely present in nature and exhibit many biological activities.¹ Among them, chiral 3-substituted 3-amino-2-oxindoles exhibiting a quaternary stereogenic center at the 3-position have been recognized as privileged substructures in new drug discovery. For example, their skeleton is present in many bioactive molecules and drug candidates for the treatment of malaria and stress related disorders as well as in marine alkaloids, such as the chartellines or in terrestrial alkaloids isolated from the leaves of the Malaysian plant *Psychotria rostrate*. The biological activity of these compounds is greatly affected by the nature of the substituent at the C3 position as well as the absolute configuration of the stereogenic center. Consequently, the development of efficient methods to synthesize such products is of great importance and constitutes a current open area of research in asymmetric catalysis,² with a special mention for organocatalysis. The principal methodology to prepare chiral quaternary 3-amino-2-oxindoles is based on enantioselective catalytic nucleophilic additions to isatin imines, such as Mannich reactions, aza-Morita–Baylis–Hillman reactions, aza-Henry reactions, additions of heteronucleophiles, Strecker reactions, or hydrophosphonylations. In addition to these classical methodologies, a range of more modern asymmetric catalytic domino

and tandem reactions have been recently developed, allowing many 3-substituted 3-amino-2-oxindoles to be achieved in high enantioselectivities. These one-pot transformations have been promoted with various chiral organocatalysts, but also chiral metal catalysts. The goal of this review is to collect the major developments in the catalytic asymmetric synthesis of 3-substituted 3-amino-2-oxindoles through enantioselective catalytic domino and tandem reactions reported since 2011. It is divided into two parts, dealing successively with enantioselective organocatalyzed reactions, and enantioselective metal-catalyzed transformations. The economic interest in combining asymmetric catalysis with the concept of domino³ and tandem reactions is obvious, and has allowed high molecular complexity to be easily reached with remarkable levels of stereocontrol on the basis of simple operational one-pot procedures, and advantages of savings in solvent, time, energy, and costs. In the last decade, an increasing number of these fascinating one-pot processes catalyzed by various types of chiral organocatalysts as well as chiral metals have been developed.⁴

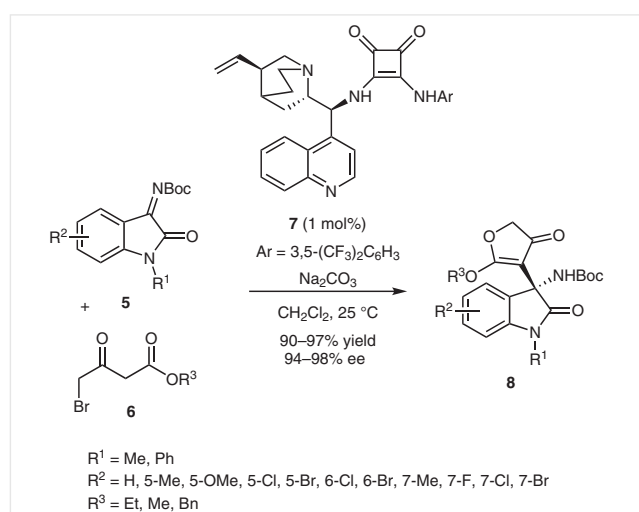
2 Organocatalyzed Reactions

Among chiral 3-substituted 3-amino-2-oxindoles, many alkaloids exhibit a spirocyclic structure.⁵ In 2011, Yuan and co-workers described the synthesis of chiral spirooxindoles **1** based on an enantioselective organocatalytic domino aldol/cyclization reaction of **2** with **3** (Scheme 1).⁶ The reaction was catalyzed at $-40\text{ }^{\circ}\text{C}$ by organocatalyst **4** in mesitylene as solvent, leading to the corresponding highly functionalized chiral domino products **1** in good to high yields (75–95%) combined with moderate to high diastereoselectivities (40–90% de) and enantioselectivities (64–98% ee). The optimal substrates were found to be acetophenones.

In 2014, Wu and co-workers reported the enantioselective domino Mannich/cyclization reaction of isatin imines **5** with 4-bromo-3-oxobutanoates **6** catalyzed by catalyst **7**



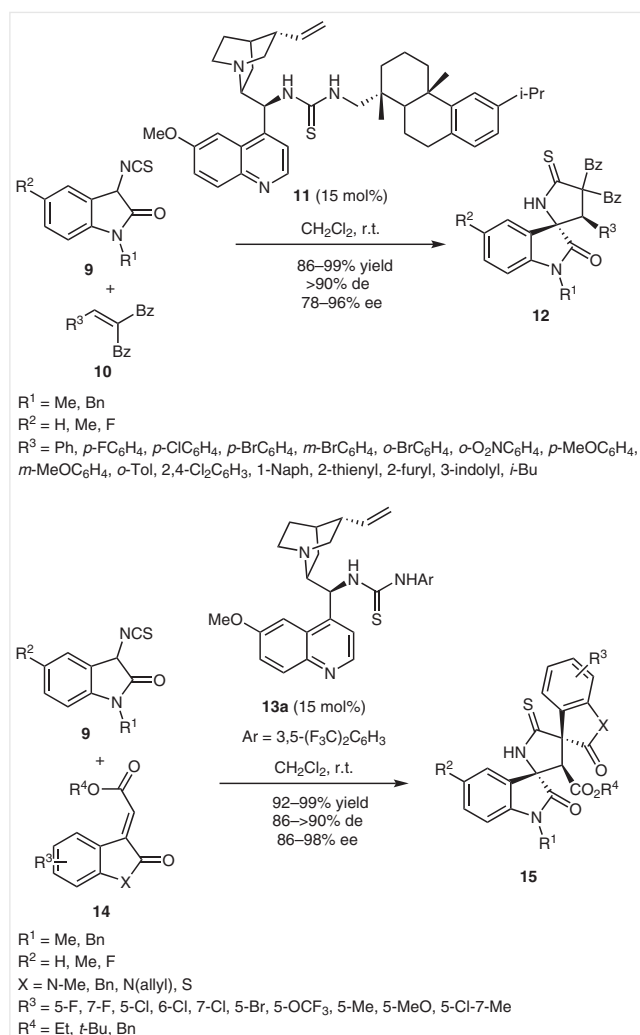
Scheme 1 Domino aldol/cyclization reaction of 3-isothiocyanato-2-oxindoles with ketones catalyzed by a thiourea



Scheme 2 Domino Mannich/cyclization reaction of isatin imines with 4-bromo-3-oxobutanoates catalyzed by a cinchona alkaloid-derived squaramide

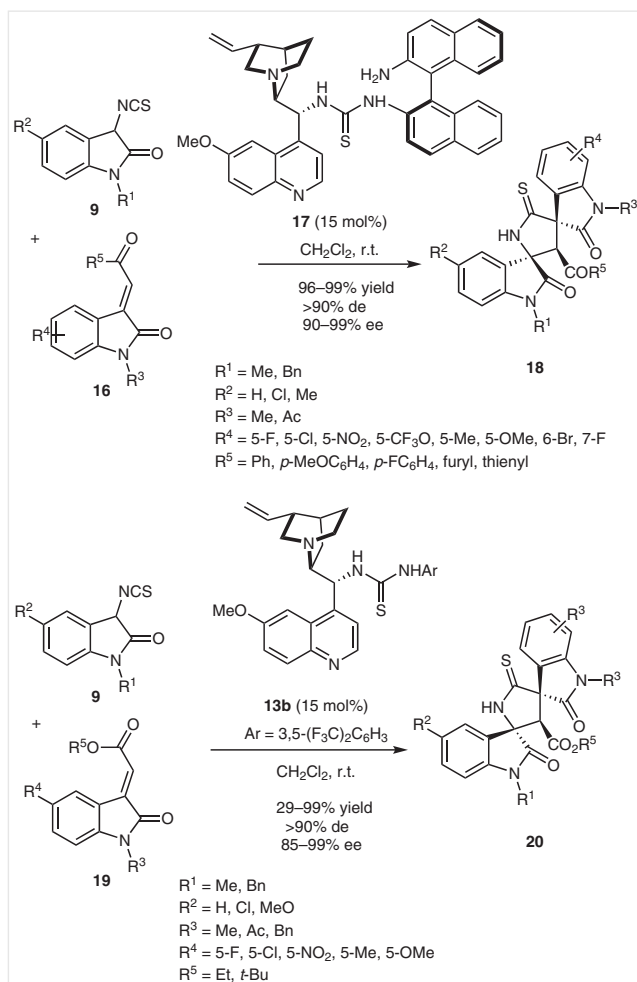
(Scheme 2).⁷ The corresponding highly functionalized chiral domino products **8** were obtained in both excellent yields (90–97%) and enantioselectivities (94–98% ee).

Enantioselective organocatalytic domino Michael/cyclization reactions have been recently developed. For example, in 2013 Wang and co-workers reported a highly diastereo- and enantioselective domino Michael/cyclization reaction of 3-isothiocyanato-2-oxindoles **9** with electron-deficient alkenes **10** catalyzed by **11** (Scheme 3).⁸ The reaction was performed in dichloromethane at room temperature and led to the corresponding chiral products **12** as almost single diastereomers (>90% de) in high to quantitative yields (86–99%) and good to excellent enantioselectivities (78–96% ee). By employing another chiral cinchona alkaloid-derived thiourea catalyst such as **13a**, the authors developed the domino reaction of **9** with 3-methylene-2-oxindoles **14** (Scheme 3), which provided the corresponding



Scheme 3 Domino Michael/cyclization reactions of 3-isothiocyanato-2-oxindoles with electron-deficient alkenes and 3-methylene-2-oxindoles catalyzed by cinchona alkaloid derived thioureas

chiral spirocyclic oxindoles **15** with uniformly excellent yields (92–99%), good to excellent diastereoselectivities (86–>90% de), and high enantioselectivities (86–98% ee).

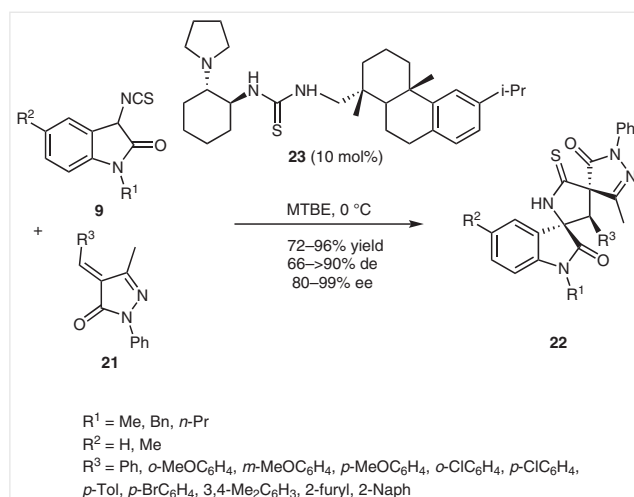


Scheme 4 Domino Michael/cyclization reactions of 3-isothiocyanato-2-oxindoles with 3-methylene-2-oxindoles catalyzed by cinchona alkaloid derived thioureas

Concurrently with this, Huang, Wang, and co-workers also developed the reactions between 3-isothiocyanato-2-oxindoles **9** with 3-methylene-2-oxindoles **16** performed in the presence of a trifunctional catalyst **17** (Scheme 4) to provide the corresponding chiral spirocyclic oxindoles **18** in uniformly excellent yields (96–99%), diastereoselectivity (>90% de), and enantioselectivities (90–99% ee).⁹ On the other hand, catalyst **13b** (Scheme 4) emerged as the optimal catalyst to promote the reaction between 3-methylene-2-oxindoles **19** bearing an ester moiety with **9** to give the corresponding domino products **20** in low to quantita-

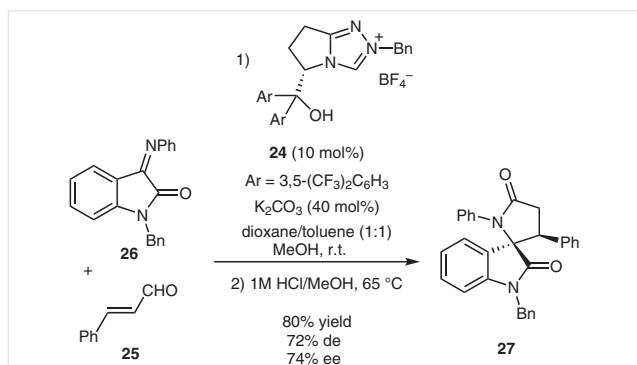
tive yields (29–99%), excellent diastereoselectivity, (>90% de), and high enantioselectivities (85–99% ee).

In 2013, an asymmetric domino Michael/cyclization reaction between 3-isothiocyanato-2-oxindoles **9** and various aryl-substituted 4-methylene-3H-pyrazol-3-ones **21** was reported by Wang and co-workers, providing the corresponding chiral products **22** exhibiting three contiguous stereogenic centers (Scheme 5).¹⁰ The process was catalyzed by **23**, and afforded these complex highly functionalized polycyclic products in good to quantitative yields (72–96%), moderate to excellent diastereoselectivities (66–>90% de), and high enantioselectivities (80–99% ee). In the case of alkyl-substituted 4-methylene-3H-pyrazol-3-ones, only low enantioselectivities were observed (11% ee) along with high yield (82%) and excellent diastereoselectivity (90% de).



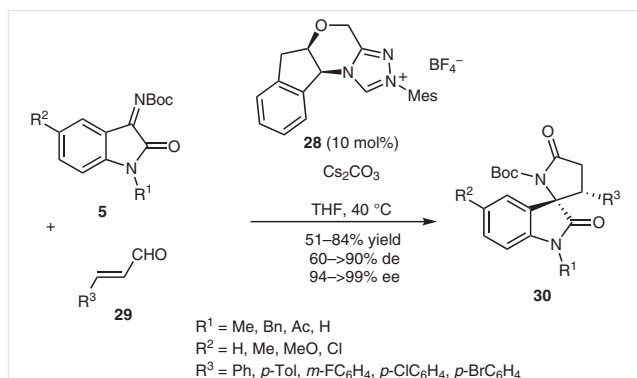
Scheme 5 Domino Michael/cyclization reaction of 3-isothiocyanato-2-oxindoles with 4-(arylmethylene)-3H-pyrazol-3-ones catalyzed by a tertiary amine-thiourea

The utility of N-heterocyclic carbenes (NHCs) as organocatalysts in domino/tandem reactions has received growing attention in the past few years.¹¹ In 2012, Jiao, Ye, and co-workers demonstrated the application of this type of organocatalysts for the addition of α,β -unsaturated aldehydes to N-arylisatin imines to develop a one-pot procedure for the synthesis of chiral spirocyclic γ -lactam oxindoles.¹² Therefore, by using 10 mol% of a NHC catalyst such as **24**, the homoenolate equivalent of **25** was added to an isatin imine **26** to give, after subsequent acid hydrolysis, the corresponding spirocyclic γ -lactam oxindole **27** in 80% yield, 72% de, and 74% ee (Scheme 6). The mechanism of this domino reaction involves the addition of the NHC catalyst to the α,β -unsaturated aldehyde to give a Breslow intermediate serving as homoenolate equivalent. Then, the latter adds to the isatin imine to give an intermediate, which then cyclizes to afford the final product.



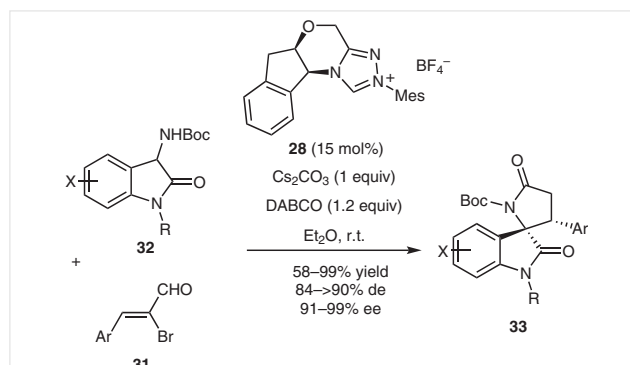
Scheme 6 NHC-catalyzed domino addition/cyclization reaction of an *N*-phenylisatin imine with cinnamaldehyde

This type of reactions was also investigated by Chi and co-workers by using another NHC catalyst such as **28** (Scheme 7).¹³ Thus, the reaction of various α,β -unsaturated aldehydes **29** with a series of *N*-aryl isatin imines **5** led to the corresponding chiral spirocyclic- γ -lactams **30** in moderate to good yields (51–84%), moderate to high diastereoselectivities (60–>90% de), and uniformly excellent enantioselectivities (94–>99% ee).



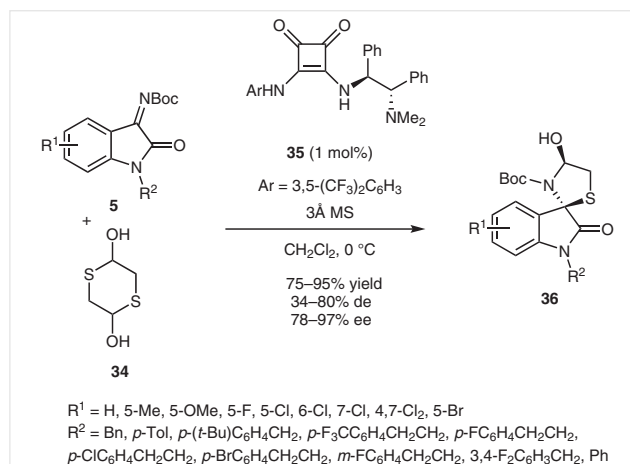
Scheme 7 NHC-catalyzed domino addition/cyclization reaction of isatin imines with α,β -unsaturated aldehydes

In 2016, Ye, Sun, and co-workers reported enantioselective NHC-catalyzed formal [3+2] annulations occurring between α -bromoenals **31** and 3-amino-2-oxindoles **32** (Scheme 8).¹⁴ The domino process was catalyzed at room temperature by 15 mol% of the same NHC catalyst **28** in the presence of two bases, such as Cs_2CO_3 and DABCO. The reaction occurs through the Michael addition of the 3-amino-2-oxindole via its enolate to an α,β -unsaturated acyl azolium, generated from the α -bromoenal and the catalyst, to give an intermediate which subsequently cyclizes through lactamization to give the final chiral product **33**. A series of these cycloadducts **33** were synthesized in moderate to quantitative yields (58–99%), high to complete diastereoselectivities (84–>90% de), and uniformly excellent enantioselectivities (91–99% ee).



Scheme 8 NHC-catalyzed domino Michael/lactamization reaction of 3-amino-2-oxindoles with α -bromoenals

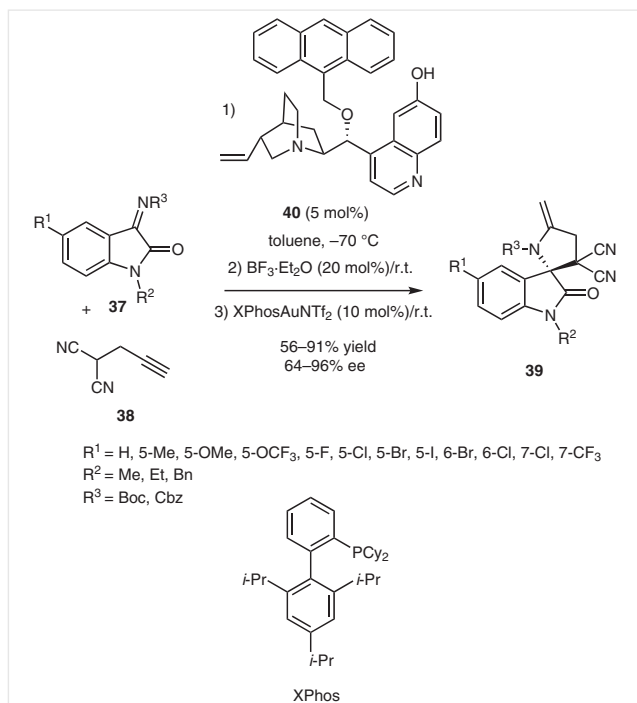
The same year, Li, Li, and co-workers described an efficient asymmetric [3+2] annulation reaction between isatin imines **5** and 1,4-dithiane-2,5-diol (**34**) as equal equivalent of 2-mercaptoacetaldehyde (Scheme 9).¹⁵ The domino reaction catalyzed by **35** begins with the addition of **34** to **5**, leading to an aldehyde intermediate that subsequently cyclizes into the corresponding chiral product **36**. A range of these products were achieved in high yields (75–95%), low to good diastereoselectivities (34–80% de), and good to high enantioselectivities (78–97% ee).



Scheme 9 Domino addition/cyclization reaction of isatin imines with 1,4-dithiane-2,5-diol catalyzed by a squaramide

Earlier in 2013, an asymmetric tandem Mannich/hydroamination reaction was developed by Jiang, Liu, and co-workers.¹⁶ It occurred between isatin imines **37** and propargylated malononitrile **38** (Scheme 10), affording the corresponding densely functionalized chiral tricyclic products **39** in moderate to high yields (56–91%) and enantioselectiv-

ities (64–96% ee). This multicyclic process involved the sequential treatment of the substrates with chiral cinchona alkaloid catalyst **40**, $\text{BF}_3 \cdot \text{Et}_2\text{O}$, and XPhosAuNTf_2 .

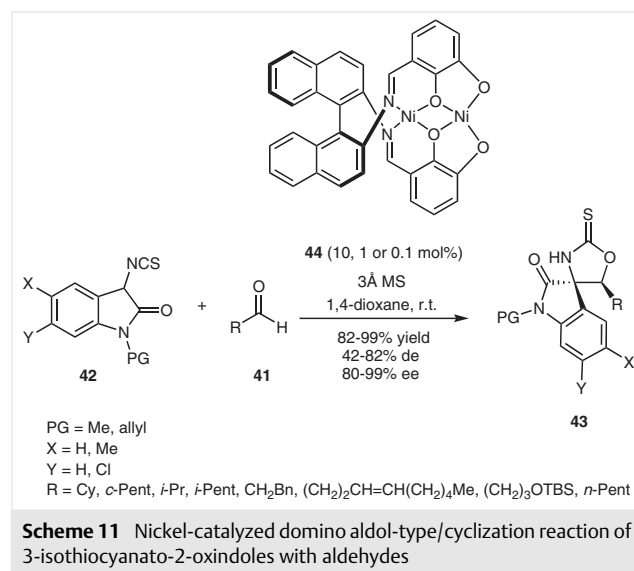


Scheme 10 Tandem multicyclic Mannich/hydroamination reaction of isatin imines with a propargylated malononitrile

3 Metal-Catalyzed Reactions

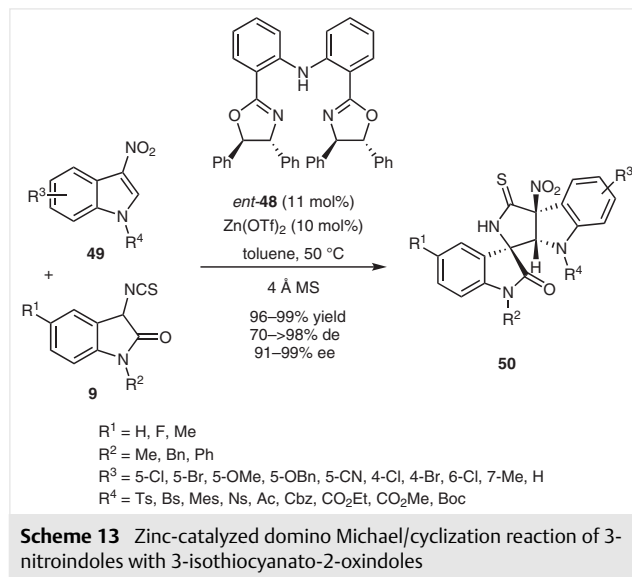
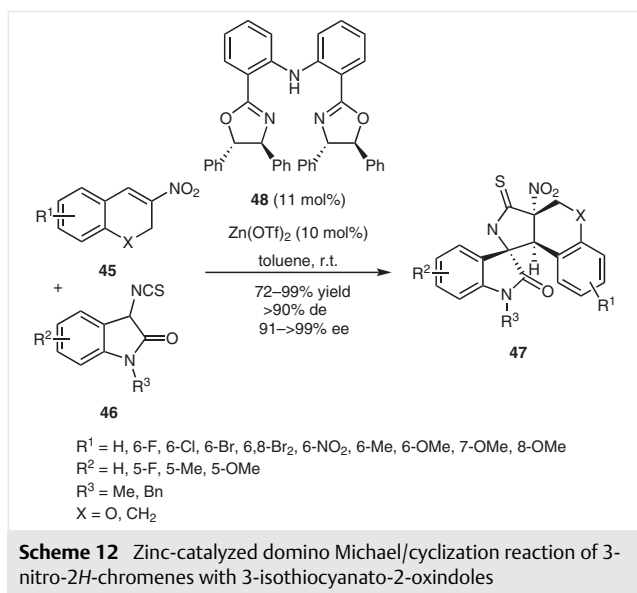
In 2013, Matsunaga, Kanai, and Kato reported the first catalytic asymmetric addition of 3-isothiocyanato-2-oxindoles to aldehydes by using a chiral dinuclear nickel Schiff base catalyst (Scheme 11).¹⁷ The reaction of a range of aliphatic aldehydes **41** with 3-isothiocyanato-2-oxindoles **42** afforded the corresponding spirooxindoles **43** in high yields and diastereo- and enantioselectivities of up to 99%, 82% de, and 99% ee, respectively. The process, evolving through a domino aldol-type/cyclization reaction, was generally promoted at room temperature by 10 mol% of **44** in 1,4-dioxane as solvent. It was demonstrated that the catalyst loading could be reduced to 0.1 mol% still providing a remarkable enantioselectivity of up to 98% ee (PG = Me, X = Y = H, R = *n*-Pent). It was found that the dinuclear nickel catalyst was much more efficient than the corresponding dinuclear copper and cobalt complexes, which provided lower enantioselectivities (2–21% ee). The scope of the domino aldol-type/cyclization reaction showed that α -branched aliphatic aldehydes led to the corresponding chiral tricyclic products in 66–78% de and 80–92% ee, while linear aliphatic aldehydes exhibited slightly higher diastereo- and enantioselectivities than the α -branched ones, providing the corresponding products in 88–99% ee and up to 82% de. In con-

trast to aliphatic aldehydes, poor results were obtained with aromatic aldehydes. For example, a low enantioselectivity of 33% ee was observed in the reaction of benzaldehyde with unsubstituted *N*-methyl-2-oxindole **42** (X = Y = H, PG = Me) in combination with a low diastereoselectivity of 10% de.



In 2014, Xiao and co-workers developed an unprecedented zinc-catalyzed asymmetric domino Michael/cyclization reaction of 3-nitro-2H-chromenes **45** with 3-isothiocyanato-2-oxindoles **46**.¹⁸ This reaction constituted an efficient access to various synthetically important polycyclic spirooxindoles **47** in a highly stereoselective manner under mild conditions. Remarkably, these complex and densely functionalized chiral products **47**, which exhibited three consecutive stereogenic centers including 1,3-nonadjacent tetrasubstituted carbon stereocenters, were achieved as almost single diastereomers (>90% de in all cases) in good to quantitative yields (72–99%) and with general excellent enantioselectivities of 91–>99% ee (Scheme 12). These results were obtained by using a combination of $\text{Zn}(\text{OTf})_2$ with ligand **48** bearing a free NH group, which could act as a Lewis base through hydrogen-bonding interaction.

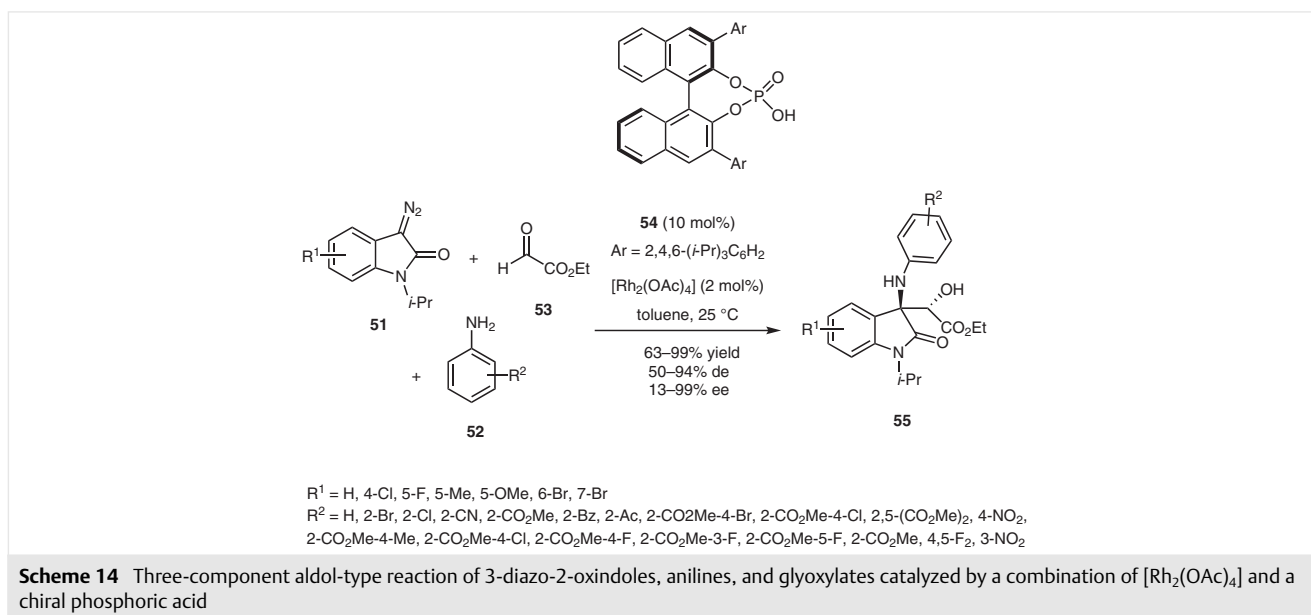
In 2015, Xu and Yuan reported related asymmetric domino Michael/cyclization reactions by using the (*R,R*)-enantiomer of the same ligand **ent-48**.¹⁹ The reaction occurred between 3-isothiocyanato-2-oxindoles **9** and 3-nitroindoles **49** to afford the corresponding chiral polycyclic derivatives **50** exhibiting three contiguous stereocenters. For example, the reaction of *N*-methyl protected 3-isothiocyanato-2-oxindoles performed in the presence of the (*R,R*)-bisoxazoline ligand combined with $\text{Zn}(\text{OTf})_2$ as precatalyst in toluene at 50 °C led to **50** in quantitative yields as single stereoisomers (>98% de and >99% ee) in almost all cases of substrates examined (Scheme 13).

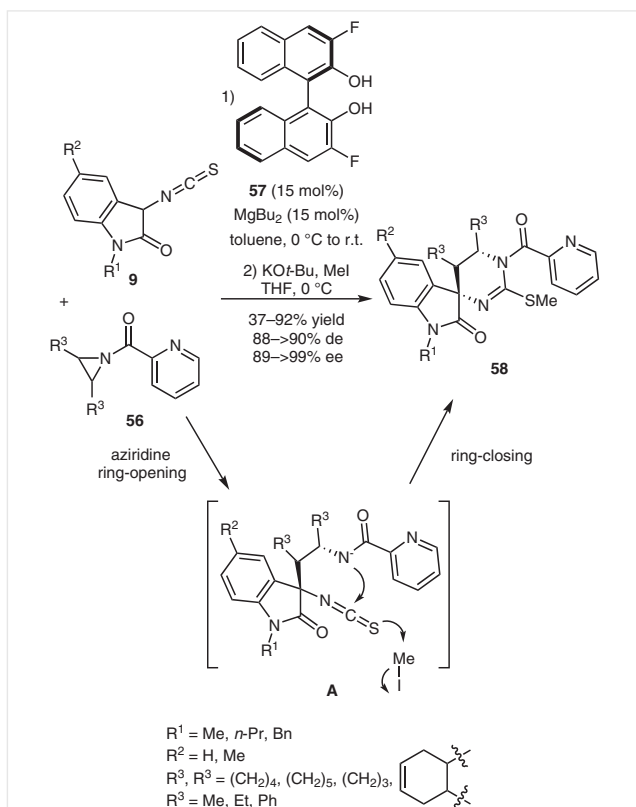


In 2013, a synthesis of chiral 3-amino-2-oxindoles was developed by Gong and co-workers on the basis of a multi-catalytic asymmetric three-component aldol-type reaction of 3-diazo-2-oxindoles **51** with anilines **52** and glyoxylate **53** (Scheme 14).²⁰ This domino process was catalyzed by a rhodium complex, such as $[\text{Rh}_2(\text{OAc})_4]$, combined with chiral phosphoric acid **54**. The reaction proceeded via rhodium-catalyzed generation of ammonium ylides from 3-diazo-2-oxindoles and anilines, followed by chiral Brønsted acid-catalyzed enantioselective aldol-type reaction with glyoxylates to give the corresponding chiral products **55** in

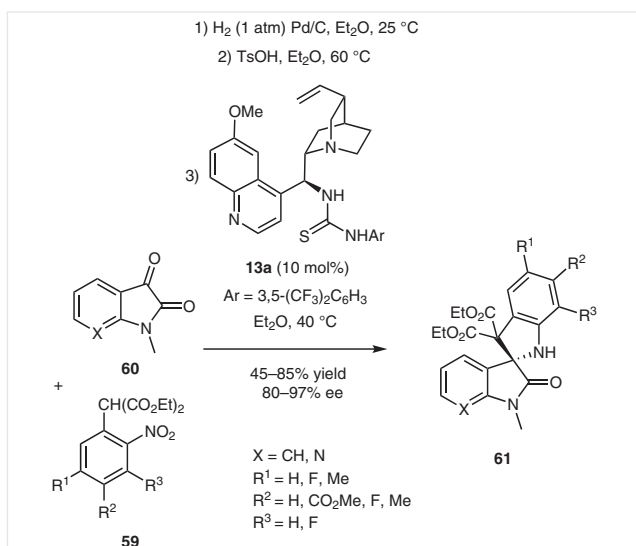
moderate to quantitative yields (63–99%), moderate to high diastereoselectivities (50–94% de), and low to excellent enantioselectivities (13–99% ee).

In 2015, an enantioselective magnesium-catalyzed tandem reaction was reported by Wang and co-workers, involving 3-isothiocyanato-2-oxindoles **9** and aziridines **56** as substrates.²¹ It constituted the first asymmetric formal [3+3] cycloaddition with aziridines. This tandem reaction was mediated by a magnesium catalyst in situ generated from MgBu_2 and ligand **57** in toluene at 0 °C to room temperature. It begins with the ring-opening of aziridines with 3-isothiocyanato-2-oxindoles to give intermediates **A** which subsequently cyclize into the final products **58** by





Scheme 15 Magnesium-catalyzed tandem ring-opening/ring-closing reaction of 3-isothiocyanato-2-oxindoles with *N*-(2-pyridyl)carbon-yl)aziridines



Scheme 16 Multicatalyzed tandem hydrogenation/imine formation/[1,5]-electrocyclic reaction of malonate-nitroarenes with isatin derivatives

treatment with *t*-BuOK/MeI (Scheme 15). This novel tandem ring-opening/ring-closing reaction allowed a range of densely functionalized chiral products **58** to be synthesized in moderate to high yields (37–92%) with uniformly high diastereo- and enantioselectivities (88–>90% de and 89–>99% ee, respectively).

In 2014, a synthesis of chiral 3-amino-2-oxindoles was based by Zhou and co-workers on a rare example of one-pot process involving asymmetric triple relay catalysis (Scheme 16).²² The sequence began with the hydrogenation of **59** into the corresponding malonate-aniline derivative. Then, this compound reacted with an isatin derivative **60** in the presence of TsOH to give the corresponding ketimine. When the latter was submitted to catalyst **13a**, it underwent a [1,5]-electrocyclic reaction to afford the corresponding chiral products **61** in moderate to good yields (45–85%) and high enantioselectivities (80–97% ee).

4 Conclusion

Chiral 3-amino-2-oxindoles are important and ubiquitous motifs in many natural products and biologically active compounds with a range of significant pharmaceutical properties. Along with more traditional methodologies to prepare these compounds, such as enantioselective nucleophilic additions to isatin imines and direct aminations of 3-substituted 2-oxindoles, this review demonstrates that more recently developed enantioselective catalytic domino and tandem reactions of various types are also highly efficient to this aim. These one-pot processes have been achieved with often excellent enantioselectivities by using both chiral metal and organocatalysts of many types. In particular, the catalytic potential of chiral organocatalysts, including cinchona alkaloids, phosphoric acids, thioureas, squaramides, and *N*-heterocyclic carbenes among others, has been successfully exploited in the last few years. Remarkable enantioselectivities of up to 99% ee have also been described in reactions catalyzed by metal complexes derived from magnesium, zinc, rhodium, and nickel. Furthermore, excellent enantioselectivities of up to 97% ee were also described in several enantioselective multicatalyzed tandem reactions. In the near future, the ever-growing need for achieving these biologically important products will prompt organic chemists to develop other enantioselective catalytic domino and tandem reactions. Indeed, designing novel catalytic asymmetric transformations by different combination of substrates and catalysts will increase the library of potentially bioactive 3-amino-2-oxindoles. In this context, a great challenge will be to design and develop efficient (organo)catalysts providing high stereoselectivity at low catalyst loading.

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