

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

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6.4.1. INTRODUCTION

The year 2011 was particularly rich in reviews involving the synthesis and transformations of a wide structural range of *O*- and *S*-6-membered heterocycles and some on their biological properties, reflecting their importance for the scientific community.

An overview on the structure and biological activity of naturally occurring withanolides <11NPR705> and a review on structure, biological activity, and synthesis of plant polyphenols <11AGE586>, marine natural products <11NPR196, 11NPR269>, and aromatic cyclopenta[*c*]pyrans <11JHC747> have appeared. The synthesis of the various structural subcategories of limonoids has also been surveyed <11EJO19>.

More specific reviews include a critical attempt to discuss a structure–activity relationship among a series of trioxane- and tetraoxane-based compounds <11CMC3889>, and a report on the bioactivity and scope of the synthetic methods of chromenopyridines, with emphasis on multicomponent and robust methodologies, as well as reactivity issues and medical applications <11CMC4761>. The powerful synthetic strategy using the thiopyran template route to the enantio- and diastereoselective synthesis of polypropionates, and some natural products or synthetic key intermediates, has been reviewed <11CC11375>.

The search for naturally occurring compounds for biological application remains an important field of research, as demonstrated for the 16-membered macrolactone leiodermatolide, isolated from a marine sponge <11AGE3219>. Despite the beneficial effect of many of these compounds, some of them are very toxic. An update of risk assessment of main marine biotoxins in the European Union <11TOX336>, as well as the traditional remedies of Ciguatera fish poisoning in Pacific <11PTR947>, has been reviewed.

The scarce natural abundance of the biologically active natural products makes them prime targets for the development of efficient and short total syntheses <11HCA2215, 11EJO7097>, for the stereoselective synthesis of some common moieties <11TL3654>, and for syntheses involving common intermediates <11TA1249, 11TL6180>.

In many cases, a dihydropyran-2-one ring is a key intermediate in the total synthesis of natural products. Ring-closing metathesis using Grubbs' catalysts has been

applied to the synthesis of (+)-goniodiol and derivatives <11TL438>, (+)-cryptofolione <11TL1003>, and (–)-cleistenolide <11T3815, 11TL2443>. Lactonization mediated by $\text{CeCl}_3 \cdot 7\text{H}_2\text{O}$ also allowed the synthesis of (–)-cleistenolide <11TL2306> and (–)-spicigerolide <11TA493>.

The synthesis of naturally occurring tetrahydropyran derivatives involves domino and metal-catalyzed reactions <11TL6550, 11T4980>, while the unified asymmetric syntheses of the natural products lachnone C (4-chromanone) and diversanol (atetrahydroxanthone) involve organocatalyzed domino reactions as the enantioselective key step <11CEJ13706>. These types of reactions comprise three of the hot and most studied transformations on O-6-membered heterocycles.

Following the dramatic growth of publications on the reactions catalyzed by gold complexes, due to their mild reaction conditions and high group compatibility, the synthesis of pyrans, and various ketals and spiroketal systems <11OBC4405>, and also di- and tetrahydropyrans, pyran-4-ones, and chromene and xanthene derivatives <11CR1657> have been discussed. The use of other metal-catalyzed reactions, namely, the intermolecular dehydrogenative Pd-catalyzed Heck reactions, to prepare pyran-4-ones, chromans, isochromans, coumarins, and isocoumarins <11CR1170>, and the application of Bi(III) compounds from the “green-chemistry” perspective in the total synthesis of several pyran derivatives <11CSR4649> have been reviewed. The asymmetric hydration of di- and tri-enoates by Sharpless asymmetric dihydroxylation combined with other metal-catalyzed reactions leads to a diverse array of structurally complex and biologically important natural products <11CC8493>.

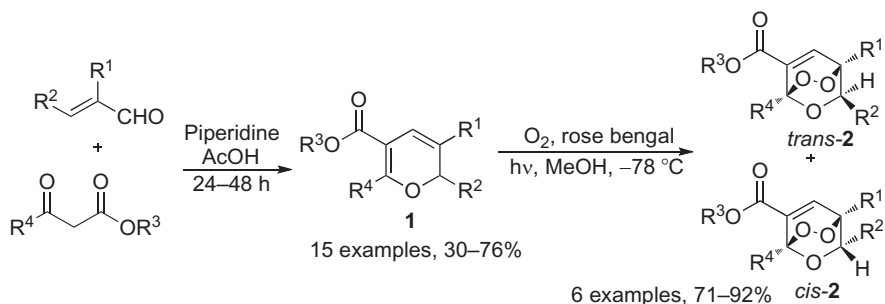
Many methodologies for the synthesis of naturally occurring coumarins have been described, including improving well-known methods <11SC1514> and the use of NbCl_5 to catalyze a known condensation <11SC1486>. Recent advances in cycloadditions of alkynes, as atom-economic controllable chemo- and regioselective reactions to construct pyranones, coumarins, and isocoumarins, were over-viewed <11COC712>. The importance of coumarins was also highlighted in a review on their antioxidant activity <11CMC3929> and in references to their application in dye-sensitized solar cells <11PC569, 11PC1929>.

Reviews on catalytic asymmetric propargylation, describing the propargylation of naphthols as a synthetic route to chromene and chroman derivatives <11CR1914>, and on the synthesis of several heterocycles (e.g., pyran-2-ones, chromenes, and isochromans) involving the electrophilic cyclization of alkynes containing a heteroatom <11CR2937> has appeared.

6.4.2. HETEROCYCLES CONTAINING ONE OXYGEN ATOM

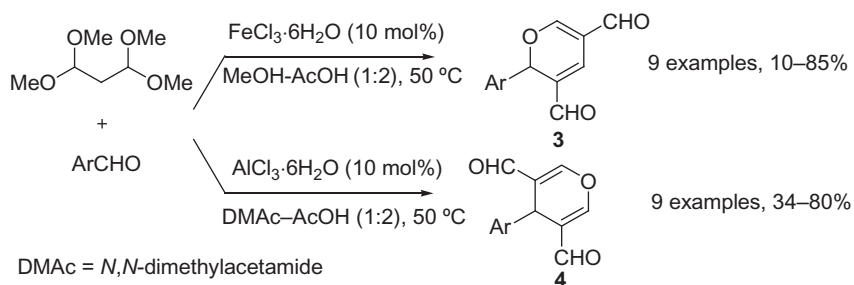
6.4.2.1 Pyrans

2,3,6-Trisubstituted 2*H*-pyran-5-carboxylates **1** are readily available from the reaction of 2-alkyl-2-enals with acetoacetates and involve a Knoevenagel condensation followed by a 6π -electrocyclization. These 2*H*-pyrans were transformed into the corresponding 1,2,4-trioxane structures **2** by cycloaddition with singlet oxygen (Scheme 1) <11EJO5469>.



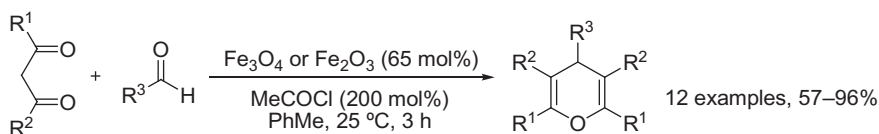
Scheme 1

2-Aryl-3,5-diformyl-2*H*-pyrans **3** are selectively prepared by FeCl₃·6H₂O-catalyzed reaction of 1,1,3,3-tetramethoxypropane with aromatic aldehydes, while using AlCl₃·6H₂O provided 4-aryl-3,5-diformyl-4*H*-pyrans **4** (Scheme 2) <11SL857>.



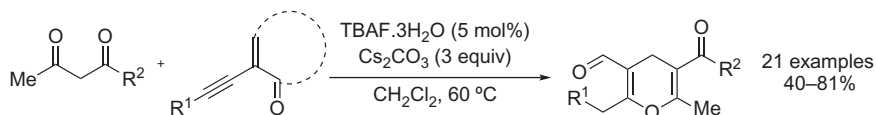
Scheme 2

Magnetite or iron(III) oxide was successfully used in the tandem formation of polysubstituted 4*H*-pyrans using the appropriate 1,3-dicarbonyl compounds and aliphatic or aromatic aldehydes (Scheme 3). This process involves an aldol condensation, a Michael-type addition, and a dehydrating annulation <11SL2017>.



Scheme 3

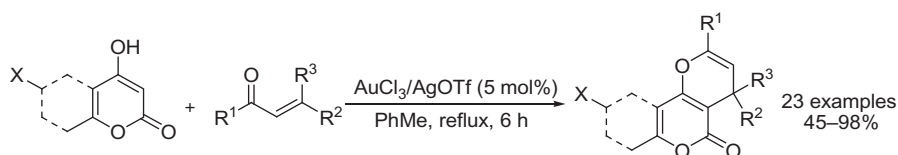
Phase transfer-catalyzed nucleophilic addition of 1,3-dicarbonyl compounds to electron-deficient 1,3-conjugated enynes followed by cyclization affords 4*H*-pyran derivatives (Scheme 4) <11OBC3375>.



TBAF = tetra-*n*-butylammonium fluoride

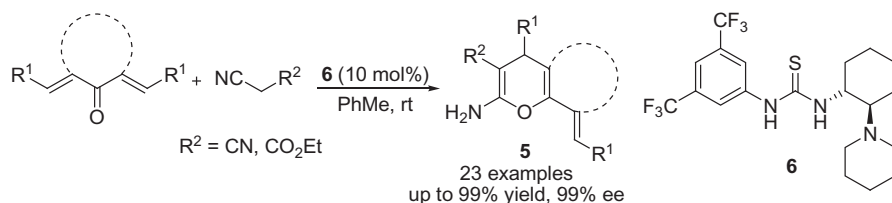
Scheme 4

A gold(III)-catalyzed regioselective tandem conjugate addition/annulation of 4-hydroxycoumarins or 4-hydroxy-6-methylpyran-2-one with α,β -unsaturated ketones furnished various coumarin or pyran-2-one-fused 4*H*-pyran derivatives (Scheme 5) <11JOC9096>.



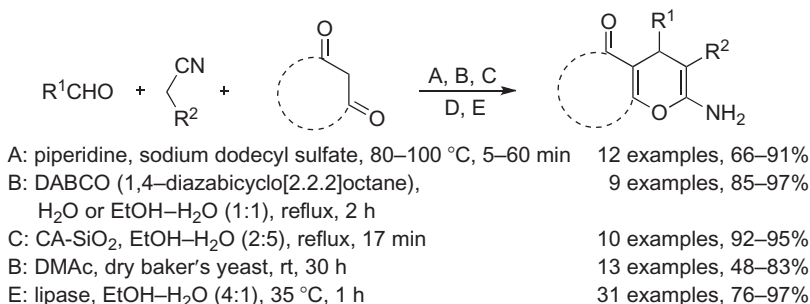
Scheme 5

An enantioselective organocatalytic conjugate addition of malononitrile or cyanoacetate to conformationally restricted dienones led to chiral 4*H*-pyrans **5**, which are potent inhibitors of *Mycobacterium tuberculosis* (Scheme 6) <11JOC3797>.



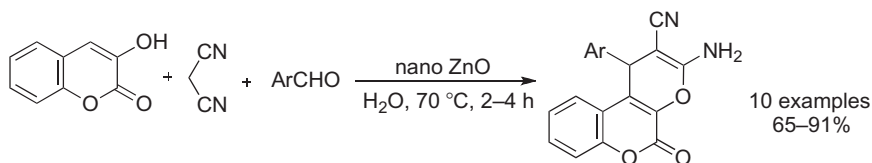
Scheme 6

Polyfunctionalized 4*H*-pyrans can be prepared by a one-pot, three-component cyclocondensation of aryl aldehydes, malononitrile/ethyl cyanoacetate, and 1,3-dicarbonyl compounds catalyzed by piperidine <11JHC124>, DABCO <11SC2701>, Caro's acid supported on silica gel (CA-SiO₂) <11SC436>, baker's yeast <11TL5817>, and the enzyme lipase (Scheme 7) <11T9582>. This type of one-pot reaction was also applied to the synthesis of biologically and pharmacologically active 4*H*-pyrans using SiO₂ nanoparticles as an inexpensive, reusable, and environmentally benign catalyst <11TL1878>. Similar compounds are obtained by a tandem Michael addition/cyclization reaction involving malononitrile, acetylene dicarboxylate, and dimedone <11T6057>. A base-catalyzed two-component approach uses α,β -unsaturated cyanoesters and dimedone derivatives <11SC2822>.



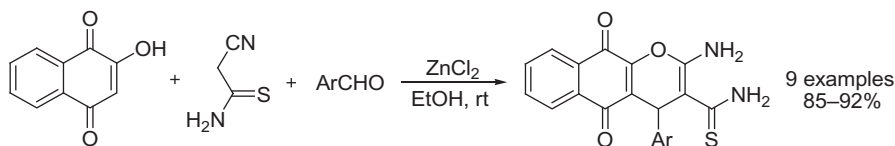
Scheme 7

Coumarin-fused 4*H*-pyran derivatives are available from a one-pot, three-component coupling reaction of aromatic aldehydes, malononitrile or ethyl cyanoacetate, and 3- or 4-hydroxycoumarin, in the presence of nano-ZnO (Scheme 8) <11TL4636>, DMAP [4-(dimethylamino)pyridine] <11TL5327>, or the ionic liquid 2-hydroxyethanaminium acetate <11SC3573>, as catalysts. Using 4-hydroxyquinolin-2-(1*H*)-one instead of hydroxycoumarins, quinoline-fused 4*H*-pyrans were obtained <11TL2597>. The same type of multicomponent reaction was used for the synthesis of other coumarin-fused pyrans by the one-pot reaction of 4-hydroxycoumarin, aldehydes, and 1,3-dicarbonyl compounds, using the ionic liquid 1,3-dimethyl-2-oxo-1,3-bis(4-sulfobutyl)imidazolidine-1,3-dium hydrogen sulfate as catalyst <11TL2601>.



Scheme 8

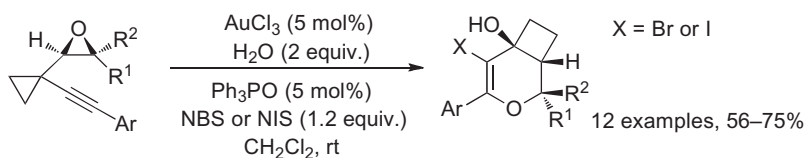
Naphtho-fused 4*H*-pyrans are formed from a three-component reaction of aromatic aldehydes, malononitrile, and α - or β -naphthol, catalyzed by disodium hydrogen phosphate under solvent-free conditions <11SC3477>. Other derivatives are prepared in moderate to good yields (65–94%) by one-pot reaction of alkyl isocyanides, dialkyl acetylenedicarboxylates, and hydroxynaphthalenes <11SC3714> or α -tropolone <11HCA371>. Some examples were also obtained by one-pot ZnCl₂-catalyzed reaction of 2-hydroxynaphthalene-1,4-dione, cyanothioacetamide, and aromatic aldehydes (Scheme 9) <11SC806>.



Scheme 9

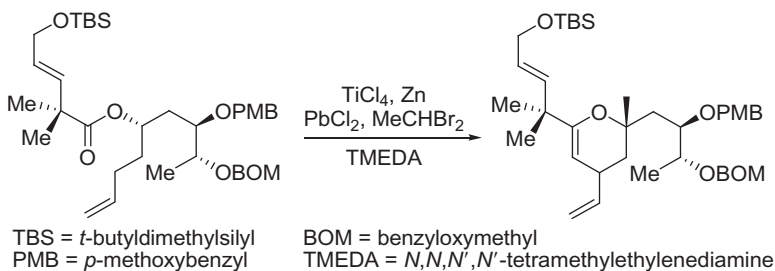
A four-component synthesis of 4*H*-pyrano[2,3-*c*]pyrazoles involved ethyl acetoacetate, hydrazines, malononitrile, and aldehydes and uses L-proline <11TL3228> or γ -alumina <11TL2523> as catalyst in aqueous media. Other combinations of catalysts and solvents are used: L-proline in ionic liquids <11SC405> and Mg-Al hydrotalcite in ethanol <11SC1320>.

An efficient gold(III)-catalyzed ring expansion of 1-oxiranyl-1-alkynylcyclopropanes in the presence of *N*-(iodo and bromo)succinimides afforded cyclobutane-fused 3,4-dihydro-2*H*-pyrans (Scheme 10) <11CC1339>.



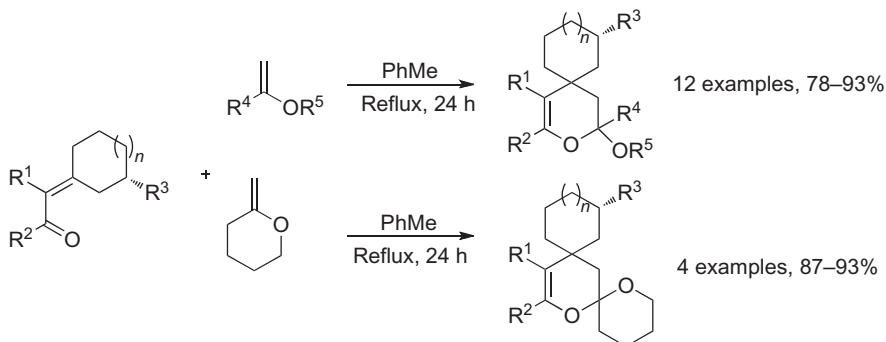
Scheme 10

The 3,4-dihydro-2*H*-pyran ring of bryostatin derivatives is prepared by a Rainier titanium-induced ring-closing metathesis (Scheme 11) <11AGE8786, 11JA744> and a Ru-catalyzed tandem enyne coupling/Michael addition <11CEJ9789>.



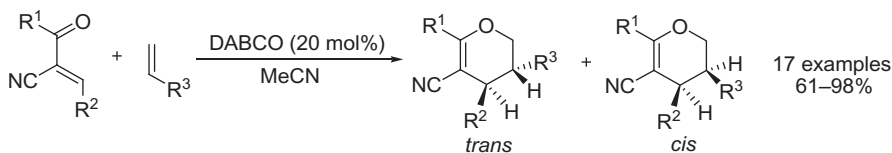
Scheme 11

Inverse-electron-demand hetero-Diels-Alder (hDA) between electron-deficient α,β -unsaturated carbonyl compounds and electron-rich dienophiles is an important strategy for the synthesis of 3,4-dihydro-2*H*-pyrans. Novel spiro- and dispirodihydropyrans were obtained in good yields by refluxing sterically hindered cycloalkylidene derivatives with cyclic enol ethers or sterically hindered cycloalkylidene cycloalkanes in refluxing toluene (Scheme 12) <11T1422>. An asymmetric hDA reaction of six-membered cyclic ketones provided *trans,cis*-2,3,4-trisubstituted 3,4-dihydropyrans as major products <11AGE3484>.



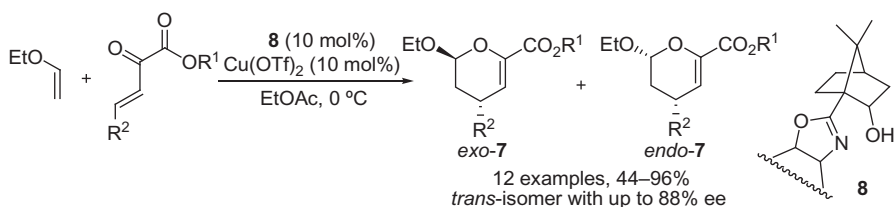
Scheme 12

Densely functionalized *trans*- and *cis*-3,4-dihydro-2*H*-pyran isomers are obtained by tandem cross-Rauhut–Currier/cyclization reactions of α,β -unsaturated ketones and activated alkenes (Scheme 13) <11T1768>.



Scheme 13

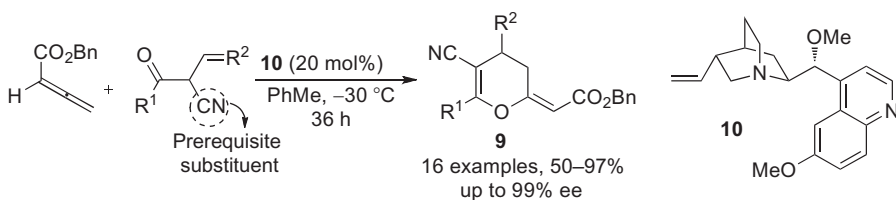
The reaction of β,γ -unsaturated α -keto esters with ethyl vinyl ether catalyzed by copper(II) complexes of (+)-*S*-ketopinonic acid-based hydroxy oxazoline **8** afforded *trans*-2,4-disubstituted chiral 3,4-dihydro-2*H*-pyrans **7**, with up to 88% ee (Scheme 14) <11SL1592>. A similar procedure used $\text{Eu}(\text{fod})_3$ as catalyst for a highly diastereoselective and facially controlled hDA reaction to prepare dihydropyran derivatives that are intermediates in the synthesis of natural lignans <11TL1608>. A remarkably low catalyst loading (up to 0.05 mol%) of a chiral $\text{Er}(\text{OTf})_3/N,N'$ -dioxide complex was used in the synthesis of 3,4-dihydro-2*H*-pyrans in excellent yields, diastereo- and enantioselectivities <11CEJ8202>. Visible light photocatalysis of symmetrical and unsymmetrical bis(enones) afforded 3,4-dihydro-2*H*-pyrans, as single regioisomers in an intramolecular hDA reaction <11T4442>.



Scheme 14

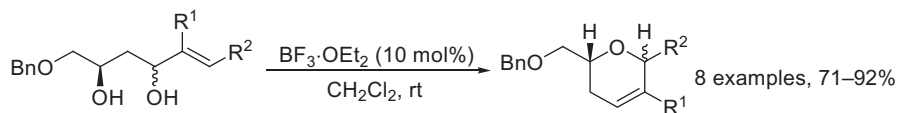
A new Ni-iminophosphine complex catalyzes the [4+2] cycloaddition of enones with allenes, providing highly substituted 3,4-dihydro-2*H*-pyrans, via oxidative cyclization of the enone with a Ni(0) complex <11CC6150>.

An asymmetric [4+2] annulation of allenates with oxadienes in the presence of a cinchona organocatalyst **10** led to 3,4-dihydro-2*H*-pyrans **9** with good to excellent enantioselectivities (Scheme 15) <11AGE5361>. A similar annulation (a formal [4+2] cycloaddition) was used to make novel 3,4-dihydro-2*H*-pyran derivatives. Using other cinchona alkaloids as catalysts, an asymmetric center at C-4 was created with efficient stereocontrol <11OL5732>, while DABCO <11OL1138> and DMAP <11OL1142> afforded dihydropyran-fused indoles and isatin derivatives, respectively.



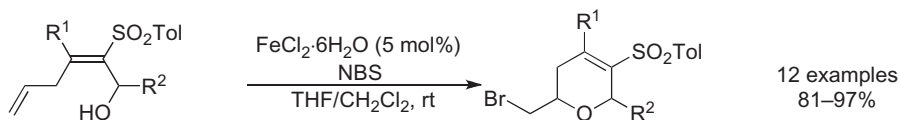
Scheme 15

The synthesis of 3,6-dihydro-2*H*-pyrans proceeds through a Lewis acid-catalyzed 6-*endo-trig* cyclization of β -hydroxy- γ,δ -unsaturated alcohols (Scheme 16) <11T9870>.



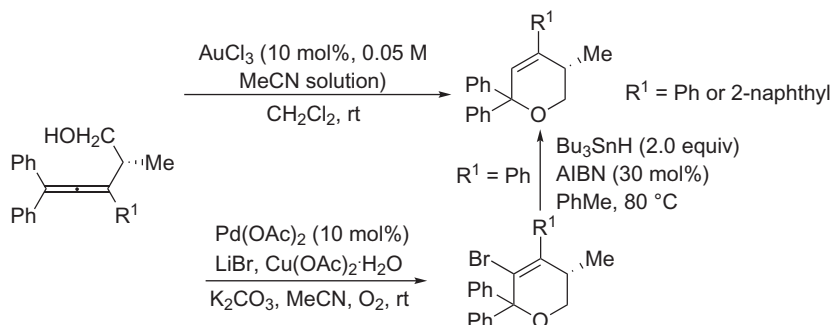
Scheme 16

The FeCl₃·6H₂O-catalyzed bromocyclization of polysubstituted 2-tosyl-2,5-hexadien-1-ol provides 2,3,4,6-tetrasubstituted 3,6-dihydro-2*H*-pyrans (Scheme 17) <11SC1227>.



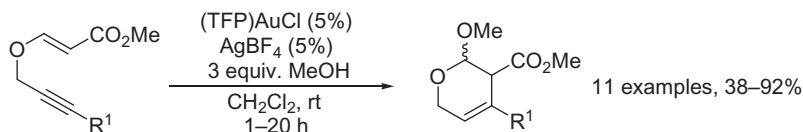
Scheme 17

Allenyl alcohols underwent Au(III)-catalyzed cyclization to give 3,6-dihydro-2*H*-pyrans, which were also obtained by an asymmetric bromoetherification of the same allenyl alcohols followed by a debromination reaction (Scheme 18) <11CEJ5796>.



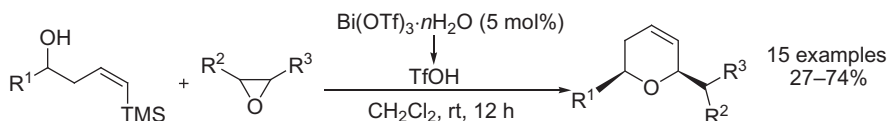
Scheme 18

3,6-Dihydro-2*H*-pyrans were obtained using the Toste protocol for the cyclization of propargylic ethers, with tris-2-furylphosphine (TFP) as ligand (Scheme 19) <11CC9390>.



Scheme 19

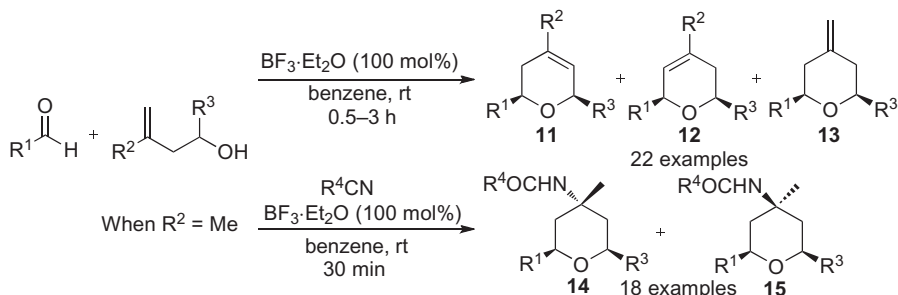
Catalytic Bi(OTf)₃ initiates a cascade reaction involving an epoxide rearrangement into an aldehyde electrophile, followed by an intermolecular addition of the *Z*- δ -hydroxyalkenylsilanes and an intramolecular silyl-modified Sakurai reaction to afford *cis*-2,6-disubstituted 3,6-dihydro-2*H*-pyrans (Scheme 20) <11JOC9269>.



Scheme 20

An efficient and diastereoselective synthesis of 4-alkyl/aryldihydro-**11,12** and 4-methylene tetrahydropyrans **13** is obtained from the reaction of aldehydes and homoallyl alcohols mediated by BF₃·Et₂O. 4-Amido tetrahydropyrans **14,15** are obtained when nitrile nucleophiles are included in the reaction medium (Scheme 21) <11OBC3428>. This oxonium-ene cyclization reaction can also be

applied in the synthesis of oxabicyclo[3.3.1]nonenes and substituted tetrahydropyrans from the reaction of geraniol and aldehydes or epoxides <11OBC4626>.



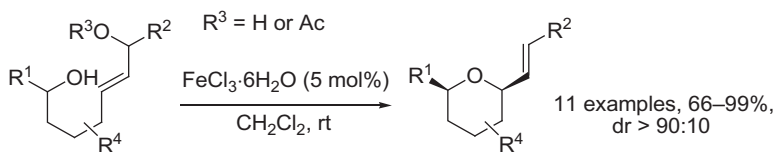
Scheme 21

Ring opening of diarylvinyliidencyclopropanes connected to alcohol-bearing chains is promoted by halo reagents affording a variety of halogenated tetrahydropyrans in moderate to good yields (Scheme 22) <11SL995>.



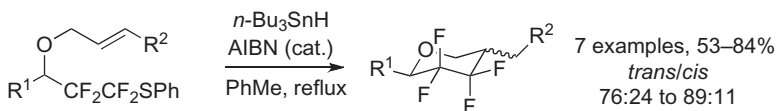
Scheme 22

Substituted *cis*-2,6-tetrahydropyrans can be chemo- and diastereoselectively prepared from ζ -hydroxy allylic alcohols (or acetates) with a catalytic $FeCl_3 \cdot 6H_2O$ (Scheme 23) <11T5024>.



Scheme 23

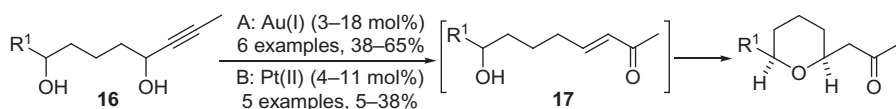
Tetrafluorotetrahydropyrans are obtained by a 6-*exo* radical cyclization of the appropriate allyl ethers (Scheme 24) <11EJO4528>.



Scheme 24

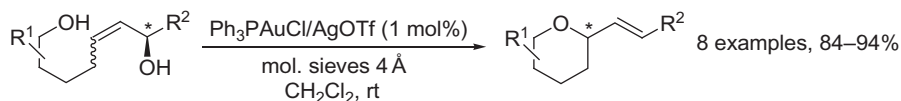
cis-2,6-Disubstituted tetrahydropyranylacetic acids can be prepared by a diastereoselective Pd-cyclization–carbonylation–hydroxylation of hept-6-en-2-ols or hexen-5-en-1,3-diols in moderate to good yields (40–88%) <11T4980>.

2,6-Disubstituted tetrahydropyrans were synthesized from 6-alkynyl-1,5-diols **16** in a gold(I)- (most efficient) or platinum(II)-mediated reaction. The metal induces an initial Meyer–Schuster rearrangement yielding hydroxyenones **17** that undergo an oxa-Michael addition (Scheme 25) <11SL1523>.



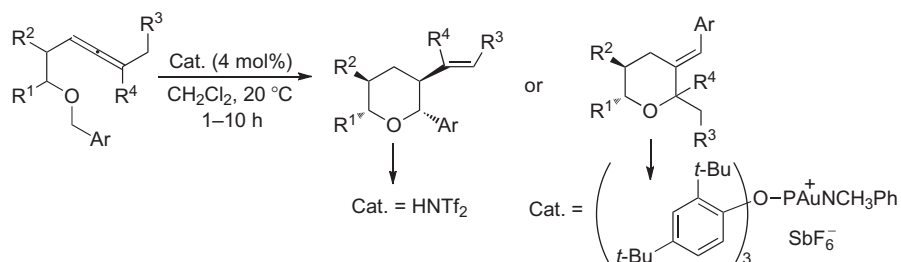
Scheme 25

Gold(I)-catalyzed cyclization of monoallylic diols forms tetrahydropyrans. The allylic alcohol stereochemistry controls the facial selectivity (Scheme 26) <11OL1330>. Gold(I)-catalyzed *exo*-cyclization of 3,3-disubstituted 1,4-diyne provides the corresponding 2-methylene tetrahydropyrans <11OL224>.



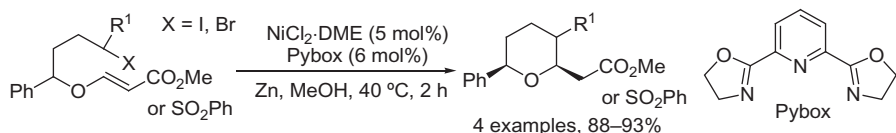
Scheme 26

A series of allenyl ethers were transformed into two types of tetrahydropyrans following a hydride shift–cyclization sequence catalyzed by a Au(I) complex or Brønsted acid. A clear-cut divergence in product selectivity depended on the catalyst (Scheme 27) <11JA7696>.

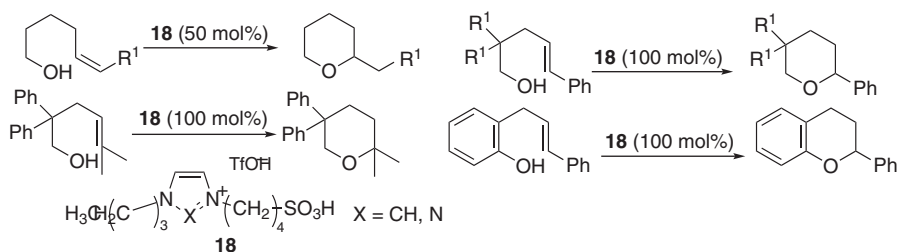


Scheme 27

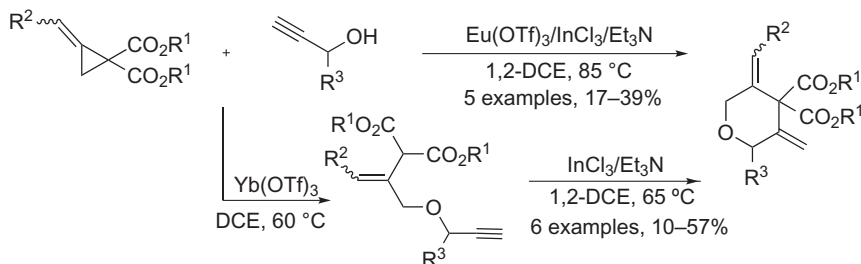
A mild and convenient Ni-catalyzed radical cyclization of organohalides led to *cis*-2,6-substituted tetrahydropyrans; zinc in methanol is the reductant (Scheme 28) <11OL2050>.

**Scheme 28**

Both SO₃H-tethered imidazolium and tetrazolium ionic liquids are effective in an intramolecular hydroalkoxylation of appropriate alkenyl alcohols giving tetrahydropyran derivatives (Scheme 29) <11OBC374>.

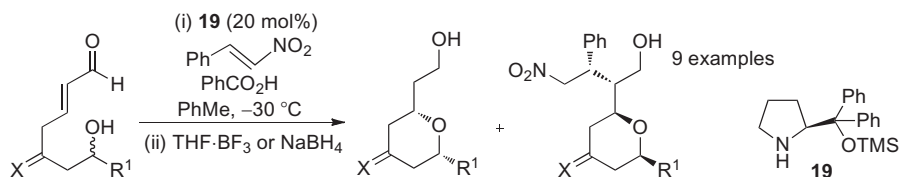
**Scheme 29**

Several Lewis acids catalyze the ring opening of methylenecyclopropanes with propargyl alcohols and subsequent intramolecular Conia-ene reaction, leading to 3,5-dimethylenetetrahydropyrans, in a stepwise or a one-pot process (Scheme 30) <11T763>.

**Scheme 30**

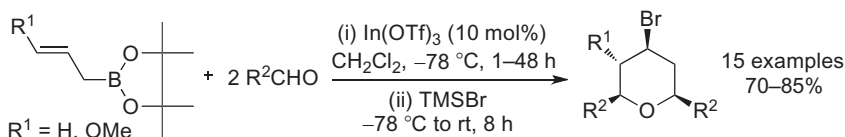
Intra- and intermolecular oxa-Michael additions of alcohols are well-known methods for the preparation of pyrans, but their enantioselective versions are complicated due to a possible reversibility. Therefore, enantioselective versions were used in the synthesis of some natural products <11CC7200, 11CC7440, 11OL5816>. An organocascade kinetic resolution, initiated by a highly selective and rapidly reversible oxa-Michael addition, provided enantioenriched *cis*-2,6-tetrahydropyrans (Scheme 31) <11OL6460>. *cis*-2,6-Disubstituted tetrahydropyrans resulted from a stereoselective Brønsted acid-catalyzed intramolecular oxa-conjugate cyclization of α,β -unsaturated

thioesters <11OL1820> and ketones <11T5034, 11TL1003>. A similar diastereoselective oxa-Michael strategy was used to prepare naphthopyrans, which are intermediates in the synthesis of several pyranonaphthoquinone antibiotics <11OBC5423>.



Scheme 31

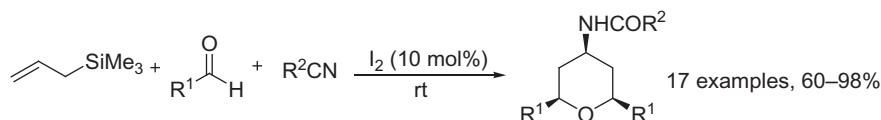
The classical Prins cyclization reaction is one of the major methods for the synthesis of di- and tetrahydropyrans. Two modifications, involving the triflate-trapped Prins-adduct and the Mukayama-aldol-silyl-Prins reaction, were employed in the total synthesis of (±)-civet <11T5107>. Tri- and tetrasubstituted tetrahydropyrans can be prepared by an In(OTf)₃-catalyzed one-pot reaction, involving allyl-*E*-crotylboration-Prins cyclization reactions (Scheme 32). The *Z*-crotylboration-Prins cyclization gives all *syn*-configuration <11TL4378>.



Scheme 32

Cu(OTf)₂-bisphosphine catalyzes synthesis of tetrahydropyrans from 5-methyl-5-hexen-1-ol and appropriate aldehydes mildly, in good yields and excellent *trans*-diastereoselectivity via an olefin migration followed by a Prins cyclization <11OL4328>.

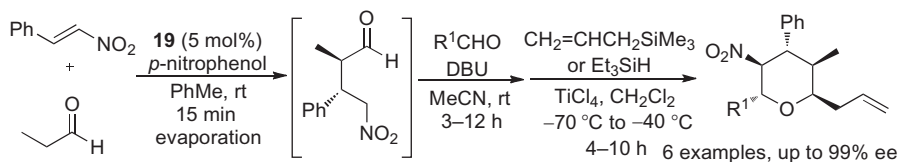
4-Amidotetrahydropyrans result from a Sakurai-Prins-Ritter sequence between allyltrimethylsilane, aldehydes, and nitriles, with iodine as catalyst (Scheme 33) <11ISC8>.



Scheme 33

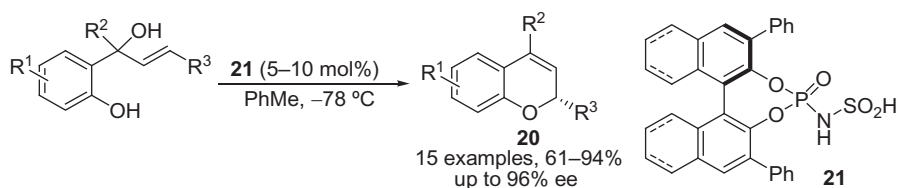
An asymmetric one-pot, four-component coupling reaction led to tetra- and penta-substituted tetrahydropyrans with excellent enantioselectivity through a Michael-Henry reaction-acetalization-Lewis acid-mediated allylation reaction. The

synthesis started with an organocatalyzed diastereoselective Michael reaction (Scheme 34) <11AGE3774>.

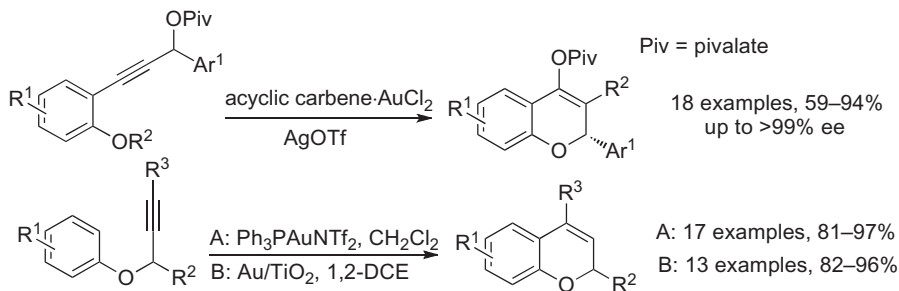


6.4.2.2 [1]Benzopyrans and Dihydro[1]benzopyrans (Chromenes and Chromans)

2*H*-Chromenes **20** can be synthesized by an enantioselective metal-free intramolecular allylic substitution catalyzed by a chiral Brønsted acid **21** (Scheme 35) <11JA3732>, radical aromatic substitution of (2-halobenzyl)aryl ethers mediated by *t*-BuOK <11CC9813, 11OL3242>, or Rauhut–Currier reaction of nitroolefins with tethered α,β -unsaturated esters <11CEJ6484>.

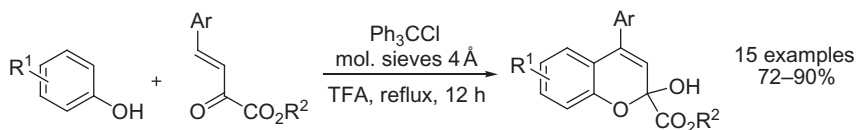


Substituted 2*H*-chromenes are available from iron(III)-mediated intramolecular alkyne-carbonyl metathesis <11JOC3539> and annulation tandem reaction <11OL14>. Selective cycloisomerization of propargylic esters <11JA12972> and aryl propargyl ethers <11CC803, 11EJO2334>, promoted by gold catalysts, also affords a wide range of functionalized chromenes (Scheme 36).



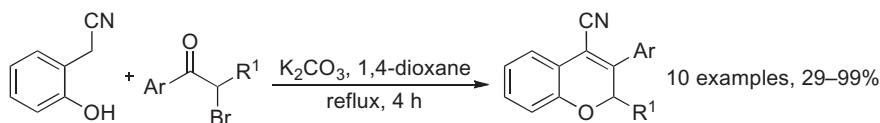
Knoevenagel condensation of carbonyl compounds with β -keto esters is a metal-free approach for the synthesis of 2*H*-chromenes <11HCA248>. Domino oxa-Michael–aldol condensation of α,β -unsaturated carbonyl compounds with salicylaldehydes in the presence of an organocatalyst <11EJO5031> and polystyrene resin-supported salicylaldehydes in the presence of potassium carbonate <11SL161> provide various 2*H*-chromene-type derivatives.

Under acidic conditions, the reaction of β,γ -unsaturated α -keto esters with phenols allows the one-pot synthesis of 4-aryl-2*H*-chromenes (Scheme 37). This selective cascade reaction is a complex process involving Friedel–Crafts alkylation, cyclodehydration, intermolecular hydrogen transfer, and hydration <11OBC2868>.



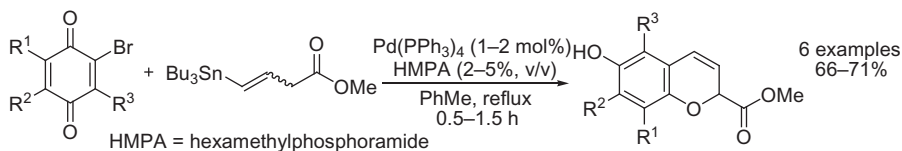
Scheme 37

2,2-Dimethyl-2*H*-chromenes are obtained by a one-pot Re(I)-catalyzed reaction of substituted phenols with 2-methyl-3-butyn-2-ol <11TL3926>, whereas 4-carbonitrile derivatives can be prepared by a one-pot tandem reaction of 2-(2-hydroxyphenyl)acetonitrile with 1-aryl-2-bromoethanones in the presence of base (Scheme 38) <11H(83)1355>.



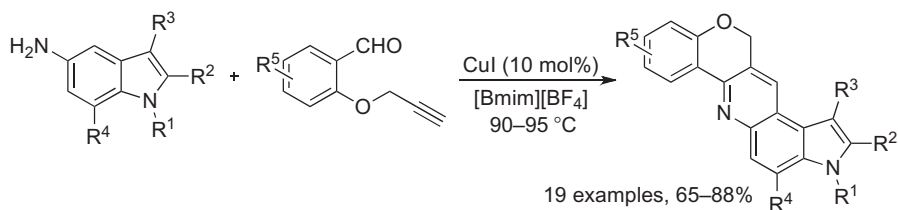
Scheme 38

Highly functionalized 2*H*-chromenes are obtained by a one-pot formal [3 + 3] cycloaddition reaction of bromobenzoquinones and vinyl stannanes (Scheme 39). The three-step sequence likely involves Stille coupling, HMPA-mediated enolization, and thermal electrocyclic ring closure <11T9779>.

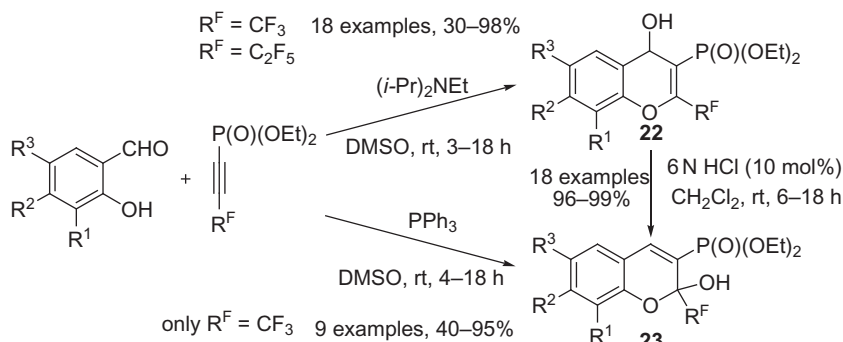


Scheme 39

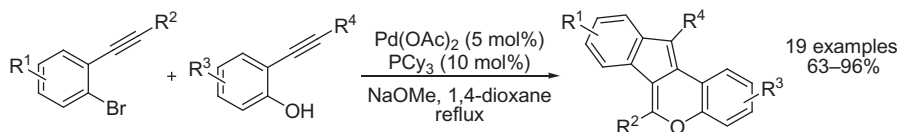
The CuI-promoted aza-Diels–Alder reaction of 5-aminoindoles with *O*-propargyl salicylaldehydes is performed in the ionic liquid [Bmim][BF₄], and a range of dihydro-2*H*-chromenes are obtained (Scheme 40) <11TL4857>.

**Scheme 40**

The synthesis of 2-perfluoroalkyl 4*H*- and 2*H*-chromenylphosphonates is conditions-controlled: regioselective cycloaddition of salicylaldehydes with perfluoroacetylphosphonates using *i*-Pr₂NEt furnishes 4*H*-chromenes **22**, while in the presence of triphenylphosphine, 2*H*-chromenes **23** are obtained as major products (Scheme 41). The acid-catalyzed isomerization of the formed 4*H*-chromenes also affords the corresponding 2*H*-regioisomers <11JOC71>.

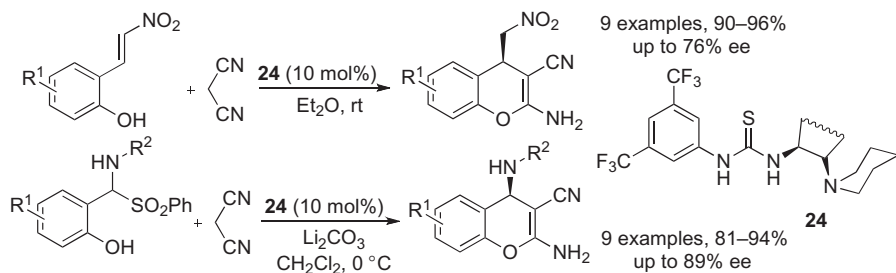
**Scheme 41**

2-Bromobenzyl bromide and β-keto esters undergo a Cu(I)-catalyzed domino *C*-benzylation–*O*-arylation reaction in ligand-free conditions leading to the selective formation of 4*H*-chromenes <11OL1972>. The Pd-catalyzed cascade reaction of 2-alkynylhalobenzenes with 2-alkynylphenols in the presence of tricyclohexylphosphine (PCy₃) as ligand affords indeno[1,2-*c*]chromenes (Scheme 42) <11CC5298>.

**Scheme 42**

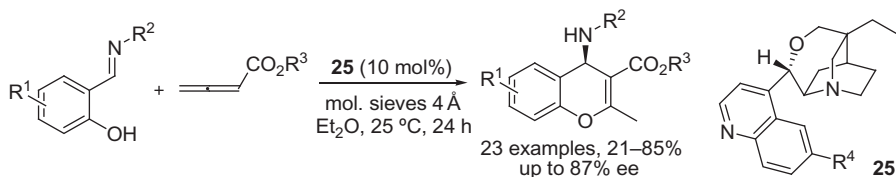
2-Amino-5-hydroxy-4*H*-chromenes are obtained in good yields (73–90%) by a one-pot condensation reaction of resorcinols with 2-benzylidenemalononitriles <11T8271>. Asymmetric 2-amino-4*H*-chromenes are readily accessible via a Mannich cyclization–tautomerization cascade sequence in which malononitrile acts as both

nucleophile and electrophile, in the presence of bifunctional chiral thiourea organocatalyst **24** (Scheme 43) <11CEJ7781, 11TL6137>. Related derivatives result from tandem Michael addition–cyclization reactions of cyclohexane-1,3-diones and 2-cyano-3-phenylacrylates catalyzed by a Salen–Co(II) complex <11EJO137> or a bifunctional chiral thiourea organocatalyst <11TL6792>, in good yields and enantioselectivity.



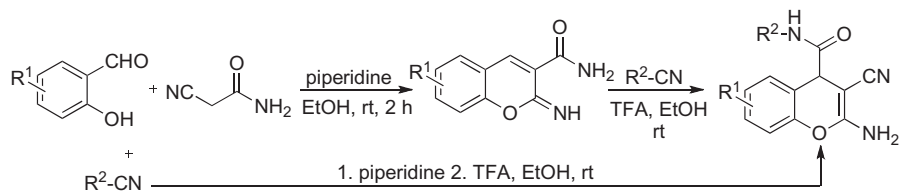
Scheme 43

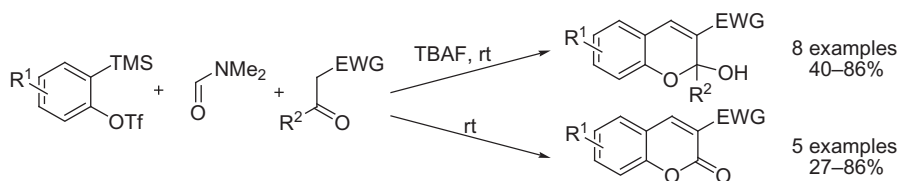
Several cinchona-based catalysts **25** have been designed to perform the asymmetric [4 + 2] cycloaddition reaction of salicyl *N*-tosylimine derivatives with allenic esters. The corresponding 4*H*-chromenes are obtained in moderate to good yields and high enantioselectivity (Scheme 44) <11TA1239>.



Scheme 44

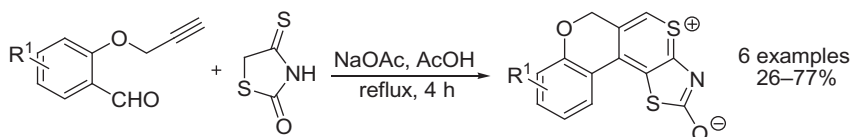
The synthesis of 4-substituted 2-amino-3-cyano-4*H*-chromenes can be achieved by a one- or two-step sequence starting from salicylaldehydes, cyanoacetamide, and isocyanides in moderate to good overall yields (30–77%) (Scheme 45) <11EJO848>. Similar products arise from the Zn-catalyzed reaction of salicylaldehydes, malononitrile, and indoles, in good yields (37–89%) and enantioselectivity (up to 90% ee) <11OL4910>. Other multicomponent coupling reactions of triflates as aryne precursors, DMF and ketones, or β -keto esters as active methylenes afford, respectively, chromenes and coumarins (Scheme 46) <11AGE6638>.





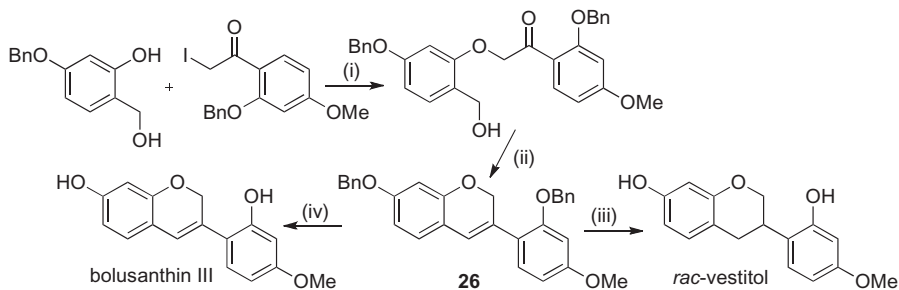
Scheme 46

Chromeno[4',3':4,5]thiopyrano[2,3-*d*][1,3]thiazol-4-ium-2-olates are obtained by a domino Knoevenagel–hDA oxidation reaction based on the appropriate 2-(2-propynyloxy)benzaldehydes and 4-thioxo-1,3-thiazolidin-2-one (Scheme 47) <11TL2324>. When *N*-substituted 4-thioxo-1,3-thiazolidin-2-one <11TL2324> and 5-pyrazolones <11TL2853> react with 2-(2-propynyloxy)benzaldehydes, only the Knoevenagel–hDA reaction took place. Indole-annulated dihydropyran[3,4-*c*]chromenes result from the Knoevenagel condensation of *O*-propargylated salicylaldehydes with indolin-2-ones followed by a microwave-assisted intramolecular hDA reaction of the formed adducts <11TL4337>.



Scheme 47

The total syntheses of two chromene natural products were accomplished in a multistep sequence having the 2*H*-chromene **26** as the key intermediate. Racemic vestitol was obtained in a 29% overall yield and bolusanthin III in 21% overall yield (Scheme 48) <11SL1605>.



Reagents: (i) K_2CO_3 , Me_2CO , reflux 16 h, 78%; (ii) (a) $\text{PPh}_3\cdot\text{HBr}$, MeCN , rt, 1 h; (b) *t*-BuOK, MeOH , reflux, 24 h, 70%; (iii) Pd/C , EtOAc , H_2 (2.4 bar), rt, 14 h, 84%; (iv) BCl_3 , pentamethylbenzene, CH_2Cl_2 , -78°C , 15 min, 61%

Scheme 48

The asymmetric total synthesis of (–)-variabilin and (–)-glycinol involves a Buchwald–Hartwig coupling reaction <11OL3686>, whereas forming the tricyclic skeleton of phomactin A involved a Prins/Conia-ene cascade cyclization (Figure 1) <11JOC6534>.

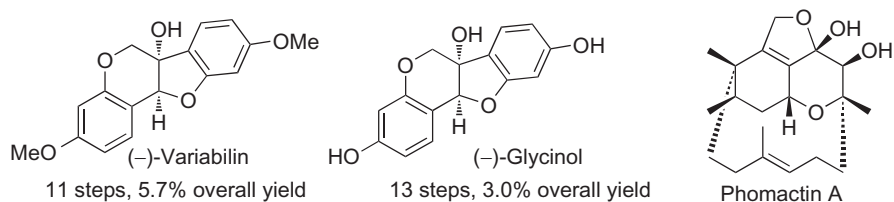
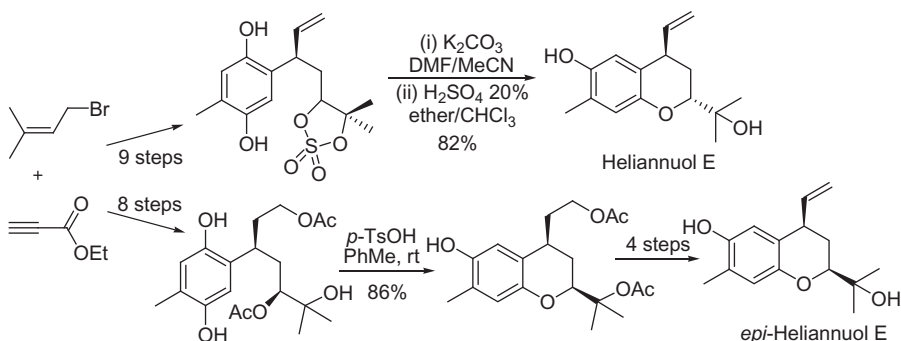


Figure 1

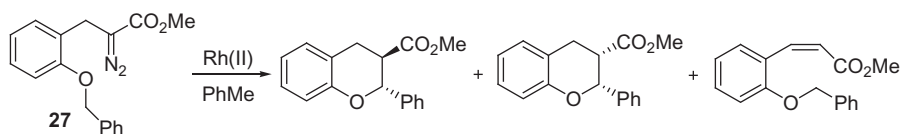
For heliannuol E and *epi*-heliannuol E, the asymmetric total synthesis needed 10 and 13 steps, respectively (Scheme 49). The formation of the chroman motif is considered to proceed through an intramolecular phenol attack on the sulfate moiety in the former case and an acyl transfer–secondary carbocation capture sequence in the latter <11TL5802>.



Scheme 49

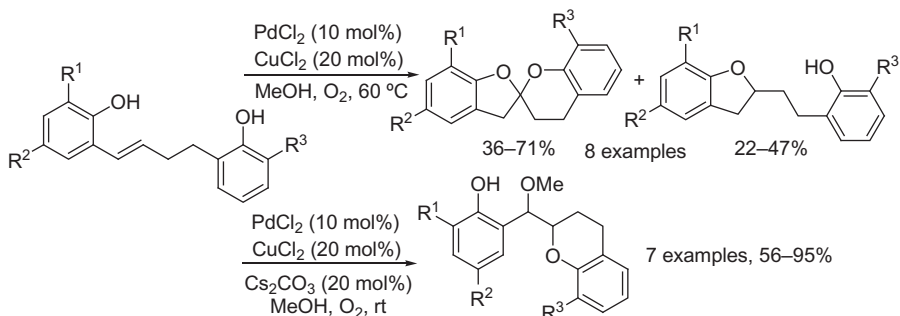
2*H*-Chromans are obtained by a diastereoselective $Et_3SiH/TMSOTf$ reductive deoxygenation of substituted 2-sulfinylmethylchroman-2-ols and their methyl ketals <11EJO3864>.

Several Rh(II) catalysts have been used for the decomposition of diazo compound 27; a diastereomeric mixture of chromans, resulting from the intramolecular 1,6-C–H insertion, and the *Z*-isomer of the β -elimination are the products obtained (Scheme 50) <11T3071>.



Scheme 50

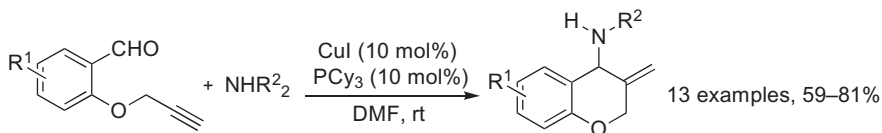
Bimetallic tandem cyclization of olefins proved to be a versatile route for the synthesis of substituted chromans <11TL3509>; the presence or absence of base is responsible for the chemoselectivity (Scheme 51). [5,6]-Bisbenzannelated spiroketals are the major products of the tandem Wacker cyclization–aroxylation in the absence of base, whereas 2-substituted chromans are formed in the presence of base through tandem Wacker cyclization–Michael addition reaction <11SL1579>.



Scheme 51

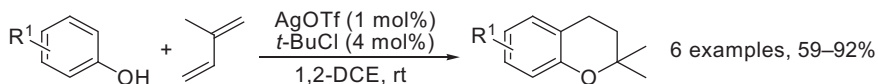
A stereoselective PtCl_4 -catalyzed cycloaddition reaction of *o*-quinone methides with olefins affords *trans*-2,3-disubstituted chromans. The intramolecular version led to decreased diastereoselectivity <11T3904>.

4-Amino-3-methylenechromans result from the CuI-catalyzed reaction of *O*-propargyl salicylaldehydes with dialkylamines (Scheme 52). The mechanism involves the formation of an iminium ion followed by intramolecular inverse electron demand ene-type reaction, with the loss of an alkyl group from the dialkylamine <11T1617>.



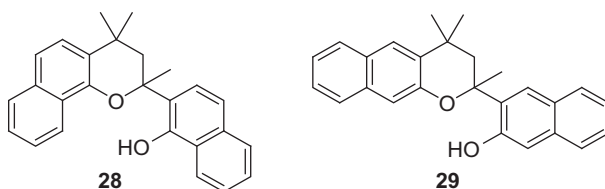
Scheme 52

A (1:4) mixture of $\text{AgOTf}/t\text{-BuCl}$ is implicated in the synthesis of 2,2-dimethylchromans through a hidden Brønsted acid catalysis rather than a silver catalysis of phenols and isoprene (Scheme 53) <11JOC9353>. A number of 2-arylchromans were synthesized through the aldol condensation of salicylaldehydes and acetophenones and subsequent reduction and cyclization of the 2'-hydroxychalcone intermediates <11TL6716>.

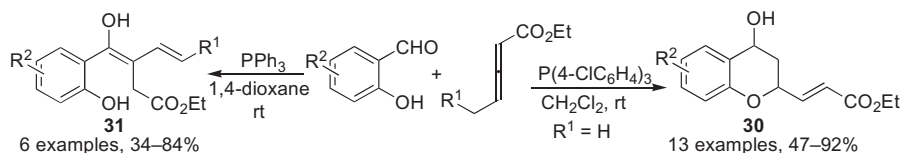


Scheme 53

2-Acetyl-1-naphthol and ethyl 3-hydroxy-2-naphthoate undergo a Grignard reaction, dehydration of the corresponding tertiary alcohols, and hDA dimerization giving benzochromans **28** and **29** <11JHC952>.

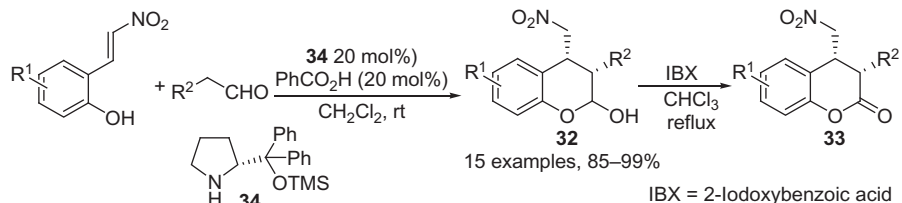


Phosphine-mediated annulation of salicylaldehydes and γ -substituted allenates provided several functionalized chromans **30** and 1,3-dienes **31**, depending on the phosphine catalyst (Scheme 54) <11T1053>.



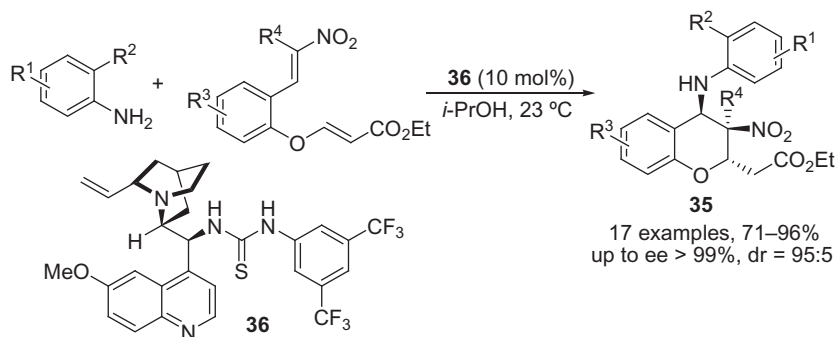
Scheme 54

Amine **34**-catalyzed reaction of 2-(2-nitrovinyl)phenols with aldehydes led to 4-nitromethylchroman-2-ols **32**, as asymmetric cascade Michael-acetalization adducts. Further oxidation furnished the corresponding 3,4-dihydrocoumarins **33** (Scheme 55) <11OBC2715>. Using **34**, β -tetralones reacted with α,β -unsaturated aldehydes giving 4-substituted benzo[*f*]chroman-2-ols (50–99%) with high enantioselectivity (up to 96%) <11OBC7510>.



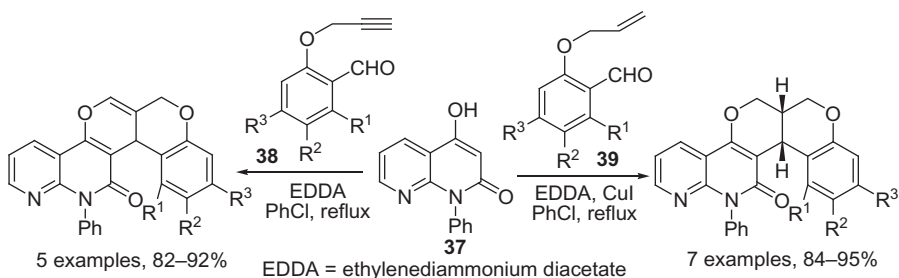
Scheme 55

Aziridination of aryl cinnamyl ethers followed by intramolecular arylation provided a one-pot synthesis of 3-amino-4-arylchromans. This regio-, diastereo- ($dr > 99:1$), and enantioselective (up to 95%) methodology used copper catalysts and chiral bis-oxazoline ligands <11JOC7334>. An enantioselective synthesis of 4-aminochromans **35** was achieved by an organocatalyzed asymmetric cascade aza-Michael–Michael addition reaction of anilines with nitroolefin enoates (Scheme 56) <11OL808>.



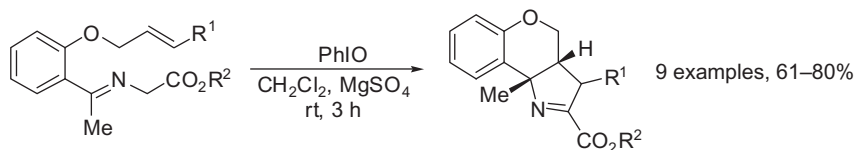
Scheme 56

A series of novel structurally complex chroman derivatives resulted from a domino Knoevenagel–hDA reaction of 4-hydroxy-1-phenyl-1,8-naphthyridin-2(1*H*)-one **37** with *O*-propargylated **38** and *O*-allylated salicylaldehydes **39** (Scheme 57) <11S3716>. In aqueous solution, 3-hydroxy-2-naphthalenemethanol and several vinyl ethers <11JA5573> and immobilized vinyl ethers <11JA15730> undergo light-induced hDA reaction to give benzo[*g*]chromans.



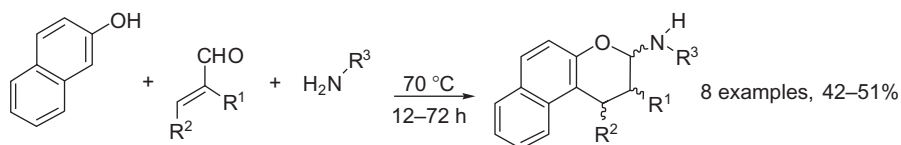
Scheme 57

Benzochromans are isolated from the reaction of salicylaldehydes and glycols <11CC10266>, and of salicylaldimines and 2,3-dihydro-2*H*-furan <11CEJ13684>, using Sc(OTf)₃ as catalyst, and by FeCl₃·6H₂O-mediated dimerization of 2*H*-chromenes <11OL6480>. Other examples were obtained after oxidative cyclization of vinylogous esters in the presence of iodobenzene and *m*-chloroperbenzoic acid <11CC11778>, and formal intramolecular 1,3-dipolar cycloaddition of ketoimines under an organic Lewis acid *cum* oxidant (Scheme 58) <11CC1285>.



Scheme 58

In solvent-free conditions, the one-pot, three-component reaction of 2-naphthol, α,β -unsaturated aldehydes, and amines results in the preparation of enantiopure benzo[*f*]chroman-2-amine derivatives (Scheme 59) <11TA1542>.

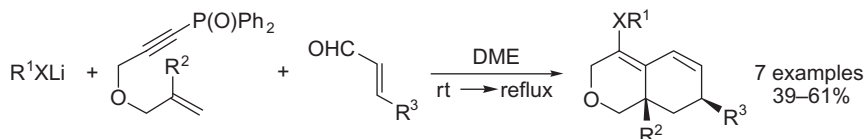


Scheme 59

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

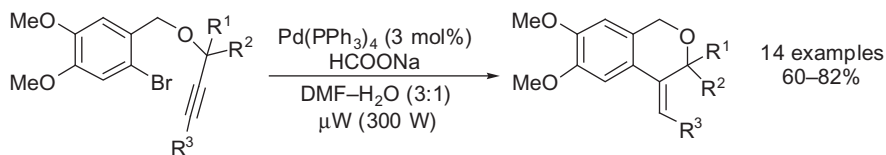
A facile one-pot synthesis of isochromenes is carried out in the presence of a rhodium catalyst in the oxidative coupling reaction of benzyl alcohols and alkynes <11JOC9548>. Gold(I)-catalyzed rearrangement of arylmethoxylated alkynylaziridines gave spiro[isochroman-4,2'-pyrrolines] in high yields <11CC6665>, while the enantioselective cycloisomerization of alkynols is substrate controlled: electron-donating groups provide isochromenes as major products via 6-*endo-dig* cyclization, while electron-withdrawing groups induce 5-*exo-dig* cyclization to afford 1,3-dihydroisobenzofurans <11JOC8869>.

Tetrahydro-1*H*-isochromenes are obtained by a stereoselective sequential three-component Michael addition–aldol–Horner–Wadsworth–Emmons–[4 + 2] cycloaddition reaction (Scheme 60) <11JOC1440>.

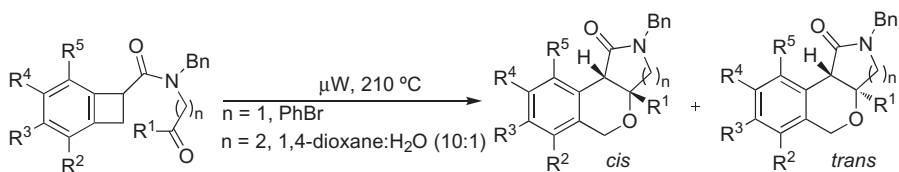


Scheme 60

Microwave irradiation was applied to the cyclization of 2-bromobenzyl propargyl ethers using $\text{Pd}(\text{PPh}_3)_4$ as catalyst and sodium formate as reducing agent. A wide range of isochromans were prepared in a regio- and stereoselective process (Scheme 61) <11SL2733>. The intramolecular hDA reaction of benzocyclobutenes gave pyrrole- and pyridine-fused isochromans under microwave (μW) conditions (Scheme 62) <11TL3413, 11TL3417>.



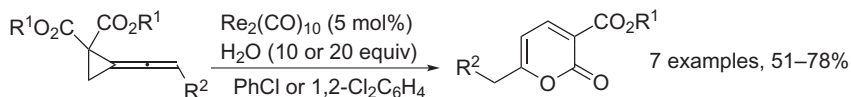
Scheme 61



Scheme 62

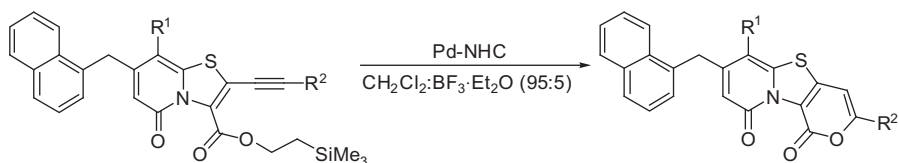
6.4.2.4 Pyranones

2*H*-Pyran-2-ones are accessible from an intramolecular ring-opening reaction of vinylidenecyclopropane diesters catalyzed by $\text{Re}_2(\text{CO})_{10}$ (Scheme 63). The regioselective carbon–carbon bond cleavage is sensitive to the electronic properties of the substituents <11EJO1099>.



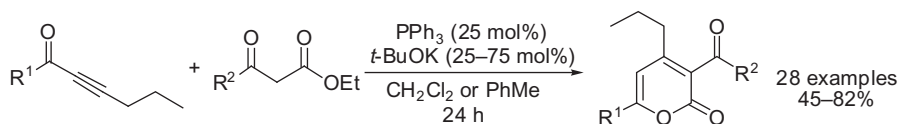
Scheme 63

A Pd–NHC complex catalyzes the completely selective 6-*endo-dig* cyclization of internal alkynes to the corresponding pyran-2-ones in quantitative yields, using the appropriate acidic conditions (Scheme 64) <11JOC9817>. Other pyran-2-one-fused heterocyclic compounds were obtained by an alkaline CuI-mediated tandem coupling oxacyclization reaction of β -iodo- α,β -unsaturated carboxylic acids with terminal alkynes <11JOC8347, 11OBC1212>.



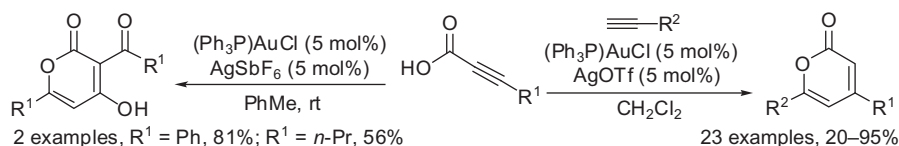
Scheme 64

Functionalized pyran-2-ones are obtained by annulation of alkynyl ketones with β -keto esters in the presence of *t*-BuOK (Scheme 65) <11SC3147> or with active methylene compounds in the presence of sodium hydride in DMSO at room temperature <11SC2738>.



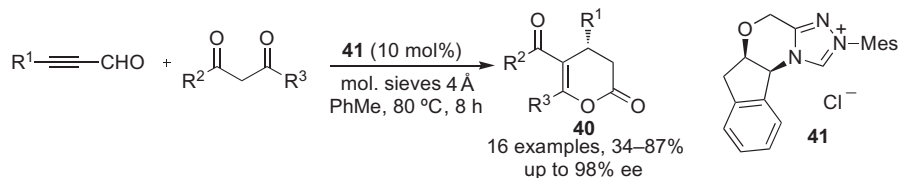
Scheme 65

Gold(I)-catalyzed cascade reaction of propiolic acids and terminal alkynes provided substituted pyran-2-ones. Dimerization of propiolic acids occurred when a different counter ion is used (Scheme 66) <11OL2834>.



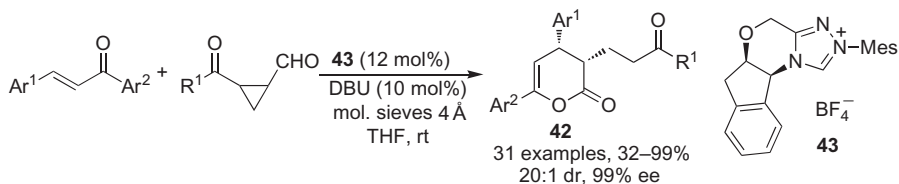
Scheme 66

Dihydropyran-2-ones are readily available through a cascade epoxide opening and lactonization of γ -epoxy- α,β -enals, catalyzed by a thiazolium salt <11OBC5948>. NHC-mediated annulation reactions of enals with vinyl ketones also afford several 3,4-dihydro-2*H*-pyran-2-one derivatives <11TL5992>, the asymmetric version occurring in a highly regio- and stereoselective way <11AGE1910>. The reaction mechanism is consistent with a Michael-type addition and subsequent intramolecular cyclization. Several chiral NHC-catalyzed annulation reactions of α,β -unsaturated enals <11OL4080, 11OL4966> and ynals <11CC8670> with 1,3-diketones (Scheme 67) allow the synthesis of functionalized 3,4-dihydro-2*H*-pyran-2-ones **40**, with excellent enantioselectivities.



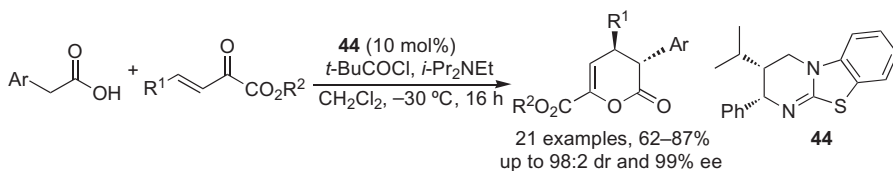
Scheme 67

With NHCs as catalyst, the formal DA reaction of 2-(*p*-nitrobenzoyloxy)-3-phenylpropanal with an β,γ -unsaturated α -ketoester affords a 3,4-dihydropyran-2-one with lower diastereoselectivity <11CC373>. However, a similar approach using chiral NHC catalyst **43** for the reaction of chalcones with formylcyclopropanes provides 3,4-dihydropyran-2-ones **42** with high diastereo- and enantioselectivities (Scheme 68) <11OL5366>. DA reaction of enals and alkylidene diketones, catalyzed by **43** <11OL4708>, and of α -halo- β -substituted propanal derivatives with α,β -unsaturated ketones, catalyzed by **41** <11T9329>, gives 3,4-dihydropyran-2-ones with excellent enantioselectivity. A wide range of derivatives are also formed by the enantio- and diastereoselective cycloaddition reactions of β,γ -unsaturated α -ketoesters with azalactones catalyzed by a chiral Brønsted base <11CEJ1760>, or with oxazolones catalyzed by cinchona alkaloid derivatives <11T3337>, and Brassard-type diene with aliphatic aldehydes catalyzed by a In(III) complex <11OL3868>.



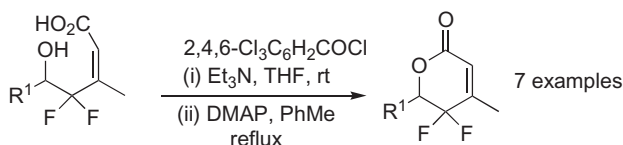
Scheme 68

An isothiourea catalyzes the highly diastereo- and enantioselective intermolecular Michael addition–lactonization of arylacetic acids with β,γ -unsaturated α -keto esters giving the *anti*-3,4-disubstituted pyran-2-ones (Scheme 69) <11JA2714>.



Scheme 69

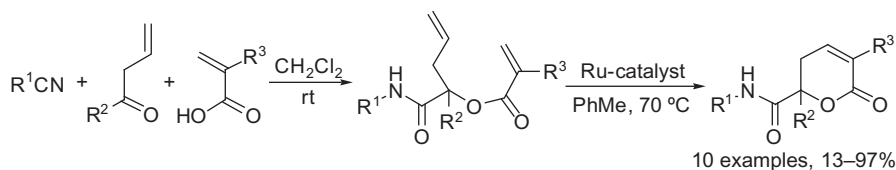
The preparation of fluorinated 5,6-dihydro-2*H*-pyran-2-ones is accomplished in a multistep sequence starting from the fluoroallylboration of aldehydes. In the final step, the lactonization of the δ -hydroxy acids formed under Yamaguchi conditions leads to the 5,5-difluoropyran-2-ones (Scheme 70) <11OL1302>. Oxidative ring closure of 4-fluoro-1,5-dienols also affords 5-fluorodihydropyran-2-ones <11JOC6525>. Regioselective cyclization of γ,δ -unsaturated carboxylic acids provides 5,6-dihydropyran-2-ones using diphenyl diselenide as catalyst and hypervalent iodine reagents as oxidants <11OL6504>.



Scheme 70

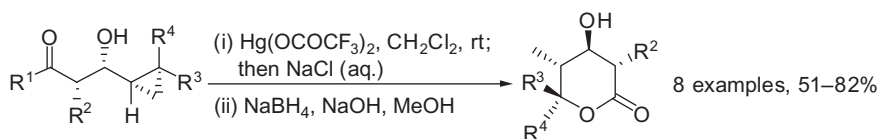
3,3-Dichloro-4-hydroxytetrahydro-2*H*-pyran-2-ones were prepared by cathodic reduction of 2,2-disubstituted 3-oxopropyl trichloroacetates in dichloromethane–tetraethylammonium chloride. If the reaction mixture is kept at 3 °C for 18 h, 3-chloro-4-hydroxy-5,6-dihydropyran-2-ones are the products obtained <11EJO4681>.

A three-component reaction of isocyanides, allyl ketones, and α,β -unsaturated carboxylic acids followed by Ru-catalyzed ring-closing metathesis led to 5,6-dihydropyran-2-ones (Scheme 71) <11EJO4335>. The same type of ring-closing metathesis was applied in the synthesis of biologically active 5,6-dihydro-2*H*-pyran-2-ones <11TA1749>.



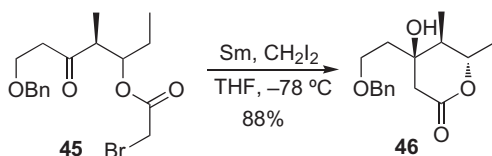
Scheme 71

The asymmetric bromolactonization of alkenoic acids catalyzed by thioureas and thiocarbamates provides 3-bromotetrahydropyran-2-ones in moderate to good yields (up to 95% ee) <11OL2738, 11TL4892>. A one-pot procedure, involving oxymercuration, cyclopropane ring opening, and reductive demercuration of cyclopropyl aldols, resulted in a series of chiral tetrahydropyran-2-ones (Scheme 72) <11OL3592>.



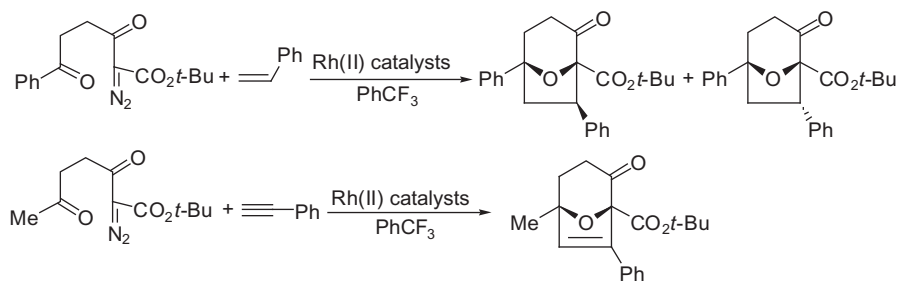
Scheme 72

In the synthesis of leiodermatolide, the tetrahydropyran-2-one **46** motif was prepared by a samarium-catalyzed internal Reformatsky–aldol reaction of ester **45** (Scheme 73) <11SL191>. In the synthesis of gonithalamin analogues, the Pd-catalyzed allylic oxidation of hept-6-enoic acids provides the appropriate tetrahydropyran-2-one, under White conditions <11SL2689>. Lactonization to the tetrahydropyran-2-one nucleus of leiocarpin C <11HCA1102> and (+)-tanikolide <11H(83)2601> was carried out with Amberlyst 15 in MeCN, while for simplactones A and B Bu₄NF in THF was used <11HCA1481>.

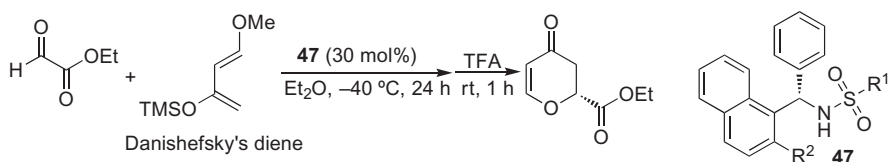


Scheme 73

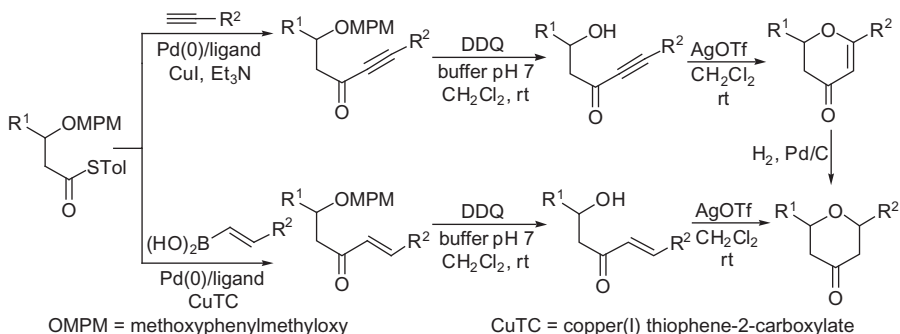
The construction of the 2,6-dihydropyran-3-one nucleus in (+)-bresisamide was based on a key Achmatowicz rearrangement of a properly functionalized furan moiety <11OL3636>. Tetrahydropyran-3-ones can be synthesized through a tandem carbonyl ylide formation–cycloaddition reaction of 2-diazo-3,6-diketoesters with styrene or phenylacetylene, catalyzed by polymer-supported dirhodium(II) complexes, under continuous flow conditions (Scheme 74) <11CEJ13992>.

**Scheme 74**

Several sulfonamide organocatalysts have been developed for the asymmetric hDA reaction of ethyl glyoxylate with Danishefsky's diene. Treating the adduct with trifluoroacetic acid gives the corresponding 2,3-dihydropyran-4-one (Scheme 75) <11H(83)2525>. Enantioselective salen-Cr(III)-catalyzed reaction of this diene with an aldehyde allows the preparation of the 2,3-dihydropyran-4-one core, an intermediate in the synthesis of galantinic acid <11EJO1223>.

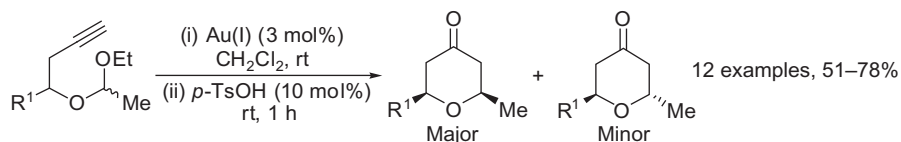
**Scheme 75**

The synthesis of several di- and tetrahydropyran-4-ones was accomplished by palladium cross-coupling reactions of thioesters with terminal alkynes or alkenylboronic acids. In the final step, an intramolecular AgOTf-promoted oxa-conjugate cyclization of the alkynyl and alkenyl derivatives led to dihydropyran-4-ones and tetrahydropyran-4-ones, respectively (Scheme 76) <11T4995>. A similar reaction was used in the synthesis of a dihydropyran-4-one precursor of diospongin A <11TA1725>.

**Scheme 76**

Tetrahydropyran-4-ones are available through an enantioselective Maitland-Japp reaction using Chan's diene as nucleophile. The reaction accommodates a wide

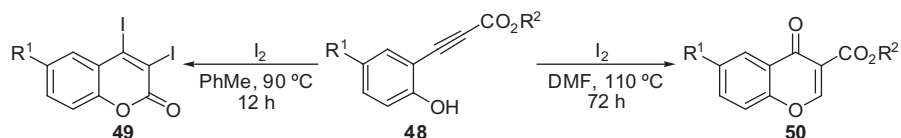
range of substituents in either the first or in the second aldehyde reaction partner <11T4960>. Several gold(I)-phosphine complexes have been involved in the asymmetric synthesis of tetrahydropyran-4-ones, in a two-step sequence starting from 4-(alkoxyalkyl)oxy-1-butyne, with a phosphine ligand playing an important role in the diastereoselectivity (Scheme 77) <11CEJ1433>.



Scheme 77

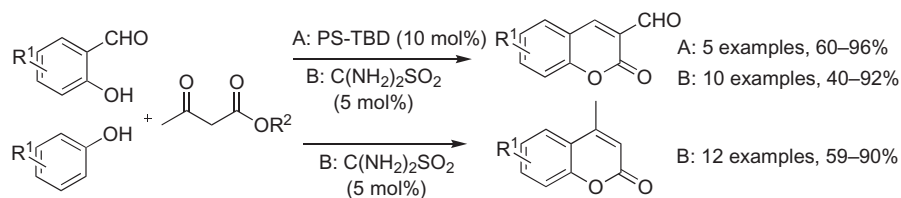
6.4.2.5 Coumarins

A tandem ring-closing metathesis-allylic oxidation sequence of *o*-allyloxystyrenes led to the formation of polysubstituted coumarins <11CC5879>. Several coumarins were also prepared by a Lewis acid-promoted cyclization of aryl 3-(dimethylamino)prop-2-enoates, which are obtained from the appropriate phenols <11HCA185>. The selective reaction of 3-(2-hydroxyphenyl)propioates **48** with iodine is conditions-controlled: using toluene as solvent gives coumarins **49**, while DMF affords chromones **50** (Scheme 78) <11TL4164>.



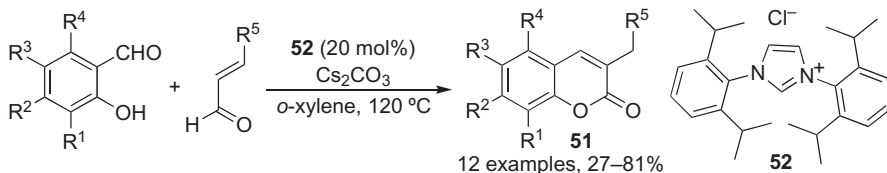
Scheme 78

Under solvent-free conditions, the synthesis of coumarins can be accomplished either by hydroarylation of propioates with substituted phenols using ZnCl_2 as catalyst <11S3692> or by Knoevenagel condensation of salicylaldehydes/phenols and β -keto esters using a supported base as catalyst <11EJO2874> or mediated by thiourea dioxide (Scheme 79) <11OBC6943>. Coumarin-type compounds are obtained by a chemoselective Knoevenagel condensation of aromatic aldehydes with Meldrum's acid to give the corresponding 5-arylidene derivatives followed by a tandem enol lactonization with a variety of active methylene compounds, using poly(ethyleneglycol)-stabilized Ni nanoparticles as catalyst <11TL3666>.



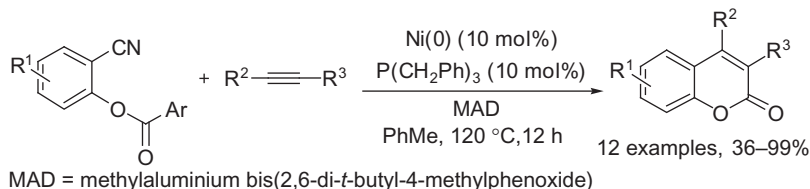
Scheme 79

Highly functionalized coumarins **51** result from the reaction of salicylaldehydes with α,β -unsaturated aldehydes in the presence of catalytic amounts of NHC generated *in situ* from **52** (Scheme 80). This synthesis involves a redox esterification of α,β -unsaturated aldehydes with simultaneous aldol condensation <11SL635>. 8-Formylcoumarins were also synthesized from salicylaldehydes and dimethyl acetylenedicarboxylate by an aromatic electrophilic substitution mediated by a vinyltriphenylphosphonium salt <11SL694>.



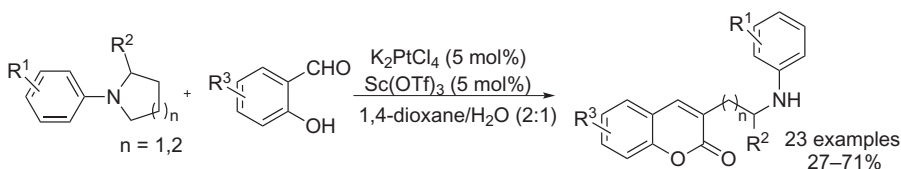
Scheme 80

Ni-promoted cycloaddition of *o*-aroyloxybenzonitriles with alkynes offers direct access to coumarins (Scheme 81). The mechanism involves an unusual cleavage of two independent C—CN and C—CO bonds <11JA11066>.



Scheme 81

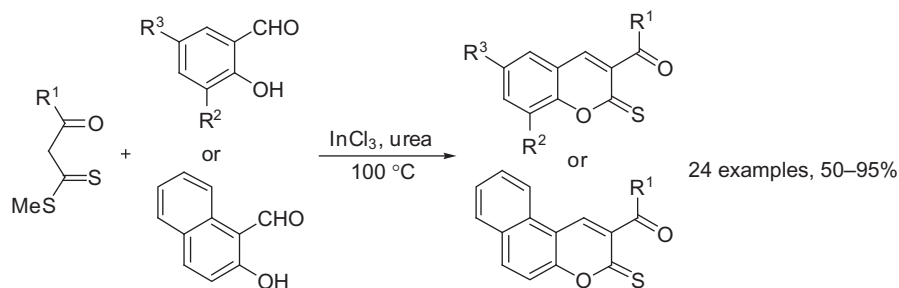
A bimetallic Pt/Sc system catalyzed the reaction of tertiary amines and substituted salicylaldehydes (Scheme 82). This one-pot synthesis involves aldol condensation, cyclization, and ring-opening processes to obtain the 3-(aminoalkyl)coumarins <11JOC342>.



Scheme 82

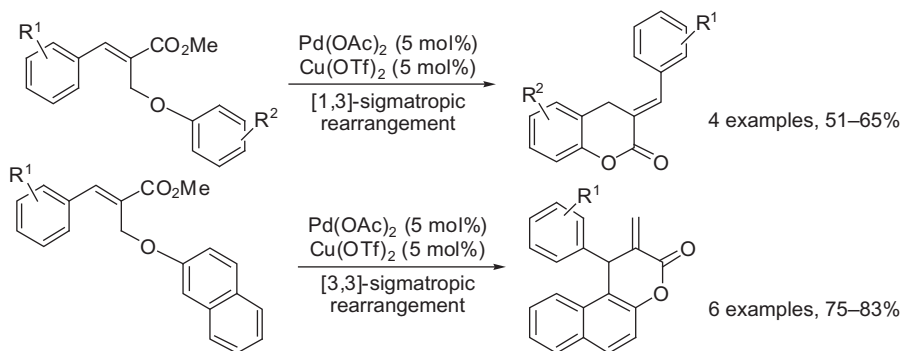
A three-component coupling reaction of benzyne, DMF, and active methylene compounds readily formed 3-substituted aliphatic and aromatic coumarins <11CC8512>.

In solvent-free conditions, several 2*H*-chromene-2-thiones result from the reaction of β -oxodithioesters and various salicylaldehyde-type compounds, catalyzed by InCl_3 in the presence of urea (Scheme 83) <11T584>.



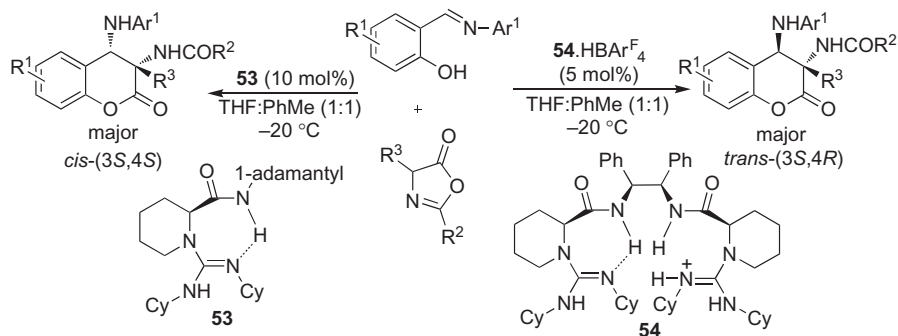
Scheme 83

A bimetallic Pd/Cu system catalyzes the regiospecific synthesis of 3,4-dihydrocoumarins involving a [1,3]- or [3,3]-sigmatropic rearrangement and cyclization of 2-(aryloxymethyl)prop-2-enoates (Scheme 84) <11SL3026>.



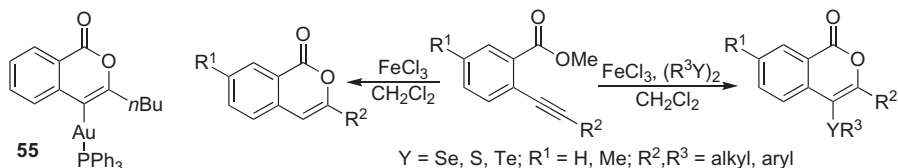
Scheme 84

Highly enantio- and diastereoselective synthesis of 3,4-dihydrocoumarins is achieved via a domino reaction of oxazolones with *o*-hydroxy aromatic aldimines in the presence of organocatalysts. The *cis*- and *trans*-isomers are obtained as the major products in the presence of guanidine **53** and bisguanidium salt **54**, respectively (Scheme 85) <11OL5060>. The asymmetric domino Michael–acetalization reaction of 2-hydroxynitrostyrenes and 2-oxocyclohexancarbaldehydes also provides 3,4-dihydrocoumarins with good diastereo- and enantioselectivities <11OBC382, 11OL5758>.



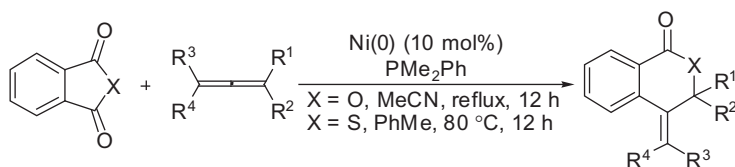
Scheme 85

The isolation and characterization of the isochromen-4-yl-gold(I) complex **55** allows rationalization of the glycosylation mechanism of glycosyl *o*-alkynylbenzoates <11AGE8329>. The oxidative cyclization of *o*-alkynylbenzaldehydes promoted by atmospheric oxygen and an NHC provides a mixture of phthalides and isocoumarins <11OL2228>. Regioselective cyclization of 2-alkynylaryl esters promoted by FeCl₃ for the synthesis of several isocoumarins is conditions-controlled: the presence and absence of a diorganyl dichalcogenide favor the presence and absence of the chalcogen motif in the structure, respectively (Scheme 86) <11JOC6789>. Isocoumarins are also obtained by regiocontrolled rearrangement of isobenzofurans <11OL2086> and photochemical rearrangement of 3-(2-formylphenyl)acrylates <11CC11098>. A diastereomeric mixture of 3,4,4*a*,7,8,8*a*-hexahydroisocoumarins is produced by an intramolecular DA cycloaddition of ester-tethered 1,3,9-decatrienes under microwave irradiation. The same types of isocoumarin are formed by a microwave-assisted tandem Wittig–intramolecular DA cycloaddition of 3,5-hexadien-1-yl α -bromoacetates with glyoxal derivatives or formaldehyde, in the presence of PPh₃ and 2,6-lutidine <11T179>.



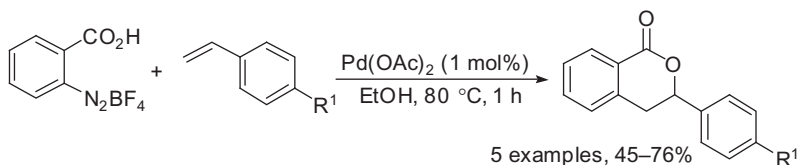
Scheme 86

(Thio)isocoumarins result from a one-pot decarboxylative cycloaddition of (thio)phthalic anhydride with allenes catalyzed by a nickel(0) catalyst (Scheme 87) <11OL1374>.



Scheme 87

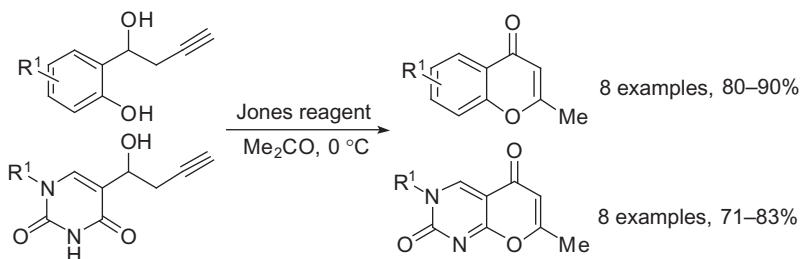
3,4-Dihydroisocoumarins are formed through a Heck–Matsuda reaction of an *o*-carboxybenzenediazonium salt with styrenes. Palladium acts as Heck arylation catalyst, and in some cases, there is an acid-catalyzed cyclization reaction (Scheme 88) <11TL6342>.



Scheme 88

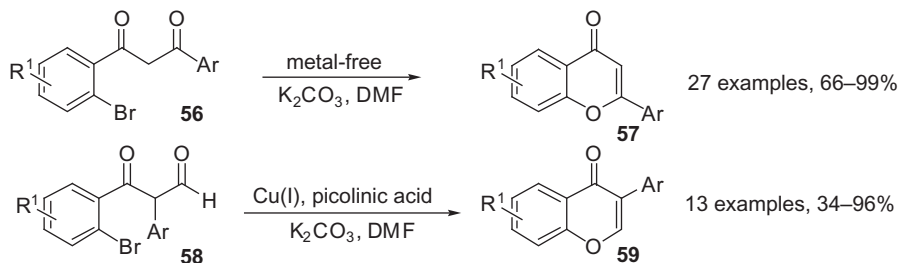
6.4.2.6 Chromones and Chromanones

Treating propargyl alcohols with Jones reagent provides a number of 2-methylchromone-type compounds, in good yields (Scheme 89). The mechanism may involve an oxidation to give propargyl ketones which isomerize to the corresponding 1,2-allenic ketones followed by an intramolecular cyclization process <11JOC982>.



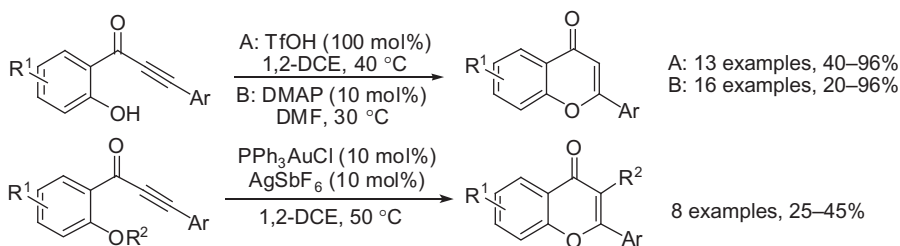
Scheme 89

The intramolecular *O*-arylation of appropriate substituted 1-(2-bromoaryl)propane-1,3-diones **56** under metal-free conditions provides a synthesis of 2-arylchromones **57** <11AGE3769>, and of 3-(2-bromoaryl)-3-oxopropanal derivatives **58** with a Cu(I) catalyst gives a range of 3-arylchromones **59** (Scheme 90) <11HCA1304>.



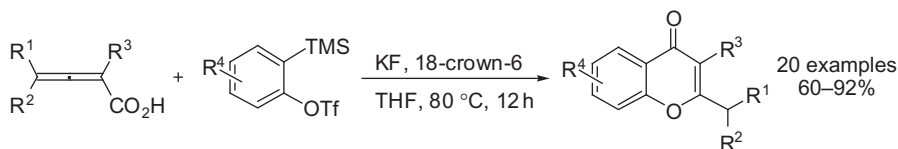
Scheme 90

2-arylchromones are obtained by regioselective cyclization of *o*-alkynoylphenols mediated by trifluoromethanesulfonic acid (TfOH) <11OL4526> or DMAP <11T9993> and by gold-catalyzed intramolecular cyclization of similar *O*-protected phenols, involving an electrophilic carbon-substituent transfer (Scheme 91) <11TL2476>. The preparation of the naturally occurring flavonoid luteolin involves a similar cyclization, catalyzed by cesium carbonate <11T4344>. The intramolecular oxa-Michael oxidation of chalcones mediated by CuI-ionic liquid <11OBC6930> or Yb(OTf)₃ <11JHC1356> affords a number of substituted 2-arylchromones in good yields.



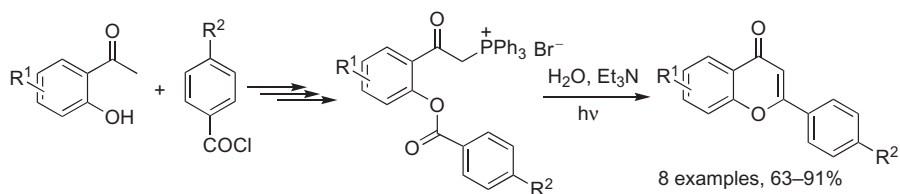
Scheme 91

Several polysubstituted chromones have been synthesized from the reaction of 2,3-allenoic acids with arynes generated *in situ*. Nucleophilic addition is followed by a rearrangement of the formed intermediate to give *o*-(1-oxo-2,3-allenyl)phenoxide, which after an oxa-Michael addition and hydrolysis gives chromones (Scheme 92) <11OL5196>. A few examples were also synthesized from an intramolecular heteroannulation of substituted 2'-hydroxy-2-nitroacetophenones with carbon disulfide in the presence of base <11TL254>.



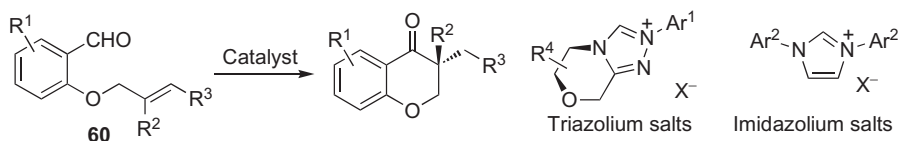
Scheme 92

A mixture of flavones (51–63%) and 3-arylflavones (23–11%) is obtained from the one-pot Baker-Venkataraman rearrangement of 2'-hydroxyacetophenones, 3 equiv. of aryl chlorides, and potassium carbonate in wet refluxing acetone <11TL3120>. An alternative multistep approach uses the same starting materials for the formation of the substituted phosphonium bromides followed by an intramolecular photochemical Wittig reaction in water (Scheme 93) <11TL7189>. A number of functionalized derivatives also result from the one-pot reaction of salicylaldehydes and arylacetylenes under dual catalysis of piperidine and FeCl₃, in good yields (74–87%) <11TL5610>.



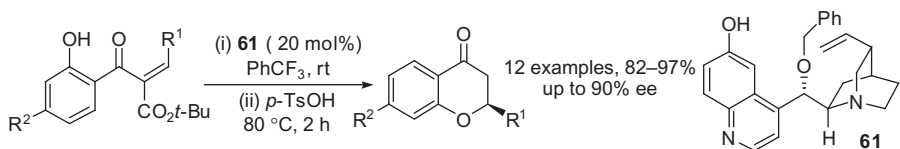
Scheme 93

Chiral triazolium salts have been used for an efficient asymmetric intramolecular Stetter reaction of oxygen substrates **60**, providing chroman-4-ones in excellent yields with up to 97% ee (Scheme 94) <11AGE4983, 11AGE7982, 11OBC2072, 11SL1033>. Studies on the mechanism of this reaction prove that the first step is an irreversible proton transfer process <11OL1742>. Similar compounds are obtained using imidazolium salts <11CEJ5965>.



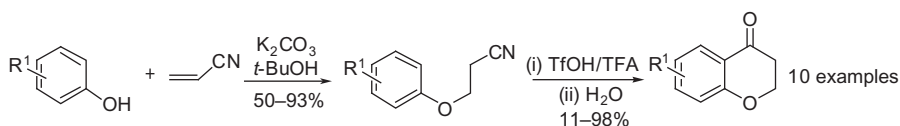
Scheme 94

Tandem intramolecular oxa-Michael addition of α -alkylidene β -ketoesters followed by decarboxylation in the presence of cinchona alkaloid **61** leads to further examples of chiral chroman-4-one derivatives (Scheme 95) <11T5389>.



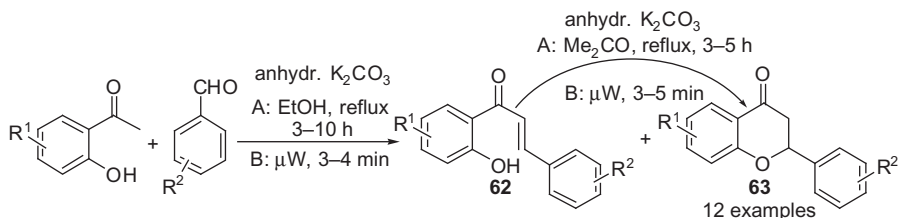
Scheme 95

3-Aryloxypropanenitriles are available by oxa-Michael addition of phenols to acrylonitriles, and their one-pot superacid-mediated Friedel-Crafts reaction affords the corresponding chroman-4-ones (Scheme 96) <11TL4824>.



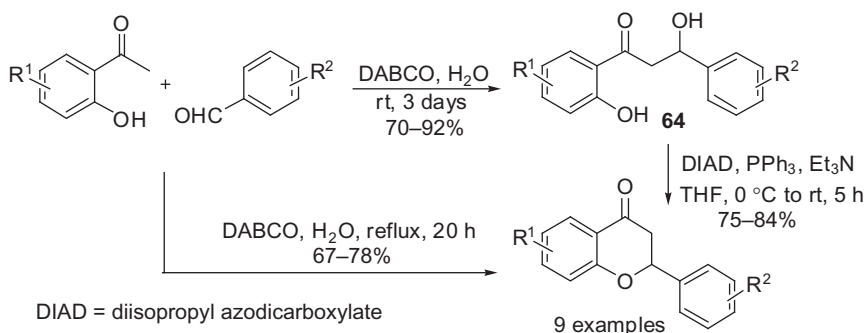
Scheme 96

Using anhydrous K_2CO_3 as catalyst, the aldol condensation of *o*-hydroxyacetophenones with benzaldehydes in ethanol affords a mixture of chalcones **62** and flavanones **63**. The chalcones **62** also cyclize to give the corresponding flavanones **63** (Scheme 97) <11TL5020>.



Scheme 97

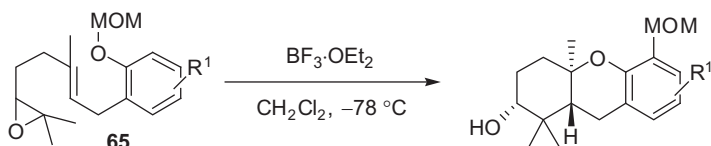
An alternative approach involves a DABCO-catalyzed Claisen–Schmidt reaction of *o*-hydroxyacetophenones with benzaldehydes to provide intermediates **64**, followed by an intramolecular dehydration with the modified Mitsunobu reaction to afford the expected flavanones. The one-pot version occurs at reflux temperature (Scheme 98) <11T4155>.



Scheme 98

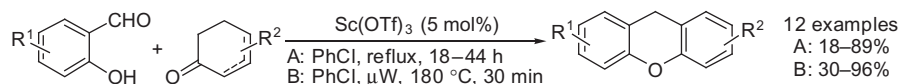
6.4.2.7 Xanthenes and Xanthones

Hexahydroxanthenes are readily attained by tandem MOM-based cascade cyclization–aromatic substitution when epoxides **65** are treated with $BF_3 \cdot OEt_2$ (Scheme 99) <11JOC909, 11TL1628> and by cyclization of 2-geranylphenol derivatives, catalyzed by a salt of a chiral phosphonous acid diester with FSO_3H <11OL3130>.



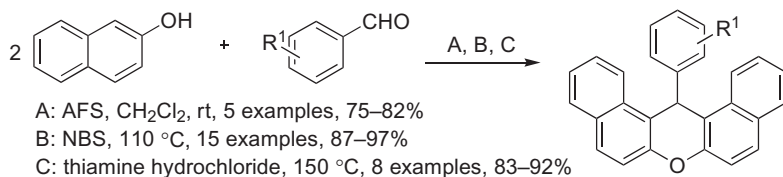
Scheme 99

A facile one-pot synthesis of substituted xanthenes involves the Lewis acid-promoted Knoevenagel condensation of salicylaldehydes and cyclohexenone derivatives followed by a sigmatropic hydrogen shift (Scheme 100) <11OBC1744>.



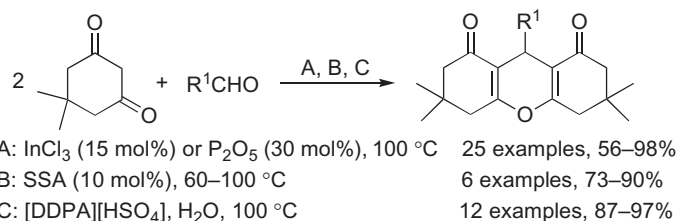
Scheme 100

A metal-free approach for the preparation of 14-aryldibenzoxanthenes was achieved by the condensation of 2-naphthol with aromatic aldehydes using a modified acid functionalized mesoporous silica (AFS) <11CC6677>, *N*-bromosuccinimide (NBS) <11HCA429> or thiamine hydrochloride <11SC3424> as catalysts (Scheme 101). A Ru(III)-catalyzed reaction of 2-naphthol with aliphatic aldehydes gave the same type of 14-substituted dibenzoxanthenes <11SC1427>.



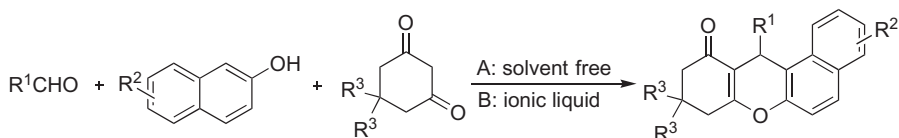
Scheme 101

A range of 9-substituted tetramethyloctahydroxanthene-1,8-diones were synthesized from dimedone and a variety of aliphatic and aromatic aldehydes using either InCl_3 and P_2O_5 <11T3698> or silica sulfuric acid (SSA) as catalysts <11SC347> or the ionic liquid 3-(*N,N*-dimethyldodecylammonium)propanesulfonic acid hydrogen sulfate ([DDPA][HSO_4]) (Scheme 102) <11JHC468>. Similar xanthene derivatives were also prepared in a solid-phase synthesis using resin-bound propiolic acid as Michael acceptor and cyclohexanediones <11S4027>.



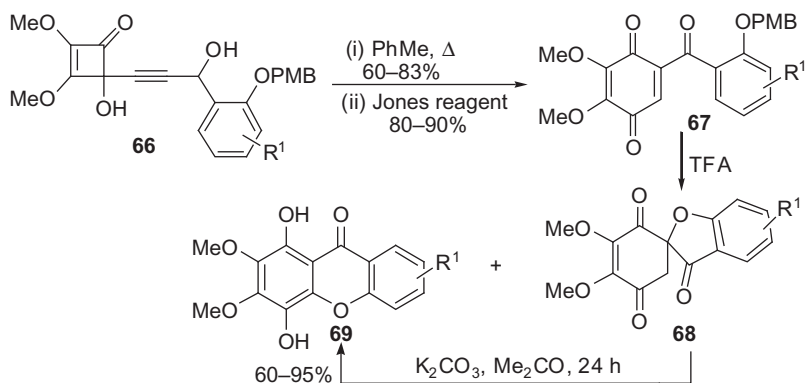
Scheme 102

Multicomponent reactions of aldehydes, 2-naphthols, and dione derivatives have been used for the synthesis of 12-aryltetrahydrobenzo[*a*]xanthen-11-ones catalyzed by Caro's acid/SiO₂ <11SC307>, Cu/SiO₂ <11SC2763>, and NH₄Cl <11SC2663> under solvent-free conditions, and even using *p*-toluenesulfonic acid as catalyst and an ionic liquid as solvent (Scheme 103) <11JHC1388>. Similar xanthen derivatives are obtained via a comparable three-component reaction involving aldehydes, 3,4-methylenedioxyphenol/2-naphthol, and 4-hydroxycoumarin/2-hydroxy-1,4-naphthoquinone <11JHC83, 11JHC1379>.



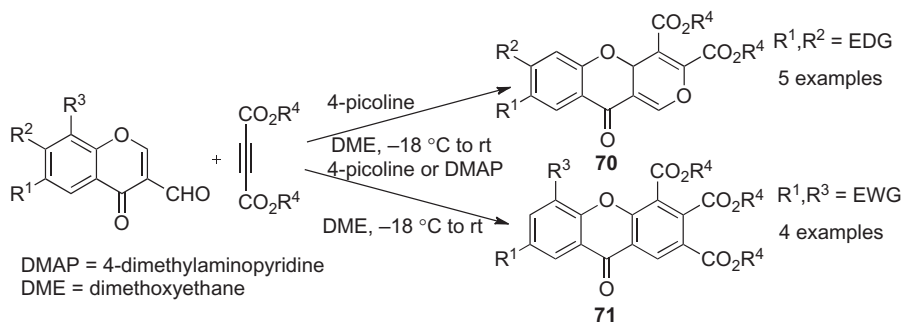
Scheme 103

1,4-Dioxygenated xanthenes **69** were prepared by a novel application of the Moore cyclization to a squarate derivative **66** leading to benzoylbenzoquinones **67** followed by oxidation and acid-catalyzed deprotection/cyclization. In some cases, the last step gave mixtures of the desired xanthenes **69** and spirocyclic ketones **68**; the latter could be converted into the former by treatment with potassium carbonate <Scheme 104> <11OL4696>.



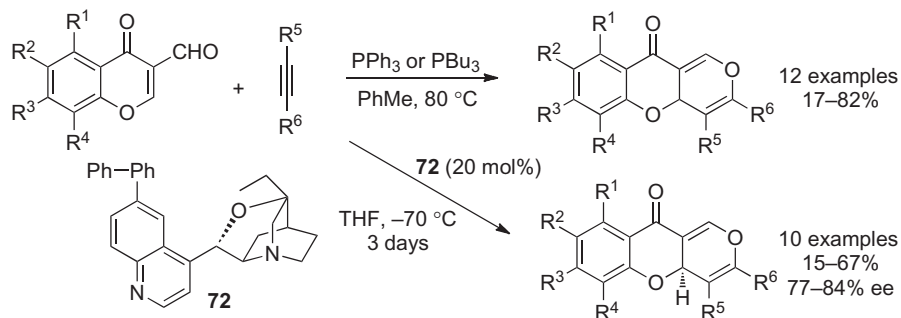
Scheme 104

The products obtained from the hDA reaction of substituted 3-formylchromones and acetylenedicarboxylates depend not only on the organocatalyst but also on the substrate. Electron-donating groups (EDG) in the presence of 4-picoline led to the formation of pyrano[4,3-*c*]chromene derivatives **70**, while electron-withdrawing groups (EWG) in 4-picoline or DMAP produced xanthenes **71** (Scheme 105) <11S97>.



Scheme 105

A Lewis base-catalyzed [4+2] annulation reaction of electron-deficient chromone-derived oxadienes and acetylene dicarboxylates afforded a series of xanthone-type compounds [<11CEJ5130, 11TL2265>](#). In the asymmetric version, modified cinchona alkaloid **72** was used as enantiodifferentiating Lewis base catalyst ([Scheme 106](#)) [<11CEJ5130>](#).



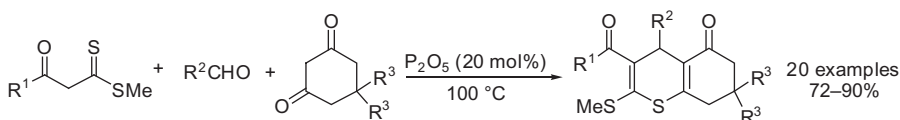
Scheme 106

The heterocyclic ring of tetrahydroxanthone and related structures are obtained by a Pd-catalyzed cyclization of 2-(bromoaryl)cyclohexanones [<11OL1056>](#). The parent xanthene and xanthone are obtained as by-products from the reaction of dimethyl(thio)formamide with *o*-trimethylsilylphenyl triflate, in the presence of CsF [<11BCJ328>](#).

6.4.3. HETEROCYCLES CONTAINING ONE SULFUR ATOM

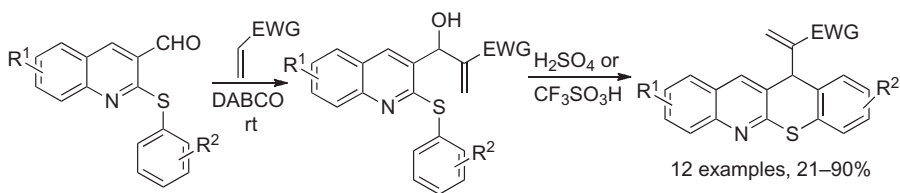
6.4.3.1 Thiopyrans and Analogues

A range of tetrahydrothiochromen-5-ones were obtained in a one-pot, three-component reaction of β -oxodithioesters, aldehydes, and cyclic 1,3-diketones, under solvent-free conditions ([Scheme 107](#)) [<11OL3762>](#).



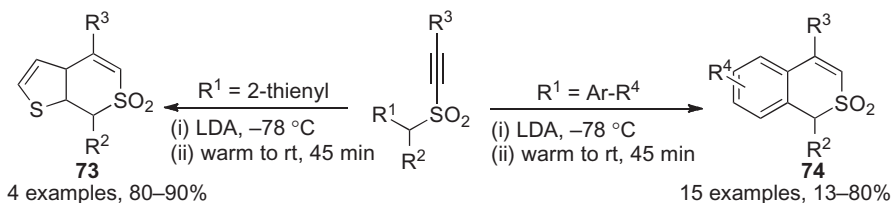
Scheme 107

An intramolecular Friedel–Crafts reaction is the key step for the preparation of an enantioenriched thiochroman-4-one from a sulfur–Michael addition of a thiol to a trifluorocrotonate <11OL4426>. The same key reaction was used in the synthesis of several thioxanthene-type compounds from Morita–Baylis–Hillman adducts, obtained by the reaction of 2-arylthioquinoline-3-carbaldehydes with activated alkenes (Scheme 108) <11T3509>, and from diarylmethyl carbinols, resulting from the addition of Grignard reagents to 4-(phenoxy or thiophenoxy)-2*H*-chromen-3-carbaldehydes <11TL5951>.



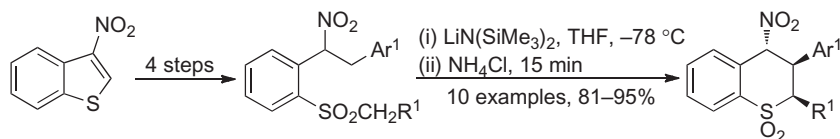
Scheme 108

LDA induces the cyclization of benzyl or 2-thienylmethyl hindered 1-propynylsulfones to afford 7*H*-thieno[2,3-*c*]thiopyran *S,S*-dioxides **73** and 1*H*-2-benzothiopyran *S,S*-dioxides **74**, respectively (Scheme 109) <11OL5330>.



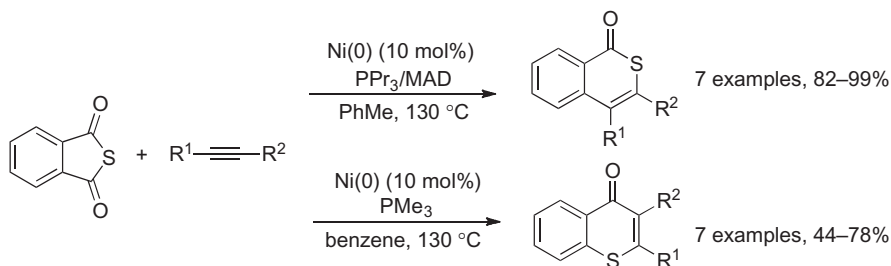
Scheme 109

The five-membered ring of 3-nitrobenzo[*b*]thiophene is enlarged to six-membered ring in a ring-opening–ring-closing procedure, where the cyclization conditions led to a diastereoselective synthesis of 4-nitrothiochroman *S,S*-dioxides (Scheme 110) <11T8160>.



Scheme 110

Heating a mixture of thiophthalic anhydride with substituted alkynes led to sulfur-containing compounds. Selective synthesis of thioisocoumarins is accomplished using Ni(0)/PPr₃ as catalyst in the presence of a Lewis acid, whereas Ni(0)/PMe₃ as catalyst afforded thiochromones (Scheme 111) <11OL1912>.

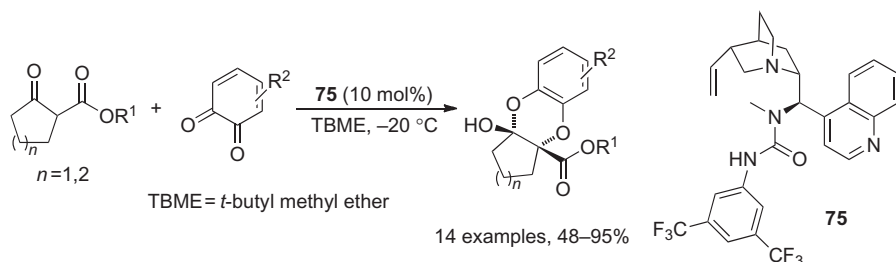


Scheme 111

6.4.4. HETEROCYCLES CONTAINING TWO OR MORE OXYGEN ATOMS

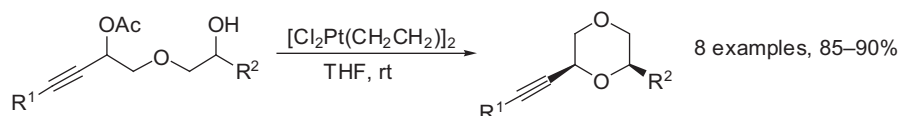
6.4.4.1 Dioxins and Dioxanes

Chiral tricyclic dioxins result from the aryloxylation of cyclic β-keto esters with a range of *o*-quinones in the presence of a cinchona alkaloid derivative **75** as catalyst (Scheme 112) <11S1880>. The manganese(III) catalytic aerobic oxidation of tetroinic acids (a type of cyclic β-keto esters) and 1,1-disubstituted alkenes affords fused 1,2-dioxins <11H(83)1783>.



Scheme 112

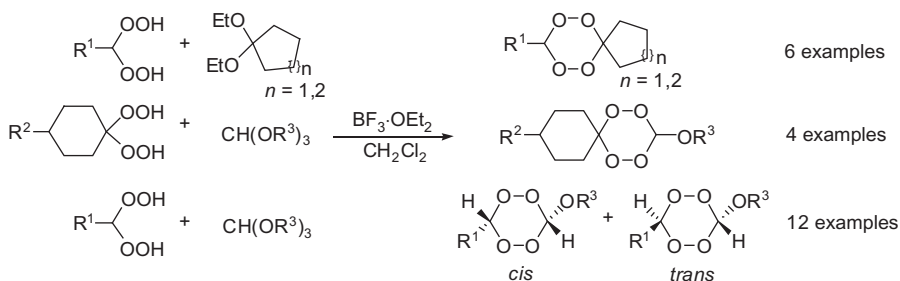
The intramolecular platinum-catalyzed cyclization of ω-hydroxy propargylic acetates in THF at room temperature provides *cis*-2,6-disubstituted 1,4-dioxanes in excellent yields (Scheme 113) <11T5046>. The reaction of enantiopure 1,2-diols and vinyl selenones as Michael acceptors followed by intramolecular displacement of the PhSeO₂ group also affords enantiopure 1,4-dioxanes <11CEJ993>.



Scheme 113

6.4.4.2 Tetraoxanes

Novel substituted 1,2,4,5-tetraoxanes were prepared through the reaction of *gem*-dihydroperoxides and cyclic acetals or orthoformates (Scheme 114) <11TL107>.

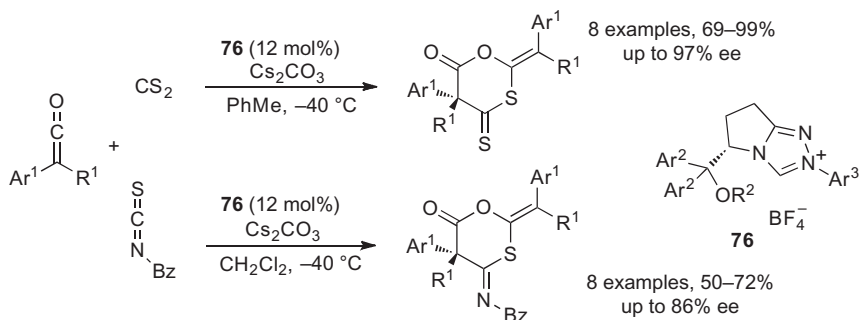


Scheme 114

6.4.5. HETEROCYCLES CONTAINING BOTH OXYGEN AND SULFUR IN THE SAME RING

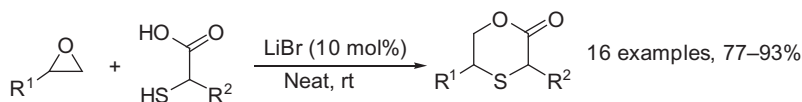
6.4.5.1 Oxathianes

NHC **76** catalyzes the [2+2+2] cycloaddition reaction at -40°C of ketenes with carbon disulfide <11CC8388> or isothiocyanates <11OL6382> to obtain the corresponding 1,3-oxathiane-6-ones in good yields and enantioselectivities (Scheme 115).

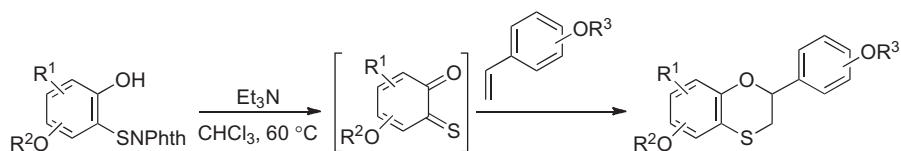


Scheme 115

Under solvent-free conditions, LiBr promotes a ring-opening–ring-closing cascade of terminal epoxides with α -mercaptocarboxylic acids for the synthesis of 1,4-oxathiane-2-ones (Scheme 116) <11TL3614>. 4-Thiaflavanes are prepared through an hDA reaction of *o*-thioquinones with styrenes (Scheme 117) <11CEJ12396, 11OBC1352>.



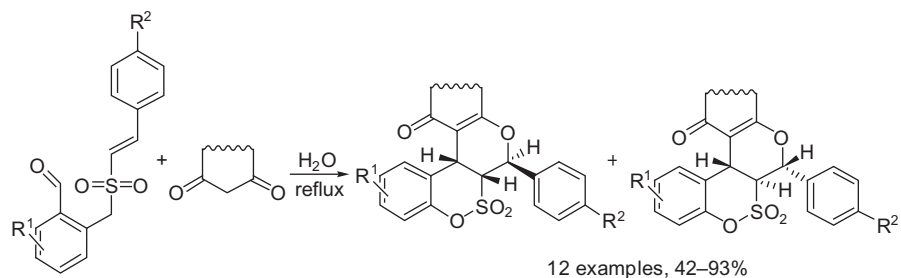
Scheme 116



Scheme 117

6.4.5.2 Sultones

Several functionalized benzo- δ -sultones were synthesized through a domino Knoevenagel–hDA reaction of substituted sulfonates and 1,3-diones in water (Scheme 118) <11T8484>.



Scheme 118

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