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学术动态

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(YOUJI HUAXUE)

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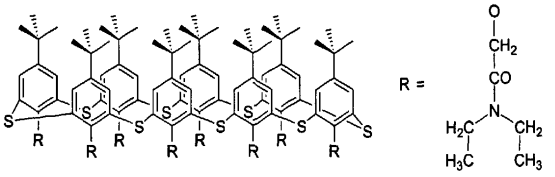
CONTENTS

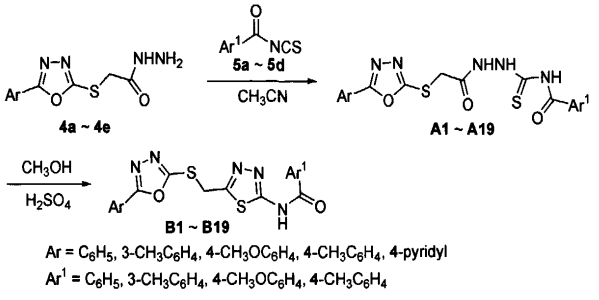
Research on Synthesis of Thiacalix[8]-arene Derivative and Its Selective Recognition of Metal Ion



A new derivative matter amidothiacalix[8]arene [TC8A-$CH_2CON(CH_2CH_3)_2$] has been synthesized by modified the lower rims of *p-tert*-butylthiacalix[8]arene (TC8A) in acetone by using *p-tert*-butylthiacalix[8]arene and *α*-chloro-*N*,*N*-diethyl-acetamide as substrate, and cesium carbonate as catalyst. The metal extraction properties of TC8A and amidothiacalix[8]arene from automotive catalyst residue solution containing platinum-group metal (PGM) cations have been investigated.

Li, Chunbin; Yue, Yulian; Liu, Baoquan; Fan, Shengdi*
Chin. J. Org. Chem. **2011**, *31*(6), 819

Synthesis of Sulfur Ethers Containing 1,3,4-Oxadiazole and 1,3,4-Thiadiazole



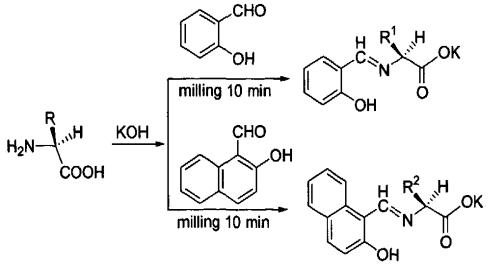
Ar = C_6H_5, 3-$CH_3C_6H_4$, 4-$CH_3OC_6H_4$, 4-$CH_3C_6H_4$, 4-pyridyl

Ar^1 = C_6H_5, 3-$CH_3C_6H_4$, 4-$CH_3OC_6H_4$, 4-$CH_3C_6H_4$

The biacyl thiosemicarbazide derivatives **A1**～**A19** were synthesized by the reaction of corresponding compounds **4a**～**4e** with aroyl isothiocyanates **5a**～**5d**. The target compounds **B1**～**B19** were prepared by cyclization of compounds **A1**～**A19** in the presence of CH_3OH/H_2SO_4.

Wu, Xianli; Zhu, Chunfeng; Lü, Zhidan; Wei, Chengshi; Liao, Xincheng*
Chin. J. Org. Chem. **2011**, *31*(6), 824

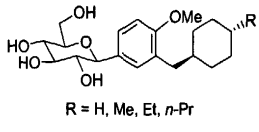
Solvent-Free Synthesis and Characterization of *L*-Amino Acids Schiff Bases



A new solvent-free method was described for the synthesis of *L*-amino acids Schiff bases by the reactions of *L*-alanine, *L*-serine, *L*-valine, *L*-leucine, *L*-isoleucine, *L*-phenylglycine, *L*-phenylalanine with salicylaldehyde or 2-hydroxy-1-naphthaldehyde, respectively, in the present of KOH at the room temperature.

You, Jun; Wang, Xin; Liu, Bo*; Chen, Yanqiong
Chin. J. Org. Chem. **2011**, *31*(6), 832

Design, Synthesis and Anti-hyperglycemic Activity of *trans*-Cyclohexane-Bearing *C*-Glucosides as SGLT2 Inhibitors

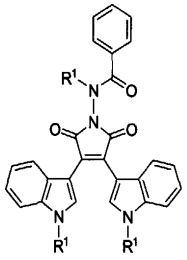


R = H, Me, Et, *n*-Pr

A series of *trans*-cyclohexane-bearing *C*-glucosides were designed and synthesized as SGLT2 inhibitors, with the key step being the regioselective Friedel-Crafts alkylation of *trans*-cyclohexanecarbonyl chloride and 4-bromoanisole mediated by anhydrous aluminum chloride. All the synthesized compounds were characterized by ^1H NMR and HRMS techniques. Evaluation of these compounds by mice oral glucose tolerance test (OGTT) revealed that all the synthesized compounds **11a**～**11d** exhibited potent antihyperglycemic activity, with **11c** being more potent than the positive control dapagliflozin, which is a promising agent for the treatment of type 2 diabetes.

Shao, Hua; Gao, Yunlong; Lou, Yuanyuan; Wang, Yuli; Liu, Wei; Xu, Weiren; Wang, Jianwu; Zhao, Guilong*; Tang, Lida*
Chin. J. Org. Chem. **2011**, *31*(6), 836

Synthesis and Fluorescence Properties of *N*-Benzamidobisindolylmaleimide Derivatives

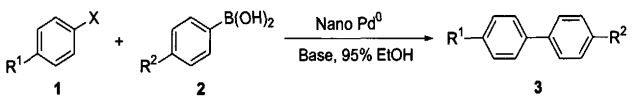


Five novel *N*-benzamidobisindolylmaleimide derivatives were designed and synthesized from succiminide by bromination, indole addition, hydrazine reaction, acylation, alkylation, and so on. Their ultraviolet, fluorescent properties and thermal behavior have been studied. The effect of structure of the compounds on fluorescent properties was also discussed.

Fan, Mei; Xie, Zhixiong; Xu, Zhihan; Ping, Bing; Zhao, Shengyin*

Chin. J. Org. Chem. **2011**, *31*(6), 843

Suzuki Reaction Catalyzed by Nanopalladium Stabilized by Gemini Quaternary Ammonium Salt

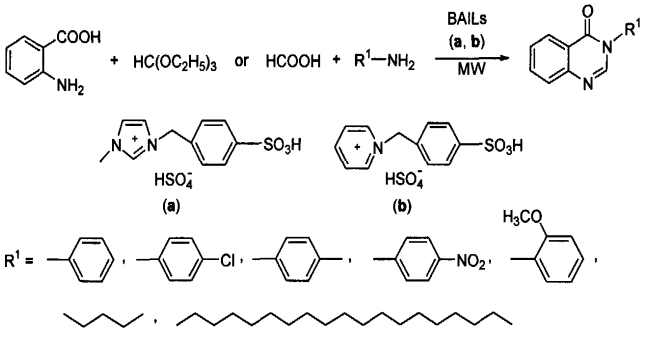


Palladium nanoparticles with the size ranged from 5 nm to 25 nm confirmed by transmission electron microscopy (TEM), generated *in situ* from the reaction of palladium(II) chloride and stabilized by Gemini quaternary ammonium salt, have been demonstrated to be an efficient catalyst for Suzuki carbon-carbon cross coupling reaction. A variety of aryl bronic acid and aryl halides have been proceed smoothly to afford the corresponding biaryls in high yields by using palladium nanoparticles in the presence of potassium carbonate without any ligands.

Zhou, Bo; Li, Yiqun*

Chin. J. Org. Chem. **2011**, *31*(6), 850

One-Pot Synthesis of 3-Substituted-4(3*H*)-quinazolinones in Acdic Ionic Liquids under Microwave Irradiation Conditions

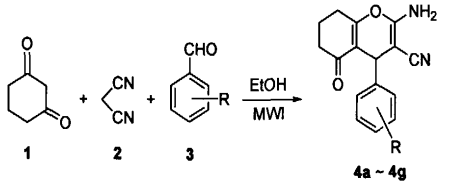


A rapid and efficient one-pot approach for the synthesis of 3-substituted-4(3*H*)-quinazolinones were developed. The reaction carried out in Brønsted acidic ionic liquids 1-(4-sulfonic)-benzyl-3-methylimidazolium hydrogen sulfate (**a**) and 1-(4-sulfonic)-benzyl pyridium hydrogen sulfate (**b**) under microwave irradiation using anthranilic acid, triethyl orthoformate or formic acid and aromatic or aliphatic amines as starting materials. The reactions completed within 4～6 min with the yields of 74%～94%. The ionic liquids could be recovered easily and recycled three times without any significant loss in catalytic activity.

Li, Xinzhong*; Lin, Qi; Eli, Wumanjiang

Chin. J. Org. Chem. **2011**, *31*(6), 855

One-Pot Synthesis of 2-Amino-4-aryl-5-oxo-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitriles under Microwave Irradiation without Catalyst



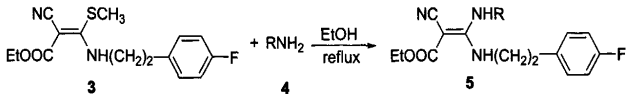
4a R = C_6H_5; **4b** R = 4-$CH_3OC_6H_4$; **4c** R = 4-$NO_2C_6H_4$; **4d** R = 2,4-$Cl_2C_6H_3$; **4e** R = 2-FC_6H_4; **4f** R = 3,4-$F_2C_6H_3$; **4g** R = 3-$CH_3C_6H_4$

Compounds **4a**～**4g** were prepared by using 1,3-cyclohexanedione, aromatic aldehydes and malononitrile in anhydrous alcohol under microwave irradiation without catalyst.

Luan, Changjun; Wang, Jianqiang; Zhang, Guohua; Wang, Wei; Tang, Shigui; Guo, Cheng*

Chin. J. Org. Chem. **2011**, *31*(6), 860

Synthesis and Bioactivity of Ethyl 2-Cyano-3-substituted-amino-3-[2-(4-fluorophenyl)-ethylamino]-acrylates



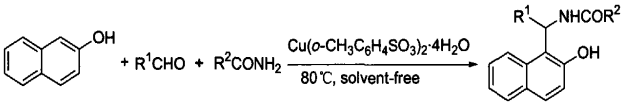
3 4 5

Zhou, Jinkang*; Song, baoan; Xue, Wei; Jin, Linhong; Gan, Zhengyang

Chin. J. Org. Chem. **2011**, *31*(6), 865

A series of ethyl 2-cyano-3-substituted-amino-3-[2-(4-fluorophenyl)-ethylamino]-acrylates were synthesized in three steps. Firstly, ethyl 2-cyano-3,3-dimethythioacrylate (**1**) was prepared from reactions of ethyl cyanoacetate with carbon disulfide and dimethyl sulfate. Secondly, it reacted with 4-fluorophenethylamine to yield ethyl 2-cyano-3-methythioacrylate-3-[2-(4-fluorophenyl)-ethylamino]-acrylate (**3**). Thirdly, the title compounds **5a**~**5h** were obtianed by the reactions of **3** with corresponding alkylamines. The structures of as-synthesized compounds were confirmed by elemental analysis, IR, ^{1}H NMR and ^{13}C NMR techniques. Preliminary bioassay test indicated that some compounds showed moderate antivirus activity against Tobacco mosaic virus.

One-Pot Catalytic Synthesis of Amidoalkyl Naphthols Derivatives under Solvent-Free Conditions

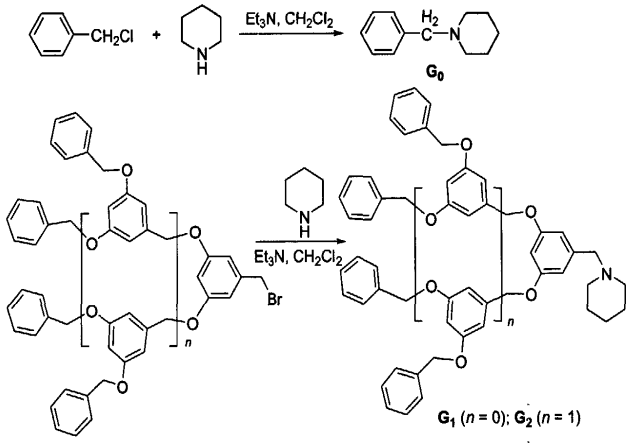


Song, Zhiguo*; Zhao, Shuang; Wan, Xin

Chin. J. Org. Chem. **2011**, *31*(6), 870

Amidoalkyl naphthol derivatives were efficiently synthesized by a three components one-pot reaction of 2-naphthol, aldehydes and amines in the presence of copper *o*-toluenesulfonate without solvent.

Study on Henry Reaction Catalyzed by Dendritic Amines

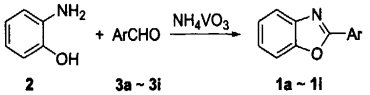


Yi, Bing*; Zhang, Yang; Shu, Xiaoma; Yang, Yan; Du, Wenle; Liu, Zhanpeng

Chin. J. Org. Chem. **2011**, *31*(6), 874

With the Henry reaction of benzaldehyde and nitromethane as the model, the catalytic ability of some weak basic catalysts was explored. The first- and the second-generation polyphenyl ether type dendritic piperidine catalysts, which were characterized by ^{1}H NMR and MS techniques, were synthesized, and their catalytic abilities were investigated in Henry reaction. The obtained results indicated that the lower generation dendritic piperidine provided preferable catalytic activity in the case of nitromethane, and the higher generation one showed negative dendritic effect.

Synthesis of 2-Substituted Aryl Benzoxazoles Catalyzed by Ammonium Metavanadate



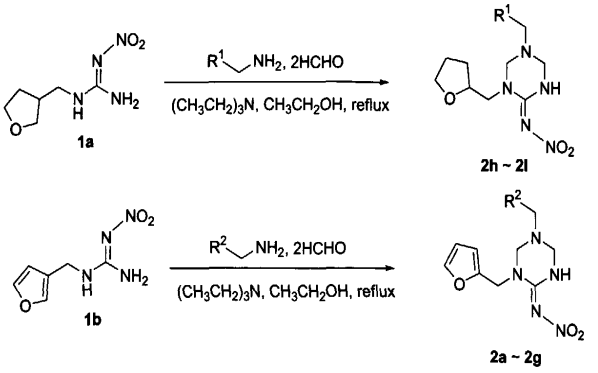
2 3a ~ 3i 1a ~ 1i

Xiao, Liwei

Chin. J. Org. Chem. **2011**, *31*(6), 878

Ammonium metavanadate was found to be a practical catalyst for synthesizing 2-substituted aryl benzoxazoles, starting from 2-amino-phenol and aromatic aldehydes in ethanol at room temperature. The method was proved to be simple and convenient, and the product was isolated with excellent yields.

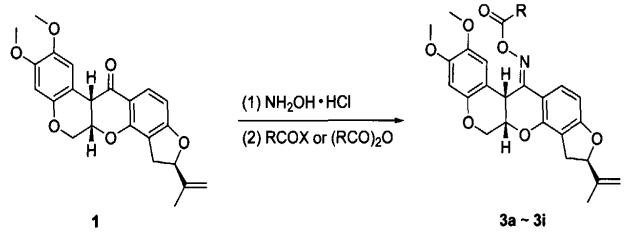
Synthesis and Insecticidal Activities of 1,5-Disubstituted-hexahydrotriazine-2-*N*-nitroimines



Twelve novel 1,5-disubstituted-hexahydrotriazine-2-*N*-nitroimines (**2a**~**2l**) were synthesized with *N*-nitro-*S*-methylisothiourea and substituted benzylamines as raw materials via nucleophilic substitution and Manich reactions in turn. Their structures were elucidated and confirmed by ^{1}H NMR, IR spectra and elemental analysis. The preliminary insecticidal activity tests indicated that all the target compounds have preferable insecticidal activity at the concentration of 500 mg/L.

Xue, Sijia*; Ma, Xubo; Bu, Hongfei
Chin. J. Org. Chem. **2011**, *31*(6), 881

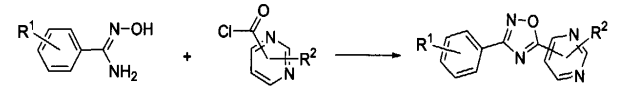
Synthesis and Biological Activity of Rotenone Oxime Esters



Nine rotenone oxime ester derivatives were synthesized from natural rotenone with two steps. Their structures were clearly established by ^{1}H NMR, IR spectra and elemental analysis. The preliminary bioassays indicated that three compounds exhibited strong insecticidal activity.

Ye, Jiao; Peng, Junmei; Hu, Aixi*; Wang, Chao; Zou, Meng; Ou, Xiaoming
Chin. J. Org. Chem. **2011**, *31*(6), 886

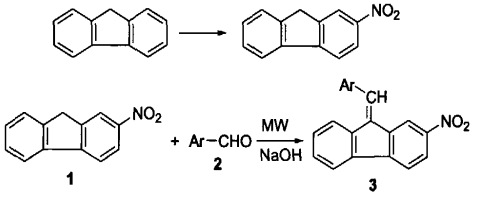
Synthesis and Biological Activity of 5-Pyrimidinyl-1,2,4-oxadiazole Derivatives



A series of 5-pyrimidinyl-1,2,4-oxadiazole derivatives were prepared by the reaction of substituted benzamidoxime with pyrimidinecarbonyl chloride. Preliminary bioassay showed that most of title compounds have good herbicidal activity.

Huang, Tonghui; Tu, Haiyang*; Liu, Ming; Hou, Changjian; Zhang, Aidong*
Chin. J. Org. Chem. **2011**, *31*(6), 891

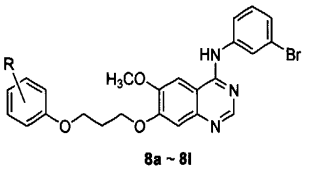
Microwave-Assisted Knoevenagel Reaction of 2-Nitrofluorene with Aromatic Aldehyde



A series of novel Knoevenagel products of 2-nitrofluorene to a series of aromatic aldehydes were obtained in the presence of sodium hydroxide under microwave irradiation (500 W) within short reaction times (5~8 min) in high yields (78%~89%). The structures of the products were characterized by IR, ^{1}H NMR, MS techniques and elemental analysis. The UV absorption and fluorescent spectra of compounds **3a**~**3d** were detected and showed that they had fluorescence.

Hu, Weibing*; Feng, Fu; Li, Yunpan; Zhou, Hongyan; Wang, Hui; Tian, Dating
Chin. J. Org. Chem. **2011**, *31*(6), 897

Synthesis, Characterization and X-ray Structure of 4-Phenylamino Quinazoline Derivatives

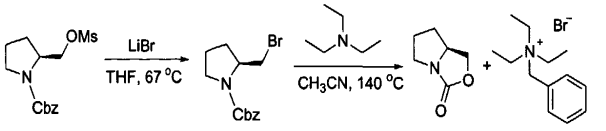


8a ~ 8l

Twelve new compounds, 4-phenylamino quinazoline compounds **8a**~**8l**, were synthesized from vanillin by seven steps reaction. Their structures were characterized by ^{1}H NMR, ^{13}C NMR, IR and MS techniques. Compound **8b** was investigated with X-ray crystallography. The crystal of **8b** belongs to Triclinic, space group *P*-1 with cell parameters a=0.8868(5) nm, b=1.0239(6) nm, c=1.4515(9) nm, α=71.02(2)°, β=82.42(2)°, γ=71.78(2)°, V=1.1831 nm^3, Z=2, D_c=1.430 Mg/m^3, μ(Mo Kα)=1.773 mm^{-1}, λ=0.071073 nm, F(000)=522.

Yan, Heping; Ouyang, Guiping*
Chin. J. Org. Chem. **2011**, *31*(6), 901

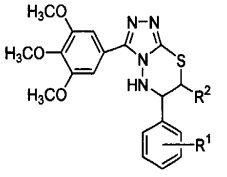
Stability of Cbz or Boc Protected *L*-Prolinol Derivatives



The stability of Cbz or Boc protected *L*-prolinol derivatives is investigated. It was found that when the hydroxyl group of Boc protected *L*-prolinol is replaced by easily leaving group, it is unstable and cyclization proceeds effectively to give γ-butyrolactone at 67 ℃; however, Cbz protected *L*-prolinol derivatives are more stable, similar cyclization will happen at 140 ℃ in the presence of triethylamine.

Wang, Ge; Cao, Xiaohui; Sun, Huichao; Chen, Ligong*
Chin. J. Org. Chem. **2011**, *31*(6), 908

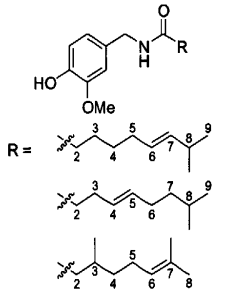
Synthesis and Antitumor Activity of 5,6-2*H*-[1,2,4]-Triazolo[3,4-*b*][1,3,4]thiadiazine Derivatives



A series of 4-(substituted-3-phenylideneamino)-3-(3,4,5-trimethoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thiones (**3**) were synthesized by the condensation reaction of 4-amino-3-(3,4,5-trimethoxyphenyl)-1*H*-1,2,4-triazole-5(4*H*)-thione (**2**) with various substituted benzaldehydes. Alkylation of **3** with alkyl halides afforded [1,2,4]-triazolo[3,4-*b*][1,3,4]thiadiazine derivatives **4**. Their structures were confirmed by ^{1}H NMR, ^{13}C NMR, IR spectra and elemental analyses. Preliminary bioassay indicated that some compounds posed antitumor activity to human prostate cancer cell line (PC3) *in vitro* by microculture tetrazolium (MTT) method. The antiproliferation activity of compound **4a** to PC3 cells at the concentration of 10 μmol• L^{-1} was 75.9%.

Zheng, Yuguo; Xue, Wei*; Guo, Qingqing; Lu, Ping; Wang, Zhenchao; Yuan, Kai
Chin. J. Org. Chem. **2011**, *31*(6), 912

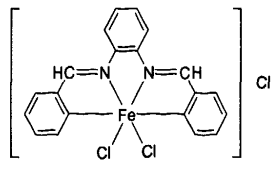
Synthesis and Characterization of New Members of Capsaicinoids and Preliminary Research on the Relationship between the Strcture and Pungency



Two new members of capsaicinoids, *N*-(4-hydroxy-3-methoxybenzyl)-8-methylnon-*trans*-4-enamide and *N*-(4-hydroxy-3-methoxybenzyl)-3,7-dimethyloct-6-enamide, were synthesized, and their structures were confirmed by elemental analysis, FT-IR, ^{1}H NMR, ^{13}C NMR *etc*. Meanwhile, their pungencies were determined through scoville organoleptic test and compared with capsaicin, norcapsaicin and homocapsicin. The double bond location, branch chain and length of the alcyl chain were confirmed to be the structural factors that could affect the pungency remarkably.

Wang, Junlian; Zhou, Shengze; Peng, Zhenghong; Zhou, Xiangfeng; Zhang, Xiaobin; Peng, Bixian*
Chin. J. Org. Chem. **2011**, *31*(6), 917

Epoxidation of Cyclohexene Catalyzed by Metal Iron Schiff Base Complexes



One of pyridinecarboxaldehyde iron(III) Schiff base complexes of [Fe(PA$_2$OPd)Cl$_2$]Cl was synthesized and its catalytic activities for epoxidation of cyclohexene were studied. The complex of [Fe(PA$_2$OPd)Cl$_2$]Cl has the highest catalytic activity for cyclohexene epoxidation using hydrogen peroxide as an oxygen source. At optimum reaction condition (ethyl acetate as a solvent, 25 ℃, pH=5.0, 6.0 h), the conversion rate of cyclohexene reached 90.5%, the selectivity of cyclohexene oxide was 97.2%, and the TON (moles of cyclohexene oxide produced per mole of [Fe(PA$_2$OPd)Cl$_2$]Cl) was 1759.

Zhang, Fuli*; Tang, Kun
Chin. J. Org. Chem. **2011**, *31*(6), 921