

有机化学

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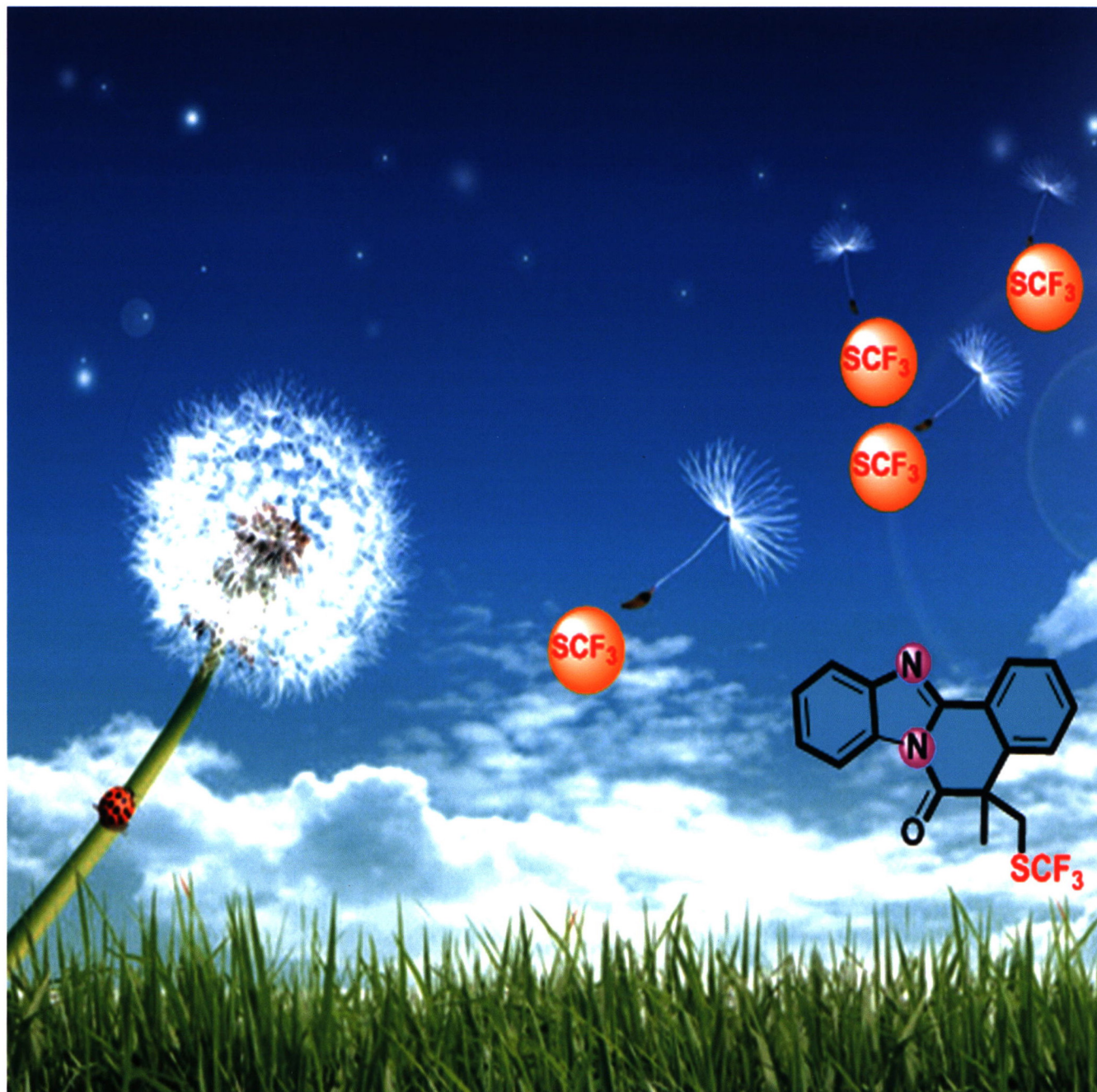
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有机化学 (月刊)

Chinese Journal of Organic Chemistry

(YOUJI HUAXUE)

第 42 卷 第 5 期 (总 402 期) 2022 年 5 月

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* 通讯联系人.

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研究简报

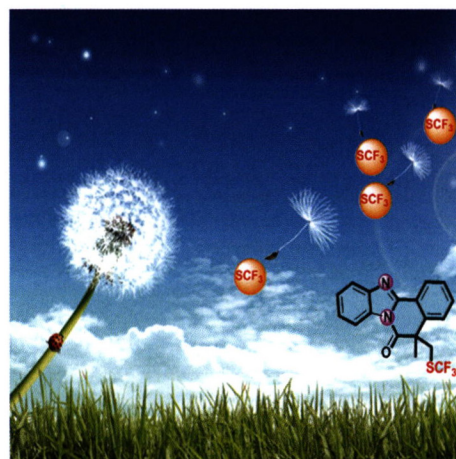
一步法合成 1,2,4-三氮唑[3,4- <i>i</i>]嘌呤类化合物	楚治良 陈晖娟 单 帅 王晓娜 高春芳 渠桂荣 刘忠于* 郭海明*	(1551)
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光镍协同催化的不对称还原交叉偶联反应	崔 坤 李公强* 夏纪宝*	(1567)
钯/Xu-Phos 催化对映选择性多米诺 Heck/远程 C(sp ²)-H 烷基化反应合成手性螺环化合物	王曼曼*	(1569)

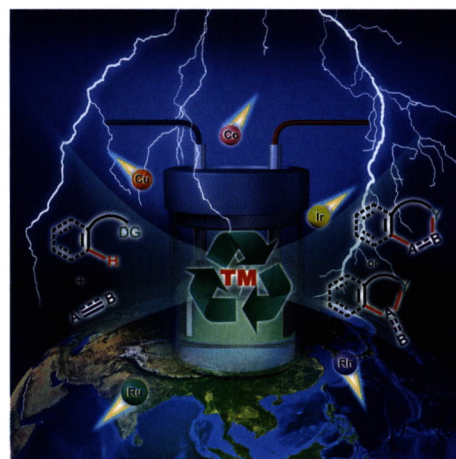
Cover Picture: Silver-Mediated Radical Trifluoromethylthiolation Cyclization: Access to F₃CS-Containing Benzimidazole[2,1-*a*]isoquinolines

Structurally diverse polycyclic benzimidazole[2,1-*a*]isoquinolines are important synthetic intermediates and key structural motifs in biologically active molecules. In this work, a AgSCF₃-mediated oxidative radical cascade cyclization is reported by Liu, Wang, Sun, Tang and Wang on page 1387, which features ease of handling, a wide substrate scope, good functional group compatibility, easily scaled-up operation, and facile derivatization.



Inside Cover: Electrooxidative Annulation of Unsaturated Molecules via Directed C—H Activation

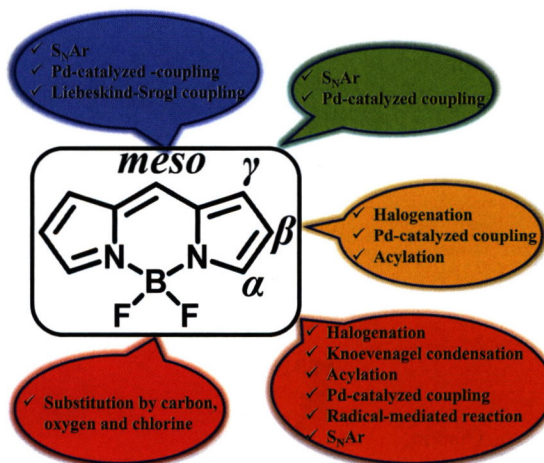
The recent progress of transition-metal catalyzed electrooxidative annulation of unsaturated molecules such as alkynes, olefins, carbon monoxide and isocyanogens via directed C—H activation is reviewed by Xie, Chen, Li, Lin, Chen and Shi on page 1286. The characteristics and mechanism of these reactions are systematically summarized.



REVIEWS

Progress in the Synthesis of Boron Dipyrromethene (BODIPY) Fluorescent Dyes

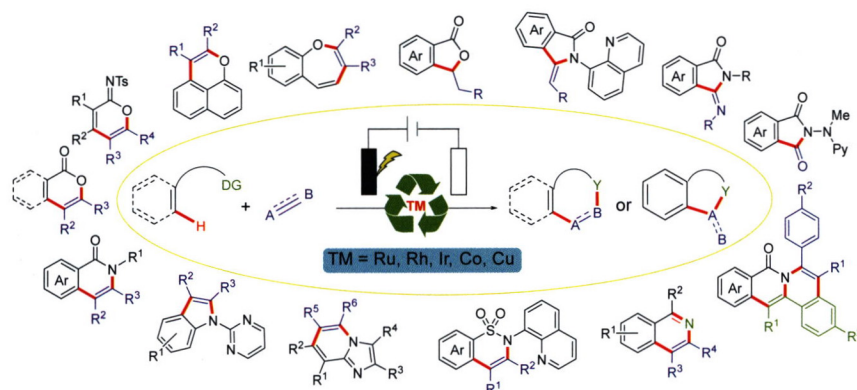
Liu, Bin-Kai; Teng, Kun-Xu; Niu, Li-Ya*; Yang, Qing-Zheng*
Chin. J. Org. Chem. **2022**, 42(5), 1265



The modification strategies of boron dipyrromethene (BODIPY) are summarized, including α , β , γ , *meso*-positions and boron atom. Then Pd-catalyzed cross-coupling reactions and oxidative coupling reactions are discussed individually for their importance.

CONTENT

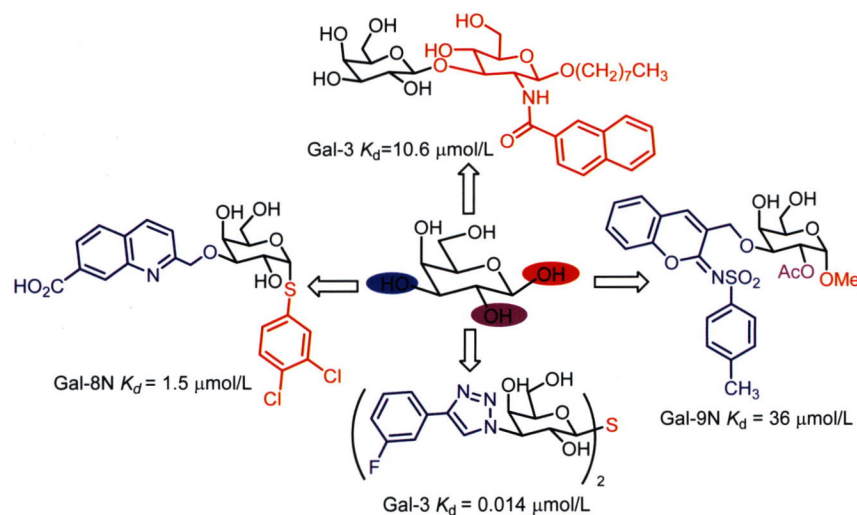
Electrooxidative Annulation of Unsaturated Molecules via Directed C—H Activation



The recent progress of transition-metal catalyzed electrooxidative annulation of unsaturated molecules such as alkynes, olefins, carbon monoxide and isocyanogens via directed C—H activation is reviewed. The reaction conditions and mechanism reaction mechanisms of these transformations are discussed. Finally, the challenges and the future development on the area are also prospected.

Xie, Wucheng*; Chen, Xu; Li, Yunyue; Lin, Jiuling; Chen, Wanwen; Shi, Junjun*
Chin. J. Org. Chem. **2022**, 42(5), 1286

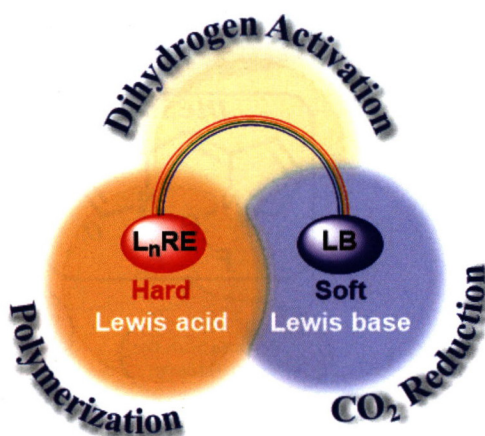
Research Progress on the Synthesis and Activity of *D*-Galactose Derived Small Galectin Inhibitors



The synthesis and biological activities of small galectin inhibitors derived from *D*-galactose is reviewed according to the difference of derivatization sites, which is expected to provide research thought for designing galectin inhibitors with high affinity and high selectivity, and afford reference for development of new drug candidates targeting galectins.

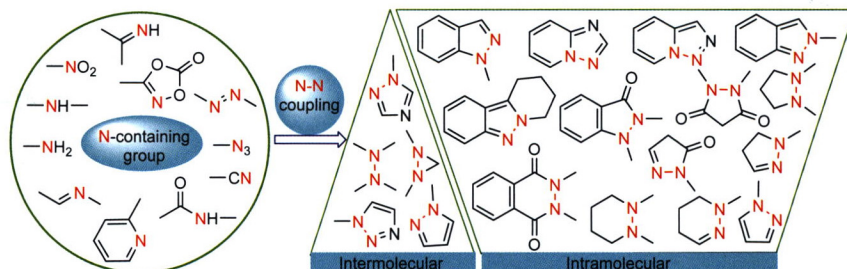
Yong, Can; Li, Yun; Bi, Tao; Chen, Guofeng; Zheng, Dongxia; Wang, Zhouyu*; Zhang, Yuanyuan*
Chin. J. Org. Chem. **2022**, 42(5), 1307

Progress in Rare-Earth Metal-Based Lewis Pair Chemistry



The syntheses of diverse rare-earth metal-based Lewis pairs and their application in activation of small molecules and catalysis, including polymerization of polar alkenes and hydrosilylative reduction of carbon dioxide, are reviewed. At the same time, new prospects for the future development of rare-earth metal based Lewis pairs are put forward.

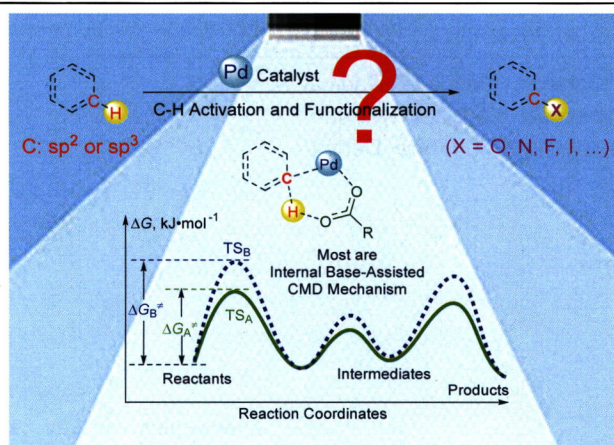
Guan, Yiwen; Chang, Kejian; Sun, Qianlin; Xu, Xin*
Chin. J. Org. Chem. **2022**, 42(5), 1326

Advances on the Synthesis of N—N
Bonds

Zhao, Weizhe; Xu, Jiali; Yang, Fan; Zeng, Xianghua*

Chin. J. Org. Chem. **2022**, 42(5), 1336

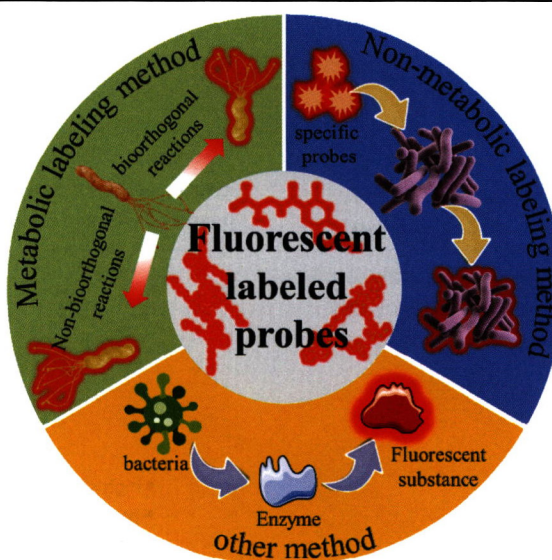
The intermolecular and intramolecular formation of N—N bond in recent years is summarized. Additionally, the difficulties and future development of this strategy are prospected.

Research Progress on Density Functional
Theory Study of Palladium-Catalyzed
C—H Functionalization to Form C—X
(X=O, N, F, I, ...) Bonds

Shi, Yubing; Bai, Wenji; Mu, Weihua*; Li, Jiangping; Yu, Jiawei; Lian, Bing

Chin. J. Org. Chem. **2022**, 42(5), 1346

The latest density functional theory research results on palladium-catalyzed C—H functionalization in constructing C—X (X=O, N, F, I, ...) bonds are summarized, with emphasis on the corresponding computational results about microcosmic reaction mechanism and selectivity controlling. The present issues and prospects of future development in this field are also summarized and forecasted in the end.

Recent Progress on Strategies and Ap-
plications of Imaging for Intestinal Micro-
flora

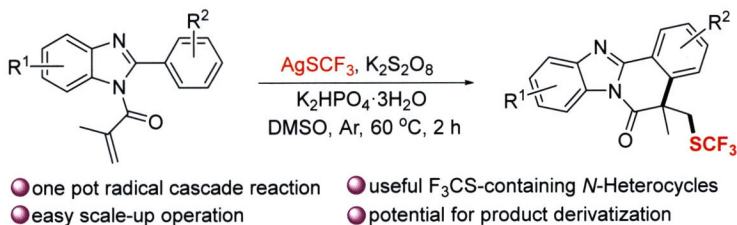
Li, Na; Tan, Xiaofeng*; Yang, Qinglai*

Chin. J. Org. Chem. **2022**, 42(5), 1375

The microflora in the mammalian gut plays an essential role in maintaining the physiological states and pathological changes of the host, and it is of significance for host health to detect the microflora in the gut. The fluorescent-labeled probes based on the metabolic/non-metabolic labelling and specific metabolite labeling methods, with the advantages of non-invasive, less tissue damage, higher specificity, and sensitivity, have shown greater potential in the intestinal microflora detection.

ARTICLES

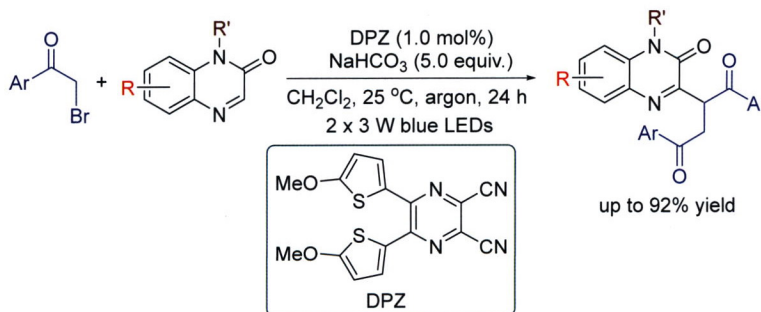
Silver-Mediated Radical Trifluoromethylthiolation Cyclization: Synthesis of CF₃S-Containing Benzimidazole[2,1-*a*]isoquinolines



A practical Ag-catalyzed trifluoromethylthiolation cyclization reaction was developed. Various structurally diverse CF₃S-containing benzimidazo[2,1-*a*]isoquinolines were obtained for the first time in moderate to good yields. Mechanistic studies suggested that the catalytic reaction proceeds via a SCF₃-radical-triggered cascade cyclization pathway.

Liu, Bing*; Wang, Zhichuang; Sun, Kai; Tang, Shi; Wang, Xin*
Chin. J. Org. Chem. **2022**, 42(5), 1387

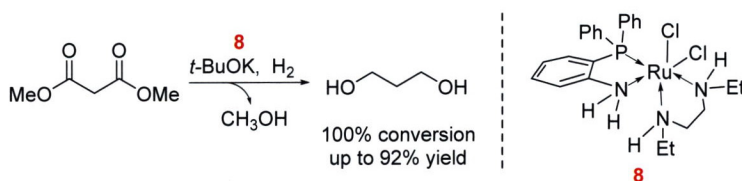
Photoredox Catalytic Cascade Radical Addition to Construct 1,4-Diketone-Functionalized Quinoxalin-2(1*H*)-one Derivatives



A new photocatalytic cascade reaction strategy towards the synthesis quinoxalin-2(1*H*)-ones has been developed. The method provides an efficient approach to access valuable 1,4-diketone-functionalized quinoxalin-2(1*H*)-one derivatives, wherein 2-bromo-1-arylethan-1-ones are the reaction partners of quinoxalin-2(1*H*)-ones.

Sun, Xin; Qu, Chaofan; Ma, Chaorui; Zhao, Xiaowei; Chai, Guobi*; Jiang, Zhiyong*
Chin. J. Org. Chem. **2022**, 42(5), 1396

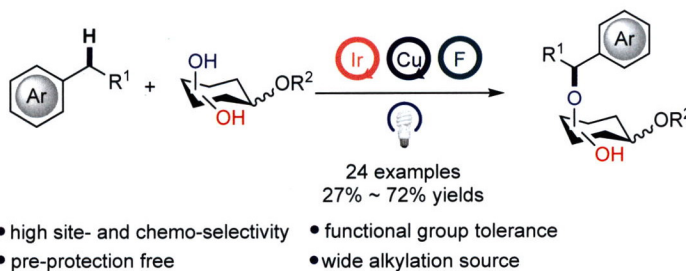
Homogeneous Catalytic Hydrogenation of Dimethyl Malonate into 1,3-Propanediol



A series of *o*-PPh₂C₆H₄NH₂-Ru(II) complexes were successfully applied in the catalytic hydrogenation of dimethyl malonate into 1,3-propanediol, in which the secondary amino ligand-constituted complex **8** performed the highest efficiency.

Fang, Xiaolong*; Li, Bin; Jin, Jie; Duan, Ning
Chin. J. Org. Chem. **2022**, 42(5), 1407

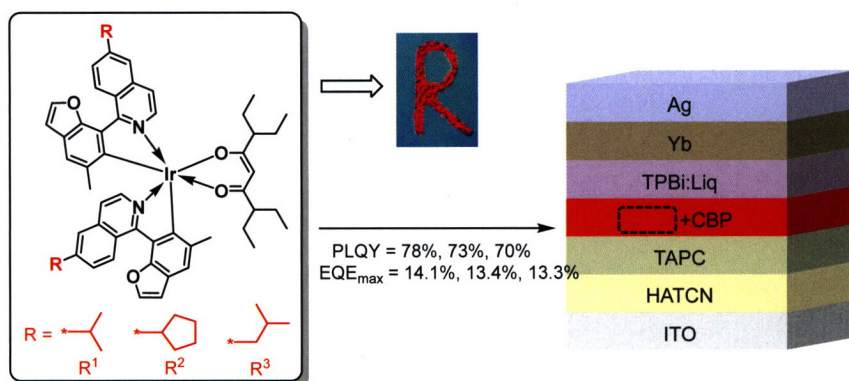
Photoredox-Copper Dual-Catalyzed Site-Selective *O*-Alkylation of Glycosides



A photoredox-copper dual-catalyzed cross dehydrogenative coupling reaction of glycosides with benzylic C—H substrates has been developed. The reaction proceeds smoothly under mild reaction conditions and features the using of readily accessible starting materials, which allows the highly site-selective synthesis of diverse glycosides *O*-alkylation products in 27%~72% yields, providing a new synthetic tool for the site-selective modification of glycosides.

Sun, Tianyi; Zhang, Yifan; Meng, Yuanjie; Wang, Yi; Zhu, Qifeng*; Jiang, Yuxin*; Liu, Shihui*
Chin. J. Org. Chem. **2022**, 42(5), 1414

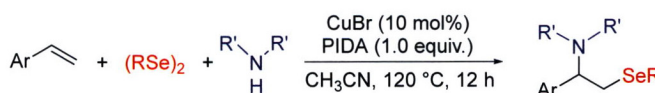
Synthesis and Electroluminescent Properties of Red-Emitting Iridium Complexes Based on Benzofuran-Isoquinoline



Nie, Fei; Huang, Guanbo; Dai, Lei; Chen, Shaofu; Ji, Shaomin; Chen, Jiaxiong*; Huo, Yanping*

Chin. J. Org. Chem. **2022**, 42(5), 1423

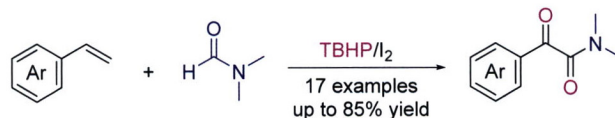
Three red phosphorescent iridium complexes were designed and synthesized, and the organic light-emitting devices prepared by using them as light-emitting materials showed good electroluminescent properties.

Radical Aminoselenation of Styrenes: Facile Access to β -Amido-selenides

Yin, Yifan; Li, Chen; Sun, Kai*; Liu, Yingjie; Wang, Xin*

Chin. J. Org. Chem. **2022**, 42(5), 1431

An efficient protocol for the intermolecular amidoselenation of alkenes with diphenyl diselenides and alkyl amines that affords a series of β -amido-selenides in high yields has been developed.

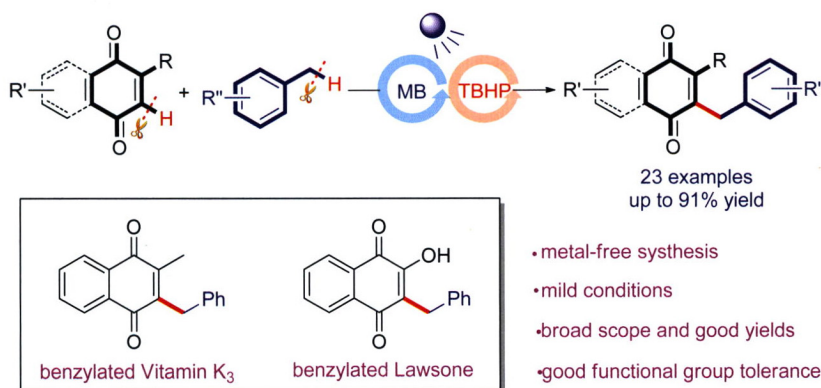
*I*₂/*t*-Butylhydroperoxide (TBHP)-Mediated Oxo-amidation of Alkenes with *N,N*-Dimethylformamide: A Facile Access to Aryl- α -ketoamide Derivatives

Xiao, Duoduo; Liu, Hailing; Zhou, Peng; Zhang, Jiantao*; Liu, Weibing*

Chin. J. Org. Chem. **2022**, 42(5), 1438

Difunctionalization of alkenes providing for the synthesis of aryl- α -ketoamide derivatives is developed by using *N,N*-dimethylformamide (DMF) as the solvent and the source of dimethylamine. This procedure is catalyzed by I₂ and *t*-butylhydroperoxide (TBHP) as oxidant. A wide range of aryl- α -ketoamide derivatives are generated in good yields.

Visible Light-Induced Metal-Free Benzoylation of Quinones via Cross Dehydrogenation Coupling Reaction



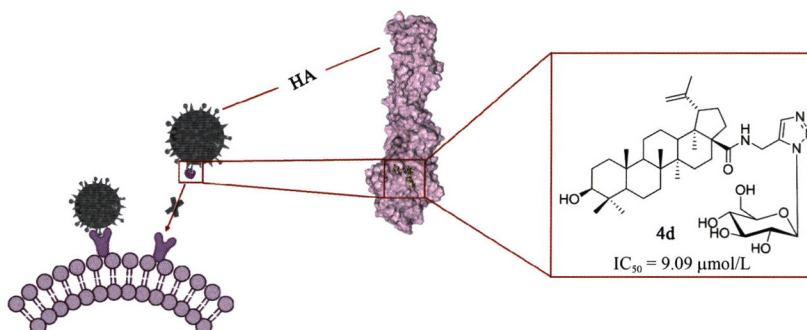
Wang, Xinyao; Zhang, Qingqing; Liu, Shuyang; Li, Min*; Li, Haifang*; Duan, Chunying; Jin, Yunhe*

Chin. J. Org. Chem. **2022**, 42(5), 1443

The development of visible light-induced metal-free benzoylation of quinones via cross dehydrogenation coupling reaction was reported. The method exhibits many advantages, including mild conditions, a broad scope with good functional group tolerance, low cost, and avoidance of metal remaining in products. This method may bring novel inspiration and approach for the synthesis of bioactive quinones.

CONTENT

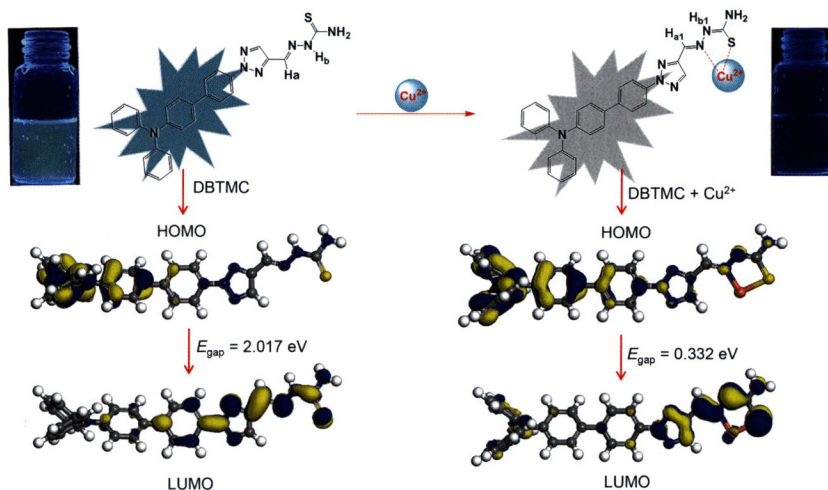
Design, Synthesis of Pentacyclic Triterpenoid Glucose Conjugate and *in vitro* Activity against Influenza Virus



Cai, Ming; Shao, Liang; Yang, Fan; Zhang, Jihong; Yu, Fei*
Chin. J. Org. Chem. **2022**, 42(5), 1453

Compound **4d** can inhibit the interaction of hemagglutinin (HA) and sialic acid receptor from the source, preventing influenza virus from infecting host cells.

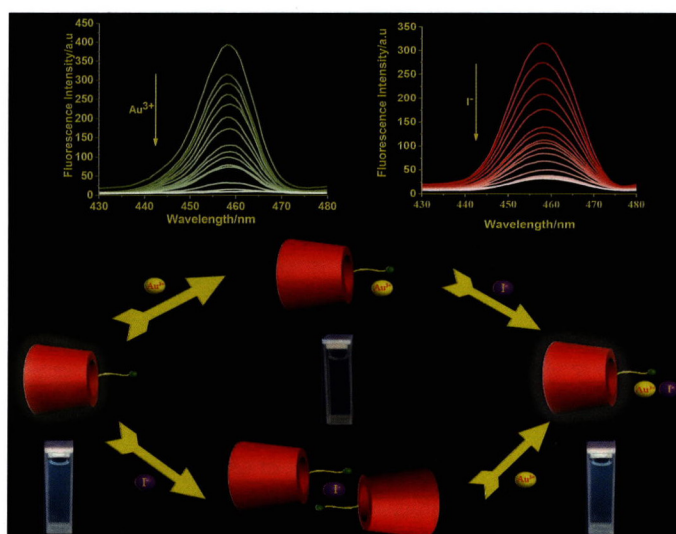
Synthesis of Triazole Functionalized Triphenylamine Cu^{2+} Fluorescent Probe and Its Application in Detection and HeLa Cells



Wen, Yiping; Xie, Zhengfeng*; Shi, Tianzhu; Chu, Yicheng; Zhou, Ronggui; Tao, Yishan; Liang, Huanmin; Qiu, Haiyan; Zhao, Yunhui*
Chin. J. Org. Chem. **2022**, 42(5), 1463

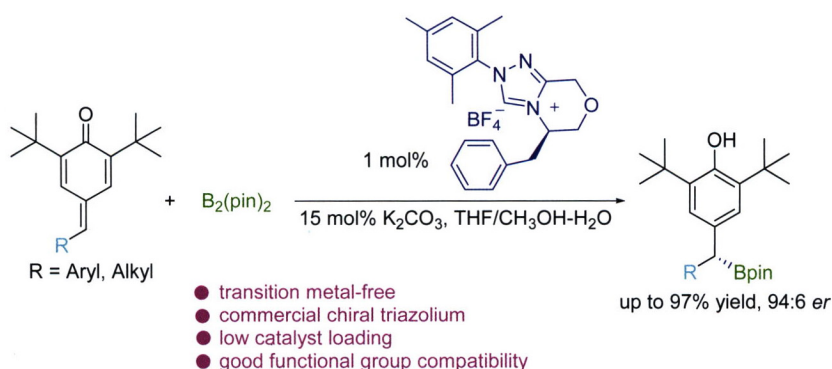
A novel fluorescence probe $N'-((2-(4'-(\text{diphenyl})-[1,1'-\text{biphenyl}]-4\text{-yl})-2H-1,2,3\text{-triazole-4-yl)methylene)hydrazine-1-thioamide}$ (DBTMC) was designed and synthesized from (4-(diphenylamino)-phenyl)boronic acid, (4-bromophenyl)-2H-[1,2,3]-triazole-4-carbaldehyde and thiosemicarbazide.

Mono-(6-diethylenetriamine-6-deoxy)- β -cyclodextrin Supramolecular Fluorescent Switch Constructed Based on Au^{3+} and I^-



Lu, Jiajia; Yang, Junli; Gu, Jie; Yang, Ju; Gao, Zhenjie; Su, Lijiao; Tao, Xin; Yuan, Mingwei*; Yang, Lijuan*
Chin. J. Org. Chem. **2022**, 42(5), 1474

Mono-(6-diethylenetriamine-6-deoxy)- β -cyclodextrin (3N- β -CD) was synthesized by green and simple method. A supramolecular fluorescent probe based on 3N- β -CD was constructed using Au^{3+} and I^- as fluorescence switches.

Asymmetric Boration of *para*-Quinone
Methides Catalyzed by *N*-Heterocyclic
Carbene

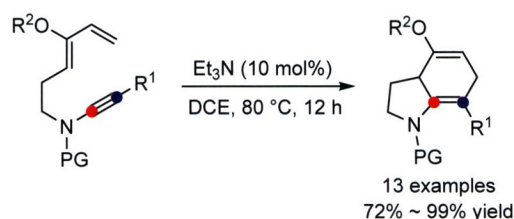
Wu, Yuzhu; Shen, Panpan; Duan, Wenzeng;
Ma, Yudao*
Chin. J. Org. Chem. **2022**, 42(5), 1483

Organocatalytic enantioselective boration of *para*-quinone methides was achieved by using a commercial chiral triazolium as the catalyst.

Visible Light-Induced Hydroxyalkylation
of Heteroarenes with Aliphatic Alcohols

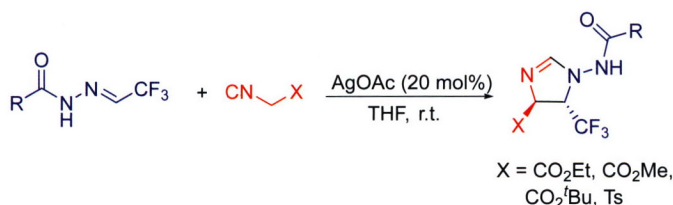
Xu, Dongping; Huang, Fei; Tang, Lin;
Zhang, Xinming; Zhang, Wu*
Chin. J. Org. Chem. **2022**, 42(5), 1493

An efficient visible light-induced direct cross-dehydrogenative coupling of heteroarenes with aliphatic alcohols in aqueous solution at ambient temperature was developed. This protocol was highlighted by photocatalyst-free, green solvent, mild conditions, readily available starting materials and wide functional group tolerance.

Synthesis of Tetrahydroindole Derivatives
via Metal-Free Intramolecular [4+2] An-
nulation of Ynamides

Zhang, Zhixin; Zhai, Tongyi; Zhu, Bohan;
Qian, Pengcheng*; Ye, Longwu*
Chin. J. Org. Chem. **2022**, 42(5), 1501

A metal-free intramolecular [4+2] annulation of 4-siloxy-3,5-diene ynamides has been developed. Under mild reaction conditions and without metal catalyst, various 2,3,3a,6-tetrahydroindoles were obtained in good to excellent yields from readily available 4-siloxy-3,5-diene ynamides, thus providing an efficient and convenient protocol for the preparation of synthetically useful tetrahydroindole motif.

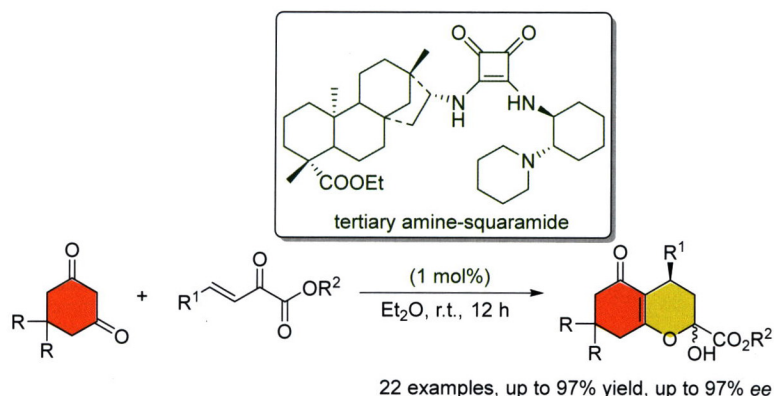
Silver-Catalyzed Synthesis of CF₃-Sub-
stituted 2-Imidazolines

Yang, Ming; Huang, Danfeng*; Wang, Kehu;
Han, Tongyu; Zhao, Pengfei; Wang, Feng;
Wang, Junjiao; Su, Yingpeng; Hu, Yulai*
Chin. J. Org. Chem. **2022**, 42(5), 1509

The silver salt catalyzed [3+2] cycloaddition reaction of trifluoromethylated *N*-acylhydrazones and ethyl isocyanoacetate was investigated. The reaction proceeds quickly to produce a series of trifluoromethylated 2-imidazoline compounds in high yields with excellent stereoselectivity, which provides a novel and efficient method for the synthesis of trifluoromethylated 2-imidazoline compounds.

CONTENT

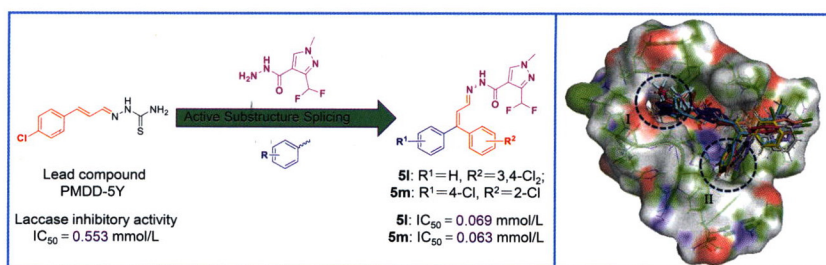
Chiral Squaramide Catalyzed Enantioselective Michael Addition of Cyclic 1,3-Diketones to β,γ -Unsaturated α -Keto Esters



Ma, Zhiwei*; Chen, Xiaopei; Wang, Chuan-chuan; Wang, Jianling; Tao, Jingchao; Lü, Quanjian*
Chin. J. Org. Chem. **2022**, 42(5), 1520

In the presence of a newly designed bifunctional tertiary amine-squaramide organocatalyst, the Michael addition between cyclic 1,3-diketones and β,γ -unsaturated α -ketoesters occurred smoothly to provide the desired products with high to excellent yields and enantioselectivities.

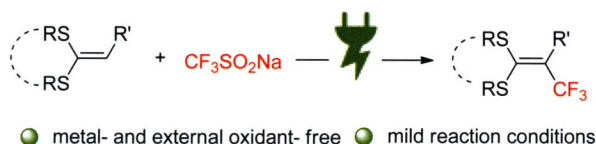
Design, Synthesis and Bioactivity of Novel Fluoropyrazole Hydrazides



Wang, Changkai; Sun, Tengda; Zhang, Xuebo; Yang, Xinling; Lu, Xingxing; Xu, Huan; Shi, Fasheng; Zhang, Li; Ling, Yun*
Chin. J. Org. Chem. **2022**, 42(5), 1527

15 new fluoropyrazole hydrazides were designed and synthesized and their bioactivities were investigated. The results of laccase inhibitory activity test showed that all the prepared compounds have good activity, and compounds **5l** and **5m** had the half-maximal inhibiting concentration (IC_{50}) values of 0.069 and 0.063 mmol/L respectively, which were significantly better than that of lead compound PMDD-5Y ($IC_{50} = 0.553 \text{ mmol/L}$) and positive control cysteine ($IC_{50} = 0.298 \text{ mmol/L}$).

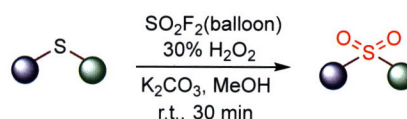
Electrochemical Oxidative Trifluoromethylation of α -Oxoketene Ketene Dithioacetals with CF_3SO_2Na



Gu, Qingyun; Cheng, Zhenfeng; Zeng, Xiaobao*
Chin. J. Org. Chem. **2022**, 42(5), 1537

The electrochemical trifluoromethylation of α -oxoketene ketene dithioacetals with CF_3SO_2Na in an undivided cell under catalyst- and exogenous redox reagent-free conditions with cheap electrode material was developed, leading to a variety of α -trifluoromethylated α -oxoketene ketene dithioacetals in moderate to good yields.

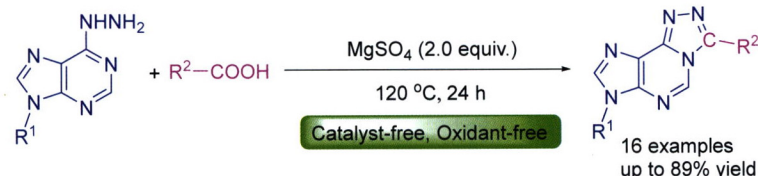
Oxidation of Sulfides with SO_2F_2/H_2O_2 /Base



Zhou, Yi; Li, Zhuojun; Hu, Minghui; Yan, Zhaohua*; Lin, Sen*
Chin. J. Org. Chem. **2022**, 42(5), 1545

A novel oxidation system SO_2F_2/H_2O_2 /base was investigated for the oxidation of sulfides and the corresponding sulfones were smoothly generated in good to excellent yields. The effects of base and solvent on oxidation were studied. The results showed that this oxidation system is tolerable to a variety of functional groups. A distinct advantage of this method is that by-products resulting from the use of the oxidation system are all water-soluble inorganic compounds, and can be conveniently removed after work-up process, therefore purification of the products was greatly simplified.

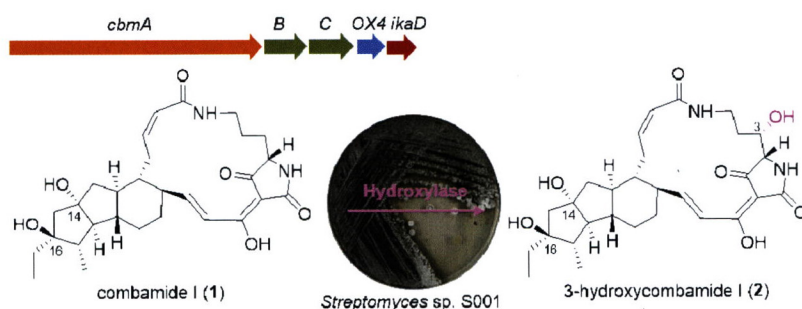
NOTES

One-Step Synthesis of 1,2,4-Triazolo[3,4-*i*]purine Derivatives

Chu, Zhiliang; Chen, Huijuan; Shan, Shuai;
Wang, Xiaona; Gao, Chunfang; Qu, Guirong;
Liu, Zhongyu*; Guo, Haiming*
Chin. J. Org. Chem. **2022**, 42(5), 1551

A new one-step method for the preparation of 1,2,4-triazolo[3,4-*i*]purine derivatives via 6-hydrazinyl-*N*⁹-substituted purine and aliphatic monocarboxylic acid was developed. Seventeen novel 1,2,4-triazolo[3,4-*i*]purine derivatives were synthesized in good to excellent yields. The method is simple to operate and does not need any catalyst or oxidant reagent

Discovery of a New Polycyclic Tetramate Macrolactam 3-Hydroxycombamide I

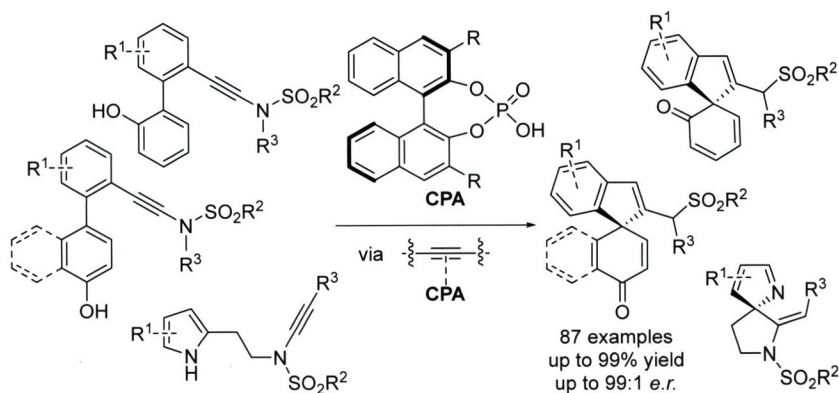


Yan, Yaqian; Wang, Haoxin; Li, Yaoyao*
Chin. J. Org. Chem. **2022**, 42(5), 1557

A new polycyclic tetramate macrolactam 3-hydroxycombamide I (2) was identified from the recombinant strain S001-*cbm-OX4-ikaD*. The hydroxylation of 2 at C-3 was proposed to be catalyzed by a hydroxylase of *Streptomyces* sp. S001.

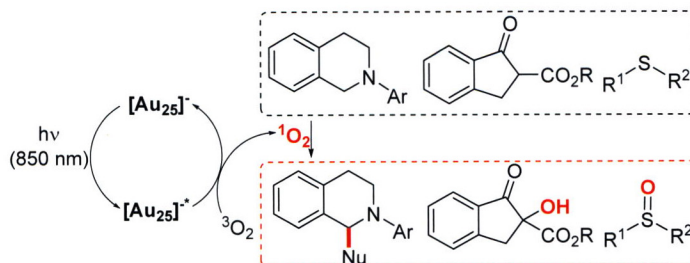
HIGHLIGHTS

Catalytic Asymmetric Dearomatization (CADA) through Activation of Ynamide by Chiral Brønsted Acids



Zhang, Wenzhao; Luo, Sanzhong*
Chin. J. Org. Chem. **2022**, 42(5), 1562

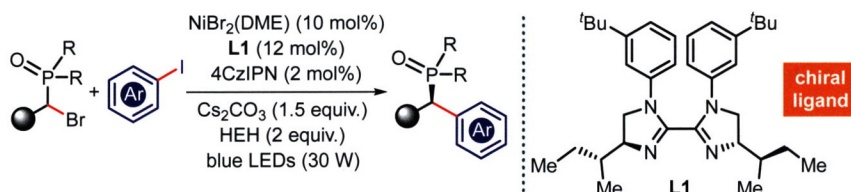
Near-Infrared Photocatalytic Oxidation Functionalization Mediated by Gold Nanoclusters



Yi, Rongnan; He, Weimin*
Chin. J. Org. Chem. **2022**, 42(5), 1565

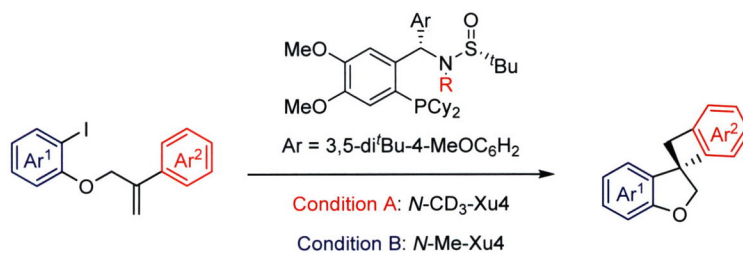
CONTENT

Photoredox Nickel-Catalyzed Asymmetric Reductive Cross Coupling



Cui, Kun; Li, Gongqiang; Xia, Ji-Bao*
Chin. J. Org. Chem. **2022**, 42(5), 1567

Synthesis of Chiral Spirocycles by the Enantioselective Domino Heck/Remote C(sp²)—H Alkylation Reaction Catalyzed by Palladium/Xu-Phos



Wang, Manman*
Chin. J. Org. Chem. **2022**, 42(5), 1569



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