PROGRESS IN HETEROCYCLIC CHEMISTRY

VOLUME 30

Edited by

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CHAPTER 6.4

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

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6.4.1 INTRODUCTION

A large variety of publications involving *O*- and *S*-6-membered ring systems have appeared in 2017. The importance of these heterocyclic compounds is highlighted by the huge number of publications on the total synthesis of natural oxygen derivatives and of other communications dedicated to synthetic derivatives.

Reviews on stereoselective organocatalytic synthesis of tetrahydropyrans (17EJO4666), of tetrahydropyrans and their application in total synthesis of natural products (17CSR1661), on the synthesis of the less thermodynamically stable 2,6-*trans*-tetrahydropyrans (17S4899), on enantioselective synthesis of polyfunctionalized pyran and chromene derivatives (17TA1462), and on enantioselective and racemic total synthesis of camptothecins, including the formation of their pyran-2-one ring (17SL1134), have appeared.

Advances in the transition metal-catalyzed synthesis of pyran-2/4-ones (17TL263), *N*-heterocyclic carbene (NHC)-catalyzed achiral synthesis of pyran-2-one, coumarin and (thio)chromone derivatives (17OBC4731), on the synthesis and transformation of 2*H*-pyran-2-ones (17T2529) and 2-styrylchromones (17EJO3115) into other heterocyclic compounds, have been surveyed. The strategies to build up the tetrahydropyranyl core of brevisamide (17H(95)81) and the reactions of ketyl radicals, generated from carbonyl derivatives under transition-metal photoredox-catalyzed conditions, leading to isochromen- and chroman-type compounds (17CC13093) were disclosed. Developments in the synthesis of pentafluorosulfanyl(chromene and coumarin) derivatives (17TL4803), photoswitchable Δ^9-tetrahydrocannabinol derivatives (17JA18206), and aminobenzopyranoxanthenes with nitrogen-containing rings (17JOC13626) have been studied.

Progress in Heterocyclic Chemistry, Volume 30
ISSN 0959-6380, https://doi.org/10.1016/B978-0-08-102788-2.00015-5

Discussions of specific reactions were accomplished and include carbonylation reactions for the synthesis of coumarin and (thio)chromone derivatives (17SL175), Prins spirocyclization for the synthesis of spiropyrans (17EJO5484), ring-closing metathesis (RCM) to obtain bryostatin analogs (17OBC2768), transannular oxa-6π-electrocyclization induced by UVA light for the synthesis of briarane diterpenoids (17OL576), triple diene-transmissive Diels–Alder cycloaddition reactions centered on the synthesis of javanicin analogs (17H(95)894).

Special emphasis was given to the total synthesis of natural oxygen derivatives. Examples are the 5,6-dihydro-2*H*-pyrans diplopyrone (17JOC4561), leptolyngbyolides (17CEJ8500), leustroduscin B (17EJO6804), mandelalide A and isomandelalide A (17JA770); tetrahydropyrans 7-*des*-*O*-pivaloyl-7-*O*-benzylbryostatin 10 (17OBC9497), (±)-decytospolides A and B (17SL249); (+)-Greek tobacco lactone (17OL1478), goniothalesdiol A (17TL1037), (+)-herboxidiene (17OBC1842), phomonol (17TL2898), and zincophorin methyl ester (17JA4568); chroman-type rennellianone B (17TL556); chroman-type (+)-lophirone H and its pentamethyl ether (17OL2486), myrtucommulone K (17TL1817), (+)- and (−)-paeoveitol (17OL429); isochromenes (+)-γ-actinorhodin (17AGE3383), (−)-arizonin B1 and C1 (17EJO2512), and (±)-chaetophenol C (17OL4387); pyranones (−)-angiopterlactone B (17OL2199), aroncin B (17CEJ16525), brevipolide M (17OBC6393), (6*R*,7*R*,8*S*)-8-chlorogoniodiol (17S2483), 8-chlorogoniodiol and parvistone A (17TA246), obolactone 1 (17T5547), parvistone C and its C-8 epimer (17SC1879), pellasoren A (17OL2394), pironetin analogs (17OBC220), (+)-synargentolide B (17T6443) and (+)-wortmannin (17JA6815); coumarins lamellarin D and H (17JOC4998) and G (17OL2262) derivatives; isocoumarin-type cruentaren B (17TL2685) and exserolide (17OL2074); chromone-type gonytolide C (17TL4479), puerarin derivatives (17TL2835), (−)-rotenone and (−)-dalpanol (17AGE182) and (±)-sanggenol F (17T3485); xanthene-type (+)-cyclospongiaquinone-1 and (−)-dehydrocyclospongiaquinone-1 (17EJO901), hongoquercins A and B and chromazonarol (17EJO1143) and psiguadial B (17AGE13776); xanthone derivatives ascherxanthone A (17OL1834), (+)-blennolide C (17TL4479), citreamicin η (17OL790) and dicerandrol C (17CEJ2299). Improved syntheses of cortistatin A analogs (17T1342) and nigricanin (17CPB1078), and a short enantiospecific semisynthesis of puupehedione-type marine natural products (17JOC12914) have been disclosed.

Developments on the stereoselective synthesis of tetrahydropyran-containing C-1—C-14 subunit of thiocoumarinols (17T2814) and the C-30—C-40 subunit of pectenotoxin 2 (17OL5154) have also been achieved.

Herein, we provide a personal overview of the most relevant transformations on *O*- and *S*-6-membered heterocycles, published in 2017.

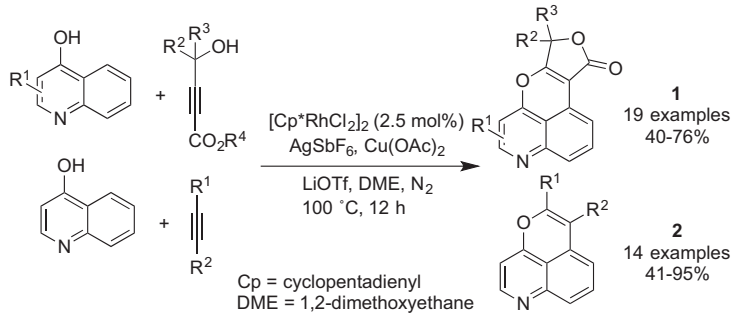
6.4.2 HETEROCYCLES CONTAINING ONE OXYGEN ATOM

6.4.2.1 Pyrans

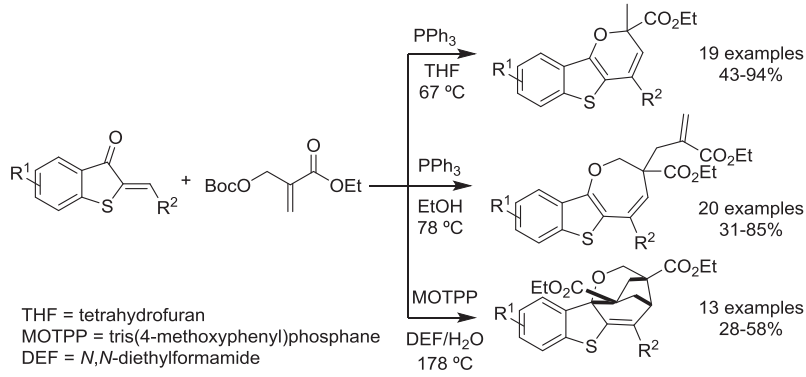
A wide range of 2-azido-quinolino[3,2-*c*]-2*H*-pyrans were chemoselectively prepared through 6-*endo*-*dig* electrophilic cyclization by a [3+2] cycloaddition reaction of 2-alkynylquinoline-3-carbaldehydes with sodium azide mediated by AgOTF. Replacing AgOTF by iodine and potassium carbonate, some 2-azido-5-iodo-quinolino[3,2-*c*]-2*H*-pyrans were obtained (17JOC6388). Rh(III)-catalyzed cascade C—H activation/annulation/lactonization reactions of quinolin-4-ols with alkyl 4-hydroxyalk-2-ynoates led to the synthesis of tetracyclic furan-2-one-fused quinolino [4,4a,5-*bc*]-2*H*-pyrans **1**, while with simple alkynes, a range of quinolino [4,4a,5-*bc*]pyrans **2** were obtained (Scheme 1; 17CC7824).

Domino reactions of 2-alkylidenebenzothiophene-3(2*H*)-ones with Morita—Baylis—Hillman (MBH) carbonates are solvent-controlled: in the presence of PPh_3 in tetrahydrofuran (THF) this leads to benzothiophene-fused α-pyrans while in ethanol benzothiophene-fused 2,3-dihydrooxepines are formed and using tris(4-methoxyphenyl)phosphane as catalyst and a 7:1 mixture of *N*,*N*-diethylformamide (DEF/H_2O) produces oxatricyclodecene derivatives (Scheme 2; 17OL6084).

A series of functionalized 3,4-dihydropyrans result from the cross-coupling intramolecular Michael addition of functionalized terminal



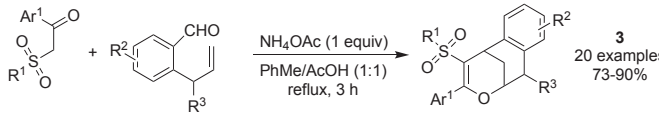
Scheme 1



Scheme 2

alkynes and diazo compounds carried out in the presence of copper(I) chloride and a phosphine ligand (17CC4350). α-Aryl-δ-keto malononitriles underwent dynamic kinetic asymmetric transfer hydrogenation–cyclization tandem reactions catalyzed by a chiral (mesitylene)RuCl(monosulfonated diamine) to give chiral 6-amino-3,4-dihydro-2*H*-pyran-5-carbonitriles (17CC6113). Enantioselective oxa-[4+2] cycloaddition reaction of α-substituted allenones with perfluoroalkyl α,β-unsaturated ketones promoted by a ferrocene-derived bifunctional phosphine-catalyst led to perfluoroalkylated 3,4-dihydropyrans, in high yields and with excellent enantioselectivity (17CEJ13587). A similar protocol used non-activated allenes with β,γ-unsaturated α-keto esters mediated by a chiral cationic indium complex (17AGE10867). A quinine-derived primary amine and benzoic acid catalysts offer high enantioselectivity in the asymmetric inverse electron demand (IED) hetero-Diels–Alder (hDA) reaction of linear deconjugated enones with α-cyano oxadienes to afford polysubstituted 3,4-dihydropyran-5-carbonitriles (17EJO871). An organocatalytic asymmetric aza-Michael-IED–hDA cascade reaction of ethyl (*E*)-4-[2-(4-methylphenylsulfonamido)phenyl]-2-oxobut-3-enoate with enals furnished quinolino[3,4-*c*]-3,4-dihydro-2*H*-pyrans in good yields with excellent diastereo- and enantio-selectivities (17OBC9630). Domino Knoevenagel/DA cyclocondensation reaction of β-ketosulfones with 2-allylbenzaldehydes mediated by NH_4OAc in a 1:1 mixture of toluene/acetic acid leads to sulfonyl tricyclic system **3** containing a 3,4-dihydropyran moiety (Scheme 3; 17JOC13324).

Various 2,3-dihydropyrrolizino[3,4-*c*]-3,4-dihydro-2*H*-pyrans can be synthesized through DMAP-catalyzed addition/(4+2) annulation reactions

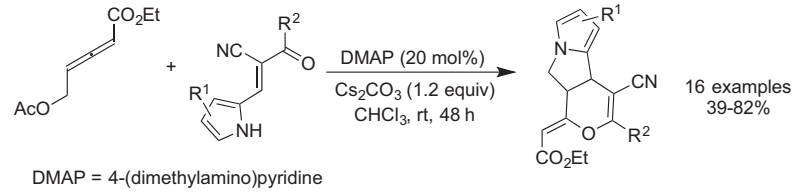


Scheme 3

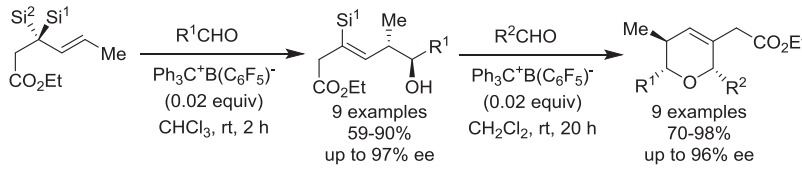
of a δ-acetoxy allenoate with 3-(pyrrol-2-yl)acrylonitriles in the presence of cesium carbonate in chloroform (Scheme 4; 17OBC4807).

Allylic ethers, formed by allylation of the corresponding homoallylic alcohols, undergo RCM to give *trans*-2,3-diaryl(heteroaryl)-3,6-dihydro-2*H*-pyrans (17EJO3343).

Palladium-catalyzed addition of homopropargylic alcohols to alkoxyallenes and a subsequent gold(I)-catalyzed cycloisomerization reaction furnishes both 2,6-*cis*- and 2,6-*trans* 4-alkoxy-5,6-dihydro-2*H*-pyrans, according to the stereochemistry of the ligand used in the first step (17OL242). Synthesis of a series of functionalized 6-aryl-5,6-dihydro-2*H*-pyrans was achieved through an oxa-DA reaction of aromatic aldehydes with unactivated polysubstituted 1,3-butadienes catalyzed by $AlCl_3$ in dry CCl_4 at room temperature (17T4039). An enantioselective version uses an imidodiphosphorimidate catalyst in methylcyclohexane at low temperatures (17JA13656). Highly substituted chiral 5,6-dihydro-2*H*-pyrans are obtained through asymmetric Sakurai allylation of crotyl geminal bis(silane) derivatives with aldehydes and subsequent Prins cyclization of the obtained products with other aldehydes, both reactions being catalyzed by $Ph_3C^+B(C_6F_5)_4^-$ (Scheme 5; 17CC3078).



Scheme 4

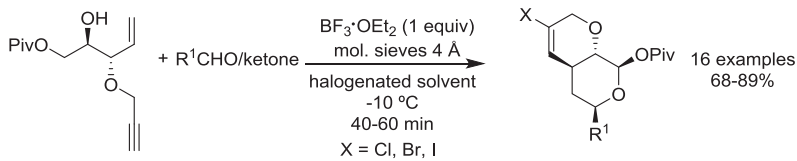


Scheme 5

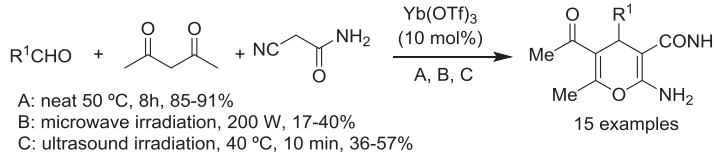
A few examples of dimethyl 3-substituted-5,6-dihydro-2*H*-pyran-2,2-dicarboxylates arise from the cascade reaction of dimethyl (but-3-yn-1-yloxy)/2-(pent-4-yn-2-yloxy)malonate with diazoacetates promoted by CuI and a bispyridine ligand (17JOC5492). Stereoselective synthesis of 3-halogenated tetrahydropyrano[3,4-*b*]-5,6-dihydro-2*H*-pyrans occurs through Prins cyclization of *O*-propargylated D-mannitol-derived homoallylic alcohols with several aldehydes or cyclohexanone in the presence of $BF_3 \cdot OEt_2$ and a halogenated solvent (Scheme 6; 17EJO5986).

Excellent enantioselectivity is achieved in the formal [3+3] annulation reaction of δ-acetoxy allenoates with 1*C*,3*O*-bisnucleophiles promoted by a tertiary amine organocatalyst to afford highly substituted 4*H*-pyrans (17OL1890, 17T3347). One-pot three-component reaction of aromatic aldehydes with malononitrile and β-keto esters carried out in the presence of hexamethylenetetramine in water gives access to polyfunctionalized 2-amino-4-aryl-4*H*-pyran-3-carbonitriles (17JHC1598, 17JHC1880). Further derivatives arise from the reaction of 2-tosyloxybenzaldehyde with malononitrile and cyclic and acyclic ketones in the presence of trimethylamine in ethanol, ammonium hydroxide in methanol, or under ultrasound irradiation (17JHC1442). Examples of 4-alkyl/aryl-2-amino-4*H*-pyran-3-carboxamides are produced from an ytterbium triflate-mediated three-component reaction of aliphatic/aromatic aldehydes with acetylacetone and cyanoacetamide. This reaction occurs at 50 °C under solvent-free conditions and applying microwave and ultrasound irradiation, the first protocol being the most efficient (Scheme 7; 17TL1659).

Visible light—induced cascade cyclization reactions of acyl chlorides with 2-(alkyl/arylethynyl)phenyl methacrylate or *N*-methyl-*N*-[2-(alkyl/arylethynyl)phenyl] methacrylamide mediated by [*fac*-^{III}Ir(ppy)$_3$] (in which ppy = 2-phenylpyridine) provides, respectively, coumarin-fused or 2-quinolone-fused 2,6-disubstituted 4*H*-pyrans (17OL512). Highly functionalized pyrazole-fused 4*H*-pyrans are prepared through [3+3] tandem reaction of 1-aryl-3-methylpyrazol-5-ones with 3-aryl-2-(arylethynyl)



Scheme 6

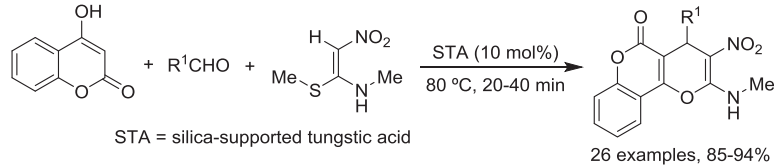


Scheme 7

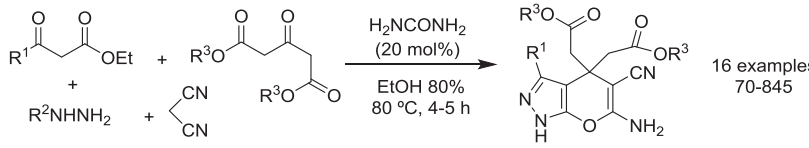
propen-1-ones mediated by a tertiary amine squaramide catalyst (17TA1708). A series of coumarin-fused 4-alkyl/aryl-2-amino-3-nitro-4*H*-pyrans result from a three-component condensation reaction of 4-hydroxycoumarin, aldehydes, and (*E*)-*N*-methyl-1-(methylthio)-2-nitroethenamine in the presence of a catalytic amount of silica-supported tungstic acid (Scheme 8; 17T5163).

A range of pyrazole-fused 2-amino-4*H*-pyran-3-carbonitriles can be obtained through a three-component reaction of aromatic aldehydes with malononitrile and 3-methyl-1*H*-pyrazol-5(4*H*)-one using montmorillonite K-10 as catalyst in aqueous ethanol at room temperature (17JHC89), of 2-acetylfuran/thiophen with malononitrile and 3-phenyl/methyl-1*H*-pyrazol-5(4*H*)-ones carried out in the presence of sodium ethoxide (17JHC2313), and a four-component reaction of ethyl acetoacetate with hydrazines, malononitrile, and 3-oxo-pentanedioic acid dialkyl esters carried out in the presence of urea and aqueous ethanol at 80 °C (Scheme 9; 17T164) or electrolytically with aromatic aldehydes at a constant current in the presence of sodium bromide as supporting electrolyte (17TL1245).

A series of spiroxindole 2-quinolone-fused 2-amino-4*H*-pyran-3-carboxylates/carbonitriles result from a three-component condensation



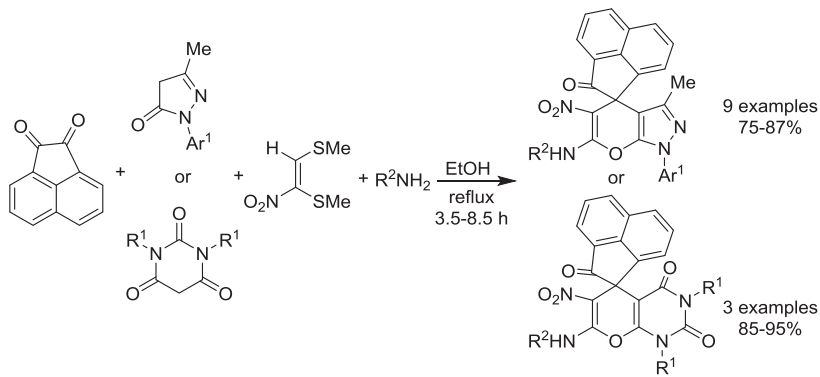
Scheme 8



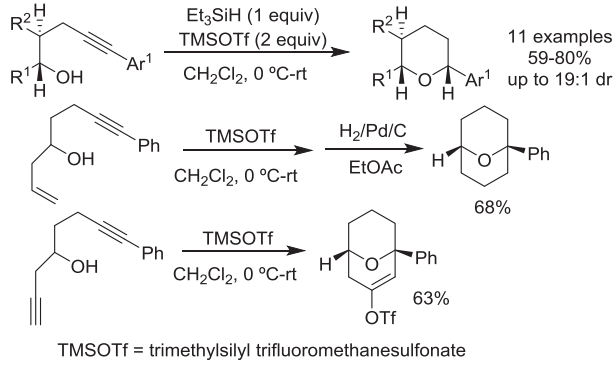
Scheme 9

reaction of 1-methylquinoline-2,4(1*H*,3*H*)-dione with ethyl cyanoacetate/malononitrile and isatins in the presence of the ionic liquid 1,8-diazabicyclo[5.4.0]undec-7-en-8-ium acetate (17JHC2326). The synthesis of spirooxindole pyrazole-fused 2-amino-4*H*-pyran-3-carbonitriles occurs via a one-pot four-component reaction of diethyl oxaloacetate sodium salt with hydrazine hydrate, isatins, and malononitrile in the presence of triethylamine in a 1:1 mixture of acetic acid:ethanol (17TL134). An alternative protocol uses β-ketoesters, various hydrazines, isatins, and malononitrile or methyl(ethyl) cyanoester in the presence of *N*,*N*,*N*′,*N*′-tetrabromobenzene-1,3-disulfonamide (TBBDA) or poly(*N*,*N*′-dibromo-*N*-ethylbenzene-1,3-disulfonamide) (PBBS) as catalyst (17JHC465). A one-pot four-component condensation reaction of acenaphthoquinone, pyrazolones or barbituric acids, 1,1-bis(methylthio)-2-nitroethene, and alkylamines in refluxing ethanol provides spiroacenaphthylene pyrazole-fused 2-amino-3-nitro-4*H*-pyrans or 2-amino-3-nitro-4*H*-chromene type compounds, respectively (Scheme 10; 17TL4260).

Alk-2-en-1,7-diols underwent a Re_2O_7-catalyzed dehydrative cyclization reaction to achieve 2,6-disubstituted tetrahydropyrans in high yields and with good diastereoselectivity (17AGE10900). Further derivatives are obtained from 6-*endo*-*dig*-hydroalkoxylation-reduction reaction of alk-4-yn-1-ols carried out using trimethylsilyl trifluoromethanesulfonate (TMSOTf) and Et_3SiH in dichloromethane. Using TMSOTf, a hydroalkoxylation−Prins cyclization cascade reaction of octa-1-en/yn-7-yn-4-ols occurs to provide a couple of oxa-bicyclic derivatives (Scheme 11; 17OL6534). A three-component reaction of 6-methylhept-5-en-2-ol with arylaldehydes and nitriles or thiols in the presence of a tetrafluoroboric acid



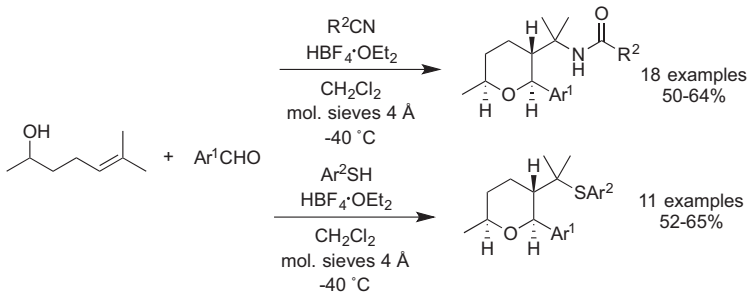
Scheme 10



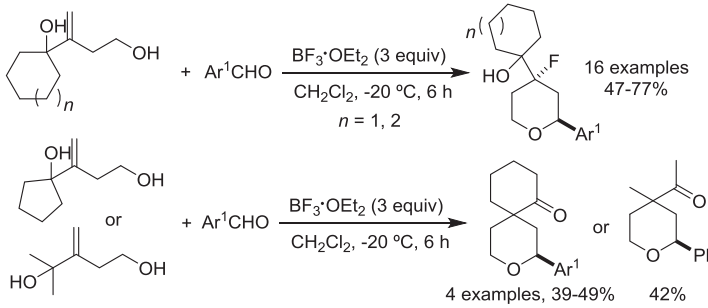
Scheme 11

diethyl ether complex affords 2,3,6-trisubstituted tetrahydropyrans, respectively, via Prins–Ritter and Prins–arylthiolation cyclization reactions (Scheme 12; 17OBC2003). Various 2,6-disubstituted 4-(*N*-acetyl)tetrahydropyrans are available by Prins–Ritter cyclization of hepta-1,6-dien-4-ol with aliphatic/aromatic aldehydes, promoted by bismuth(II) triflate in acetonitrile at room temperature (17S5197).

Cyclization reactions of 1-cycloalkyl-2-methylenebutane-1,4-diols with aldehydes in the presence of BF_3 is dictated by the ring-strain: cyclohexane and cycloheptane derivatives suffer fluorinating Prins cyclization to afford 4-fluoro-2,4-disubstituted tetrahydropyrans while cyclopentane and an acyclic derivative undergo semipinacol rearrangement reactions to give spirocyclohexan-2-one or 4-acetyl-4-methyltetrahydropyrans (Scheme 13; 17OBC6478). A diastereomeric mixture of dimethyl 2-aryl-5-substituted tetrahydropyran-4,4-dicarboxylates is obtained from the [3+3] annulation reaction of γ-hydroxyenones with dimethyl 2-arylcyclopropane-1,1-dicarboxylates mediated by $Sc(OTf)_3$ in dichloromethane at 35 °C. A



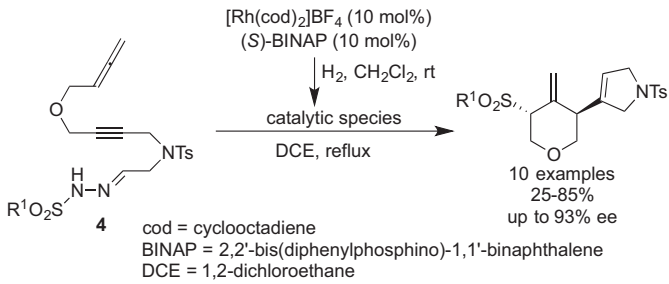
Scheme 12



Scheme 13

preliminary asymmetric version uses a chiral PyBox ligand but with poor enantioselectivity (17EJO534).

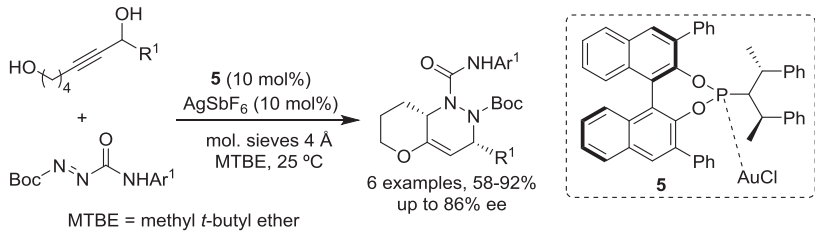
Tuning the olefinic geometry of α-silyloxy homoallylsilanes in the silyl-oxa-Prins cyclization with various aldehydes, selective *syn*- and *anti*-isomeric 2-silyltetrahydropyranol derivatives are obtained: (*Z*)-isomers in the presence of methanesulfonic acid or trifluoroacetic acid lead to *syn*-tetrahydropyran-4-mesylate or tetrahydropyran-4-ols, respectively; (*E*)-isomers in the presence of trifluoroacetic acid afford *anti*-tetrahydropyran-4-ols (17EJO933). Under the influence of catalytic species obtained from the mixture of a Rh(I) complex and 2,2'-bis(diphenylphosphino)-1,1'-binaphthalene (*S*-BINAP) treated with hydrogen, several allene oxygen-tethered alkynes **4** undergo cascade reactions to give 3,5-disubstituted 4-methylenetetrahydropyrans (Scheme 14; 17CC9922). The syntheses of 2,6-disubstituted 4-chloro-3-chloromethyltetrahydropyrans are accomplished via tandem S_N2'—Prins cyclization reactions of 3-chloroalk-4-en-1-ols with aliphatic/aromatic aldehydes in the presence of $FeCl_3$ and trimethylsilyl chloride in dry dichloromethane (17OL4834).



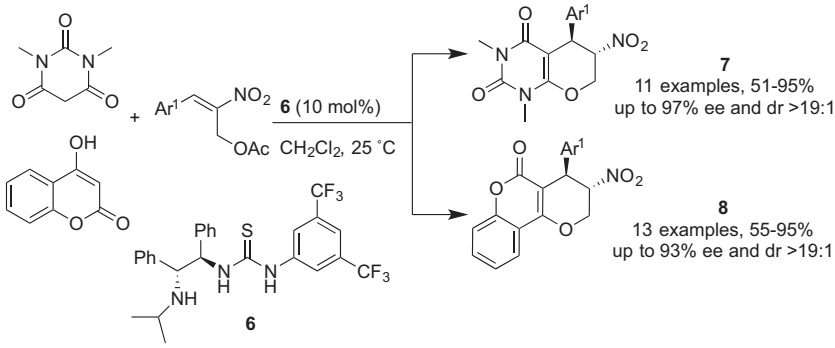
Scheme 14

Homoallyl and homopropargyl alcohols bearing benzofuran and benzothiophene moieties undergo, respectively, *O*-allylation and *O*-propargylation to afford the corresponding enyne systems which suffer intramolecular Pauson–Khand reactions to afford cyclopentenone-fused benzofuran- and benzothiophene-substituted tetrahydropyrans (17TA479). Tandem dehydrative cyclization/aza-hDA reaction of propargyl alcohols with urea-based diazenes catalyzed by gold(I) complex **5** bearing BINOL-derived chiral phosphoramidite ligand provides pyridazine-fused tetrahydropyrans (Scheme 15; 17S151).

Trifluoroacetic acid–catalyzed oxa-Pictet–Spengler reaction of 3-(2-substituted-2-hydroxyethyl)indoles with acetals produces indolo[2,3-*c*] tetrahydropyrans in high yields, enantioselectivities, and diastereoselectivities (17TL129). Bifunctional secondary amine-thiourea **6** catalyzes the domino Michael–Michael addition reaction of *N*,*N*′-dimethylbarbituric acid or 4-hydrocoumarin with MBH acetates of nitroalkenes, giving access to pyrimidine-fused, **7**, or coumarin-fused, **8**, 4-aryl-3-nitrotetrahydropyrans, respectively (Scheme 16; 17JOC13594).



Scheme 15



Scheme 16

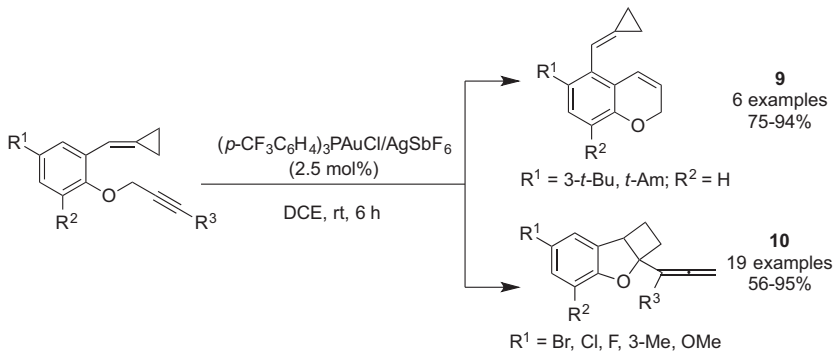
6.4.2.2 [1]Benzopyrans and Dihydro[1]benzopyrans

6.4.2.2.1 Chromenes and Chromans

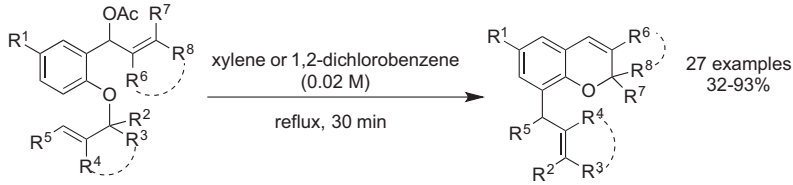
Cycloisomerization of *o*-(propargyloxy)arylmethylenecyclopropanes catalyzed by $(p\text{-}CF_3C_6H_4)_3PAuCl/AgSbF_6$ system is controlled by adjacent substituents on the aromatic ring: with sterically bulky substituents, methylenecyclopropane migration takes place to produce 5-methylenecyclopropane 2*H*-chromenes **9**; in the presence of Me, MeO, or a halogen atom as substituents, methylenecyclopropane ring enlargement and rearrangement of the propargyl group occurs to give cyclobutane-fused benzofurans **10** (Scheme 17; 17CEJ6845).

A series of 2,2-diaryl-4-bromo/iodo-2*H*-chromenes result from cascade cyclization of 1,1-diarylprop-2-ynolphenols in the presence of HBr in 1,2-dichloroethane or HI in dichloromethane, respectively (17TL3049). Regioselective hydroarylation of 3-substituted phenyl 1,1-disubstituted propargyl ethers catalyzed by cationic Au(I) complexes leads to a mixture of 2,2-disubstituted 5- and 7-substituted 2*H*-chromenes, depending on the ligand and solvent dielectric constant (17JA4035). 3-Acetyloxy-3-(2-alkoxyaryl)prop-1-enes undergo cascade Claisen rearrangement, *o*-quinone methide formation, and electrocyclization reactions to give a wide range of polysubstituted 2*H*-chromenes (Scheme 18; 17CC6021).

A one-pot synthesis of 2,2,3-trisubstituted 2*H*-chromenes can be achieved through [4+2] cycloaddition of salicylaldehydes with arylalkynes, via *o*-quinone methides, carried out in the presence of trimethyl orthoformate and TfOH in toluene followed by the addition of methanol (17T6456). Enyne metathesis of some *o*-allyloxy(phenylethynyl)benzene derivatives using Grubbs' second-generation catalyst affords the corresponding 4-(1-phenylvinyl)-2*H*-chromenes (17EJO2359). Palladium-mediated tandem



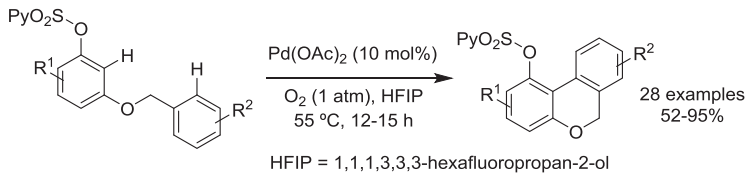
Scheme 17



Scheme 18

oxidative arylation/vinylation reactions of 3-arylpropargyloxybenzenes with styrene or acrylate derivatives provided 4-aryl-3-substituted 2*H*-chromenes in moderate to good yields (17CEJ793). Under solvent-free conditions, $Ca(OTf)_2$-promoted cascade annulation of propargyl alcohols with ambident enols (4-hydroxycoumarin, cyclohexane-1,3-dione, and 5,5-dimethylcyclohexane-1,3-dione) leads to structurally diverse 2*H*-chromenes or benzochromenes. This process involves etherification, Claisen-type rearrangement, allene formation, and endocyclization (17TL4642). The synthesis of 6*H*-benzo[*c*]chromenes occurs via intramolecular dehydrogenative coupling reaction of *O*-benzyloxyphenols catalyzed by palladium(II) acetate in the presence of 1,1,1,3,3,3-hexafluoropropan-2-ol (HFIP) and using molecular oxygen as terminal oxidant (Scheme 19; 17OL798).

A wide range of indeno[1,2-*c*]chromenes were prepared through cascade reactions of 5-[2-(2-oxoalkoxy)aryl]pent-4-yn-1-ols mediated by zinc bromide in toluene at 130 °C. It involves a 5-*exo*-*dig*-cyclization, Friedel—Crafts reaction, and ring-opening sequence (17OL488). Similar iodinated or brominated derivatives arise from a cascade cyclization reaction of aryldiynes, 1-methoxy-2-[2-(arylethynylphenyl)]ethynylbenzene derivatives, with iodine in dichloromethane or copper(II) bromide in acetonitrile (17JOC6071). A one-pot synthesis of cyclopenta[*h*]chromenes was accomplished through a cascade reaction of 1,3,8,10-tetraynes with α,β-unsaturated aldehydes in toluene at room temperature. The reaction is extended to the synthesis of pyrrolidino

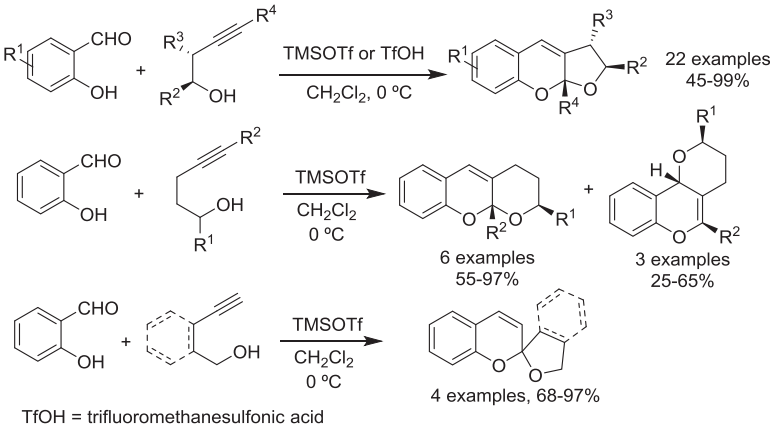


HFIP = 1,1,1,3,3,3-hexafluoropropan-2-ol

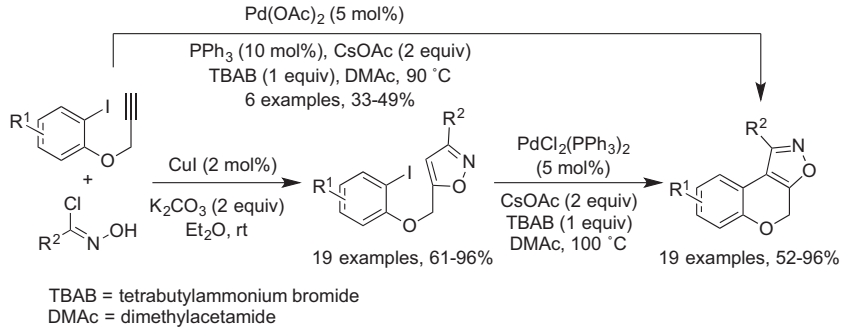
Scheme 19

[3,4-*h*]chromenes starting from the corresponding alkyl-substituted *N*-tetraynes (17CEJ6264). Hydroalkoxylation-formal [4+2] cycloaddition cascade reaction of salicylaldehydes with alk-3-ynols or alk-4-ynols in the presence of TMSOTf or trifluoromethanesulfonic acid (TfOH) gives, respectively, tetrahydrofuran- or tetrahydropyran-fused 2*H*-chromenes. The reaction was extended to the reaction of terminal alkynes to afford spirocyclic 2*H*-chromenes (Scheme 20; 17CEJ10007).

Treating {2-[(prop-2-ynylimino)methyl]aryloxy}acetonitriles with 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) in refluxing DMF led to pyrazino[2,3-*c*]chromenes, in moderate yields (17EJO1489). Intramolecular one-pot [4+2] cycloaddition reactions of *O*-propargylated salicylaldehydes with 3-aminoquinoline mediated by copper(I) iodide and ytterbium triflate in refluxing acetonitrile with molecular sieves provides a range of naphthyridine-fused chromenes (17TL449). The synthesis of isoxazole-fused 2*H*-chromenes is achieved in a two-step protocol involving copper(I) iodide-catalyzed 1,3-dipolar cycloaddition reactions of 2-propargyloxyiodobenzenes with *N*-hydroxybenzimidoyl chloride derivatives and subsequent cyclization reactions promoted by $PdCl_2(PPh_3)_2$. The one-pot procedure uses $Pd(OAc)_2$ as catalyst in the presence of PPh_3, CsOAc, and tetrabutylammonium bromide (TBAB) in dimethylacetamide (Scheme 21; 17S1356). Several examples of thiazol-2-one[4,5-*b*]chromenes were provided by the reaction of 4-aminothiazol-2(5*H*)-one with salicylaldehydes carried out in the presence of sodium acetate in a 1:1 mixture of acetic acid:water (17SL811).



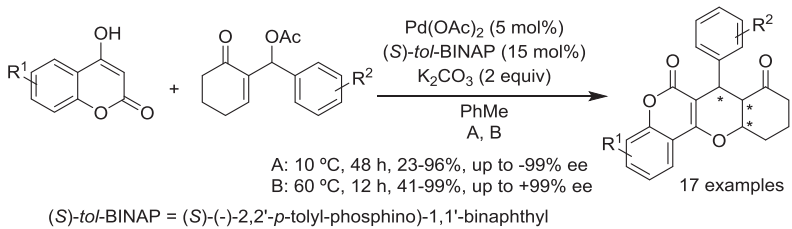
Scheme 20



Scheme 21

The synthesis of 3-acyl-4*H*-chromene derivatives occurs via manganese(III)-catalyzed asymmetric addition of 1,3-dicarbonyl compounds to *o*-quinone methides, generated in situ by catalytic aerobic oxidation of 2-alkylsubstituted phenols (17OL4588) or the reactions of push–pull enaminoketones with *o*-quinone methides, generated in situ from 2-(aminomethyl)phenols or corresponding trimethylammonium salts, in refluxing acetic acid (17JOC1517). Further derivatives arise from a nitro-Michael addition, hemiacetalization reaction of 2-(2-nitrovinyl)phenols with acyclic 1,3-dicarbonyl compounds in neat conditions and subsequent dehydration in the presence of *p*-toluenesulfonic acid (PTSA) in toluene (17JOC8444). Autoinductive reversal of enantioselectivity is temperature-dependent in the palladium-mediated [3+3] annulation reaction of 4-hydroxycoumarin with MBH acetates. Modifying the reaction temperature from 10 to 60 °C, coumarin-fused hexahydrochromene adducts with the opposite absolute configurations were formed (Scheme 22; 17CC4441).

A series of pyrimidine-fused 4*H*-chromenes were synthesized in a one-pot procedure from the reaction of salicylaldehydes with malononitrile and various cyclic amines promoted by TBBDA and PBBS (17JHC215).

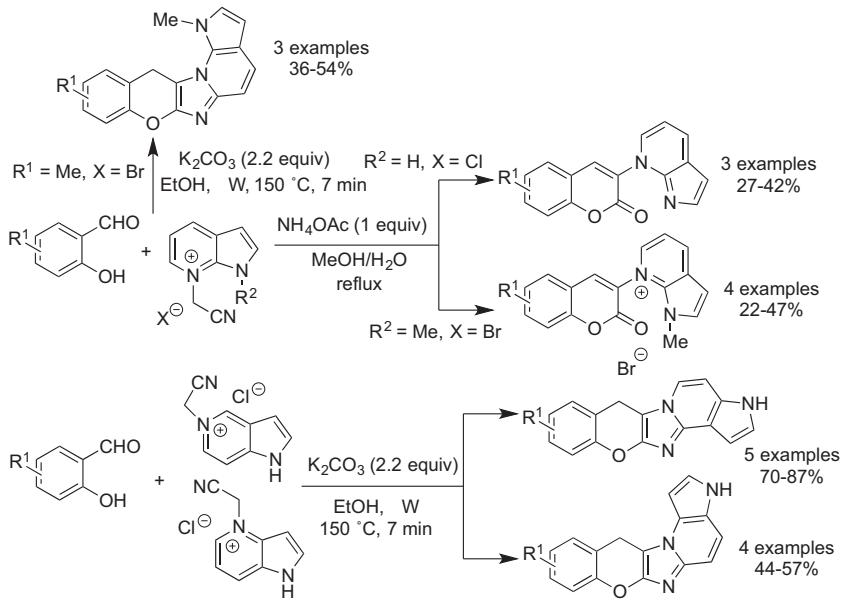


Scheme 22

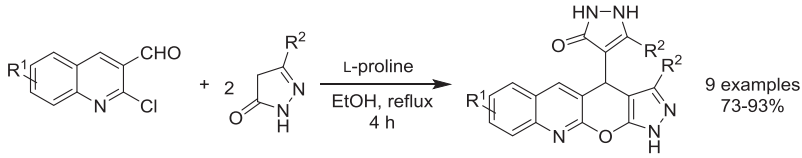
Enantiopure pyrazole-fused hexahydro-4*H*-chromenes are attained by an organocatalytic domino Michael/hemiacetalization reaction of cyclohexanone with alkylidene pyrazolones (17OBC8032). Domino reactions of salicylaldehydes with 7-azaindolium salts promoted by NH_4OAc led to 3-(7-azaindole)substituted coumarins, while microwave-assisted reaction with 4-, 5- and 7-azaindolium salts in the presence of potassium carbonate afforded imidazolopyrrolopyridine-fused chromenes (Scheme 23; 17S2753).

Niobium pentachloride catalyzes multicomponent combination of 4-hydroxycoumarin, aliphatic/aromatic aldehydes, and cyclohexane-1,3-dione giving rise to coumarin-fused tetrahydrochromene derivatives in moderate to good yields (17TL894). Uncatalyzed four-component reaction of benzaldehydes with ethyl acetoacetate, hydrazine hydrate, and thiobarbituric acid in ethanol affords pyrazolo-fused chroman-type compounds (17SC111). High yields of pyrazolo-fused benzo[*g*]aza-4*H*-chromene derivatives result from the reaction 2-chloroquinoline-3-carbaldehydes with 3-substituted pyrazol-5-ones mediated by L-proline in refluxing ethanol (Scheme 24; 17T2116).

The syntheses of some 2-aminotetrahydrochromene-3-carbonitriles were accomplished via three-component reactions of cyclohexane-1,3-diones



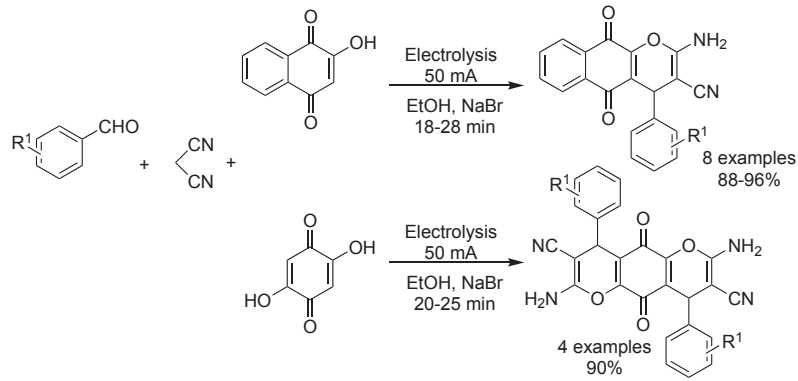
Scheme 23



Scheme 24

with 2-acetylfuran and malononitrile carried out in the presence of sodium ethoxide in refluxing ethanol (17JHC2313). Other 2-aminotetrahydrochromene-3-carboxylates/carbonitriles arise from tandem Michael addition–cyclization reaction of cyclohexane-1,2- and 1,3-diones with (*E*)-3-aryl-2-cyanoacrylate or alkylidene malononitrile derivatives, respectively, promoted by cinchona alkaloid–derived bifunctional organocatalysts (17JHC677). The synthesis of bis(2-aminotetrahydrochromene-3-carbonitriles) can be accomplished via one-pot, multicomponent reaction of bisaldehydes with malononitrile and dimedone in the presence of a catalytic amount of piperidine in refluxing ethanol (17JHC1854) or mediated by chitosan in refluxing ethanol or under microwave irradiation (17JHC305). Electrochemical condensation of aromatic aldehydes with malononitrile and 2-hydroxynaphthalene-1,4-dione or 2,5-dihydroxycyclohexa-2,5-diene-1,4-dione in ethanol at room temperature under constant current density leads to benzo[*g*]chromeno- and pyrano[2,3-*g*]chromene-type compounds (Scheme 25; 17TL4323).

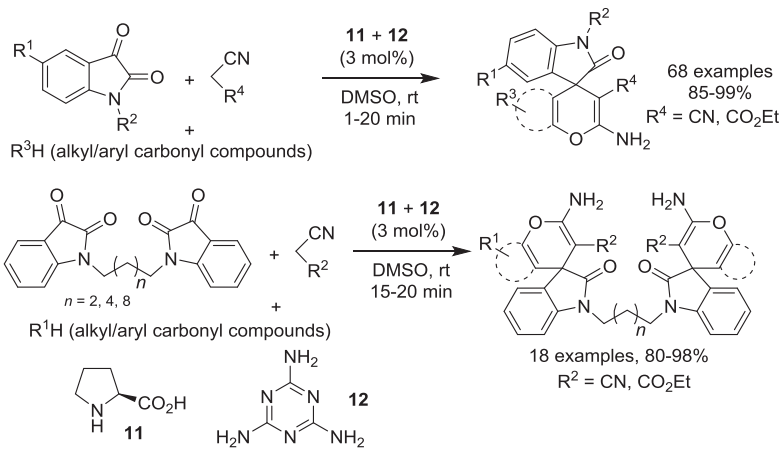
The synthesis of spiroxindole 2-aryl-4*H*-chromenes is accomplished through the reaction of 3-hydroxy-3-(2-hydroxyaryl)indolin-2-one with



Scheme 25

terminal arylacetylenes promoted by Lewis acids $Sn(OTf)_2$ or $FeCl_3$, in dichloromethane (17EJO3078). A three-component reaction of 6*H*-pyrrolo[3,2,1-*de*]acridine-1,2-dione with cyclohexane-1,3-diones/barbituric acid derivatives and malononitrile/ethyl cyanoacetate in the presence of triethylamine in refluxing ethanol leads to spiropyrroloacridone 2-aminotetrahydrochromene derivatives (17JHC2223). Electrocatalytic multicomponent reaction of dimedone with malononitrile and isatins in an undivided cell in the presence of sodium bromide as an electrolyte affords spiroxindole 2-aminotetrahydrochromene-3-carbonitriles in high yields (17JHC1763). Under solvent-free conditions, spiroindoloquinazoline cyclic—fused 2-aminotetrahydrochromene-3-carbonitriles are prepared in high yields from a three-component reaction of C—H-activated carbonyl compounds with malononitrile and tryptanthrin in the presence of ammonium acetate. The fused motifs result from the starting carbonyl compounds: 4-hydroxycoumarin, triacetic acid lactone, cyclohexane-1,3-dione, dimedone, and 3-methylpyrazol-5-one derivatives (17TL1947). The same (and other) type of carbonyl compounds underwent three-component reaction with isatins or bis-isatins and malononitrile/ethyl cyanoacetate using a mixture of L-proline **11** and melanine **12** to afford the corresponding spiroxindole or bis-spiroxindole chromene derivatives (Scheme 26; 17TL4200).

Under a dual catalyst system of 3-cyano-1-methylquinolinum photocatalyst and cobaloxime, intramolecular alkoxylation of 3-arylpropanols furnishes A-ring—substituted chromans (17OL2206). A three-step

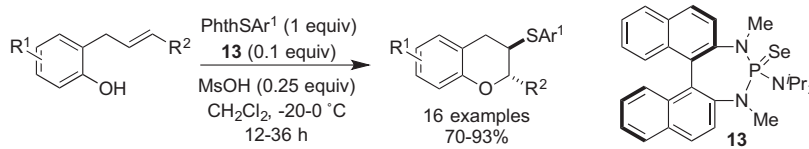


Scheme 26

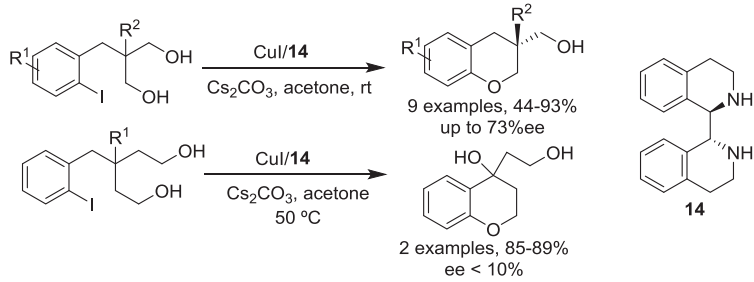
sequence involving a Heck cross-coupling reaction of 2-iodophenols with allylic alcohols followed by reduction and Mitsunobu cyclization gives 2-alkyl/arylchromans (17S657). 2-Aryl-3-sulfonylchromen-2-ols, formed from Knoevenagel condensation of β-ketosulfones and salicylaldehydes promoted by NH_4OAc, undergo $NaBH_4$ promoted regio- and stereo-controlled reduction to provide 2-aryl-3-sulfonylchromans (17JOC12631). A series of 2-substituted-3-sulfenoarylchromans are produced from sulfenocyclization reaction of 2-(prop-2-en-1-yl)phenols with electrophilic sulfanophthalimides using a BINAM-based phosphoramide Lewis catalyst **13** (Scheme 27; 17JOC3192).

Cinchona alkaloid-catalyzed enantioselective [4+2] annulation reactions of allenic esters and *o*-quinone methides, generated in situ from 2-(tosylmethyl)phenols (17AGE3689) or *O*-protected 2-(1-aryl-1-chloromethyl)phenols (17JOC5433) provide 2-methylene-4-substituted chromans. Further derivatives can be prepared starting from *o*-quinone methides and allenic ketones in the presence of a phosphine catalyst (17OL4126). High diastero- and enantio-selectivity is achieved in the [4+2] cycloaddition reactions of aromatic vinyl sulfides with *o*-quinone methides, generated in situ from *o*-hydroxybenzyl alcohols, mediated by a chiral phosphoric acid (CPA) catalyst to afford 4-substituted 2-thioarylchromans (17OL2334). Various (*E*)-2-[3-bromo-1-hydroxy-4-(phenylselanyl)]but-3-en-1-yl)phenols underwent trimethylsulfoxonium iodide-catalyzed intramolecular 6-*exo*-*trig*-coupling reactions giving rise to 2-methyleneselanylchroman-4-ols (17T7200). Asymmetric desymmetrization of 2-(2-iodobenzyl)-1,3-diols and of 3-(2-iodoaryl)-1,5-diols, promoted by CuI and a chiral cyclic diamine ligand **14** gives, respectively, 3-substituted 3-(1-hydroxymethyl)chromans and 4-substituted 4-(2-hydroxyethyl)chroman-4-ols in moderate to excellent yields and with modest enantioselectivity (Scheme 28; 17JOC1458).

Palladium(0)-catalyzed silaborative carbocyclization of 1-ethynyl-2-allyloxybenzenes with (chlorodimethylsilyl)pinacolborane provides chromans substituted with silicon and boron functions at C-3 and C-4,



Scheme 27



Scheme 28

respectively (17OL308). Prenyloxy- and allyloxy-benzenes undergo carbocyclization reactions with PhSeCl in the presence of triethylamine as base to produce 4,4-dimethyl- and 4-phenyl-3-phenylselenochromans, respectively (17TL371). A squaramide-containing tertiary amine base bifunctional organocatalyst confers high enantioselectivity on the vinylogous Michael–Michael cascade reactions of 3-alkylidene oxindoles with ethyl 3-(2-nitrovinylaryloxy)prop-2-enoates to give polysubstituted chromans (17JOC7317). Further derivatives are synthesized via enantioselective annulation of acyclic enecarbamates with *o*-quinone methides, generated in situ from *o*-hydroxybenzyl alcohols, mediated by a BINOL-based phosphoric acid catalyst (17OBC7272). Iron(III) chloride-promoted both the DA reaction of alkenes or 1,3-dicarbonyl compounds with *o*-quinone methides, generated in situ from *o*-hydroxybenzyl alcohols, and a multicomponent reaction of phenols with aldehydes/acetals and alkenes (17OL1878). A range of 3-aroyl-2-aryl-4-ethynylchromans were prepared from [4+2] cycloaddition reactions of chalcones with quinone methides, derived from trimethylsilyl (TMS) protected acetylene benzyl acetates **15**, mediated by platinum(IV) chloride (Scheme 29; 17JOC2672).

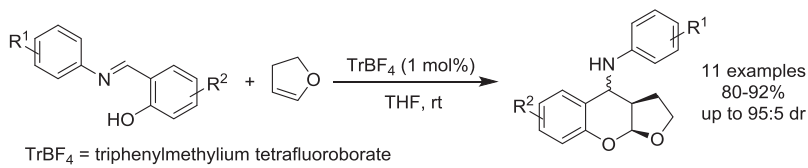
DMAP-catalyzed addition/(4+2) annulation reactions of δ-acetoxy allenoate with 2-hydroxybenzylidene 1,3-dicarbonyl compounds carried in the presence of cesium carbonate in chloroform affords 3,4-dihydro-2*H*-

Scheme 29

pyrano[3,4-*c*]chromans in moderate to good yields (17OBC4807). The synthesis of pyran-2-one[3,4-*c*]chromans is achieved in three steps via 6-*exo*-*trig*-Michael addition-lactonization reactions of (*E*)-2-[2-(4-oxo-4-alkyl/arylbut-2-en-1-yloxy)aryl]acetic acids promoted by isothiourea tetramisole (17CC2555). Formal [4+2] cycloaddition reactions of alkyl-quinones with 1,3-disubstituted indoles in the presence of triethylamine produce a variety of indolo[2,3-*b*]chromans (17OBC3472). High yields of indan-1-one[2,3-*c*]chromans are accomplished in the domino reaction of 2-(3-oxobut-1-ynyl)benzaldehydes with phenols mediated by PTSA or *p*-chlorophenylsulfonic acid (17T3310). A wide range of 4-amino-substituted furo[2,3-*b*]chromans are obtained through the reaction of salicylaldimines with 2,3-dihydrofuran catalyzed by triphenylmethylium tetrafluoroborate (Scheme 30; 17EJO3996), Brønsted acids of anionic chiral cobalt(III) complexes (17OBC9077) and $NaHSO_4$ supported on silica gel (17SC2352). Asymmetric [4+2] cycloaddition reaction of *o*-quinone methides, generated from salicylaldehydes, with alk-3,5-dien-1-ols mediated by a confined chiral imidodiphosphoric acid catalyst furnishes furo[3,2-*c*]chromans in high yields and with excellent diastereo- and enantio-selectivities (17AGE4936).

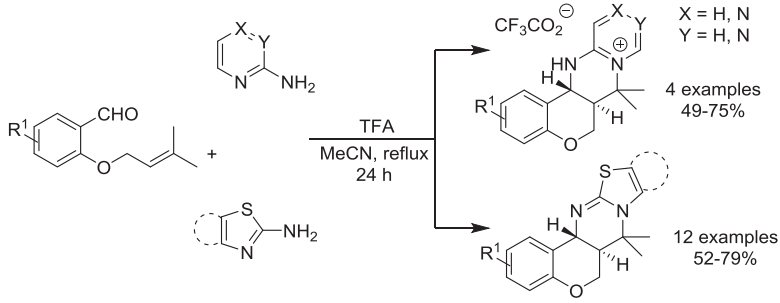
Several 2,8-dioxabicyclo[3.3.1]nonane derivatives arise from the reaction of 3-arylpropiolaldehydes or α,β-dibromocinnamaldehydes with two equivalents of phloroglucinol catalyzed by PTSA in acetonitrile (17TL4609) and of 2-hydroxychalcones with dimedone, 4-hydroxycoumarin, 2-hydroxynaphthoquinone, 2-naphthol, or 1-naphthol, using the sulfonated polystyrene resin Amberlyst-15, as heterogeneous catalyst (17SC2195). Diaza-DA reaction of *o*-prenyloxybenzaldehydes with aromatic 2-aminoazines carried out in the presence of trifluoroacetic acid in refluxing acetonitrile provides a series of polyheterocyclic-fused chromans (Scheme 31; 17CEJ4137).

Stereoselective intramolecular dearomatizative [4+2] cycloaddition reactions of 2-{2-[2-(naphth-2-ol-1-yl)ethynyl]phenyl}benzofurans using a catalytic amount of triethylamine in chloroform affords polycyclic



$TrBF_4$ = triphenylmethylium tetrafluoroborate

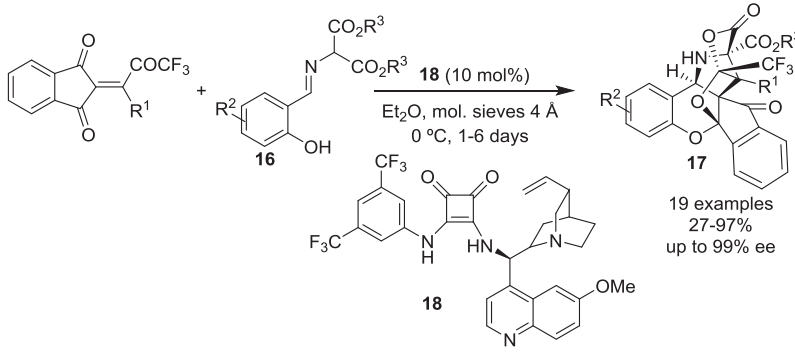
Scheme 30



Scheme 31

benzofuran-indane-fused benzochromans (17EJO6914). Low loading of the catalyst Cp*Rh(III) is applied in the coupling reaction of *N*-phenoxyacetamides with 7-azabenzonorbornadienes to give polycyclic-fused chromans (17AGE1381). Rhodium-catalyzed C−H activation/annulation reactions of naphthalene aldehydes with internal alkynes provide a series of four-ring-fused phenalenyl-based pyrylium cations in moderate to good yields (17AGE13094). High yields and enantioselectivity are achieved in the palladium(II)-mediated cyclization of aniline-tethered alkynyl cyclohexadienones to afford indole-fused dihydrochroman derivatives (17AGE14698). Structurally complex products **17**, bearing five quaternary stereocenters, are obtained from azomethine imine **16** and 2-benzylidene indane-1,3-diones in the presence of a cinchona alkaloid−derived organocatalyst **18** (Scheme 32; 17CC7649).

Under catalyst-free conditions, thermal cyclization of 5-(2-aryloxyarylidene)barbituric acid and its derivatives occurs at 118 to 240 °C to form 3-spiropyrimidine 2-arylchromans (17T542). A synergistic



Scheme 32

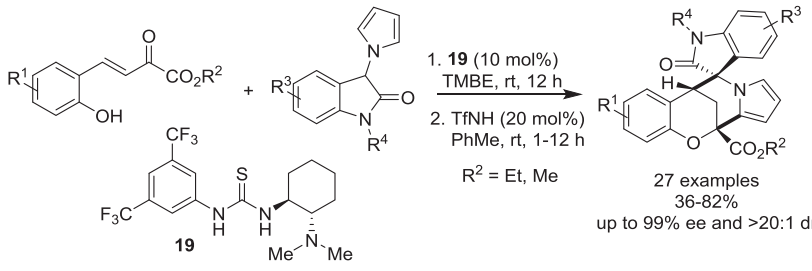
Au(I)/Sc(OTf)$_3$-catalyzed [4+2] cycloaddition reaction of *o*-quinone methides, generated in situ from *o*-hydroxybenzhydryl alcohol, with alkynyl alcohols or amides gives in high yields and excellent diastereoselectivity 2-spirofuran/benzofuran/pyrrolo/isoindole chromans (17OL2526). A diastereoselective Michael addition/ketalization sequence of 2-hydroxychalcones with 3-hydroxyoxindoles catalyzed by TfOH in ethyl acetate produced various spiroxindole methanobenzodioxepines, in moderate to excellent yields (17CC11201). Bridged and spiroheterocyclic skeletons, bearing a chroman moiety, are achieved by an asymmetric Michael/hemiketalization/oxa-Pictet–Spengler cyclization reaction of ethyl/methyl 4-(2-hydroxyaryl)-2-oxobut-3-enoates with pyrrol-3-ylindolin-2-ones cooperatively catalyzed by a Takemoto thiourea catalyst **19** and a triflimide (Scheme 33; 17OL6626).

6.4.2.3 [2]Benzopyrans and Dihydro[2]benzopyrans

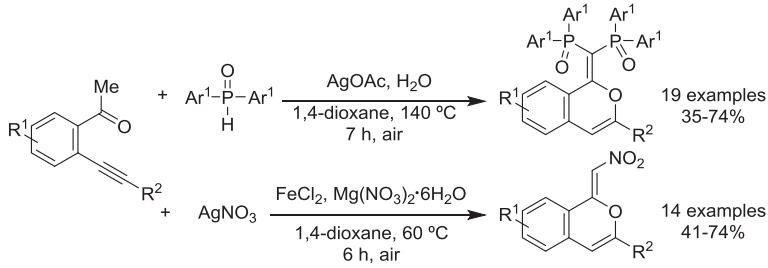
6.4.2.3.1 Isochromenes and Isochromans

Under the dual catalysis of Cu(OTf)$_2$ and a chiral cationic ruthenium-diamine complex, a series of *o*-(alkynyl)aryl ketones underwent asymmetric hydrogenation to give polysubstituted 1*H*-isochromenes in high yields and enantioselectivity (17AGE4135). Further *o*-(alkynyl)aryl ketones undergo selective C–H functionalization and 6-*endo*-*dig*-oxo-cyclization reactions using different radical sources: in the presence of diphenylphosphine oxides and silver acetate as both catalyst and oxidant affords biphosphonylated isochromenes, while the presence of silver nitrate as both catalyst and nitrating reagent provides nitro-terminated isochromenes (Scheme 34; 17OL754).

The synthesis of 1,3-disubstituted 1*H*-isochromenes is achieved via tandem reactions of a wide range of *o*-alkynylbenzaldehydes with thiophenes mediated by a gold(III) catalyst in acetonitrile (17SC463), with



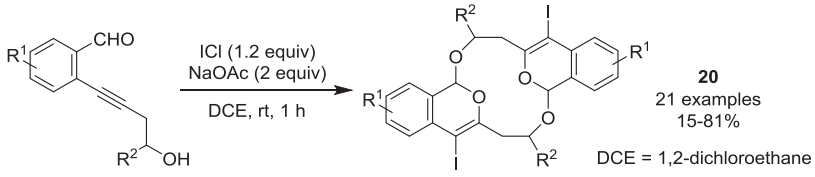
Scheme 33



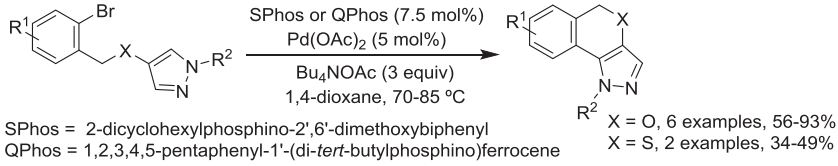
Scheme 34

trialkyl orthoformates mediated by a racemic 1,1′-binaphthalene-2,2′-diol (BINOL)-gold(III) complex (17AGE3074), with enaminones carried out in the presence of $AgNO_3$ in *N*,*N*-dimethylacetamide (17T5731), with substituted ketones promoted by silver triflate (17EJO1425) or under dual catalysis of silver carbonate and diphenyl phosphate in *n*-hexane (17S1243). Electrophilic cyclization and intermolecular acetalization of 2-(4-hydroxy-1-yn-1-yl)benzaldehyde derivatives with ICl and sodium acetate in 1,2-dichloroethane delivers diiodinated macrocyclic bis(isochromene)-type compounds **20** (Scheme 35; 17JOC10641).

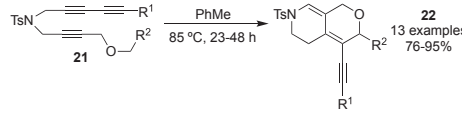
Examples of pyrazole-fused (thio)isochromenes were obtained from palladium(II)-catalyzed cyclization of 4-[(2-bromoaryl) (thio)oxy]-1-methyl-1*H*-pyrazoles using 2-dicyclohexylphosphino-2′,6′-dimethoxybiphenyl (SPhos) or 1,2,3,4,5-pentaphenyl-1′-(di-*tert*-butylphosphino) ferrocene (QPhos) as ligand (Scheme 36; 17TL4587). Thermally promoted cyclization of ynamide-tethered 1,3,8-triynes **21** affords isochromene-type



Scheme 35



Scheme 36

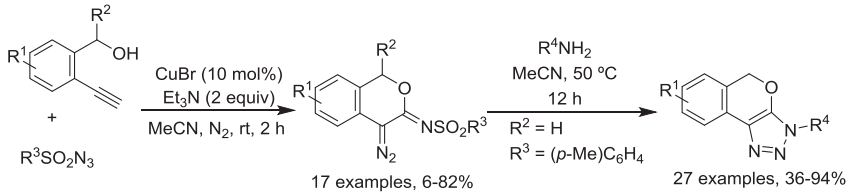


Scheme 37

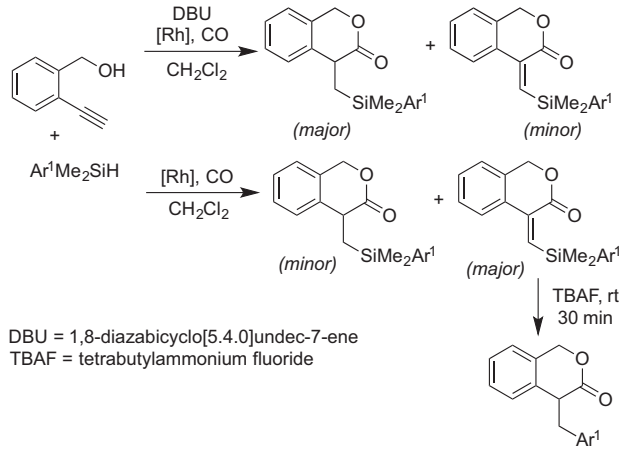
compounds **22** (Scheme 37). The key steps involve the formation of a strained keteniminium or a biradical intermediate followed by a 1,5-hydride or 1,5-hydrogen shift (17CEJ8161). The synthesis of quinolino[3,4-*c*]isochromenes can be accomplished through copper(II) triflate-mediated cascade annulation reactions of alkynols with 2-azidobenzaldehydes, in moderate to good yields (17JOC7032).

Copper(I)-catalyzed cascade reaction of (2-ethynylphenyl)methanols with sulfonyl azides carried out in the presence of CuBr and triethylamine in acetonitrile provides 4-diazoisochroman-3-imines which can react with primary amines to afford triazole-fused isochromenes (Scheme 38; 17CC3769). Several 1,1,4-trisubstituted and some 1,1,4,4-tetrasubstituted isochromans were prepared via tandem condensation/[1,3]-hydride transfer/Friedel–Crafts reaction of 2-arylacetaldehydes with substituted alcohols mediated by $Sc(OTf)_3$ in refluxing toluene (17CC10652).

The synthesis of isochroman-3-one derivatives through rhodium-mediated silylcarbocyclization of 2-ethynylbenzyl alcohol with different arylsilanes are conditions-dependent: in the presence of DBU, 4-(aryldimethylsilylmethyl)isochroman-3-ones are the main products, generated from hydrogenation of the corresponding methyleneisochroman-3-ones; without base, (*Z*)-4-[(aryldimethylsilyl)methylene]isochroman-3-ones are obtained as major products, which can undergo desilylation/aryl migration reactions by treatment with an excess of tetrabutylammonium fluoride to give 4-(arylmethyl)isochroman-3-ones (Scheme 39; 17EJO3473). Palladium-catalyzed intermolecular tandem cyclization of (2-hydroxymethylaryl)propargylic carbonates with various 2-iodobenzyl alcohols furnishes 3*H*-spiro



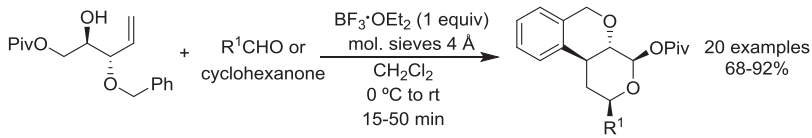
Scheme 38



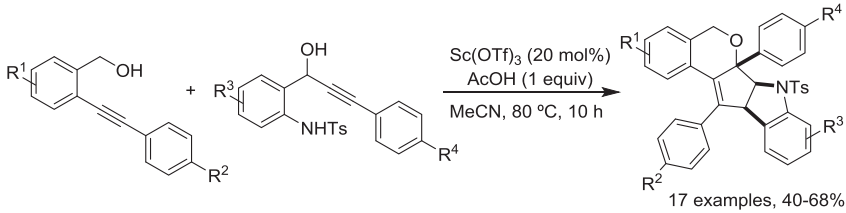
Scheme 39

[isobenzofuran-1,3′-isochromans]. It involves decarboxylative allenylpalladium formation, nucleophilic attack, arylpalladium addition, and intramolecular nucleophilic attack (17OBC2403).

A series of benzo[*f*]isochromans were synthesized via one-pot 6-*endo*-*dig*-alkynyl-Prins cyclization, Friedel—Crafts alkenylation, dehydration, aromatization cascade reaction of 1-arylhex-3-yne-2,6-diol derivatives with various aldehydes carried out in the presence of $BF_3 \cdot OEt_2$ in dichloromethane (17OBC7584). A few examples of 3-chloropyran-2-one- or 3-chloropyridin-2-one-fused isochromans arise from direct arylation of 4-benzyloxy-3-chloro(pyran-2-ones or pyridin-2-ones), respectively, mediated by palladium(II) acetate, TBAB, and potassium carbonate in THF (17EJO5119). Stereoselective synthesis of tetrahydropyran-fused isochromans occurs through Prins cyclization of D-mannitol-derived homoallylic alcohols (Scheme 40; 17EJO5986) or other homoallylic alcohols (17SL1346) with several aldehydes or cyclohexanone in the presence of $BF_3 \cdot OEt_2$. A cascade reaction involving alkynol cycloisomerization, intermolecular substitution with 1-(2-tosylaminoaryl)-3-arylprop-2-ynols, intermolecular addition with alkynols, and consequent cyclizations



Scheme 40



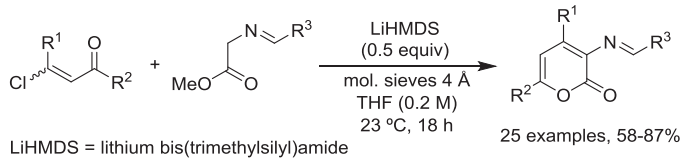
Scheme 41

leads to a tricyclic-fused isochromans, in moderate to good yields (Scheme 41; 17CC8608).

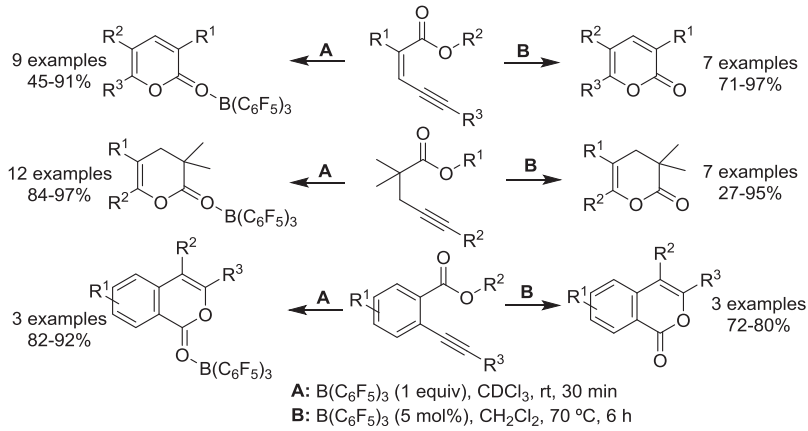
6.4.2.4 Pyranones

An NHC catalyst promotes the reaction of aroyl-substituted nitromethanes with enals to give a range of 4,6-disubstituted-2*H*-pyran-2-ones in moderate to good yields. It involves an addition, elimination, and lactonization sequence (17S121). 5,6-Disubstituted 2*H*-pyran-2-ones result from 6-*endo*-*dig*-transesterification and subsequent alkenylation of enynoates with electron-deficient alkenes promoted by $PdCl_2$ as catalyst and XPhos as ligand (17TL1387). Highly functionalized 3-imino-2*H*-pyran-2-ones arise from a direct conjugate addition reaction of imino esters to β-chlorovinyl ketones in the presence of substoichiometric amount of lithium bis(-trimethylsilyl)amide (Scheme 42; 17OL4904). A series of alkynyl carboxylic acids and esters underwent intramolecular cyclization in the presence of $B(C_6F_5)_3$ to provide polysubstituted 2*H*-pyran-2-ones, 3,4-dihydro-2*H*-pyran-2-ones, or isocoumarins. Using stoichiometric amounts of the catalyst, a range of boron-oxygen adducts are obtained while using catalytic amounts of $B(C_6F_5)_3$; the boron-free adducts were isolated (Scheme 43; 17AGE11995).

Several examples of pyrazole-fused 2*H*-pyran-2-ones were shown to arise from three-component reaction of ethyl acetoacetate, substituted hydrazines, and 3-oxopentanedioic acid dimethyl ester carried out in the presence of urea and aqueous ethanol at 80°C (17T164). The synthesis of



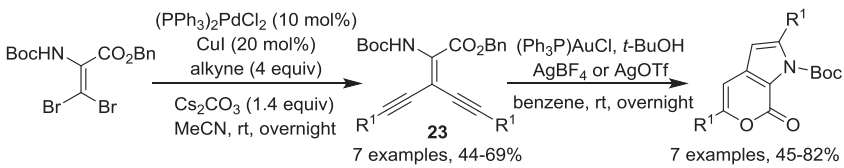
Scheme 42



Scheme 43

pyrrole-fused 2*H*-pyran-2-ones is accomplished through one-pot two-step nucleophilic 5-*endo*-*dig* and 6-*endo*-*dig*-cyclizations of dyines **23**, prepared via double Sonogashira cross-coupling reaction of β,β-dibromodehydroalanine derivatives, promoted by a cationic gold complex (Scheme 44; 17OBC7290). Rhodium(III)-catalyzed coupling reactions of indoles with diazo esters carried out in the presence of $Zn(OTf)_2$ and acetic acid in dichloroethane at 100°C affords indole-fused pyran-2-ones in moderate to good yields (17OL6184). Several heterocyclic carboxylic acids undergo ruthenium(II)-promoted oxidative coupling reaction with internal alkynes to provide a range of heterocyclic-fused pyran-2-ones. These heterocyclic systems include thiophene, furan, pyrrole, pyridine, and quinolone units (17OBC8904).

A wide range of polysubstituted 3,4-dihydro-2*H*-pyran-2-ones were enantioselectively achieved through [4+2] cycloaddition reaction of aliphatic aldehydes with β,γ-unsaturated α-ketoesters promoted by a proline-derived catalyst and subsequent oxidation with pyridinium chlorochromate (PCC) (17TA1591); Knoevenagel condensation/Michael addition/hemiacetalization reaction of aldehydes with β-ketoesters mediated by a proline

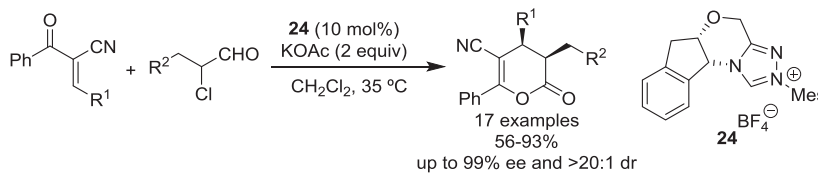


Scheme 44

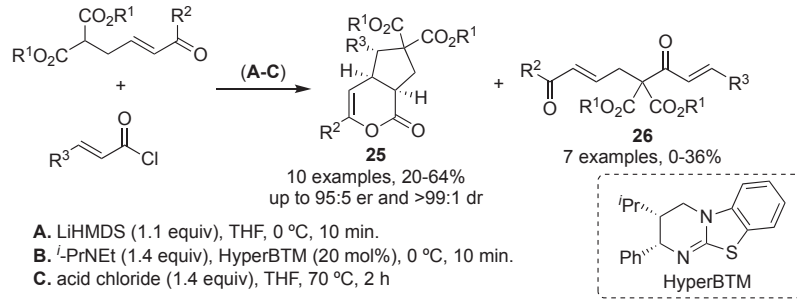
catalyst followed by oxidation with Dess–Martin periodinane (17TA153); NHC-catalyzed redox [4+2] hDA reaction of α-aroyloxyaldehydes with β,γ-unsaturated α-ketoesters or with α,β-unsaturated γ-ketoesters (17TA355), and NHC-catalyzed asymmetric [4+2] annulation reaction of (*E*)-2-benzoyl-3-arylacrylonitriles with α-chloroaldehydes carried out in the presence of potassium acetate (Scheme 45; 17S4861). Further enantiopure derivatives arise from a one-pot sequential three-component reaction of β-nitroalkenes with two different aldehydes, employing two NHC catalysts (17OL6076).

Various 4-spiroxindole 3,4-dihydro-2*H*-pyran-2-ones are produced from NHC-promoted formal [3+3] annulation reactions of isatin-derived α,β-unsaturated acids with 1,3-dicarbonyl compounds (17T3249). Low-loading of another NHC catalyst is applied to the enantioselective synthesis of pyrrolidone-fused 3,4-dihydro-2*H*-pyran-2-ones, via [4+2] cycloaddition reactions of pyrrolidone-derived cyclic enones with α-haloaldehydes, carried out in the presence of sodium bicarbonate and THF as solvent (17CC6875). α,β-Unsaturated acyl ammonium intermediates, generated in situ from addition of isothiourea catalyst HyperBTM into α,β-unsaturated acid chlorides, underwent a Michael–Michael–lactonization cascade sequence with enone-malonates to afford mainly cyclopentane-fused 3,4-dihydro-2*H*-pyran-2-ones **25** and as minor product, the acylation product **26** of the malonate and the acid chloride (Scheme 46; 17S409). A dirhodium(II) 2-phthalimide carboxylate mediates intramolecular cyclopropanation of (*Z*)-1,3-dienyl aryldiazoacetates giving access to cyclopenta[2,3]cyclopropa[1,2-*c*]pyran-4-ones with high regio-, diastereo- and enantio-selectivities (Scheme 47; 17OL1306).

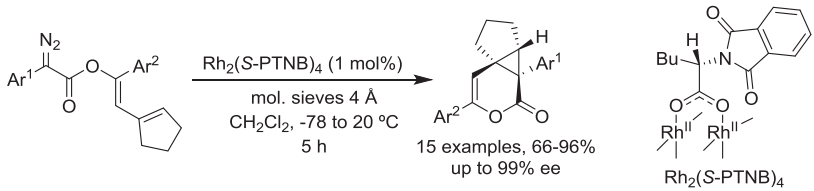
Some examples of 5-alkylated 5,6-dihydro-2*H*-pyran-2-ones were attained through cross-aldol reactions of formaldehyde with alkyl aldehydes and subsequent (*Z*)-selective Horner–Wadsworth–Emmons reaction and lactonization, conducted in the presence of triethylsilyl diphenylprolinol as catalyst (17OL3592). Highly substituted 5,6-dihydro-2*H*-pyran-2-ones are produced from the *anti* selective aldol reaction of chiral alkyl



Scheme 45



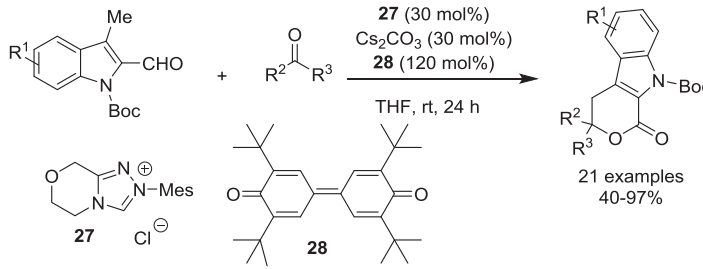
Scheme 46



Scheme 47

(*E*)-4-benzyloxy-3-(pyrrolidin-1-yl)but-2-enoates with aliphatic aldehydes and using lithium diisopropylamide (LDA) as base in THF (17TA1573). Further derivatives were prepared from the reaction of substituted styrylmalonates with aromatic aldehydes in the presence of $BF_3 \cdot OEt_2$ (17OL3731). An NHC-bound *o*-quinodimethane intermediate is generated under oxidative conditions starting from substituted 2-formyl-3-methylindoles to react with various activated ketones to afford indole-fused 6,6-disubstituted 5,6-dihydro-2*H*-pyran-2-ones (Scheme 48; 17JOC13342).

Cerium-catalyzed aerobic coupling reaction of β-oxoesters with enol acetates gives access to a mixture of tetrahydro-2*H*-pyran-2-ones **29** as main



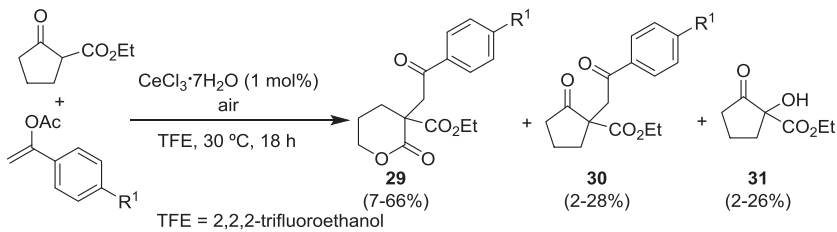
Scheme 48

products and also 1,4-diketones **30** and α-hydroxylated derivative **31** as byproducts (Scheme 49; 17CEJ7245). One-pot microwave-assisted synthesis of 3-ylidenetetrahydro-2*H*-pyran-2-ones starting from (*Z*)-2-(bromomethyl)alk-2-enoates (derived from MBH reaction) has been accomplished in aqueous medium. The protocol includes regioselective base-mediated allylation of ethyl acetoacetate with (*Z*)-2-(bromomethyl)alk-2-enoates followed by decarboxylative hydrolysis, carbonyl reduction of the keto carboxylate intermediate, and acid-mediated cyclization of the resulting δ-hydroxy acids (17S667). A two-step strategy was applied to the synthesis of 5-sulfonyl tetrahydro-2*H*-pyran-2-ones starting from NaH-promoted Michael addition of β-ketosulfones to methyl acrylate derivatives in refluxing THF and subsequent $NaBH_4$-mediated stereoselective reduction/lactonization reaction of the δ-ketoesters formed, in refluxing MeOH (17T46). A couple of 4-hydroxy-6-substituted tetrahydro-2*H*-pyran-2-ones were obtained from the cyclization of 3,5-dihydroxyalkanoates in the presence of PTSA in dry dichloromethane (17TA181).

The synthesis of 5,6-disubstituted 4-spiroindolin-2-one tetrahydro-2*H*-pyran-2-ones occurs by a two-step approach, involving palladium- or iridium-mediated Tsuji–Trost allylation of oxindoles with 3-allylindolecarbonate and subsequent dihydroxylation followed by lactonization promoted by PTSA (17T888).

Nickel-catalyzed cycloaddition reactions of oxetan-3-ones with unsymmetrical alkynes furnishes a mixture of 2*H*-pyran-3(6*H*)-one regioisomers, the regioselectivity being determined by the alkyne substituents (17S3582).

Electrophilic condensation of β-ketoesters mediated by triflic anhydride gives direct access to 4*H*-pyran-4-ones in moderate to good yields (17OBC680). Through asymmetric aldol/vinylogous aldol reaction of 4-aroyloxypent-3-en-2-ones with arylaldehydes and subsequent cyclization that 2,6-disubstituted 2,3-dihydro-4*H*-pyran-4-ones were prepared. This



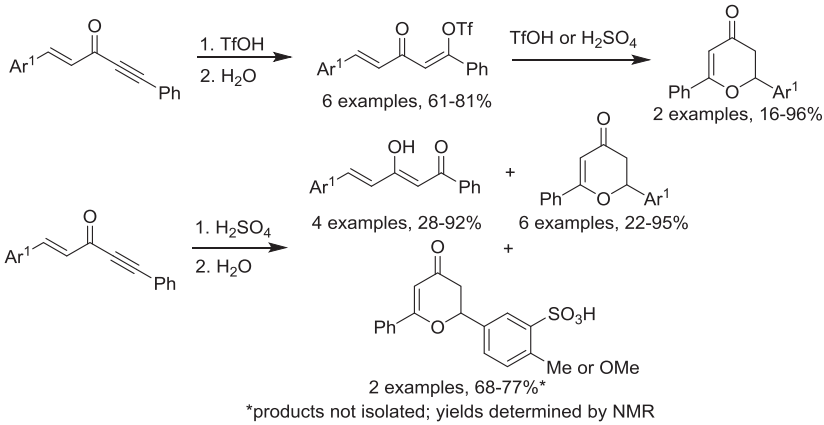
Scheme 49

procedure occurs in the presence of silicon tetrachloride, (*S*)-2,2′-bis(diphenylphosphino)-1,1′-binaphthyl dioxide and *N*,*N*-diisopropylethylamine (DIPEA) in dichloromethane at −60°C (17CPB989).

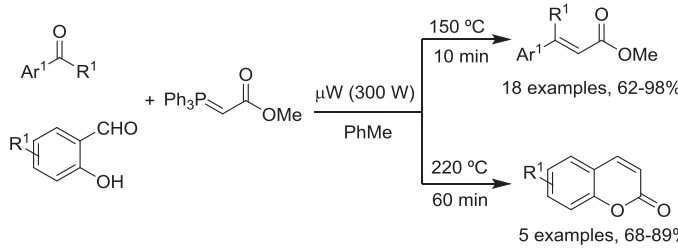
Treating diaryl-substituted cross-conjugated enynones with triflic acid led to the corresponding vinyl triflates which undergo acid-promoted cyclization to afford 2,3-dihydro-4*H*-pyran-4-ones, whereas in the presence of sulfuric acid, a mixture of α,β-unsaturated 1,3-diketones, generated from vinyl sulfate hydrolysis during the aqueous work-up, and 2,3-dihydro-4*H*-pyran-4-ones are obtained (Scheme 50; 17EJO3635).

6.4.2.5 Coumarins

One-pot microwave-assisted Wittig reaction of aromatic aldehydes and ketones with a stabilized ylide, carried out in tolune at 150 °C, provides cinnamic acid derivatives, while the reaction of salicylaldehydes with the same ylide in toluene at 220 °C affords 3,4-unsubstituted coumarins (Scheme 51; 17EJO5204). Other salicylaldehydes underwent a condensation–ring opening–annulation cascade reaction with *N*-Boc-indolin-2-ones and benzofuran-2(3*H*)-ones promoted by tetramethylguanidine to give, respectively, 3-(2-aminoaryl)- and 3-(2-hydroxyaryl)coumarins (17OBC7505). One-pot regioselective Michael addition of aliphatic and aromatic thiols with alkenyl *p*-benzoquinones in methanol at −40 °C followed by cyclization furnishes 3-substituted 5-alkyl/aryl sulfides of coumarins in a one-to-two minute reaction time (17T2591). Further 3-substituted coumarins (3-acetyl, 3-cyano and ethyl 3-carboxylates) arise from Knoevenagel condensation and subsequent intramolecular cyclization



Scheme 50

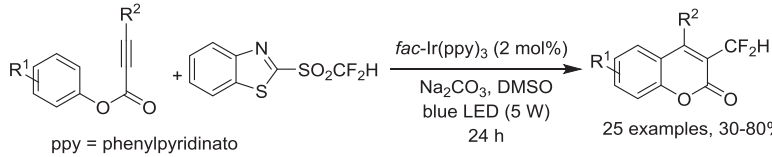


Scheme 51

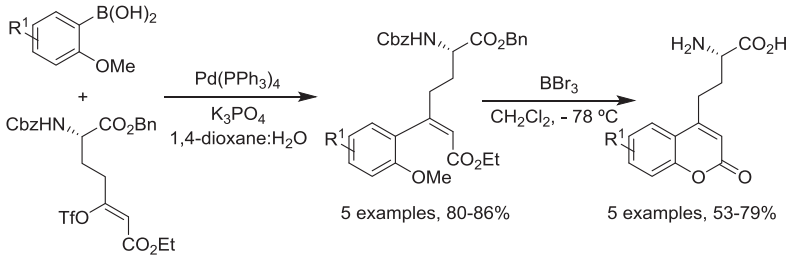
reactions of salicylaldehydes with ethyl acetoacetate/cyano ethyl acetate/diethyl malonate mediated by phenyliododiacetate (17TL3183) or carried out in ethanol at reflux and using ultrasonic irradiation (17S2677). In the last reaction conditions, the mixture of salicylaldehyde with Meldrum's acid provided coumarin-3-carboxylic acid, while the mixture of resorcinol with ethyl acetoacetate afforded 7-hydroxy-3-methylcoumarin (17S2677).

A range of 3-trifluoromethylcoumarins were achieved via a CuI--mediated cascade reaction of *o*-hydroxycinnamic esters with the Togni reagent as the CF_3 source, in DMF at 80°C (17EJO271). Through a visible light-mediated bromo radical addition/spirocyclization/ester migration cascade reaction, alkynoates react at room temperature with NBS to afford 3-bromo-4-substituted coumarins (17OBC8820). Using difluoromethyl sulfone instead of NBS, a range of 3-difluoromethyl-4-substituted coumarins are obtained (Scheme 52; 17OBC9057). Under dual catalysis of PPh_3 and iodine, one-pot esterification and cyclization reactions of salicylaldehydes and 2′-hydroxyacetophenones with aryl acetic acids at room temperature give, respectively, 3-arylcoumarins and 3-aryl-4-methylcoumarins (17SL825).

4-Substituted coumarins arise from palladium(0)-promoted coupling reactions of *o*-methoxyboronic acids with a glutamic acid-derived (*Z*) vinyl triflate followed by total deprotection with boron tribromide (Scheme 53; 17OL2797). Highly functionalized coumarins arise from an NHC-mediated



Scheme 52

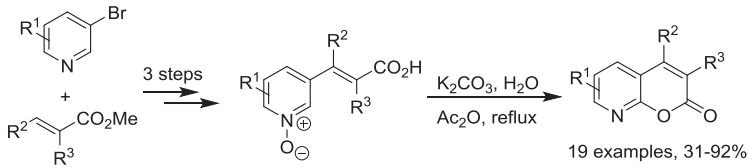


Scheme 53

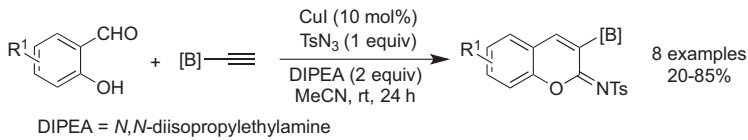
unusual formal [5+5] reaction of enals with methyl (*E*)-2-[5-oxofuran-2(5*H*)-ylidene]acetate in the presence of a quinone as oxidant and K_3PO_4 as base, in acetonitrile at room temperature (17OL6188).

A one-pot synthesis of 8-azocoumarins occurs via lactonization of pyridine *N*-oxides, obtained from Heck reaction of 3-bromopyridine derivatives with methyl acrylates followed by hydrolysis and chemoselective oxidation, in the presence of acetic anhydride, acting as both the solvent and the activation agent. The lactonization involves a conjugate addition, nucleophilic aromatic substitution, and elimination sequence (Scheme 54; 17OL984). Further derivatives can be obtained from three-component reactions of chalcones with ethyl 2-substituted acetates and ammonium acetate under ultrasonic irradiation and grinding conditions (17JHC2003).

Several 3-boryliminocoumarins are accomplished through reaction of salicylaldehydes with ethynyl (*N*-methyliminodiacetic acid) boronate, and tosyl azide carried out in the presence of CuI and DIPEA in acetonitrile at room temperature (Scheme 55; 17CEJ9711). Another copper(I)-mediated



Scheme 54



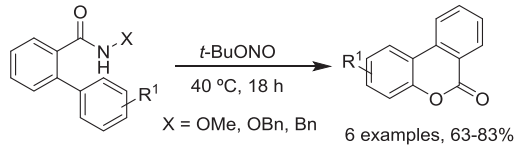
Scheme 55

three-component reaction of 2-hydroxybenzonitriles with aliphatic/aromatic terminal alkynes and tosyl/phenyl azide delivers a wide range of polysubstituted 4-aminoiminocoumarins (17EJO102).

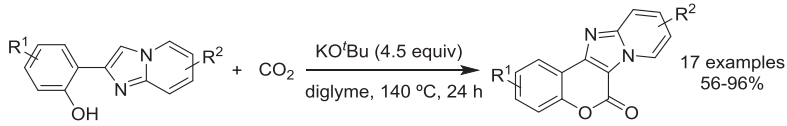
Palladium-mediated direct oxidative annulation of benzoic acids with phenols leads to a series of benzo[*c*]coumarins in moderate to good yields (17OL1326). More analogs arise from double C—H activation/oxygen insertion of 2-arylbenzaldehydes using a Cu(0)/Selectfluor catalytic system (17T154) and one-pot conversion of *N*-substituted 2-arylbenzamides in the presence of *t*-BuONO at 40 °C, in metal-, oxidant- and solvent-free conditions (Scheme 56; 17JOC5769).

Benzo[*c*]coumarins and thieno[*c*]coumarins are accessed through the reaction of methyl anthranilates and methyl 2-aminothiophene carboxylate, respectively, with sodium nitrite in HCl to afford the corresponding diazonium salts, which undergo Meerwein reaction with benzoquinone and subsequent reduction of the obtained arylquinones using either sodium sulfide in aqueous ethanol or zinc powder in glacial acetic acid (17SC2399).

Rhodium(III)-catalyzed C—H activation/cyclization reactions of benzamides with diazonaphthalen-2(1*H*)-ones carried out in the presence of silver acetate in toluene provides naphthalene-fused coumarins (17OL4002). It is through a transition metal-free lactonization process that various 2-(imidazo[1,2-*a*]pyridin-2-yl)phenols react with CO_2 to furnish imidazopyridine-fused coumarins. The reaction occurs in the presence of potassium *t*-butoxide and diglyme at 140°C (Scheme 57; 17OL396). A range of pyrazole-fused coumarins result from double C—H activation/oxygen insertion of 5-arylpyrazole-4-carbaldehydes using the Cu(0)/Selectfluor catalytic system (17T154). Ru(II)-catalyzed oxidative annulation of enamides with alkynes leads to highly functionalized pyrroles, which



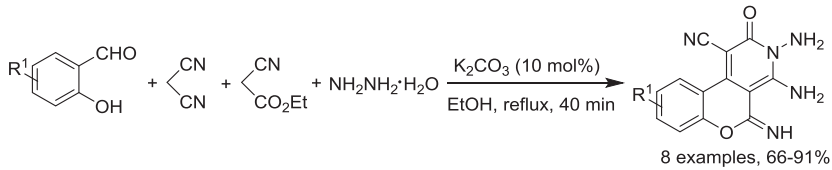
Scheme 56



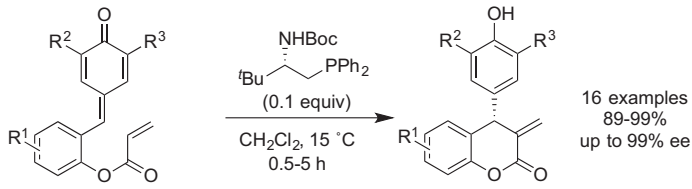
Scheme 57

undergo Suzuki–Miyaura reaction with boronic acids to give some examples of pyrrole-fused coumarins (17SL1715). Functionalized pyridin-2-one-fused iminocoumarins are synthesized through a four-component reaction of salicylaldehydes with malononitrile, ethyl cyanoacetate, and hydrazine hydrate in the presence of potassium carbonate in refluxing ethanol (Scheme 58; 17H(94)1143).

An asymmetric organocatalyzed oxa-Michael–Michael cascade reaction of 2-hydroxycinnamaldehydes with *trans*-nitroolefins in the presence of Hantzsch ester and *p*-methylbenzoic acid followed by oxidation with PCC led to 3-(1-aryl-2-nitroethyl)-3,4-dihydrocoumarins (17JOC4774). High yields and enantioselectivity of 4-aryl-3-methylene-3,4-dihydrocoumarins are achieved through intramolecular vinylogous Rauhut–Currier reaction of *p*-quinone methides, using a bifunctional chiral amine-phosphine catalyst (Scheme 59; 17OL3207). Several examples of 3-aminosubstituted 4-aryl-3,4-dihydrocoumarins arise from the NHC-mediated reaction of phenols with enals (17OL1318), reaction of phenols with azlactones in the presence of $AlCl_3$ (17JOC5524), reaction of *o*-quinone methides, generated in situ from 2,6-di-*tert*-butyl-4-(2-hydroxybenzylidene)cyclohexa-2,5-dien-1-one, with azlactones mediated by a CPA (17OBC8743), reaction of *o*-quinone methides, generated in situ from 2-(1-tosylalkyl/aryl) phenols, with azlactones promoted by a bifunctional squaramide catalyst (17CC3531), and the reaction of α-chloro aldehydes with *o*-hydroxybenzhydryl amines, to generate respectively, azolium ester enolates and *o*-quinone methides, under an NHC and acid cooperative catalytic system (17OL5892).



Scheme 58

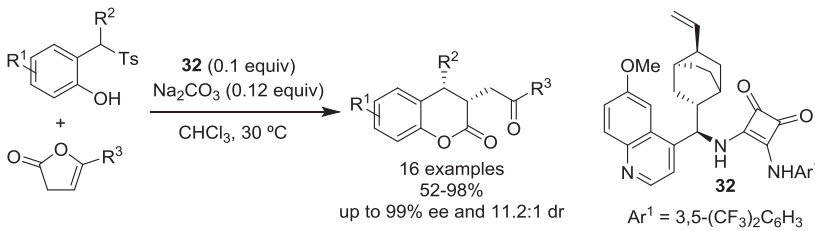


Scheme 59

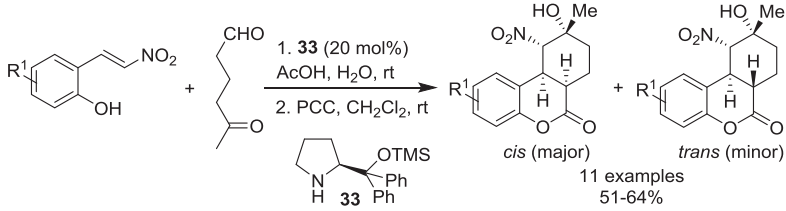
A thiourea-derived cinchona alkaloid catalyzes the reaction of cyclohexane-1,3-diones with 3-arylidenefuran-2(3*H*)-ones (17OBC7286), and NHC catalyst promotes the reaction of cyclohexane-1,2-dione with ynals (17OBC1329) to afford 3,4-dihydrocoumarin-type compounds in high yields and enantioselectivities. 3-Aryl-4-trifluoromethyl-3,4-dihydrobenzocoumarins are prepared in high yields and with excellent diastereo- and enantio-selectivies from Friedel—Crafts alkylation/lactonization reactions of naphthols with *N*-Boc protected 3-trifluoroethylidene oxindoles promoted by low loading (2.5 mol%) of a quinine-derived squaramide catalyst (17AGE338). A different quinine-derived squaramide catalyst **32** promotes the enantioselective synthesis of 3-substituted 4-aryl-3,4-dihydrocoumarins through regioselective α-addition of 2-(1-tosylaryl)naphthols with γ-substituted deconjugated butenolides (5-substituted furan-2(3*H*)-ones) in the presence of sodium carbonate in chloroform at 30 °C (Scheme 60; 17AGE4006).

The synthesis of quinolone-fused 3,4-dihydrocoumarins is attained through intramolecular Povarov-type reaction of aldehydes tethered to alkenes with primary anilines mediated by a CPA (17AGE10573). Organocatalyzed enantioselective Michael–acetalization–Henry reaction of 2-hydroxynitrostyrenes with 5-oxohexanal and subsequent oxidation with PCC furnishes *cis*- and *trans*-cyclohexane-fused 3,4-dihydrocoumarins, the *cis* isomers being the major products (Scheme 61; 17JOC12840).

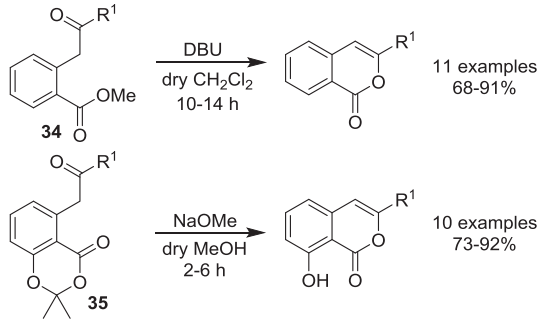
Treating ketoesters **34** with DBU in dry dichloromethane gives 3-substituted isocoumarins, while the reaction of ketolactones **35** with sodium methoxide in dry methanol leads to 8-hydroxy-3-substituted isocoumarins (Scheme 62). This route is also applied to the synthesis of 3-glycosylated derivatives (17EJO34). Palladium(II)-catalyzed carbonylation reactions of 1-(2-iodo-3,5-dimethoxyphenyl)alkan-2-ones in the presence of sodium acetate affords a couple of 6,8-dimethoxy-3-pentylisocoumarin derivatives (17H(94)1542), while 1-aryl/heteroaryl-2-(2-bromoaryl)ethan-1-ones in the presence of 1,1′-bis(diphenylphosphine)ferrocene as ligand and



Scheme 60



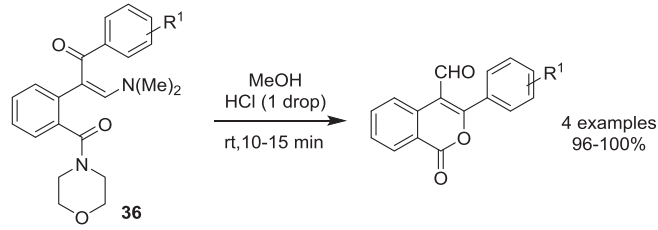
Scheme 61



Scheme 62

phenyl formate as CO source provides 3-aryl/heteroarylisocoumarins (17OBC1628). Other 3-alkyl/arylisocoumarins arise from palladium(II)-mediated sequential nucleophilic addition/oxidative annulation reaction of benzoic acids with bromoalkyl/arylalkynes (17OL4440), copper(I)-promoted annulation reactions of 2-bromobenzoic esters(17TL2433) and 2-iodobenzoic acids (17EJO6131) with terminal alkynes, and copper nanoparticles catalyzed coupling reaction of 2-halobenzoic acids/amides with 1,3-diketones (17TL3164).

Annulation reactions of carboxylic acids with alkynes mediated by the Cp*Co(III) catalyst affords a range of 3,4-disubstituted isocoumarins in good to excellent yields (17OL2544). A few examples of 3-aryl-4-formylisocoumarins have been accomplished through cyclization of enaminoketones of 2′-carboxamidodeoxybenzoins **36** achieved with one drop of HCl in methanol (Scheme 63; 17TL245). The synthesis of 4-halo-3-substituted isocoumarins is achieved from the reaction of phenyl 2-alkynylbenzoates with zinc halide, TBAB, and oxone in a 1:1 mixture of DCE:H_2O (17OBC4867). Iodolactonization of 2-alkynylbenzoic acids is dependent on the nature of the ionic liquid medium: using *N*-ethyl-*N*-methylmorpholinium dicyanamide [$Mor_{1,2}N(CN)_2$], an anti-5-*exo*-*dig*-cyclization occurs to afford mainly (*E*)-3-(iodomethylene)

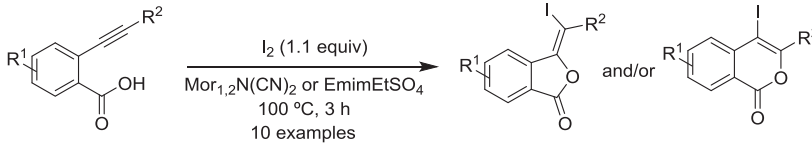


Scheme 63

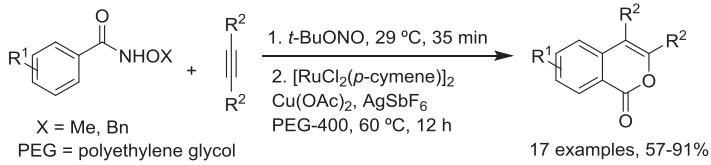
isobenzofuran-1(3*H*)-ones, while the use of 1-ethyl-3-methylimidazolium ethyl sulfate ($EmimEtSO_4$) tended to favor the 6-*endo*-*dig*-cyclization to give 4-iodo-3-substituted isocoumarins (Scheme 64; 17OBC4831).

A variety of 3,4-disubstituted isocoumarins are available through rhodium(III)-catalyzed annulation reactions of aryl carboxylic acids with alkynes (17EJO341) and from palladum(II)-mediated reactions of 2-halobenzoates with ketones (17JOC8296). In the last case, the addition of iodide anions to the reaction mixture played an important role in the yield and selectivity when 2-bromobenzoates were used as starting materials (17JOC8296). Under Ru(II)/PEG-400 catalytic system, the reaction of *N*-methoxy/benzyloxy aromatic amides with alkynes and using *t*-BuONO as oxygen source provided 3,4-disubstituted isocoumarins (Scheme 65; 17JOC5769).

A series of indole type—fused isocoumarins were synthesized through domino reactions of 2-amino-1,4-naphthoquinones with ninhydrin in the presence of PTSA in acetic acid (17H(94)237). It is through a



Scheme 64



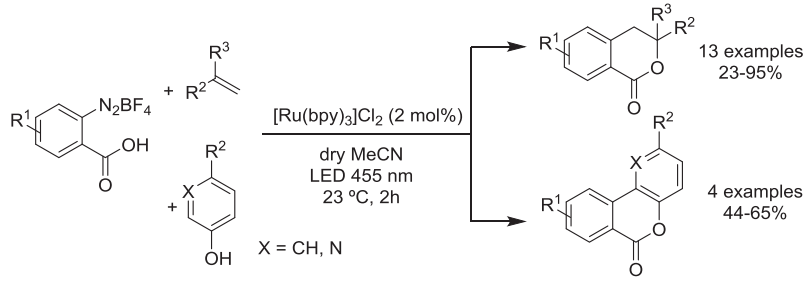
Scheme 65

rhodium(III)-catalyzed C—H activation of benzoic acids and subsequent intermolecular esterification reaction with cyclic 2-diazo-1,3-diketones that a variety of cyclohexanone-fused isocoumarins were prepared (17JOC2081). Photocatalytic Meerwein synthesis of 3-substituted or 3,3-disubstituted 3,4-dihydroisocoumarins involves the reaction of diazonium salts of substituted anthranilic acids with alkenes. The reaction was also applied to the synthesis of (hetero)aromatic-fused isocoumarins starting from 4-substituted (hetero)aromatic alcohols instead of alkenes (Scheme 66; 17EJO2147). Highly enantioselective [4+2] annulation reactions of 2-(halomethyl)benzaldehydes with (perfluoroalkyl)ketones under cooperative catalysis of an NHC and a CPA provides 3-aryl-3-(perfluoroalkyl)-3,4-dihydroisocoumarins (17S293). Examples of 3-substituted-3-trifluoromethyl 3,4-dihydroisocoumarins arise from asymmetric benzylation and 1,2-addition reactions of 2-methyl-3,5-dinitrobenzaldehyde with α,β-unsaturated trifluoromethyl ketones promoted by a tertiary amine-thiourea catalyst and subsequent oxidation with PCC (17CEJ519).

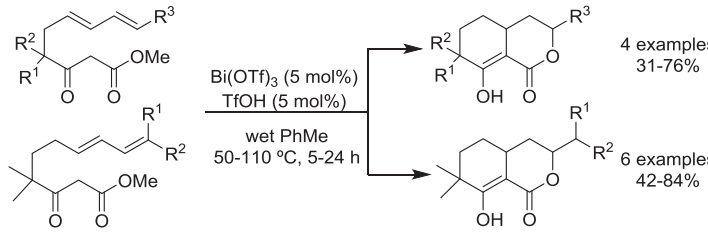
Thermal hDA reaction of benzocyclobutenones with isatins in *m*-xylene at 150°C led to 3-spiroxindole 3,4-dihydroisocoumarins in good to excellent yields (17JOC13751). Under $Bi(OTf)_3$/TfOH dual catalysis, the intramolecular addition of 1,3-dienyl β-ketoesters produced a series of 3,4-dihydroisocoumarin-type compounds (Scheme 67; 17OBC584).

6.4.2.6 Chromones and Chromanones

Tetrakis(triphenylphosphine)palladium(0) catalyst and XPhos (2-dicyclohexylphosphino-2′,4′,6′-triisopropylbiphenyl) were applied to the synthesis of 2-substituted 4*H*-chromen-4-ones via intramolecular acylation of *O*-(2-bromoalkenyl)salicylaldehydes carried out in the presence of potassium carbonate in 1,4-dioxane (17JOC5481). Under white



Scheme 66

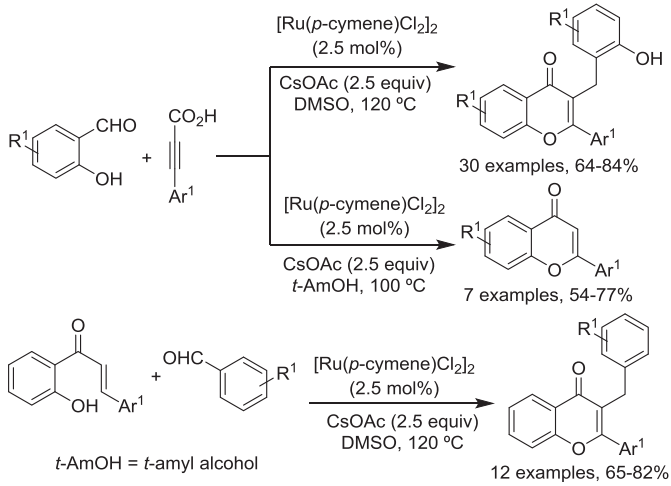


Scheme 67

LED irradiation (18 W), a series of *o*-hydroxyaryl enaminones with $BrCF_2CO_2Et$ or Ph_2SCF_3OTf in the presence of $Ir(ppy)_3$ and sodium acetate in acetone underwent radical-triggered tandem cyclization to furnish 4*H*-chromen-4-ones bearing 3-CF_2CO_2Et or 3-CF_3 substituents, respectively (17OL146).

A wide range of 2-aryl-3-hydroxy-4*H*-chromen-4-ones were synthesized through Algar–Flynn–Oyamada reaction of highly substituted of 2′-hydroxychalcones conducted in the presence of five equivalents of sodium carbonate and 2.5 equivalents of hydrogen peroxide in a 2:1 mixture of MeOH:H_2O at 0 °C for 30 min followed by the increase of temperature to 27 °C for 24 hours (17T4822). 2-Aryl-3-[(pyridin-3-yl)methyl]-4*H*-chromen-4-ones are readily formed in a one-pot procedure by base-catalyzed aldol condensation of aromatic aldehydes with 2′-hydroxyacetophenones in ethanol at 40 °C to afford the corresponding 2′-hydroxychalcones, which undergo tandem Michael-aldol reaction with nicotinaldehydes (17CPB784). The reaction of salicylaldehydes with arylpropiolic acids in the presence of a ruthenium catalyst and cesium acetate as base are solvent-dependent: in DMSO, various 2-aryl-3-benzyl-4*H*-chromen-4-ones were exclusively obtained, while 2-aryl-4*H*-chromen-4-ones were dominantly formed in the presence of *t*-AmOH as solvent. In addition, reacting 2′-hydroxychalcones with salicylaldehydes in DMSO also provides 2-aryl-3-benzyl-4*H*-chromen-4-ones (Scheme 68; 17OL6606).

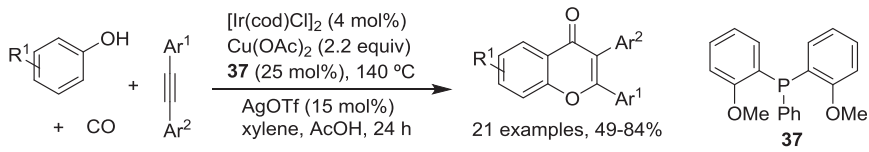
Functionalized 2,3-disubstituted 4*H*-chromen-4-ones are prepared via palladium(0)-catalyzed dehydrogenation of *o*-acyl phenols, in the presence of *N*,*N*-dimethylacetamide as solvent and 1 atm of nitrogen at 150 °C (17OL976), from the reaction of 1-(2-haloaryl)prop-2-yn-1-one derivatives with 1-aryl-2-phenylketones and cesium carbonate in DMF at 80 °C (17OBC2497), Friedel–Crafts acylation of alkyl/arylalkynes with 2-methoxyaroyl chloride and subsequent intramolecular cyclization of the in situ generated β-chlorovinyl ketones (17OL312), and from the



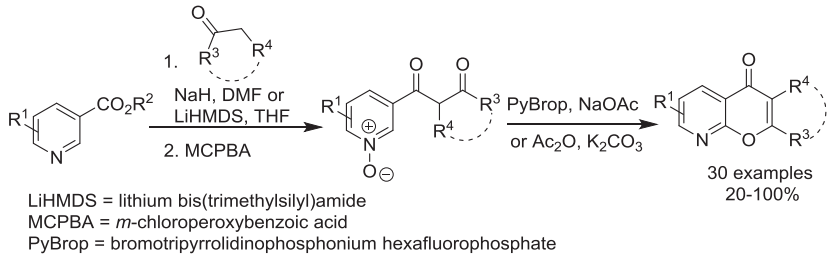
Scheme 68

iridium-catalyzed carbonylative annulation reaction of simple phenols with internal alkynes (Scheme 69; 17CEJ3276). Further derivatives are achieved via a four-step synthesis starting from 2′-hydroxyacetophenones: (1) *O*-allylation with allyl bromide in the presence of potassium carbonate and TBAI; (2) Claisen condensation with aliphatic and aromatic methyl esters; (3) cyclopropanation of the formed 1,3-diketone with *trans*-1,4-dibromobut-2-ene or bromosulfonium bromide in the presence of potassium carbonate; and (4) formation of the chromone ring through the reaction of the cyclopropane moiety with tetrakis(triphenylphosphine) palladium(0) and potassium carbonate (17JOC5317).

3-(Propane-1,3-dione)pyridine *N*-oxides, prepared in two steps via base-catalyzed Claisen condensation of nicotinates with 1,2-disubstituted ethan-1-ones followed by oxidation with *m*-chloroperoxybenzoic acid, underwent intramolecular *O*-arylation under PyBrop (bromo-tripyrrolidinophosphonium hexafluorophosphate) or Ac_2O activation conditions to afford a range of 8-aza-4*H*-chromen-4-ones (Scheme 70;



Scheme 69



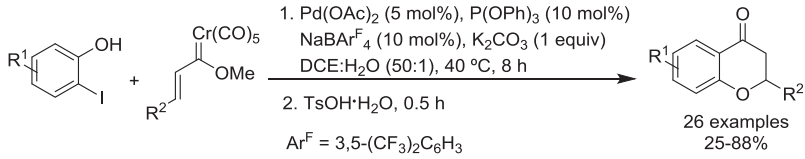
Scheme 70

17JOC11275). The synthesis of pyrazole-fused 4*H*-chromen-4-ones occurs via copper(I)-mediated tandem *O*-arylation-oxidative cross-coupling of 2,4-dihydro-3*H*-pyrazol-3-ones with *o*-halo arylcarboxaldehydes (17JOC2926).

Asymmetric oxa-Michael addition of 2′-hydroxychalcones catalyzed by CPAs modified magnetite nanoparticles inside and outside carbon nanotubes leads to 2-substituted 4*H*-chroman-4-ones (17CC6029). Other analogs arise from palladium(II)-mediated [3+3] annulation of vinyl chromium(0) Fischer carbene complexes with 2-iodophenols (Scheme 71). This strategy involves carbene migratory insertion and an intramolecular Tsuji–Trost reaction as key steps (17AGE13140). Highly functionalized 2-amino-substituted 3,3-difluoro-4*H*-chroman-4-ones are obtained from tandem cyclization of *o*-hydroxyaryl enaminones carried out in the presence of Selectfluor and sodium acetate in acetone at room temperature (17JOC9837). NHC-mediated intramolecular nucleophilic substitution reactions of *O*-(4-bromobut-2-ene)salicylaldehydes and DBU in THF provide 3-substituted-3-vinyl-4*H*-chroman-4-ones in moderate to excellent yields and with excellent enantioselectivity (17CEJ2783).

6.4.2.7 Xanthenes and Xanthones

High yields of 9-hydroxy-9-trifluoromethyl-9*H*-xanthenes are achieved via tandem insertion-nucleophilic cyclization reactions of arynes, generated in situ from *o*-trimethylsilylphenyl triflates and CsF as fluoride source, with

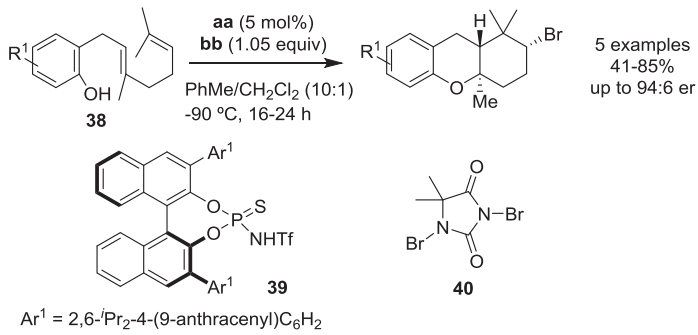


Scheme 71

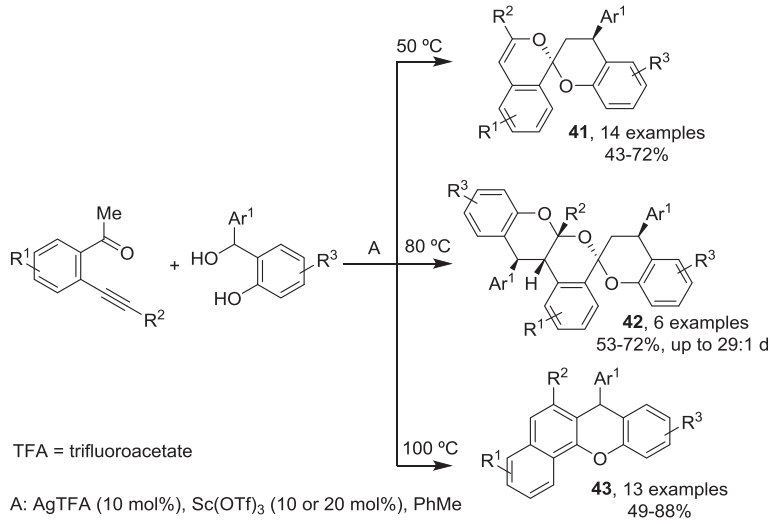
2-trifluoroacetylphenols in refluxing THF (17TL2964). Other arynes, generated in situ from *o*-trimethylsilylphenyl triflates and KF, undergo DA reaction with stable *o*-quinone methides in THF at room temperature to give 9-substituted 9*H*-xanthenes (17TL1137). Asymmetric bromocyclization of geranylphenols **38** conducted in the presence of a chiral BINOL-derived thiophosphoramide catalyst **39** and 1,3-dibromo-5,5-dimethylhydantoin **40** as an electrophilic bromine source gives access to hexahydro-9*H*-xanthenes (Scheme 72; 17JA1460).

Under dual catalysis of AgTFA and $Sc(OTf)_3$, the reaction of 2′-(2-arylethynyl)acetophenones with *p*-quinone methides in toluene at 80 °C produces 11-substituted benzo[*c*]xanthenes, via benzoannulation, 1,6-addition, and cyclization cascade reactions (17JOC11524). Using the same bicatalytic system and adjusting the temperature, the reaction of β-alkynyl ketones with *o*-hydroxybenzyl alcohols in toluene provides (1) at 50 °C a range of spiro[chromane-2,1′-isochromene] derivatives **41**, (2) at 80 °C a series of spiro[chromane-2,5′-isochromeno[3,4-*b*]chromene] derivatives **42**, and (3) at 100 °C, several benzo[*c*]xanthenes **43** (Scheme 73; 17CC10692).

The syntheses of 9-substituted tetrahydro-9*H*-xanthen-1-ones can be accomplished through copper(I)-catalyzed reaction of diethyl 2-(2-bromobenzylidene)malonates with cyclic 1,3-diketones, involving Michael addition and intramolecular Ullmann-type C(aryl)–O bond formation (17TL168). Under palladium(0) catalysis, the cross-coupling reaction of 1-bromo-2-[2-(bromomethyl)phenoxy]benzene with *N*-tosylarylhydrazones followed by intramolecular Heck reaction, using a combination of *t*-BuOLi and *t*-BuONa as base in 1,4-dioxane at 120 °C produces 9-benzylidene-9*H*-xanthenes in moderate to good yields



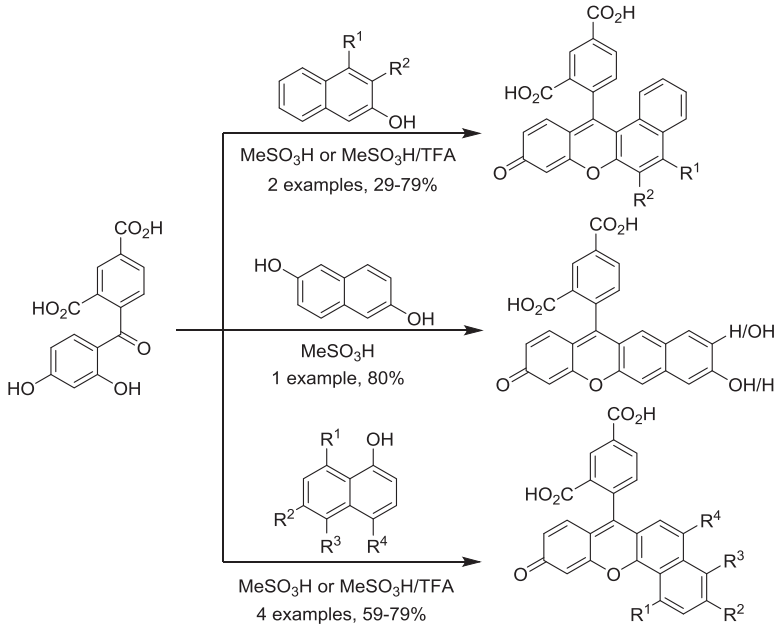
Scheme 72



Scheme 73

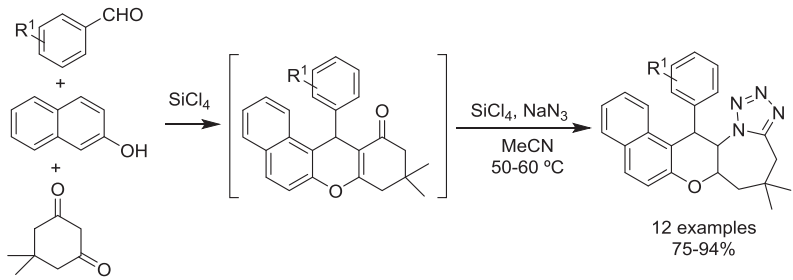
(17OL2034). Enantioselective syntheses of (2,3,4,9-tetrahydro-1*H*-xanthen-1-ylidene)anilines are accomplished via cyclization of *o*-quinone methides, generated in situ from *o*-hydroxystyrenes at high temperatures in the presence of a CPA organocatalyst, with dimedone-derived enaminones (17S2035). Condensation reaction of 4-(2,4-dihydroxybenzoyl)isophthalic acid with naphthol derivatives in methanosulfonic acid or in a 1:1 mixture with TFA provides a series of 5-carboxyseminaphthofluoresceins (Scheme 74; 17TL1611).

A wide range of diversely substituted xanthene-type compounds arise from one-pot three-component reactions of alkyl/aryl aldehydes with two equivalents of cyclohexane-1,3-diones or cyclohexane-1,3-diones and barbituric acid catalyzed by L-proline in water (17T3497) or by sulfated perborate in solvent-free conditions (17TL2859); reaction of alkyl/aryl aldehydes with two equivalents of 2-naphthol or with 2-naphthol and dimedone catalyzed by sulfated perborate in solvent-free conditions (17TL2859); reaction of 1,2-diarylethan-1,2-diols with four equivalents of 4-hydroxycoumarins using lead tetraacetate as oxidizing agent (17JHC1543); reaction of aromatic aldehydes with barbituric acid and 4-hydroxyquinoline using a heterogenous sulfamic acid catalyst in a 4:1 mixture of water: ethanol (17JHC2206); reaction of aromatic aldehydes with cyclohexane-1,3-dione and 3,5-dimethoxyphenol mediated by niobium pentachloride (17S2402); reaction of salicylaldehydes with malononitrile and secondary amines



Scheme 74

promoted by TiO_2—SiO_2, under solvent-free conditions (17JHC2598); reaction of salicylaldehydes with cyclohexanones and anilines under dual catalysis of $Yb(OTf)_3$ and 1,1′-binaphthyl-2,2′-diyl hydrogenphosphate (17OBC4933). Further examples are achieved from a four-component reaction of benzaldehydes with 2-naphthol and dimedone in tetrachlorosilane followed by the addition of azidochlorosilane, prepared in situ from sodium azide and tetrachlorosilane in acetonitrile (Scheme 75) (17JHC2463). Treating 5,6-dihydro-*4H*-pyrrolo[3,2,1-*ij*]quinoline-1,2-dione with two equivalents of cyclohexane-1,3-diones/barbituric acid

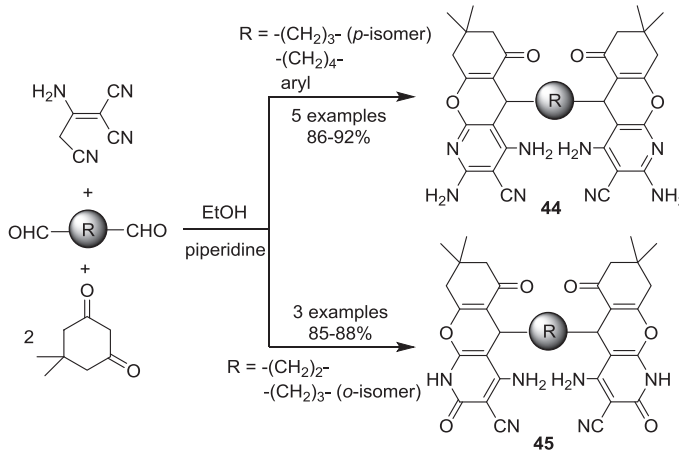


Scheme 75

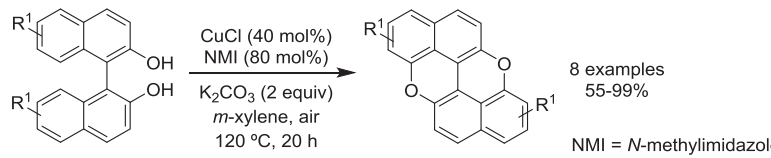
derivatives in the presence of PTSA in refluxing water produces spiropyrroloquinoline xanthene–type derivatives (17JHC944).

Depending on the length and position of the spacer in the bis-aldehyde, a three-component reaction with malononitrile dimer and two equivalents of dimedone leads to bisxanthene derivatives **44** or **45** (Scheme 76; 17JHC2844).

A practical synthesis of *peri*-xanthenoxanthenes carried out in the presence of air as oxidant and potassium carbonate as base involves C–H/C–O cyclization reactions of 2,2′-binaphthols performed with CuCl and *N*-methylimidazole as the catalytic system (Scheme 77) (17OL2714). Under aerobic solvent-free conditions, a range of 9*H*-xanthen-9-ones arise from intramolecular oxidative dehydrogenative reaction of 2-aryloxybenzaldehydes promoted by carbon tetrabromide at 140 °C. This reaction was applied to a 10-g scale-up for the synthesis of the parent xanthone (17OBC1589).



Scheme 76



Scheme 77

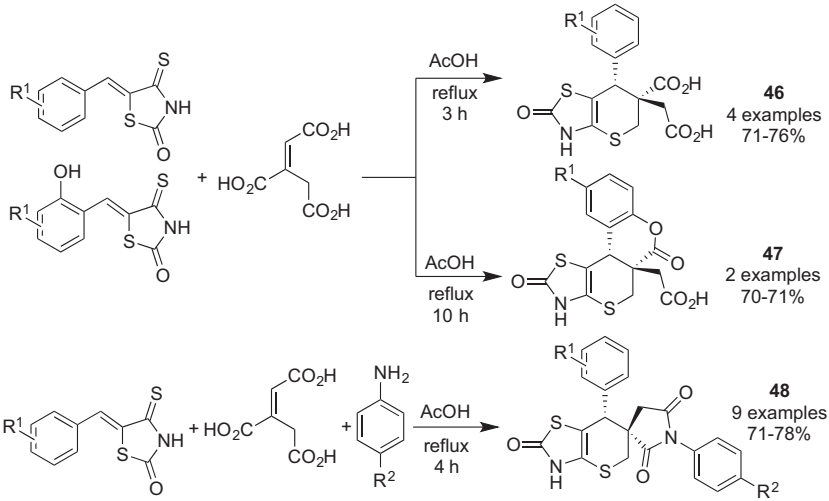
6.4.3 HETEROCYCLES CONTAINING ONE OR TWO SULFUR ATOMS

6.4.3.1 Thiopyrans and Analogs

A series of 3,4-dihydro-2*H*-thiopyrans can be prepared from an organocatalyzed *o*-regioselective IED–hDA reaction of dienamines, generated in situ from β,β-disubstituted enals and an amino catalyst, as dienophiles and thiochalcones as heterodienes (17CC11472). Asymmetric [4+2] thia-DA reaction of thioketones as heterodienophiles with 2,4-dienals mediated by the organocatalyst diphenylprolinol trimethylsilyl ether provides polysubstituted 5,6-dihydro-2*H*-thiopyrans in moderate to good yields (17EJO950). A wide range of thiopyran[2,3-*b*]indoles are synthesized through formal [3+3] annulation reactions of β′-acetoxy allenoates with indoline-2-thiones carried out in the presence of 1,4-diazabicyclo[2.2.2] octane as catalyst and potassium carbonate as additive (17CC2567). [4+2] hDA reaction of 5-arylidene-4-thioxo-2-thiazolidinones with *trans*-aconitic acid affords tetrahydro-2*H*-thiopyrano[2,3-*d*][1,3]thiazole derivatives **46**, but when an *o*-phenolic group is presented at the arylidene moiety, a couple of tetrahydro-2*H*,5*H*-chromeno[4′,3′:4,5]thiopyrano[2,3-*d*]thiazoles **47** are obtained. Moreover, a three-component reaction of 5-arylidene-4-thioxo-2-thiazolidinones with *trans*-aconitic acid and aromatic amines gives spiro-substituted thiopyrano[2,3-*d*]thiazoles **48** (Scheme 78) (17TL1751).

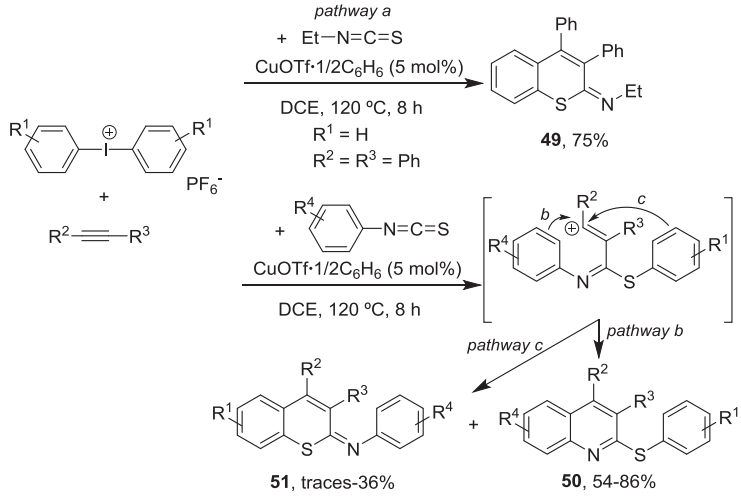
High yields of thiopyranindole annulated [3,4-*c*]quinolone derivatives are achieved through domino Knoevenagel–hDA reaction of indoline-2-thiones with *N*-acrylated anthranilaldehydes with the assistance of zinc bromide as catalyst in refluxing ethanol (17T3040).

A wide range of 2*H*-thiochromenes are obtained from a sulfa-Michael/Julia–Kocienski olefination cascade reaction of 2-mercaptobenzaldehydes with vinyl(1-alkyl/aryl-1*H*-tetrazol-5-yl)sulfones mediated by diphenylprolinol trimethylsilyl ether (17JOC4851), thiol-Michael–aldol cascade reaction of 2-mercaptobenzaldehydes with methyl cinnamates mediated by tetramethylguanidine (17SL429) and Cu(II)-catalyzed domino reaction of 2-halobenzaldehydes with chalcone derivatives and potassium ethyl xanthate as an odorless sulfur surrogate (17JOC1936). CuOTf-catalyzed three-component reaction of diaryliodonium salts with isothiocyanates and alkynes depend on the substitution pattern of isocyanate. Thus, using ethyl isocyanate, thiochromene **49** was obtained (pathway a), while in the presence of aryl isocyanates, two possible routes may occur. The first



Scheme 78

possibility is the nucleophilic attack of the phenyl ring of arylisothiocynate on the cationic species through [4+2] annulation reaction to give 2-sulfhydrylquinolines **50** (pathway b); the second one is the nucleophilic attack of the phenyl ring of diaryliodonium salt on the cationic species through [2+2+2] annulation reaction to form thiochromenes **51** (pathway c; Scheme 79; 17OL2694).

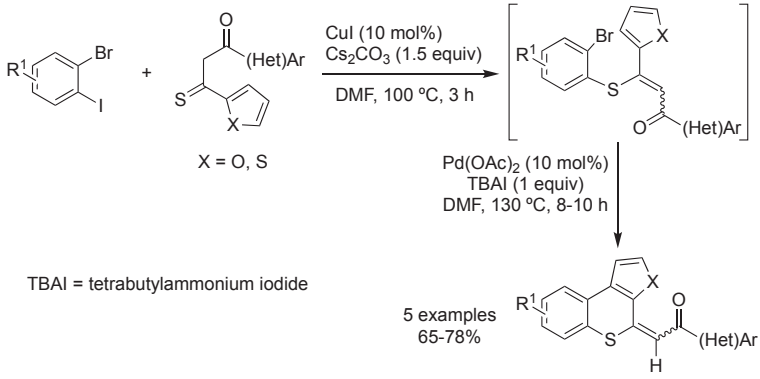


Scheme 79

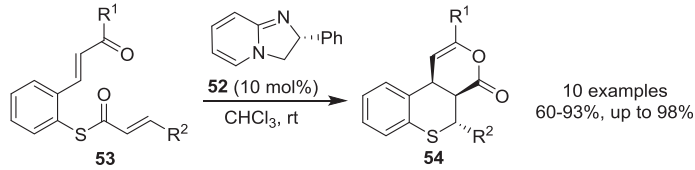
The synthesis of thiochroman-4-ols can be accomplished through the reactions of 2-mercaptobenzaldehydes with *gem*-difluoroolefins in the presence of DBU at room temperature (17JOC11348) or with nitroalkenes mediated by a bis(imidazolidine)iodobenzene catalyst (17SL122). Tandem sulfa-Michael–aldol reaction of 2-mercaptobenzaldehydes with 3-ylideneoxindoles promoted by potassium carbonate affords spiroxindole thiochroman-4-ols (17TL3401). Reductive cyclization of β-arylthioketones carried out in the presence of SmI_2 as catalyst and hexamethylphosphoramide as additive leads to dihydrothiochroman-4-ols, and further oxidation with 2,3-dichloro-5,6-dicyano-1,4-benzoquinone gives the corresponding thiochroman-4-ols (17OBC6157). A one-pot synthesis of furano- and thieno-fused 2-(het)aroylethylidenethiochromenes arise from a copper-catalyzed Ullmann-type intermolecular C—S coupling reaction of *o*-bromoiodoarenes with 1,3-bis(het)arylmonothio-1,3-diketones followed by in situ palladium-catalyzed intramolecular Heck reaction of the obtained β-(arylthio)vinyl ketones (Scheme 80; 17OL1512).

Amidine-based catalyst **52** promotes an acyl transfer—initiated cascade reaction of thioester **53** to provide pyran-2-one-fused thiochromans **54** in high yields and with excellent enantioselectivity (Scheme 81; 17OL6486). A series of CF_3S-containing spirocyclopentanone thiochromans results from one-pot electrophilic trifluoromethylthiolation–sulfur–Michael/aldol cascade reaction of cycloalkenones with 1-(trifluoromethylthio)pyrrolidine-2,5-dione and 2-mercaptobenzaldehydes mediated by a bifunctional squaramide catalyst (17OL1036).

The reaction of α-substituted *o*-[(*t*-butylsulfanyl)methyl]styrenes with three equivalents of iodine and sodium bicarbonate in acetonitrile at 0 °C



Scheme 80

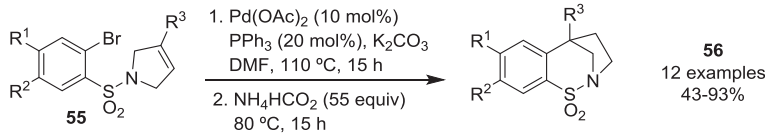


Scheme 81

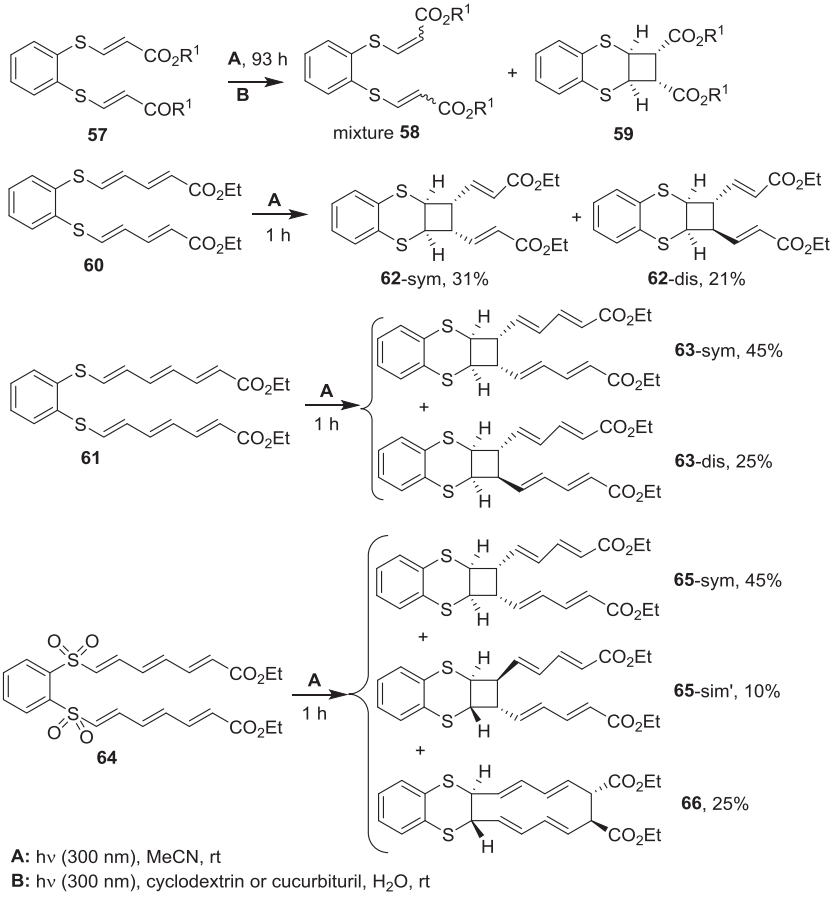
gives access to isothiochromenes in moderate to good yields (17H(94) 2065). Friedel–Crafts acylation of alkyl/arylalkynes with 2-(methylthio) benzoyl chloride provides β-chlorovinyl ketones, which undergo intramolecular cyclization to afford 4*H*-thiochromen-4-ones in good yields (17OL312). Further derivatives arise from sequential nucleophilic addition, elimination and S_NAr reaction of β-chlorovinyl aromatic ketones, and sodium hydrogen sulfide carried out in the presence of cesium carbonate in DMSO (17S4309). The synthesis of cyclic sulfonamides **56** occurs via one-pot intramolecular Heck reaction of *N*-(*o*-bromoaryl)sulfonyl dihydropyrroles **55** followed by transfer hydrogenation sequence in the presence of ammonium formate (Scheme 82; 17TL4559).

6.4.3.2 Dithiin Analogs

Direct irradiation of bis-acrylates (R^1 = Me) **57** in acetonitrile provides a mixture of isomers **58** and the cycloadduct cyclobutyl derivative **59**. Photocycloaddition of bis-diene **60** and bis-triene **61** affords the two stereoisomeric cyclobutyl products **62**-sym and **62**-dis and **63**-sym and **63**-dis, respectively. Under the same conditions, bis-sulfone **64** gives **65**-sym isomer as major product and two new compounds, **65**-sym' and the macrocycle **66**. Using water as solvent and cyclodextrin or cucurbituril as host, the irradiation of bis-acrylates **57** (R^1 = Me) also provides a mixture of isomers **58** and the cycloadduct cyclobutyl derivative **59**. In these conditions, the acid derivatives **58** (R^1 = H) led mainly to cycloadduct cyclobutyl derivative **59** and to the mixture of isomers **58**, in minor amounts (Scheme 83; 17OBC4180).



Scheme 82

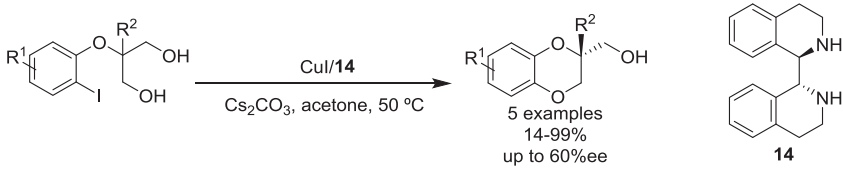


Scheme 83

6.4.4 HETEROCYCLES CONTAINING TWO OR MORE OXYGEN ATOMS

6.4.4.1 Dioxanes

Asymmetric desymmetrization of iodophenols-tethered 1,3-diols promoted by CuI and a chiral cyclic diamine ligand **14** gave several examples of 2-hydroxymethyl-1,4-benzodioxanes in moderate to excellent yields and with modest enantioselectivity (Scheme 84; 17JOC1458).



Scheme 84

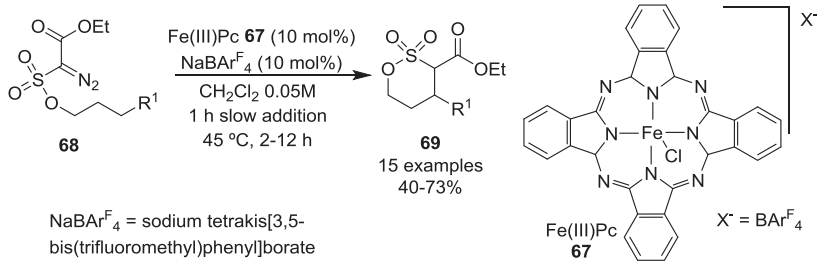
6.4.5 HETEROCYCLES CONTAINING BOTH OXYGEN AND SULFUR IN THE SAME RING

6.4.5.1 Oxathianes

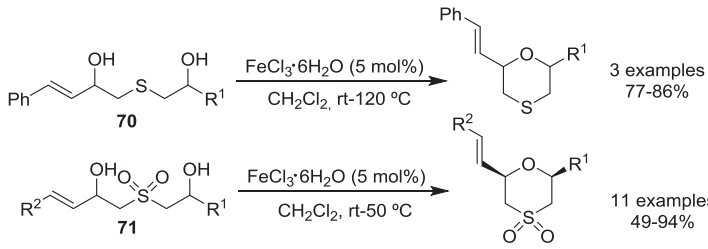
Under microwave irradiation, the reaction of thiiranes with α-diazo-β-dicarbonyl compounds in the presence of copper sulfate provides 3-acyl-5,6-dihydro-1,4-oxathiines (17TL1651). Iron-phthalocyanine **67** mediates C—H alkylation of allylic and benzylic diazoesters **68** to afford 1,2-oxathiine 2,2-dioxides **69** (Scheme 85; 17JA13624).

Several examples of 1,4-oxathianes arose from iron-catalyzed cyclization of bis diol thioethers **70** in dichloromethane at 50 °C. The same procedure was applied to the synthesis of 1,4-oxathiane 4,4-dioxides starting from the corresponding bis diol sulfones **71** (Scheme 86; 17JOC4020).

Iron-mediated coupling reactions of internal alkynes bearing electron-withdrawing groups [CF_3, CO_2R, R(O)R] with thiosalicylic acids in the presence of 1,10-phenanthroline in a 1:1 mixture of toluene and hexafluoroisopropyl alcohol led to benzo[1,3]oxathiines in moderate to excellent yields (17OL4299). The synthesis of fluorinated benzo[*b*][1,4]oxathiines occurs through perfluorophenylthiolation, and cyclization reactions of activated α-methylene ketones (β-ketoesters, 1,3-diketones, and β-keto sulfones) carried out in the presence of perfluorophenyl diethylaminosulfur difluoride (17OL1012).



Scheme 85



Scheme 86

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