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BINOL 酸作用下 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应

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摘要: 探讨了价廉易得、环境友好和无毒性的 BINOL 酸作用的 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应. 在回流条件下,于二甲亚砜中,当 BINOL 酸的摩尔分数为 15% 时,α-EWG 二硫缩烯酮与二苯甲醇能有效进行偶联反应,高产率地生成 α-EWG 二硫缩烯酮衍生物.

关键词: BINOL 酸; 二苯甲醇; 二硫缩烯酮; 偶联反应
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0 引言

C—C 键的形成是构建有机分子碳骨架的重要途径之一[1],在有机合成中有重要的作用,由碳亲核体(RH)直接取代醇分子(R′OH)中的羟基基团[2-3],在此种类型的反应中,醇被看作是绿色环保的试剂,因为它们反应的副产物只是水[4]. 近年来,已有很多由 RH 和 R′OH 直接作用形成碳碳键结构骨架的反应陆续被报道. 在这些报道中,烯丙基或苄基醇与活性亚甲基吲哚或烷氧基酮之间的偶联反应已被证明是成功的[5-7]. 最近,烷硫基活化的内烯烃二硫缩烯酮的偶联反应引起人们的关注,利用烷硫基的推电子作用和 α-位的吸电子基团的拉电子作用使二硫缩烯酮的 α-位呈现较强亲核能力的性质,Zhang Qian 等和巴哈尔古丽·别克吐尔逊等成功实现了 $BF_3 \cdot Et_2O$ 作用的二硫缩烯酮和二苯甲醇的偶联反应[2-3]. 然而,他们所使用的催化剂都有一定的毒性,或者产物的产率比较低,这些反应的缺点不仅限制了它们的应用,而且不符合绿色合成的理念

α-羰基二硫缩烯酮是易制备和具有多反应中心的重要有机合成中间体[8-10],在构造芳环[11-12]、杂环化合物的合成[13],多取代烯烃的合成[14]和用作无气味的代硫醇试剂[15-17]中有广泛的应用. 鉴于 α-羰基二硫缩烯酮在合成中的广泛应用和醇在有机合成中的重要性,进行基于 α-羰基二硫缩烯酮与醇的偶联反应是一项非常有意义的工作. 有机小分子在催化反应时具有条件温和、操作简单、环境友好且反应完全后易回收等特点,这更加充分体现了现代化学中绿色催化这一重要概念. 有机小分子催化剂具有毒性小、高效、高选择性且在空气中稳定等优点,被广泛应用于有机合成中[18-19]. 本文在张前等[2]和于海丰等[20]工作的基础上,开展了 BINOL 酸作用的 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应研究.

1 实验部分

1.1 仪器与试剂

X-6 数字显示显微熔点测定仪(北京泰克仪器有限公司); Unity-400MHz 核磁共振仪(TMS 为内标,$CDCl_3$ 为溶剂,美国 Bruker 公司); 所用试剂均为分析纯; 水为蒸馏水; 按文献[21-22]制备 BINOL 酸; 按文献[2,23-26]制备 α-EWG 二硫缩烯酮 **1**.

1.2 实验过程

1.2.1 BINOL 酸的制备　由 3-羟基萘-2-甲酸甲酯经 CuCl(OH)·TMEDA 催化氧化偶联得黄色颗粒状晶体,用 NaOH 的甲醇溶液($2\ mol \cdot L^{-1}$,80 mL)加热回流 12 h,冷却,然后加入一定量的水,再用 HCl($2\ mol \cdot L^{-1}$)酸化至 pH 值为 1,抽滤、晾干得到淡黄色固体 2,2′-羟基-1,1′-联二萘-3,3′-二甲酸,

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合成路线见图 1. ^{1}H NMR 和 ^{13}C NMR 的数据与文献[21-22]报道一致.

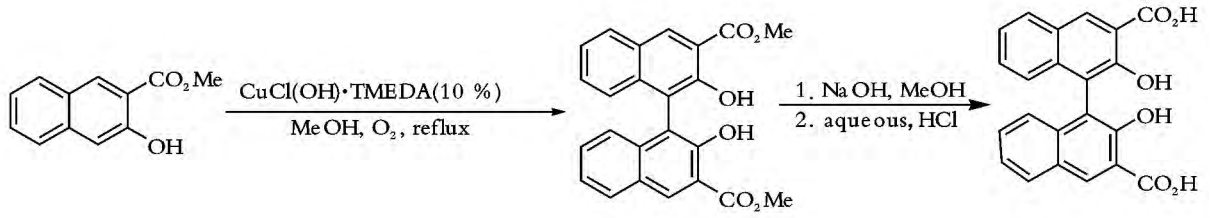


图 1 BINOL 酸的合成路线

1.2.2 二苯甲醇的合成 向 250 mL 的圆底烧瓶中加入二苯甲酮 9.10 g(50 mmol),用 50 mL 无水 C_2H_5OH 溶解,在冰水浴冷却条件下,向里分 3 次加入 $NaBH_4$ 0.57 g(15 mmol). 常温搅拌 60 min,TLC 监测反应直至完成,将反应液倾入盛有 300 mL 冰蒸馏水的烧杯中,充分搅拌,析出大量白色固体. 抽滤、水洗、晾干得二苯甲醇,产率为 99%. 用同样方法制备 4-甲基二苯甲醇,产率为 98%.

1.2.3 BINOL 酸作用下 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应(以 1a 与 2a 反应为例) 在 25 mL 的圆底烧瓶中依次加入 1a 0.124 0 g (0.500 mmol)、二苯甲醇 2a 0.147 2 g(0.800 mmol)、BINOL 酸 0.028 1 g(0.075 mmol)和 2 mL DMSO. 回流 5 h,通过 TLC 检测底物消失,然后在反应液中加入 15 mL 的水,转移至分液漏斗用二氯甲烷萃取(10 mL×3),有机相用 8 mL 饱和的碳酸钠溶液洗涤,无水硫酸镁干燥,然后加入 1 g 硅胶,通过旋转蒸发除去溶剂,最后柱层析[洗脱剂 v(石油醚):v(乙酸乙酯)=7:1]纯化后得到 3a 0.186 3 g,产率 90%. 用相同方法得到化合物 3b~3t,实验结果列于表 1.

1.2.4 化合物表征 **3a: 黄色晶体**,m. p.: 152~154 ℃[7,18]. ^{1}H NMR(400 MHz,$CDCl_3$),δ: 3.35(d,J=8.0 Hz,4H),5.94(s,1H),6.68(d,J=16.0 Hz,1H),7.13(s,2H),7.24~7.34(m,13H),7.48(d,J=16.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 36.0,38.9,54.9,15.3,125.3,126.8(2C),127.6,128.1(2C),128.6(6C),129.0(4C),129.6,135.4,141.3,141.5(2C),156.4,186.5.

3b: 淡黄色晶体,m. p.: 79~80 ℃. ^{1}H NMR (400 MHz,$CDCl_3$),δ: 2.31(s,3H),3.33(d,J=8.0 Hz,4H),5.89(s,1H),6.69(d,J=16.0 Hz,1H),7.13(s,6H),7.24~7.30(m,8H),7.48(d,J=16.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 21.1,36.1,38.9,54.6,125.4,126.7,127.8,128.1(2C),128.6(4C),128.9(2C),128.9(2C),129.3(2C),129.6,135.5,136.4,138.2,141.1,142.0,165.2,186.6.

3c: 黄色晶体,m. p.: 151~153 ℃[7]. ^{1}H NMR (400 MHz,$CDCl_3$),δ: 3.36(d,J=4.0 Hz,4H),5.93(s,1H),6.62(d,J=16.0 Hz,1H),7.02(d,J=8.0 Hz,2H),7.19(d,J=8.0 Hz,2H),7.24~7.32(m,10H),7.40(d,J=16.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 36.0,38.9,54.7,125.8,126.8(2C),127.5,128.6(4C),128.8(2C),128.9(4C),129.2(2C),133.9,135.3,139.6,141.5(2C),165.9,86.3.

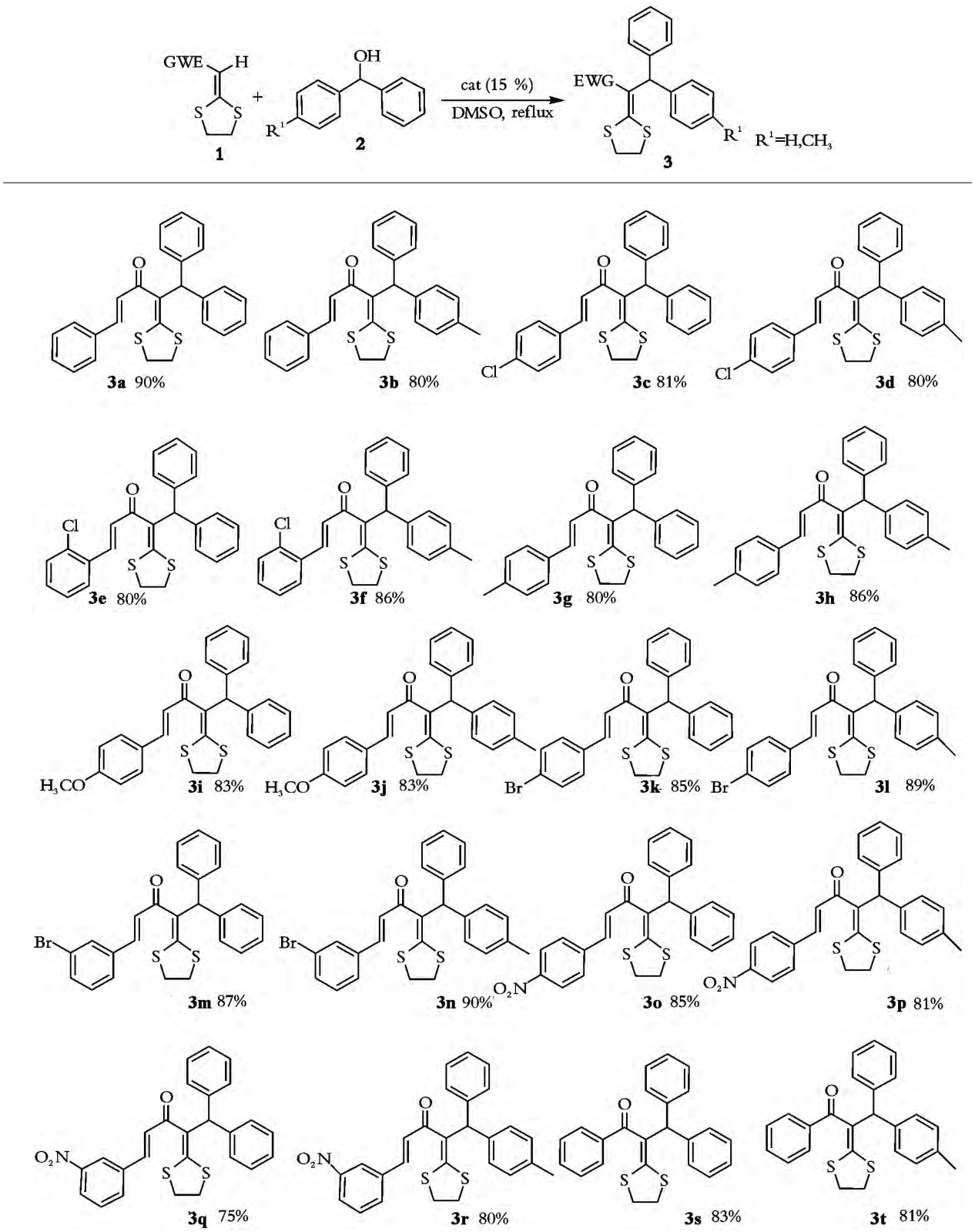
3d: 淡黄色晶体,m. p.: 156~158 ℃. ^{1}H NMR (400 MHz,$CDCl_3$),δ: 2.31(s,3H),3.34(d,J=8.0 Hz,4H),5.89(s,1H),6.63(d,J=16.0 Hz,1H),7.03(d,J=4.0 Hz,2H),7.13~7.22(m,9H),7.29(d,J=8.0 Hz,2H),7.39(d,J=12.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 21.1,36.1,39.0,54.5,125.9,126.7,127.7,128.6(2C),128.8(2C),128.8(2C),128.9(2C),129.2(2C),129.3(2C),134.0,135.3,136.5,139.2,139.5,142.0,165.7,186.3.

3e: 黄色晶体,m. p.: 161~163 ℃. ^{1}H NMR (400 MHz,$CDCl_3$),δ: 3.37(d,J=8.0 Hz,4H),5.94(s,1H),6.65(d,J=16.0 Hz,1H),6.85(d,J=4.0 Hz,1H),7.04(s,1H),7.14~7.18(m,1H),7.25~7.33(m,11H),7.88(d,J=16.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 29.7,35.8,38.6,54.7,126.6,126.8(2C),127.4,127.6,127.6,128.6(4C),129.0(4C),129.9,130.2,133.7,135.1,137.0,141.5,166.3,196.0.

3f: 黄色晶体,m. p.: 130~132 ℃. ^{1}H NMR (400 MHz,$CDCl_3$),δ: 2.32(s,3H),3.35(d,J=12.0 Hz,4H),5.89(s,1H),6.65(d,J=12.0 Hz,1H),6.85(d,J=8.0 Hz,1H),7.04(s,1H),7.13(s,5H),7.26(d,J=24.0 Hz,6H),7.87(d,J=16.0 Hz,1H). ^{13}C NMR(100 MHz,$CDCl_3$),δ: 21.1,

36.0,39.0,54.5,126.6,126.7,127.6(2C),127.7,128.6(2C),128.9(2C),128.9(2C),129.3(2C),129.9,130.2,133.8,135.0,136.5,136.8,138.2,142.0,166.0,186.1.

表 1　BINOL 酸作用下 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应



3g: 黄色晶体, m. p.: 118 ~ 120 ℃[7]. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.29 (s, 3H), 3.35 (d, J = 4.0 Hz, 4H), 5.93 (s, 1H), 6.64 (d, J = 16.0 Hz, 1H), 7.03 (s, 4H), 7.26 ~ 7.32 (m, 10H), 7.47 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.4, 36.0, 38.8, 55.1, 124.4, 126.7 (2C), 127.7, 128.1 (2C), 128.6 (4C), 129.0 (4C), 129.3 (2C), 132.7, 140.0, 141.4, 141.6 (2C), 164.9, 196.6.

3h: 淡黄色晶体, m. p.: 147 ~ 149 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.30 (d, J = 8.0 Hz, 6H), 3.32 (d, J = 8.0 Hz, 4H), 5.89 (s, 1H), 6.65 (d, J = 16.0 Hz, 1H), 7.03 (s, 4H), 7.13 (s, 4H), 7.22 ~ 7.30 (m, 5H), 7.47 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.1, 21.4, 36.0, 38.9, 54.6, 124.5, 126.6, 127.9, 128.1 (2C), 128.5 (2C), 128.9 (2C), 129.0 (2C), 129.3 (4C), 132.8, 136.4, 138.2, 140.0, 141.3, 142.1, 164.8, 186.8.

3i: 黄色晶体, m. p.: 144 ~ 146 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.34 (d, J = 4.0 Hz, 4H), 3.78 (s, 3H), 5.93 (s, 1H), 6.65 (d, J = 16.0 Hz, 1H), 6.75 (d, J = 8.0 Hz, 2H), 7.09 (d, J = 8.0 Hz, 2H), 7.26 ~ 7.32 (m, 10H), 7.46 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 36.0, 38.9, 54.9, 55.3, 114.0 (2C), 123.2, 126.7 (2C), 127.7, 128.2, 128.5 (4C), 129.0 (4C), 129.7 (2C), 141.1, 141.6 (2C), 161.0, 164.6, 186.6.

3j: 淡黄色晶体, m. p.: 136 ~ 137 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.31 (s, 3H), 3.32 (d, J = 8.0 Hz, 4H), 3.77 (s, 3H), 5.89 (s, 1H), 6.57 (d, J = 16.0 Hz, 1H), 6.75 (d, J = 8.0 Hz, 2H), 7.08 ~ 7.14 (m, 6H), 7.22 ~ 7.30 (m, 5H), 7.45 (d, J = 12.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.1, 36.0, 38.9, 54.6, 55.3, 114.0 (2C), 123.3, 126.6, 127.9, 128.3, 128.5 (2C), 128.9 (2C), 129.0 (2C), 129.3 (2C), 129.7 (2C), 136.3, 138.3, 141.0, 142.1, 160.9, 164.3, 186.8.

3k: 黄色晶体, m. p.: 114 ~ 116 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.28 (d, J = 4.0 Hz, 4H), 5.85 (s, 1H), 6.56 (d, J = 16.0 Hz, 1H), 6.88 (d, J = 8.0 Hz, 2H), 7.04 (s, 2H), 7.15 ~ 7.28 (m, 10H), 7.38 (d, J = 16.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 35.0, 38.0, 53.7, 122.6, 124.9, 125.9 (2C), 126.4, 127.6 (4C), 127.9 (4C), 128.3 (2C), 130.7 (2C), 133.3, 138.6, 140.5 (2C), 164.9, 184.9.

3l: 黄色晶体, m. p.: 170 ~ 172 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.32 (s, 3H), 3.35 (d, J = 8.0 Hz, 4H), 5.88 (s, 1H), 6.64 (d, J = 16.0 Hz, 1H), 6.96 (d, J = 12.0 Hz, 2H), 7.13 (s, 4H), 7.22 ~ 7.35 (m, 7H), 7.39 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.1, 36.0, 39.0, 54.5, 123.6, 126.0, 126.7, 127.6, 128.6 (2C), 128.8 (2C), 128.9 (2C), 129.3 (2C), 129.4 (2C), 131.7 (2C), 134.4, 136.5, 138.2, 139.5, 142.0, 165.9, 186.3.

3m: 黄色晶体, m. p.: 204 ~ 205 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.30 (d, J = 4.0 Hz, 4H), 5.96 (s, 1H), 6.54 (d, J = 16.0 Hz, 1H), 6.97 (s, 1H), 7.00 ~ 7.07 (m, 2H), 7.15 ~ 7.31 (m, 12H)). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 29.9, 35.9, 39.2, 55.0, 122.6, 126.7, 126.8, 126.9 (2C), 127.5, 128.7 (4C), 128.9 (4C), 130.0, 130.5, 132.2, 137.7, 139.4, 141.6, 166.6, 186.0.

3n: 黄色晶体, m. p.: 120 ~ 122 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.33 (s, 3H), 3.35 (d, J = 8.0 Hz, 4H), 5.89 (s, 1 H), 6.60 (d, J = 16.0 Hz, 1H), 7.05 ~ 7.14 (m, 7H), 7.24 (d, J = 8.0 Hz, 3H), 7.36 (d, J = 16.0 Hz, 4H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.3, 36.0, 39.0, 54.6, 122.7, 126.8, 126.9, 126.9, 127.7, 128.7 (2C), 128.8 (2C), 128.8 (2C), 129.4 (2C), 130.0, 130.4, 132.2, 136.5, 137.8, 138.4, 139.3, 142.0, 166.3, 186.0.

3o: 淡黄色晶体, m. p.: 194 ~ 196 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.32 (d, J = 8.0 Hz, 4H), 5.87 (s, 1H), 6.66 (d, J = 16.0 Hz, 1H), 7.11 ~ 7.26 (m, 12H), 7.35 (d, J = 12.0 Hz, 1H), 8.00 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 29.7, 35.7, 39.0, 54.7, 123.8 (2C), 127.0 (2C), 127.3, 128.4 (2C), 128.7 (4C), 128.9 (4C), 129.2, 137.7, 141.4, 141.8, 147.8, 167.6, 185.3.

3p: 淡黄色晶体, m. p.: 161 ~ 163 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.32 (s, 3H), 3.38 (d, J = 8.0 Hz, 4H), 5.90 (s, 1H), 6.74 (d, J = 16.0 Hz, 1H), 7.14 ~ 7.32 (m, 11H), 7.42 (d, J = 16.0 Hz, 1H), 8.07 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.1, 36.1, 39.0, 54.5, 123.8 (2C), 126.9, 127.5, 128.4 (2C), 128.7 (2C), 128.8 (2C), 128.8 (2C), 129.3, 129.4 (2C), 136.7, 137.6,

138.1,141.9,141.9,147.8,167.4,185.6.

3q: 淡黄色晶体, m. p.: 217 ~ 219 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.32 (d, J = 8.0 Hz, 4H), 5.88 (s, 1H), 6.65 (d, J = 16.0 Hz, 1H), 7.18 ~ 7.40 (m, 13H), 7.77 (s, 1H), 8.01 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 13.0, 21.6, 28.6 (2C), 34.9, 37.7, 53.6, 120.9, 122.6, 125.9, 127.1, 127.7 (4C), 127.8 (4C), 128.5, 132.9, 136.3, 136.9, 140.4, 147.3, 184.6.

3r: 黄色晶体, m. p.: 191 ~ 194 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.32 (s, 3H), 3.38 (d, J = 8.0 Hz, 4H), 5.91 (s, 1H), 6.72 (d, J = 16.0 Hz, 1H), 7.14 ~ 7.25 (m, 7H), 7.33 ~ 7.47 (m, 5H), 7.84 (s, 1H), 8.08 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.0, 36.0, 39.0, 54.6, 121.8, 123.6, 126.9, 127.5, 128.2, 128.7 (2C), 128.8 (4C), 129.5 (3C), 134.0, 136.7, 137.4, 137.9, 138.2, 142.0, 149.4, 167.2, 185.7.

3s: 淡黄色晶体, m. p.: 141 ~ 142 ℃[18]. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 3.25 (d, J = 4.0 Hz, 4H), 5.59 (s, 1H), 7.17 ~ 7.19 (m, 2H), 7.25 (s, 8H), 7.32 (d, J = 7.7 Hz, 2H), 7.41 (d, J = 7.3 Hz, 1H), 7.55 (d, J = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 37.3, 38.6, 56.3, 126.4 (2C), 128.1 (4C), 128.2 (2C), 128.6 (2C), 129.5 (4C), 131.6 (2C), 138.9, 141.0 (2C), 154.7, 195.3.

3t: 淡黄色晶体, m. p.: 112 ~ 114 ℃. ^{1}H NMR (400 MHz, $CDCl_3$), δ: 2.29 (s, 3H), 3.23 (d, J = 8.0 Hz, 4H), 5.56 (s, 1H), 7.05 (d, J = 8.0 Hz, 2H), 7.14 ~ 7.33 (m, 9H), 7.41 (d, J = 8.0 Hz, 1H), 7.56 (d, J = 7.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$), δ: 21.1, 37.5, 38.7, 56.1, 126.4, 128.0, 128.1 (2C), 128.2 (2C), 128.6 (2C), 128.9 (2C), 129.4 (2 C), 129.5 (2C), 131.6, 136.0, 137.9, 139.0, 141.3, 154.9, 195.3.

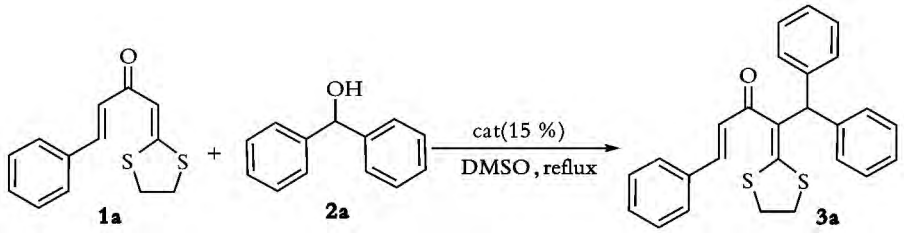
2　结果与讨论

2.1　反应条件优化

2.1.1　**催化剂和溶剂**　**1a** 0.5 mmol, **2a** 0.8 mmol, 在回流条件下反应 5 h, 其余反应条件同 1.2.3 节所述, 研究催化剂和溶剂对 **3a** 产率的影响, 结果见表 2. 由表 2 可见, No. 1 ~ No. 4 为催化剂对化合物 **3a** 产率的影响, 结果表明: **3a** 产率最高 (No. 2, 90%), 可能是萘酚酸和水杨酸酸性较弱, 而对甲苯磺酸酸性强, 不利于反应进行. 因此, 选用 BINOL 酸为催化剂. No. 5 ~ No. 8 为溶剂对 **3a** 产率的影响, 结果表明: 反应不能进行, **3a** 产率为 0. 可能是因为该反应为非均相反应, 催化剂 BINOL 酸在二氯甲烷等溶剂中不能溶解. 因此, 选用二甲基亚砜为溶剂. 化合物 **3a** 的合成路线见图 2.

表 2　催化剂和溶剂对 **3a** 产率的影响[a]

No.	Solvent (2 mL)	Cat (15 %)	t/h	Yield /%
1	DMSO	Naphthol acid	5	62
2	DMSO	BINOL acid	5	90
3	DMSO	Salicylic acid	5	54
4	DMSO	*p*-Toluenesulfonic acid	5	80
5	CH_2Cl_2	BINOL acid	20	–
6	CH_3CN	BINOL acid	20	–
7	H_2O	BINOL acid	20	–
8	–	BINOL acid	20	–



[a]**1a** 0.5 mmol, **2a** 0.8 mmol, reflux 5 h, 其余反应条件同 1.2.3.

图 2　化合物 **3a** 的合成路线

2.1.2　**催化剂用量**　DMSO 2 mL, 回流条件下反应 5 h, 其余反应条件同 2.1.1 节所述, 研究 BINOL 酸的量对 **3a** 产率的影响, 结果见表 3.

当 BINOL 酸的摩尔分数由 20% 降至 5% 时, 在 5 h 内, 该反应能有效完成, 几乎定量得到 **3a** (No. 1 ~ No. 4); 当 BINOL 酸的摩尔分数为 1% 时, 反应 5 h, **3a** 的产率仅为 20% (No. 5); 而没有 BINOL 酸存在时, 反应 5 h, **3a** 的产率仅为 7% (No. 6). 因此, 选用

BINOL 酸的摩尔分数为 15%.

2.2 BINOL 酸作用下 α-EWG 二硫缩烯酮与二苯甲醇的偶联反应

在最佳反应条件下,α-EWG 二硫缩烯酮(1)与二苯甲醇(2)能有效进行偶联反应,生成一系列 α-EWG 二硫缩烯酮衍生物 3a ~ 3t(见表 1).

表 3 催化剂的量对 3a 产率的影响[a]

序号	催化剂的摩尔分数/%	产率/%
1	20	90
2	15	90
3	10	80
4	5	35
5	1	20
6	–	7

[a]**1a** 0.5 mmol,**2a** 0.8 mmol,reflux 5 h,其余反应条件同 2.1.1.

3 结论

以 α-EWG 二硫缩烯酮(1)和二苯甲醇(2)为原料,BINOL 酸为催化剂,DMSO 为溶剂,在回流条件下高产率合成了 α-EWG 二硫缩烯酮衍生物(3a ~ 3t).

本文以 3a 为例,用单因素法研究了催化剂、溶剂和催化剂用量对 3a 产率的影响. 结果表明: 在最优反应条件(**1a** 0.5 mmol,**2a** 0.8 mmol,联萘酚酸摩尔分数为 15%,DMSO 2 mL,回流 5 h)下,3a 的产率为 90%. 该反应具有反应条件温和、环境友好和易操作等优点.

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The BINOL Acid-Mediated Coupling Reaction of α-EWG Ketene Dithioacetals with Diphenylmethanol

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Abstract: An inexpensive and environmentally friendly BINOL acid-mediated coupling reaction of α-EWG ketene dithioacetals with diphenylmethanol has been developed. The reaction is efficiently performed in the presence of BINOL acid(15 mmol%) at reflux in DMSO, forming α-EWG ketene dithioacetals derivatives in good yields.

Key words: BINOL acid; diphenylmethanol; ketene dithioacetals; coupling reaction

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