ELSEVIER

PROGRESS IN HETEROCYCLIC CHEMISTRY

VOLUME 29

EDITORS
Gordon W. Gribble and
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CHAPTER 6.4

Six-Membered Ring Systems: With O and/or S Atoms

Clementina M.M. Santos*, Artur M.S. Silva§,1
*Polytechnic Institute of Bragança, Bragança, Portugal; §University of Aveiro, Aveiro, Portugal
[1]Corresponding author: E-mail: artur.silva@ua.pt

6.4.1 INTRODUCTION

In 2016, the large variety of publications focused on the synthesis of *O*- and *S*-6-membered ring systems highlights the importance and popularity of these heterocyclic compounds.

Reviews on natural products covered the origins and medicinal use of avermectins (16AGE10190), discovery, isolation, characterization and application of artemisinin (16AGE10210), biosynthesis of fungal meroterpenoids (16NPR26), isolation, biological properties, and synthesis of dimeric pyranonaphthoquinones (16EJO5778), and peroxide and peroxide-derived polyketide metabolites from marine sponges (16NPR861). Late-stage convergent annulation reactions (16T3345) and rhodium-catalyzed annulation reactions of arenes with alkynes via weak chelation-assisted C—H activation (16CC2872) for the synthesis of several natural six-membered oxacycles have also been reviewed.

New approaches for the synthesis of naturally occurring bioactive pyran derivatives have appeared. Different routes have been established for the total synthesis of tetrahydropyran-containing natural compounds brevisamide (16EJO2300, 16JOC3799), (±)-centrolobine (16SL2221), (−)-clavosolide A (16AGE2498), (+)-decarestrictine L (16TL4368), decytospolide A, B, and their C-3 epimers (16S765), goniodomin A (16JOC2213), herboxidiene (16OBC6212), irciniastatins (16JOC1930), (−)-lasonolide A (16JA11690), (−)-luminacin D (16JOC3818), (−)-mandelalide A (16JA770, 16JA3675), isomandelalide A (16JA770), thailanstatin A (16JA7532); of the 2*H*-pyran-2-ones sibirinone, (*E*)-6-(pent-1-en-1-yl)-2*H*-pyran-2-one and (*E*)-6-(hept-1-en-1-yl)-2*H*-pyran-2-one (16JOC10357); of the 5,6-dihydropyran-2-one derivatives cryptomoscatone F1 (16S1561), cryptorigidifoliol B (16TL2100), cryptorigidifoliol E (16S4213), 8-methoxygoniodiol (16S4300),

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pectinolides A, C, and H (16HCA247); and of the tetrahydropyran-2-one derivatives (−)-(*S*)-goniothalamin and (−)-leiocarpin (16SC187), helicascolides A, B, and C (16AGE5765), isodaphlongamine H (16AGE2577), and (−)-malyngolide and its C-5 epimer (16HCA267).

The total synthesis of natural carbazole[2,1-*b*]-2*H*-pyrans koenidine and mahanimbine (16JA13870), koenine, koenimbine, koenigine, and koenigicin (16S150); of chroman-type bryostatins (16JA13415), 3,14-didehydroxyisogarcinol and garcimultiflorone A stereoisomers (16JA14789), paeoveitol (16OL3698), and xiamenmycin A (16OBC1805); of polycyclic bischroman-type kuwanon X and Y and kuwanol A (16OL360); of biscoumarins valoneic and woodfordinic acids (16SL859); of isocoumarin legioliulin (16TL3942); of 3,4-dihydroisocoumarin isocladosporin and 3-*epi*-isocladosporin (16TL53); of flavone derivatives such as artocapin (16H(93)310), 2′,3,5′,6,7,8-hexamethoxyflavone (16SL1725), and houttuynoid B (16CEJ2935); of cytotoxic hexahydro-1*H*-xanthene (+)-psiguadial B (16JA9803); of pyrano[2,3-*b*]chroman-type teadenols A and B (16OBC10783), mirabiquinone A, and its methyl derivatives (16S761); of xanthene-type compound (−)-siccanin (16SL96); of three *C*-glycosyl xanthones mangiferin, homomangiferin, and neomangiferin (16OBC8821) and the prenylated xanthone elliptoxanthone A (16SL2229); of cytotoxic polycyclic tetrahydroxanthone kibdelone C and its simplified derivatives (16JA10561); and of HIV-1 integrase inhibitor containing a 1,3-dioxine moiety integrastatin B (16OL1458) have also been surveyed.

Developments on the stereoselective synthesis of tetrahydropyran-containing C1–C13 subunit of amphidinolide N (16TL3532), C1–C16 subunit of bryostatins (16OBC9650), C1–C12 subunit of laulimalide (16AGE13210), C1–C10 subunit of madeirolide A (16OL2154), C1–C13 subunit of mandelalide A (16S917), C14–C29 subunit of mirabalin (16OL4534); of 3,6-dihydro-2*H*-pyran-containing C7–C18 subunit of scytophycin C (16TL446), C1–C15 subunit of natural antibiotic sorangicin A (16JOC10698); and of tetrahydropyran-2-one-containing C16–C28 subunit of hemicalide (16CC4632) and a key fragment of elaiophylin (16HCA506) have been achieved.

An overview on enantioselective organocatalyzed transformations of β-ketoesters (16CR9375) and enantioselective alcohol C—H functionalization (16JA5467) for the synthesis of various *O*-6-membered heterocycles were disclosed. A specific review on microwave-assisted synthesis of xanthene-type compounds in ionic liquids (16JHC1697) has also appeared.

Discussions on specific reactions include metal-catalyzed intramolecular cyclization of acyclic precursors to give 3,4- and 3,6-dihydro-2*H*-pyrans (16S3470), transition-metal-catalyzed asymmetric aryl C—O coupling reactions of aryl halides with nucleophiles for the synthesis of chromans and 1,4-dioxanes (16SL664), transition-metal-catalyzed synthesis of coumarins (16S2303), radical C—H functionalization in the synthesis of coumarins and xanthones (16CC2220), ring-closing alkene metathesis in the synthesis of natural pyrans (16OBC5875), intramolecular cyclization of aryltriazenes for the synthesis of coumarins and 2-hydroxy-4*H*-chroman-4-ones (16SL1318), and inverse electron demand hetero-Diels–Alder (IED-hDA) reaction of *o*-thioquinones with bis-dienophiles to afford helical-shaped bis-1,4-benzoxathiines (16EJO5386).

Certain reaction types have merited special attention. Ring-closing metathesis was applied to the synthesis of 3,6-dihydro-2*H*-pyrans starting from diene and enyne skeletons using Grubbs catalysts (16TA954, 16EJO3900) and also to construct the 5,6-dihydropyran-2-one ring in many natural compounds including cryptocaryol (16AGE5049), cryptomoscatone D1 and (5*R*,7*S*)-kurzilactone (16TL1087), the C1–C27 subunit of hemicalide (16JOC11275), 12 withanolide analogs (16S48), and in the synthesis of eight pure stereoisomers of pironetin–dumetorine hybrids, a new scaffold for tubulin binders (16EJO2029). Prins cyclization was used to build the tetrahydropyran core of four diarylheptanoids from *Dioscorea villosa* (16TL3505), while oxa-Michael cyclization reactions were used to prepare the tetrahydropyran rings of (−)-gilbertine (16JOC4566), neopeltolide (16JOC415), rhopaloic acid A (16AGE3455), and fragments of various polyketides (16AGE6280).

The importance of specific reagents, namely, quinones in catalytic asymmetric reactions (16OBC6913), *o*-quinone methides as electrophiles in metal-free catalysis (16JOC10145), Morita–Baylis–Hillman (MBH) and Rauhut–Currier adducts of nitroalkenes (16SL2425) to prepare pyran-, chromene-, and xanthene-type compounds; and of coumarin as a building block in the synthesis of pentacyclic coumarin–chromene-containing compounds (16SC569); 3-methyl-2-vinylindoles as dienophiles in asymmetric IED-hDA reaction with *o*-hydroxybenzyl alcohols catalyzed by a chiral phosphoric acid to give 2-(indol-2-yl)chromans (16SL2515); ytterbium triflate as catalyst in the synthesis of several *O*- and *S*-6-membered heterocycles (16S4305); silica-supported polyphosphoric acid (16SC197); and MCM-41-anchored sulfonic acid (16SC1713) as a heterogeneous catalyst in the synthesis of thiochroman, chromene, and xanthene-type compounds have been disclosed.

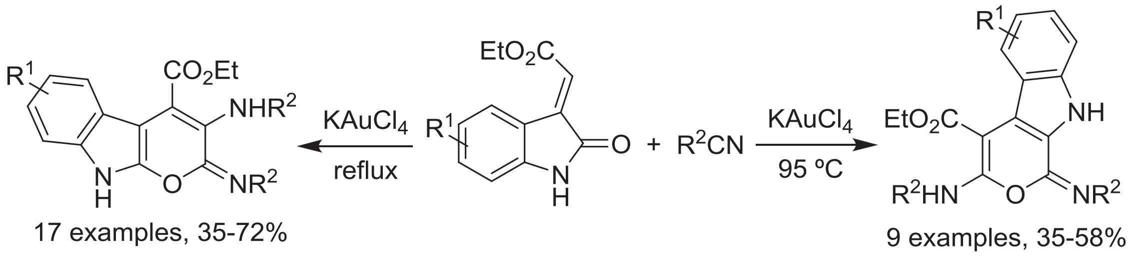
Herein, we will provide a personal overview of the most studied transformations on *O*- and *S*-6-membered heterocycles that were published in 2016.

6.4.2 HETEROCYCLES CONTAINING ONE OXYGEN ATOM

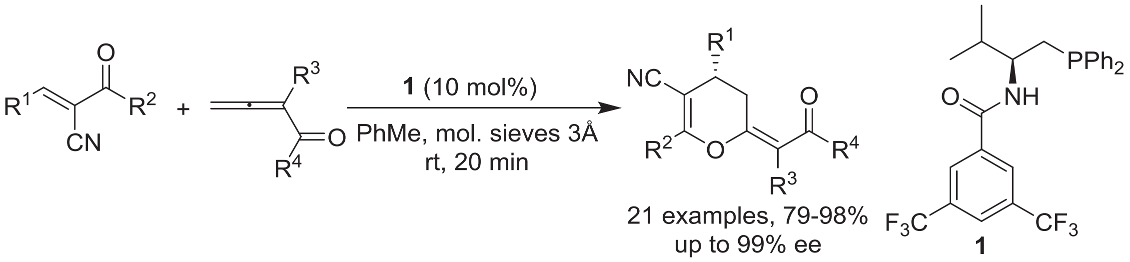
6.4.2.1 Pyrans

3-Propargyloxyfuran-2-ones are converted into furan-2-one-fused [3,4-*b*]-2*H*-pyrans via gold(I)-catalyzed Claisen rearrangement/6-*endo*-*trig*-cyclization reactions (16OL680). The same catalyst was used in the reaction of 3-propargyloxyfuran-2-one-type compounds with several nucleophiles to afford various products, according to the substitution pattern of the substrate. Furyl and thienyl substituents provided dihydrofuran-2-one-fused 5,6-dihydro-2*H*-pyrans, while aryl substituents led to furan-2-one-fused dihydrofurans (16CC6233). The reaction of isocyanides with oxindolydeneacetates promoted by $KAuCl_4$ is temperature controlled: in refluxing toluene, double isocyanide insertion occurs to give indolo[2,3-*b*]-2-imino-2*H*-pyrans; at 95°C, also in toluene, double insertion with oxygen migration takes place to furnish indolo[2,3-*c*]-2-imino-2*H*-pyrans (Scheme 1) (16OL840). A series of quinolino[3,2-*c*]-2*H*-pyrans arises from $FeCl_3 \cdot 6H_2O$-mediated 6-*endo*-*dig*-cyclization of 2-(arylethynyl)quinoline-3-methanols (16TL615) and from the reaction of 2-alkynylquinoline-3-carbaldehydes with various nucleophiles in the presence of AgOTf, via hydroarylation and regioselective 6-*endo*-*dig*-cyclization reactions (16S2178). Examples of quinolin-2-one-fused [4,3-*b*]-3-iodo-2*H*-pyrans result from electrophilic cyclization of 4-propargyloxyquinolin-2-ones with iodine using nitromethane as solvent (16T4288).

$FeCl_3$-mediated intramolecular *O*-alkylation of 2-(3-methylbut-2-en-1-yl)-1,3-dicarbonyl compounds in 1,2-dichloroethane at room temperature affords 2,2-dimethyl-5,6-disubstituted 3,4-dihydro-2*H*-pyrans (16OL1310). Several



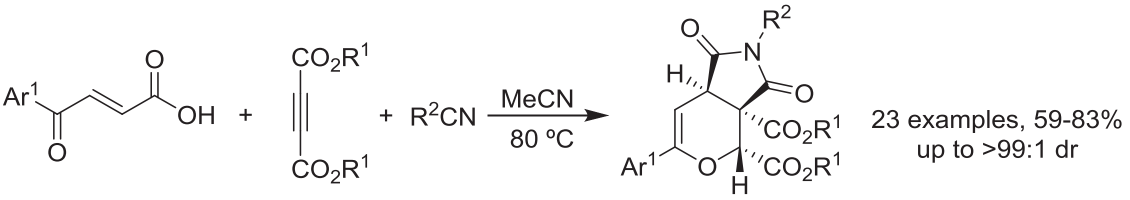
Scheme 1



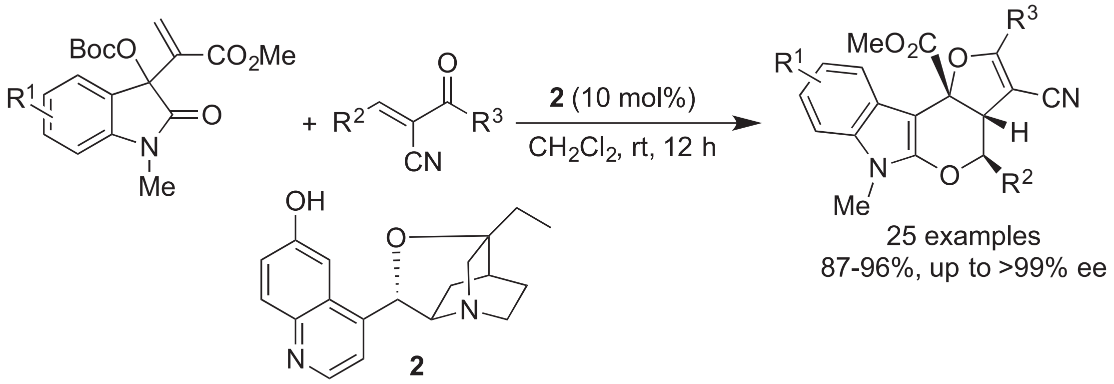
Scheme 2

examples of 6-aryl-2,2-dimethyl-5-sulfonyl-3,4-dihydro-2*H*-pyrans were synthesized through $Fe(OTf)_3$-promoted one-pot [3 + 3]-cycloaddition of 1-aryl-2-sulfonylethan-1-ones with prenyl alcohol in nitromethane followed by intramolecular Friedel–Crafts alkylation (16T518). Ring-closing metathesis was used to prepare 3,3-disubstituted 3,4-dihydro-2*H*-pyrans in high yields starting from vinyl pent-4-en-1-yl ethers in benzene at 60–70°C (16SL2611). An enantioselective formal [4 + 2]-cycloaddition reaction of ethyl 2,3-butadienoate with 1,1,1-trifluoro- and 4,4,4-trifluorobut-3-en-2-ones mediated by a quinidine catalyst led to 6- and 4-trifluoromethyl-substituted 3,4-dihydro-2*H*-pyrans, respectively (16EJO3619). Highly substituted 3,4-dihydro-2*H*-pyrans were produced enantioselectively from the reactions of α-cyano-α,β-unsaturated ketones with allenones promoted by an L-valine-derived amide–phosphine catalyst **1** (Scheme 2) (16OL2138) and with allylic ketones promoted by a *Cinchona*-derived primary amine catalyst and a benzoic acid derivative (16OL6480) or from the reaction of penta-1,4-dien-3-ones with allenoates mediated by a chiral amine (16OL3976). One-pot three-component reaction of β-keto perfluoroalkanesulfones with aliphatic/aromatic aldehydes and vinyl ethers carried out in the presence of ammonium acetate delivers 2,4,5,6-tetrasubstituted 3,4-dihydro-2*H*-pyrans (16TL2171).

A dipeptide phosphine-mediated [4 + 2]-annulation reaction of 3-aroylcoumarins with allenones in toluene at room temperature provides coumarino[4,3-*c*]-3,4-dihydro-2*H*-pyrans in high yields and enantioselectivity (16OBC5059). An organocatalytic cascade of α-arylacetylenyl-β-aryl nitroolefins with 4-hydroxycoumarins/4-hydroxy-6-methylpyran-2-one led to coumarin/pyran-2-one-fused 4-aryl-2-methylene-3-nitro-3,4-dihydro-2*H*-pyrans. It involves chiral squaramide-mediated conjugate addition, allene formation, intramolecular oxa-Michael 6-*endo*-*dig*-cyclization, and 1,4-diazabicyclo[2.2.2]octane (DABCO)-mediated olefin isomerization (16OL3098). Good stereoselectivities were obtained in a similar reaction with chiral-free catalysts (16OBC1940). A wide range of pyrazolo [5,4-*b*]-3,4-dihydro-2*H*-pyrans were produced via enantioselective reaction



Scheme 3



Scheme 4

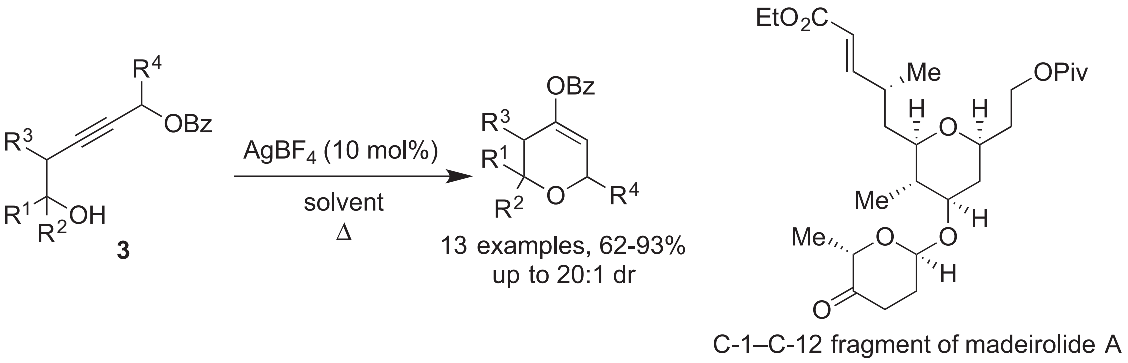
of nitroalkene-derived MBH adducts with pyrazolin-5-ones mediated by chiral tertiary-amine squaramides (16JOC4340, 16T4047). Further examples include the reaction of 4-alkylidenepyrazol-5-ones with α,β-unsaturated aldehydes carried out in toluene at room temperature and using 4-hydroxydinaphthylprolinol as catalyst (16OL556). Highly substituted pyrrolo[3,4-*c*]-3,4-dihydro-2*H*-pyrans were formed through three-component bicyclization reactions of 3-aroylacrylic acids with dialkyl acetylenedicarboxylates and isocyanides in acetonitrile (Scheme 3) (16CC900) and of tetracyanoethylene with ketones bearing α-protons and aliphatic/aromatic aldehydes in acetic acid (16OL1940).

Rhodium(II)-promoted reactions of 3-diazoindolin-2-imines with α,β-unsaturated aldehydes affords indole-fused 4-imino-3,4-dihydro-2*H*-pyrans, while with aromatic aldehydes and subsequent treatment with base, indole-fused 4*H*-chromen-4-ones were formed (16OL3682). A series of thiazolo[4,5-*b*]-3,4-dihydro-2*H*-pyrans were attained through [2 + 4]-annulation reactions of allenoates with 5-alkylidenethiazol-4-ones mediated by dimethylphenylphosphine. The asymmetric version used Kwon's phosphine as chiral ligand (16OL3418). Cinchona-derived tertiary amine **2** catalyzes the annulation of MBH carbonates of isatins and methyl acrylate with α-cyano-α,β-unsaturated ketones to give dihydrofurano indolo[2,3-*b*]-3,4-dihydropyrans (Scheme 4) (16CC11104).

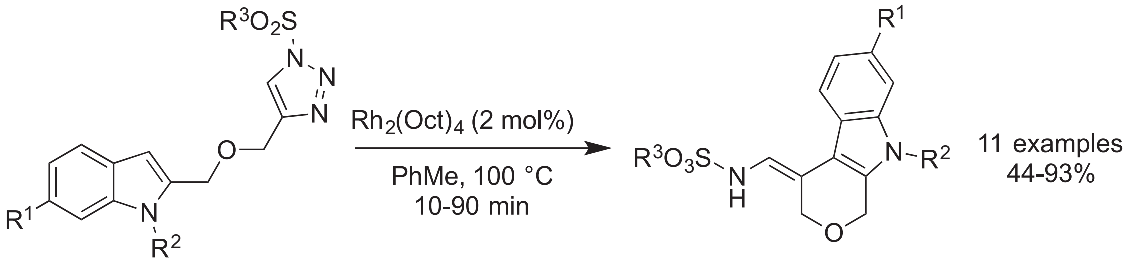
An enantioselective cascade sequence of β,γ-unsaturated α-ketoesters with alkynyl alcohols or amides affords spirotetrahydrofuran or spiropyrrolidine 3,4-dihydro-2*H*-pyrans, respectively, under dual catalysis of a gold(I)/*N*,*N*′-dioxide nickel(II) complex (16AGE6075). A wide range of dihydrospiro[indoline-3,4′-pyrano[2,3-*c*]pyrazole] derivatives were attained through enantioselective Michael/hemiketalization reaction of isatylidene β,γ-unsaturated α-ketoesters with pyrazolin-5-ones mediated by a chiral L-proline-derived squaramide organocatalyst (16OL1354). Gold(I)-catalyzed [2 + 2 + 2]-cycloaddition of 2-ethynyl-1-prenyl-1*H*-indole with aromatic aldehydes led to some tetracylic pyrroloindole-fused 3,6-dihydro-2*H*-pyrans in moderate-to-good yields (16T3647).

High yields of 6-aryl-3,4-dimethyl-5,6-dihydro-2*H*-pyrans were accomplished via [4 + 2]-hDA reaction of aromatic aldehydes with 2,3-dimethylbuta-1,3-diene using a zwitterionic metal–organic framework (MOF) catalyst (16JA10293). Diastereoselective $AgBF_4$-mediated cyclization of propargyl benzoates **3** provides diversely substituted 5,6-dihydro-2*H*-pyrans. This strategy is applied to the synthesis of C-1–C-12 subunit of natural madeirolide A (Scheme 5) (16OL1744).

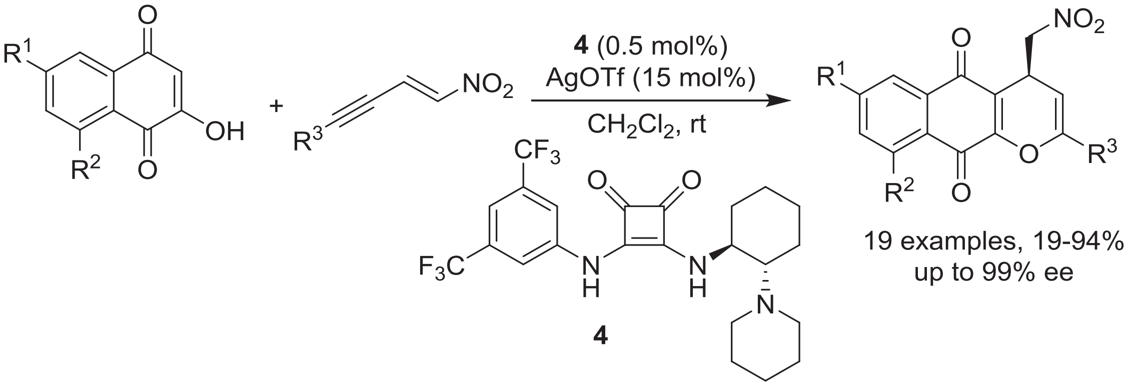
A variety of 2,6-disubstituted 5,6-dihydro-2*H*-pyrans result from a Prins cyclization/elimination reaction of but-4-en-2-ol with aldehydes in a two-step or in a one-pot procedure. In the former case, cerium(III) chloride and lithium iodide were added to produce the corresponding 4-iodotetrahydropyrans, which in the presence of indium(III) acetate and lithium iodide afford the corresponding 5,6-dihydro-2*H*-pyrans. The one-pot protocol used a TMSBr/lithium iodide/indium(III) acetate catalytic system to give the 2,6-disubstituted 5,6-dihydro-2*H*-pyrans in moderate yields (16T318). Rhodium(II)-promoted intramolecular annulation of 4-(indol-2-ylmethoxymethyl)-1-sulfonyl-1,2,3-triazoles in toluene at 100°C led to indolo[2,3-*c*]-5,6-dihydro-2*H*-pyrans (Scheme 6) (16T3014).



Scheme 5



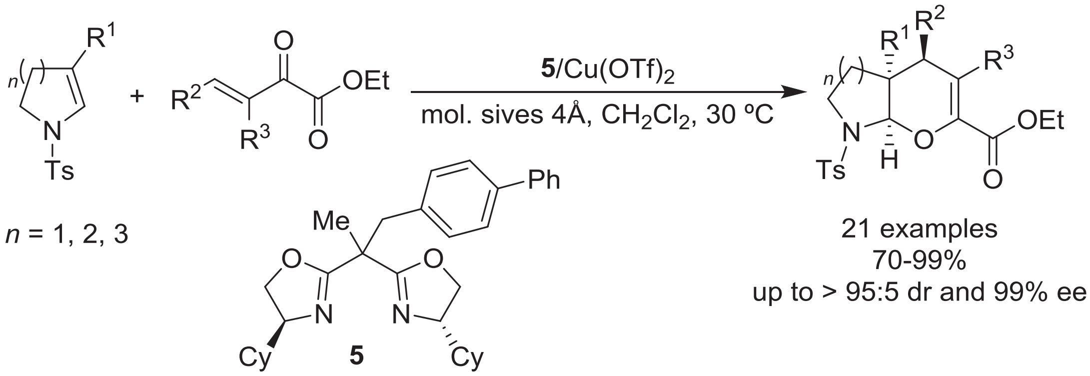
Scheme 6



Scheme 7

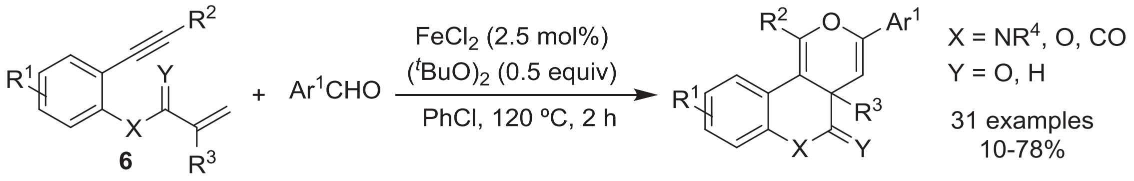
The synthesis of pentasubstituted 4*H*-pyrans can be achieved via multicomponent reactions of 1,3-dicarbonyl compounds with aromatic aldehydes promoted by $NbCl_5$ (16TL5050). Other chiral derivatives arise from [3 + 3]-annulation reaction of β′-acetoxy allenoates with 3-oxonitriles mediated by a *Cinchona* alkaloid-based catalyst in the presence of sodium carbonate and using ethyl acetate as solvent (16JA7872). Under dual catalysis of a *Cinchona*-derived squaramide and AgOTf, one-pot Michael addition/hydroalkoxylation reactions of 2-hydroxy-1,4-naphthoquinones with 1-nitrobut-1-en-3-ynes provide a wide range of naphthoquinono[2,3-*b*]-4*H*-pyrans (Scheme 7) (16CC1669).

The synthesis of coumarino[4,3-*b*]-2,4-diaryl-4*H*-pyrans can be achieved through the reaction of 4-hydroxycoumarin with various chalcones using catalytic amounts of SiO_2–$ZnCl_2$ in neat conditions (16CPB399) and mediated by a quinine-derived primary amine in the presence of *N*-Boc-D-phenylglycine (16OBC623) or through microwave-assisted three-component reaction of 4-hydroxycoumarin with benzaldehydes and methyl ketones, under catalyst- and solvent-free conditions (16TL476). Asymmetric hDA reaction of β,γ-unsaturated α-ketoesters with cyclic enamines catalyzed by Cy-SaBOX **5**/$Cu(OTf)_2$ system provides highly functionalized nitrogen heterocycle-fused 4*H*-pyrans in high yields and diastereo- and enantioselectivities (Scheme 8) (16AGE9220).

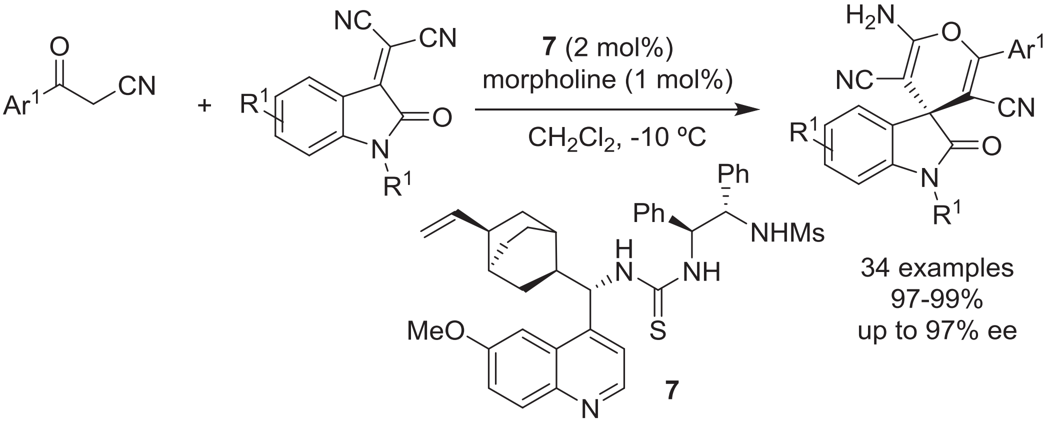


Scheme 8

Quinine-derived catalysts were used in the enantioselective synthesis of pyrazolo[5,4-*b*]-4*H*-pyrans via annulation reactions of pyrazolin-5-ones with β′-acetoxy allenoates (16JA7872) and with chalcones (16OBC623). Diversely substituted pyrazolo[5,4-*b*]-2-amino-4*H*-pyran-3-carbonitriles arise from three-component reactions of pyrazolin-5-ones with benzaldehydes and malononitrile promoted by sulfonic acid-functionalized magnetic $Fe_{3-x}Ti_xO_4$ nanoparticles (16SC745) and from four-component reaction of benzaldeydes with malononitrile, hydrazines, and ethyl acetoacetate in choline chloride/urea deep eutectic solvent (16SC220) or mediated by 1,5,7-triazabicyclo-[4,4,0]-dec-1-ene-anchored mesoporous silica nanoparticles in a refluxing 1:1 mixture of ethanol/water (16AJC1117) and other β-ketoesters catalyzed by L-proline under ultrasonic irradiation (16T7599). Four-component reactions of dialkyl acetylenedicarboxylates with isocyanides, hydrazines, and ethyl acetoacetate deliver pyrazolo[4,5-*b*]-2-aminosubstituted-4*H*-pyran-3,4-dicarboxylates under catalyst- and solvent-free conditions (16TL1435). A wide range of thiazolo[4,5-*b*]-2-amino-4*H*-pyran-3-carbonitriles was produced through [2 + 4]-annulation reactions of malononitrile with 5-ylidenethiazol-4-ones in the presence of a squaramide catalyst in diethyl ether at room temperature (16TA1056). Tandem annulation reactions of 2-ethynylanilines with benzaldehydes and nitriles using triflic acid (TfOH) as catalyst and 1,2-DCE as solvent provided quinolino[4,3-*b*]-4*H*-pyrans in moderate-to-good yields (16T4423). Quinolino[4,3-*b*]-2-amino-4-aryl-4*H*-pyran-3-carbonitriles were synthesized via three-component reactions of 1-methylquinoline-2,4(1*H*,3*H*)-dione with benzaldehydes and malononitrile/ethyl cyanoacetate promoted by 1,8-diazabicyclo(5.4.0)undec-7-en-8-ium acetate in refluxing water (16JHC1911). Three-component reactions of 2*H*-pyrido[1,2-*a*]pyrimidine-2,4(3*H*)-diones with isocyanides and dialkyl acetylenedicarboxylates



Scheme 9



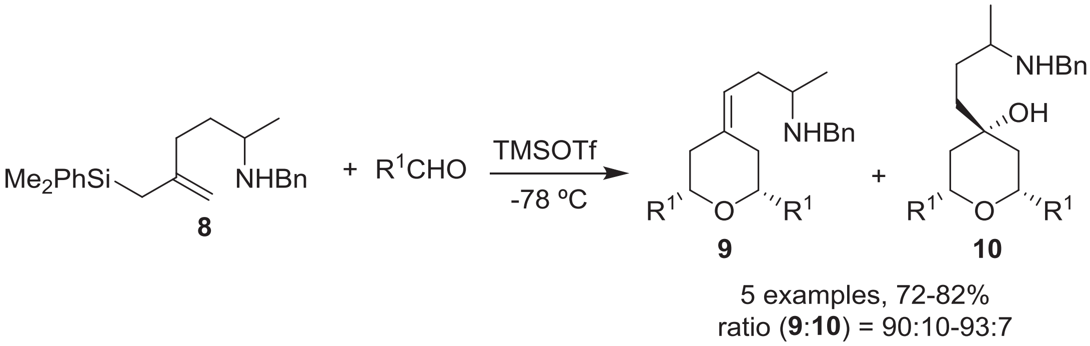
Scheme 10

in dimethylformamide (DMF) at 100°C led to pyridopyrimidine-fused 2-aminosubstituted-4*H*-pyran-3,4-dicarboxylates (16TL100). Various (hetero)hexacyclic-fused 4*H*-pyrans were synthesized through $FeCl_2$-catalyzed radical [2 + 2 + 2]-annulation of benzene-linked 1,7-enynes **6** with aromatic aldehydes in the presence of di-*t*-butyl peroxide and using chlorobenzene as solvent (Scheme 9) (16OL2264).

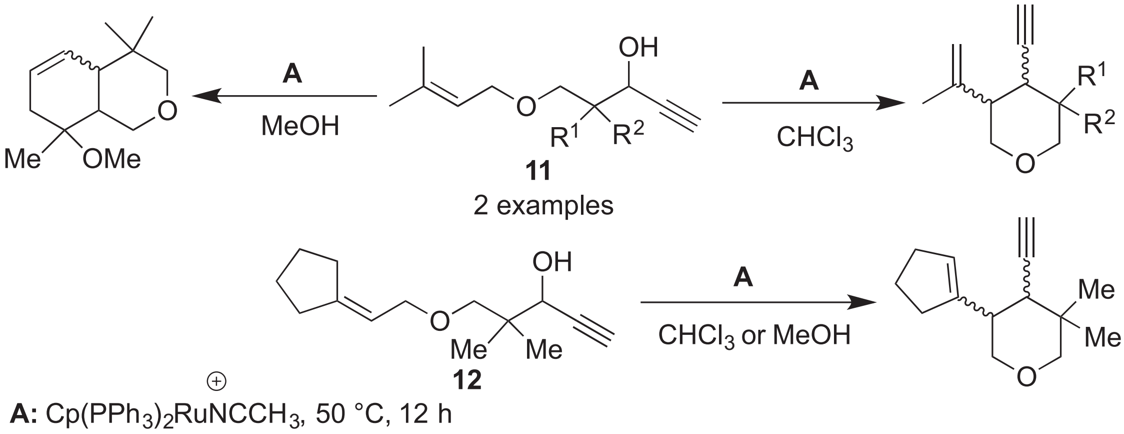
Excellent yields of chiral spirooxindole 2-amino-6-aryl-4*H*-pyran-3,5-dicarbonitriles are obtained from the organocatalytic cascade reaction of 1-aryl-2-cyanoethan-1-ones with isatylidene malononitriles in the presence of morpholine in dichloromethane at −10°C (Scheme 10) (16EJO3983). Other isatylidene malononitriles undergo organocatalyic asymmetric Michael/cyclization cascade reaction with pyrazolin-5-ones to construct spiro[indoline-3,4′-pyrano[2,3-*c*]pyrazole] derivatives (16OBC8346).

The synthesis of 2,3-disubstituted tetrahydropyrans can be accomplished through tandem vinylcyclopropane ring-opening/Prins cyclization reactions of (*E*/*Z*)-5-cyclopropylpent-4-en-1-ol with aliphatic/aromatic aldehydes carried out in the presence of $HBF_4 \cdot OEt_2$ in dichloromethane (16TL1889). $AlCl_3$-promoted Prins cyclization reaction of but-3-en-1-ol with benzaldehydes and using trimethylsilyl (TMS) bromide/chloride or $BF_3 \cdot OEt_2$ as halide sources provides mainly *cis*-2-aryl-4-halotetrahydropyrans (16JOC5144). A regio- and enantioselective synthesis of 2-alkyl/aryl-4-methylenetetrahydropyrans used a highly acidic confined imino-imidodiphosphate catalyst to promote the reaction of 3-methylbut-3-en-1-ol with aliphatic/aromatic aldehydes (16JA10822). A mixture of 2,4,6-trisubstituted tetrahydropyrans **9** and **10** results from tandem Sakurai–Prins cyclization reactions of allylsilyl amines **8** with aldehydes mediated by trimethylsilyl trifluoromethanesulfonate (TMSOTf) with subsequent proton elimination or addition of water, respectively (Scheme 11) (16OL1972). Ruthenium-catalyzed domino cyclization of 1,8-enynes **11** bearing two geminal methyl groups at the double bond is solvent dependent: in chloroform, tetrahydropyrans are formed, while in methanol tetrahydroisochromans result. In the case of 1,8-enyne **12**, only a tetrahydropyran derivative was formed in either solvent (Scheme 12) (16JOC4494).

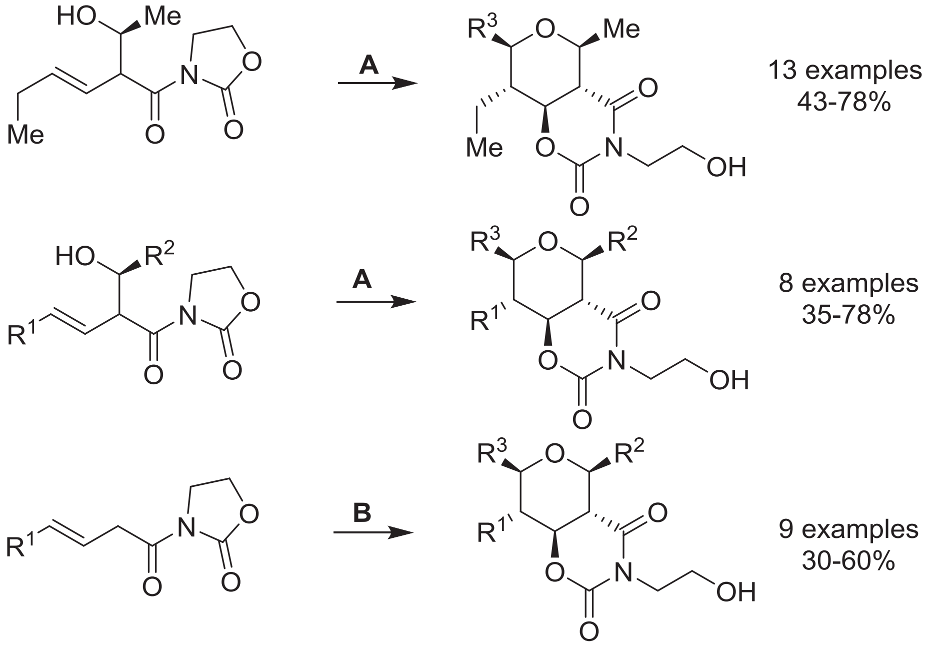
Gold(I)-catalyzed intramolecular hydroalkoxylation of 1-(but-3-yn-1-yl)tetrahydropyran-2-ols in the presence or absence of 1,1,1,3,3,3-hexafluoropropan-2-ol in tetrahydrofuran (THF) affords a mixture of tetrahydropyrano[3,2-*b*]tetrahydropyrans and tetrahydroxepino[3,2-*b*]tetrahydropyrans (16CPB845). Various examples of piperidino[3,4-*c*]tetrahydropyrans arise from cascade reactions of (*E*)-5-(3-phenylprop-2-yn-1-ylamino)pent-3-en-1-ols with aliphatic/aromatic aldehydes in the presence of $BF_3{\cdot}OEt_2$ in dichloromethane at 0°C (16OBC11396). Highly substituted



Scheme 11



Scheme 12



A: R^3CHO (1.5 equiv), $BF_3 \cdot OEt_2$ (2.5 equiv), CH_2Cl_2,rt, 30 min

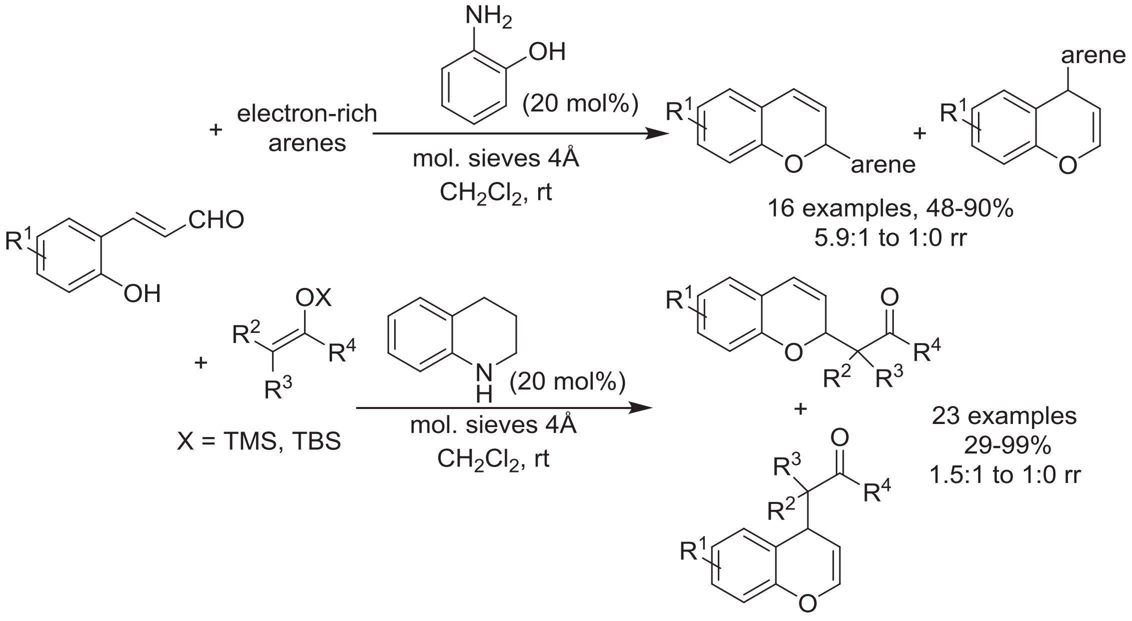
B: 1. i) TEA (1.3 equiv), $^nBu_2BOTf$ (1.2 equiv), CH_2Cl_2, -78 °C, 30 min
ii) 0 °C, 20 min; then, -78 °C
iii) R^2CHO (1 equiv), -78 °C to rt, 15 h
2. R^3CHO (1 equiv), $BF_3 \cdot OEt_2$ (2.5 equiv), rt, 30 min

Scheme 13

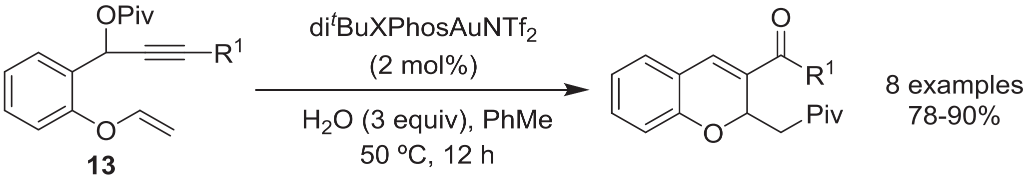
heterocycle-fused tetrahydropyrans are produced via two steps in a one-pot Evans–Aldol–Prins cyclization reaction of β,γ-unsaturated *N*-acyl oxazolidin-2-ones with aldehydes in moderate-to-good yields (Scheme 13) (16CC3380). TMSOTf promotes a domino reaction of (*R*)-3-hydroxyhex-5-enoic acid with aliphatic/aromatic aldehydes in dichloromethane to provide a variety of 7-alkyl/aryl-2,6-dioxabicyclo[3.3.1]nonan-3-ones (16OBC8832). The synthesis of 3-spirooxindole 2,5-disubstituted tetrahydropyran-4-carboxylates can be accomplished through an organocatalyzed Michael/Aldol/hemiacetalization cascade of 2-(2-oxoindolin-3-ylidene) esters with aliphatic aldehydes in moderate-to-good yields and with high diastereo- and enantioselectivities (16OL2387).

6.4.2.2 [1]Benzopyrans and Dihydro[1]benzopyrans (Chromenes and Chromans)

Structurally diverse 2-substituted 2*H*-chromenes result from an arylamine-catalyzed cyclization/substitution cascade reaction of 2-hydroxycinnamaldehydes with various nucleophiles, such as indoles, pyrroles, naphthols, phenols, and silyl enol ethers (Scheme 14) (16CEJ9240). Vinyl ethers of



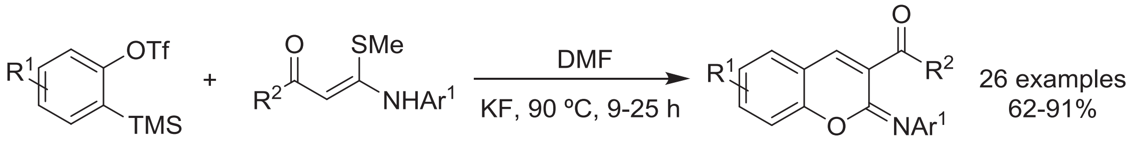
Scheme 14



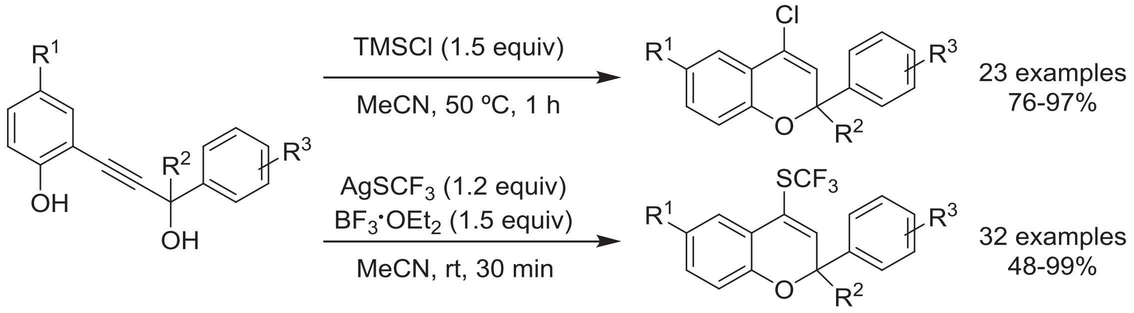
Scheme 15

1,7-enyne esters **13** undergo gold(I)-mediated cycloisomerization reactions, with concomitant pivaloyl group migration during formation of the final products by 7687 carbon–gold displacement, to afford 2,3-disubstituted 2*H*-chromenes in high yields (Scheme 15) (16CC7687).

A three-component coupling reaction of arynes with *N*,*S*-keteneacetals and DMF carried out in the presence of KF provides 3-aroyl-2-arylimino-2*H*-chromenes (Scheme 16) (16JOC5942). Using a solvent-free ball-milling procedure, an oxa-Michael/Henry cascade, of salicylaldehydes with nitrostyrenes produced a series of 2-aryl-3-nitro-2*H*-chromenes in 60%–97% yields (16S407). A wide variety of 2,3-diaryl-2-methoxy-2*H*-chromenes were obtained through [4 + 2]-cycloaddition reactions of salicylaldehydes with diarylacetylenes promoted by TfOH in the presence of trimethyl orthoformate in toluene at 60°C, followed by the addition of methanol (16TL5914). Under dual catalysis of an arylboronic acid and a Brønsted acid, phenols react with α,β-unsaturated carbonyl compounds to give 2,2- and 2,4-disubstituted and also 2,2,4-trisubstituted 2*H*-chromenes (16OBC6703). Further examples arise from a successive *o*-alkenylation/*O*-alkenylation of phenols with



Scheme 16

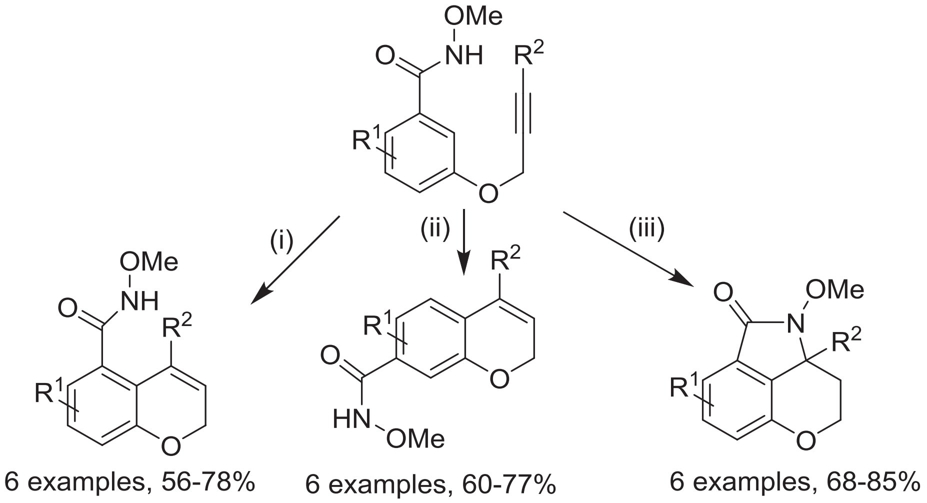


Scheme 17

phenylacetylene and subsequent cyclization catalyzed by the Ph_3PAuCl–$SnCl_2$ composite (16EJO5678). Various 3-(2-hydroxyaryl)propargyl alcohols undergo TMSCl- and $AgSCF_3$-$BF_3{\cdot}OEt_2$-mediated cascade cyclization reactions to give 2-aryl-, 2,2-diaryl-4-chloro- (16TL4519), and 4-(trifluoromethyl)thio-2*H*-chromenes (16OL1514), respectively (Scheme 17).

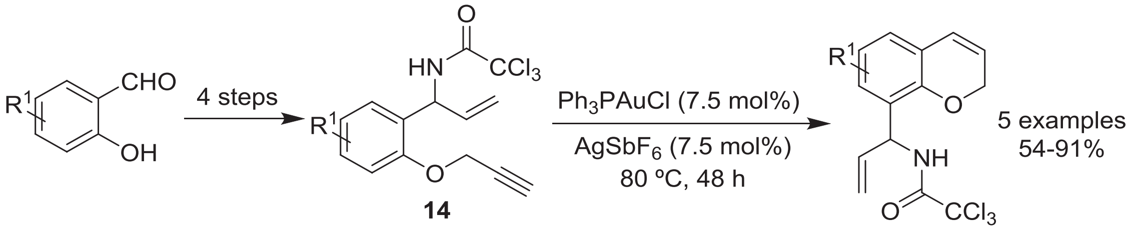
One-pot syntheses of 4-alkyl/aryl-3-iodo-2*H*-chromenes can be achieved though *O*-arylation of 3-alkyl/arylprop-2-yn-1-ols with diaryliodonium triflates and *t*-BuONa followed by iodocyclization carried out in the presence of *N*-iodosuccinimide (NIS) and $BF_3{\cdot}OEt_2$ (16OL944). The reaction of (3-arylprop-2-yn-1-yl)oxybenzenes with trifluoromethanesulfanylamide mediated by $BiCl_3$ in 1,2-DCE at 50°C led to the regioselective synthesis of 4-aryl-3-(trifluoromethylthio)-2*H*-chromenes (16T1472). Gold(I)-catalyzed intramolecular hydroarylation of alkynes used different phosphine ligands to promote regiodivergent cyclization at *o*- or *p*-positions to give 5- or 7-substituted 2*H*-chromenes (Scheme 18). Exclusive *p*-cyclization occurs with the assistance of a rigid electron-abundant phosphine ligand-based gold(I) catalyst. Combining gold and silver catalysts produced a series of tricyclic chromans (Scheme 18) (16JA5218).

An $AlCl_3$-catalyzed annulation reaction of terminally substituted ynamides with *o*-quinone methides led to 4-amino-2*H*-chromenes, while terminally unsubstituted ynamides underwent a [4 + 2]-cyclization to afford 2-amino-4*H*-chromenes (16OL5022). Allylic trichloroacetimidates **14** bearing a 2-propargyloxyaryl group, prepared in a four-step synthesis from salicylaldehydes, underwent Overman rearrangement and subsequent



(i) (2,4-^{t}Bu$_2$PhO)$_3$PAuOTf (5 mol%), 1,2-DCE, 80 °C, 8 h
(ii) XphosAuNf$_2$ (5 mol%), 1,2-DCE, 80 °C, 8 h
(iii) (2,4-^{t}Bu$_2$PhO)$_3$PAuCl (5 mol%), AgOTf (10 mol%), PhCl, 110 °C, 12 h

Scheme 18

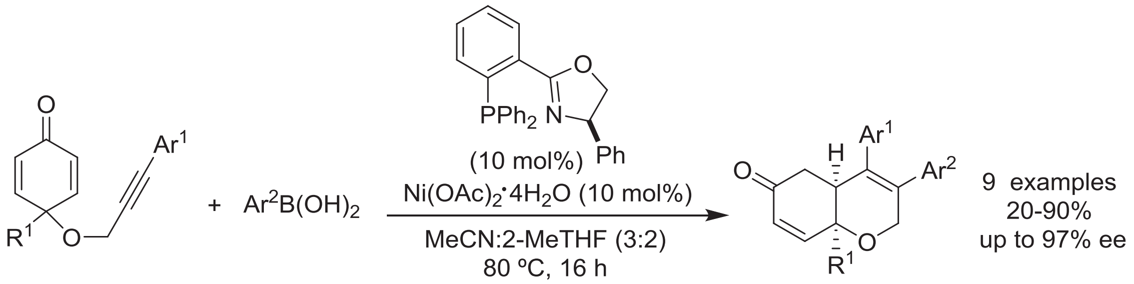


Scheme 19

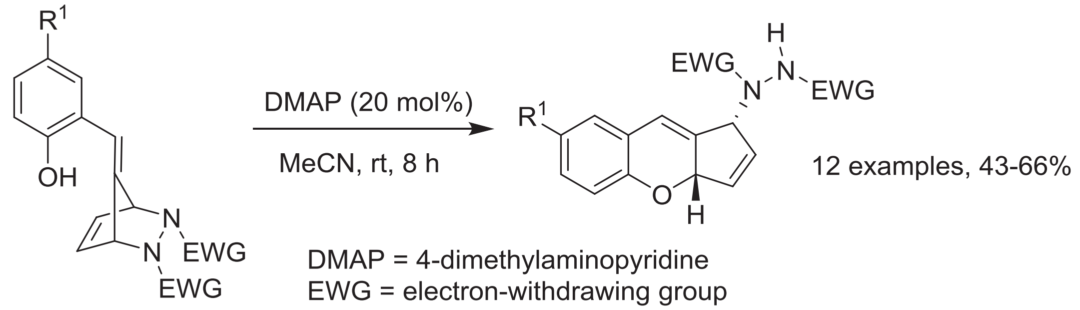
gold(I)-catalyzed hydroarylation to give 8-functionalized 2*H*-chromenes (Scheme 19) (16JOC9810).

Intramolecular direct arylation of 2-(aryloxymethyl)-1-bromobenzenes occurs in the presence of *N*,*N*-dimethylacetamide (DMA), potassium acetate, and PdO–Fe_3O_4 at 140°C for 48 h giving rise to 6*H*-benzo[*c*]chromenes (16T1043). Similar derivatives can be prepared through a palladium(0)-catalyzed Suzuki coupling reaction of 2-hydroxyarylboronic acids with ethyl 3-(2-bromovinyl/aryl)propenoates followed by Michael addition, in a one-pot protocol (16JHC919). Several 3,4-diaryl-4a,8a-dihydro-2*H*-chromen-6(5*H*)-ones were made by enantioselective nickel(II)-promoted reaction of arylboronic acids with arylpropargyloxy tethered to cyclohexa-1,3-dienones, in 20%–90% yields and up to 97% enantiomeric excess (ee) (Scheme 20) (16JA8068).

Using an intramolecular ring-opening/ring-closure strategy allowed a series of phenol-substituted fulvene-derived azabicyclic olefins to rearrange in the presence of 4-dimethylaminopyridine (DMAP) in acetonitrile at

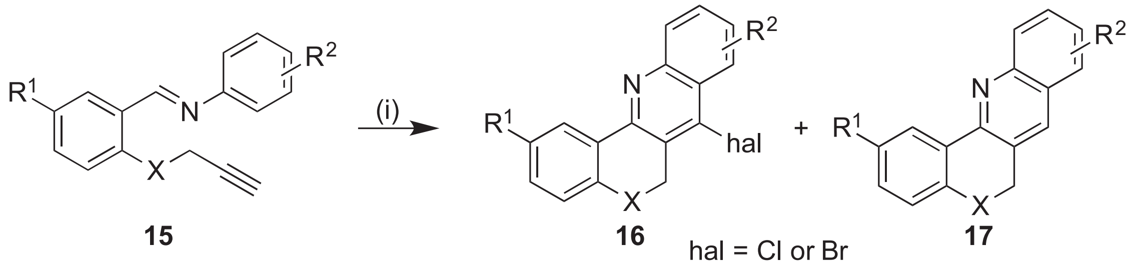


Scheme 20



DMAP = 4-dimethylaminopyridine
EWG = electron-withdrawing group

Scheme 21



(i) Cu_2O (10 mol%), chloranil or bromanil (2 equiv), NaCl (1.5 equiv), 1,2-dimethoxyethane, 80 °C, 8 h

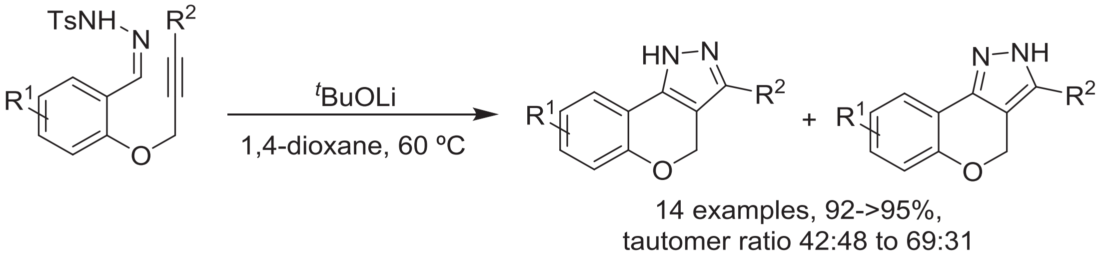
X = O, hal = Cl, 9 examples, **16** 38-65%, **17** 0-26%
X = O, hal = Br, 1 example, **16** 56%, **17** 24%
X = S, hal = Cl, 10 examples, **16** 21-57%, **17** 0-27%
X = S, hal = Br, 1 example, **16** 45%, **17** 23%

Scheme 22

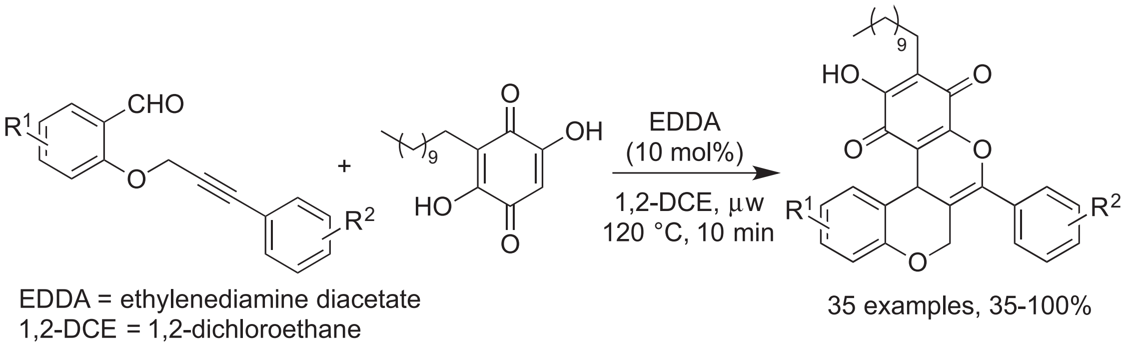
room temperature to cyclopenta[*b*]chromenes (Scheme 21) (16TL2965). Tricyclic glycofused chromene derivatives arise from organocatalyzed domino oxa-Michael addition/aldol condensation of salicylaldehydes with protected 3-oxoglucal (16EJO1660). A copper(I)-catalyzed cascade reaction of Schiff bases **15** with chloranil or bromanil gave a mixture of haloquinoline-fused chromenes **16** as major products along with small amounts of the nonhalogenated derivatives **17** (Scheme 22). Replacing the oxygen atom on the starting material by sulfur led to similar halogenated and nonhalogenated quinolino[3,2-*c*]thiochromenes (Scheme 22) (16OL2491).

Intramolecular [3 + 2]-cycloaddition reactions of propargyloxy-tethered *N*-tosylhydrazones carried out in the presence of *t*-BuOLi in 1,4-dioxane at 60°C delivers a mixture of tautomers of pyrazolo[4,5-*c*]- and [4,3-*c*]chromenes in excellent yields (Scheme 23) (16JOC11072). Further examples arise from condensation of substituted *O*-propargyl salicylaldehydes with tosylhydrazine followed by 1,3-dipolar cycloaddition in a one-pot synthesis (16S4207). Other substituted *O*-propargyl salicylaldehydes underwent tandem [3 + 2]-cycloaddition/oxidation reactions with proline or pipecolic acid under an oxygen atmosphere to prepare pyrrolizino[3,2-*c*]- or indolizino[3,2-*c*]chromenes, respectively (16BCSJ1331). Microwave-assisted one-pot Knoevenagel condensation of *O*-(arylpropynyloxy)salicylaldehydes with embelin promoted by ethylenediamine diacetate (EDDA) and a subsequent intramolecular hDA reaction provided highly substituted chromene-type-[3,4-*c*]chromene derivatives (Scheme 24) (16JOC9738).

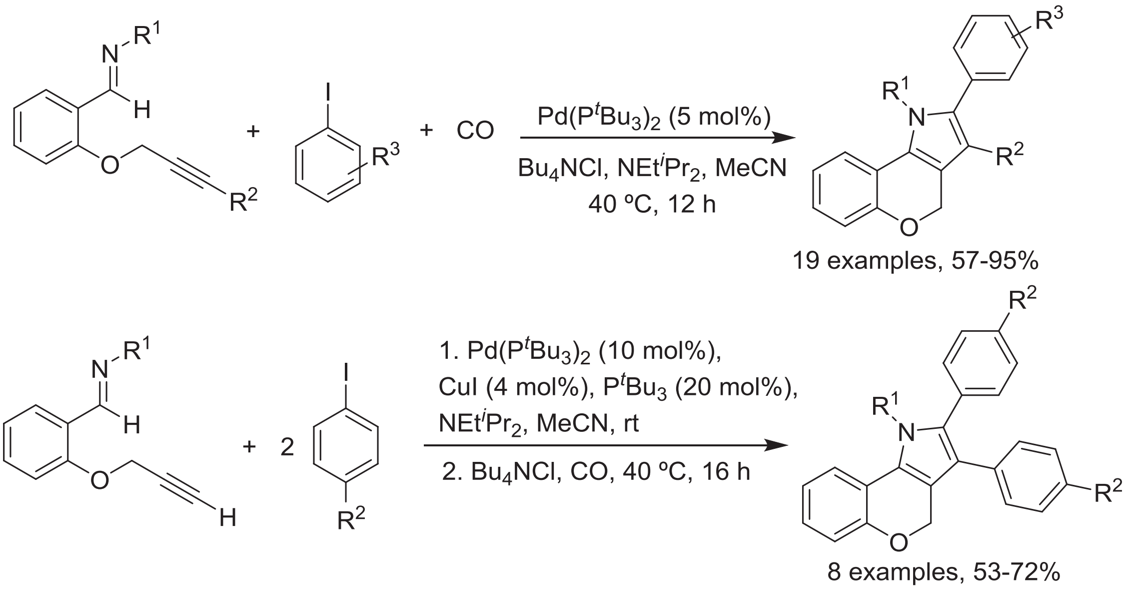
A variety of pyrrolo[3,2-*c*]chromenes has been synthesized through multicomponent reaction of 2-propargyloxy benzyl imines with (hetero)aryl iodides and carbon monoxide mediated by a palladium(0) catalyst (Scheme 25) (16JOC11145). Several 1-aryloxy-3-(2-iodoaryloxy)propan-2-ones undergo BCl_3-mediated regioselective ring closure to give 3-[(2-iodoaryloxy)methyl] benzofurans, which suffer palladium(II)-catalyzed intramolecular direct arylation to afford 6*H*-benzofuro[3,2-*c*]chromenes (16OBC8074).



Scheme 23



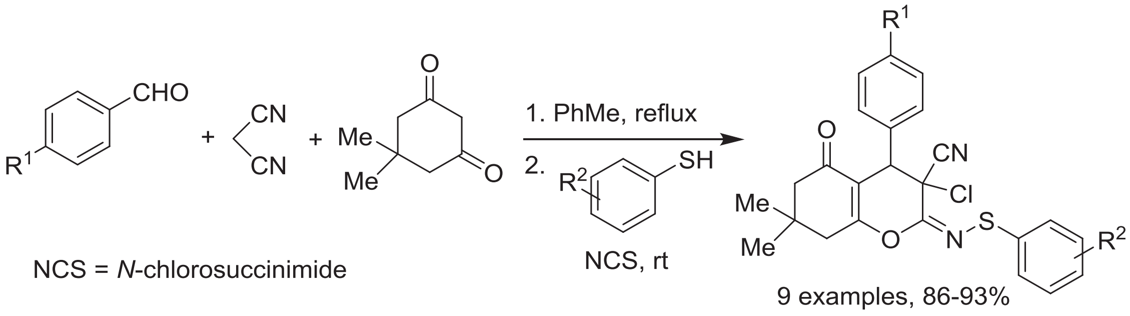
Scheme 24



Scheme 25

An unexpected K_3PO_4-mediated intramolecular Rauhut–Currier-type reaction of chalcones, bearing an enolate of a 1,3-dicarbonyl compound as substituent, using THF as solvent, allows the preparation of 3,4-disubstituted 4*H*-chromenes (16OBC5612). A variety of 2,4-diaryl-4*H*-chromenes were produced from enantioselective nucleophilic addition of phenols to benzopyrylium salts (16JA9775). High yields and enantioselectivity were achieved in the Michael addition/cyclization reactions of 2-(nitrovinyl) phenols with malononitrile mediated by a C_3-symmmetric cinchonine–squaramide catalyst to prepare 2-amino-4-(nitromethyl)-4*H*-chromene-3-carbonitriles (16TA670). The synthesis of 2-amino-4-aryl-4*H*-chromene-3-carbonitrile derivatives are promoted by the reaction of benzaldehydes with malononitrile and 3-substituted phenols using catalytic amounts of piperidine in refluxing ethanol (16JHC1778) and using a bifunctional nanostructured molten salt [4,4′-bipyridine]-1,1′-diium tricyanomethanide catalyst, under solvent-free conditions (16SL1418). One-pot multicomponent reaction of salicylaldehydes with malononitrile and 2-cyanoacetohydrazide carried out in the presence of K_3PO_4 in refluxing ethanol provides 2-amino-4-(3-amino-5-hydroxy-4*H*-pyrazol-4-ylidene)-4*H*-chromene-3-carbonitriles in high yields (16SL1720).

Structurally diverse 2-substituted 4-alkyl/aryl-4*H*-chromene-3-carbonitrile/carboxylate-type derivatives arise from one-pot three-component reaction of cyclic 1,3-dicarbonyl compounds with aliphatic/aromatic aldehydes and activated cyanomethylene compounds in the presence of ammonium acetate in ethanol at room temperature (16HCA724), of chitosan in

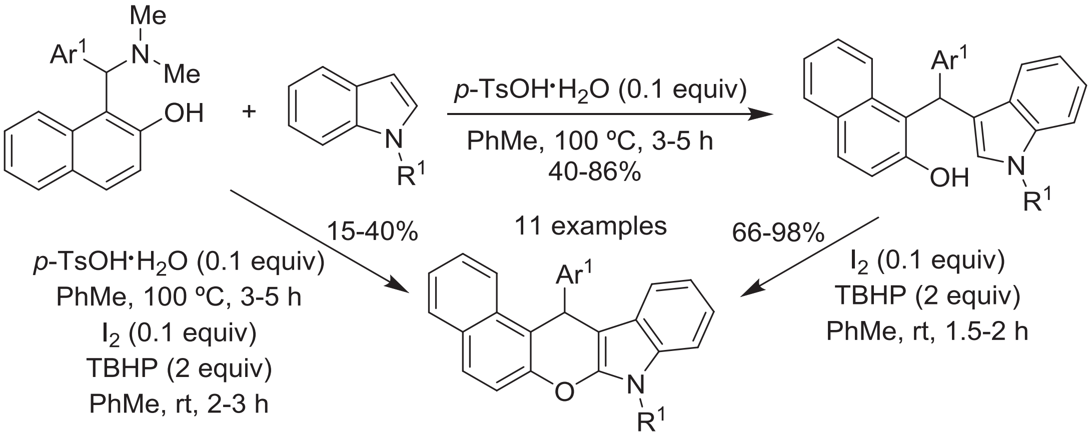


Scheme 26

refluxing 1,4-dioxane (16JHC1892), of polyethylene glycol (PEG-600) in water (16SC1665), of ionic liquid diethylene glycol-bis(3-methylimidazolium) dihydroxide in water at room temperature (16TL361), of sulfonic acid-functionalized magnetic $Fe_{3-x}Ti_xO_4$ nanoparticles in a refluxing 1:1 mixture of ethanol/water (16SC745), of *N*,*N*-dimethylbenzylamine in ethanol at 45°C (16H(92)75), or using polystyrene-supported *p*-toluenesulfonic acid (16TL1289) and potassium-exchanged zirconium hydrogen phosphate (16S1533) as catalysts under solvent-free conditions. Similar 2-sulfenylimine chroman-type derivatives are obtained through one-pot reaction of benzaldehydes with malononitrile and dimedone in refluxing toluene followed by the addition of thiophenol and *N*-chlorosuccinimide at room temperature (Scheme 26) (16OBC582).

A series of bicyclic and tricyclic hexahydro-4*H*-chromene-type compounds results from intramolecular annulation reactions of, respectively, α-geranyl and α-farnesyl β-ketosulfones mediated by tin triflate in nitromethane (16T5121). Under catalyst-free conditions, a wide range of 4-aryl-*N*-methyl-3-nitro-1*H*-benzo[*f*]chromene-3-amine derivatives were achieved through one-pot multicomponent reactions of naphth-2-ol with aldehydes and (*E*)-*N*-methyl-1-(methylthio)-2-nitroethenamine in a 1:1 mixture of ethanol/water (16T6484). The synthesis of indolo[2,3-*b*]benzo[*f*]chromenes occurs in one step or two steps involving the reaction of 1-(dimethylaminomethyl)naphth-2-ols with indoles in the presence of *p*-TsOH monohydrate followed by cyclization promoted with molecular iodine and *t*-butyl hydroperoxide (TBHP) (Scheme 27) (16EJO3441).

A wide variety of spirooxindole 2-amino-4*H*-chromene-3-carbonitrile-type derivatives arise from one-pot three-component reaction of isatin with cyclic 1,3-dicarbonyl compounds and malononitrile carried out in the presence of PEG-600 (16SC1665) and diethylene glycol-bis-(3-methylimidazolium) dihydroxide and potassium carbonate (16TL361) in water.

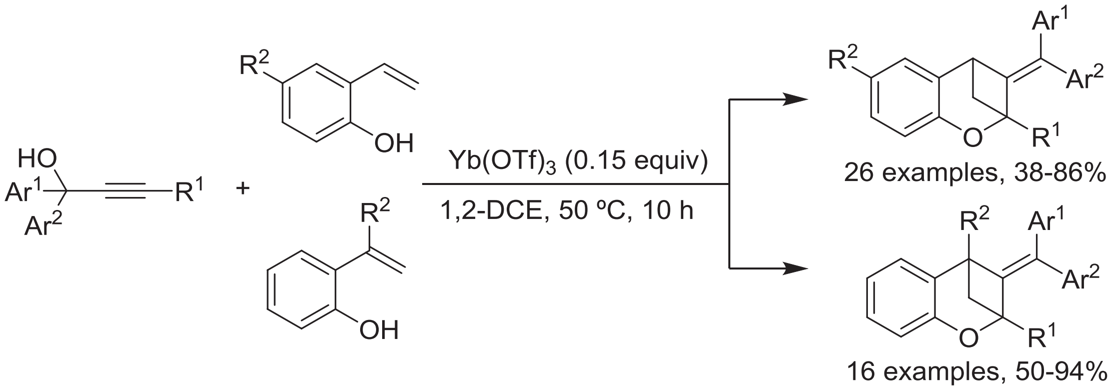


Scheme 27

Bis(spirooxindoles) are attained by replacing isatins by bis-isatins in ethylene glycol (16SC1880).

Using $FeCl_3$ and $AgBF_4$ as the catalytic system, the reaction of phenols with isoprene in 1,2-DCE at 60°C leads to 2,2-dimethylchromans in moderate-to-good yields (16EJO2925). A wide range of 2,3-bismethylenechromans are readily accessible through palladium(0)-catalyzed annulation reactions of alkynyl aryl ethers with allyl pivalates carried out in the presence of tricyclohexylphosphine and cesium carbonate in toluene at 140°C (16AGE8701). Knoevenagel condensation and cyclization reactions of benzaldehyes with 2-(2-hydroxyaryl)-1-nitroethanes mediated by KF, DABCO, and Me_2NH_2Cl in refluxing toluene provided several examples of *trans*-2-aryl-3-nitrochromans (16EJO2720). Enantioselective synthesis of 2-(indol-3-yl)-2-methyl-4-(nitromethyl)chromans was successful using organocatalyzed Michael addition/hemiketalization reactions of 2-[(*E*)-2-nitrovinyl]phenols with acetone followed by Friedel–Crafts alkylation of indoles promoted with $BF_3{\cdot}OEt_2$, in a one-pot protocol (16T240). A multistep approach was established for the synthesis of optically active *trans*-flavan-3-ol gallates starting from appropriate aryl bromides and epichlorohydrin (16CEJ13050). Calcium triflate/Bu_4NPF_6 promotes one-pot dehydrative coupling reactions of styrenes with 2-hydroxybenzylic alcohols followed by oxacyclization to afford 2-aryl-4-substituted chromans (16TL5841). Highly functionalized 3-diarylmethylidene-2,4-methanochromans were produced via cycloaddition of propargylic alcohols to 2-vinylphenols mediated by $Yb(OTf)_3$ (Scheme 28) (16OL3866).

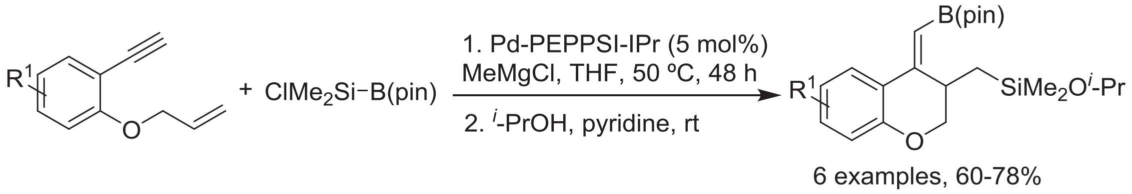
o-Quinone methides, formed in situ from 2-benzylphenols, undergo [4 + 2]-cycloaddition reactions with substituted styrenes in the presence of Ag_2O and 4 Å molecular sieves in toluene giving rise to 2,4-diarylchromans



Scheme 28

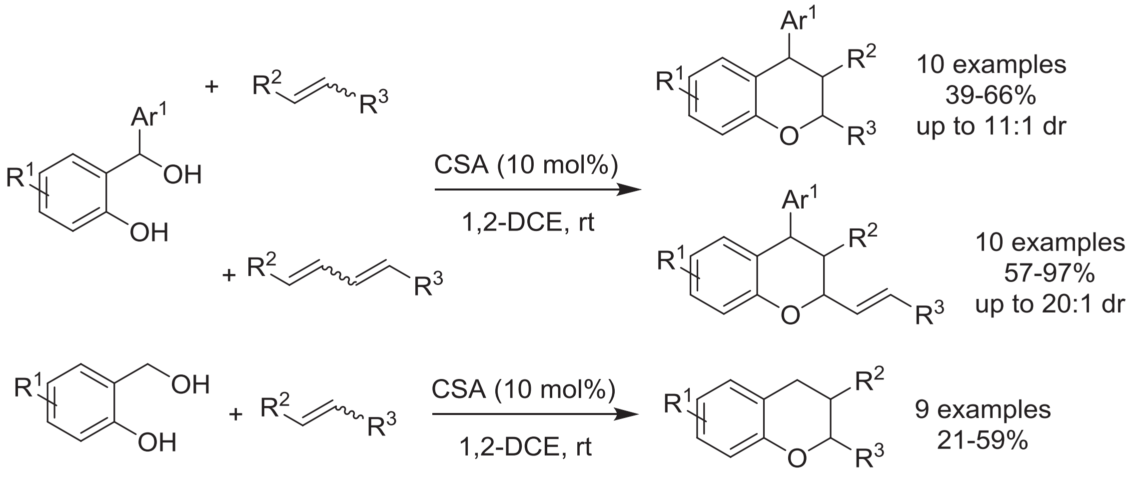
(16T2748). Various examples of 2,2,4-triarylchromans came from a three-component reaction of aromatic aldehydes with phenols and 1,1-diarylethylenes carried out in the presence of a sulfone-containing Brønsted acid ionic liquid as catalyst (16T479). The same catalyst was used in the three-component reaction of salicylaldehydes with 1,3-diphenylethylene and indoles to afford 2,2-diphenyl-4-(indol-3-yl)chromans and in the two-component reaction of salicylaldehydes with 1,1-diarylethylenes to give 2,2-diaryl-4-(2,2-diarylvinyl)chromans (16T479). Silaborative cyclization of 1-ethynyl-2-(prop-2-en-1-yloxy)benzenes occurs in the presence of (chlorodimethylsilyl)pinacolborane and a palladium(0) catalyst to furnish 3-methylsilyl-substitued chromans with boron attached to the exocyclic olefin at C-4 (Scheme 29) (16OL308). Several examples of 4-methyl-(4-methylborylated) 2-substituted chromans were made via diastereoselective palladium(0)-catalyzed domino Heck and arylborylation of 2-iodoaryl 1,3-disubstituted-but-3-enyl ethers in the presence of bis(pinacolato)diboron and potassium acetate in DMF (16S1483). Well-decorated chromans bearing a 3-selenofunctionality were prepared by a TMSOTf-catalyzed polystyrene-supported succinimidyl selenide intramolecular selenoarylation of alk-2-enyloxyarenes (16H(92)708).

A series of 3-benzyl/benzylidene-4-hydroxychromans are formed by photoredox-promoted formal hydroacylation of 2-(cinnamyloxy/arylpropargyloxy)benzaldehydes carried out in the presence of an iridium(III) catalyst and *N*,*N*-diisopropylethylamine (DIPEA) in acetonitrile (16JOC6959). (±)-*trans*-2-(Aryloxymethyl)-3-(halo)aryloxiranes undergo stereo- and regioselective intramolecular Friedel–Crafts epoxy-arene cyclization using *p*-TsOH monohydrate as catalyst to prepare (±)-*trans*-4-(halo)aryl-3-hydroxychromans (16T4878). A wide range of multisubstituted chromans was produced via a metal-free approach involving the reaction of



Pd-PEPPSI-IPr = dichloro-*N*,*N*-bis[2,6-(diisopropyl)phenyl]imidazolium (3-chloropyridine) palladium
B(pin) = pinacolborane

Scheme 29



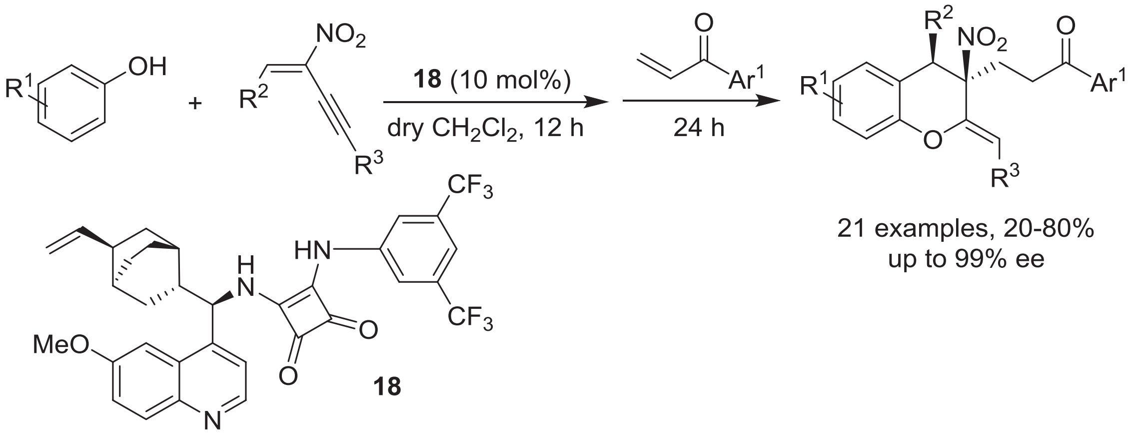
CSA = (+)-10-camphorsulfonic acid

Scheme 30

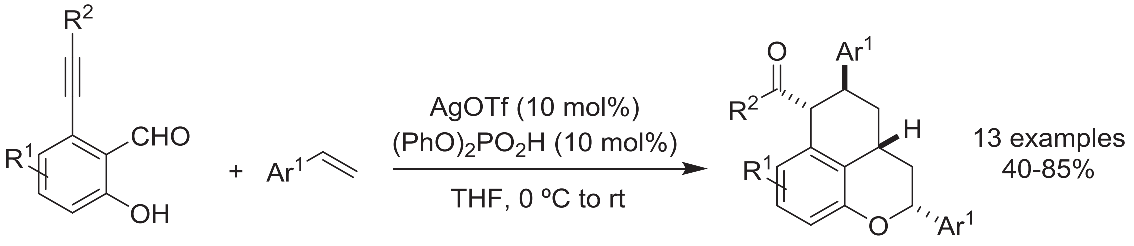
2-(hydroxybenzyl)phenols with alkenes and of *o*-hydroxybenzyl alcohols with monoenes or dienes using (+)-10-camphorsulfonic acid (CSA) as catalyst (Scheme 30) (16T7642). Highly functionalized chromans are obtained through one-pot three-component tandem reactions of electron-rich phenols with 2-nitro-1,3-enynes and vinyl ketones, mediated by bifunctional squaramide **18** in dry dichloromethane (Scheme 31) (16OL6276).

Regio- and diastereoselective synthesis of 2-hydroxy-2-trifluoromethytetrahydrochroman-3-carboxylate derivatives can be achieved through one-pot reaction of dimedone with aromatic aldehydes in the presence of a catalytic amount of piperidine in ethanol followed by the addition of methyl 4,4,4-trifluorobut-2-ynoate. Refluxing these chroman derivatives with *p*-TsOH gives the corresponding 4*H*-chromenes (16S2863). Silver triflate in the presence of diphenyl hydrogen phosphate catalyzes a cascade reaction of *o*-alkynylsalicylaldehydes with styrenes giving rise to tetrahydrobenzo[*de*]chromans in moderate-to-good yields (Scheme 32) (16CC13405).

Acyclic and cyclic ketones reacted with *O*-prenylsalicylaldehyde derivatives in the presence of 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) and glycerol at 120°C to furnish pyrano[3,4-*c*]chromans via domino Aldol/hDA reactions (16JOC4955). The synthesis of quinolino[3,2-*c*]chromans occurs

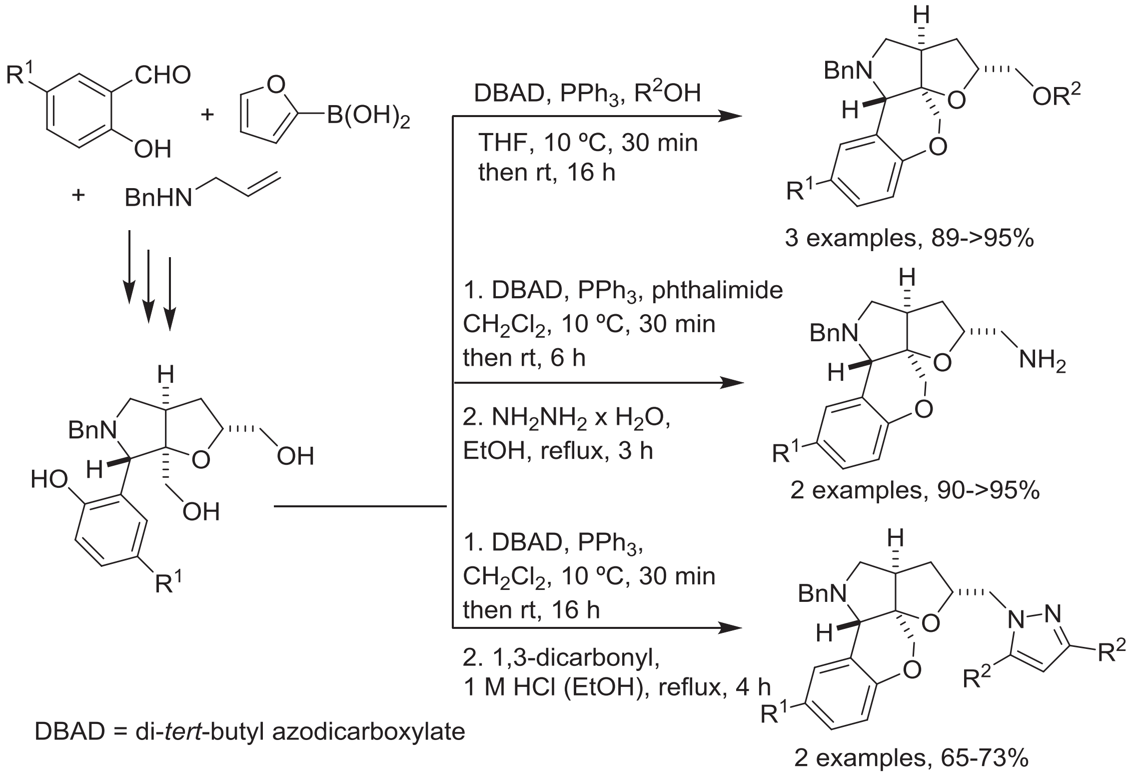


Scheme 31

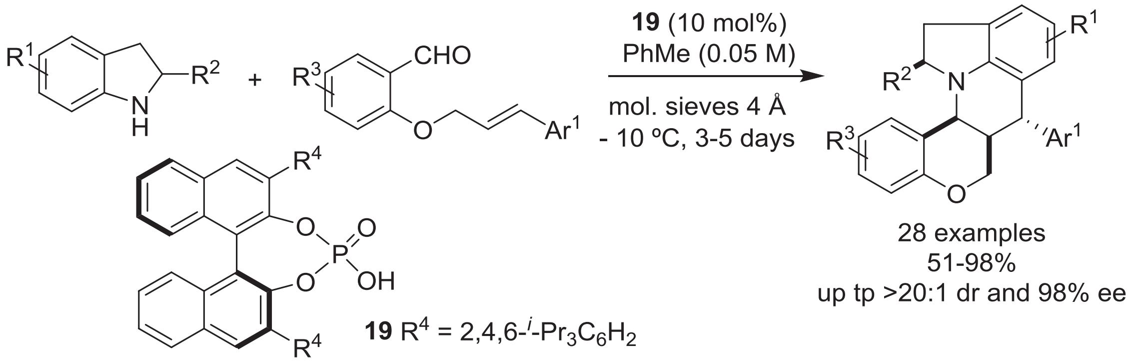


Scheme 32

through copper(I) chloride-mediated oxidative arylation/cyclization reactions of (3-arylpropargyloxy)benzonitriles with arylmesityliodonium triflates (16JOC920). Similar quinoline derivatives can be accessed by the reaction of 2-cinnamyloxybenzaldehydes with nitrobenzenes in aqueous citric acid using iron as reductant and montmorillonite K10 as catalyst (16EJO5706). Palladium(0)-catalyzed domino reactions of 1-iodo-2-[(3-methylbut-3-en-1-yl)oxy]benzenes with iodobenzenes in the presence of tetrabutylammonium bromide (TBAB) and potassium phosphate in *N*-methyl-2-pyrrolidone provides tetracyclic naphtha[*de*]chromans (16EJO5616). Other complex tetracyclic chromans result from a multistep strategy starting from salicylaldehydes, 2-furyl boronic acid, and *N*-benzylprop-2-en-1-amine. It involves a Petasis three-component reaction, DA reaction, oxidative cleavage, reduction, and Mitsunobu reactions (Scheme 33) (16OBC4943). A wide variety of pentacyclic chroman derivatives were obtained in high diastereo- and enantioselectivities through intramolecular Pavaroc reactions of racemic 2-alkyl/aryl indolines with 2-cinnamyloxybenzaldehydes using chiral phosphoric acid **19** in toluene at −10°C (Scheme 34) (16CEJ10817). One-pot condensation of aliphatic/aromatic aldehydes with 2-(5-hydroxy-3-methylidenepent-1-yl) phenol carried out in the presence of $BF_3 \cdot OEt_2$ in dichloromethane followed by Prins spirocyclization affords tetrahydrospiro[chroman-2,4′-pyran] derivatives in good-to-excellent yields (16OBC3234). Structurally diverse



Scheme 33

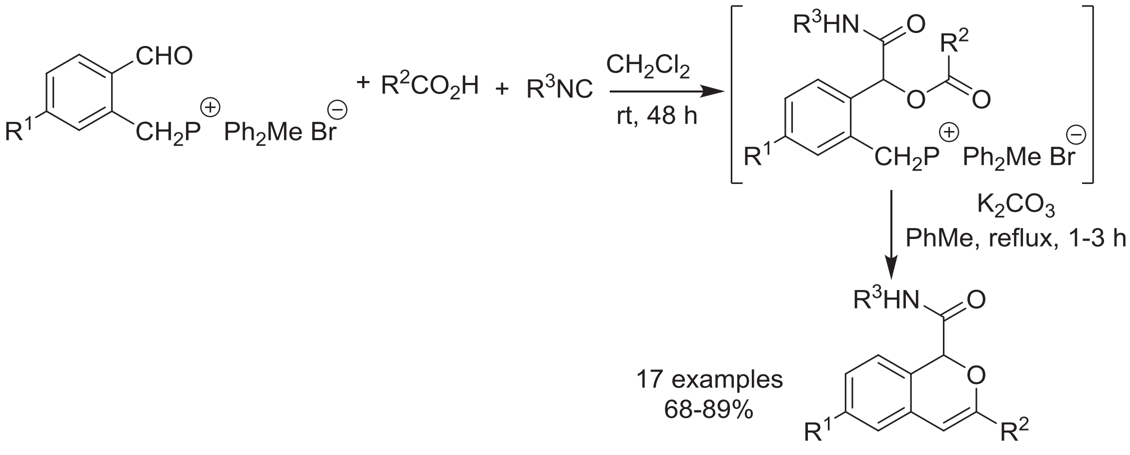


Scheme 34

spirooxindole tetrahydrofuran[3,2-*c*]chromans have been synthesized via three-component cascade reactions of 3-diazooxindoles with methyl 3-(2-formylphenoxy)propenoates and various nitroolefins promoted by $Rh_2(OAc)_4$ (16EJO2671).

6.4.2.3 [2]Benzopyrans and Dihydro[2]benzopyrans (Isochromenes and Isochromans)

A squaramide-containing tertiary amine base bifunctional organocatalyst confers high enantioselectivity on the intramolecular oxa-Michael reaction of *o*-homoformyl chalcones to chiral 1*H*-isochromenes (16JOC4654). Tandem

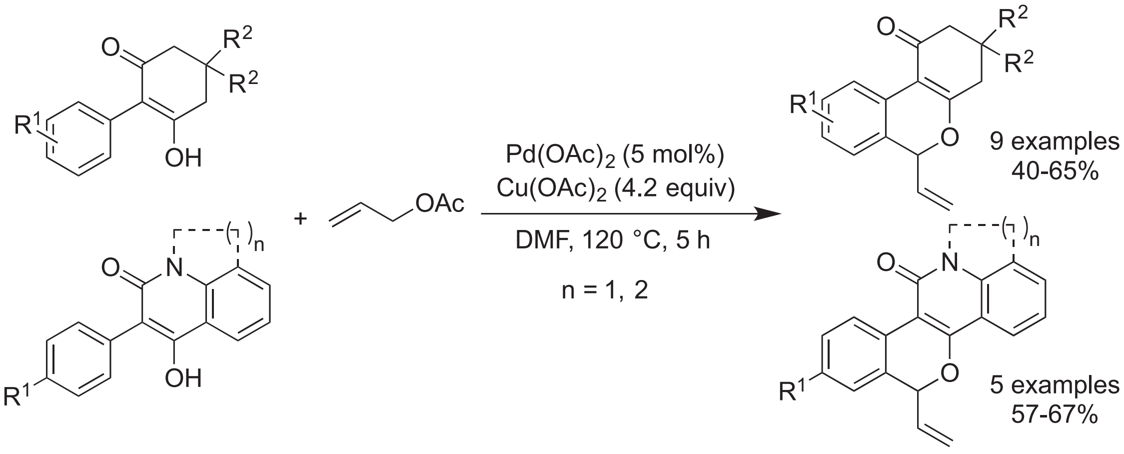


Scheme 35

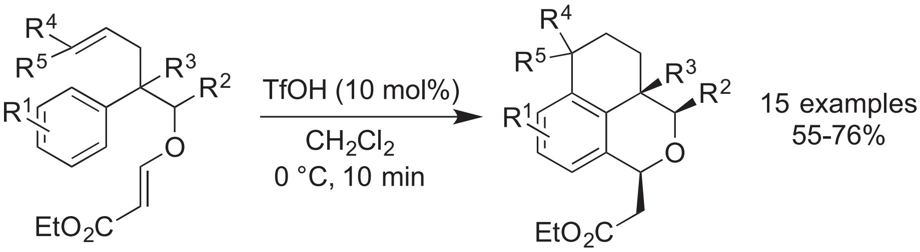
copper(I) bromide-mediated *N*-arylation and regioselective 6-*endo*-*dig*-cyclization reactions of 2-alkynylbenzamides with diaryliodonium salts carried out in the presence of 2,6-di-*t*-butylpyridine give access to 1-arylimino-1*H*-isochromenes (16EJO541). One-pot multicomponent reactions of benzaldehyde-derived phosphonium salts with aliphatic/aromatic acids and isocyanides in dichloromethane at room temperature followed by treatment with base provided 3-alkyl/aryl-1-amido-1*H*-isochromenes (Scheme 35) (16OBC2413). Highly functionalized 3-methyl-1-(nitromethyl)-1*H*-isochromenes arise from a three-step synthesis involving Henry reaction of 2-allylbenzaldehydes with NH_4OAc and nitromethane, aerobic palladium(II)-catalyzed Wacker-type oxidation, and finally intramolecular Michael cyclization mediated by potassium carbonate (16T547).

Palladium(II)-promoted oxidative coupling reactions of 2-aryl-1,3-dicarbonyl compounds with allyl acetate in DMF at 120°C leads to tri- and tetracyclic-fused 1-vinyl-1*H*-isochromenes (Scheme 36) (16TL2488). The synthesis of polysubstituted isochromans occurs through oxa-Pictet–Spengler reactions of ethyl (*E*)-2-(2-arylethoxy)acrylate mediated by TMSOTf in dry dichloromethane (16S1727) and 2-arylethan-1-ols with aliphatic/aromatic aldehydes in the presence of a nitrated confined imidophosphoric acid catalyst (16JA9429). A palladium(II)/chiral sulfoxide system was used in the enantioselective allylic C—H oxidation of 2-(2-allylaryl) ethan-1-ols to prepare a range of 1-vinylisochromans in good yields (16AGE9571).

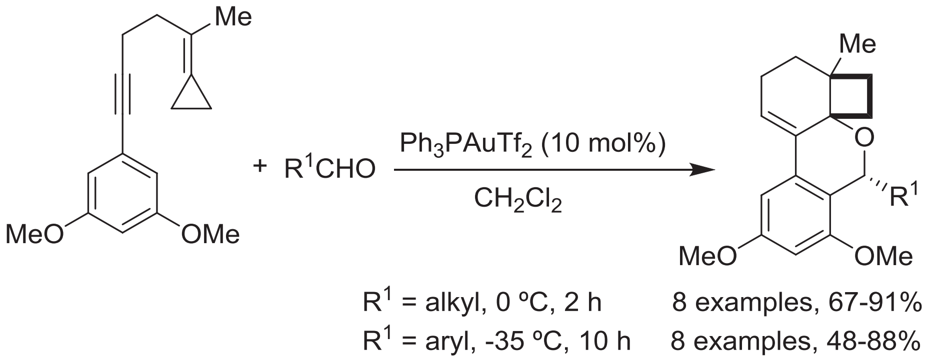
The asymmetric synthesis of pryan[*de*]octahydroisochromans can be achieved in a one-pot procedure by reacting 3-alkoxyallyl alcohols, nitroalkenes, and acrolein under a combination of lanthanide catalysis with a chiral secondary amine organocatalyst (16AGE16153). Hexahydrobenzo[*de*]



Scheme 36



Scheme 37



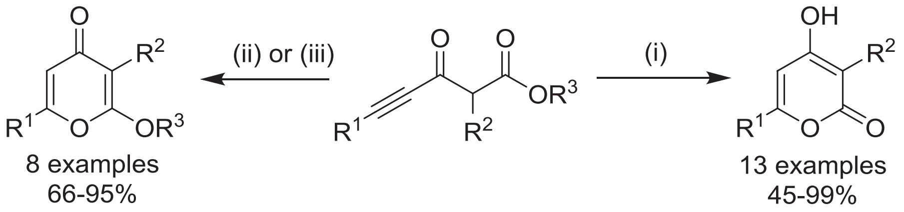
Scheme 38

isochromans are readily accessible from Friedel–Crafts and oxa-Pictet–Spengler diastereoselective reactions of ethyl (*E*)-3-(2-arylpent-4-en-1-yl) acrylates mediated by TfOH (Scheme 37) (16OBC970). Diastereoselective gold(I)-catalyzed cascade reactions of cyclopropylidene-bearing 6-aryl-1,5-enynes with aliphatic/aromatic aldehydes provide tetracyclic-fused isochromans (Scheme 38) (16OBC11261). A different gold(I) catalyst promotes homodimerization of *o*-alkynylbenzaldehyes through a sequence of 6-*endo*-*dig*-cyclization, amine addition, and [4 + 2]-cycloaddition to afford tetracyclic dihydronaphthaleno[2,3-*c*]isochromans (16CC14133).

6.4.2.4 Pyranones

Highly functionalized 2*H*-pyran-2-ones arise from ruthenium(III)-promoted oxidative cyclization of *N*-tosylacrylamides with diazoacetoacetate-type compounds in methanol at 60°C. In the presence of silver oxidants, a series of furan derivatives are obtained (16CC1661). A palladium(0) catalyst-mediated three-component reaction of methyl ketones with aryl/alkenyl bromides and 2-bromoacrylates provides 3,5,6-trisubstituted 2*H*-pyran-2-ones. It involves α-arylation, subsequent α-alkenylation, alkene isomerization, and dienolate lactonization reactions (16OL5724). According to the ester terminus, gold or silver catalysts can be used in the 6-*endo-dig*-cyclization of 3-oxo-4-alkynoic acid esters to afford either 4-hydroxy-2*H*-pyran-2-ones or 2-alkoxy-4*H*-pyran-4-ones (Scheme 39) (16CEJ237).

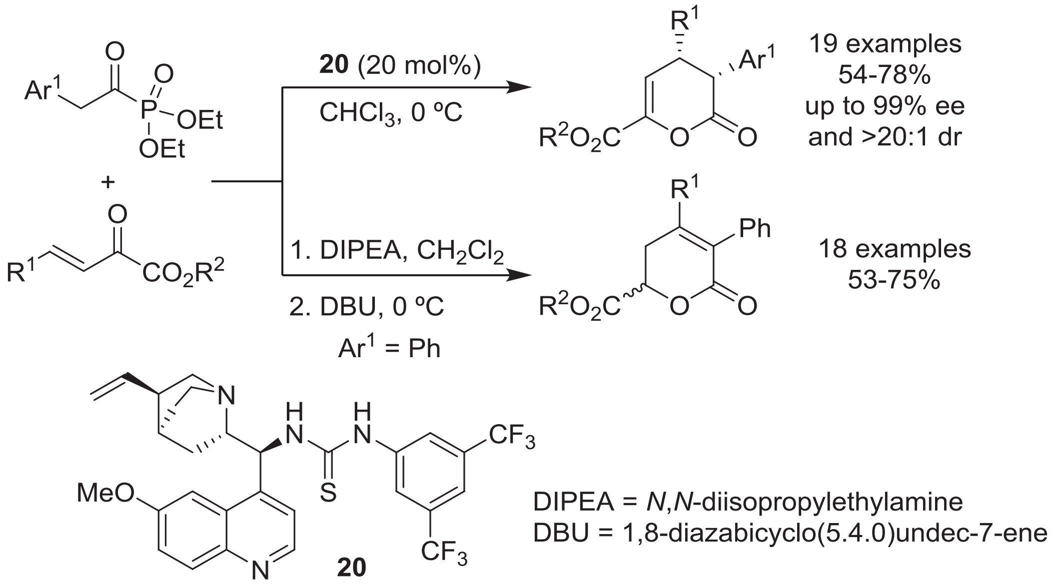
High yields of 3,4-dihydro-2*H*-pyran-2-ones can be achieved through enantioselective green aerobic annulation reactions of α,β-unsaturated aldehydes, bearing aliphatic/aromatic groups, with 1,3-dicarbonyl compounds carried out in the presence of *N*-heterocyclic carbene (NHC) catalysts using only oxygen (16JOC6136) or with a coupled system of electron transfer mediators that allows the use of oxygen as the terminal oxidant (16CC11571). The reaction of arylacetyl phosphonates with β,γ-unsaturated α-ketoesters catalyzed by chiral bifunctional thiourea-tertiary amine **20** provides *syn*-3,4-dihydro-2*H*-pyran-2-ones, while using stoichiometric amounts of DIPEA and DBU affords 5,6-dihydro-2*H*-pyran-2-ones (Scheme 40) (16OL5110). β,γ-Unsaturated amides react with *o*-quinones by a direct enantioselective organocatalytic vinylogous aldol/cyclization cascade to afford spirocyclic 5,6-dihydro-2*H*-pyran-2-ones (16OBC6435) and 3-alkylidene oxindoles (16CC2322). α,β-Unsaturated carboxylic acids (16OBC1485) react with isatins to give spirooxindole 5,6-dihydro-2*H*-pyran-2-ones.



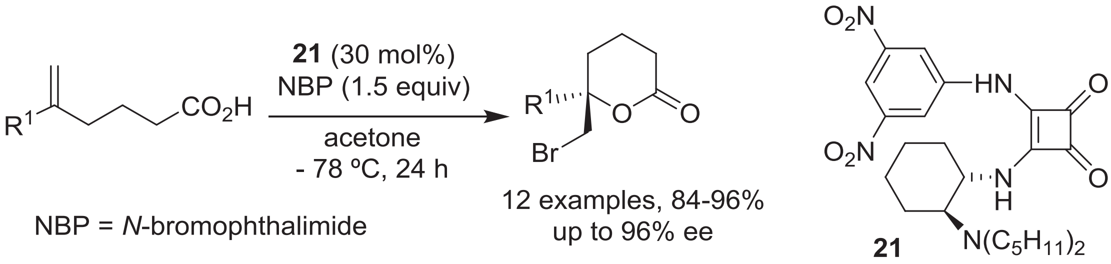
(i) $R^3 = {}^t\text{Bu}$ or $(CH_2)_2SiMe_3$	$(SPhos)AuNTf_2$ (5 mol%), AcOH	13 examples, 45-99%
(ii) $R^3 = CH_2R^4$	$(SPhos)AuNTf_2$ (5 mol%), AcOH	5 examples, 66-95%
(iii) $R^3 = {}^t\text{Bu}$	$AgNTf_2$ (5 mol%), AgOTs, DMEDA, $CHCl_3$	3 examples, 85-88%

DMEDA = *N*,*N*-dimethylethylenediamine

Scheme 39



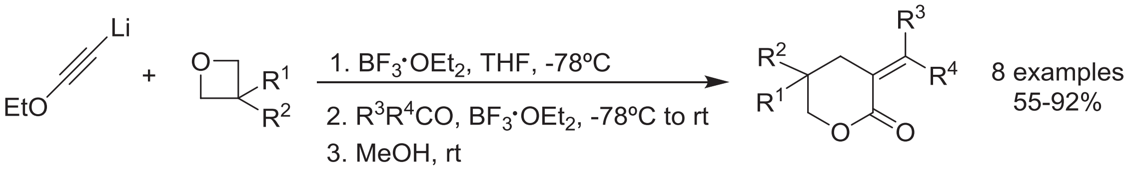
Scheme 40



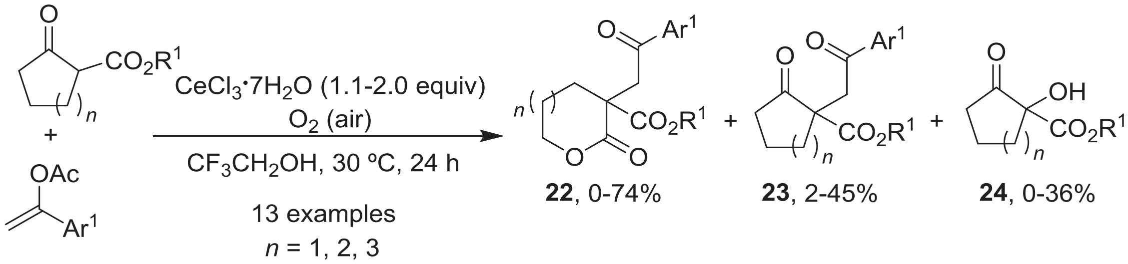
Scheme 41

Asymmetric iodolactonization of 5-arylhex-5-allenoic acids occurs in the presence of iodine and NIS using a squaramide catalyst formed some 6-aryl-6-(1-iodovinyl)tetrahydro-2*H*-pyran-2-ones in moderate yields (16TL5232). A similar catalyst promotes bromolactonization reactions of 5-alkyl/arylhex-5-enoic acids in the presence of *N*-bromophthalimide (NBP) giving rise to 6-alkyl/aryl-6-(bromomethyl)tetrahydro-2*H*-pyran-2-ones (Scheme 41) (16JOC8287). 6-Substituted 5-iodotetrahydro-2*H*-pyran-2-ones are accessible from formal intermolecular iodolactonization reaction of allylic alcohols with 2-iodoacetic/2-iodopropionic acid in the presence of lauroyl peroxide in refluxing 1,2-DCE (16EJO1739).

Reacting (ethoxyethynyl)lithium with oxetanes in the presence of $BF_3{\cdot}OEt_2$ at low temperature, addition of aliphatic/aromatic carbonyl compounds, warming the reaction mixture to room temperature, and addition of an excess of methanol led to the synthesis of 3-alkylidene and 3-benzylidene tetrahydro-2*H*-pyran-2-ones (Scheme 42) (16TL415). Other substituted tetrahydro-2*H*-pyran-2-ones result from cross-coupling reactions of allyl/propargyl ethers with δ-ketoesters mediated by a samarium(II)



Scheme 42



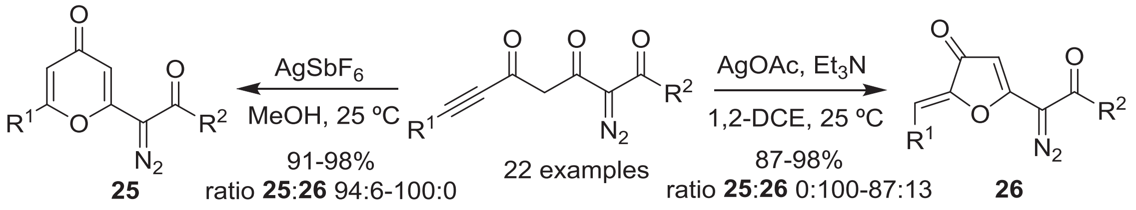
Scheme 43

cyclopentadienyl reagent (16CC13503) and palladium nanoparticles-promoted telomerization of carbon dioxide with 1,3-butadiene (16TL3163). Cerium(III) promoted the reaction of cyclopentenone-2-carboxylates with α-aryl vinyl acetates, using atmospheric oxygen as oxidant, to deliver mainly 3,3-disubstituted tetrahydro-2*H*-pyran-2-ones **22** and cyclopentenone-2-carboxylate derivatives **23** and **24** as by-products (Scheme 43) (16JOC7790).

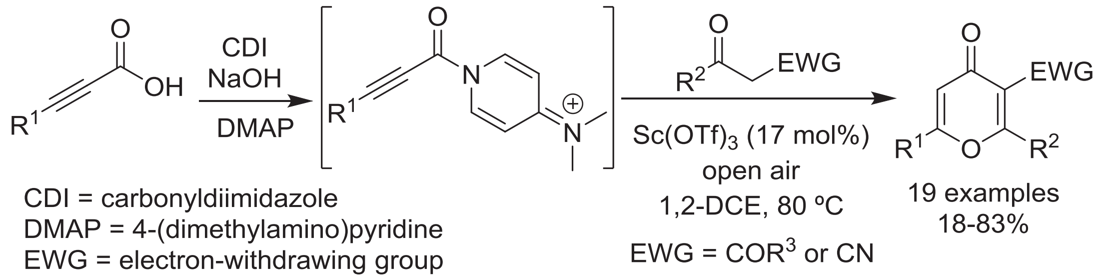
Dearomatizing cyclization of 2-(benzofuran-2-yl)propionic acids with arylboronic acids is accomplished using palladium(II) acetate and 2,2,6,6-tetramethylpiperidin-1-oxyl (TEMPO) radical at room temperature to afford 2,3-dihydrobenzofuran[2,3-*e*]tetrahydro-2*H*-pyran-2-ones. In the same reaction conditions, use of 2-(*N*-Boc-indol-2-yl)propionic acids leads to indolino[2,3-*e*]tetrahydro-2*H*-pyran-2-ones (16EJO2268).

Various 2-diazo-3,5-dioxo-6-ynoates underwent regioselective 6-*endo-dig*-cyclization promoted by $AgSbF_6$ in methanol to give mainly 4*H*-pyran-4-ones **25**, whereas 5-*exo-dig*-cyclization mediated by AgOAc in 1,2-DCE affords 3(2*H*)-furan-3-ones **26** as major products (Scheme 44) (16OL6248). Other functionalized 4*H*-pyran-4-ones have been synthesized through formal [3 + 3]-annulation reactions of alkynyl acylammoniums, generated in situ under dual activation of alkynyl acids in the presence of carbonyldiimidazole, DMAP, and $Sc(OTf)_3$, with 1,3-dicarbonyl compounds or enolizable ketones bearing an electron-withdrawing group at C-2 in 1,2-DCE at 80°C (Scheme 45) (16OL3882).

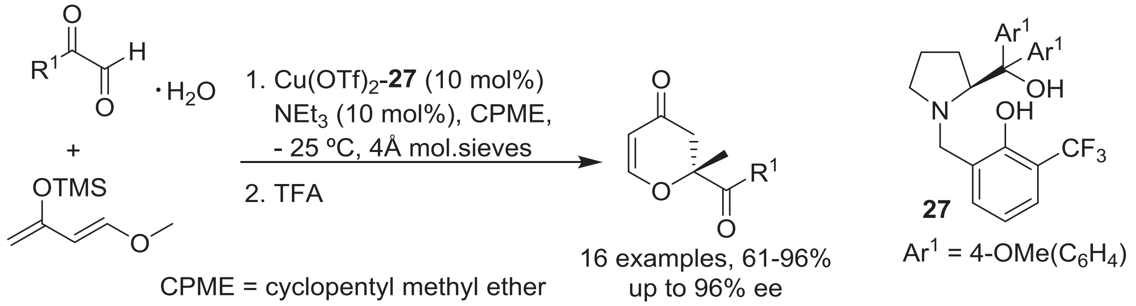
Stereoselective synthesis of 2,3-dihydro-4*H*-pyran-4-ones occurs via an asymmetric double aldol/cyclization cascade reaction of 4-*O*-alkyl/phenyl-but-3-en-2-one with aldehydes mediated by a chiral phosphine oxide catalyst in the presence of silicon tetrachloride and DIPEA (16CPB189).



Scheme 44



Scheme 45



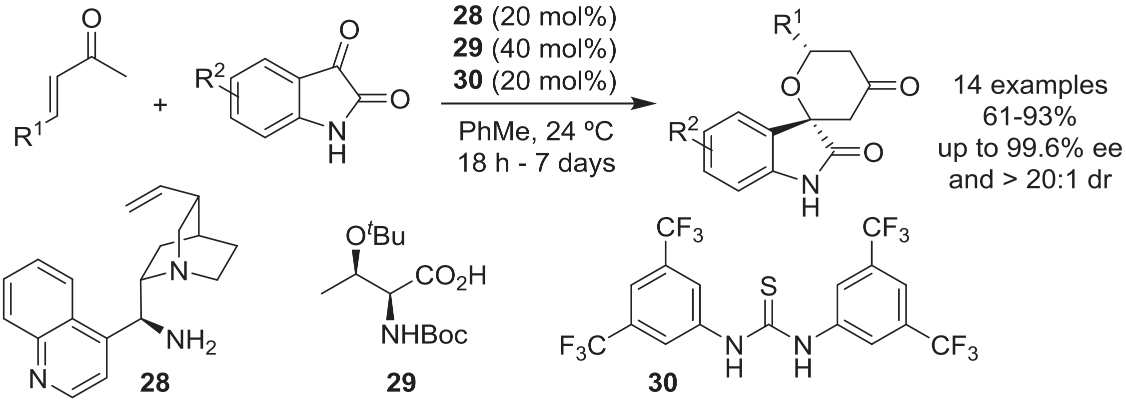
Scheme 46

Copper(II) triflate promotes enantioselective hDA reaction of Danishefsky's diene with alkyl/arylglyoxals, which leads to 2-oxo-alkyl/aryl-2,3-dihydro-4*H*-pyran-4-ones in high yields and enantioselectivity (Scheme 46) (16JOC2993). A few examples of 2,5-disubstituted 2,3-dihydro-4*H*-pyran-4-ones result from Maitland–Japp reaction of δ-hydroxy-β-ketoesters with DMF dimethyl acetal in dichloromethane at room temperature followed by treatment with $BF_3 \cdot OEt_2$ (16OBC6840).

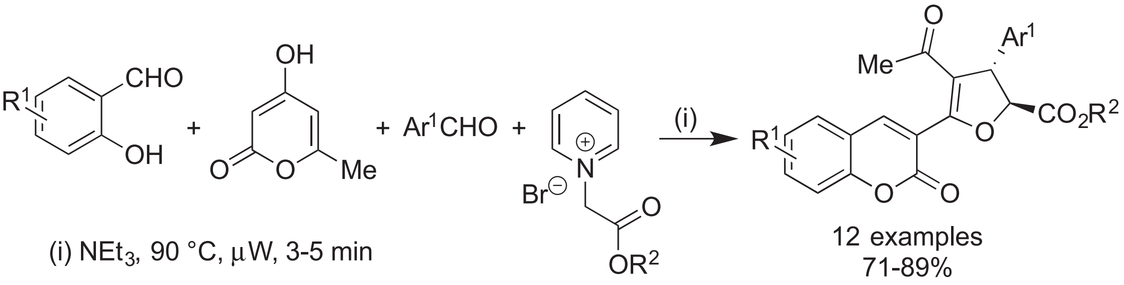
Asymmetric hDA reaction of various enones with isatins catalyzed by the combination of amine **28**, acid **29**, and thiourea **30** system provide a number of substituted spirooxindole tetrahydro-4*H*-pyran-4-ones (Scheme 47) (16OBC1777).

6.4.2.5 Coumarins

Knoevenagel/intramolecular cascade reactions of salicylaldehydes with Meldrum's acid catalyzed by aqueous KH_2PO_4 lead to coumarin-3-carboxylic



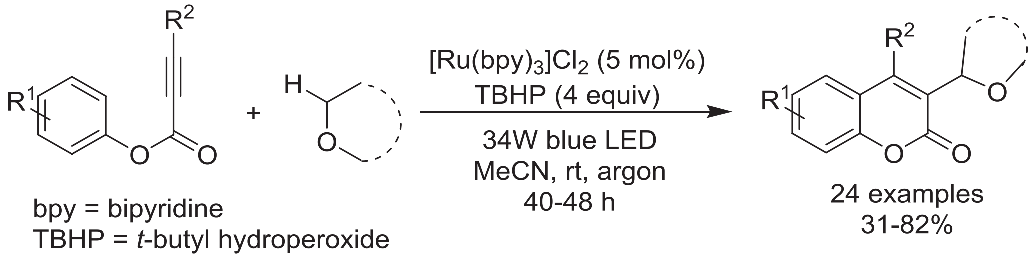
Scheme 47



Scheme 48

acids (16H(92)1698). Using diethyl malonate or ethyl acetoacetate as 1,3-carbonyl compounds and carrying out the reaction in the presence of piperidine in refluxing ethanol gives 3-acetyl-2*H*-coumarins or ethyl 2*H*-coumarin-3-carboxylates in good yields (16TL2496). Reaction with ethyl cyanoacetate in ethanol at room temperature promoted by ZIF-8 nanoparticles leads to α,β-unsaturated cyano esters, while using DMF as solvent at 80°C leads to 3-cyanocoumarins (16TL5885). Microwave-assisted, one-pot four-component reactions of salicylaldehydes with 4-hydroxy-6-methylpyran-4-one, benzaldehydes, and a pyridinium ylide carried out in the presence of triethylamine produces 3-(dihydrofuran-2-yl)-2*H*-coumarins (Scheme 48) (16SC613).

A large variety of 4-alkyl/aryl-3-substituted 2*H*-coumarins arise from transition-metal-free approaches starting from aryl alkynoates: with acetonitrile or acetone in the presence of *t*-butyl peroxybenzoate leading to 3-cyanomethyl- or 3-(2-oxopropyl)-2*H*-coumarins (16JOC11489); with xanthates and dilauroyl peroxide in 1,2-DCE leading to 3-(2-oxosubstituted)-2*H*-coumarins (16OBC9033); with *N*-sulfanylsuccinimides in the presence of $BF_3{\cdot}OEt_2$ to give 3-sulfenyl-2*H*-coumarins (16JOC11297); with aryldiazonium tetrafluoroborates and DABCO·$(SO_2)_2$ in 1,2-DCE to form 3-sulfonyl-2*H*-coumarins (16AGE11925); and with 2,4-diones in the presence of $K_2S_2S_8$ and TBAB in a 1:1 mixture of 1,2-DCE/water producing



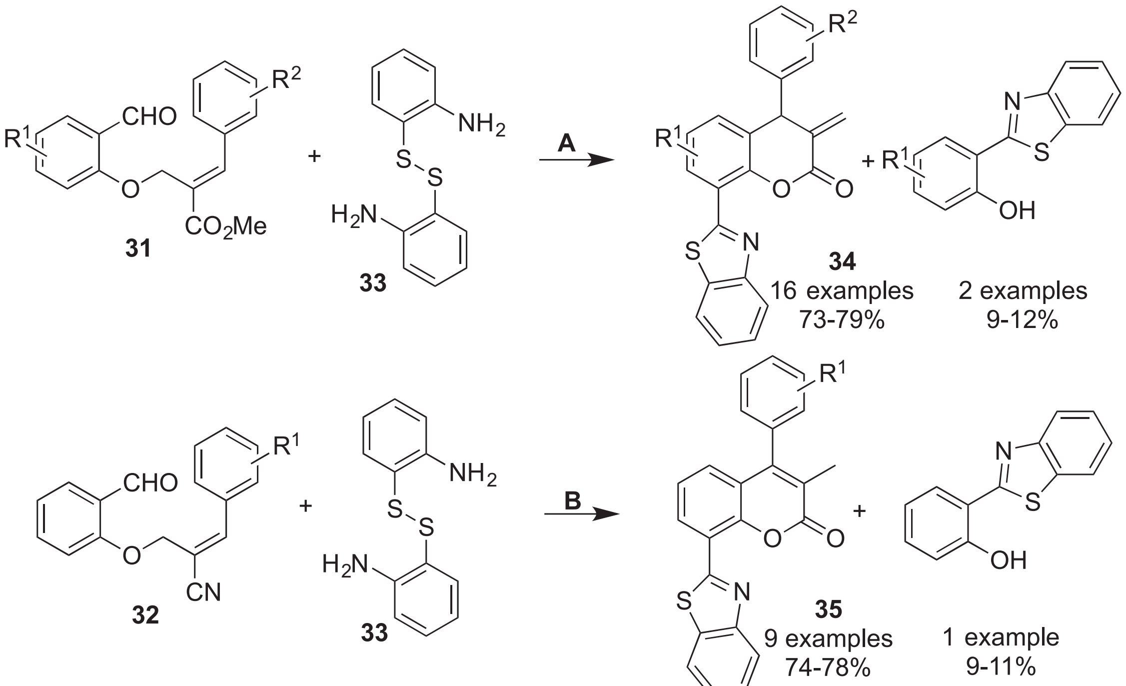
Scheme 49

other 3-functionalized 2*H*-coumarins (16T279). A transition-metal approach involves ruthenium(II)-catalyzed reaction of aryl alkynoates with simple cyclic ethers mediated by visible light to achieve 4-aryl-3-substituted-2*H*-coumarins (Scheme 49) (16OL3846).

The formation of 3-substituted-4-vinyl-2*H*-coumarins by the carbocation-initiated [4 + 2]-cycloaddition reaction of *o*-anisole-substituted propargyl silyl ethers with ynamides is efficiently promoted by zinc bromide (16CC11131). 2-Iodoaryl-2-arylacetates and arylacetylenes undergo a palladium(II)-catalyzed carbonylative Sonogashira coupling–intramolecular aldol cascade reaction, which affords 3-aryl-4-(arylethynyl)-2*H*-coumarins (16EJO4041). Further 3,4-disubstituted-2*H*-coumarins are obtained through iridium(III)-catalyzed and copper(II)-promoted carbonylative annulation reactions of simple phenols with internal alkynes at atmospheric pressure (16AGE14151).

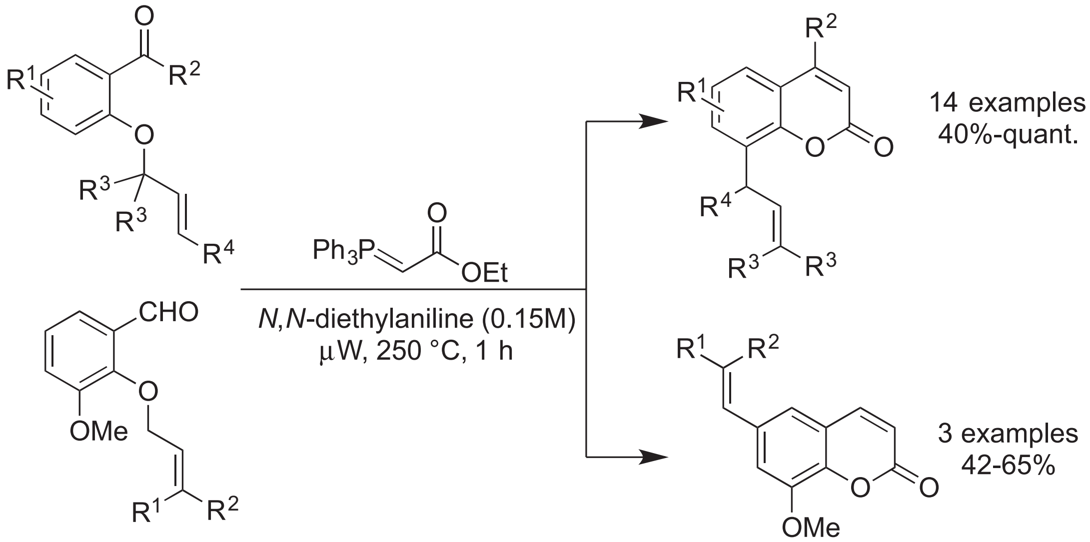
Using a solid-state melt reaction a variety of substituted 8-benzothiazole-2*H*-coumarins **34** and **35** are accessible from the reaction of *O*-substituted salicylaldehydes **31** and **32** with disulfide **33** in a one-pot transformation (Scheme 50) (16JOC3391).

(−)-Riboflavin was the photocatalyst used in the isomerization and cyclization reactions of (*E*)-cinnamic acids in a 1:1 mixture of acetonitrile/methanol at room temperature to afford 4-substituted-2*H*-coumarins (16JA1040). More examples are readily accessible from the reactions of phenols with propiolic acids promoted by TfOH in chlorobenzene at 100°C (16TL3600) or $Yb(OTf)_3$ under microwave irradiation and solvent-free conditions (16TL2939). Further derivatives arise from Pechmann condensation reaction of phenols with β-ketoesters mediated by meglumine sulfate in neat conditions under microwave and thermal heating conditions (16SC1283). *O*-Allylated/prenylated salicylaldehyde derivatives and [(ethoxycarbonyl)methylene]triphenylphosphorane in the presence of *N*,*N*-diethylaniline are converted into 6- or 8-allylated/prenylated 2*H*-coumarins (Scheme 51). This microwave-assisted tandem reaction



A: (i) melt, 180 °C, 1 h; (ii) conc. H_2SO_4, CH_2Cl_2, rt, 1 h
B: (i) melt, 180 °C, 1 h; (ii) *t*-BuOK, THF, rt, 2 h

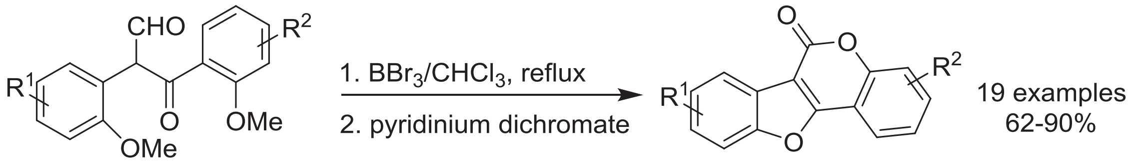
Scheme 50



Scheme 51

involves Claisen rearrangement, carbonyl olefination, and cyclization reactions (16S141).

Ruthenium(II)-promoted C—H arylation of (hetero)aryl carboxylic acids with 2-halophenols provides a range of benzo[*c*]coumarins in moderate-to-excellent yields (16OL5432). Palladium(0)-catalyzed carbonylative cascade reactions of 2-(2-bromophenyl)imidazo[1,2-*a*]pyridines in the presence of $Cu(OAc)_2$ afford imidazopyridine-fused coumarins, while in



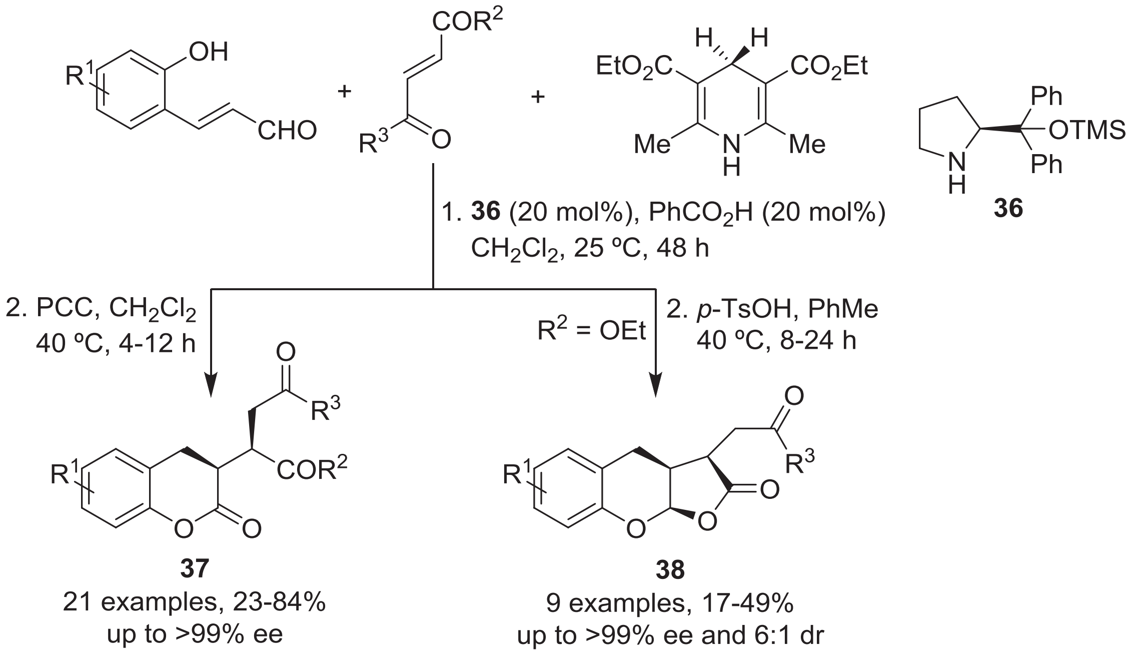
Scheme 52

the absence of copper(II) acetate, imidazopyridine-fused indenones are produced (16JOC3206).

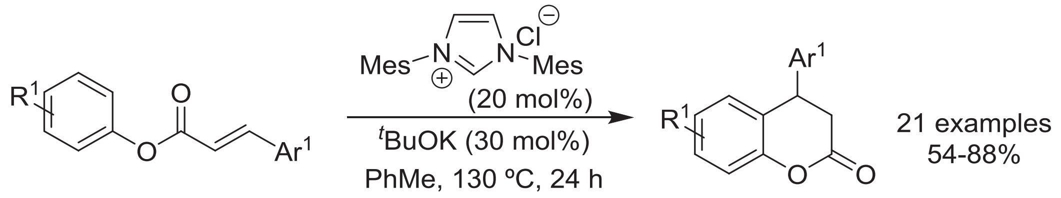
2,3-Bis(2-methoxyaryl)-3-oxopropanals undergo tandem demethylation, annulation, and oxidation reactions in the presence of BBr_3 followed by treatment with pyridinium dichromate to achieve benzofuro[3,2-*c*]coumarins in good yields (Scheme 52) (16EJO3380). Other analogs arise when the aldehyde function is replaced by ethyl esters and with no need for oxidative conditions (16T3324). Chromen-4-one-fused[3,2-*c*]coumarins can be prepared in a three-step sequence starting from 2-hydroxyacetophenones, involving esterification with 2-chloroaroyl chlorides, Baker–Venkataraman rearrangement and cyclization to the coumarin structure by reaction with methyl chloroformate, and finally chromone cyclization with K_3PO_4 (16AJC98). Benzo[*de*]coumarins result from copper(I)-Y zeolite-catalyzed reaction of *N*-sulfonylketenimines, generated in situ from *p*-toluenesulfonyl azide and alkynes, with 5-hydroxy-1,4-naphthoquinone, carried out in the presence of triethylamine in 1,4-dioxane (16CC8436).

Organocatalyzed [4+2]-annulation of aldehydes with *o*-quinone methides followed by oxidation provided 3-substituted 3,4-dihydrocoumarins in moderate-to-good yields (16TL5649). Chiral 2-hydroxychromans arise from an organocatalytic domino reaction of 2-hydroxycinnamaldehydes with *trans*-2-ene-1,4-dicarbonyl compounds and a Hantzsch ester in the presence of benzoic acid in dichloromethane at room temperature. Subsequent oxidation with pyridinium chlorochromate (PCC) leads to chiral 3,4-dihydrocoumarins **37**, while in the presence of *p*-TsOH a cyclization reaction occurs to produce compounds **38** (Scheme 53) (16OL864).

A series of 4-aryl-3,4-dihydrocoumarins has been synthesized through the Friedel–Crafts alkylation and cycloaddition reactions of *t*-butyl 3-hydroxy-3-(2-hydroxyaryl)propanoates with aromatic compounds in the presence of $HClO_4$ in acetonitrile at 0°C (16T3917). It is through cooperative catalysis of an NHC and potassium ion that α,β-unsaturated phenolic esters undergo in situ conversion into α,β-unsaturated acylazoliums and phenolates to afford 4-aryl-3,4-dihydrocoumarins (Scheme 54) (16EJO4569).



Scheme 53



Scheme 54

$[Ir(dtbbpy)(ppy)_2]PF_6$ = [4,4'-bis(1,1-dimethylethyl)-2,2'-bipyridine-*N*1,*N*1']
bis[2-(2-pyridinyl-*N*)phenyl-C]iridium(III) hexafluorophosphate

Scheme 55

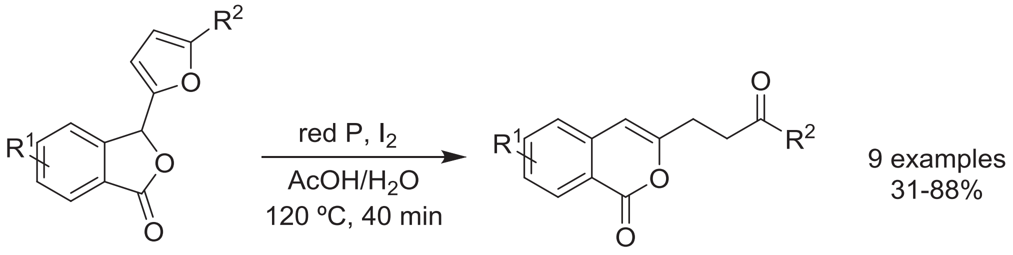
Under solvent-free conditions, tandem reaction of phenols/naphthols with olefinic thioazlactones promoted by *p*-TsOH produces *cis*-3,4-disubstituted 3,4-dihydrocoumarins (16JOC5699). High yields and diastereoselectivity of chiral 3,4-disubstituted 3,4-dihydrocoumarins result from the photocatalytic radical cascade of acryloyl ester-tethered aroylhydrazones with α-silylamines promoted by an iridium(III) catalyst (Scheme 55) (16OL6304). Further derivatives arise from the enantioselective [4 + 2]-cycloaddition reactions of azlactones with *o*-hydroxy benzhydryl alcohols promoted by a chiral phosphoric acid catalyst (16CEJ6774) and with *o*-hydroxystyrenes mediated by another chiral phosphoric acid catalyst in the presence of a chiral guanidine base, a merging of Brønsted acid and base catalysis (16JOC1681).

A wide range of densely functionalized pyrrolidino[3,2-*c*]-3,4-dihydrocoumarins arise from cascade reactions of β,γ-unsaturated-α-ketophosphonates with imines, derived from salicylaldehydes and diethyl aminomalonate, carried out in the presence of a dihydroquinine cinchona catalyst (16JOC6800). Asymmetric [3+3]-cyclization reactions of 4-arylidene-2-aryloxazol-5(4*H*)-ones with cyclohexane-1,3-diones promoted by a chiral thiourea-tertiary amine catalyst give 3-aminohexahydrocoumarin derivatives in moderate-to-good yields and with high diastereo- and enantioselectivities (16JOC5056). Further *cis*-3,4-disubstituted hexahydrocoumarin analogs arise from aldol condensation/NHC-catalyzed [2+4]-annulation reaction of cyclohexane-1,3-diones with α,β-unsaturated aldehydes (16CEJ5123).

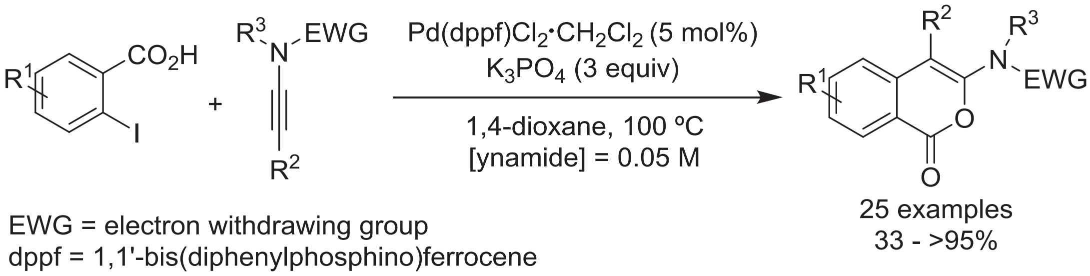
(*E*)-1,4-Diarylenynes undergo microwave-assisted annulation reactions in the presence of a catalytic amount of *p*-TsOH giving access to (*E*)-3-styrylisocoumarins (16S3382). Various 3-(3-oxoalkyl)isocoumarins can be achieved through tandem reductive rearrangement of 3-(fur-2-yl)phthalides using a red phosphorus/iodine reagent system (Scheme 56) (16TL1483). Iodine is also involved in the electrophilic 6-*endo*-*dig*-cyclization of methyl 2-(buta-1,3-diynyl)benzoates to provide 3-ethynyl-4-iodoisocoumarins (16EJO739).

A range of 4-borylated-3-substituted isocoumarins were produced by a catalyst-free electrophilic oxyboration reaction of *o*-ethynylated methyl arylates. Using alk-2-en-4-ynyl esters, 2*H*-pyran-2-ones were obtained in lower yields (16JA2126).

Regioselective synthesis of 3,4-disubstituted isocoumarins result from palladium(II)-catalyzed reactions of 2-iodobenzoic acid derivatives with ynamides (Scheme 57) (16CC6801) and *o*-(2-alkyl/aryl ethynyl)benzoic acid derivatives with alkenyl alcohols using oxygen as sole oxidant

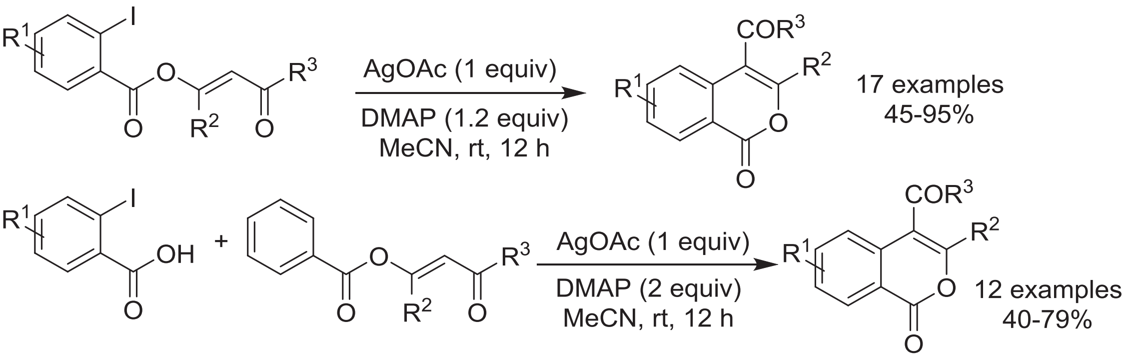


Scheme 56



EWG = electron withdrawing group
dppf = 1,1'-bis(diphenylphosphino)ferrocene

Scheme 57

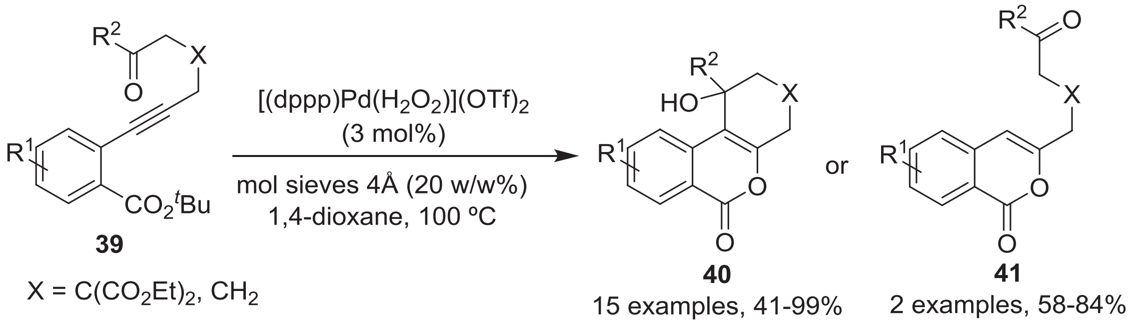


Scheme 58

(16EJO663). Further examples include silver(I)-promoted annulation reactions of 2-iodo enol esters or coupling of 2-iodobenzoic acids with enolates (Scheme 58) (16JOC1047) or ruthenium(II)-catalyzed C—H hydroarylation of propargylic alcohols with benzoic acid derivatives, followed by intramolecular lactonization (16AGE6933). High yields of 8-amido-3,4-disubstituted isocoumarins are produced from ruthenium(II)-mediated C—H activation and annulation reactions of isatins with alkynes (16CC9809).

A two-step strategy involving Suzuki coupling of 2-bromoaryl-substituted esters with 2-[(*E*)-2-ethoxyvinyl]-4,4,5,5-tetramethyl-1,3,2-dioxaborolane and subsequent microwave-assisted cyclization in the presence of trifluoroacetic acid (TFA) was used to prepare 3,4-unsubstituted isocoumarins (16EJO4171).

A range of cyclohexa[*c*]isocoumarins **40** were synthesized via a palladium(II)-mediated cascade cyclization of alkyne-tethered carbonyl compounds **39** bearing an aryl ester (Scheme 59). It involves intramolecular oxypalladation of the alkyne with the ester group followed by 1,2-addition of the formed C—P(II) bond to the carbonyl group. A couple of 3-substituted isocoumarins **41** were also isolated (Scheme 59) (16JOC3423). A few examples of pyrido[3,2-*c*]isocoumarins resulted from titanium(III)-promoted radical arylation and lactonization of 3-hydroxypyridines with *o*-methyl ester-substituted phenyldiazonium salts in a mixture of water/HCl



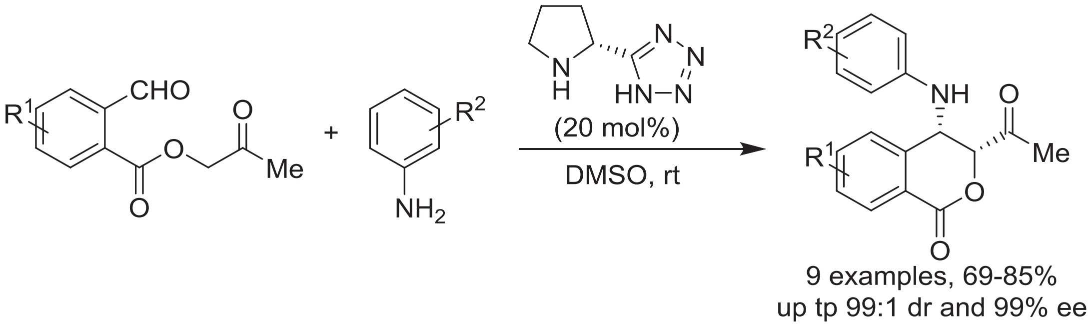
Scheme 59

(16JOC5752). Under solvent-free conditions, a condensation reaction of ninhydrin with arylhydrazones carried out on the solid surface of magnetically separable $Fe_3O_4@SiO_2–SO_3H$ nanoparticles provided pyrazole-fused isocoumarins in good yields (16TL2103). 2-Arylindenones undergo domino head-to-head DA dimerization/Baeyer–Villiger oxidation with incorporation of molecular oxygen into a reaction intermediate to afford hexacyclic indeno-fused isocoumarins (16T5903).

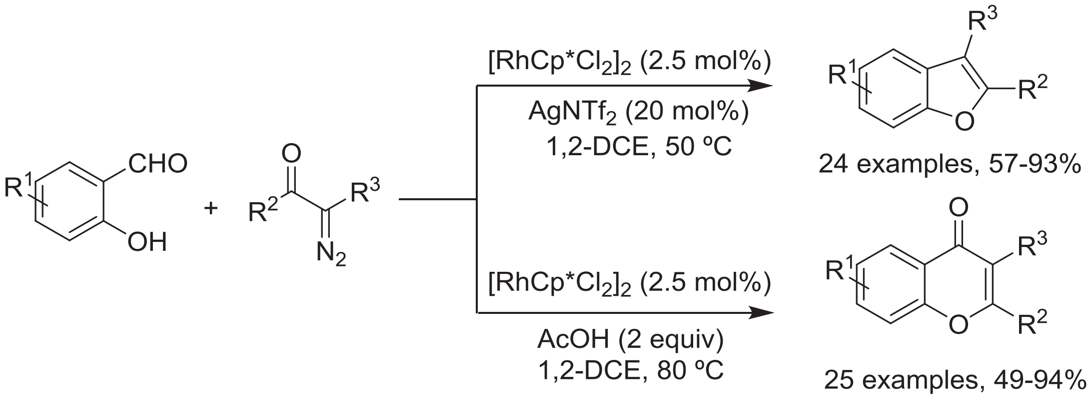
Treatment of (2-allylaryl)(pyrrolidin-1-yl)methanones with a mixture of HCl/1,4-dioxane at 110°C for 12 h produces 3-methyl-3,4-dihydroisocoumarins (16OBC7691). *o*-Quinodimethanes, generated in situ from 2-(bromomethyl)benzaldehydes and an NHC catalyst, undergo [4 + 2]-annulation reactions with ketones to achieve 3,3-disubstituted 3,4-dihydroisocoumarins (16OL4444). Regioselective 6-*endo*-bromocyclization reactions of 2-(2-arylvinyl)benzoic acids occurs in the presence of TFA and *N*-bromosuccinimide in acetonitrile at room temperature giving rise to *trans*-3-aryl-4-bromo-3,4-dihydroisocoumarins (16OBC4571). High diastereo- and enantioselectivities in the synthesis of 4-fluoro-3-substituted 3,4-dihydroisocoumarins was achieved through a fluorolactonization reaction of methyl 2-(alk-2-en-1-yl)benzoates using HF-pyridine as nucleophilic fluoride source and *meta*-chloroperoxybenzoic acid as oxidant (16JA13858). Asymmetric organocatalytic one-pot intramolecular Mannich reaction of 2-oxopropyl 2-formylbenzoates with anilines in dimethyl sulfoxide (DMSO) at room temperature provided 3-acetyl-4-aminoaryl-3,4-dihydroisocoumarins in good yields and with excellent diastereo- and enantioselectivities (Scheme 60) (16S4451).

6.4.2.6 Chromones and Chromanones

A couple of 1-(2-prenyloxyaryl)-3-arylprop-2-yn-1-ones in *N*,*N*-diethylaniline at 250°C were shown to undergo a microwave-assisted deprenylation and 6-*endo*-cyclization sequence to afford 2-aryl-4*H*-chromen-4-



Scheme 60

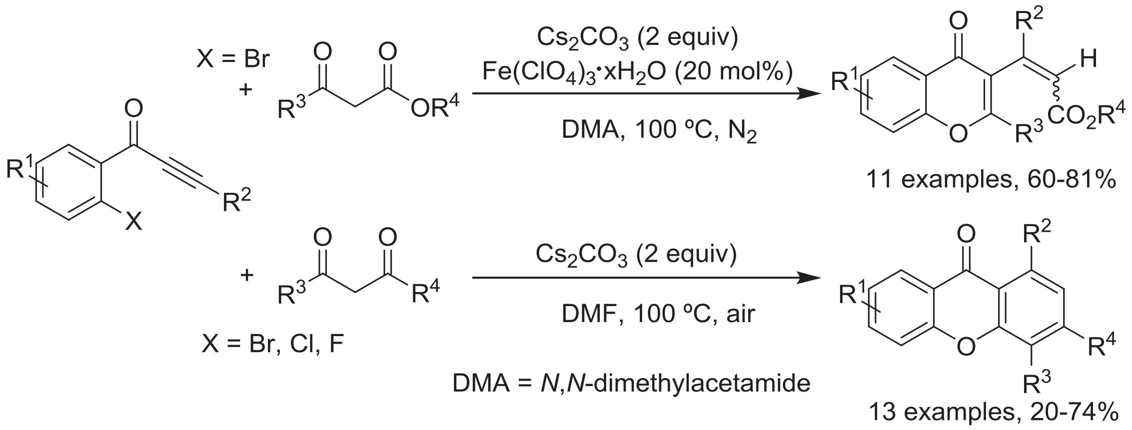


Scheme 61

ones. Using 3-methyl-1-(2-prenyloxyaryl)but-2-en-1-ones led to 2,2-dimethyl-4*H*-chroman-4-ones (16S1399).

Two-component reaction of 2′-hydroxyacetophenones with diethyl oxalate and sodium ethoxide in ethanol gives ethyl 4*H*-chromen-4-one-2-carboxylates in moderate-to-good yields (16TL3006). A wide range of 2-substituted and 2,3-disubstituted 4*H*-chromen-4-ones were attained from annulation reactions of salicylaldehydes with alkynes catalyzed by cobalt(I) (16AGE2870) or ruthenium(II) (16CC13004) complexes.

Under rhodium(III) catalysis, the annulation reaction of salicylaldehydes with 2-diazo-1,3-dicarbonyl compounds in the presence of $AgNTf_2$ led to benzofurans, while in the presence of acetic acid access to 2,3-disubstituted 4*H*-chromen-4-ones was demonstrated (Scheme 61) (16OL6464). The synthesis of 2-aryl-3-sulfenyl-4*H*-chromen-4-ones occurs through $FeCl_3$-mediated regioselective cyclization of 1-aryl-3-(alkyl/aryl)prop-2-yn-1-ones with *N*-arylthiobenzamides in dichloromethane at room temperature (16T8617). Other 2-aryl-3-substituted-4*H*-chromen-4-ones arise from cyclization reactions of 1,3-diketones carried out in the presence of *N*-triflyl phosphoramide in methanol at 40°C (16TL3841) and the formal [3 + 3]-cycloaddition reaction of 2-haloaroyl chlorides with



Scheme 62

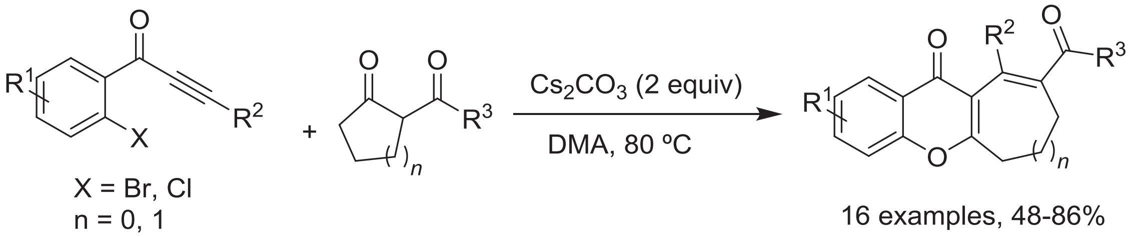
α-methyleneketones under dual catalysis of sodium *t*-butoxide and cesium carbonate in DMA (16TL2116).

Cesium carbonate also promotes the reactions of 1-(2-haloaryl)-3-(alkyl/aryl)prop-2-yn-1-ones with 3-oxobutanoates in the presence of $Fe(ClO_4)_3$ in DMA at 100°C under a nitrogen atmosphere to provide 2,3-disubstituted 4*H*-chromen-4-ones, while the reaction with pentane-2,4-diones in DMF at 100°C in air gives 9*H*-xanthen-9-ones (Scheme 62) (16CEJ12655).

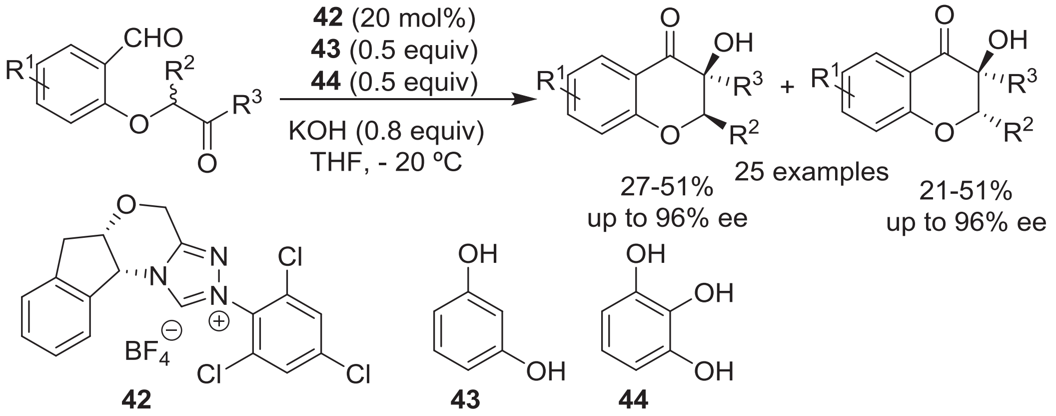
o-Hydroxyarylenaminones, available from heating in DMF-substituted 2′-hydroxyacetophenones with DMA–DMF, undergo silver triflate-catalyzed reaction with alkyl iodides in dichloromethane at room temperature to afford 3-alkyl-4*H*-chromen-4-ones (16S3364) and by gold-mediated reaction with 1-{[tris-(1-methylethyl)silyl]ethynyl]}-1,2-benziodoxol-3(1*H*)-one in 1,2-DCE to provide 3-alkynyl-4*H*-chromen-4-ones (16CC12306).

Excellent yields of indeno-fused 4*H*-chromen-4-ones result from the gold(I)-promoted tandem cyclization reaction of 3-(2-alkoxymethyl)-1-(2-hydroxyaryl)prop-2-yn-1-ones (16CC11516). Cesium carbonate mediates ring-expansion reactions of 3-alkyl/aryl-1-(2-haloaryl)prop-2-yn-1-ones with unstrained cyclic β-dicarbonyl compounds giving rise to cycloheptene- and cyclooctene-fused 4*H*-chromen-4-ones in 48%–86% yields (Scheme 63) (16CEJ17936).

A number of 2-aryl-4*H*-chroman-4-ones have been synthesized through a three-step approach involving the preparation of cinnamoyl chlorides by refluxing cinnamic acids with $SOCl_2$, their condensation with phenols promoted by $BF_3 \cdot OEt_2$ to afford the corresponding chalcones and finally base-catalyzed cyclization. The first two steps do not require solvents or a



Scheme 63

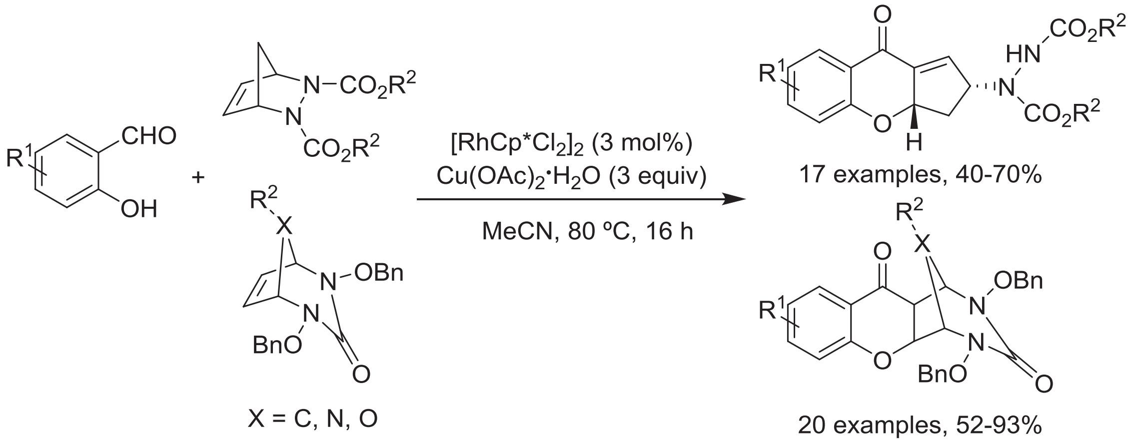


Scheme 64

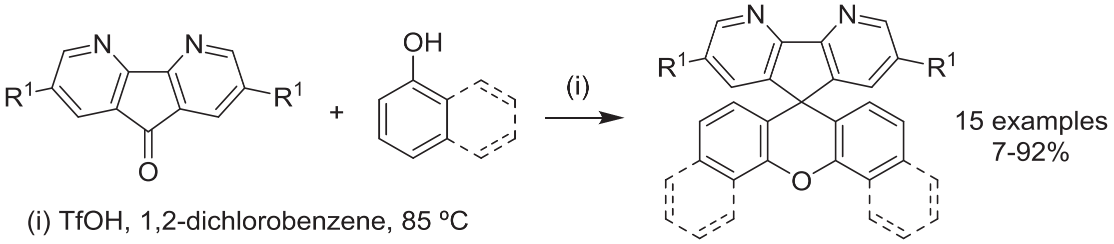
purification step (16SC1803). Bi(OTf)$_3$ promotes tandem reaction of electron-rich phenols with 3,3-dimethylacrylic acid or *trans*-crotonic acid in refluxing toluene to give a series of 2,2-dimethyl- or 2-methyl-4*H*-chroman-4-ones, respectively. It involves an esterification, Fries rearrangement, and oxa-Michael ring closure (16TL5057).

Intramolecular benzoin reactions of 2-(2-oxoalkoxy)benzaldehydes catalyzed by NHC catalysts leads to a mixture of stereoisomers of 3-substituted and 2,3-disubstituted 3-hydroxy-4*H*-chroman-4-ones in moderate-to-excellent enantioselectivity using THF as solvent (Scheme 64) (16OL3980) or in solvent-free conditions (16OL5764). In the last conditions, 2-(2-carboxymethylvinyl) benzaldehydes also afford 3-substituted 4*H*-chroman-4-ones via intramolecular asymmetric Stetter reactions (16OL5764). Other 2-allyloxybenzaldehydes undergo radical cyclization/coupling cascade reactions with dialkyl phosphites/ AgSbF$_6$/K$_2$S$_2$O$_8$, sodium azide/phenyliodine bis(trifluoroacetate) and potassium iodide/TBHP to achieve 3-(phosphonomethyl)-, 3-(azidomethyl)-, and 3-(hydroxymethyl)-4*H*-chroman-4-ones, respectively (16CC3661).

A large variety of cycloalkane-fused 4*H*-chroman-4-ones were formed through rhodium(III)-catalyzed oxidative coupling of various salicylaldehydes with bicyclic olefins using Cu(OAc)$_2$·H$_2$O as oxidant in acetonitrile at 80°C (Scheme 65) (16T4007).



Scheme 65



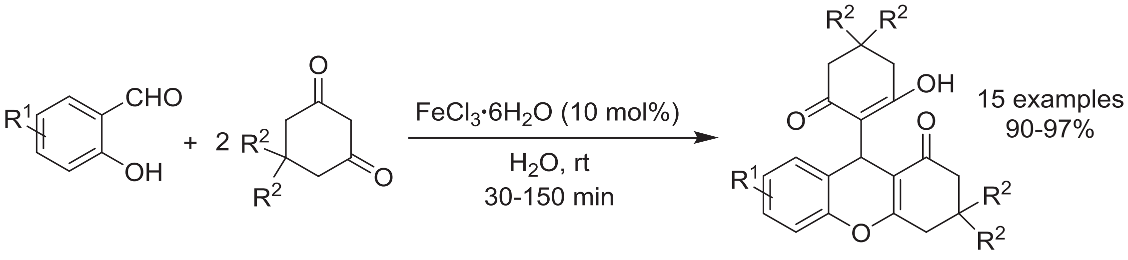
Scheme 66

6.4.2.7 Xanthenes and Xanthones

DA reaction of 3-vinylchromones with arynes is controlled by the Brønsted acid used: using 1.1 equiv of TfOH, 9-(naphthalen-2-yl)-9*H*-xanthen-9-ols are obtained as major products with small amounts of 2-phenyloxyarylnaphthalen-2-ylmethanones, while using 2.5 equiv of TFA, only 2-hydroxyarylnaphthalen-2-ylmethanones are produced (16T8565).

Regioselective synthesis of rhodamine dyes can be accomplished when various 3-aminophenols react with phthalaldehydic acids in 2,2,2-trifluoroethanol (TFE) or in a 4:1 mixture of TFE/water under an oxygen atmosphere, on gram scale with good yields (16OL5316). Various spirodiazafluorenes are attained via tandem Friedel–Crafts reaction, nucleophilic addition, and intramolecular cyclization reactions of diazafluorenones with phenols, using TfOH in 1,2-dichlorobenzene at 85°C (Scheme 66) (16OL6220).

Under solvent-free conditions, the reaction of 2-hydroxy-1,4-naphthoquinone with benzaldehydes carried out in the presence of a catalytic amount of *p*-TsOH at 80°C affords 7-aryl-6*H*-dibenzo[*b*,*h*]xanthene-5,6,8,13(7*H*)-tetraones (16JOC5525).

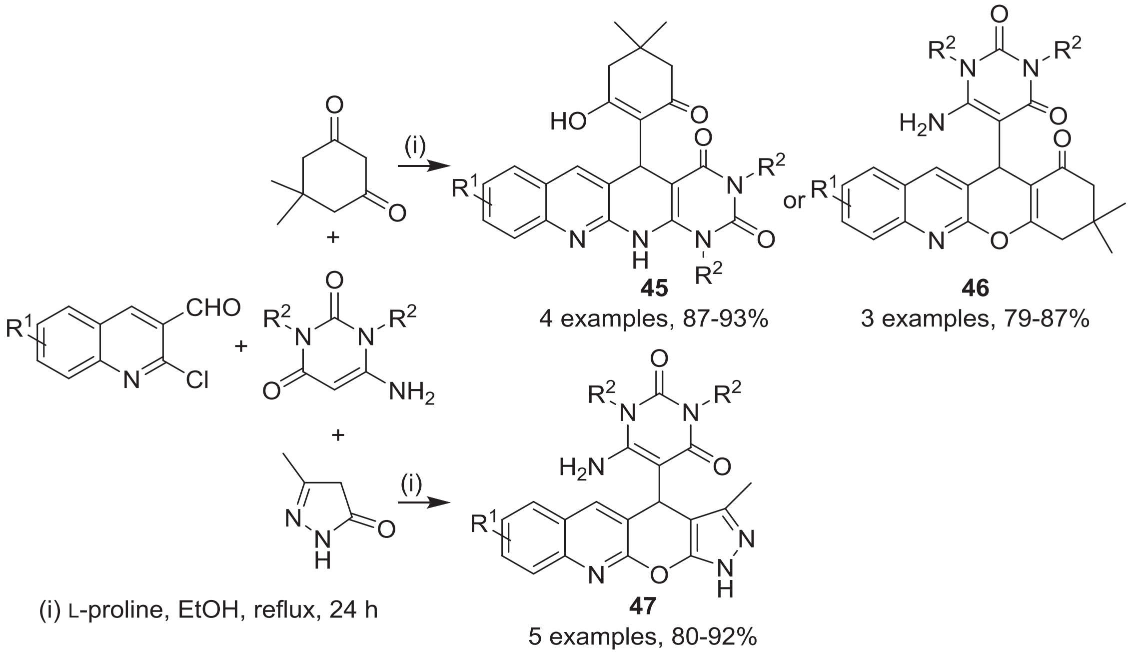


Scheme 67

A range of unsymmetrical 9-arylxanthenes arise from Ca(OTf)$_2$-catalyzed one-pot reactions of 2-[(2-hydroxyaryl)methyl]phenols with 4-hydroxycoumarin/cyclohexane-1,3-diones in refluxing water or with naphth-1/2-ols under neat conditions and with microwave irradiation (16TL2351). One-pot reactions of 1-(2-hydroxyaryl)-propargyl/diaryl amines with cyclohexane-1,3-diones promoted by anhydrous FeCl$_3$ provides 9-aryl/9-arylethynyl-2,3,4,9-tetrahydro-1*H*-xanthen-1-ones in 68%–95% yields (16JOC2062). The hydrated iron catalyst (FeCl$_3$·6H$_2$O) was used to mediate the condensation reaction of salicylaldehydes with cyclohexane-1,3-diones in water at room temperature to prepare hexahydroxanthen-1-ones (Scheme 67). The catalyst can be reused in four cycles with similar efficiency (16TL141). Several octahydroxanthene-1,8-diones were synthesized through the reaction of benzaldehydes with cyclohexane-1,3-diones catalyzed by titanium aminophosphates in a 1:1 mixture of methanol/water (16SC1671) or a cesium salt of phosphotungstic acid in water (16SC1766). Similar xanthene-type derivatives were obtained from the reactions of aromatic aldehydes with substituted thiobarbituric acids catalyzed by *p*-TsOH (16JHC1646) and of arylglyoxal monohydrates with 2,4-dihydroxyquinoline using a catalytic amount of DBU (16TL105), both in refluxing ethanol.

Under solvent-free conditions, one-pot three-component reactions of naphth-2-ols with benzaldehydes and dimedone promoted by guanidine hydrochloride led to 12-aryl-8,9,10,12-tetrahydrobenzo[*a*]xanthen-1-ones in high yields (16JHC981). Other related derivatives are attained from microwave-assisted reaction of kojic acid with aromatic aldehydes and dimedone in the presence of ytterbium perfluorooctanoate (16JHC493) and from the reaction of naphtha-2,3-diol with aromatic aldehydes and cyclic 1,3-dicarbonyl compounds carried out in the presence of formic acid (16SC1699).

One-pot reaction of 2-chloroquinoline-3-carbaldehydes with 6-aminouracils and dimedone in the presence of L-proline in refluxing

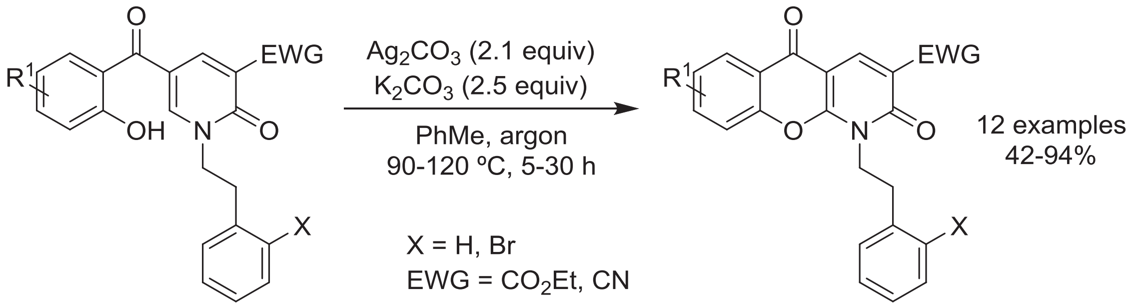


45
4 examples, 87-93%

46
3 examples, 79-87%

47
5 examples, 80-92%

Scheme 68



Scheme 69

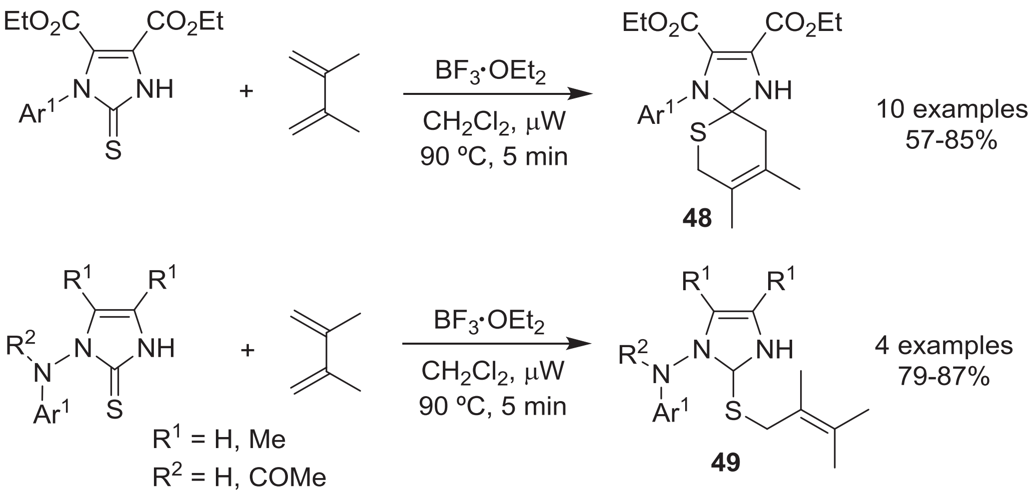
ethanol provides xanthenes **45** or **46**, according to the substitution pattern of the aldehyde. Replacing dimedone by 3-methyl-1*H*-pyrazol-5(4*H*)-one gave only compounds **47** (Scheme 68) (16TL5435). Similar derivatives arise in good yields from microwave-assisted three-component reactions of 4-hydroxycarbazole with aromatic aldehydes and 2-amino-1-ene-1,1,3-tricarbonitrile using sodium ethoxide in ethanol (16TL1985).

A palladium-catalyzed acylation S_N–Ar sequence converted 2-bromofluorobenzenes with salicylaldehydes into a series of substituted xanthones (16SL1269). Selective C-6–aryloxylation of 1-substituted 5-(2-hydroxybenzoyl)pyridin-2(1*H*)-ones occurs in the presence of silver carbonate and potassium carbonate in toluene under argon atmosphere to provide xanthone-type compounds in 42%–94% yields (Scheme 69) (16OBC3564).

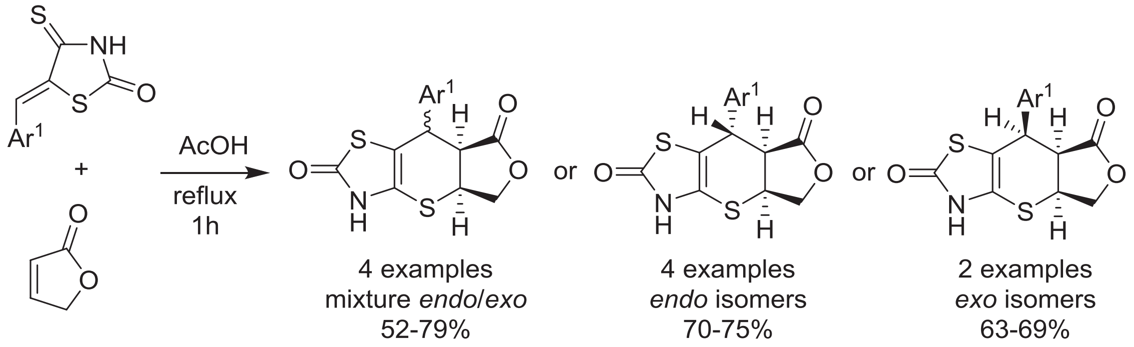
6.4.3 HETEROCYCLES CONTAINING ONE OR TWO SULFUR ATOMS

6.4.3.1 Thiopyrans and Analogs

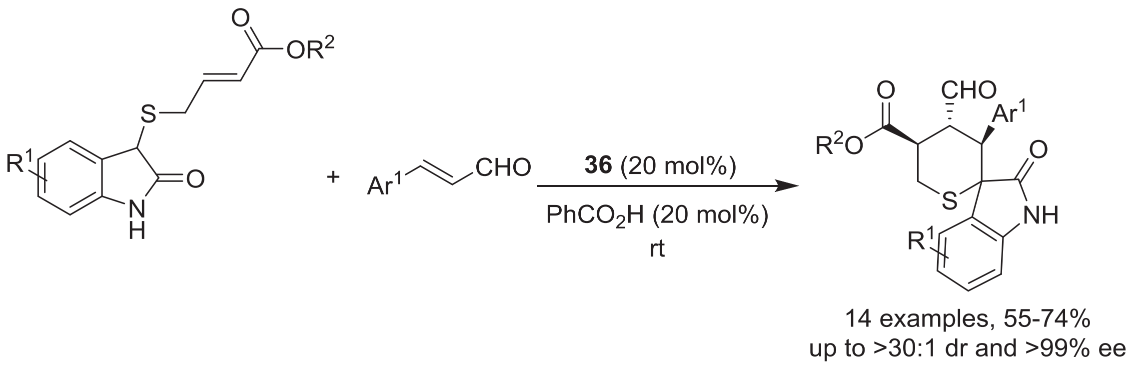
A series of pyrrolo[2,3-*b*]-2*H*-thiopyrans results from an intramolecular cyclization of 5-(prop-2-yn-1-ylsulfanyl)-1*H*-pyrrole-2-amines promoted by DMSO at 118–122°C for 5–8 min (16S4278). Microwave-assisted one-pot three-component reactions of 3-methyl-2-thioxo-1,3-thiazolidin-4-one, aromatic aldehydes, and 3-aryl-3-oxodithiopropionic acid methyl esters in the presence of the basic ionic liquid [bmIm]OH led to thiazole-2-thiono[4,5-*b*]-4*H*-thiopyrans in good yields (16JHC849). $BF_3{\cdot}OEt_2$-catalyzed hDA reaction of *N*-arylsubstituted imidazole-2-thiones with 1,3-dimethylbutadiene under microwave irradiation is substrate controlled: electron-withdrawing groups give spiroimidazole dihydro-2*H*-thiopyrans **48**, whereas electron-donating groups afford sulfur-substituted imidazole derivatives **49** (Scheme 70) (16T1742). Other hDA reaction of 5-arylideneisorhodanines with 2(5*H*)furanone in boiling acetic acid provide a mixture of *endo*/*exo* or single diastereomeric furanone-thiazole-fused 2,3-dihydro-4*H*-thiopyran adducts (Scheme 71). In the presence of an *o*-phenolic group on the arylidene moiety, a diastereoselective tandem reaction occurs to give dihydrocoumarin-thiazole-fused 2,3-dihydro-4*H*-thiopyran derivatives (16TL3318). A *Cinchona*-derived organocatalyst promotes regioselective [3 + 2]/[4 + 2]-cascade reactions of α,β-unsaturated methanesulfonamides with 3-isothiocyanato oxindole to afford spirooxindole pyrrolidine-fused 2,3-dihydro-4*H*-thiopyrans in high yields and enantioselectivity (16CEJ4733).



Scheme 70



Scheme 71

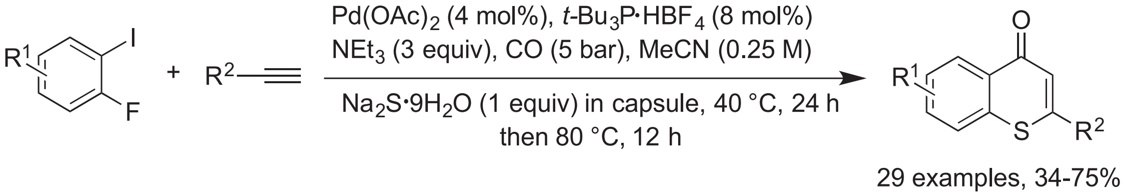


Scheme 72

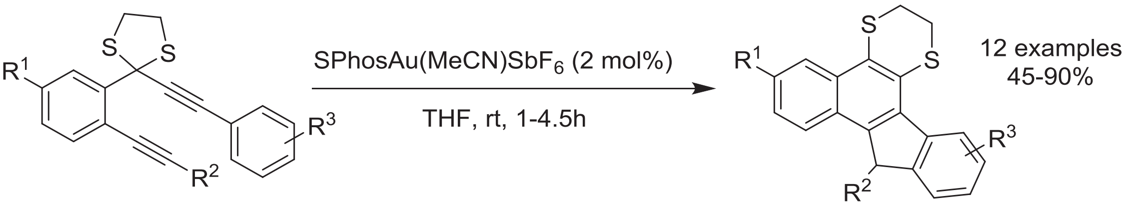
Chiral tetrahydrothiopyran-4-ols are obtained from the [3 + 3]-annulation reaction of 2-substituted cyclopropane 1,1-diesters with mercaptoacetaldehyde mediated by an *N*,*N*′-dioxide/scandium(III) complex (16OBC5914). Organocatalytic Michael/Michael cascade reactions of various 3-thiosubstituted indolin-2-ones with α,β-unsaturated aldehydes produces spirooxindole tetrahydrothiopyrans in good yields and with excellent diastereo- and enantioselectivities (Scheme 72) (16OL1028).

A series of chiral 2-substituted thiochromenes were obtained through an acyl transfer-initiated cascade reaction of *S*-cinnamoyl derivatives of benzaldehyde promoted by an amidine-based catalyst (16OL3454). Organocatalyzed Michael/aldol reactions of 2-mercaptobenzaldehyde with β-indol-3-yl-β-trifluoromethyl enones leads to 4-hydroxy-2-indol-3-yl-2-trifluoromethyl-substituted thiochromans (16OL3546) and with 3-methyleneindolin-2-ones gives 3-spirooxindole thiochroman-4-ols (16OL5824).

A large variety of thiochromen-4-ones arise from palladium-mediated carbonylative four-component reactions of 1-fluoro-2-iodobenzaldehydes with alkyl/arylacetylenes and using a paraffin wax capsule to control the release of sodium sulfide nonahydrate (Scheme 73) (16AGE5067). Rhodium(I)-catalyzed *S*-conjugate additions of β-*t*-butylthio-substituted



Scheme 73



Scheme 74

aryl, alkenyl, and alkyl aldehydes with alkynes afford thiochroman-4-ones, tetrahydro-4*H*-thiochroman-4-ones, and tetrahydrothiopyran-4-ones, respectively. Subsequent to *S*-conjugate additions, addition of an aqueous solution of hydrogen peroxide led to in situ oxidation to *S*,*S*-dioxide derivatives, whereas adding *N*-chlorosuccinimide and pyridine provided thiochromen-4-ones and dihydrothiopyrans as well as their chlorinated derivatives (16OL5676).

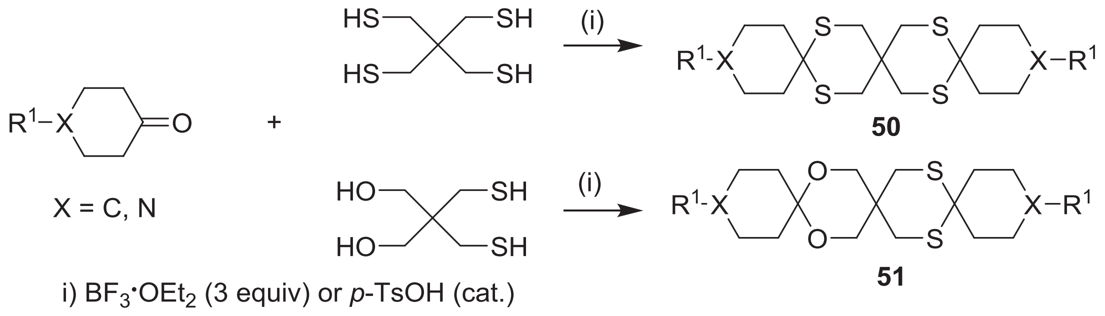
6.4.3.2 Dithiin Analogs

Rhodium-catalyzed addition reactions of sulfur with cyclic alkynes affords symmetrical 1,4-dithiines, while with dialkyl acetylenedicarboxylates in the presence of cyclic alkynes or vinyl ethers, unsymmetrical 1,4-dithiins are produced (16S3107). A range of benzo[*a*]fluorene derivatives arise from gold(I)-mediated cyclization of 1,6-diynyl dithioacetals in THF at room temperature (Scheme 74) (16CC11000). $BF_3{\cdot}OEt_2$ or *p*-TsOH promote ketal formation when cyclohexanone-type derivatives react with 2,2-bis(mercaptomethyl)propane-1,3-dithiol or 2,2-bis(mercaptomethyl)propane-1,3-diol to give trispiranes **50** and **51** (Scheme 75), respectively, used for the preparation of oligospirothioketals (16JOC1125).

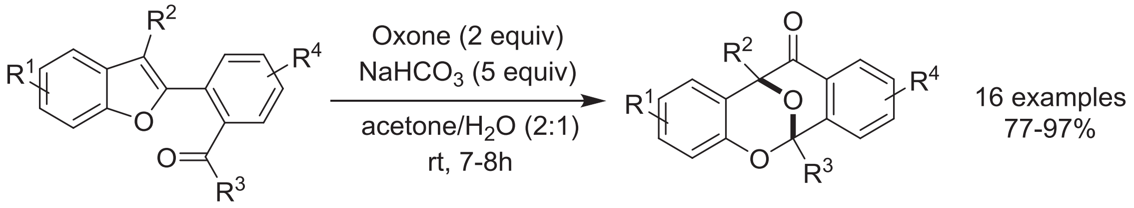
6.4.4 HETEROCYCLES CONTAINING TWO OR MORE OXYGEN ATOMS

6.4.4.1 Dioxines

Gold(I)-mediated tandem annulation reaction of 3-(2-azidoaryl)propargyl alcohols with aldehydes gives 6-(2-azidoaryl)-4*H*-1,3-dioxines. If the



Scheme 75



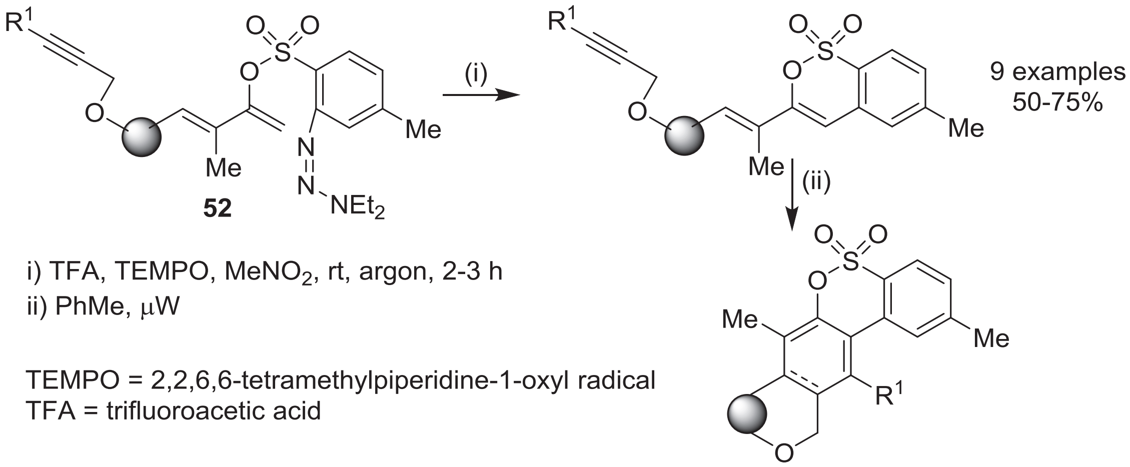
Scheme 76

aromatic ring possesses 3,5-dichloro substitution, a range of indole-fused 1,3-dioxines are formed (16EJO4265). Functionalized 2,3-dihydro-1,4-dioxines are obtained through ruthenium-catalyzed O—H insertion reactions of α-keto-diazo compounds with β-halohydrins followed by base-promoted cyclization (16JOC11477). Oxone-promoted oxidative dearomatization of 2-(2-oxoaryl)-3-methylbenzo[*b*]furans and subsequent intramolecular cycloaddition reaction of the formed *o*-quinone methides with a carbonyl group lead to tetracyclic 1,3-benzodioxanes (Scheme 76) (16OL612). Enantioselective palladium(0)-catalyzed alkene aryloxyarylation reactions of 2-[(2-alkyl/arylallyl)oxy]phenols with aromatic bromides give rise to a series of 1,4-benzodioxanes in moderate-to-good yields and with excellent enantioselectivity (16AGE5044).

6.4.5 HETEROCYCLES CONTAINING BOTH OXYGEN AND SULFUR IN THE SAME RING

6.4.5.1 Oxathianes

The synthesis of 3-alkenyl benzo[*c*][1,2]oxathiine 1,1-dioxides occurs through a selective benzoannelation–dehydrogenation process when dienes **52** bearing an aryl triazene moiety are treated with equimolar amounts of TFA and TEMPO in nitromethane at room temperature under argon. A subsequent microwave-assisted intramolecular DA reaction gives access to chroman-type-fused 1,2-oxathiine 1,1-dioxides (Scheme 77) (16CEJ285).



i) TFA, TEMPO, $MeNO_2$, rt, argon, 2-3 h
ii) PhMe, µW

TEMPO = 2,2,6,6-tetramethylpiperidine-1-oxyl radical
TFA = trifluoroacetic acid

Scheme 77

High yields of 2-benzylidene-1,4-oxathiane-3-thiones are obtained through multicomponent reactions of terminal alkynes with oxiranes and carbon disulfide, using copper(II) acetate and sodium ascorbate as the catalytic system (16SL259). Treating terminal alkynes with isothiocyanates and epoxides in the presence of silver carbonate in 1,4-dioxane at 55°C leads to 2-benzylidene-1,4-oxathian-3-imines in good-to-excellent yields (16T5301).

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