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Contents

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

6.4

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6.4.1 Introduction

A large variety of publications involving *O*- and *S*-6-membered ring systems have appeared in 2018. The importance of these heterocyclic compounds is highlighted by the large number of publications on the total synthesis of natural oxygen derivatives and of other communications dedicated to natural and synthetic derivatives.

Reviews on the ruthenium-catalyzed synthesis of various *O*-6-membered heterocycles (18SC1551), the enantioselective syntheses of 3,4-dihydropyran derivatives (18CR2080), and strategies for the synthesis of coumarins (18SC1534) have appeared. The synthesis of natural pyran-2-ones and pyran-4-ones starting from alkynes and promoted by gold complexes (18AGE4215) and cyclization strategies of substituted alkynyl acids and esters to afford pyran-2-one and isocoumarin derivatives (18SL1) have been detailed in minireviews. The synthesis of various *O*-6-membered derivatives was also achieved through ultrasound irradiation (18SC1235) and using 4-hydroxy-6-methylpyran-2-one as a building block in multicomponent reactions (18H(96)381).

Recent advances in the asymmetric synthesis of pyrans and chromenes (18TA1462), the chemistry of 2,3-dihydroxynaphthalene for the synthesis of benzochromene, benzoxanthene, and benzodioxine derivatives (18SC2305), and the synthesis of flavones (18T811) have been discussed. The potent synthetic application of Diels−Alder reactions involving *o*-quinodimethanes, aza-*o*-quinone methides, and *o*-quinone methides in the total synthesis of natural products (18CSR7926) and in asymmetric multicomponent reactions for the preparation of spiroxindolinone tetrahydropyran-2-ones and 2*H*-chromenes (18TA708) were also overviewed.

Discussions on specific reactions such as C−H activation for the synthesis of (iso) chromans (18EJO6068); ring closing metathesis (RCM) combined with enzymatic kinetic resolution applied in the synthesis of the enantiomerically pure (6*R*)-phenyl-5,6-dihydro-2*H*-pyran-2-ones (18TA809); radical reactions of aryl alkynoates for the synthesis of coumarins (18TL1309); soft-enolization Baker−Venkataraman rearrangement for the total synthesis of dichromones and related 2-substituted chromones (18OL7424); anion relay chemistry strategy for the total synthesis of

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tetrahydropyran-containing (−)-mandelalides A and L (18JOC4287); titanium(III)-mediated reductive epoxide opening-cyclization for the construction of the cyclopenta[*d*]tetrahydropyran-2-one skeleton in a few synthetic derivatives (18S3006) and also in the total syntheses of (+)-iridomyrmecin, (−)-isoiridomyrmecin, (+)-7-*epi*-boschnialactone, (+)-teucriumlactone, and (−)-dolichodial (18JOC6086); and pericyclic cascade reactions in the biosynthesis of natural isochromene-type derivative leporine C (18AGE2754) were accomplished.

The first synthesis of *O*-doped zig-zag molecular graphenes through oxidative C−C and C−O bond formations and starting from 2,6-dihydroxynaphthalene has been reported (18AGE8942).

The importance of specific reagents, namely, triethylamine as organocatalyst in the synthesis of pyran-, chromene-, coumarin-, and xanthene-type compounds (18S4145), and chiral hypervalent iodine reagents for the synthesis of dihydroisocoumarins (18H(96)563) have been disclosed.

New strategies for the synthesis of a wide variety of naturally occurring bioactive compounds have appeared. Different routes have been established for the total synthesis of tetrahydropyran derivatives 6-acetoxy-5-hexadecanolide (18JOC1627), bryostatin 8 (18AGE942), (−)-dactylolide (18TL763), decytospolide A and B (18OBC5979), 15-*epi*-exiguolide (18S3131), lasonolide A (18AGE16200), mandelalide A and isomandelalide A (18JA770), thailanstatins A-C and spliceostatin D (18JA8303); chromans bavachromanol (18TL1363), both enantiomers of equol and sativan (18T2020), (*S*)-equol, (*S*)-sativan, (*S*)-isosativan, (*S*)-vestitol and (+)-medicarpin (18TL2407), (+)-phomactin A (18OL7466), yaequinolones J1 and J2 (18OL4277); benzo[*c*]chroman derivatives (−)-machaeriol B and (−)-machaeriol D (18OL2964), naphtertin and marinone (18AGE11009); polycyclic chromans dragonbloodins A1 and A2 (18OL1819); isochromans (−)-nanaomycin A (18T4994), penicitol A and stoloniferol B (18OL3021), and pericoannosin A (18OL4475); pyran-2-ones (*ent*)-desoxygermicidin C, (+)-germicidin C, gibepyrone A, racemic gulypyrone A, nectriapyrone, phomapyrone B and (*ent*)-prolipyrone A (18EJO3144), and yangonin (18JOC4279); 5,6-dihydropyran-2-ones (+)-altholactone, (+)-7-*epi*-altholactone (18TL4024), brevipolide M and N (18TL4213), 8-chlorogoniodiol and parvistone A (18TA246), cryptocaryols A and B (18CC3428), (*S*)-dihydrokavain (18SC2382), 2,18-*seco*-lankacidinol B (18AGE13551), phostriecin (18TL454), (−)-rasfonin (18OL5062), and (−)-synargentolide B (18TL291); tetrahydropyran-2-ones (5,7,9*R*)-7,9-dihydroxy-5-decanolide (18TL2893), (−)-etharvensin (18TL4024), pellasoren A (18TL4209), and (+)-tanikolide (18T1059); isocoumarin exserolide F (18SC2403); polyhydroisocoumarins (+)-*epi*-claulansine C (18JOC382), eurotiumide A and B (18EJO4013) and salvinorin A (18OL3418); flavones carambolaflavone A (18JOC4111), isosakuranetin (18JOC4279), and sudachitin (18TL1816); chromanones blennolide D, (*ent*)-blennolide E and F (18OL2186), (+)-cryptocaryanones A and B and (+)-cryptochinones A and C (18OL1945) and viscumneoside III (18T2376); bischromanones blennolide H and phosmopsis-H76 A (18CEJ8760); xanthene-type derivative sparstolonin B (18T3787); xanthone derivatives citreamicin η(18T4981), kibdelone C (18OL2872), (−)-rotenone and (−)-dalpanol (18AGE182); and dioxine-type (−)-maldoxin (18OL3919).

The synthesis of naturally occurring analogous tetrahydropyran derivative belizentrin methyl ester (18AGE10712), chroman *des*-hydroxy paecilospirone (18SL1517) and 5,6-dihydropyran-2-one 1′,2'-(*E*)-isomer of (*ent*)-hyptenolide (18SC2333) have been disclosed.

Enantioselective total synthesis of several meroterpenes was reported, including cochlearoid B (18OBC3358), guadials B and C, guapsidial A and psiguajadial D (18OBC4793), (+)-hongoquercin A and B (18JOC13276), and (+)-psiguadial B (18JOC6066). Total enzyme syntheses of meroterpenes napyradiomycins A1 and B1 (18JA17840), concise total syntheses of monoterpene hybrids callistrilones A-E (18OL2509), and biomimetic total syntheses of callistrilones A, B and D (18OL680) and scabellone B (18SL1617) have also been achieved.

Chemoenzymatic total synthesis of natural 5,6-dihydro-2*H*-pyran-2-ones cryptocaryalactone derivatives (18TL160) was disclosed.

Total synthesis of both (−)- and (+)-artemisinin, starting from commercially available *S*-(+)- and *R*-(−)-citronellene, respectively, as well as antimalarial activity of (−)-artemisinin were reported (18AGE8293). A green chemical approach for the synthesis of artemisinin was accomplished by reacting extracts of *Artemisia annua* plants in the presence of oxygen, acid, and visible light (18AGE5525).

The convergent synthesis of sorangicin A involves ring closing metathesis (RCM) for the formation of the tetrahydropyran ring and a 5-*endo* iodoetherification to construct the dioxabicyclo[3.2.1]octane subunit (18T1071). Enantiospecific total synthesis of cryptopyranmoscatone B2 is accomplished via $FeCl_3$-catalyzed cyclization of an allyl alcohol to form the tetrahydropyran core and RCM reactions to afford the 5,6-dihydropyran-2-one moiety (18T2627). In the total synthesis of projerangolide and jerangolide E, the tetrahydropyran rings were obtained by intramolecular oxa-Michael addition while the 5,6-dihydropyran-2-one units were formed by lactonization under weakly basic conditions (18JOC14091).

The synthesis of tetrahydropyran-containing C-1−C-8 and C-9−C-19 fragments of phorboxazole A (18TL117) and the synthesis and stereochemical revision of C-31−C-67 fragment of amphidinol 3 (18AGE6060) was disclosed.

Some heterocyclic-fused chromans were synthesized and their potential application as blue-fluorescent probes was studied (18EJO4795) and a novel 2,10-bis-styryl-1-benzopyrylium dye revealed extended π-conjugation, which is very important for application as a photosensitizer in dye-sensitive solar cells (18SL1390).

Herein, we provide a personal overview of the most relevant transformations on *O*- and *S*-6-membered heterocycles, published in 2018.

6.4.2 Heterocycles Containing One Oxygen Atom

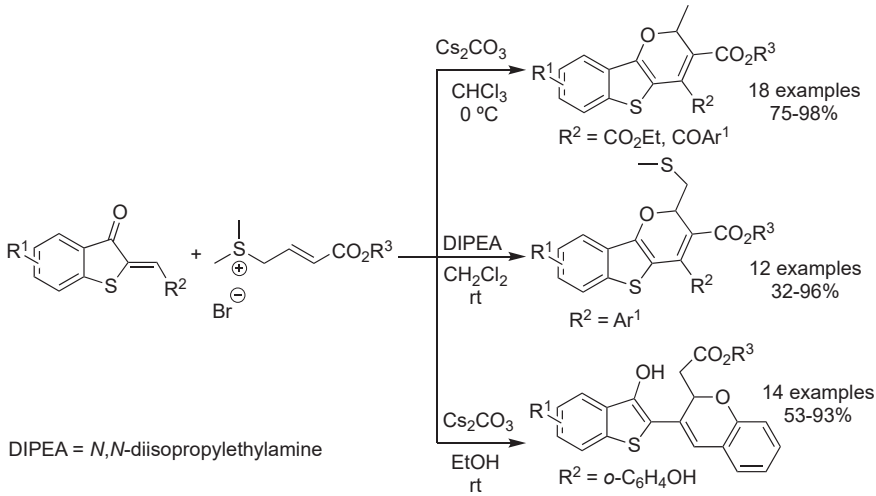
6.4.2.1 Pyrans

Gold(I)-catalyzed intramolecular cyclization through electrophilic aromatic substitution of 4-propargyloxyisoxazoles led to isoxazole-fused 2*H*-pyrans (18OL433). The synthesis of benzo[4,5]thieno[3,2-*b*]pyrans was accomplished through a phosphine-

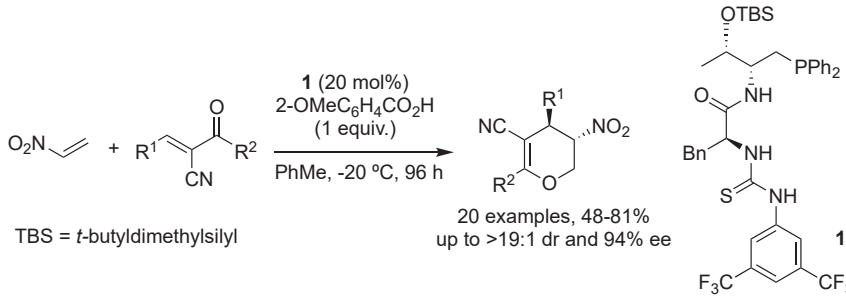
catalyzed [4 + 2] annulation reaction of 2-alkylidenebenzothiophen-3(2*H*)-ones with γ-benzyl allenoates (18OBC2885). The reaction of thioaurone-type compounds with crotonate-derived sulfur ylides is substrate-controlled: (a) compounds bearing an ester or acyl group at the α-position of the 2-alkylidene substituent undergo a [4 + 2] annulation reaction in the presence of cesium carbonate to afford benzo[4,5]thieno[3,2-*b*] pyrans; (b) compounds bearing an aryl group undergo a new [4 + 2] annulation reaction in the presence of *N*,*N*-diisopropylethylamine (DIPEA) with sulfide incorporation to give 2-[(methylthio)methyl]benzo[4,5]thieno[3,2-*b*]-3,4-dihydro-2*H*-pyrans; and finally (c) compounds bearing a 2-hydroxyaryl group, underwent vinylcyclopropane rearrangement domino reaction to provide 3-(3-hydroxybenzo[*b*]thiophen-2-yl)-2*H*-chromene derivatives (Scheme 1) (18JOC13821).

3-Alkylidene-2*H*-1,2-oxazines, obtained from regioselective [4 + 2] cycloaddition reactions of alkenylallenes with nitrosoarenes in THF, undergo triflic acid—promoted rearrangement to form pyran-3(6*H*)-imines (18OL1038). Under visible-light irradiation, trifluoromethylative intramolecular cyclization of 5-arylpent-4-yn-1-ols carried out in the presence of $Ir(ppy)_3$, Umemoto reagent, and lithium carbonate in acetonitrile provides 6-aryl-5-trifluoromethyl-3,4-dihydro-2*H*-pyrans (18OL1698). Highly substituted 3-nitro-3,4-dihydro-2*H*-pyran-5-carbonitriles are enantioselectively obtained from a formal oxa-[4 + 2] annulation reaction of nitroethylene with α-cyano-α,β-unsaturated aryl ketones in the presence of a dipeptide-based phosphine catalyst **1** and 2-methoxybenzoic acid (Scheme 2) (18OL5515).

Catalyst-controlled enantio- and *cis*-selective synthesis of *cis*-2-hydroxy-3,4-dihydro-2*H*-pyran-6-carboxylates are achieved through conjugate addition of aldehydes bearing α-protons with β,γ-unsaturated α-ketoesters promoted by a binaphthyl amino diol catalyst and benzoic acid, followed by cyclization (18CC3496). A metal-free approach for the synthesis of dimethyl 6-aryl-



Scheme 1

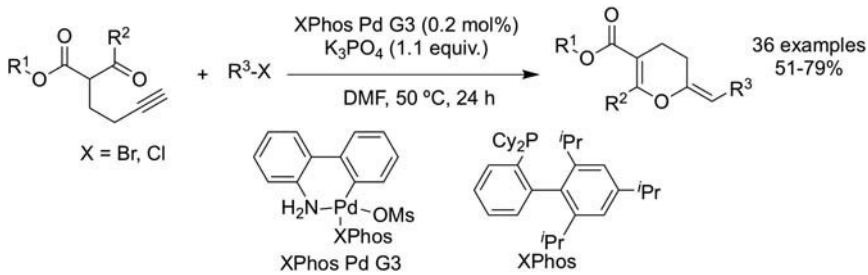


Scheme 2

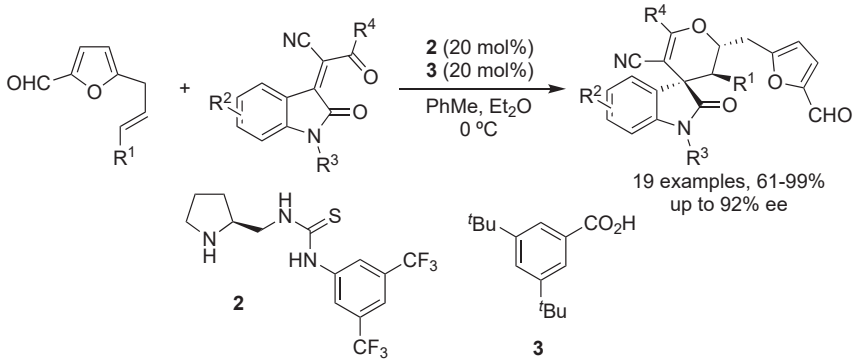
3,4-dihydro-2*H*-pyran-4,5-dicarboxylates involves [2 + 2 +2] cycloaddition of dimethyl acetylenedicarboxylate with benzaldehydes and alkenes using 2-picoline as catalyst in refluxing toluene (18OBC5965). A wide range of (*E*)-2-methylidene-3,4-dihydro-2*H*-pyran-5-carboxylates is formed through a palladium-catalyzed tandem 6-*exo-dig* oxocyclization-coupling reaction of δ-acetylenic β-ketoesters with aryl halides in the presence of potassium phosphate in DMF at 50 °C (Scheme 3) (18JOC12887).

Inverse electron-demand oxa-Diels−Alder (IED-hDA) cycloaddition reactions of 5-allyl furfurals with isatin-derived oxadienes mediated by chiral bifunctional amine-thiourea catalyst **2** and 3,5-di-*t*-butylbenzoic acid **3** in a 1:4 mixture of toluene:diethyl ether affords 4-spiroxindolinones 3,4-dihydro-2*H*-pyrans (Scheme 4) (18OL804).

Intramolecular palladium-catalyzed formal *anti*-carboalkoxylation of 3-(2-bromobenzyls or 2-bromophenols)pent-4-yn-1-ols carried out in the presence of triethylamine in DMF leads to (hetero)cyclic-fused 3,4-dihydro-2*H*-pyrans (18CEJ13446). High yields and enantioselectivity are achieved in the asymmetric Michael addition/hemiketalization domino reaction of 2-alkyl-5-hydroxy-4*H*-pyran-4-ones with β,γ-unsaturated α-ketoesters mediated by a C_2-symmetric bifunctional tertiary amine−squaramide organocatalyst to provide 4-oxopyrano[3,2-*b*]-3,4-dihydro-2*H*-pyrans (18OBC9314). 4-(Dimethylamino)pyridine (DMAP) or 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) catalyze formal [3 + 3] cycloaddition



Scheme 3

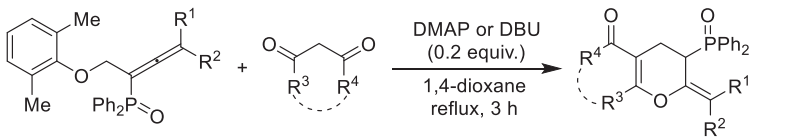


Scheme 4

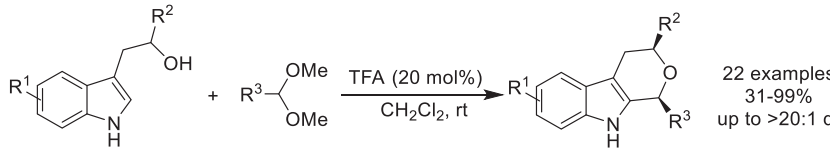
reactions of allenylphosphine oxides with acyclic and cyclic 1,3-diketones in refluxing 1,4-dioxane giving access to polysubstituted 3,4-dihydro-2*H*-pyrans or cyclic-fused 3,4-dihydro-2*H*-pyrans, respectively (Scheme 5) (18OBC6675).

Various pentacyclic 3-spiroxindole indolo[2,3-*b*]-3,4-dihydro-2*H*-pyrans result from one-pot asymmetric aldol/chloroetherification/aromatization sequence involving the reaction of 3-(indol-3-ylmethyl)oxindoles with paraformaldehyde in the presence of a chiral tertiary amine catalyst in DCE, followed by the addition of *N*-chlorosuccinimide (NCS) in the presence of 1,4-diazabicyclo[2.2.2]octane (DABCO) (18OBC6647).

Cyclization reactions of allenic alcohols with aldehydes in dichloromethane at 0 °C promoted by bismuth(III) trifluoromethanesulfonate leads to 3,6-dihydro-2*H*-pyrans while using trimethylsilyl trifluoromethanesulfonate (TMSOTf) at −45 °C provides hexahydropyrano[4,3-*b*]pyrans (18JOC14987). Some 5,6-dihydro-2*H*-pyrans are obtained from enyne metathesis using ruthenium dihydride complexes as catalysts (18TL4471). A series of indolo[2,3-*c*]-5,6-dihydro-2*H*-pyrans were prepared via intramolecular oxa-Pictet−Spengler reactions of 3-(2-vinyloxyethan-1-yl)indoles, under dual catalysis of urea and a chiral phosphoric acid in toluene at −40 °C (18AGE17225). Further derivatives arise from trifluoroacetic acid (TFA)-catalyzed oxa-Pictet–Spengler reactions of 3-(2-hydroxyethan-1-yl)indoles with acetals, prepared in situ from aldehydes and trimethyl orthoformate, in dichloromethane at room temperature (Scheme 6) (18TL129).



Scheme 5

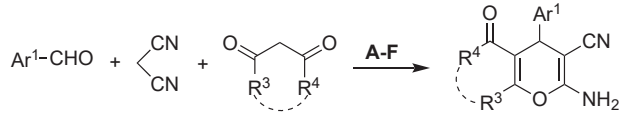


Scheme 6

N-Heterocyclic carbene (NHC)-catalyzed hDA reaction of allenoates with chalcones in the presence of cesium carbonate and under mild conditions furnishes 4,6-diaryl-2-methyl-4*H*-pyran-3-carboxylates (18JOC3361). Several 6-amino-4-aryl-5-cyano-2-methyl-4*H*-pyran-3-carboxylates were produced from multicomponent reactions of aromatic aldehydes with malononitrile and acetoacetates [5-(1,2-dithiolan-3-yl)pentyl 3-oxobutanoate or 1-benzhydrylazetidin-3-yl 3-oxobutanoate] in the presence of ammonium hydroxide in acetonitrile (18S1020). A large variety of 5,6-disubstituted 2-amino-4-aryl-4*H*-pyran-3-carbonitriles were prepared via three-component reactions of aromatic aldehydes with malononitrile and acyclic and cyclic 1,3-dicarbonyl compounds mediated by imidazole in a 1:1 mixture of ethanol:water (18JHC1189), mediated by DABCO (18JHC1678) and magnetic graphitic carbon nitride (18SL645) in ethanol, and mediated by β-cyclodextrin (18SC2046), L-valine (18SC188) or protic ionic liquid (18JHC1010) in water (Scheme 7).

Several 4-spiroxindolin-2-one 2-amino-4*H*-pyran-3-carbonitriles were synthesized through the reaction of isatylidenes with dialkyl acetone-1,3-dicarboxylates in the presence of triethylamine using ethanol as solvent (18SC582).

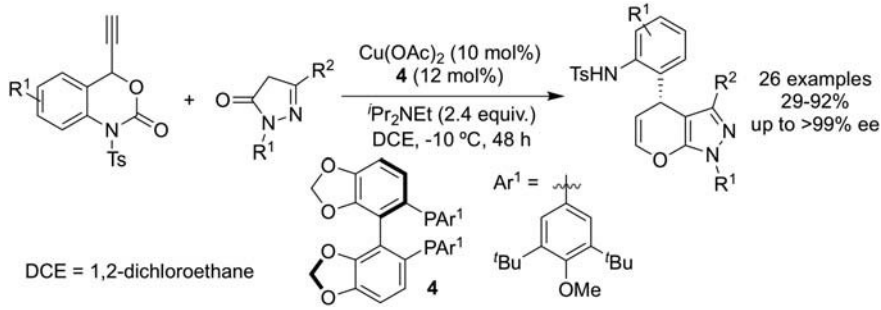
One-pot cascade heterocyclization reactions of 2-(3-oxo-1,3-diarylpropyl)malononitriles promoted by triphosgene and triphenylphosphine oxide lead to 2,4-dichloropyrimidino[4,5-*b*]-4,6-diaryl-4*H*-pyrans (18JOC6423). A series of 1,3-disubstituted pyrazolo[5,4-*b*]pyrans were synthesized via asymmetric [3 + 3] annulation reactions of 1,3-disubstituted 1*H*-pyrazol-5(4*H*)-ones with ethynyl benzoxazinanones (Scheme 8) (18OL5278), or 3-trimethylsilylpropargylic acetates (18CC12033) mediated by copper complexes of chiral *P*,*P*-bidentate or *P*,*N*,*N*-



A: imidazole, EtOH/H_2O (1:1), rt — 6 examples, 86-94%
B: DABCO, EtOH, rt — 14 examples, 82-93%
C: magnetic graphitic carbon nitride, EtOH, 60 °C — 21 examples, 72-97%
D: β-cyclodextrin, H_2O, 60-65 °C — 15 examples, 61-93%
E: L-valine, H_2O, 40 °C — 20 examples, 78-96%
F: protic liquid ionic DABCO/AcOH/H_2O (1:1:3), rt — 13 examples, 80-94%

DABCO = 1,4-diazabicyclo[2.2.2]octane

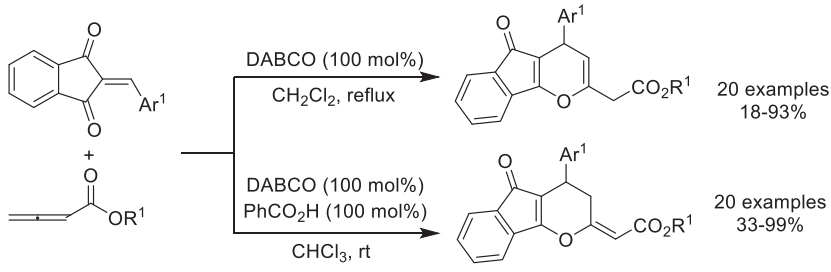
Scheme 7



Scheme 8

tridentate ligands, respectively; or with 3-(2-arylethynyl)-3-alken-2-ones promoted by a tertiary amine−squaramide catalyst (18TA1708). The synthesis of 2-amino-1,3-disubstituted pyrazolo[5,4-*b*]pyran-3-carbonitriles is accomplished via one-pot, three-component reaction of aromatic aldehydes with malononitrile and 3-methyl-1-phenyl-2-pyrazolin-5-one catalyzed by recyclable $Zn(ANA)_2Cl_2$ (ANA = 2--aminonicotinaldehyde) in water (18SC2642) and by sodium fluoride under ultrasonic irradiation (18SC1994). Four-component reactions of benzyl halides with malononitrile/ethyl cyanoacetate, diethyl acetylenedicarboxylate/ethyl acetoacetate and hydrazine hydrate promoted by *N*-methylmorpholine *N*-oxide and silver oxide in refluxing ethanol provides 2-amino-3-substituted pyrazolo[3,4-*b*]pyran-3-carbonitrile/carboxylates (18SC146).

The reaction of allenoates with 2-arylidene-1*H*-indene-1,3(2*H*)-diones occurs under dual catalysis of DABCO, as Lewis base promoting [4 + 2] cycloaddition reaction in refluxing dichloromethane to afford indanone-fused 4*H*-pyran-2-acetates and as Brønsted base, with the subsequent double bond isomerization in the presence of benzoic acid as additive in chloroform at room temperature, leading to indanone-fused 3,4-dihydropyran-2-ylideneacetates (Scheme 9) (18EJO4917). Asymmetric organocatalyzed [4 + 2] cycloaddition reaction of α-haloaldehydes with *N*-substituted 4-ylidenepyrrolidine-2,3-diones in chlorobenzene at room temperature provides polyfunctionalized pyrrolidinone-fused 3-halo-2-hydroxy-4*H*-pyrans (18SL2601).

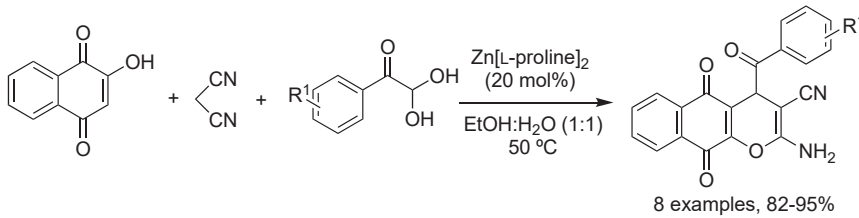


Scheme 9

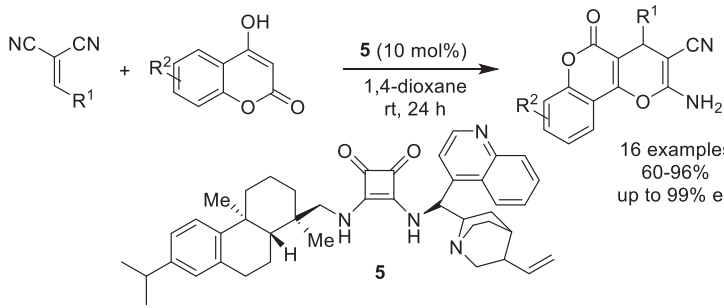
Visible light−promoted condensation reactions of 4-hydroxycoumarins with aromatic aldehydes and acetophenones in ethanol furnishes 2,4-diarylcoumarino[4,3-*b*]pyrans (18SC809). Three-component reactions of 2-hydroxy-1,4-naphthoquinone with malononitrile and different arylglyoxals in the presence of Zn[L-proline]$_2$ as catalyst leads to 2-amino-4-benzoyl-1,4-naphthoquinono[2,3-*b*]pyran-3-carbonitriles (Scheme 10) (18JHC951). Replacing 2-hydroxy-1,4-naphthoquinone by 4-hydroxyquinolin-2(1*H*)-one and using SBA-15 as a nanocatalyst, 2-amino-4-benzoyl-2-oxoquinolino[4,3-*b*]pyran-3-carbonitriles are obtained (18JHC149).

Various 2-amino-4-substituted coumarino[4,3-*b*]pyran-3-carbonitriles were synthesized through an asymmetric domino reaction of 2-methylenemalononitriles with 4-hydroxycoumarins promoted by a chiral squaramide organocatalyst **5** (Scheme 11) (18OBC472). Three-component reaction of 5,7-dihydroxy-4-methylcoumarin with aromatic aldehydes and ethyl cyanoacetate in the presence of silica sodium carbonate delivers ethyl 2-amino-4-arylcoumarino[7,8-*b*]pyran-3-carboxylates (18JHC125). A series of 2-amino-4-arylcoumarino[4,3-*b*]pyran-3-carboxamides result from three-component condensation reactions of 4-hydroxycoumarin with aromatic aldehydes and cyanoacetamide in the presence of a catalytic amount of polystyrene-supported *p*-toluenesulfonic acid in ethanol (18SC2232).

Asymmetric Michael addition/annulation reactions of 2-(2-substituted ethynyl) cyclohex-2-en-1-ones with 1,3-disubstituted pyrazol-5-ones promoted by a chiral cinchona alkaloid leads to tricyclic cyclohexanone-fused pyrazolo[5,4-*b*]-4*H*-pyrans (18CC2028). Tricyclic polysubstituted 4*H*-pyrans are prepared through microwave-assisted multicomponent reaction of secondary amines with cyclic 1,3-dicarbonyl



Scheme 10

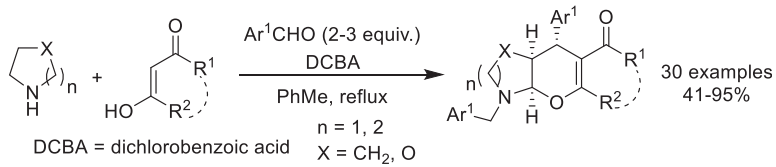


Scheme 11

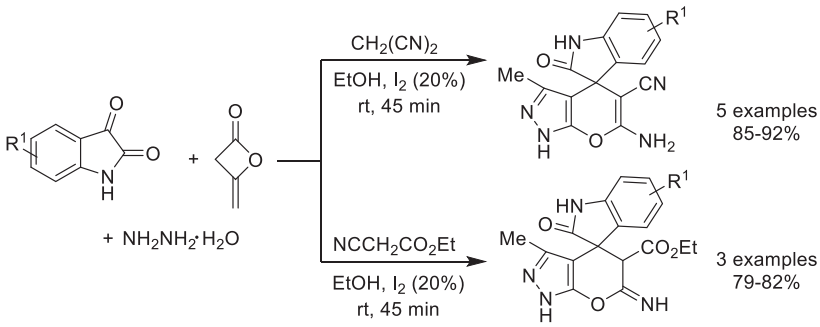
compounds, benzaldehydes, and 2,4-dichlorobenzoic acid in refluxing toluene (Scheme 12) (18JOC8874). The synthesis of pyrazolopyranopyrimidines is accomplished by a four-component domino reaction of aromatic aldehydes with barbituric acid, hydrazine derivatives, and ethyl acetoacetate using choline chloride:urea complex as deep eutectic solvent (18JHC716). High yields of bis(2-amino-4-arylcoumarino[4,3-*b*]-4*H*-pyran-3-carbonitriles) are obtained in one-stage or two-stage protocols, involving multicomponent reaction of bisarylaldehydes with malononitrile and 4-hydroxycoumarin in the presence of pyridine or acetic acid/sodium acetate medium (18JHC498).

A chiral squaramide catalyzes Michael addition/cyclization tandem reaction of dimedone with isatylidenemalononitriles to afford 4-spiroxindolin-2-one cyclohexanone-fused 2-amino-4*H*-pyran-3-carbonitriles in excellent yields and with excellent enantioselectivity (18T7148). A wide range of 4-spiroxindolin-2-one coumarin-fused 3-substituted 2-amino-4*H*-pyrans were synthesized via one-pot, three-component reactions of isatins with 4-hydroxycoumarin and active methylene compounds in γ-valerolactone as a green reaction media (18JHC2817) and of *N*-alkyl-1-(methylthio)-2-nitroethenamine, derived from the addition of various amines to nitroketene dithioacetal, with isatin derivatives and 4-hydroxycoumarin in refluxing ethanol (18JHC2693). One-pot, four-component reactions of isatins with 4-methyleneoxetan-2-one, hydrazine hydrate, and malononitrile or ethyl cyanoacetate in the presence of iodine in ethanol at room temperature provides 4-spiroxindolin-2-one pyrazole-fused 2-amino-4*H*-pyran derivatives (Scheme 13) (18JHC2772).

A few examples of 2-(alk-1-en-3-ol)tetrahydropyrans arise from cycloisomerization reactions of epoxyhex-1-en-6-ols catalyzed by tetrakis(triphenylphosphine)



Scheme 12

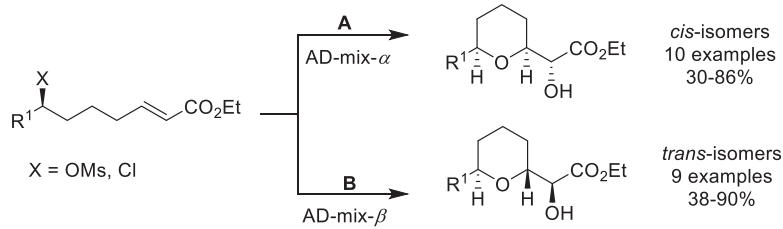


Scheme 13

palladium(0) in the presence of phosphite ligands and diphenylphosphinic acid as a Brønsted acid cocatalyst in dichloromethane (18JOC6259). 2,6-Disubstituted tetrahydropyrans can be obtained through an organocatalyzed asymmetric cycloetherification of *rac*-1-aryl-7-hydroxyhept-2-en-1-ones via kinetic resolution using a chiral phosphoric acid catalyst (18S4243). Diastereoselective tandem dihydroxylation reactions followed by S_N2 cyclization of 7-mesyloxy/7-chloro α,β-unsaturated esters led to the synthesis of *cis*- and *trans*-2,6-disubstituted tetrahydropyrans (Scheme 14) (18OL6910). Nickel-catalyzed intramolecular hydroalkenylation of *O*-tethered 1,6-dienes affords 3-methylenetetrahydropyrans in moderate to good yields (18JA7458).

Tandem Prins cyclization reactions of *N*-(4-hydroxy-2-methylenebutyl)benzamide with aldehydes catalyzed by copper(II) triflate furnishes various spiro 2-phenyl-4,5-dihydrooxazole tetrahydropyrans (18TL1084). The synthesis of bis-spiro tetrahydropyrans is achieved by Prins/Friedel—Crafts cyclization reaction of *N*-[2-(1*H*-indol-3-yl) ethyl]-*N*-(4-hydroxy-2-methylenebutyl)-2-nitrobenzenesulfonamide with aromatic and aliphatic aldehydes in the presence of TFA (Scheme 15) (18EJO1693).

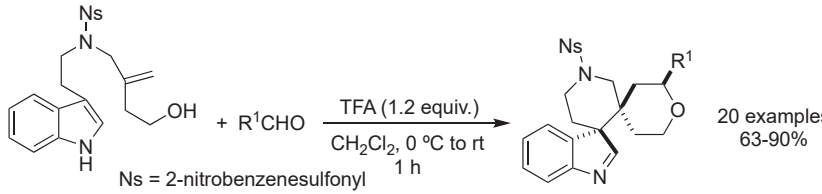
A one-step Prins-type cyclization of 2-arylcyclopropa-1-carbaldehydes with but-3-yn-1-ol promoted by $TiCl_4$ or $TiBr_4$ in dichloromethane with 4Å molecular sieves at −78 °C affords, respectively, 5-aryl-(4,4-dichloro- or 4,4-dibromo)cyclopenta[*b*] tetrahydropyrans (18OL5163). A few examples of 2-oxofurano[4,5-*b*]tetrahydropyrans arising from Prins reactions of homoallylic alcohols with 1,3,5-trioxane, were



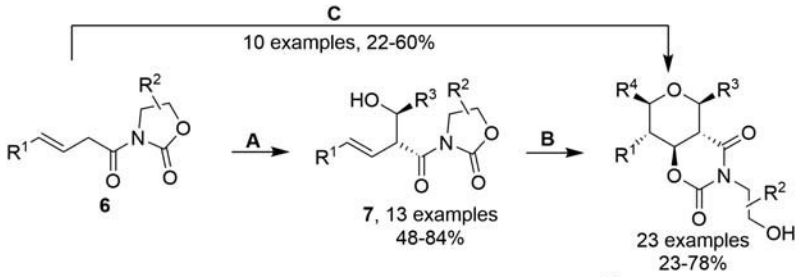
A: 1. $K_3[Fe(CN)_6]$ (3 equiv.), $K_2OsO_2(OH)_4$ (0.005 equiv.), $(DHQ)_2PHAL$ (0.01 equiv.), K_2CO_3 (6 equiv.), $CH_3SO_2NH_2$ (1 equiv.), *t*-BuOH/H_2O (1:1), 0 °C, 12 h
2. 60 °C, 12 h

B: 1. $K_3[Fe(CN)_6]$ (3 equiv.), $K_2OsO_2(OH)_4$ (0.005 equiv.), $(DHQD)_2PHAL$ (0.01 equiv.), K_2CO_3 (6 equiv.), $CH_3SO_2NH_2$ (1 equiv.), *t*-BuOH/H_2O (1:1), 0 °C, 12 h
2. 60 °C, 12 h

Scheme 14



Scheme 15



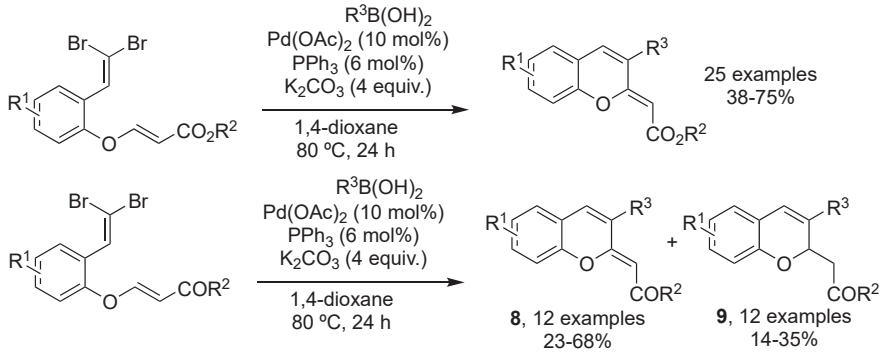
Scheme 16

carried out in the presence of various Lewis acids in dichloromethane (18H(96)453). Several bicyclic 2,3,4,5,6-pentasubstituted tetrahydropyrans were obtained from a one-pot Evans Aldol−Prins reaction of β,γ-unsaturated *N*-acyl oxazolidin-2-ones **6** with aldehydes and from a two-step protocol, which additionally permitted the isolation of β,γ-unsaturated alcohol precursors **7** bearing an *N*-acyl oxazolidin-2-one in the α position (Scheme 16) (18JOC9039).

6.4.2.2 [1]Benzopyrans and Dihydro[1]benzopyrans

6.4.2.2.1 Chromenes and Chromans

2-Amino-*N*-Boc-*O*-propargylphenols undergo platinum(II)-catalyzed intramolecular hydroarylation to give 8-(*N*-Boc)amino-2*H*-chromenes (18EJO6176). The synthesis of 2,2-disubstituted 2*H*-chromenes involves electrocyclization reactions of vinyl *o*-quinone methides catalyzed by Brønsted acid benzene-1,3,5-tricarboxylic acid, in acetonitrile at 50 °C (18S2416). Intramolecular alkyne−aldehyde metathesis of 2-(3-arylpropargyloxy)benzaldehydes mediated by boron trifluoride−etherate ($BF_3 \cdot OEt_2$) in 2,2,2-trifluoroethanol (TFE) delivers 3-aroyl-2*H*-chromenes in excellent yields (18TL4263). One-pot organocatalytic domino Henry/Michael/dehydration reactions of ethyl (*E*)-3-(2-formylphenoxy)acrylates with nitromethane promoted by a cyclohexanediamine-based Takemoto thiourea catalyst affords 2-(ethoxycarbonylmethyl)-3-nitro-2*H*-chromenes (18TL3511). Other 3-[2-(2,2-dibromovinyl)phenoxy] acrylates underwent palladium(II)-catalyzed cascade reactions with aryl boronic acids giving access to (3-aryl-2*H*-chromen-2-ylidene)acetates. Replacing acrylates by 3-[2-(2,2-dibromovinyl)phenoxy]enones, a series of 3-aryl-2*H*-chromen-2-ylidenones **8** are

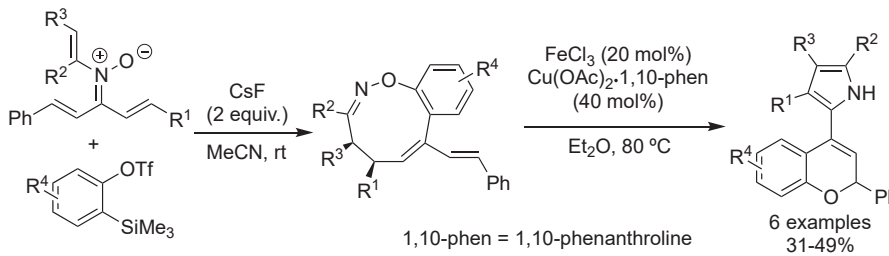


Scheme 17

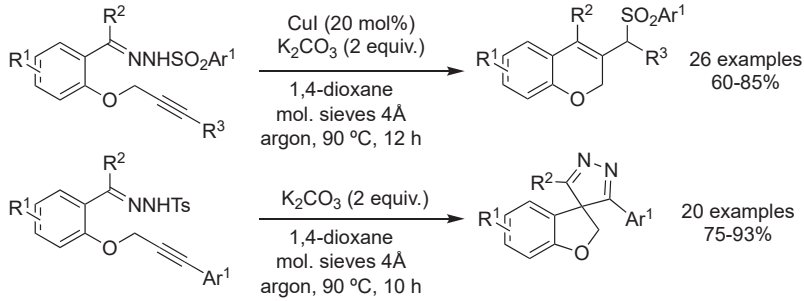
obtained as major products along with small amounts of the corresponding (3-aryl-2*H*-chromen-2-ylidene)ethanones **9** (Scheme 17) (18JOC15256).

The synthesis of 2-alkyl-2*H*-chromene-3-carboxylic acids can be accomplished through a rhodium(III)-catalyzed C−H activation/unusual [3 + 3] annulation reaction of *N*-phenoxyacetamides with methyleneoxetanones carried out in the presence of cesium acetate in acetonitrile (18OL3892). Several functionalized benzoxazonines, formed via formal [7 + 2] cycloaddition reactions of *N*-vinyl-α,β-unsaturated nitrones with arynes in the presence of cesium fluoride in acetonitrile, undergo N−O bond cleavage promoted by iron(III) chloride and copper(II) acetate to afford mainly polysubstituted 2-phenyl-4-(pyrrol-2-yl)-2*H*-chromenes (Scheme 18) (18OL4571).

A cyclopentadienyl iridium(III) complex catalyzes double C−H bond activation of anisoles with 3,3-difluoroalk-1-ynes leading to 4-alkyl-3-(1-fluorovinyl)-2*H*-chromenes in moderate to good yields (18JA5370). A divergent transformation involves alkyne-tethered *N*-sulfonyl hydrazones, which in the presence of a copper catalyst leads to 3,4-disubstituted 2*H*-chromenes while under thermal conditions 4-spiroindoline 4*H*-pyrazoles are formed (Scheme 19) (18CEJ6705). A few examples showed (*Z*)-4-(2-bromoaryl)-5,5,5-trifluoro-2-methylpent-3-en-2-ols suffering photoisomerization, using anthracene as photocatalyst in acetonitrile, followed by a cyclization cascade promoted by palladium(II) acetate in refluxing toluene to produce 2,2-dimethyl-4-trifluoromethyl-2*H*-chromenes (18OL724). Under solvent-free



Scheme 18



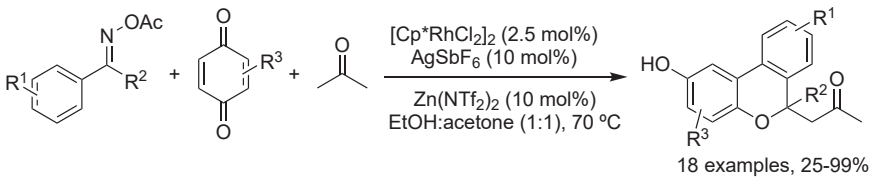
Scheme 19

conditions, one-pot domino reaction of phenols with terminal arylacetylenes mediated by zinc chloride occurs at 140 °C to give 2,2,4-trisubstituted 2*H*-chromenes while a lower temperature (80 °C) leads to 2,4,4-trisubstituted 4*H*-chromenes (18EJO2846).

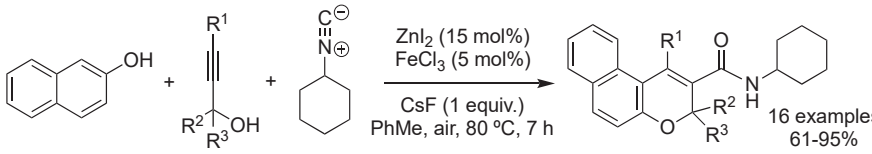
A series of chromene-type compounds arise from domino reactions of cyclohexa-1,3-diones with α,β-unsaturated aldehydes promoted by bovine serum albumin in a 9:1 mixture of propan-2-ol/water (18H(96)1740). Structurally diverse 6*H*-benzo[*c*]chromenes were prepared by photocatalytic Pschorr reactions of 2-(aryloxy)benzenediazonium tetrafluoroborates in the presence of eosin Y in acetonitrile (18SL2311), by visible-light-promoted intermolecular radical addition of $BrCF_2CO_2Et$ or 2-bromo-2,2-difluoroacetamides to biaryl vinyl ethers followed by cyclization (18JOC6151) and by rhodium(III)-catalyzed three-component annulation reactions of aryl ketone *O*-acyloximes with quinones and acetone, the latter acting both as cosolvent and as reactant (Scheme 20) (18OBC6865).

Electrochemical ruthenium(II)-catalyzed annulation reactions of 1,2-diarylethynes with 1-naphthol in protic media prompted a few examples of 2,3-diaryl-6*H*-benzo[*de*]chromenes (18CC12879). A range of 1*H*-benzo[*f*]chromenes were synthesized through rhodium(III)-catalyzed annulation reactions of (*E*)-1-benzylidene-3,4-dihydronaphthalen-2(1*H*)-ones with internal alkynes and subsequent base-promoted 1,5-*H* shift (18OL1074). Three-component coupling of 2-naphthol with propargyl alcohols and cyclohexyl isocyanide in the presence of zinc(II) iodide and iron(III) chloride under air atmosphere delivers *N*-cyclohexyl 3*H*-benzo[*f*]chromene-3-carboxamides in good yields (Scheme 21) (18T3776).

Several 2-spiroindole 2*H*-chromenes were produced via condensation reactions of 1,2,3,3-tetramethyl-3*H*-indol-1-ium iodide with salicylaldehydes in an aqueous



Scheme 20



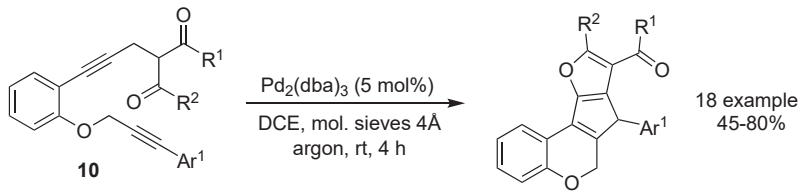
Scheme 21

solution of choline hydroxide at 80 °C (18SC208) and microwave-assisted one-pot alkylation reaction of 1,3-disubstituted 2-methylindoles with alkyl/aryl bromides in water and subsequent condensation with salicylaldehydes in ethanol (18OBC7245).

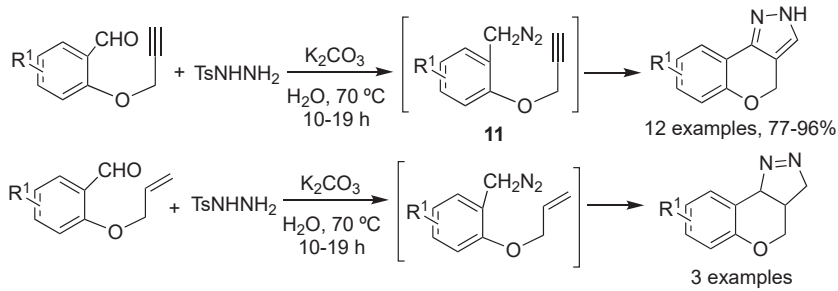
Molecular iodine promotes Friedel−Crafts alkylation/oxidative coupling reactions of indoles (in excess) with salicylaldehydes in refluxing ethanol to produce 4-indolyl indolo[2,3-*b*]chromenes (18T2383). Alkyne-tethered enynones **10** underwent palladium(0)-catalyzed cascade reactions to provide polyheterocyclic-fused chromenes, involving 5-*exo*-*dig* cyclization, carbene/alkyne metathesis and electrophilic aromatic substitution (Scheme 22) (18CC350). Another palladium(0)-catalyzed cascade reaction of 3-aryloxy-4-(2-haloaryl)but-1-ynes led to the synthesis of indano[2,1-*b*]chromenes in moderate to good yields (18AGE10610).

A range of 1,5-naphthyridine-fused chromenes were derived via domino hDA reactions of 3-aminopyridine with *O*-propargylated salicylaldehydes using the catalytic system CuI/$InCl_3$ in refluxing acetonitrile (18H(96)43). Intramolecular [3 + 2] cycloaddition reactions of an appropriate diazocompound **11** formed from *O*-propargylated salicylaldehydes with *p*-toluenesulfonyl hydrazide and potassium carbonate in water at 70 °C provides pyrazolo[4,3-*c*]chromenes. Using *O*-allylated salicylaldehydes, a series of pyrazolino[4,3-*c*]chromans were formed (Scheme 23) (18TL1501).

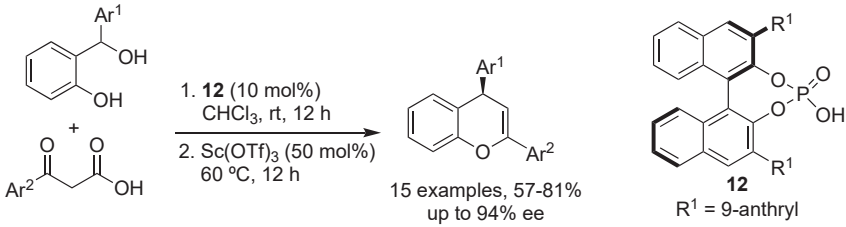
The synthesis of functionalized 2,4-diaryl-4-methyl-4*H*-chromenes can be accomplished via $FeCl_3$-promoted [4 + 2] cycloaddition reactions of 1-aryl-1-(2-hydroxyaryl)alkenes followed by an aryl group elimination (18OBC703). Further derivatives arise from Michael addition of phenols to benzylidene oxobutanoates carried out in refluxing nitromethane in the presence of TFA (18TL2347). A wide variety of 2,4-diaryl-4*H*-chromenes were obtained from enantioselective one-pot decarboxylative alkylation reactions of in situ−generated *o*-quinone methides, from 2-hydroxybenzylic alcohols, with β-aryl-β-ketoacids in the presence of a chiral phosphoric acid **12**, followed by sequential cyclization and dehydration steps promoted by $Sc(OTf)_3$ (Scheme 24) (18OL2944).



Scheme 22



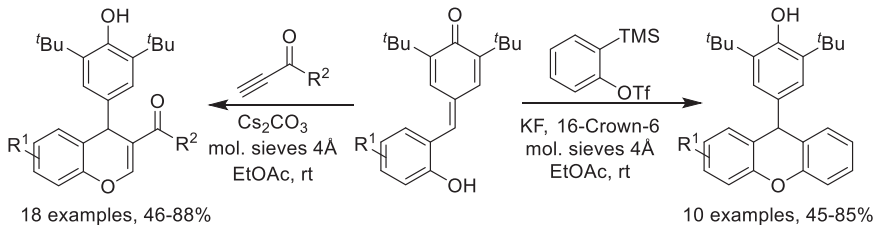
Scheme 23



Scheme 24

Molecular iodine promotes Michael addition reactions of cyclic amides (pyrazole/indolinone) to 2-hydroxychalcones followed by intramolecular cyclization and dehydrogenation processes to produce 2-aryl-4-(pyrazolo/indolinono)-4*H*-chromen-4-ylidenes in good to excellent yields (18T490). Other 2-aryl-4*H*-chromen-4-ylidenes result from tandem reactions of 2-[3-aryl-1-(piperidin-1-yl)prop-2-yn-1-yl]phenols with benzoylacetonitriles/malononitriles in the presence of $FeCl_3$, involving 1,4-conjugate addition, 6-*endo-dig* cyclization and oxidation reactions (18OBC7191). [4 + 2] Cyclization reactions of *o*-hydroxyphenyl *p*-quinone methides with ynones in the presence of cesium carbonate in ethyl acetate led to 3-(acetyl/aroyl)-4-aryl-4*H*-chromenes while the reaction with benzyne formed in situ using KF and 18-crown-6 in ethyl acetate affords 9-aryl-9*H*-xanthenes (Scheme 25) (18JOC1414).

Formal [4 + 2] annulation reactions of 2-hydroxychalcones with allenoates promoted by cesium carbonate are substrate-controlled: with terminal allenoates a series

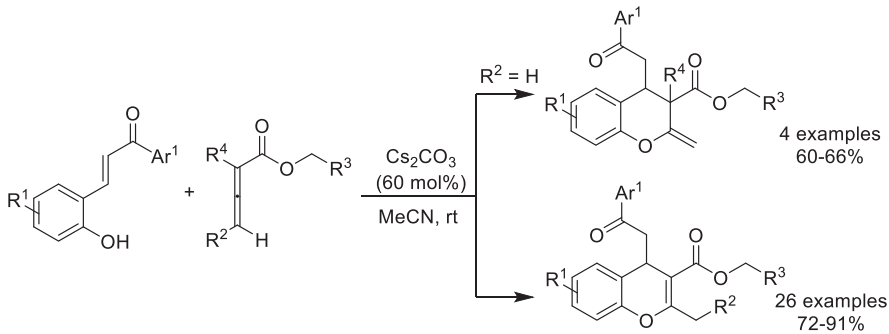


Scheme 25

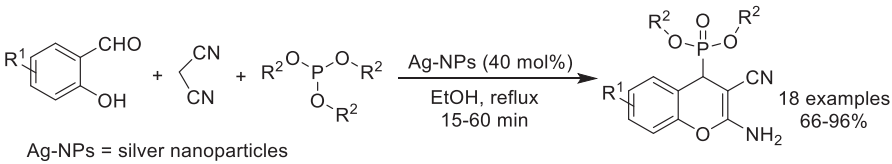
of 2-methylenechroman-3-carboxylates are obtained; with internal allenoates, 4*H*-chromene-3-carboxylates are formed (Scheme 26) (18JOC15372). Highly functionalized 2,4-diaryl-3-sulfonylamino-4*H*-chromenes are achieved through rhodium-catalyzed transannulation reactions of 4-aryl-*N*-sulfonyl-1,2,3-triazoles with in situ−generated *o*-quinone methides from 2-hydroxybenzyl alcohols in refluxing toluene (18OL3762). Various 2,4,4-trisubstituted 4*H*-chromenes were obtained from iron(III) triflate-catalyzed tandem reactions of phenols with two equivalents of ketones in 1,2-dichloroethane (DCE) at 90 °C (18S1482).

Tandem Knoevenagel/Pinner cyclization/Michael addition reaction of salicylaldehydes with malononitrile and pyrazol-5-one/indole derivatives in the presence of DABCO-based ionic liquids leads to 2-amino-4-pyrazol-5-one/4-indolo-4*H*-chromene-3-carbonitriles (18S3708). Under ultrasound conditions, similar three-component reactions of salicylaldehyde derivatives with malononitrile and pyrrole/indole derivatives in the presence of glutathione on superparamagnetic iron oxide nanoparticles in aqueous medium provides 2-amino-4-pyrrolo/4-indolo-4*H*-chromene-3-carbonitriles in excellent yields. Using dimedone instead of malononitrile, a series of xanthene-type compounds were obtained (18SC541). Polysubstituted 2-amino-3-cyano-4*H*-chromen-4-ylphosphonates can be synthesized via one-pot, three-component condensation reactions of salicylaldehydes with malononitrile and trialkyl phosphite using silver nanoparticles as catalyst in refluxing ethanol (Scheme 27) (18SC2366).

Cascade Mannich cyclization reactions of α-amido sulfones derived from salicylaldehydes with (phenylsulfonyl)acetonitrile promoted by chiral squaramide catalysts provides 2,4-diamino-3-(phenylsulfonyl)-4*H*-chromenes, whereas the oxa-



Scheme 26



Scheme 27

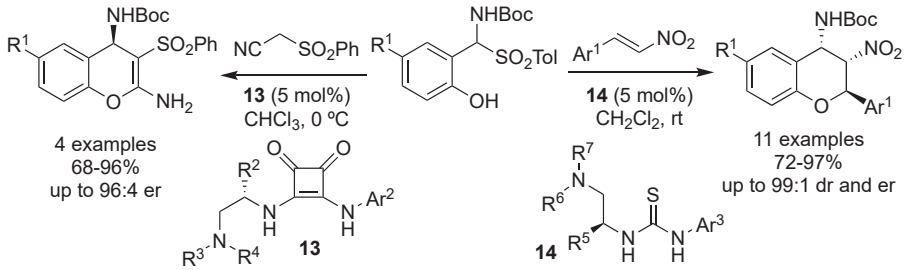
Michael−aza-Henry reaction of the same α-amido sulfones with nitrostyrenes mediated by chiral thiourea catalysts affords 4-amino-2-aryl-3-nitrochromans, in excellent diastereo- and enantioselectivities (Scheme 28) (18JOC5546).

One-pot synthesis of 3-substituted 4-spiroxoindolin-2-one 3-iodo-4*H*-chromenes can be accomplished through Friedel−Crafts alkylation reactions of phenols with 3-alkynyl-3-OBoc oxindoles in the presence of $Cu(OTf)_2$ in dichloromethane and subsequent iodocyclization carried out with molecular iodine and sodium hydrogencarbonate in DCE (18TL4344).

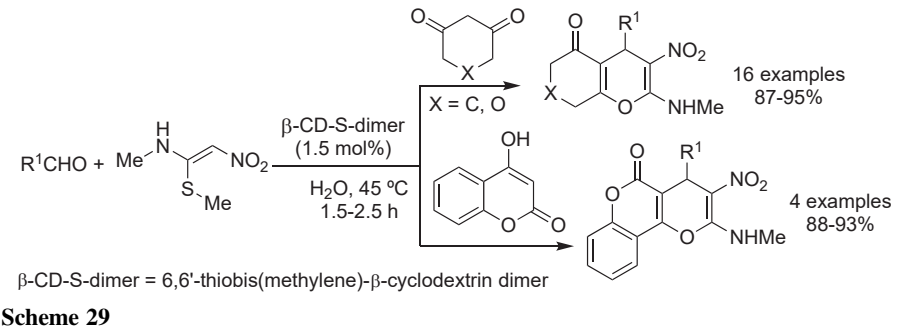
A 6,6′-thiobis(methylene)-β-cyclodextrin dimer catalyzes multicomponent reactions of (hetero)aromatic aldehydes with 1,3-dicarbonyl compounds and (*E*)-*N*-methyl-1-(methylthio)-2-nitroethenamine to yield functionalized 4-(hetero)aryl-2-methylamino-3-nitro-4*H*-chromene-type compounds (Scheme 29) (18T194). A similar protocol occurs under catalyst- and solvent-free conditions, and allows preparation and isolation of products without chromatographic purification (18SC2683>.

The synthesis of tetracyclic tropone-fused tetrahydrochromenes is achieved via rhodium-catalyzed [2 + 2 + 2 + 1] carbonylative cycloaddition reactions of triynes **15** (Scheme 30) (18OL3915).

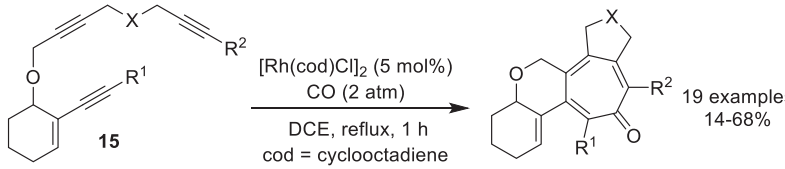
Atroposelective intramolecular [4 + 2] cycloaddition of vinylidene *o*-quinone methides, generated in situ from 1,2-bis[2-(2-hydroxynaphth-1-yl)ethyne]benzenes or 2-[2-(2-hydroxynaphth-1-yl)ethyne]-1-[2-(1-hydroxyphen-1-yl)ethyne]benzene derivatives, mediated by a quinine-derived thiourea catalyst affords indane-fused 2-



Scheme 28



Scheme 29



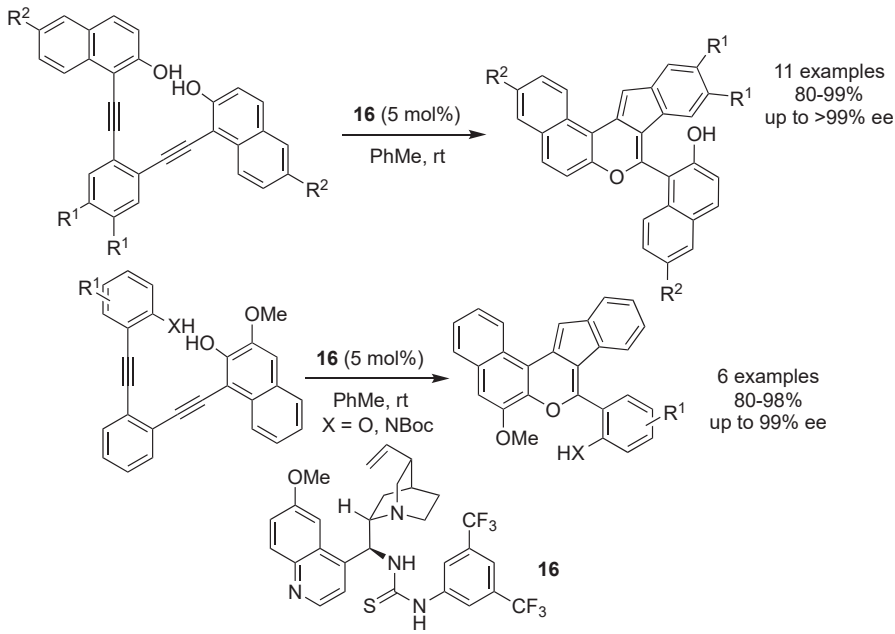
Scheme 30

(2-hydroxynaphth-1-yl)- or 2-(2-hydroxyphen-1-yl)benzo[*f*]chromene derivatives with excellent yields and enantioselectivity (Scheme 31) (18AGE6491).

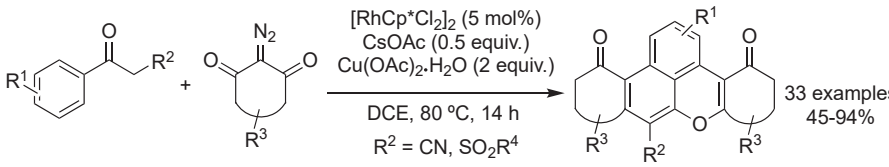
Several pyran-2-one-fused 4-(4-hydroxy-6-methyl-2-oxo-2*H*-pyran-3-yl)-4*H*-chromenes were obtained via pseudo-three-component reactions of two equivalents of 4-hydroxy-6-methyl-2*H*-pyran-2-one with salicylaldehydes promoted by $H_3PMo_{12}O_{40}$ in refluxing ethanol (18T872).

Indanone-fused tetrahydrochromene-type compounds can be synthesized via the [4 + 2] cycloaddition reactions of electron-poor 2-(arylmethylene)-1*H*-indene-1,3(2*H*)-dione heterodienes with electron-rich enaminones promoted by ionic liquid [bmim]BF_4 or acetic acid at 80 °C (18TL1493). Polycyclic benzo[*de*]chromene derivatives were prepared by a rhodium(III)-catalyzed cascade reaction of benzoylacetonitriles/methyl sulfones with cyclic 2-diazo-1,3-dicarbonyl compounds (Scheme 32) (18TL3094).

Three-component domino Knoevenagel condensation/Michael addition/intermolecular cyclization reactions of isatin derivatives with cyclohexane-1,3-diones and 2-hydroxy-



Scheme 31

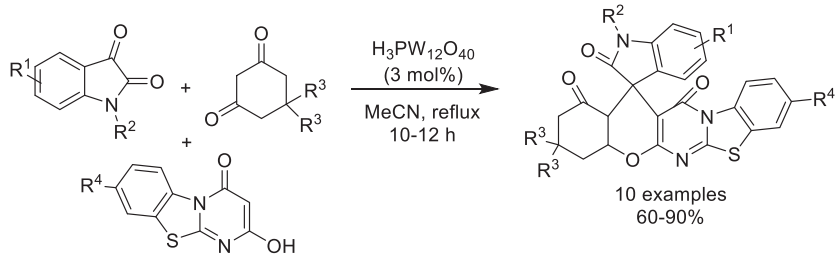


Scheme 32

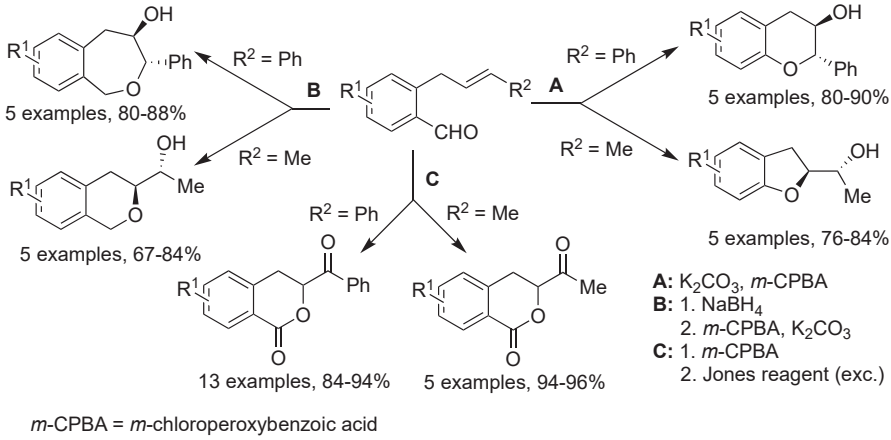
4*H*-benzo[4,5]thiazolo[3,2-*a*]pyrimidin-4-ones and using 12-tungstophosphoric acid ($H_3PW_{12}O_{40}$) as catalyst provided several spiro[benzo[4,5]thiazolo[3,2-*a*]chromeno [2,3-*d*]pyrimidine-14,30-indoline]-1,2′,13(2*H*)-triones (Scheme 33) (18T2967).

Using a combination of *m*-chloroperoxybenzoic acid (*m*-CPBA) and potassium carbonate in dichloromethane, *o*-cinnamyl arylaldehydes underwent intramolecular oxidative annulation reactions to provide 2-phenylchroman-3-ols while *o*-crotyl arylaldehydes afford 1-(2,3-dihydrobenzofuran-2-yl)ethanols. An alternative route uses $NaBH_4$ as reducing agent followed by the addition of *m*-CPBA and potassium carbonate to afford 1,3,4,5-tetrahydrobenzo[*c*]oxepin-4-ols and 1-(isochroman-3-yl) ethanols, respectively, from *o*-cinnamyl- and *o*-crotyl arylaldehydes. A third approach involves the use of *m*-CPBA followed by the addition of an excess of Jones reagent to afford 3-benzoyl- and 3-acetyl-3,4-dihydroisocoumarins respectively from *o*-cinnamyl- and *o*-crotyl arylaldehydes (Scheme 34) (18JOC14110).

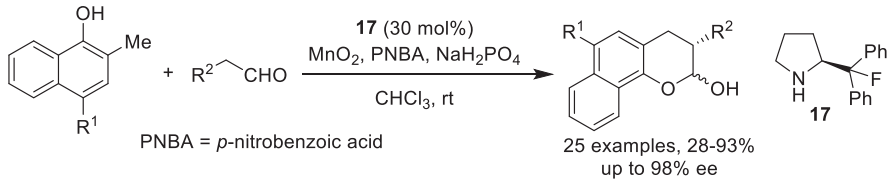
Asymmetric cascade Michael addition/hemiketalization/acyl transfer reactions of 2-hydroxycinnamaldehydes with α-nitroketones under dual catalysis of prolinol TMS ether catalyst and benzoic acid in DCE furnishes 4-(nitromethyl)chroman-2-yl esters (18OBC1598). Formal [4 + 2] cycloaddition reactions of *o*-quinone methides derived from 2-(2-hydroxyaryl)cyclopropa-1,1-diesters with terminal alkenes carried out in the presence of $BF_3 \cdot OEt_2$ in dichloromethane at room temperature gives a series of dimethyl [(2,2-disubstituted chroman-4-yl)methyl] malonates (18OBC3897). Asymmetric formal [4 + 2] annulation reactions of aldehydes bearing α-protons with oxidation-generated β-unsubstituted *o*-quinone methides (derived from *o*-methylphenols) catalyzed by a chiral secondary amine **17** furnished 3-substituted chroman-2-ols (Scheme 35) (18OL174), whereas the reaction of *o*-quinone methides (derived from 2-hydroxyarylbenzyl alcohols) with *o*-hydroxyphenyl-substituted



Scheme 33



Scheme 34

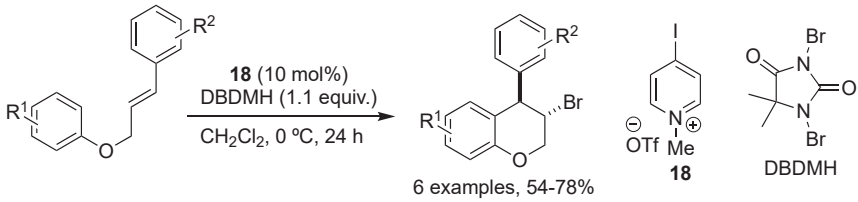


Scheme 35

α,β-unsaturated compounds using a chiral *N*,*N*′-dioxide-Sc(III) complex as catalyst delivered 4-aryl-2-(2-hydroxyaryl)-3-substituted chromans (18JOC10175).

IED cycloaddition reaction of *o*-quinone methides, generated *in situ* from salicylaldehydes, with 1, 1-disubstituted ethylenes, carried out in the presence of TfOH and $CH(OMe)_3$ in toluene is temperature-controlled: at room temperature for one hour leads to 2,2-disubstituted 4-methoxychromans; reacting one hour at room temperature followed by heating at 100 °C for 1 h gives 2,2-disubstituted (2,2-disubstituted ethen-1-yl)chromans (18TL1841). Various polysubstituted 3-oxoalkylchroman-4-ols arise from arginine-mediated cascade cyclizations of salicylaldehydes with alkyl vinyl ketones in water (18TL2356). Bromo-carbocyclization of *O*-cinnamyl phenyl ethers promoted by *N*-methyl 4-iodopyridinium triflate **18** and 1,3-dibromo-5,5-dimethylhydantoin (DBDMH) in dichloromethane at 0 °C delivers 4-aryl-3-bromochromans in moderate to good yields with high chemo- and regioselectivities (Scheme 36) (18AGE3483).

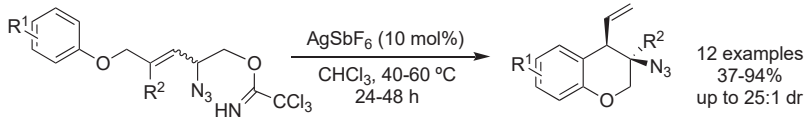
The synthesis of *trans*-4-(fluoren-3-yl)amino-3-vinylchromans is achieved via a one-pot, three-step sequence involving: (1) condensation of *O*-(4-(OBoc)but-2-en-1-yl)salicylaldehydes with 9*H*-fluoren-9-amine in dry dichloromethane, (2) removal of the solvent (without purification) and addition of NaHMDS in THF to form the corrresponding 9-fluorenyl imine product, and (3) reduction with $NaBH_4$ in acetic acid (18OL5857).



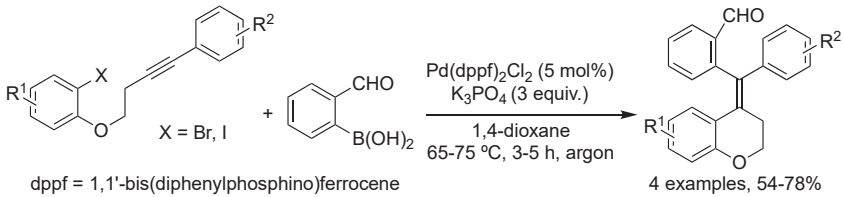
Scheme 36

Nickel-catalyzed *syn*-selective aryl nickelation and cyclization of 2-propargyloxy benzaldehydes with arylboronic acids provides 3-benzylidenechroman-4-ols while using 2-propargyloxychalcone-type compounds as starting materials, a series of 4-(acetylmethyl/benzoylmethyl)-3-benzylidenechromans are obtained (18JOC15361). Phenol-derived allylic azides bearing a pendant trichloroacetimidate underwent tandem allylic azide rearrangement/Friedel–Crafts alkylation in the presence of a catalytic amount of $AgSbF_6$ to give 3-azido-4-vinylchromans (Scheme 37) (18JA1211, 18SL1537).

Asymmetric cascade reactions of phenols with (*E*)-3-aryl-2-nitroallylic acetates in the presence of a bifunctional thiourea catalyst provided a synthesis of 4-aryl-3-nitrochromans. Replacing phenols by 4-hydroxyindole, several 4-aryl-3-nitropyrrolo[2,3-*h*]chromans were formed (18OL2190). Enantioselective synthesis of 4-amino-substituted 2-aryl-3-hydroxychromans was achieved in a multistep strategy starting from chalcones. It involved (1) Corey–Bakshi–Shibata reduction of the enone, (2) Sharpless asymmetric epoxidation of the formed allylic alcohol, (3) protection of the epoxy alcohols, (4) regioselective epoxide opening with various amines using catalytic europium(III) triflate, (5) orthogonal protection of the resulting free alcohols, (6) tandem deprotection/intramolecular nucleophilic aromatic substitution to provide the benzopyran core (18S4796). Palladium-catalyzed domino carbopalladation/cross-coupling reactions of 1-halo-2-[(4-arylbut-3-yn-1-yl)oxy]benzenes with 2-formylphenylboronic acid in the presence of K_3PO_4 in 1,4-dioxane gives access to (*Z*)-2-[chroman-4-ylidene(aryl)methyl]benzaldehydes (Scheme 38) (18JOC8139).



Scheme 37



Scheme 38

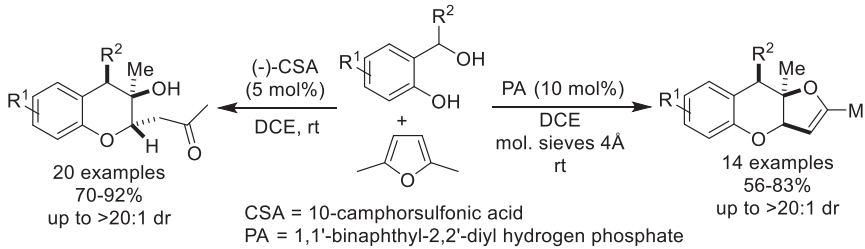
Diversely substituted chromans arise from IED-hDA reactions of salicylaldehyde acetal-derived oxocarbenium ions with vinyl ethers using a catalytic amount of a chiral pentacarboxycyclopentadiene Brønsted acid in benzene at room temperature (18JA3523) and from formal [3 + 3] cycloaddition reactions of benzo[*c*]oxepines with electron-rich phenols in the presence of potassium *t*-butoxide in DMSO (18JOC3409).

Dearomative [4 + 2] cycloaddition reactions of *o*-quinone methides, derived from *o*-hydroxybenzyl alcohols, with 2,5-dimethylfuran in the presence of (−)-10--camphorsulfonic acid [(−)-CSA)] deliver polyfunctionalized chroman-2-ols while 1,1′-binaphthyl-2,2′-diyl hydrogen phosphate (PA) in combination with 4Å molecular sieves furnishes furano[3,2-*b*]chromans (Scheme 39) (18OL6069).

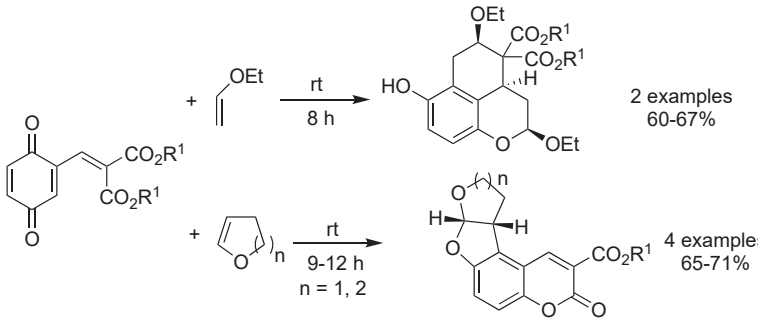
The biomimetic syntheses of the natural chroman-type hyperjapons F−I involve an intermolecular hDA of norflavesone with the appropriate monoterpene building blocks sabinene, α-pinene, and β-pinene (18AJC649). Organocatalytic reductive coupling of chiral 3-aryl-2-formylpropa-1,1-dicarboxylates with cyclic 1,3-diketones and Hantzsch ester followed by a Lewis-acid-catalyzed annulative ring-opening reaction was applied to the stereoselective synthesis of tetrahydrochroman-type compounds, as major products (18JOC9795).

Several dihydrobenzo[*c*]chromans were obtained via intramolecular [4 + 2] cycloaddition reactions of 1-allyloxy-2-(2-cyanobuta-1,3-dien-1-yl)benzenes in refluxing toluene (18OL4566). The tricyclic dihydrobenzo[*c*]chroman core of the palodesangrens was achieved by a three-step transformation, involving DA reaction of chalcones with butadienylarynes, followed by $LiAlH_4$-promoted isomerization and acid-promoted cyclization (18JOC5225). A wide variety of benzo[*de*]chroman-2-ols arise from rhodium(III)-catalyzed cascade C−H activation of benzoylacetonitriles and annulation reactions with diazo compounds (18OL1720) or sulfoxonium ylides (18OL2160). *p*-Benzoquinone alkenyl-dicarboxylates undergo several regioselective cycloaddition reactions at room temperature: (1) with ethyl vinyl ether to afford tetrahydrobenzo[*de*]chromans; (2) with dihydrofuran to give furo-furan-fused coumarins, and (3) with dihydropyran to form pyran-furan-fused coumarins (Scheme 40) (18JOC75).

The synthesis of polyfunctionalized cyclopropa[*b*]chromans can be accomplished via tandem oxa-Michael addition and Michael addition reactions of 2-hydroxychalcones with 2-aroyl-1-chlorocyclopropacarboxylates using cesium carbonate as base in DMF at room temperature (18T1486). Under a combination of



Scheme 39

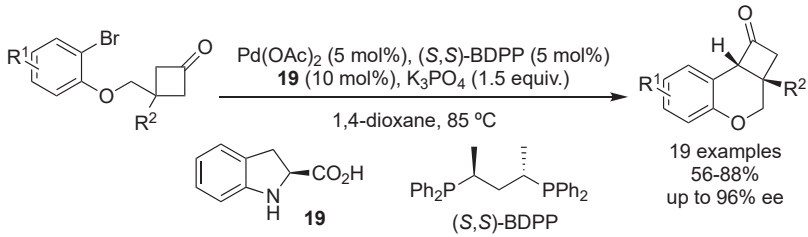


Scheme 40

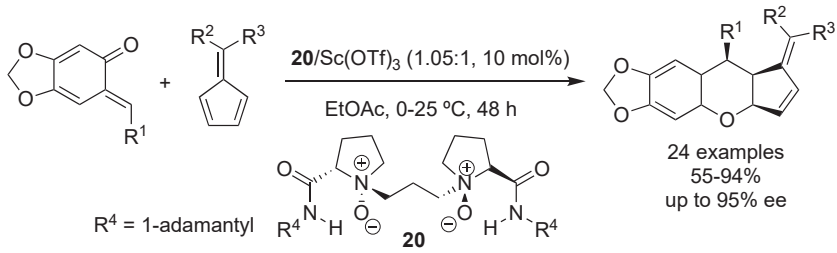
palladium(II) and chiral amine catalytic systems and (2*S*,4*S*)-2,4-bis(diphenylphosphino)pentane [(*S*,*S*)-BDPP] as ligand, enantioselective intramolecular α-arylation of (3-oxocyclobutyl)methyl 2-bromoaryl ethers delivers 3-oxocyclobuta[*c*]chromans in moderate to good yields with high enantioselectivity (Scheme 41) (18AGE2707).

The synthesis of cyclopenta[*b*]chromans is accomplished via palladium(II)-catalyzed conjugate addition of *o*-quinone methides, formed *in situ* from *o*-hydroxybenzhydryl alcohols, with cyclic β-keto esters in toluene at room temperature (18AGE14736). TFA-promoted cascade reactions of 6-(4-alkoxyaryl)-1-(2-hydroxyaryl)hex-5-yn-1-ols in the presence of Et_3SiH in dichloromethane at 0 °C provides cyclopenta[*c*]chromans with a 2,3-*syn* and 3,4-*syn* motif and bearing aryl groups at C-2 of the chroman unit (18EJO1785). Various cyclopentene-fused chromans are obtained through IED oxa-DA reactions of *o*-quinone methides with fulvenes mediated by a chiral *N*,*N*′-dioxide/Sc(III) complex as catalyst and ethyl acetate as solvent (Scheme 42) (18CC74).

Examples of pyrrolidino[2,3-*b*]chromans arise through formal [4 + 2] cycloaddition reactions of *N*-substituted 4-arylbut-3-ynamines with *o*-hydroxybenzhydryl alcohols promoted by a dual catalytic system formed from a gold complex and $BF_3 \cdot OEt_2$ in dichloromethane at room temperature (18EJO3957). Multistep cascade reactions of 2-(3-carboxymethylprop-2-en-1-yl)salicylaldehydes with primary amines, aliphatic isocyanides, and acetic acid derivatives followed by treatment with zinc bromide in DCE under microwave conditions led to pyrrolidino[3,2-*c*]chromans. A couple of



Scheme 41

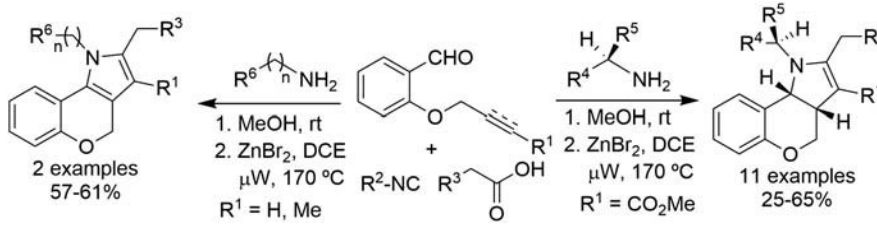


Scheme 42

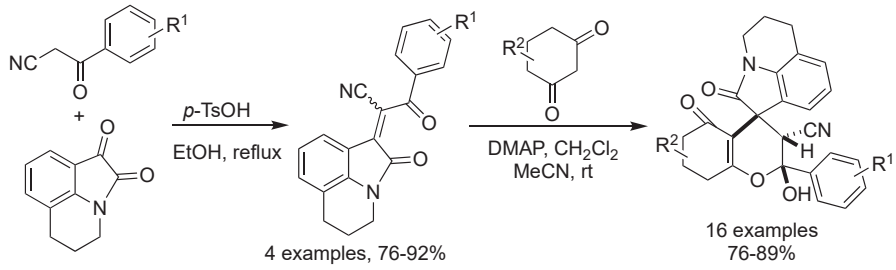
pyrrolo[3,2-*c*]chromenes were obtained when propargyloxy salicylaldehydes were used as starting materials (Scheme 43) (18OL836). Photochemical reactions of 2-(pent-4-en-1-yloxy)benzene-1-carbonitrile/1-carboxylates in methanol afforded cyclobut-2-ene[*i*]tetrahydrochromans in moderate to good yields (18JOC3069).

O-Allylated julolidines, formed through alkylation of julolidines (derivatives of salicylaldehyde) with allyl bromides in the presence of NaH, underwent intramolecular 1,3-dipolar cycloaddition reactions with *N*-substituted α-amino acids (including cyclic ones) mediated by heterogenous $MgSiO_3$ nanoparticles to give a series of pyrrolidine-fused chroman-type compounds (18SC2485). Knoevenagel condensation of 5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-1,2-dione with benzoylacetonitriles promoted by *p*-TsOH in refluxing ethanol gave 3-[aroyl(cyano)methylidene]oxindoles, which react with cyclic 1,3-diketones using DMAP in a 1:4 mixture of dichloromethane:acetonitrile to provide spiroindolin-2-one chroman-2-ol-type compounds (Scheme 44). The one-pot, three-component reaction is also applied to the synthesis of the latest compounds, starting from 5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinoline-1,2-dione, benzoylacetonitriles, and cyclic 1,3-diketones in refluxing ethanol with DMAP (18JHC226). Three-component reactions of substituted isatins with cyclic 1,3-dicarbonyls and 4-hydroxycoumarins mediated by molecular iodine provided spiroindolin-2-one coumarin-fused chroman-type compounds, in moderate to good yields (18T955).

The dioxabenzobicyclo[3.2.1]octane core, a structure found in some natural and bioactive compounds bearing a chromane moiety, can be prepared by a $Cu(OTf)_2$-catalyzed Prins reaction of salicylaldehydes with 2-aryl substituted allylic alcohols (18JOC12897). Highly strained bridged methanodibenzo[*d*,*g*][1,3]dioxocins are



Scheme 43



Scheme 44

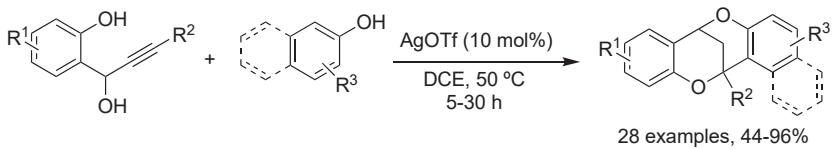
prepared through tandem reactions of 2-hydroxychalcones with phenols/naphthols promoted by a cationic-lanthanide complex $\{[Yb(CH_3CN)_9][(AlCl_4)_3]\cdot CH_3CN\}$ in refluxing chlorobenzene (18T4211). Various examples of methanodibenzo[*b*,*f*][1,5] dioxocins arise from cascade reactions of 2-hydroxycinnamaldehydes with acylphloroglucinols mediated by ethylenediamine diacetate (EDDA) in a 5:1 mixture of toluene: 1,4-dioxane (18OL546) and of alkynyl *o*-quinone methides, generated *in situ* from 1-(2-hydroxyaryl)prop-2-yn-1-ols, with electron-rich phenols in the presence of AgOTf in DCE (Scheme 45) (18OL4371).

Iron(III) chloride−catalyzed cascade reactions of indoles with 2-hydroxychalcones in acetonitrile give a wide variety of pentacyclic indole-bridged chromans (18OL3451). The synthesis of polycyclic indole−bridged spiroindolin-2-one chromans is accomplished through a diastereoselective Michael addition/condensation/Friedel−Crafts alkylation cascade of indol-3-yl-substituted indolin-2-ones with 2-hydroxychalcones promoted by trifluoromethanesulfonic acid in acetonitrile (Scheme 46) (18JOC3679). Other derivatives arise from a two-step sequence starting from indol-3-yl-substituted indolin-2-ones and cinnamaldehydes (18OL6682).

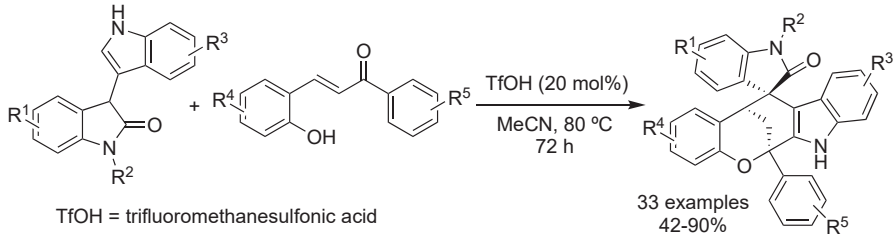
6.4.2.3 [2]Benzopyrans and Dihydro[2]benzopyrans

6.4.2.3.1 Isochromenes and Isochromans

One-pot synthesis of 4-borylated 1*H*-isochromenes is achieved through oxyboration of 2-alkynyl benzyl alcohols and subsequent intramolecular cross-coupling reactions promoted by a gold(I) catalyst (18JOC11204). A similar gold(I) catalyst is used in the synthesis of polysubstituted 1*H*-isochromenes via oxidative cyclizations of *o*-(alkynyl)aryl



Scheme 45

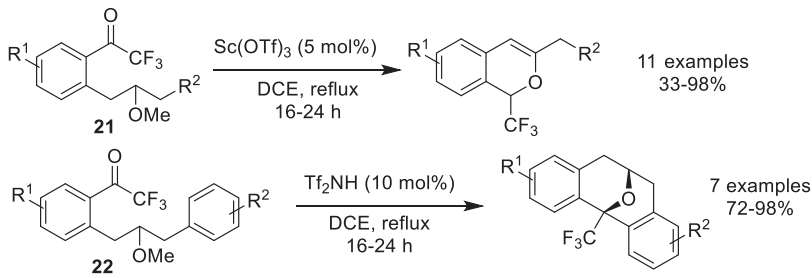


Scheme 46

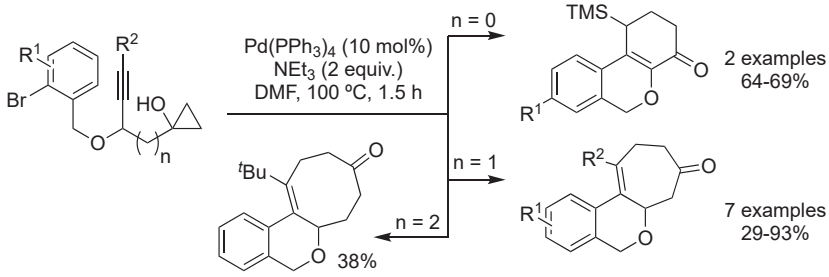
propargyl ether derivatives in the presence of 8-methylquinoline *N*-oxide as oxidant and THF as solvent, at room temperature (18OL5461). CF_3-substituted polycyclic skeletons **21** and **22** undergo divergent transformations by changing the acid catalyst: under $Sc(OTf)_3$ catalysis, selective activation of carbonyl oxygen occurs to give CF_3-substituted isochromene derivatives; using Tf_2NH, activation of the ether oxygen takes place to produce CF_3-substituted bicyclo[3.3.1]nonane derivatives (Scheme 47) (18CC6927).

A series of 1-alkylidene-3-alkoxybenzo[*de*]isochromene derivatives arise from regioselective gold(I), $SIPrAuNTf_2$, catalyzed addition/6-*exo*-*dig* cyclization reactions of 8-[(alkyl/aryl)ethynyl]-1-naphthaldehyde derivatives with aliphatic alcohols in DCE at room temperature (18OL954). The commercially available gold(I) catalyst, JohnphosAu(CH_3CN)SbF_6, mediates intramolecular bicyclization reactions of 1-(1-hydroxyalk-1-ylaryl)-2-(2-azidoaryl)ethynes in DCE to afford indolo[*c*]isochromenes, in moderate to good yields (18OL2733). Intramolecular *trans*-carbocarbonation of internal alkynes bearing a cyclopropanol and 2-bromobenzyloxy moieties undergo cascade formal *anti*-carbopalladation/cyclopropanol ring-opening reactions to give various six-, seven- and eight-membered ring-fused isochromenes (Scheme 48) (18OL7266).

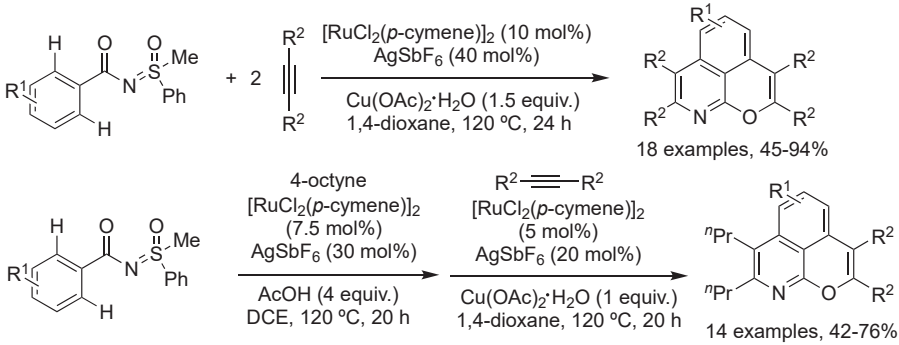
A wide range of pyridino[4,3,2-*ij*]isochromenes were synthesized through rhodium(III)-catalyzed annulation of ethyl benzimidates with α-aroyl sulfur ylides carried out in the presence of copper(II) acetate and sodium acetate in DCE at 120 °C (18OL1396) and one-pot ruthenium(II)-promoted annulation reactions of *N*-(4-benzoyl)methylphenyl sulfoximines with two equivalents of symmetrical alkynes, in a two-step strategy (Scheme 49) (18OL5144).



Scheme 47

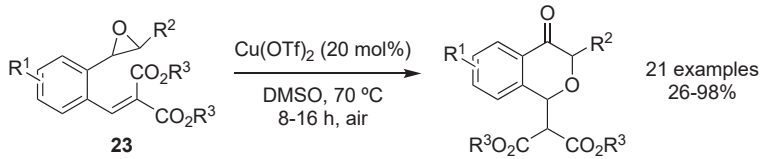


Scheme 48



Scheme 49

Iron(II) triflate−mediated oxa-Pictet–Spengler reactions of 2-arylethanols with aliphatic or aromatic aldehydes give 1-alkyl/1-arylisochromans and with ketones or acetals provide 1,1-disubstituted isochromans (18T7040). Diastereoselective synthesis of 3,3,4-trisubstituted isochromans is accomplished through $Cu(OTf)_2$-mediated cyclization reactions of 3-(2-hydroxymethylaryl)prop-2-en-1-ones with aryldiazoacetates in refluxing toluene (18CC12650). The same copper(II) catalyst is used in the synthesis of isochroman-4-ones through a one-pot protocol, involving oxidative ring opening of epoxides **23** in DMSO and subsequent oxa-Michael addition reactions (Scheme 50) (18EJO926). Direct intramolecular dehydrogenative cyclization of 2-cinnamoylbenzoic acids using a Pd/C catalytic system provides a range of 3-benzylidene-3*H*-isochroman-1,4-diones, in high yields (18CC7774).



23

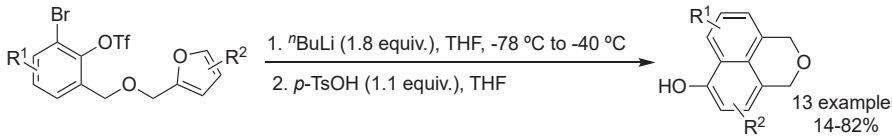
Scheme 50

Various examples of 1-alkyl/1-aryl-6-fluorohexahydro-1*H*-isochromans were synthesized through reaction of aliphatic/aromatic aldehydes with (*E*)-octa-3,7-dien-1-ol carried out in the presence of $BF_3 \cdot Et_2O$ and trimethylsilyl chloride (TMSCl) in dichloromethane, via Prins cyclization followed by diene fluorination (18H(96) 1363). Intramolecular DA reactions of 2-bromo-6-[(furan-2-ylmethoxy)methyl]aryl trifluoromethanesulfonate in the presence of *n*-butyllithium in THF and subsequent treatment with *p*-TsOH gives 1,3-dihydrobenzo[*de*]isochromen-6-ols, in moderate to good yields (Scheme 51) (18JOC4871).

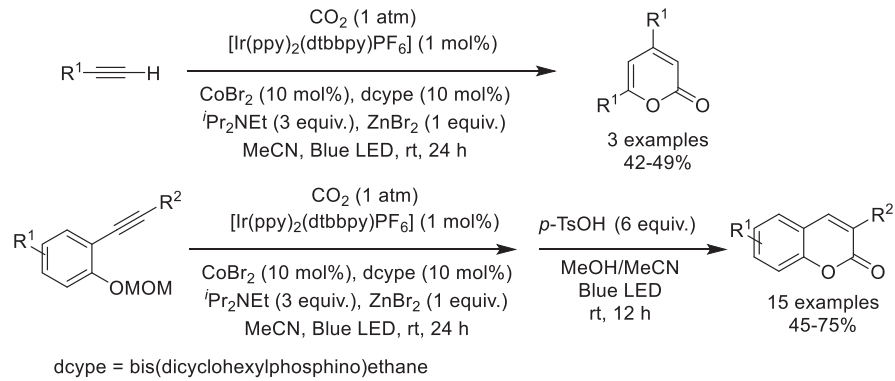
6.4.2.4 Pyranones

Platinum(II)-mediated desaturation processes were used to convert various ketones into their conjugated α,β-unsaturated counterparts. This protocol was applied to the synthesis of 2*H*-pyran-2-ones, 2*H*-chromen-2-ones, and 4*H*-chromen-4-ones (18AGE16205).

Several examples of 4,6-disubstituted 2*H*-pyran-2-ones were prepared under Ir/Co dual catalysis and visible-light-assisted hydrocarboxylation and intermolecular [2 + 2 + 2] cycloaddition reactions of two equivalents of aliphatic terminal alkynes and CO_2 at ambient pressure and temperature. Under the same conditions, followed by treatment with *p*-TsOH under blue LED irradiation, 1-(2-methoxymethoxy)arylethynes provided 3-substituted coumarins (Scheme 52) (18JA5257).



Scheme 51

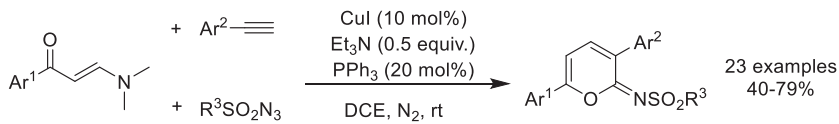


Scheme 52

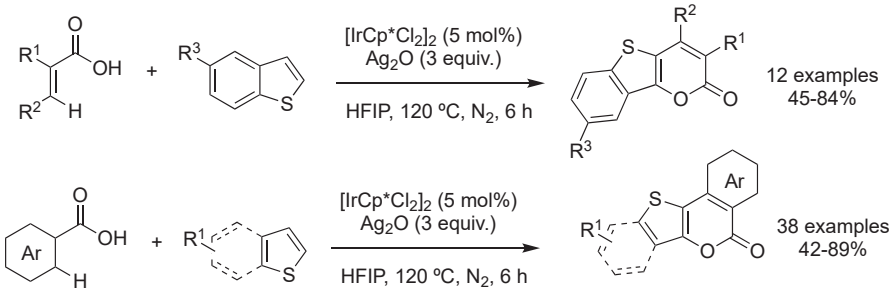
The synthesis of 6-aryl-4-trifluoromethyl-2*H*-pyran-2-ones can be accomplished by self-condensation reactions of 5-aryl-3-(trifluoromethyl)-5-methoxypenta-2,4-dienenitriles in the presence of zinc bromide, water, and HCl, while 6-aryl-4-carboxyethyl-2*H*-pyran-2-ones arise from self-condensation of ethyl 4-aryl-2-(cyanomethylene)-4-methoxybut-3-enoates in the presence of water and hydrochloric acid (18TL121). Silver-promoted 6-*endo-dig* cyclization reactions of *t*-butyl 5-aryl-2-phenylpent-2-en-4-ynoates in methanol at room temperature deliver 6-aryl-4-hydroxy-1-phenyl-2*H*-pyran-2-ones (18OL7455). Various 6-substituted or 4,6-disubstituted 5-(3-oxoalkyl)-2*H*-pyran-2-ones are provided by palladium(II)-catalyzed one-pot tandem 6-*endo*-cyclization/alkylation reactions of 5-substituted or 3,5-disubstituted pent-2-en-4-ynoates with allylic alcohols in cyclohexanone under air (18JOC13414). It is through an alkynylation−Michael addition−cyclocondensation reactions sequence that (hetero)aroyl chlorides react with terminal aromatic alkynes in the presence of $PdCl_2(PPh_3)_2$, CuI, Et_3N in 1,4-dioxane followed by the addition of dialkyl malonates in basic conditions (sodium carbonate) to give alkyl 4-aryl-6-(hetero)aryl-2*H*-pyran-2-one-3-carboxylates (18S2741). A wide range of 3,6-disubstituted 2-imino-2*H*-pyran-2-ones arise from three component reactions of arylacetylenes with enaminones and sulfonyl azides, through a cascade process involving copper-catalyzed alkyne−azide cycloaddition (CuAAC), Michael addition of metalated ketenimine, elimination and 6π electrocyclization reactions (Scheme 53) (18CC13953).

Cycloalkanone-fused 4-trifluoromethyl-2*H*-pyran-2-ones are available from Pechmann-type reactions of cyclic 1,3-diones with ethyl 4,4,4-trifluoroacetoacetate in the presence of 2-dimethylaminopyridine (DMAP) in DCE at 120 °C (18OBC9440). Under metal- and additive-free conditions, 2-alkynylquinoline-3-carbaldehydes underwent intramolecular 6-*endo-dig* cyclization and oxidation reactions promoted by *t*-butyl hydroperoxide (TBHP) to form quinolino[3,2-*c*]-2*H*-pyran-2-ones (18TL1019). Iridium(III)-catalyzed one-pot annulation reactions of (benzo)thiophenes with α,β-unsaturated or (hetero)aromatic carboxylic acids in 1,1,1,3,3,3-hexafluoropropan-2-ol (HFIP) at 120 °C provides (benzo)thiophene-fused 2*H*-pyran-2-ones or isocoumarins, respectively (Scheme 54) (18AGE6309). Under dual cooperative catalysis of ytterbium triflate and silver carbonate, cascade reaction sequences of isocyanides with enynones provide a series of pyrrole-fused 2*H*-pyran-2-imines, via double isocyanide insertion and subsequent 5-*endo-dig* cyclization reactions (18CC6412).

Several 3,4-dihydro-2*H*-pyran-2-ones were obtained from RCM of substituted vinyl pent-4-enoate derivatives conducted in the presence of a Grubbs second generation catalyst in toluene at 80 °C (18JOC8655). A highly active diradical cobalt(III) catalyst promotes cycloisomerization of alk-4-ynoic acids to give mainly vinyl



Scheme 53

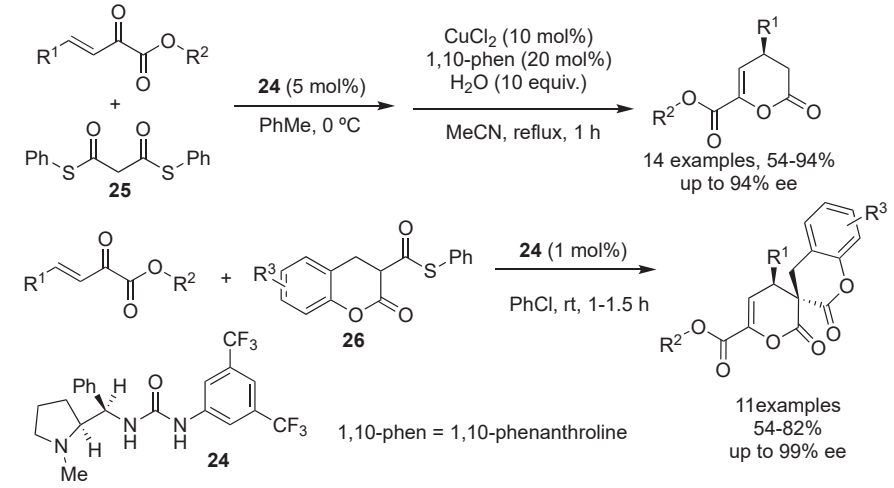


Scheme 54

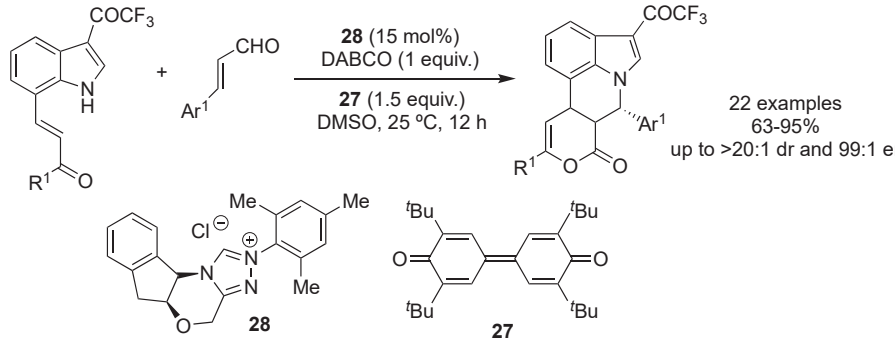
furan-2-ones with 3,4-dihydro-2*H*-pyran-2-ones as minor products (18CC8241). Excellent diastereo- and enantioselectivity is achieved in the synthesis of 3-methylsubstituted-4-(trisubstituted silyl)-3,4-dihydro-2*H*-pyran-2-ones via formal [4 + 2] annulation reactions of α,β-unsaturated aldehydes with β-silyl arylenones mediated by an NHC catalyst (18AGE4594). The same catalyst promotes [4 + 2] hetero-Diels−Alder reactions of α-aroyloxyaldehydes with β,γ-unsaturated α-ketoesters or α,β-unsaturated γ-ketoesters to afford 3,4-dihydro-2*H*-pyran-2-one-6-carboxylates or -4-carboxylates, respectively (18TA355). Further 3,4-dihydro-2*H*-pyran-2-one-6-carboxylates arise from [4 + 2] hDA reactions of aliphatic aldehydes with β,γ-unsaturated α-ketoesters promoted by a substituted proline catalyst [di(*N*,*N*-dimethylbenzylamine)prolinol silyl ether], and subsequent oxidation with pyridinium chlorochromate (PCC) (18TA1591). Another substituted proline catalyst promotes sequential Knoevenagel condensation–Michael addition–hemiacetalization reactions of aldehydes with β-ketoesters or β-ketocarbonitriles, which undergo subsequent oxidation with Dess−Martin periodinane to afford 3,4-dihydro-2*H*-pyran-2-one 5-carboxylates or 5-carbonitriles, respectively (18TA153). Under a low loading of proline-derived urea catalyst **24**, asymmetric Michael addition−lactonization of β,γ-unsaturated α-ketoesters with thioesters **25** provides 3,4-dihydro-2*H*-pyran-2-ones, while with dihydrocoumarin-containing thioesters **26** and subsequent hydrolysis and decarboxylation, spiro-3,4-dihydrocoumarin 3,4-dihydro-2*H*-pyran-2-ones are formed (Scheme 55) (18OL1584).

Organocatalytic cascade reactions of β,γ-unsaturated-α-ketophosphonates with cyclic 1,3-dicarbonyl compounds in chloroform led to cyclic-fused 3,4-dihydro-2*H*-pyran-2-ones in high yields and enantioselectivity (18TL2636). Other enantioselective NHC-catalyzed cascade reactions of 5-alkenylthiazolones with α-chloroaldehydes carried out in the presence of sodium acetate in dichloromethane led to thiazole-fused 3,4-dihydro-2*H*-pyran-2-ones (18S1047), while of 7-(3-oxoprop-1-enyl)indoles with cinnamaldehydes using DABCO and bisquinone **27** as oxidant provides pyrroloquinoline-fused 3,4-dihydro-2*H*-pyran-2-ones (Scheme 56) (18OL6998).

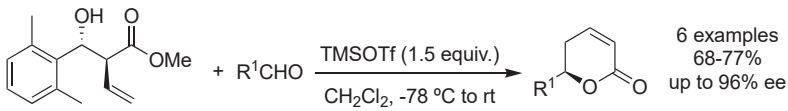
A wide range of 6-substituted 5,6-dihydro-2*H*-pyran-2-ones result directly from vinylogous aldol–lactonization reactions of β,γ-unsaturated esters with aliphatic and



Scheme 55



Scheme 56



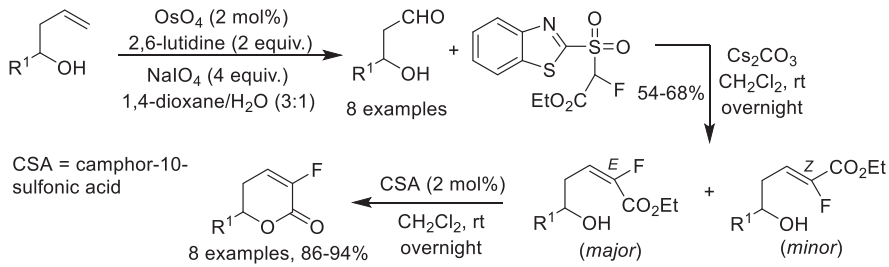
Scheme 57

aromatic aldehydes mediated by a copper(I) catalyst (18JA12270). Enantioselective one-pot synthesis of 5,6-dihydro-2*H*-pyran-2-ones is achieved by 2-*oxonia*-Cope rearrangment of vinylogous aldolation synthons and aldehydes (Scheme 57) (18OL1448). A multi-step protocol is used in the synthesis of 6-substituted 3-fluoro-5,6-dihydro-2*H*-pyran-2-ones. It involves oxidation of homoallylic alcohols with $OsO_4 \cdot NaIO_4$ in the presence of 2,6-lutidine to afford the corresponding β-hydroxyaldehydes;

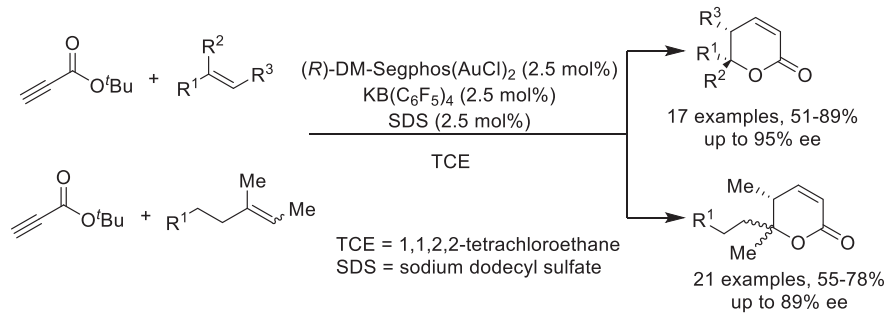
condensation with benzothiazole sulfone using cesium carbonate leading to a mixture of (*E*)- and (*Z*)-isomers of ethyl 2-fluoro-5-hydroxy-5-alkyl/arylpent-2-enoates; and finally lactonization of the mixture with camphor-10-sulfonic acid (CSA) in dichloromethane at room temperature (Scheme 58) (18SL75).

Examples of 4,6-disubstituted 5,6-dihydro-2*H*-pyran-2-one 6-phosphonates arise from NHC-catalyzed enantioselective formal [4 + 2] cycloaddition reactions of α,β-unsaturated aldehydes with α-ketophosphonates, using sodium acetate as base in THF (18CC6040). Several 5,6,6-trisubstituted 5,6-dihydro-2*H*-pyran-2-ones result from gold(I)-catalyzed intermolecular [4 + 2] annulation reactions of *t*-butyl propiolate with trisubstituted alkenes, in the presence of sodium dodecyl sulfate (SDS) and using 1,1,2,2-tetrachloroethane (TCE) as solvent (Scheme 59) (18AGE13130).

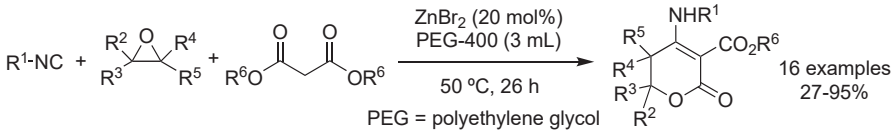
Asymmetric [4 + 2] cycloaddition reactions of β,γ-unsaturated α-ketoesters with cyclobutenones catalyzed by the Lewis acid [*N*,*N*′-dioxide/Yb(III) complex] led to polysubstituted 5,6-dihydro-2*H*-pyran-2-ones in good yields and with excellent diastereo- and enantioselectivities (18CC3375). Further derivatives arise from vinylogous aldol-lactonization cascade reactions of *N*-Boc 3-alkylideneindol-2-ones with trifluoromethyl aromatic ketones (18JOC12440) and of β,γ-unsaturated α-ketoesters with β,γ-unsaturated pyrazole amides (18T3557), promoted by amine-thiourea organocatalysts. Highly functionalized 5,6-dihydro-2*H*-pyran-2-ones are readily available from multicomponent reaction of isocyanides with oxiranes and dialkyl malonates promoted by zinc bromide in PEG-400 at 50 °C (Scheme 60)



Scheme 58



Scheme 59



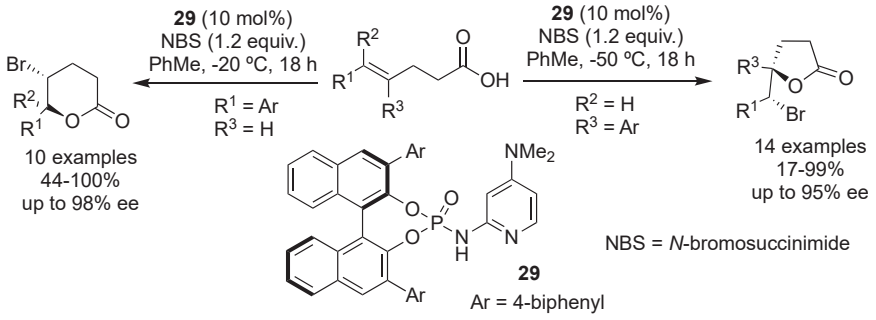
Scheme 60

(18SL894). Various examples of 4-aryl-6-(2-oxo-3-spirooxindolinyl)-5,6-dihydro-2*H*-pyran-2-ones arise from asymmetric NHC-catalyzed [4 + 2] cycloaddition reactions of β-(fluoromethyl)cinnamaldehydes with isatins, in the presence of sodium carbonate in toluene at 0 °C(18CC1567).

Fe(III)-catalyzed hydroallylation of allylic alcohols with 2-aryl-substituted Morita−Baylis−Hillman adducts in the presence of $PhSiH_3$ and ethanol in THF furnishes 3-benzylidenetetrahydropyran-2-ones (18OL1355). Under dual catalysis of a rhodium complex and a chiral phosphoric acid, asymmetric cascade reactions of aliphatic/aromatic aldehydes with allyl boronates and syngas (H_2/CO) provides 5,6-disubstituted tetrahydropyran-2-ones, in moderate to excellent yields and excellent enantioselectivities (18CEJ7626). Chiral pyridyl phosphoramide **29** catalyzes enantioselective bromolactonization of disubstituted 4-arylhex-4-enoic acid derivatives, via 5-*exo* cyclization at −50 °C, to provide 5-bromomethyl-substituted 5-substituted dihydrofuran-2-ones while through 6-*endo* cyclization of 5-arylhex-4-enoic acid derivatives at −20 °C, 5-bromo-6,6-disubstituted tetrahydropyran-2-ones are produced (Scheme 61) (18CEJ18880).

The synthesis of 3-(spiro-1-oxocyclopent-2-yl)tetrahydropyran-2-ones is achieved using a triple catalytic system of iron and copper complexes and an organocatalyst, in the Michael addition of cyclopentan-1-one-2-carboxylates with allylic alcohols and a subsequent DBU-promoted lactonization (18S785).

An aerobic protocol for the synthesis of 2-alkyl-6-hydroxy-2*H*-pyran-3(6*H*)-ones involves selective oxidation of 1-(2-furyl)alkanols, in an Achmatowicz-type ring expansion, promoted by chloroperoxidase in combination with glucose and glucose oxidase, as oxygen-activating biocatalyst (18EJO2717). Further derivatives arise through Achmatowicz rearrangements of 1-(2-furyl)alkanols in the presence of tetrabutylammonium bromide (TBAB) as catalyst and phthaloyl peroxide as oxidant and



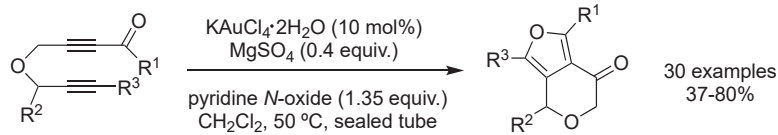
Scheme 61

of under visible-light photocatalytic reactions using $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ as catalyst and 4,5-dichlorophthaloyl peroxide (Cl-PPO) as oxidant (18OBC5566). Gold(III)-catalyzed regioselective oxidation/cycloisomerization of 4-(prop-2-yn-1-yloxy)but-2-yn-1-one derivatives carried out in the presence of magnesium sulfate and pyridine *N*-oxide as oxidant provides mainly furano[3,4-*d*]pyran-3-ones in moderate to good yields (Scheme 62) (18OL4622).

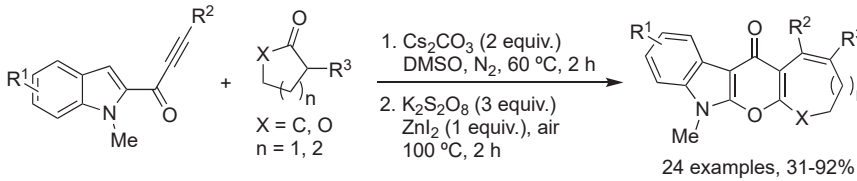
Microwave-assisted reaction of α-oxoketenes, prepared *in situ* by a thermal Wolff rearrangement of 2-diazo-1,3-dicarbonyl compounds, with ketone- and aldehyde-stabilized Wittig ylides leads to functionalized 4*H*-pyran-4-ones, while reacting with ester-stabilized Wittig ylides provides 4*H*-pyranylidenes (18CEJ11110). The synthesis of 4*H*-pyran-4-ones fused to indole units and 7- and 8-membered rings is accomplished via cesium carbonate-mediated C−C bond cleavage and 1,2-acyl migration reactions of 1-(1-methyl-1*H*-indol-2-yl)-3-alkyl/3-arylprop-2-yn-1-ones with cyclic ketones (such as β-ketoesters, β-diketones or α-cyanoketones) followed by zinc iodide/$K_2S_2O_8$-promoted C−O bond formation, in a one-pot strategy (Scheme 63) (18OL6130).

Enantioselective hDA reaction of *N*-substituted 4-benzylidene-2,3-dioxopyrrolidines with Danishefsky's diene promoted by a chiral copper(II) catalyst resulted in 2-spiro(4-benzylidene-2-oxopyrrolidin-3-yl)-2,3-dihydro-4*H*-pyran-4-ones in good yields and with excellent enantioselectivity (18JOC8464).

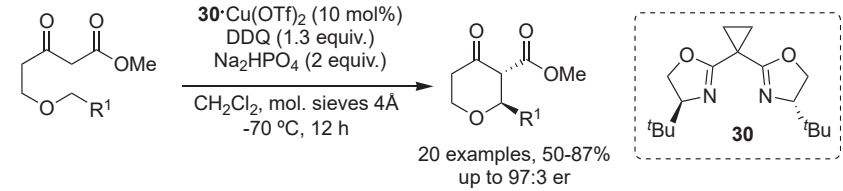
Cross-dehydrogenative coupling catalysis of methyl 5-alkoxy-3-oxopentanoates carried out in the presence of a copper(II)-bisoxazoline complex and DDQ in dichloromethane gives acess to chiral 2-substituted 3-methoxycarbonyltetrahydro-4*H*-pyran-4-ones (Scheme 64) (18JA6212). A series of spiroindolinone tetrahydro-4*H*-pyran-4-ones can be synthesized through one-pot, two-step reactions of isatins with 4-methylpent-3-en-2-one using diethyl amine as catalyst in methanol at room temperature for 24 h, followed by addition of tartaric acid and hydrochloric acid at reflux for 12 h (18SC1033).



Scheme 62



Scheme 63



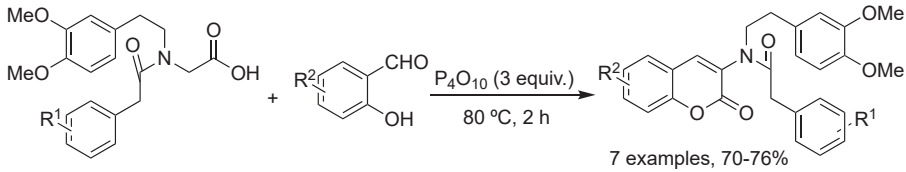
DDQ = 2,3-dichloro-5,6-dicyano-1,4-benzoquinone

Scheme 64

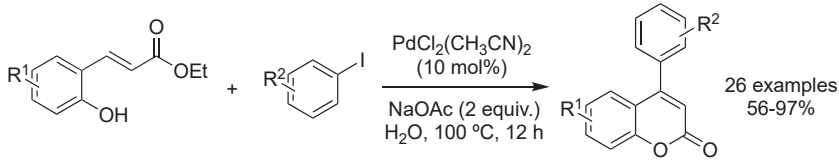
6.4.2.5 Coumarins

One-pot synthesis of 3,4-unsubstituted coumarins is accomplished via Wittig reaction of salicylaldehydes with methyl (triphenylphosphoranylidene)acetate in methanol at room temperature followed by irradiation with a low pressure UV mercury lamp. Changing the ylide to methyl 2-(triphenylphosphoranylidene)propanoate, a series of 3-methylcoumarins are formed (18JHC1183). The synthesis of 3-[*N*-(3,4-dimethoxyphenethyl)]coumarin-3-(2-phenylacetamides) occurs through Perkin reaction of *N*-(3,4-dimethoxyphenethyl)-*N*-(2-phenylacetyl)glycine with salicylaldehydes in the presence of P_4O_{10} at 80 °C (Scheme 65) (18EJO6665).

Under solvent-free conditions, Pechmann reaction of phenols with ethyl acetoacetate promoted by ionic liquid *N*-methylimidazolium sulfomethylsulfonate ([Hmim][$HO_3SCH_2SO_3$]) at 80 °C provides 4-methylcoumarins in moderate to good yields (18SC692). A wide range of 4-arylcoumarins are synthesized through palladium(II)-catalyzed Heck-arylation/cyclization cascade reaction of ethyl 2-hydroxycinnamates with aryl iodides using sodium acetate in water, under aerobic conditions (Scheme 66) (18TL2526) and palladium(II)-catalyzed carbonylative annulation reactions of phenols with terminal alkynes carried out in the presence of benzoquinone as oxidant and $BF_3 \cdot OEt_2$ as additive (18OL3422). Other derivatives



Scheme 65

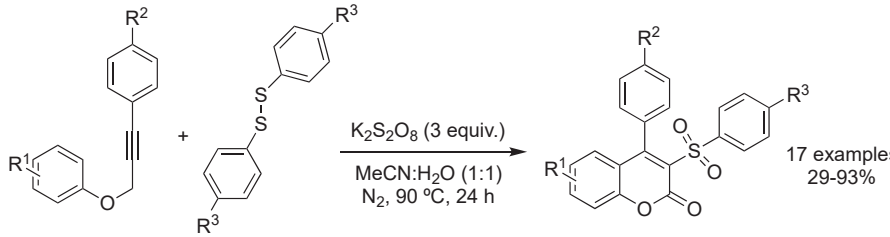


Scheme 66

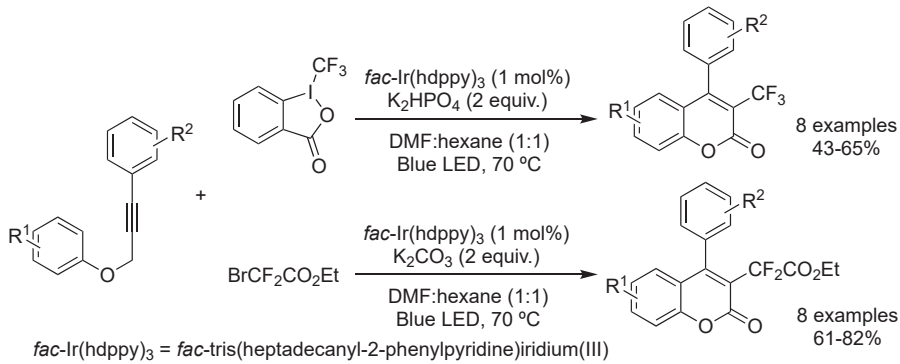
arise from electrochemical dehydrogenative lactonization reaction of diaryl acrylic acids in an undivided cell equipped with a Pt anode and a Pt cathode, using *n*-tetrabutylammonium acetate in a 7:1 mixture of acetonitrile:methanol as supporting electrolyte, under constant current conditions (18OL252).

Microwave-assisted tandem reaction of allyloxy- or 1,1-dimethylallyloxysalicylaldehyde derivatives and the stabilized ylide [(ethoxycarbonyl)methylene]triphenylphosphorane in *N*,*N*-dimethylaniline at 250 °C provides, respectively, 8-allyl- or 8-prenyl-4-substituted coumarins. It involves Claisen rearrangement, Wittig olefination, isomerization and cyclization reactions (18JOC5210). One-pot synthesis of 3-(furan-2-yl)-4-hydroxycoumarins is achieved by reaction of ethyl 3-(2-hydroxyaryl)-3-oxopropanoates with 2,5-dimethoxy-2,5-dihydrofuran conducted in presence of K10 montmorillonite clay heterogeneous catalyst at 80 °C under solvent-free conditions followed by the addition of sodium hydroxide in refluxing ethanol (18T4712). A catalyst-free approach for the synthesis of 4-aryl-3-sulfonylcoumarins uses aryl 3-arylpropiolates with disulfides and potassium persulfate in a 1:1 mixture of acetonitrile:water, via *ipso*-cyclization/1,2-ester migration reactions (Scheme 67) (18T4435).

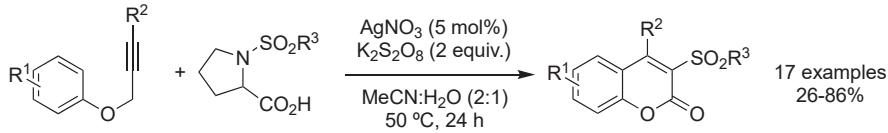
A large variety of 3-acyl-4-arylcoumarins was prepared via visible-light-mediated domino radical addition/cyclization reactions of aryl 3-arylpropiolates with aldehydes promoted by a ruthenium(II) catalyst and *t*-butyl hydroperoxide (TBHP) (18OBC8196), promoted by 2-*t*-butylanthraquinone as catalyst and benzoyl peroxide



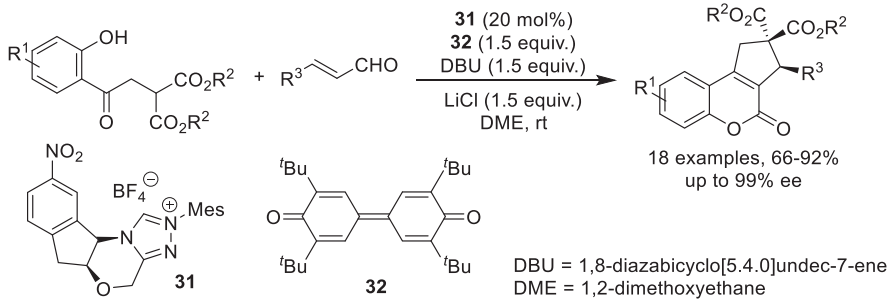
Scheme 67



Scheme 68



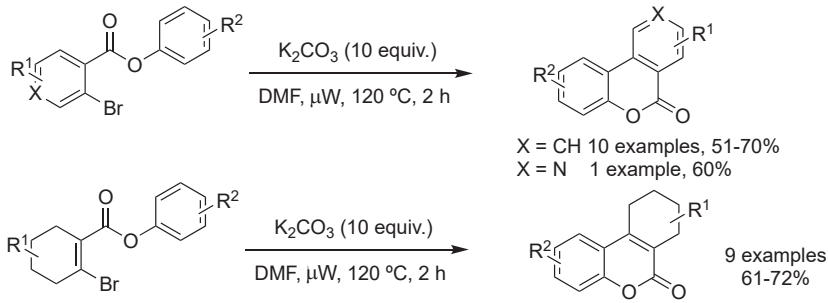
Scheme 69



Scheme 70

(BPO) as oxidant (18JOC1988), and from the reaction of aryl 3-arylpropiolates with acyl chlorides promoted by tris(2-phenylpyridine)iridium(III) [Ir(ppy)$_3$] and 2,6-lutidine in acetonitrile (18TL2038). A similar protocol uses aryl 3-alkyl/3-arylpropiolates and bromoacetonitrile promoted by *fac*-Ir(ppy)$_3$ and lithium carbonate to provide 4-alkyl/4-aryl-3-cyanomethylcoumarins (18OBC5788). Other visible-light photoredox cascade reactions of aryl 3-alkyl/3-arylpropiolates with CF_3SO_2Cl promoted by $Ru(bpy)_3Cl_2$ affords 4-alkyl/4-aryl-3-trifluoromethylcoumarins (18JOC8607), while the reaction of aryl 3-arylpropiolates with Togni's reagent or $BrCF_2CO_2Et$ promoted by *n*-heptadecanyl bound *fac*-Ir(ppy)$_3$ catalyst and a base led to 3-trifluoromethylated or 2-difluoromethylated 4-arylcoumarins, respectively (Scheme 68) (18T7358).

Sulfonyl radicals, generated from α-amino acid sulfonamides, undergo silver-catalyzed reactions with aryl 3-alkyl/3-arylpropiolates in a 2:1 mixture of acetonitrile: water to produce various 4-alkyl/4-aryl-3-sulfonylcoumarins (Scheme 69)



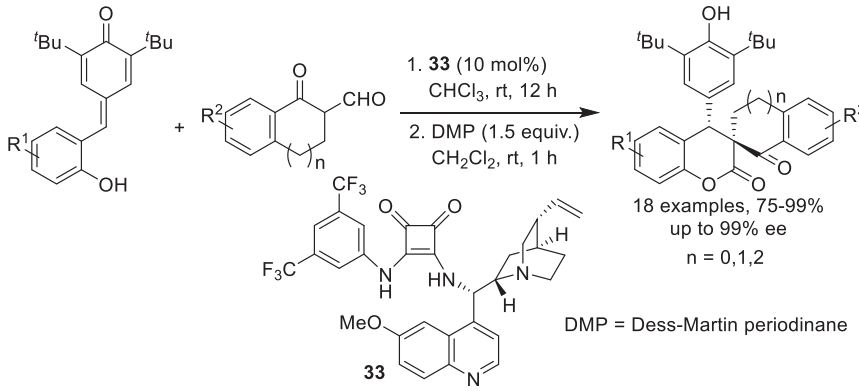
Scheme 71

(18EJO5905). One-pot synthesis of 3-aryl-*N*-arylsulfonyl-2-iminocoumarins is achieved via three-component reactions of salicylaldehydes with arylacetonitriles and aryl sulfonyl chlorides using DABCO as base and 2-methyltetrahydrofuran as solvent (18T1900).

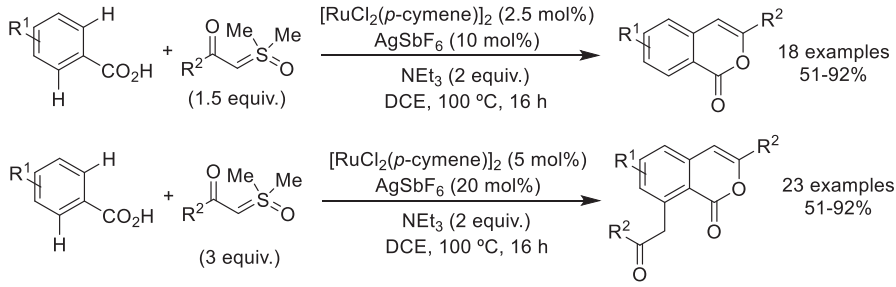
The asymmetric synthesis of cyclopenta[*c*]coumarins is achieved by the NHC-catalyzed reaction of 2′-hydroxyacetophenones bearing malonates at C-2 with enals in the presence of DBU and LiCl in DME (1,2-dimethoxyethane). It involves a Michael/aldol/lactonization/dehydration domino sequence (Scheme 70) (18AGE17100). Microwave-assisted reaction of 4-propargyloxysalicylaldehyde derivatives with ethyl 2-(triphenylphosphoranylidene)acetate in *N*,*N*-diethylaniline at 250 °C give pyrano[2,3-*h*]coumarins in moderate to good yields (18EJO223).

A wide variety of benzo[*c*]coumarin-type compounds arise from electrochemical dehydrogenative lactonization of 2-(hetero)arylbenzoic acids promoted by $LiClO_4$ in acetonitrile (18CEJ6932), or by *n*-Bu_4NBF_4 in a 7:1 mixture of acetonitrile:water (18OL252), carried out in the presence of DDQ, 2,6-lutidine, *n*-Bu_4NClO_4, and HFIP (18S2924), and in an oxidant-free approach using sodium hydroxide in a 10:1 mixture of methanol:water (18JOC3200). Further derivatives were produced from visible-light-assisted protocols mediated by (−)-riboflavin as photocatalyt and oxygen as terminal oxidant (18OL1316), and by a combination of 9-mesityl-10-methylacridinium perchlorate and cobaloxamine as catalysts (18JOC3582). Under microwave irradiation, aryl 2-bromobenzoates and aryl 2-bromocyclohex-1-enecarboxylates undergo cyclization in the presence of potassium carbonate and dimethylformamide at 120 °C to give several benzo[*c*]coumarins (Scheme 71) (18JOC4140).

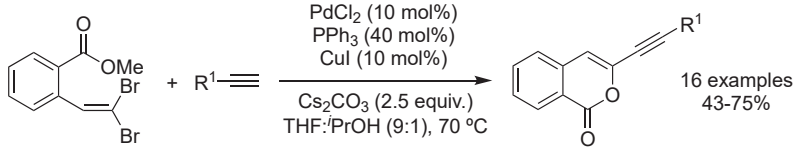
A series of polysubstituted 4-aryl-3,4-dihydrocoumarins was obtained from the reaction of phenols with cinnamic acids mediated by HFIP and HCl, generated *in situ* from acetyl chloride (18EJO306). Stereoselective synthesis of *cis*-3,4-diaryl-3,4-dihydrocoumarins is achieved via phosphoric acid−catalyzed formal [4 + 2] cycloaddition reactions of *o*-quinone methides, generated *in situ* from 2-hydroxybenzhydryl alcohols, with aryl acetaldehydes followed by the addition of PCC (18OL4769). Another asymmetric formal [4 + 2] cycloaddition reaction involved the reaction of *o*-quinone methides with β-keto acylpyrazoles and used a



Scheme 72



Scheme 73

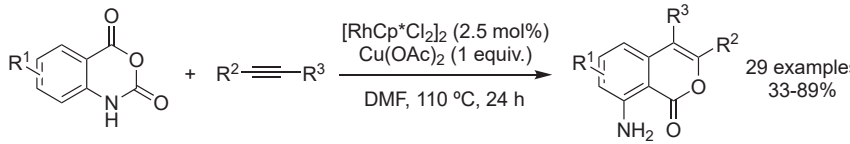


Scheme 74

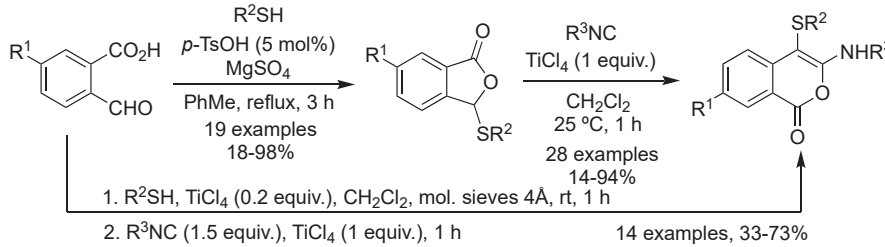
quinine-based chiral squaramide as catalyst to afford *trans*-3,4-disubstituted 3,4-dihydrocoumarins (18JOC4221). A wide range of 3,3,4-trisubstituted 3,4-dihydrocoumarins was obtained from diastereoselective domino reactions of *o*-hydroxyaryl *p*-quinone methides with azlactones mediated by diphenyl hydrogen phosphate (18S1307) and a chiral phosphoric acid catalyst (18JOC364). The same type of *p*-quinone methides underwent asymmetric 1,6-addition/acetalization reactions with 1-oxotetralin-2-carbaldehyde promoted by a chiral squaramide **33** giving access to spiro 3,4-dihydrocoumarins (Scheme 72) (18JOC2714).

Regiodivergent [3 + 2] cycloaddition reactions of azomethine ylides with 2-hydroxybenzylidene indane-1,3-diones carried out in the presence of DMAP in chloroform, followed by acetalization and lactonization provides spiroindane-1,3-dione pyrrolidino[2,3-*c*]dihydrocoumarins (18CC9921).

Sulfoxonium ylides underwent C−H activation/annulation reactions with benzoic acids promoted by ruthenium(II)/$AgSbF_6$ catalyst (Scheme 73) (18CEJ16548) and with *N*-methoxybenzamides mediated by a rhodium(III) complex (18CC670), giving access to 3-substituted isocoumarins. The synthesis of 3-alkynylisocoumarins can be achieved through palladium(II)/copper(I)-catalyzed tandem Sonogashira/lactonization reactions of methyl 2-(2,2-dibromovinyl)benzoate with terminal alkynes carried out in the presence of triphenylphosphine and cesium carbonate in a 9:1 mixture of THF:



Scheme 75

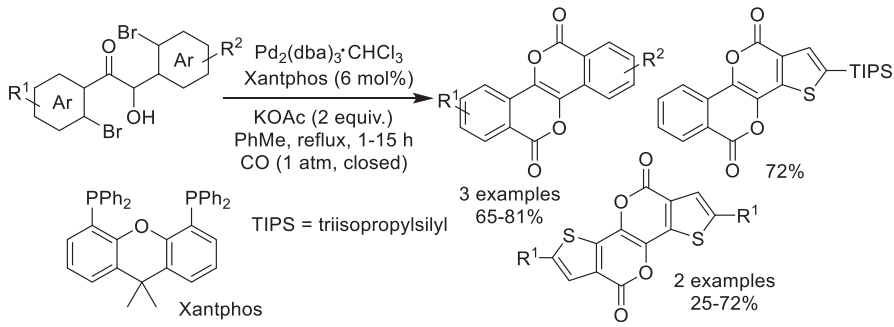


Scheme 76

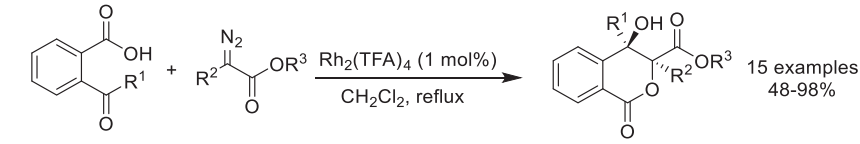
propan-2-ol (Scheme 74) (18TL3151). Various 3-substituted isocoumarin-type compounds arise from cyclization of 2-alkynylbenzoates under dual catalysis of silver triflate and *p*-toluenesulfonic acid (18OBC3213).

Rhodium(I)-catalyzed decarbonylative aerobic oxidation of substituted *o*-naphthoquinones using a phosphine ligand in chlorobenzene or toluene leads to 3,4-disubstituted isocoumarins (18OL942). High yields of 4-iodo-3-substituted isocoumarins are obtained through oxidative iodocyclization of 2-alkynoates promoted by tetrabutylammonium iodide (TBAI) and oxone in a 1:4 mixture of DCE/water (18SC1786). A wide variety of 3,4-disubstituted isocoumarins are prepared through the reactions of benzoic acids with alkynes mediated by cobalt(II) hexafluoroacetylacetonate (18AGE1688) and by an electroxidative process promoted by a ruthenium(II) catalyst (18AGE5818). Other examples arise from the palladium(II)-catalyzed reaction of 2-bromobenzaldehydes with aryl diazoesters (18AGE319) and rhodium(III)-catalyzed decarbonylative annulation reactions of isatoic anhydrides with substituted alkynes (Scheme 75) (18CC11889).

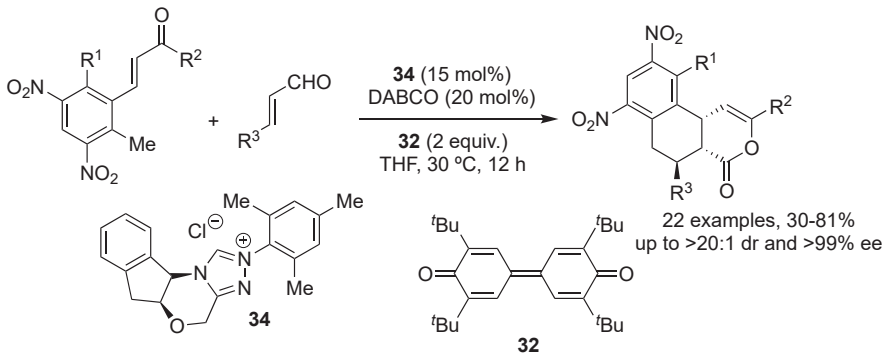
The synthesis of 3-amino-4-sulfanyl isocoumarins is accomplished from the reaction of 3-sulfanyl phthalides, formed from the reaction of 2-formylbenzoic acids with thiols in the presence of *p*-TsOH, with isocyanides mediated by $TiCl_4$ and by a one-pot strategy, starting directly from the reaction of 2-formylbenzoic acids with thiols promoted by $TiCl_4$ and subsequent addition of isocyanides and $TiCl_4$ in dichloromethane at room temperature (Scheme 76) (18S1331). A metal-free protocol for the synthesis of 4-fluoroalkylselenolated isocoumarins involves 6-*endo-dig* cyclization



Scheme 77



Scheme 78

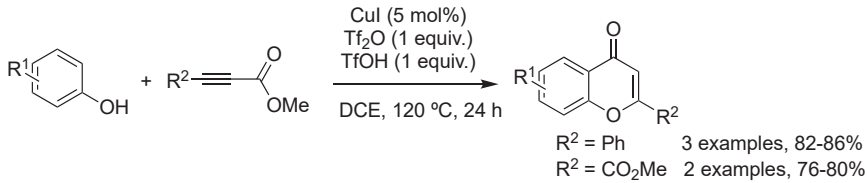


Scheme 79

reaction of methyl 2-alkynylbenzoates in the presence of RCF_2SeCl (R = F, H, CO_2Me, $PhSO_2$) derivatives in THF (18OL56).

Rhodium(III)-catalyzed tandem reactions of primary benzamides with cyclic 2-diazo-1,3-diketones carried out in the presence of cesium acetate in DCE furnishes 1-oxocyclohexa[3,2-*c*]isocoumarins in moderate to good yields (18T7082). A variety of benzothieno[3,2-*c*]isocoumarins were obtained from Ir(III)-catalyzed oxidative annulation reactions of arylglyoxylic acids with benzo[*b*]thiophenes, using Ag_2O as oxidant, 1-adamantanecarboxylic acid as additive and HFIP as solvent (18OL3001). Palladium(II)-catalyzed double carbonylative cyclization of 1,2-bis[2-bromo(hetero)aryl)]-2-hydroxyethanones in the presence of Xantphos as ligand in refluxing toluene provided several examples of (hetero)aryl-fused isocoumarins (Scheme 77) (18OL7442).

Cobalt(II) hexafluoroacetylacetonate mediates coupling reactions of benzoic acids with terminal alkenes using $(TMS)_2NH$ as base, oxygen as oxidant and cesium sulfate as cooxidant to afford 3-substituted 3,4-dihydroisocoumarins (18AGE1688). Further derivatives arise through photoinduced regioselective lactonization of 2-iodobenzoic acids with terminal alkenes in the presence of sodium hydrogencarbonate in DMSO (18SL131) and lactonization reactions of 2-(2-aryl-2-hydroxyethyl)benzonitriles in a 1:1 mixture of methanol:conc. HCl at 70 °C for five hours (18S2617). The synthesis of 4-(2-hydroxyethyl)-3,4-dihydroisocoumarins is achieved through palladium(II)-catalyzed Heck−Matsuda arylation reactions of methyl benzoates bearing diazonium salts at C-2 with 2,5-dihydrofuran and a subsequent $NaBH_4$-reduction/lactonization sequence (18CEJ17691). A chiral bifunctional dialkyl sulfide catalyst promotes bromolactonization of 2-styrylbenzoic acids in the presence of NBS to give 3-aryl-



Scheme 80

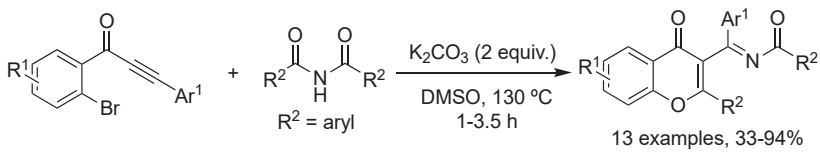
4-bromo-3,4-dihydroisocoumarins in a regio-, diastereo- and enantioselective manner (18CEJ16747). Functionalized 3,4-dihydroisocoumarins result from a $Rh_2(TFA)_4$-catalyzed cascade reaction of 2-acylbenzoic acids with 2-diazoesters in refluxing dichloromethane (Scheme 78) (18OL7585).

A range of cycloalkyl-fused dihydroisocoumarins were synthesized through microwave-assisted nickel(II)-catalyzed C−H coupling reactions of 8-aminoquinoline-derived benzamides with various epoxides in the presence of diglyme at 200 °C (18AGE11797). NHC-catalyzed Michael/Michael/lactonization cascade reactions of 4-(2-methyl-3,5-dimethylphenyl)prop-2-en-1-one derivatives with α,β-unsaturated aldehydes provide benzo[*f*]tetrahydroisocoumarins (Scheme 79) (18OL2952).

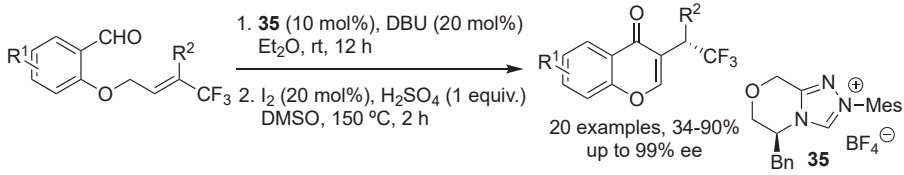
6.4.2.6 Chromones and Chromanones

Under microwave irradiation, the synthesis of 2,3-unsubstituted 4*H*-chromen-4-ones occurs through cyclization of 2-hydroxyaryl enaminones promoted by propylphosphonic anhydride at 90 °C (18SL1087).

The synthesis of a wide variety of 2-aryl-4*H*-chromen-4-ones is achieved in one-pot syntheses via metal-free cascade cyclization/oxidative radical reactions of 1-aryl-3-(2-hydroxyaryl)propargylic alcohols in aqueous HI-air system (18EJO5548); NHC-catalyzed dehydrative acetylation of substituted salicylaldehydes with α-haloketones in a 10:1 mixture of THF:*t*-butanol, followed by intramolecular heterocyclization reactions (18EJO537); carbonylative Sonogashira annulation reactions of 2-iodophenols with arylacetylenes in the presence of $Pd(OAc)_2$ as catalyst and $Mo(CO)_6$ as CO source (18TL2025) or in the presence of $Ni(OAc)_2 \cdot 4H_2O$ as catalyst, triethylamine and two bar of CO (18TL3283). CuI catalyzed the one-pot reaction of phenols with methyl 3-phenylpropynoate or dimethyl butynedioate carried out in the presence of Tf_2O and TfOH in DCE to lead to 2-phenyl-4*H*-chromen-4-ones or methyl 4*H*-chromen-4-one-3-carboxylates, respectively (Scheme 80) (18OL1893).



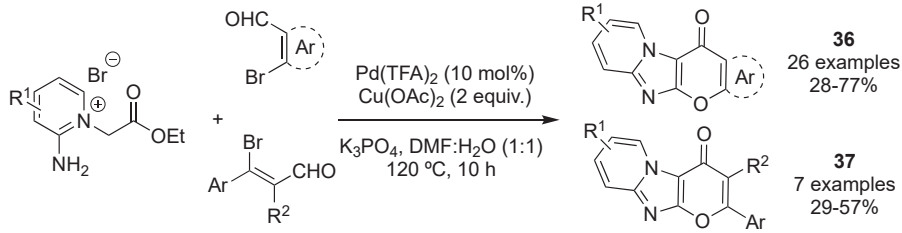
Scheme 81



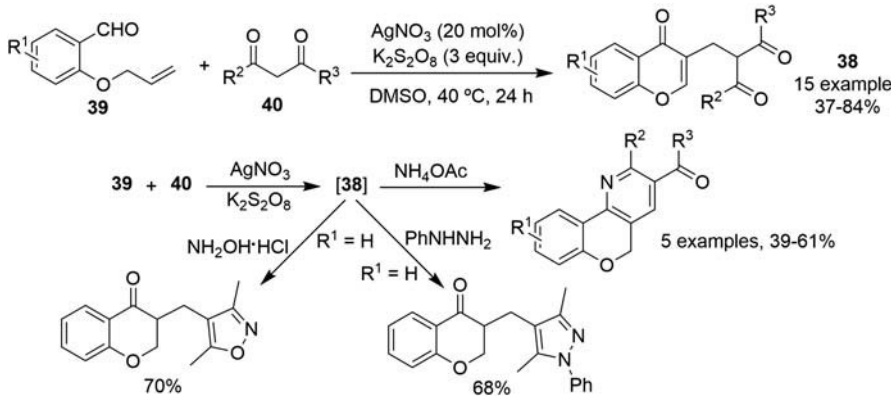
Scheme 82

The selective fluorination of 3-aryl-1-(2-hydroxyaryl)propa-1,3-diones with Selectfluor and a small amount of acetonitrile at room temperature, followed by cyclization and dehydration in the presence of a trace amount of concentrated sulfuric acid gives access to 2-aryl-3-fluoro-4*H*-chromen-4-ones (18OBC2479). Examples of 2-aryl-3-iodo-4*H*-chromen-4-ones arise from dehydrogenative cyclization and α-iodination of 2′-hydroxychalcones mediated by molecular iodine and from 2′-allyloxychalcones, with an initial allyl deprotection step (18SC1299). Domino intermolecular Friedel–Crafts acylation/oxa-Michael addition/demethylation reaction sequences are involved in the synthesis of 2,3-diaryl- and 2-aryl-3-TMS-4*H*-chromen-4-ones starting from 2-methoxyaroyl chlorides and 1,2-diaryl- or 1-aryl-2-TMS-acetylenes, promoted by aluminum tribromide in dichloromethane at −78 °C (18JOC9929). Various 3-enamide substituted 2-aryl-4*H*-chromen-4-ones were selectively prepared from the reaction of 3-aryl-1-(2-bromoaryl)prop-2-yn-1-ones with aromatic imides, carried out in the presence of potassim carbonate in DMSO (Scheme 81) (18CC6192). The synthesis of 3-aryl-2-(ω-carboxyalkyl)-4*H*-chromen-4-ones is achieved via base-catalyzed condensation of 2,4-dihydroxy-substituted deoxybenzoins with cyclic anhydrides, under dual catalysis of triethylamine and DBU in 1,4-dioxane (18EJO5460).

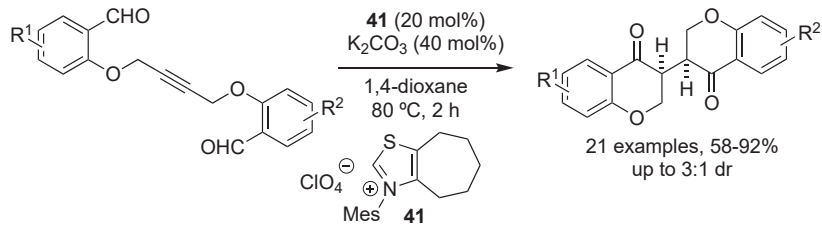
Under an inert atmosphere, silver-catalyzed decarboxylative C–C cross-coupling reactions of 2-hydroxyaryl tertiary enaminones with α-keto acids in 2:1 mixture of DMSO:water at 60 °C provides 3-acyl-4*H*-chromen-4-ones (18EJO6867). Other 2-hydroxyaryl tertiary enaminones undergo cross-coupling reactions with aroyl peroxides in ethanol, at room temperature, to provide 3-acyloxy-4*H*-chromen-4-ones, in a catalyst-free protocol (18OL3971). A range of 3-selenocyanato-4*H*-chromen-4-ones were synthesized by a sequential one-pot reaction of 2′-hydroxyacetophenones with *N*,*N*-dimethylformamide dimethyl acetal (DMF-DMA) to deliver 2-hydroxyaryl enaminones which undergo addition with triselenodicyanide, formed in situ from a 3:1 mixture of selenium oxide:malononitrile in DMSO (18SL1215). High yields and



Scheme 83



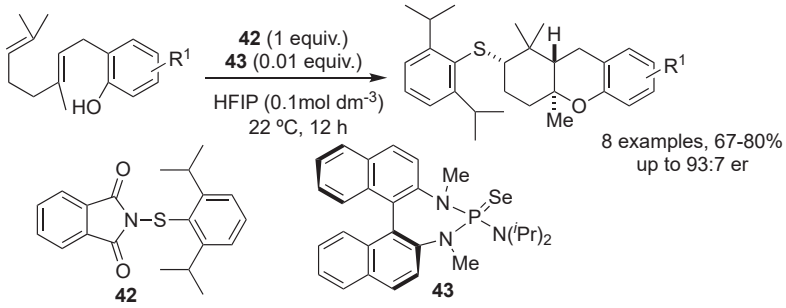
Scheme 84



Scheme 85

enantioselectivity are accomplished in the synthesis of 3-[1-(trifluoromethyl)]alkyl-4*H*-chromen-4-ones through NHC-catalyzed Stetter reactions of trifluoromethyl-substituted 2-allyloxybenzaldehydes using DBU as base in diethyl ether and subsequent treatment with iodine and sulfuric acid in DMSO (Scheme 82) (18OL6012). Copper(II) acetate mediates [4 + 2] annulation reactions of sulfonylacetylenes with salicylic acids in the presence of benzotriazol-1-yloxy tri(dimethylamino) phosphonium hexafluorophosphate (BOP, Castro's reagent) and DMAP in nitromethane, afford 2-alkyl/2-aryl-3-sulfonyl-4*H*-chromen-4-ones (18JOC2361).

Tandem reaction of 3-aryl-1-(2-haloaryl)prop-2-yn-1-ones with indene-1,3-dione carried out in the presence of potassium carbonate in DMSO at 110 °C result in benzocycloheptanone-fused 4*H*-chromen-4-ones (18OL1744). A wide range of imidazo [1,2-*a*]pyridine-fused 4*H*-chromen-4-one-type compounds **36** were obtained from tandem reactions of 2-amino-1-(2-ethoxy-2-oxoethyl)pyridinium salts with 2-bromoarylaldehydes using $Pd(TFA)_2$ as catalyst and $Cu(OAc)_2$ as oxidant (Scheme 83). The sequence involves amidation, Knoevenagel condensation, palladium-catalyzed Wacker-type oxidation, and an intramolecular C—O coupling reaction. This protocol was extended to 3-aryl-3-bromoacrylaldehydes to afford imidazo[1,2-*a*]pyridine-fused 4*H*-pyran-4-ones **37** (Scheme 83) (18JOC8026)



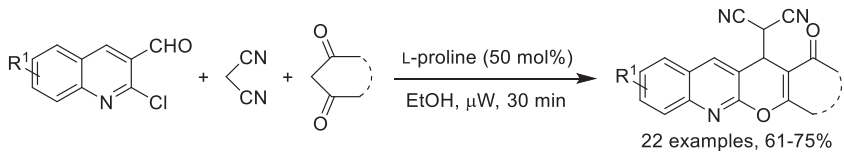
Scheme 86

Annulation of 2′-hydroxy-2-sulfonylacetophenones with ynones in the presence of potassium carbonate and copper(I) iodide in DMSO furnishes 2-(2-oxoalkyl)-3-sulfonyl-4*H*-chroman-4-ones in good yields (18OL1824). Silver nitrate catalyzes radical cascade reactions of 2-allyloxybenzaldehydes with 1,3-dicarbonyl compounds in the presence of $K_2S_2O_8$ to give 4*H*-chroman-4-ones containing 1,3-dicarbonylmethyl moieties at C-3 **38**. In addition, treating the crude reaction mixture with NH_4OAc, $NH_2OH \cdot HCl$, or $PhNHNH_2$ affords, respectively, pyridino[3,2-*c*] chromenes, isoxazole- , and pyrazole-containing 4*H*-chroman-4-ones (Scheme 84) (18OL6157). Other 2-allyloxybenzaldehydes underwent intramolecular trifluoromethylation promoted by CF_3SO_2Na and using $K_2S_2O_8$ as oxidant to provide 3-(2,2,2-trifluoroethyl)-4*H*-chroman-4-ones. Using the same conditions, selective trifluoromethylarylation of 2-(but-3-en-1-oxy)benzaldehydes affords 4-(2,2,2-trifluoroethyl)chromans (18OL6520).

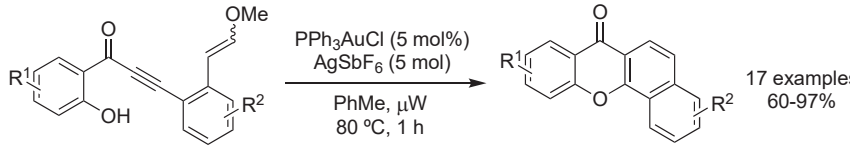
A series of 3,3-bis(4*H*-chroman-4-ones) was obtained through an NHC-catalyzed intramolecular hydroacylation/Stetter reaction cascade reaction of 2,2'-[but-2-yne-1,4-diylbis(oxy)]dibenzaldehydes and potassium carbonate in 1,4-dioxane (Scheme 85) (18OL2676).

6.4.2.7 Xanthenes and Xanthones

Enantioselective sulfenocyclization of 2-geranylphenols promoted by sulfenylation agent **42** and chiral Lewis basic catalyst **43** in HFIP delivers 2-thioarylhexahydro-1*H*-xanthenes in good yields (Scheme 86) (18JA3569). Various 9-aroyl-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-diones can be synthesized through condensation reactions of arylglyoxals with two equivalents of 1,3-diketones



Scheme 87

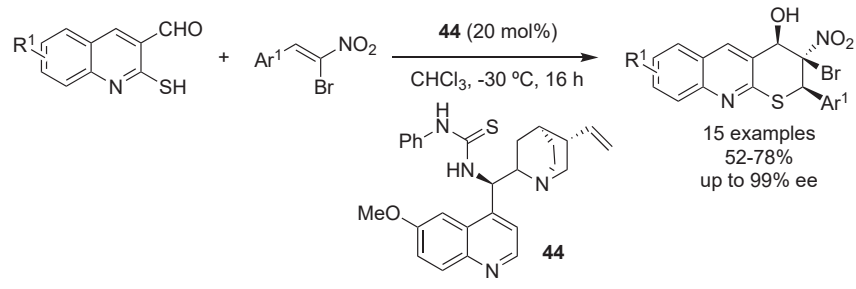


Scheme 88

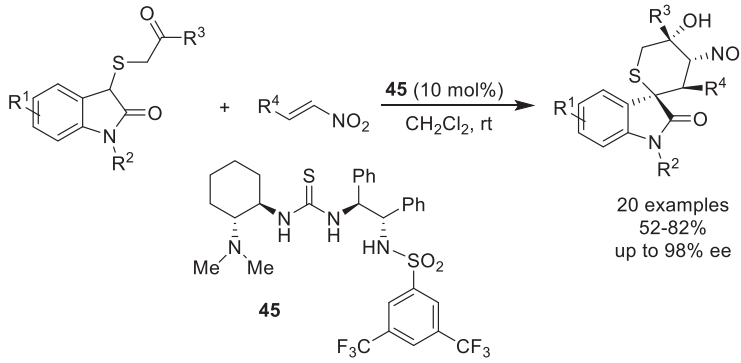
(cyclohexane-1,3-dione or dimedone) and using tetrapropylammonium bromide (TPAB) as catalyst in the presence of a 3:1 mixture of ethanol:water at 70 °C (18JHC1324).

One-pot three-component condensation of salicylaldehydes with active methylene compounds and a carbon-based nucleophile (including 2-naphthol, 1,5-dihydroxynaphthalene, 2,7-dihydroxynaphthalene, 2,3-dihydroxynaphthalene; phenol, indole, and 4-hydroxycoumarin) in the presence of a catalytic amount of $ZrOCl_2 \cdot 8H_2O$ in water at 55 °C delivers xanthene-type compounds (18JHC522). Further derivatives arise from microwave-assisted three-component reaction of 2-chloroquinoline-3-carbaldehydes with malononitrile and 1,3-dicarbonyl compounds carried out in the presence of L-proline in ethanol (Scheme 87) (18H(96)1977). Under solvent-free conditions and with microwave irradiation, condensation of aromatic aldehydes with two equivalents of β-naphthol using benzyltrimethylammonium dichloroiodate (BTADCI) as catalyst furnishes 14-aryl-14*H*-dibenzo[*a*,*j*]xanthenes (18JHC1499). Solvent-free organocatalyzed condensation of 4-hydroxycoumarin with benzaldehydes and 2-aminoprop-1-ene-1,1,3-tricarbonitrile at 90 °C delivers benzo[*c*]xanthenes (18TL3567).

1-[2-(1,2-Dichlorovinyloxy)]arylpenta-2,4-dien-1-ones, generated from the condensation reaction of 2'-(1,2-dichlorovinyloxy)acetophenones with cinnamaldehydes and other 2-substituted prop-2-enal derivatives using potassium carbonate as base and propan-1-ol as solvent at 70 °C, underwent intramolecular cycloaddition reaction and subsequent aromatization by increasing the temperature to 120−140 °C to afford functionalized 9*H*-xanthen-9-ones (18T5715). High yields of 12*H*-benzo[*c*]xanthen-12-ones can be achieved via a microwave-assisted Au(I)-catalyzed Michael addition/6-*endo-trig* cyclization/aromatization cascade annulation reaction



Scheme 89



Scheme 90

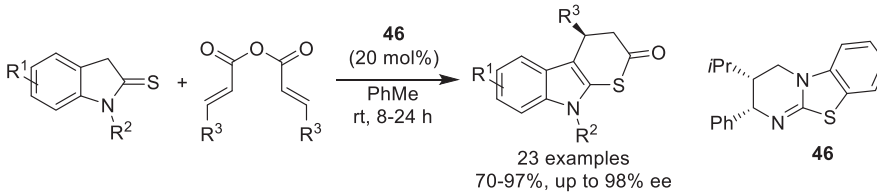
of 1-(2-hydroxyaryl)-3-[2-(2-methoxyvinyl)aryl]prop-2-yn-1-ones in toluene at 80 °C (Scheme 88) (18OBC7361). Bikaverin analogs, benzo[*b*]xanthone derivatives, were prepared by halogenation of 12-(3-hydroxy-1,4-naphthoquinon-2-yl)-6*H*-benzo[*b*] xanthene-6,11-(12*H*)-diones, prepared from 2-hydroxyphthazarins substituted benzaldehydes, followed by treatment with water under atmospheric oxygen (18S3931).

6.4.3 Heterocycles Containing One or Two Sulfur Atoms

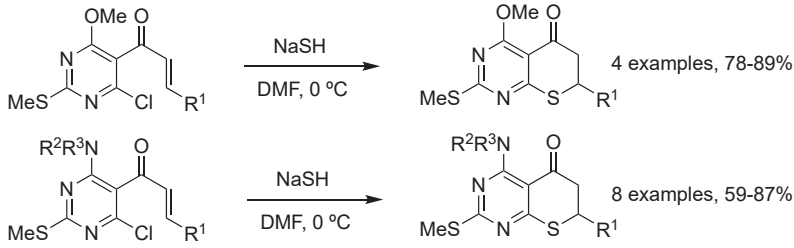
6.4.3.1 Thiopyrans and Analogues

Various conjugated dienes, such as cyclohexa-1,3-diene, cyclohepta-1,3,5-triene, and anthracene, underwent DA reactions with linear and cyclic perfluorinated olefins in the presence of sulfur and cesium/potassium fluoride to give perfluorinated dihydrothiopyran-type compounds (18CC9298). Organocatalytic asymmetric double Michael reactions of 2-oxo-2-arylethyl 4-oxo-4-arylbut-2-en-1-yl thioethers with *trans*-α-cyano-α,β-unsaturated ketones in methyl *t*-butyl ether (MTBE) as solvent at room temperature for five days provides a wide variety of 2,4-diaroyl-5-(2-oxo-2-arylethyl)-3-aryltetrahydro-2*H*-thiopyran-4-carbonitriles (18SL576). The synthesis of 2-aryl-3-bromo-3-nitro-3,4-dihydro-2*H*-thiopyrano[2,3-*b*]quinolin-4-ols is accomplished via asymmetric domino Michael/Henry reaction of 2-mercaptoquinoline-3-carbaldehydes with β-aryl-α-bomonitroalkenes mediated by a bifunctional thiourea **44** in chloroform at −30 °C (Scheme 89) (18SL603).

A different approach uses lemon juice as a natural acid to assist DA reaction of 5-arylmethylene-3-phenyl-4-thioxothiazolidin-2-ones with *N*-arylmaleimides, in order to prepare polysubstituted thiopyrano[2,3-*d*][1,3]thiazole derivatives (18SC2496). One-pot cascade reactions of 3-*S*-(2-oxoalkyl)substituted indolin-2-ones with nitroalkenes promoted by a bifunctional quinine-thiourea catalyst **45** (Scheme 90) (18CEJ62) or with α,β-unsaturated aldehydes promoted by L-proline (18OBC625) led to a series of highly substituted spirooxindole tetrahydrothiopyrans.



Scheme 91



Scheme 92

$Cu(OTf)_2$ mediates cascade annulation reactions of 2-alkynylthioanisoles with unsaturated α-bromocarbonyls in acetonitrile at 80 °C to give cyclopentene-fused thiochromans, in moderate to good yields (18JOC13726).

The synthesis of 2*H*-thiochroman 1,1-dioxides involves a multi-step strategy: (1) treatment of α-substituted *o*-bromostyrenes with butyllithium in THF to afford the corresponding α-substituted *o*-lithiostyrenes, (2) reaction successively with sulfur and methyl iodide to give α-substituted *o*-(methylsulfanyl)styrenes, (3) oxidation with *m*-chloroperbenzoic acid (MCPBA) to afford α-substituted *o*-(methylsulfonyl) styrenes, and (4) cyclization promoted by LDA in THF at −78 °C and subsequent warming of the reaction mixture to 0 °C (18H(96)1570). Another multistep strategy was applied in the synthesis of 4-aryl-1*H*-2-thiochromene-3-carboxylic acid/3-carbonitrile derivatives, starting from *o*-bromobenzyl mercaptans. It involves their reaction with two equivalents of butyllithium, subsequent coupling with aromatic aldehydes and 2-bromoacetonitrile/*t*-butyl 2-bromoacetate, oxidation with PCC and finally reaction with sodium hydride for the thiochromene ring formation (18H(96)1430).

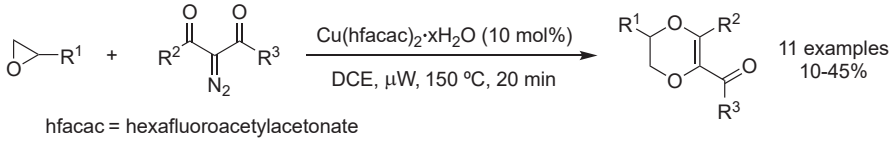
A wide range of isothiochroman-3-ones is obtained from metal-free oxidative cyclization of internal or terminal alkynyl benzyl thioethers, carried out in the presence of $HNTf_2$ as catalyst and 2,6-dibromopyridine *N*-oxide as oxidant in 1,2-dichloroethane (DCE) (18OL7721). High yields and enantioselectivity were achieved in the formal [3 + 3] cycloaddition reactions of α,β-unsaturated anhydrides with indoline-2-thiones to afford indolo[3,2-*e*]-3,4-dihydrothiopyran-2-ones (Scheme 91) (18T6804).

The synthesis of functionalized thiochromen-4-ones is accomplished through Darzens reactions of thioisatins with sulfonium salts in the presence of cesium carbonate (18OBC3487), rhodium-catalyzed [3 + 2 + 1] cyclization reactions of aromatic sulfides with alkynes and carbon monoxide (18JOC13612), and palladium(II)-catalyzed reactions of 1-bromo-2-fluorobenzenes with terminal alkynes and *tert*-butyl isocyanide as carbonyl source, subsequent addition of $Na_2S \cdot 9H_2O$, and finally hydrolysis promoted by oxalic acid (18SL621). Examples of (*E*)-*N*-aryl-4*H*-thiochromen-4-imines arise from cascade reactions of 2-bromobenzothioamides with terminal alkynes, using CuI as catalyst, L-proline as ligand, and cesium carbonate as base (18JOC9504).

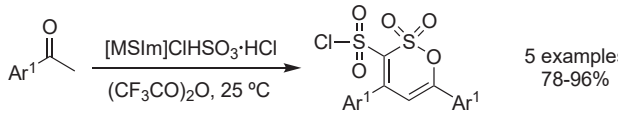
Treating 1-[2-(methylthio)phenyl]-3-(hetero)arylprop-2-yn-1-ones with 2-(2-fluoropyridinium-1-yl)-1,1-bis[(trifluoromethyl)sulfonyl]ethan-1-ide in acetonitrile at room temperature gives bis(triflyl)thioflavones while refluxing toluene affords triflylbenzothienopyrans. Variation of the benzene linker was also performed using pyridine-, cyclohexene-, indole-, and thiophene-tethered alkynones to give the corresponding thioflavone-type compounds. The reaction in acetonitrile at room temperature was extended to oxygen-substituted derivatives such as methoxyalkynones and hydroxyalkynones to achieve bis(triflyl)flavone-type compounds (18CEJ8186).

A series of functionalized 6,7-dihydro-5*H*-thiopyran[2,3-*d*]pyrimidin-5-ones was synthesized through reaction of 1-[4-chloro-6-(methoxy/dialkylamino)pyrimidin-5-yl]alk-2-en-1-ones with sodium hydrogensulfide in DMF at 0 °C (Scheme 92) (18H(96)287). Intramolecular Friedel—Crafts cyclization reactions of 2-(thioaryl) diarylmethanols carried out in the presence of *N*-triflylphosphoramide as organocatalyst in THF at room temperature delivers substituted 9*H*-thioxanthenes (18SC2177).

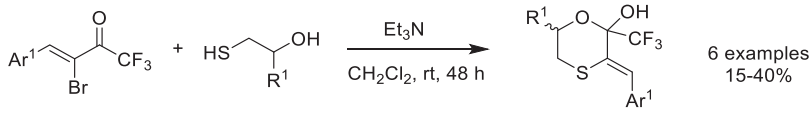
6.4.4 Heterocycles Containing Two or More Oxygen Atoms



Scheme 93



Scheme 94



Scheme 95

6.4.4.1 Dioxanes

Microwave-assisted ring expansion of oxiranes with α-diazo-β-dicarbonyl compounds using copper hexafluoroacetylacetonate as catalyst in DCE led to 2-acyl-5,6-dihydro-1,4-dioxines, in modest yields (Scheme 93) (18T1613). A range of 4*H*-benzo[*d*][1,3] dioxin-4-ones were obtained via an organocatalyzed Michael addition cascade of salicylic acids with 4-arylbut-3-yn-2-ones carried out in the presence of morpholine in dichloromethane at room temperature (18OBC5533).

6.4.5 Heterocycles Containing Both Oxygen and Sulfur in the Same Ring

6.4.5.1 Oxathianes

Nickel-promoted reaction of 2-substituted ethenesulfonyl fluorides with 2-acetylazaarenes or 6,7-dihydroquinolin-8(5*H*)-one provides, respectively, 4,6-disubstituted or quinoline-fused 1,2-oxathiines, via an annulative sulfur(VI) fluoride exchange process (18CC9011). Several examples of 4,6-diaryl-3-(chlorosulfonyl)-1,2-oxathiine-2,2-dioxides were obtained from the one-pot *pseudo* four-component reaction of two equivalents of simple acetophenones with the ionic liquid [MSIm] $ClSO_3 \cdot HCl$ and trifluoroacetic anhydride at room temperature (Scheme 94) (18T4047).

Microwave-assisted copper(II) sulfate−catalyzed ring expansion of thiiranes with α-diazo-β-dicarbonyl compounds in DCE gives 3-acyl-5,6-dihydro-1,4-oxathiines (18T1613). The synthesis of 3-benzylidene-2-(trifluoromethyl)-1,4-oxathian-2-ols is accomplished through Michael addition of β-mercaptoalcohols with 4-aryl-3-bromo-1,1,1-trifluorobut-3-en-2-ones in the presence of triethylamine in dichloromethane at room temperature (Scheme 95) (18EJO3716).

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