Asymmetric Synthesis of BINOL Derivatives

Subjects: Chemistry, Organic

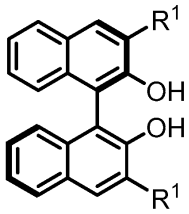
Contributor: Everton Machado da Silva , Hérika Danielle Almeida Vidal , Marcelo Augusto Pereira Januário , Arlene Gonçalves Corrêa

The chirality resulting from restricted rotation around a single bond is called atropisomerism (axial chirality). This phenomenon was first described by Christie and Kenner in 1922 when investigating the biaryl 6,6'-dinitro-2,2'-diphenic acid, and the term "atropisomer", derived from the Greek where "a" means "not" and "tropes" means "turn", was created by Kuhn. Atropisomers belong to the class of axially chiral compounds; however, here, the enantiomers exist due to restricted rotation around a single bond. Axial chirality has also been considered as an important structural element of many natural products and bioactive compounds, whose enantiomers generally exhibit different pharmacological activities and metabolic processes in vivo and in vitro.

BINOL chirality transfer metal-mediated enantioselective coupling organocatalysis

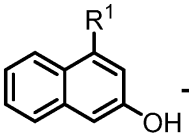
1. Metal-Mediated Oxidative Enantioselective Coupling

Among the available methods for the synthesis of optically active BINOLs, one of the most explored is the oxidative dimerization of 2-naphthols mediated by complexes of Cu [1][2][3][4][5][6][7][8] Fe [9][10][11], V [12][13][14][15][16][17], Ru [18], and chiral ligands (very often amines), normally generated in situ. In this regard, excellent reviews discussing these methods have been reported by Brunel [19], Wang [20], Bryliakov et al. [21], and Liao et al. [22] (Scheme 1 and Scheme 2).



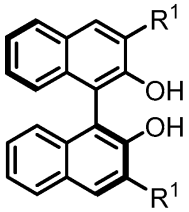
(*R*)-**1**

III, VII, IX, X



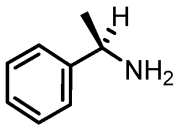
3a

I,II, IV, V, VI,VI



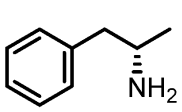
(*S*)-**1**

I



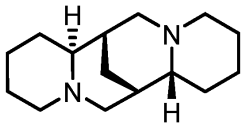
S-phenylethylamine, 1 Eq.
Cu(II)$(NO_3)_2.3H_2O$, 1 Eq.,
N_2 atm.,MeOH,20ºC
(*S*)-**1**
63% yield
3% ee
Wynberg *et al.*, 1978

II



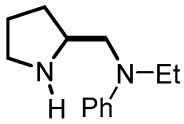
S-amphetamine, 8 eq.
Cu(II)$(NO_3)_2.3H_2O$, 2 eq.
MeOH, 20ºC, N_2 atm.,
MeOH, 20ºC
(*S*)-**1**
98% yield
96% ee
Brussee *et al.*, 1985

III



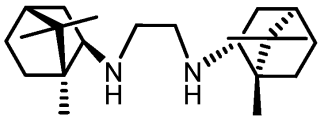
(-)sparteine 10 mol%
CuCl_2 10%mol,
1.1 eq. AgCl,MeOH, 20ºC, 72h
(*R*)-**1**
70% yield
3% ee
Kdovskf, *et al.*, 1991

IV

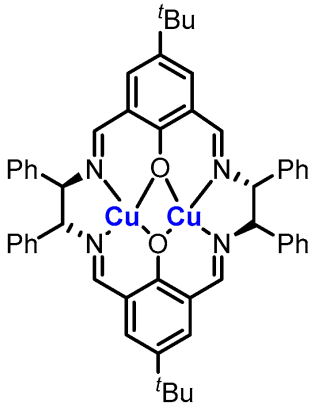


Cat. 10 mol%,
CuCl 10 mol%
O_2, DCM, reflux, 24 h
(*S*)-**1**
69 – 89% yields
17 – 78% ee
Hashimoto *et al.*, 1999

V



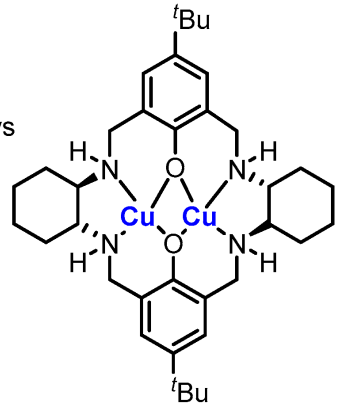
1*R*,2*R*,4*R*
Cat 10 mol %
Cu$(OTf)_2.C_6H_6$, 10 mol%
40ºC, 5-48 h, DCM/MeCN
(*S*)-1
48 – 90% yields
9 – 65% ee
Pilati *et al.*, 2003



Cat. A
(*S*)-**1**
75 – 80% yield
69 – 84% ee

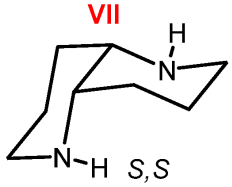
VI

Cat. A or B, 10 mol%
O_2, CCl_4, 0ºC, 3-7 days
Martell *et al.*, 2003



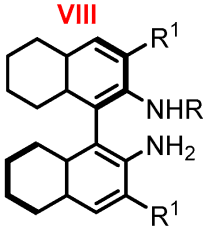
Cat. B
(*S*)-**1**
85 – 92% yield
84 – 87% ee

VII



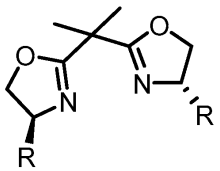
Cat. 10 mol%
CuCl/**Cu**I, 10 mol %,
40ºC, ACN, O_2, 24-48h
(*R*)-**1**
77 – 85% yield
38 – 91% ee
Kozlowsk *et al.*, 2003

VIII



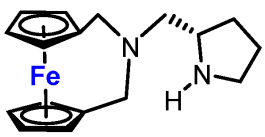
CuCl(9%), O_2, DCM,48h, r.t.
molecular sieves
(*S*)-**1**
72 – 98% yield
4 – 94% ee
Ha *et al.*, 2004

IX



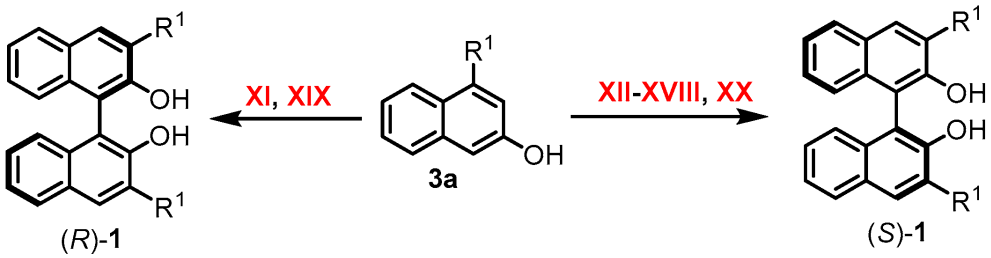
R= Ph, Bn
Cat. 40 mol%,
CuCl
THF, r.t., O_2
(*R*)-**1**
55 – 86% yield
28 – 65% ee
Temma *et al.*, 2005

X

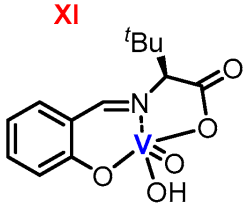


Cat.10 mol%
CuCl 10 mol %, 15ºC, ACN,O_2
(*R*)-**1**
19 – 79% yield
53 – 92% ee
Wu *et al.*, 2014

Scheme 1. Chiral Cu-amine catalytic systems employed in the construction of BINOL derivatives with axial chirality [1][2][3][4][5][6][7][8][9].

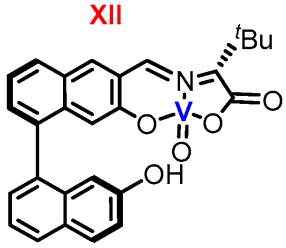


XI



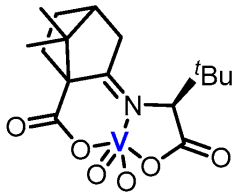
Cat. 10 mol%
CCl_4, O_2 , r.t., 3 -1 2days
(*R*)-**1**
75 – 100% yields
68% ee
Chen *et al.*, 2001

XII



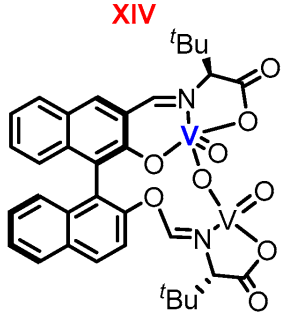
Cat. 5 mol%
O_2, $CHCl_3$, 8 - 36 h
(*S*)-**1**
74 – 96 % yields
18 – 72% ee
Chu *et al.*, 2001

XIII



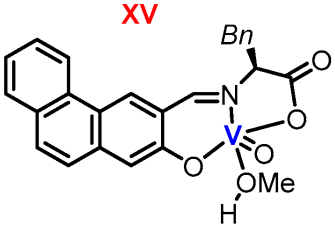
Cat. 3 mol%, O_2, 40 - 45 ºC,
CCl_4, 7 - 10 days
(*S*)-**1**
61 – 99% yields
42 – 87% ee
Chen *et al.*, 2002

XIV



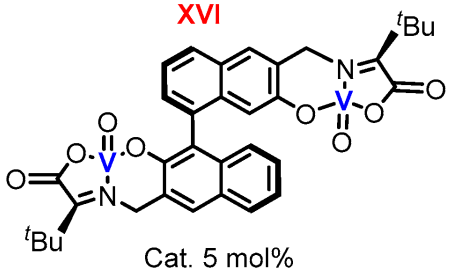
Cat.10mol%, O_2, CCl_4, 5 - 8 days
(*S*)-**1**
88 – 95% yields
83 – 98% ee
Luo *et.al.*, 2002

XV



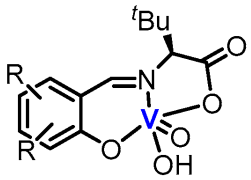
Cat. 10 mol%
TfOH, 10% mol
DCM, O_2, 6 - 24 h
(*S*)-**1**
Up 100%
68 – 87% ee
Uang *et al.*, 2001

XVI



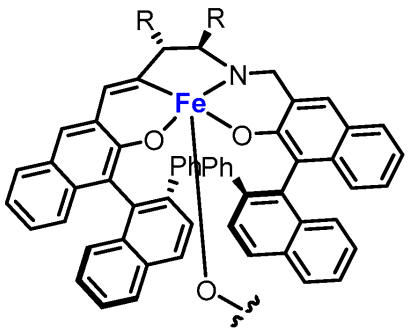
Cat. 5 mol%
DCM, air, 1 - 3 days
(*S*)-**1**
35 – 99% yields
45 – 93% ee
Sasai *et al.*, 2004

XVII



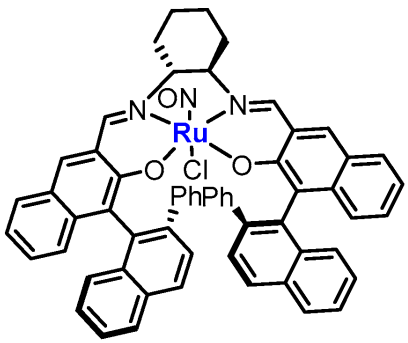
Cat. 10 mol%
CCl_4, O_2, 0ºC, 5 - 8 days
(*S*)-**1**
20 – 93 yields
6 – 83% ee
Gong *et al.*, 2007

XVIII



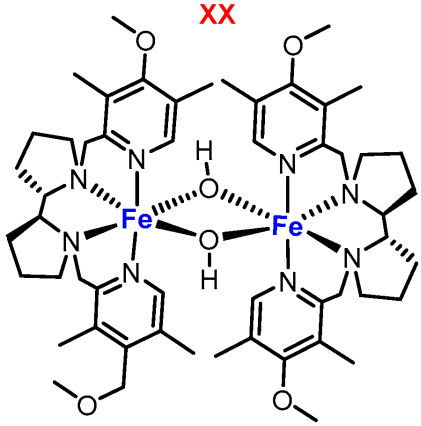
Cat.
A) R = -$(CH_2)_4$-/B) Ph
4 mol%
toluene, air,60 ºC,
(*R*)-**1**

XIX



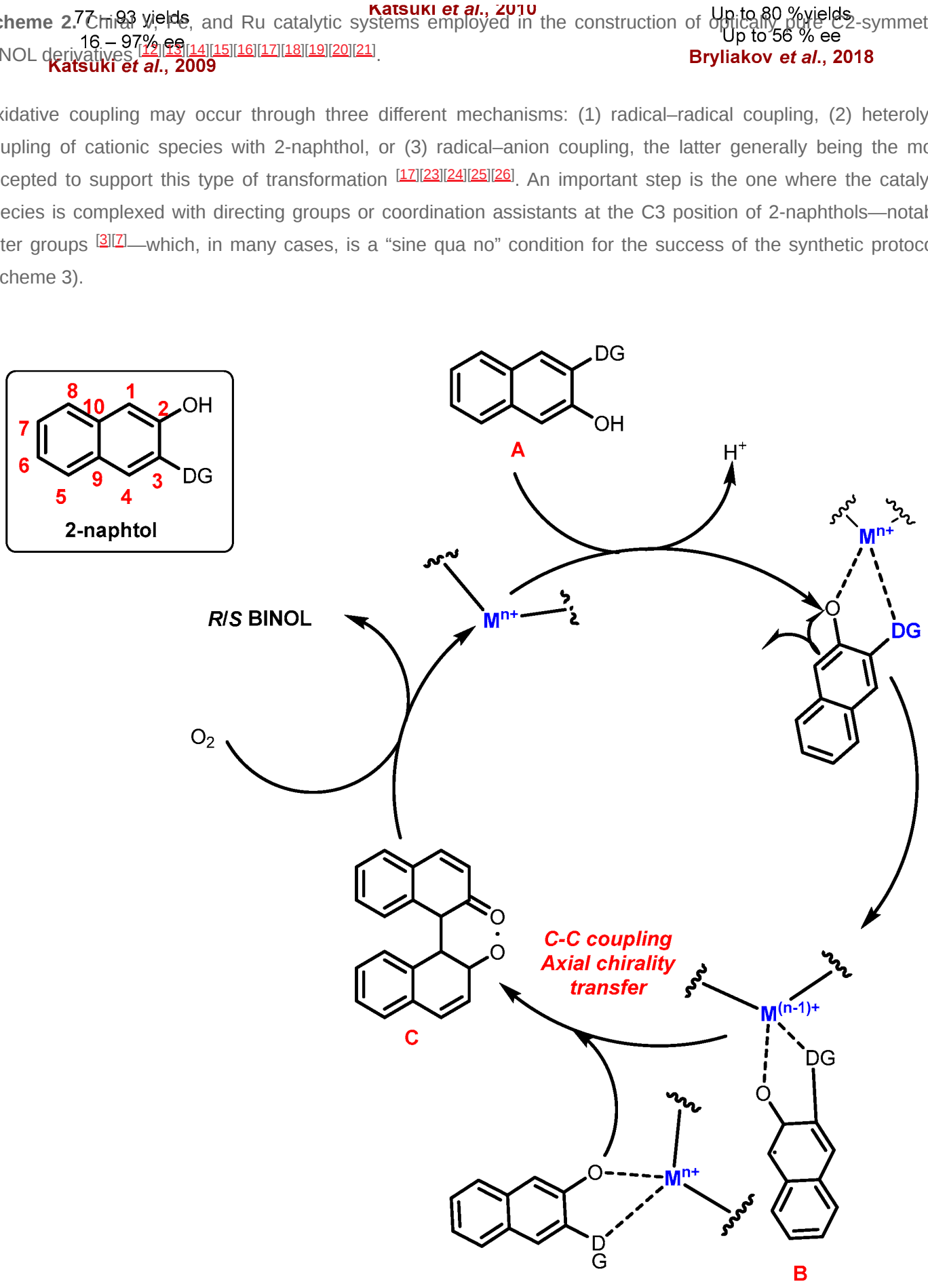
Cat. 2-5 mol% mol%
toluene, air,hv, 25 ºC, 5 - 8 days
(*S*)-**1**
72 – 93 yields
33 – 69% ee
Katsuki *et al.*, 2010

XX



Cat.1 mol%
Chlorobenzene, air,50 ºC, 24 h
(*S,R*)-**1**

Scheme 2. Chiral V, Fe, and Ru catalytic systems employed in the construction of optically pure C2-symmetric BINOL derivatives [12][13][14][15][16][17][18][19][20][21].

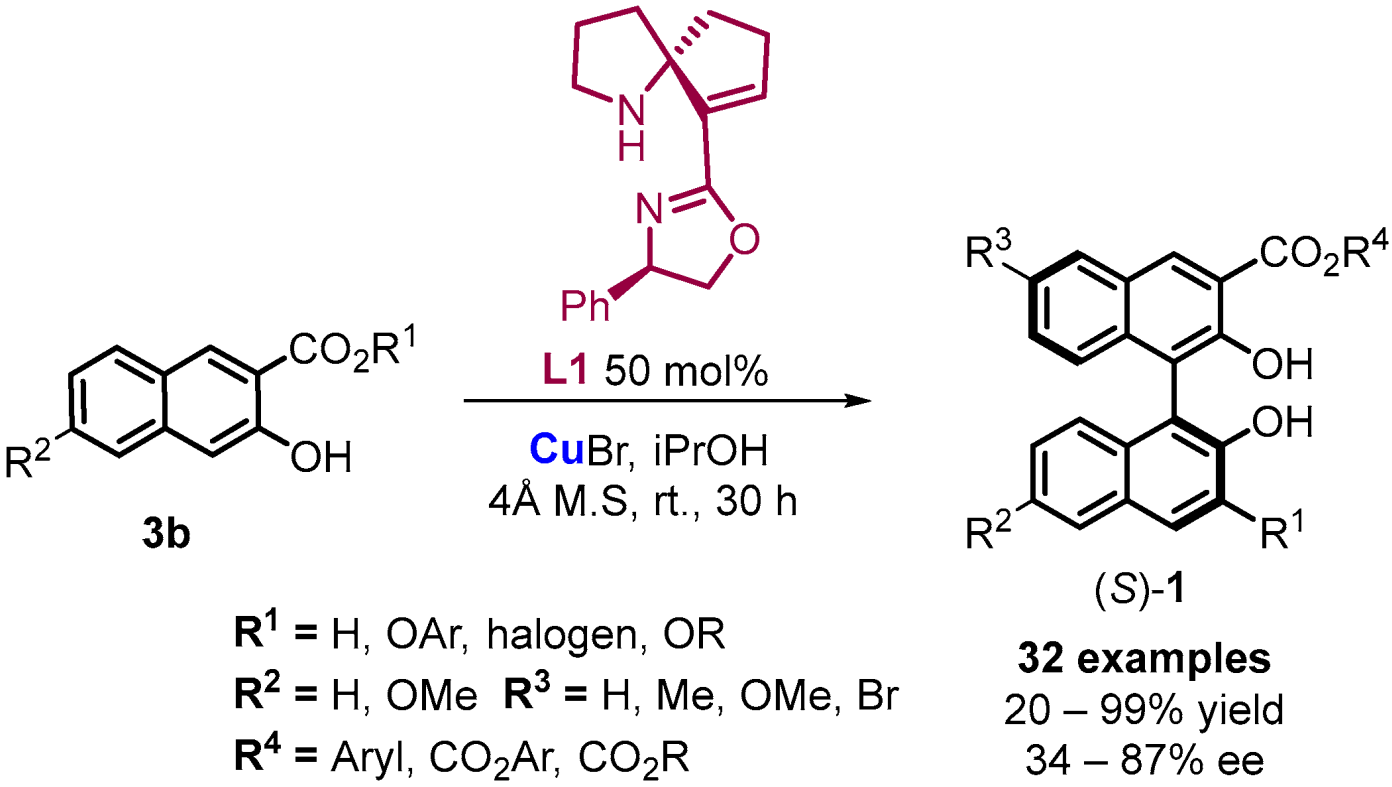


Oxidative coupling may occur through three different mechanisms: (1) radical–radical coupling, (2) heterolytic coupling of cationic species with 2-naphthol, or (3) radical–anion coupling, the latter generally being the most accepted to support this type of transformation [17][23][24][25][26]. An important step is the one where the catalytic species is complexed with directing groups or coordination assistants at the C3 position of 2-naphthols—notably ester groups [3][7]—which, in many cases, is a “sine qua no” condition for the success of the synthetic protocols (Scheme 3).

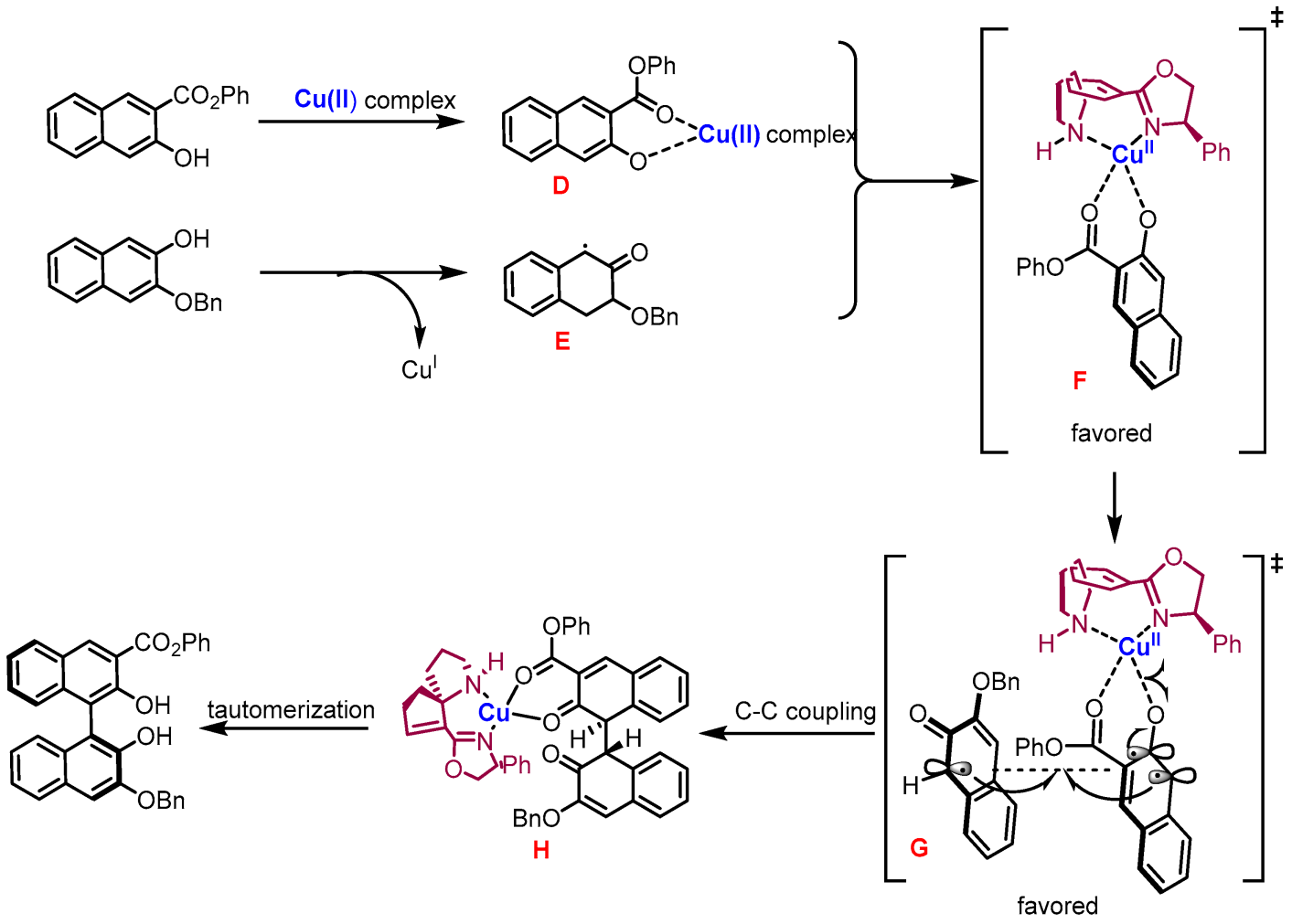
Scheme 3. General aspects of the mechanism for aerobic radical–anion coupling of 2-napthols in the presence of metallic chiral complex catalysts (M^{n+}). DG: coordination aids. Adapted from Brunel et al [19].

With due recognition of the particularities of each case, the radial–anion mechanism [17][23][24][25][26][27] (Scheme 3) usually proceeds via generation of the radical species **B** resulting from an oxidation of 2-naphthol **A** by a metal catalyst (M^{n+}). **B** is then added to another neutral 2-naphthol molecule to form a new C-C bond and generate the **C**-radical, which is further oxidized by O_2 to restore aromaticity.

The most recent methods for obtaining enantiomerically pure BINOLs are still based on the catalytic dyad metal–chiral ligands. In this sense, Chen and colleagues [28] (Scheme 4) have developed a new chiral 1,5-*N*,*N*-bidentate ligand based on a spirocyclic skeleton of pyrrolidine oxazoline and CuBr to couple 2-naphtols **3b**. The efficient catalytic species formed in situ allows for (*S*)-BINOL derivatives (**1**) with high enantioselectivity (up to 99% ee) and good yields (up to 87%) to be obtained. Based on experimental results and the literature, the authors proposed that this coupling proceeds via radical–anion coupling, where the complex generated in situ coordinates to form species **D** in the presence of air, which couples with radical **E** (generated through an electron transfer from the outer sphere with another Cu(II) complex) to form the intermediate **F** (Scheme 5). The coupling product is obtained after tautomerization of **H**. The authors found experimental evidence that, during the coupling process, the attack from **E** to **F** by the *Si* face was favored, probably because of the greater steric impediment to attack by the *Re* face.

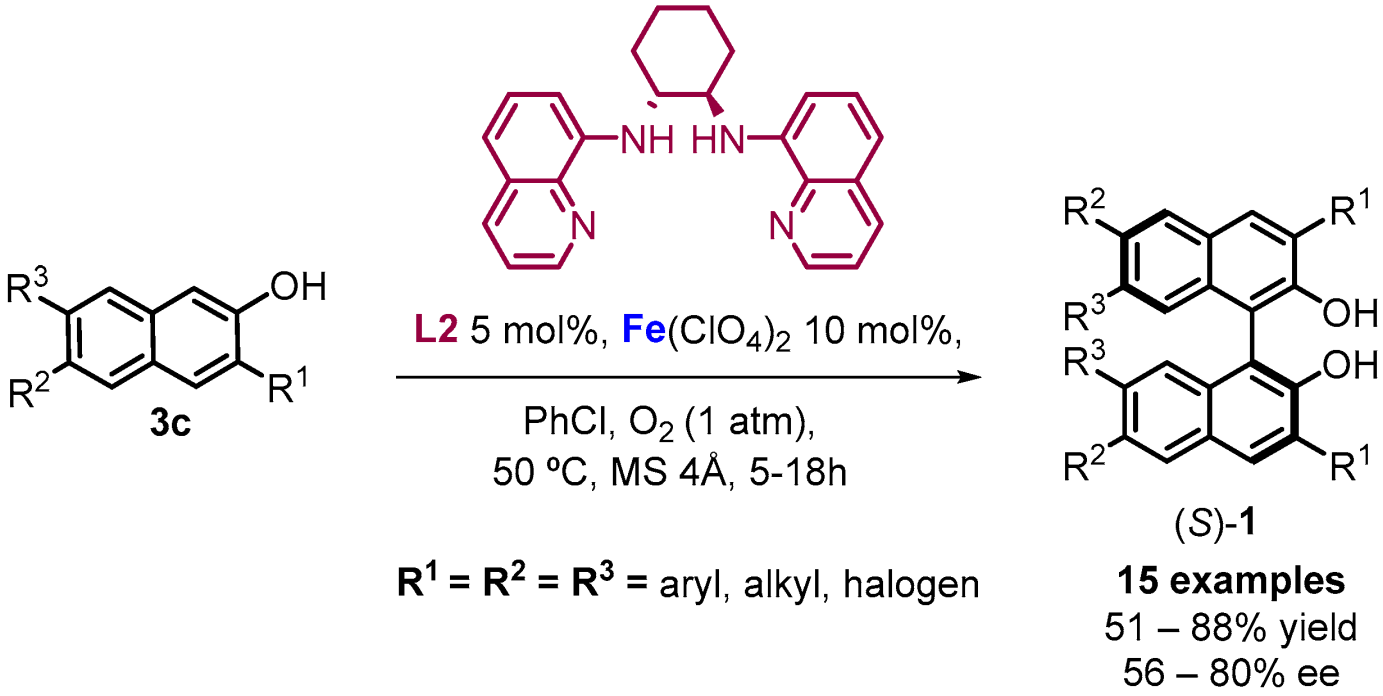


Scheme 4. Recent synthetic protocols for the construction of axially chiral BINOL derivatives.



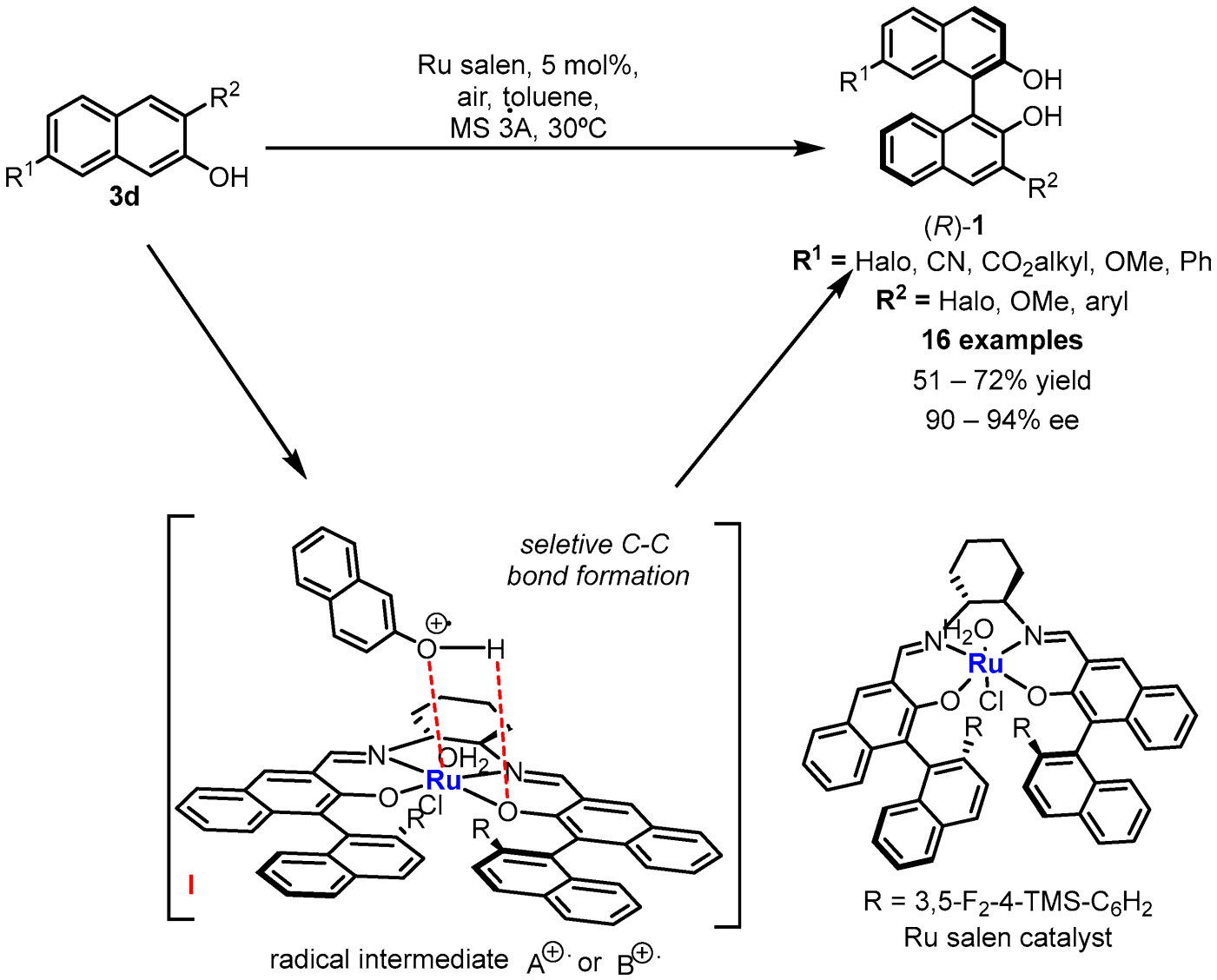
Scheme 5. Mechanistic proposal for the enantioselective aerobic coupling of 2-naphthols based on a ligand catalytic system containing a spirocyclic skeleton of pyrrolidine oxazoline/CuBr.

Che and co-workers introduced a chiral aminopyridine-like ligand—bisquinolyldiamine [(1*R*,2*R*)-N^1,N^2-di(quinolin-8-yl)cyclohexane-1,2-diamine (BQCN)]— and applied it to the iron-catalyzed asymmetric *cis*-dihydroxylation of alkenes [29]. Inspired by this work, Liu's group [30] (Scheme 6) established a methodology for the asymmetric oxidative homo-coupling of 2-naphthols (**3c**), leading to the synthesis of (*S*)-BINOL derivatives (**1**) mediated by a Fe complex and generated in situ from $Fe(ClO_4)_2$ and the BQCN ligand. Excellent yields (up to 99%) and enantiomeric excesses (up to 81%) have been reported.



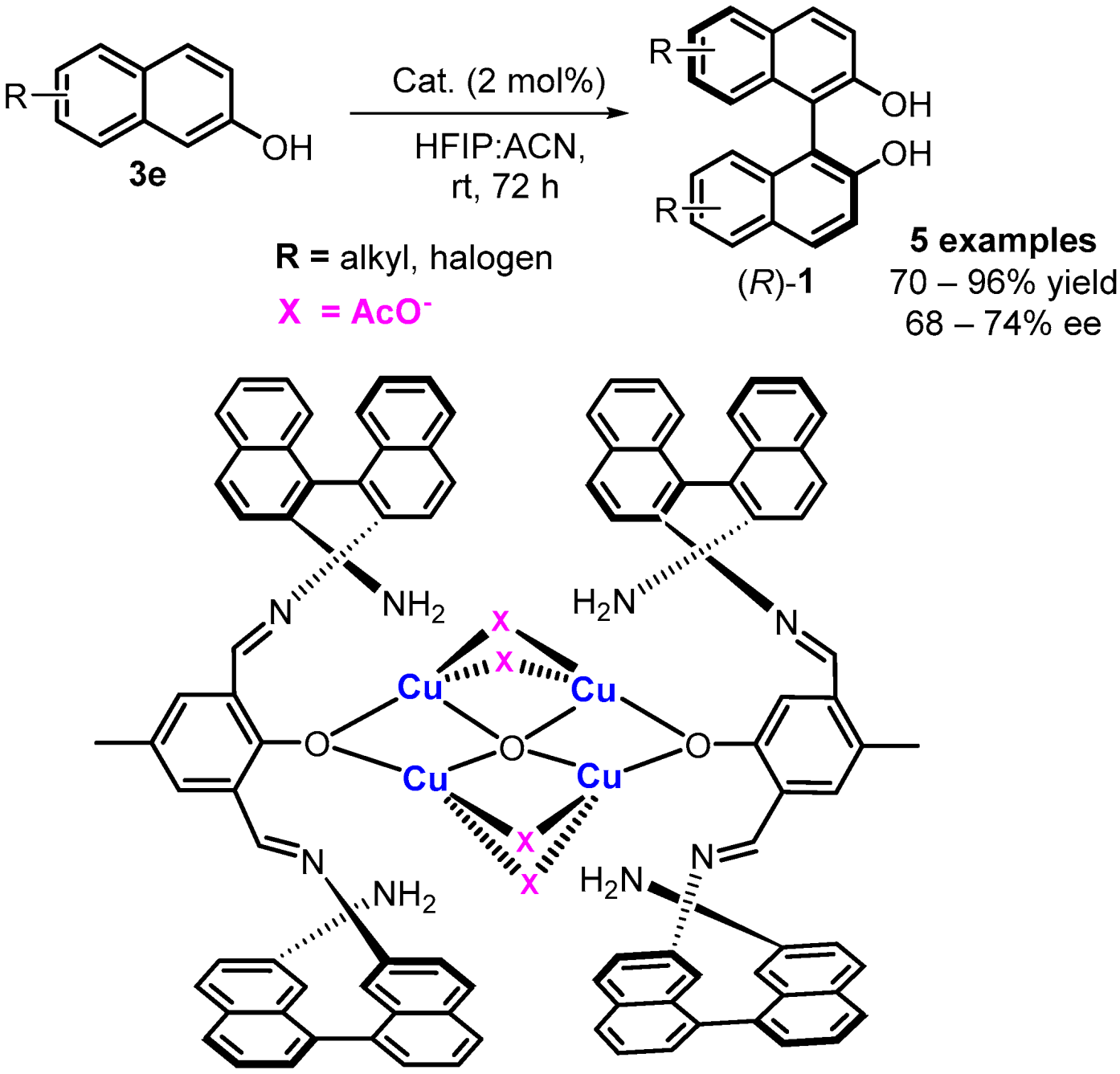
Scheme 6. Enantioselective coupling between 2-naphthols (**3c**) mediated by an iron/bisquinolyldiamine ligand complex.

From the same perspective, Uchida's group [31] (Scheme 7) developed remarkable enantioselective aerobic coupling between 2-naphthols **3d** in the presence of the (aqua)ruthenium complex (salen). The protocol provided (*R*)-BINOLs (**1**) with yields between 55 and 85% and enantiomeric excesses up to 94%. Through mechanistic studies, these researchers concluded that, in this case, cross-coupling selectivity is dominated by steric rather than electronic effects, which can be controlled by chemoselective oxidation via single electron transfer (SET) and oxidative carbon–carbon bond formation, a process for which ruthenium(salen) catalyst proved to be suitable [24]. Therefore, the authors have proposed that this transformation proceeds via oxidation of one of the coupling partners to the electrophilic intermediate radical **I**, which is converted to the desired BINOL after chemoselective coupling [24].



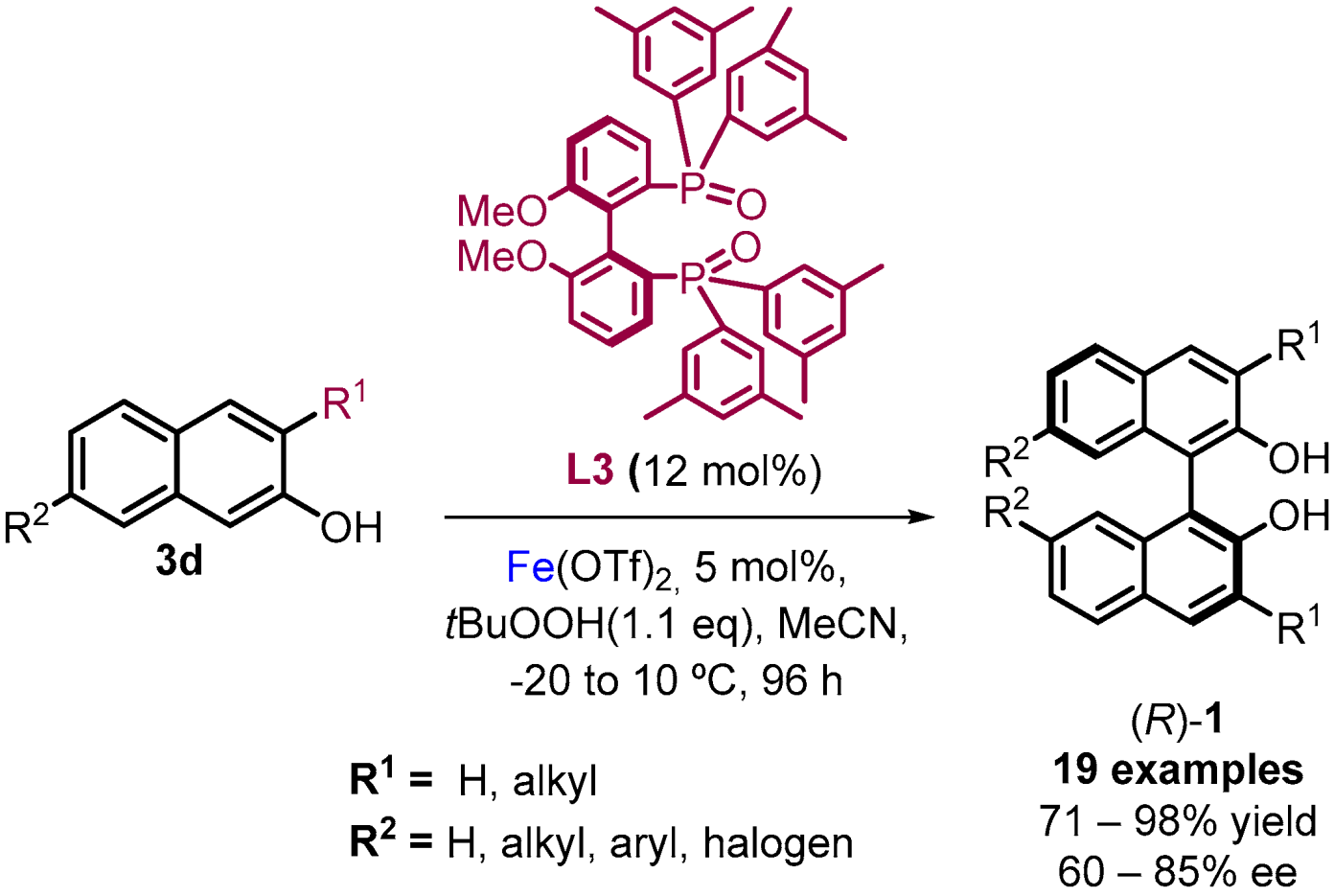
Scheme 7. (Aqua)ruthenium (salen) catalyzed enantioselective aerobic coupling between 2-naphthols for access to *C*1-symmetric BINOL derivatives.

Recently, Subramanian et al. [32] (Scheme 8) developed a Cu(II)-2+4-μ4-oxo tetranuclear open frame macrocyclic/BINAN complex and employed it in the asymmetric oxidative coupling of 2-naphthols **3e**, obtaining (*R*)-BINOL derivatives (**1**) with good to excellent yields (70–96%) and enantiomeric excesses between 68 and 74%.



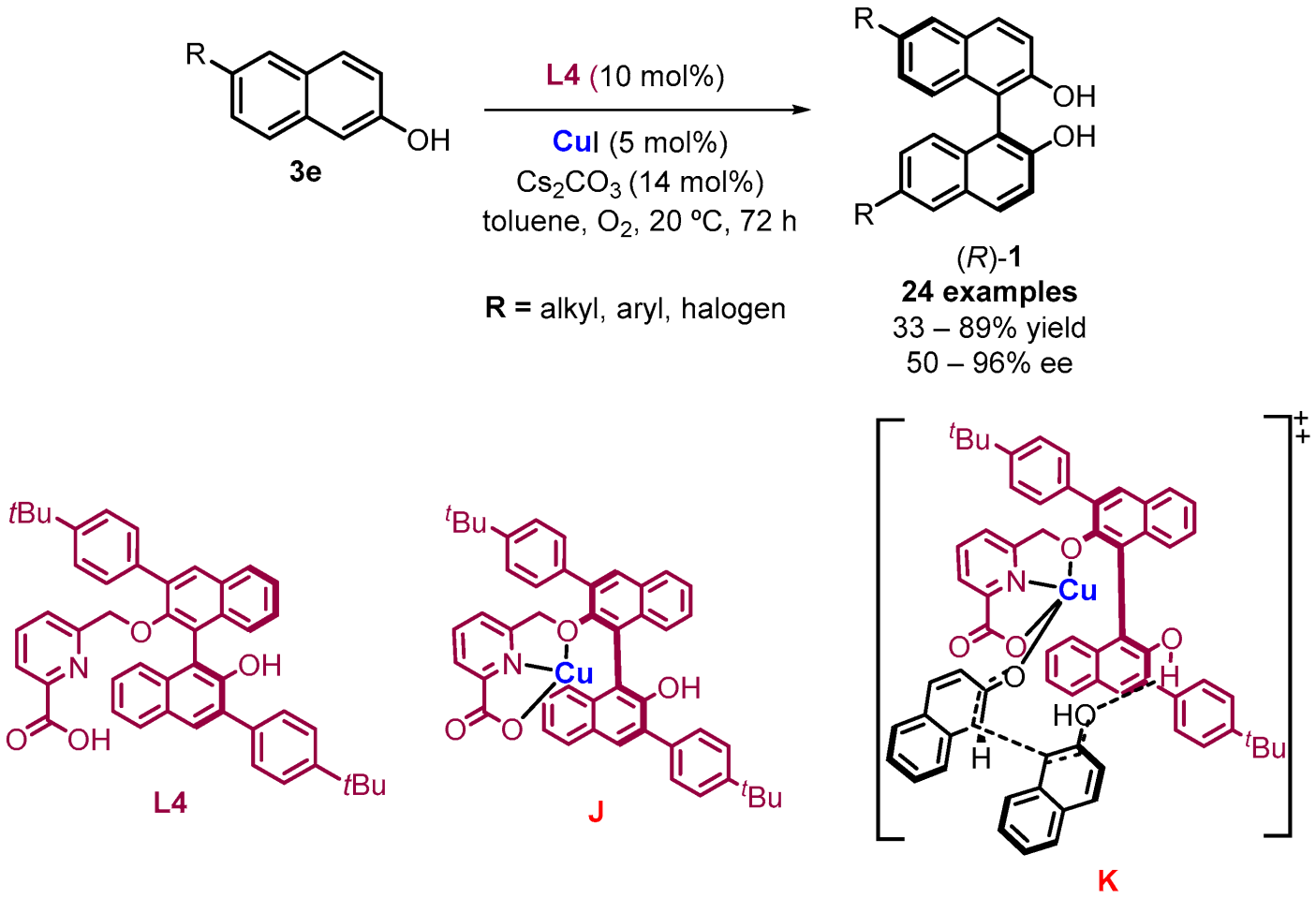
Scheme 8. Asymmetric oxidative coupling of 2-naphthols mediated by a macrocyclic Cu(II) complex.

Ishihara and co-workers [33] (Scheme 9) developed a method for enantioselective oxidative coupling of 2-naphthol derivatives **3d** in the presence of a chiral Fe(II)-diphosphine oxide complex. The products of interest were obtained with yields up to 98 % and enantiomeric excesses between 60 and 85%.



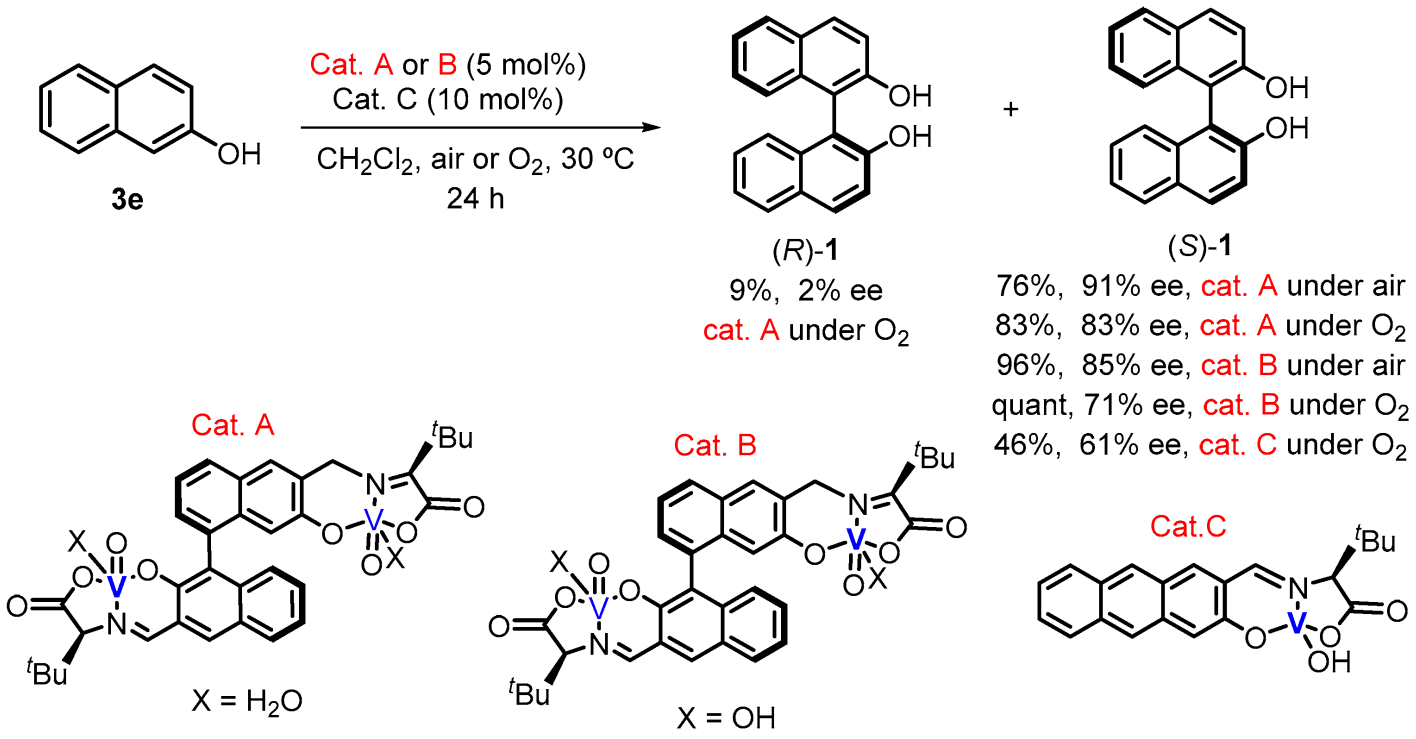
Scheme 9. Chiral diphosphine oxide-iron(II) complex catalyzed enantioselective aerobic coupling between 2-naphthols to access *C*1-symmetric BINOL derivatives.

A copper catalyst prepared in situ from a ligand synthesized by the fusion of chelating picolinic acid/substituted BINOLs and CuI was employed in the asymmetric oxidative coupling of 2-naphthols (**3e**). In this work, published by Zhang et al. [34] (Scheme 10), 6,6'-disubstituted (*R*)-BINOLs (**1**) were obtained with yields of up to 89% and excellent enantioselectivities (up to 96% ee). The reaction was accompanied by Mass Spectroscopy, and identification of a peak corresponding to the complex **J** allowed the authors to propose a mechanism pathway through the transition state **K**.



Scheme 10. Copper-catalyzed asymmetric oxidative coupling of 2-naphthols for the synthesis of 6,6′- disubstituted BINOLs.

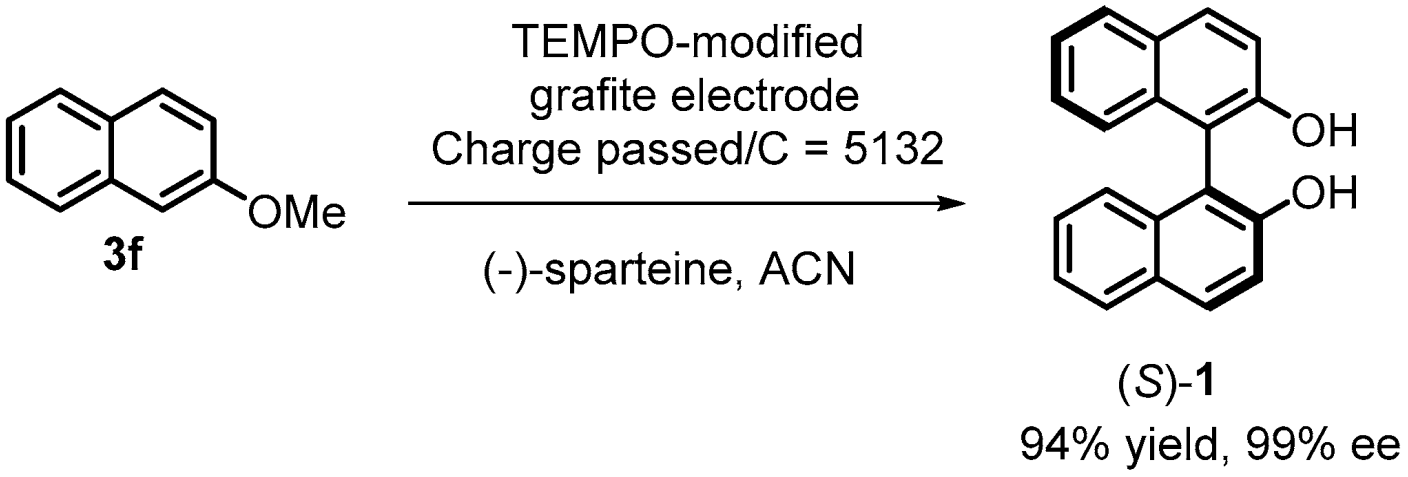
Continuing work involving multifunctional chiral catalysis via double activation, Takizawa's group [35] developed complexes **A**-**C** (Scheme 11)—from $VOSO_4$ and Schiff base ligands generated via condensation of (*S*)-tert-leucine and 3,3 '-formyl-(R)-BINOL—which have been successfully applied in the synthesis of (*R*)- and (*S*)-BINOL (**1**) with yields between 46 and 76%, in addition to enantiomeric excesses of up to 91%.



Scheme 11. Atroposelective synthesis of (*R*)- and (*S*)-BINOLs (**1**) via mono- and binuclear vanadium catalysts.

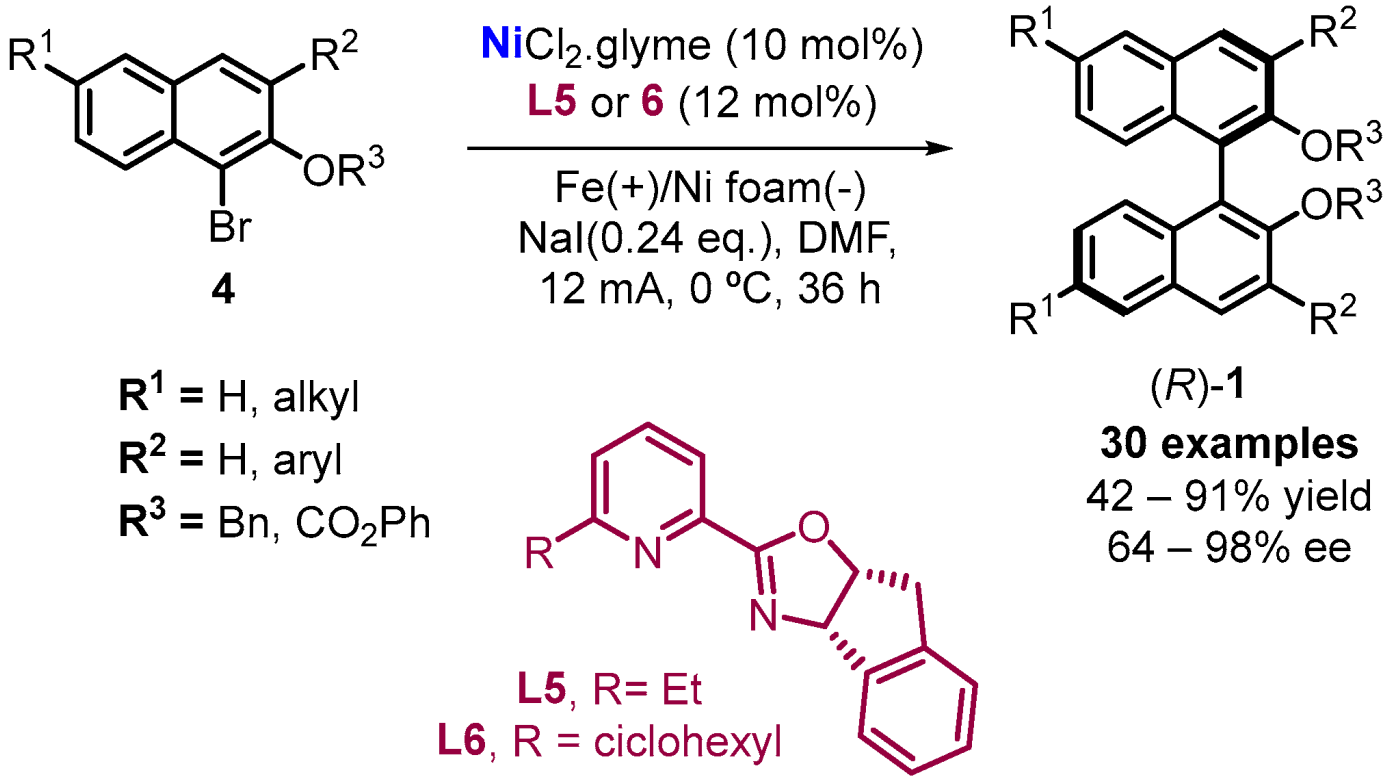
2. Electrochemical Synthesis

Despite the inherent advantages of electrochemical synthesis, notably in terms of sustainability [36], few examples of enantioselective coupling for the construction of chiral BINOLs have been reported so far. In 1994, which appears to be the first record of this type of synthesis, Bobbitt et al. [37] (Scheme 12) established a method for enantioselective coupling of 2-naphthols (**3f**) on a TEMPO-modified graphite electrode in the presence of (-)-sparteine in acetonitrile to afford (*S*)-BINOL (**1**) with excellent yields and enantiomeric excesses.



Scheme 12. Electrochemical synthesis of (*S*)-BINOL (**1**) using a TEMPO-modified graphite electrode.

Recently, Mei's group [36] (Scheme 13) demonstrated the first example of a Ni-catalyzed enantioselective electrochemical reductive coupling of 2-naphtols (**4**) in an undivided cell for the construction of axially chiral BINOL derivatives (**1**) with good yields (up 91%) and enantiomeric excess of up to 98%.



Scheme 13. Enantioselective Ni-promoted electrochemical synthesis of (*R*)-BINOL derivatives (**1**).

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