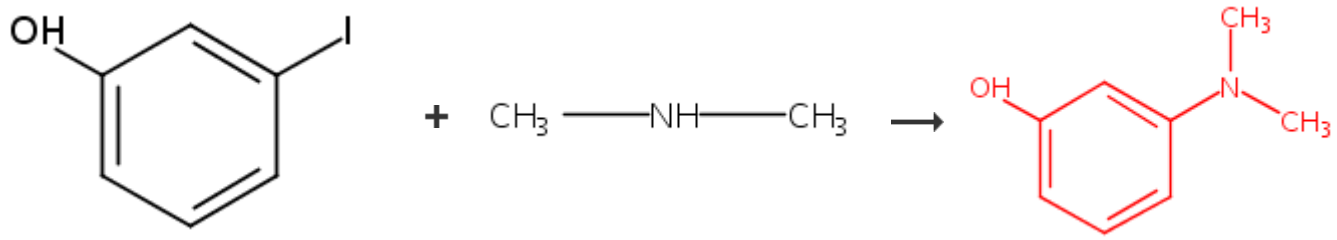
1. Single Step



91%

Overview

Steps/Stages

1.1 R:KOH, C:58509-59-4, C:CuI, S:H_2O, 24 h, 25°C

Notes

sealed tube used, alternative preparation shown, oxime catalyst prepared and used, green chemistry, Reactants: 2, Reagents: 1, Catalysts: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

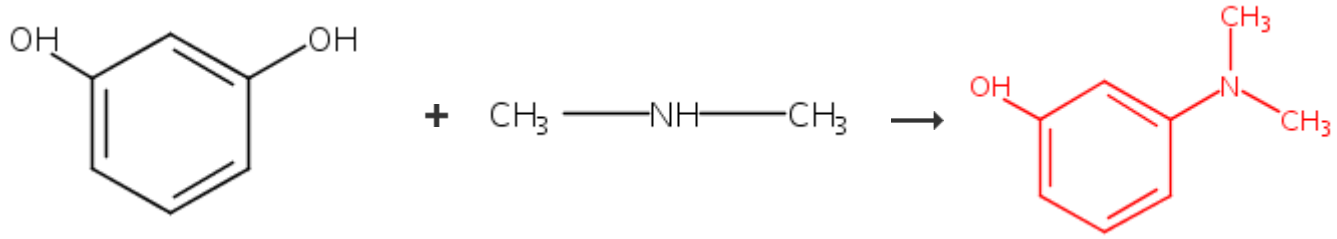
References

Room-Temperature Copper-Catalyzed Arylation of Dimethylamine and Methylamine in Neat Water

By Wang, Deping et al

From Advanced Synthesis & Catalysis, 357(4), 714-718; 2015

2. Single Step



93%

Overview

Steps/Stages

1.1 R:H_2, C:Ni, S:H_2O, rt, 0.05 MPa; rt → 200°C; 3 h, 200°C

Notes

thermal, autoclave used, Raney nickel used, alternative reaction conditions shown, Reactants: 2, Reagents: 1, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

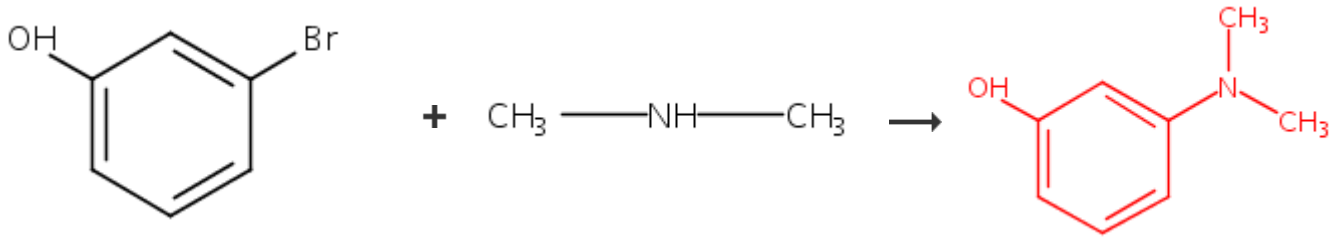
References

Mechanism study on Raney nickel-catalyzed amination of resorcinol

By Ge, Xin et al

From Catalysis Communications, 46, 201-207; 2014

3. Single Step



89%

Overview

Steps/Stages

1.1 R:KOH, C:58509-59-4, C:CuI, S:H_2O, 24 h, 85°C

Notes

sealed tube used, alternative preparation shown, oxime catalyst prepared and used, green chemistry, Reactants: 2, Reagents: 1, Catalysts: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

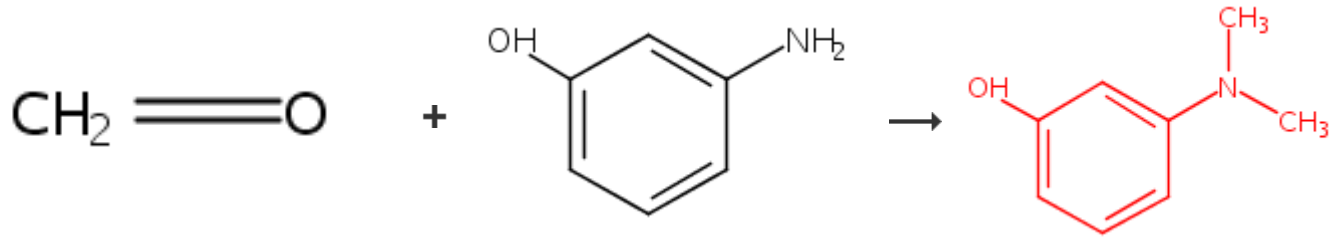
References

Room-Temperature Copper-Catalyzed Arylation of Dimethylamine and Methylamine in Neat Water

By Wang, Deping et al

From Advanced Synthesis & Catalysis, 357(4), 714-718; 2015

4. Single Step



96%

Overview

Steps/Stages

Notes

1.1 R:$NaBH_4$, S:MeOH

Reactants: 2, Reagents: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

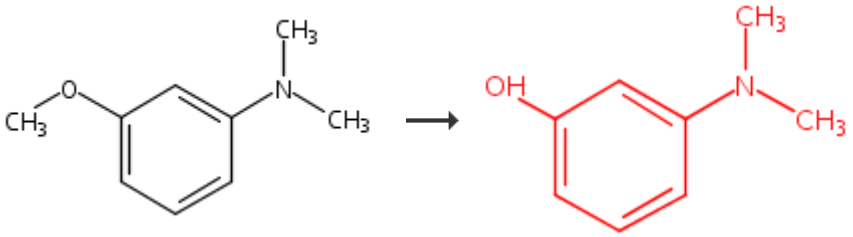
References

Oxyaniliniums as acetylcholinesterase inhibitors for the reversal of neuromuscular block

By Grove, Simon J. A. et al

From Bioorganic & Medicinal Chemistry Letters, 12(2), 193-196; 2002

5. Single Step



95%

Overview

Steps/Stages

Notes

1.1 R:$Bu_4N^+ \cdot I^-$, S:CH_2Cl_2

1.2 R:BCl_3, S:CH_2Cl_2

1.3 R:H_2O

Reactants: 1, Reagents: 3, Solvents: 1, Steps: 1, Stages: 3, Most stages in any one step: 3

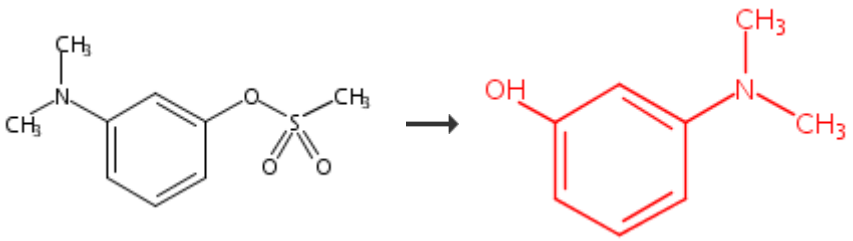
References

Boron trichloride/tetra-n-butylammonium iodide: a mild, selective combination reagent for the cleavage of primary alkyl aryl ethers

By Brooks, Paige R. et al

From Journal of Organic Chemistry, 64(26), 9719-9721; 1999

6. Single Step



89%

Overview

Steps/Stages

Notes

1.1 R:LiN(Pr-*i*)$_2$, S:THF

Reactants: 1, Reagents: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

I. Cholesterol-Absorption Inhibitors II. Toward the Synthesis of (+)-Resiniferatoxin via Photorearrangement of Cross-Conjugated Cyclohexadienones

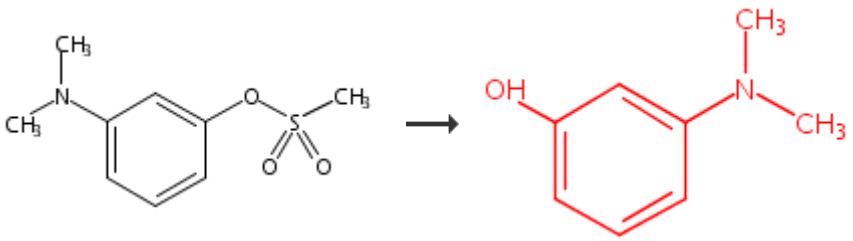
By Ritter, Tobias

From null, , No pp.; 2004

Experimental Procedure

3-Dimethylamino-phenol (179): To a solution of methanesulfonic acid 3-dimethylamino-phenyl ester (432 mg, 2.00 mmol, 1.00 equiv.) in THF (2.0 mL) at 0 degC was added a freshly prepared solution of LDA in THF (3.20 mmol, 1.60 equiv.). After 1 min NH4Cl (0.5 g) was added and the suspension was concentrated in vacuo. To the residue was added CH2Cl2 and water. The phases were separated and the aqueous phase was extracted with CH2Cl2. The combined organic phases were washed with brine, dried (Na2SO4) and concentrated in vacuo. The residue was purified by chromatography on silica gel eluting with hexane/ethyl acetate (2:1) to afford the title compound in 89 % yield.

7. Single Step



89%

Overview

Steps/Stages

1.1 R:LiN(Pr-*i*)$_2$, S:THF, S:Me(CH$_2$)$_4$Me, 1 min, 0°C

1.2 R:NH$_4$Cl

Notes

Reactants: 1, Reagents: 2, Solvents: 2, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Mild cleavage of aryl mesylates. Methanesulfonate as potent protecting group for phenols

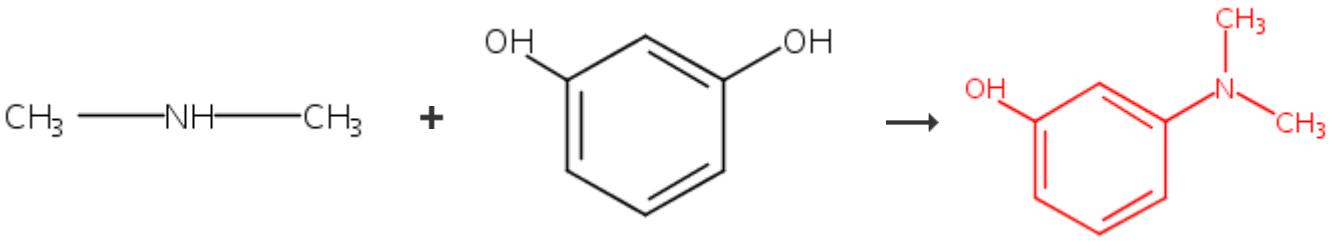
By Ritter, Tobias et al

From Organic Letters, 6(9), 1513-1514; 2004

Experimental Procedure

General/Typical Procedure: General Procedure for the Deprotection of Arylmethanesulfonates. To a solution of the methanesulfonate aryl ester (1.00 equiv) in THF (conc = 0.1 M-1.0 M) at -78 °C to 23 °C is added a freshly prepared solution of LDA (1.60-1.80 equiv per mesylate group) in THF. After 1-30 min 5% aqueous HCl or aqueous NH$_4$Cl is added followed by ethyl acetate. The phases are separated and the aqueous phase is extracted with ethyl acetate or CH$_2$Cl$_2$. The combined organic phases are washed with brine, dried (Na$_2$SO$_4$) and concentrated in vacuo. The residue is purified by chromatography on silica gel or recrystallized to afford the pure phenols. 89% yield. ^{1}H-NMR (300 MHz, CDCl$_3$): δ 7.13-7.07 (m, 1 H), 6.37-6.32 (m, 1 H), 6.24-6.18 (m, 2H), 4.94 (s, 1 H), 2.92 (s, 6H).

8. Single Step



84%

Overview

Steps/Stages

1.1

Notes

Reactants: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

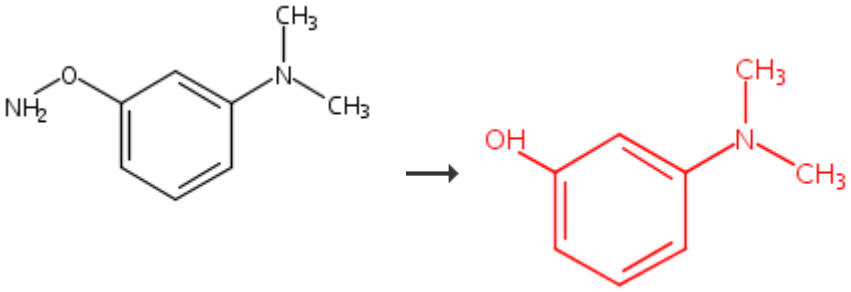
References

Preparation of N,N-dialkylaminophenols from dihydroxybenzene

By Kuwabara, Masahiro et al

From Jpn. Kokai Tokkyo Koho, 05085994, 06 Apr 1993

9. Single Step



- HCl

Overview

Steps/Stages

1.1 R:AcONa, S:D_2O, 1 h, rt

Notes

46% conversion of starting material, photochemical, Reactants: 1, Reagents: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

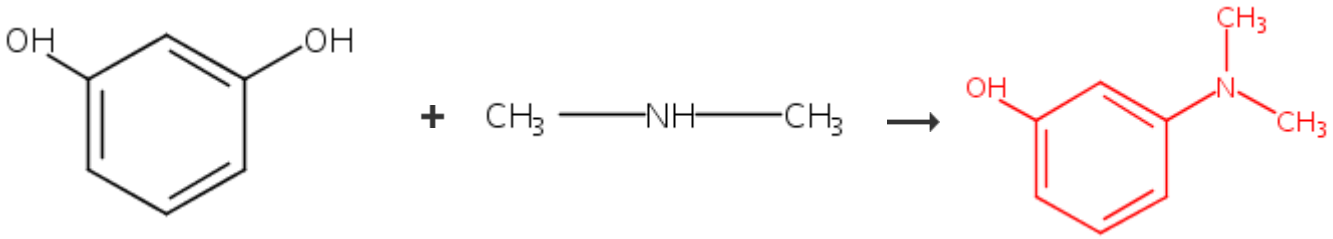
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

10. Single Step



Overview

Steps/Stages

1.1 C:16652-03-2, rt → 120°C, 1.6 MPa
1.2 < 160°C; 160°C → 200°C; 3 h, 200°C; cooled

Notes

autoclave used, alternative reaction conditions shown, high pressure, Reactants: 2, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

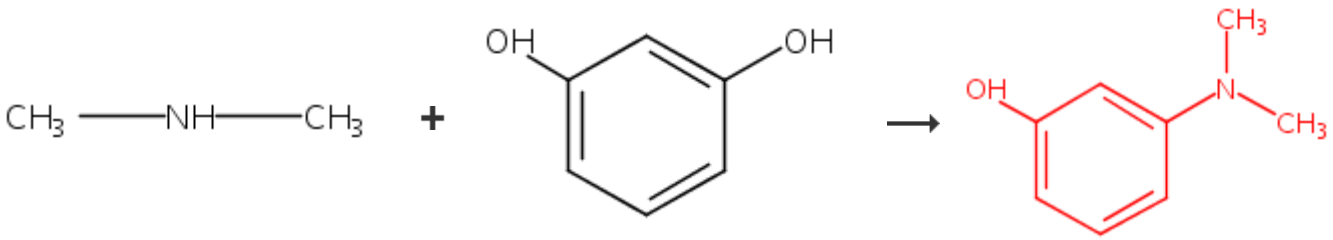
References

Method for preparing m-dimethylaminophenol from m-resorcinol

By Pan, Yi

From Faming Zhuanli Shenqing, 102924304, 13 Feb 2013

11. Single Step



Overview

Steps/Stages

1.1 S:H_2O, rt → 175°C; 175°C → 30°C

1.2 R:HCl, S:H_2O, pH 6-7

Notes

other reaction conditions also tried, Reactants: 2, Reagents: 1, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

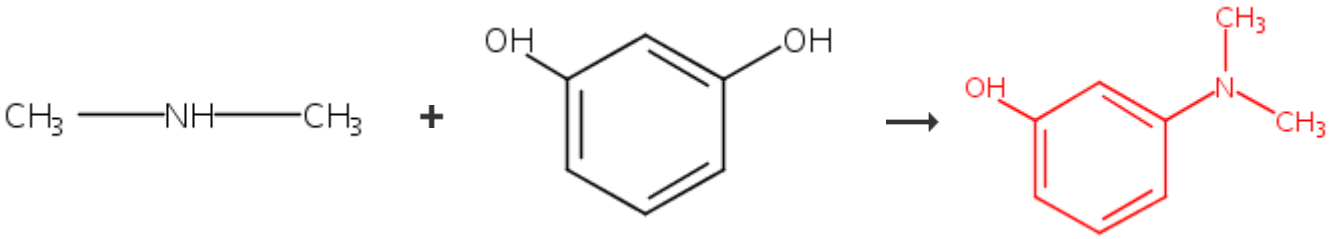
References

Preparation of 3-(N,N-dimethylamino)phenol

By He, Chengxiang et al

From Faming Zhuanli Shenqing, 102381993, 21 Mar 2012

12. Single Step



61%

Overview

Steps/Stages

1.1 R:H_2O, R:H_3PO_4, S:PhOH

Notes

Reactants: 2, Reagents: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

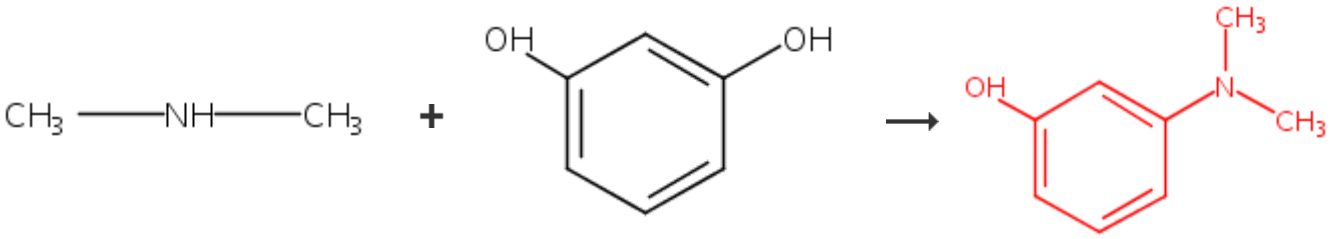
References

Preparation of N-substituted aminophenols

By Kondo, Masahiro and Hirowatari, Noriyuki

From Jpn. Kokai Tokkyo Koho, 03099042, 24 Apr 1991

13. Single Step



Overview

Steps/Stages

1.1

Notes

Go to Science of Synthesis, a critically reviewed reference work of synthetic methodology, for more information., Reactants: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

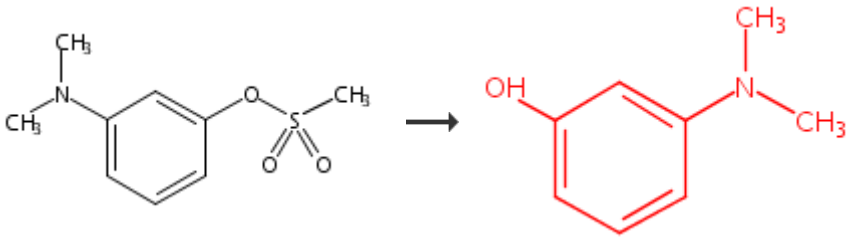
References

Arylamines

By Scholz, U. and Schlummer, B.

From Science of Synthesis, 31b, 1565-1678; 2007

14. Single Step



Overview

Steps/Stages

1.1

Notes

Go to Science of Synthesis, a critically reviewed reference work of synthetic methodology, for more information., Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

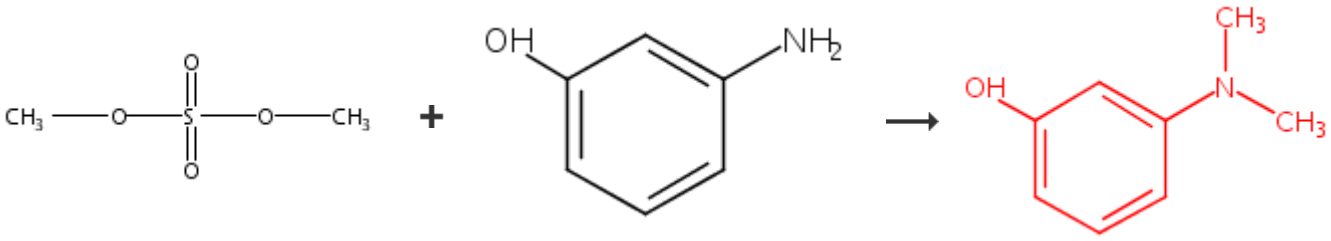
References

Product class: monohydric phenols and corresponding phenolates - synthesis by elimination

By Gonzalez-Bello, C. and Castedo, L.

From Science of Synthesis, 31a, 305-318; 2007

15. Single Step



51%

Overview

Steps/Stages

1.1 S:Benzene

Notes

Reactants: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

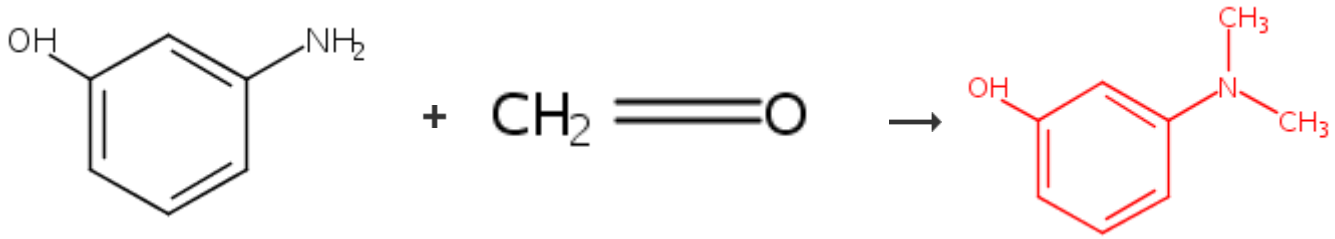
References

Synthesis of edrophonium chloride

By Lu, Jin-Rong and Mo, Li-Rong

From Zhongguo Yiyao Gongye Zazhi, 31(6), 243-244; 2000

16. Single Step



21%

Overview

Steps/Stages

1.1 R:H_2, C:Ni, S:MeOH, rt → 180°C; 8 h, 180°C, 1.7 MPa

Notes

thermal, high pressure, autoclave used, Raney nickel used, paraformaldehyde used, reusable catalyst, Reactants: 2, Reagents: 1, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

RANEY nickel-catalyzed reductive N-methylation of amines with paraformaldehyde: theoretical and experimental study

By Ge, Xin et al

From RSC Advances, 4(81), 43195-43203; 2014

Reaction Protocol

Procedure

1. Add paraformaldehyde (350 mmol), aromatic amine (152 mmol) and RANEY® Ni (445 mg) to methanol (100 ml) in a 250 ml autoclave.
2. Purge the autoclave with nitrogen gas three times, followed by hydrogen gas three times.

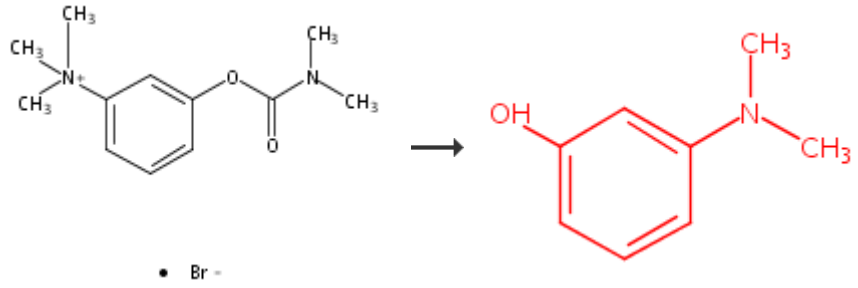
View more...

Available Experimental Data

^{1}H NMR, ^{13}C NMR, Mass Spec

View with MethodsNow

17. Single Step



Overview

Steps/Stages

1.1

Notes

Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

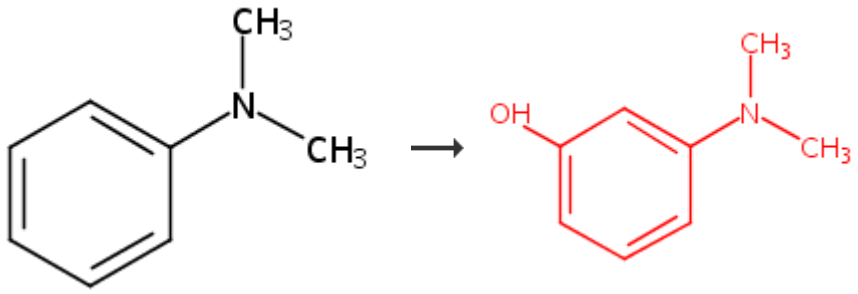
References

Structure of degradation products of neostigmine bromide

By Porst, Hella and Kny, L.

From Pharmazie, 40(5), 325-8; 1985

18. Single Step



44%

Overview

Steps/Stages

1.1 R:H_2O_2, R:SbF_5, R:HF

Notes

Reactants: 1, Reagents: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

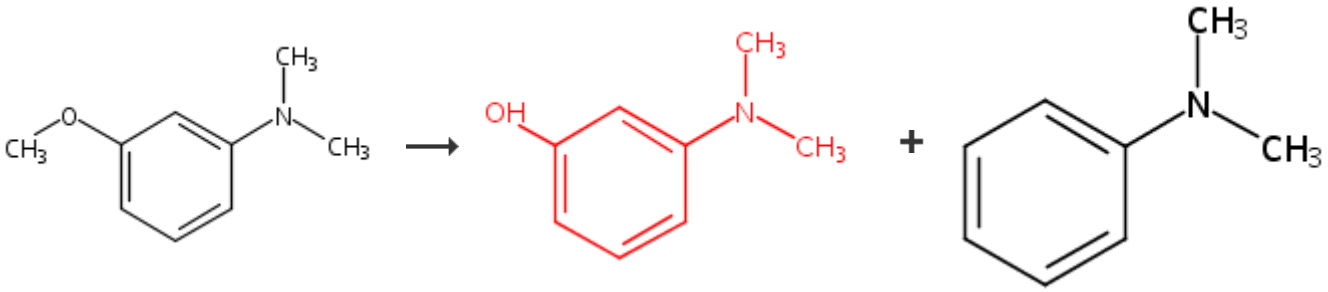
References

Direct conversion of anilines into aminophenols

By Jacquesy, Jean Claude et al

From Tetrahedron Letters, 25(14), 1479-82; 1984

19. Single Step



Overview

Steps/Stages

1.1 R:K, C:Naphthalene, S:*i*-BuCMe$_3$, S:THF, 24 h, rt; rt → 0°C

1.2 R:H_2O, 0°C

Notes

Reactants: 1, Reagents: 2, Catalysts: 1, Solvents: 2, Steps: 1, Stages: 2, Most stages in any one step: 2

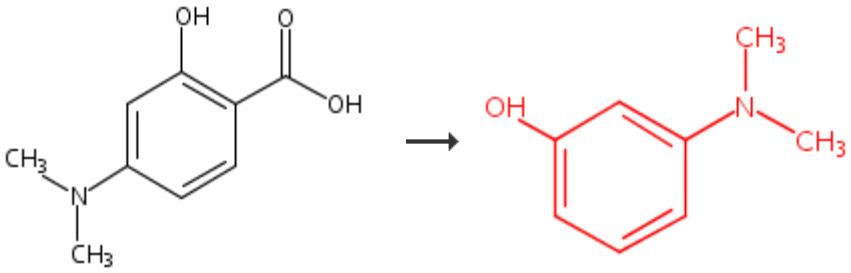
References

Electron-transfer-induced reductive dealkoxylation of alkyl arylethers. III. Reductive cleavage of methoxy-substituted N,N-dimethylanilines (N,N-dimethylanisidines)

By Azzena, Ugo et al

From ARKIVOC (Gainesville, FL, United States) [online computer file], (5), 181-188; 2002

20. Single Step



Overview

Steps/Stages

Notes

1.1 S:H_2O

Classification: Decarboxylation; # Conditions: pH<3; NaOH H2O, Reactants: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

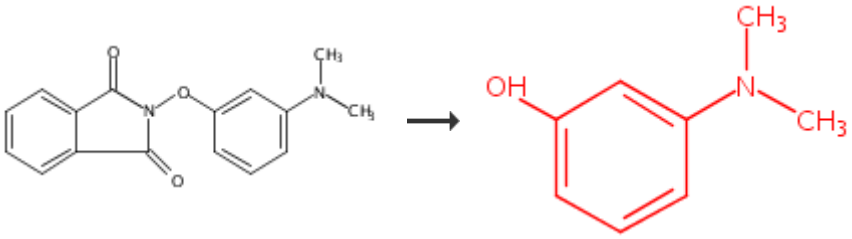
References

Ultraviolet absorption spectra of p-aminosalicylic acid and some related compounds. IV. Ampholytic forms of p-aminosalicyclic acid and some structurally related compounds and their decarboxylation

By Rekker, R. F. and Nauta, W. Th.

From Journal of Medicinal & Pharmaceutical Chemistry, 2, 281-97; 1960

21. 2 Steps



Overview

Steps/Stages

1.1 R:N_2H_4-H_2O, S:MeOH, S:CH_2Cl_2, 12 h, rt

1.2 R:HCl, S:Et_2O, rt

2.1 R:AcONa, S:D_2O, 1 h, rt

Notes

2) 46% conversion of starting material, photochemical, Reactants: 1, Reagents: 3, Solvents: 4, Steps: 2, Stages: 3, Most stages in any one step: 2

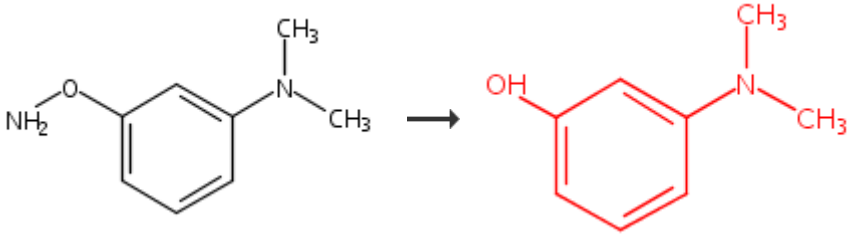
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

22. 2 Steps



Overview

Steps/Stages

1.1 R:HCl, S:Et_2O, rt

2.1 R:AcONa, S:D_2O, 1 h, rt

Notes

2) 46% conversion of starting material, photochemical, Reactants: 1, Reagents: 2, Solvents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

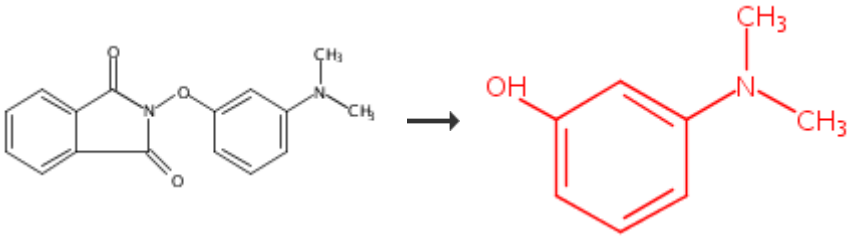
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

23. 3 Steps



Overview

Steps/Stages

1.1 R:N_2H_4-H_2O, S:MeOH, S:CH_2Cl_2, 12 h, rt

2.1 R:HCl, S:Et_2O, rt

3.1 R:AcONa, S:D_2O, 1 h, rt

Notes

3) 46% conversion of starting material, photochemical, Reactants: 1, Reagents: 3, Solvents: 4, Steps: 3, Stages: 3, Most stages in any one step: 1

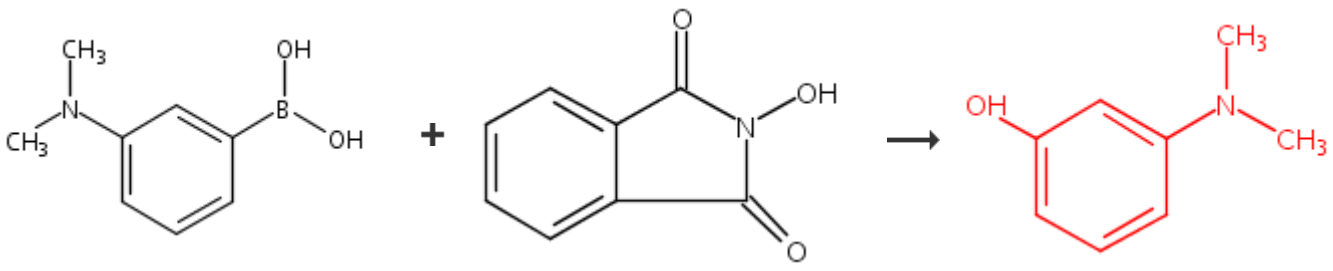
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

24. 3 Steps



Overview

Steps/Stages

1.1 R:C_5H_5N, R:CuCl, S:$ClCH_2CH_2Cl$, 72 h, rt

2.1 R:N_2H_4-H_2O, S:MeOH, S:CH_2Cl_2, 12 h, rt

2.2 R:HCl, S:Et_2O, rt

3.1 R:AcONa, S:D_2O, 1 h, rt

Notes

1) molecular sieves used, 3) 46% conversion of starting material, photochemical, Reactants: 2, Reagents: 5, Solvents: 5, Steps: 3, Stages: 4, Most stages in any one step: 2

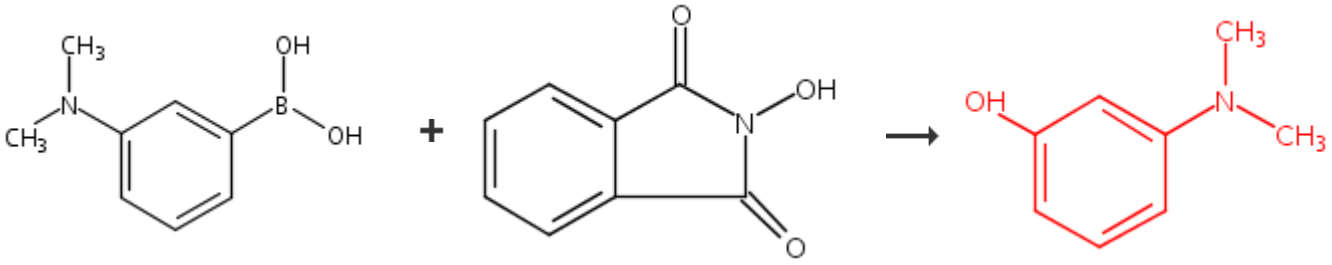
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

25. 4 Steps



Overview

Steps/Stages

1.1 R:C_5H_5N, R:CuCl, S:$ClCH_2CH_2Cl$, 72 h, rt

2.1 R:N_2H_4-H_2O, S:MeOH, S:CH_2Cl_2, 12 h, rt

3.1 R:HCl, S:Et_2O, rt

4.1 R:AcONa, S:D_2O, 1 h, rt

Notes

1) molecular sieves used, 4) 46% conversion of starting material, photochemical, Reactants: 2, Reagents: 5, Solvents: 5, Steps: 4, Stages: 4, Most stages in any one step: 1

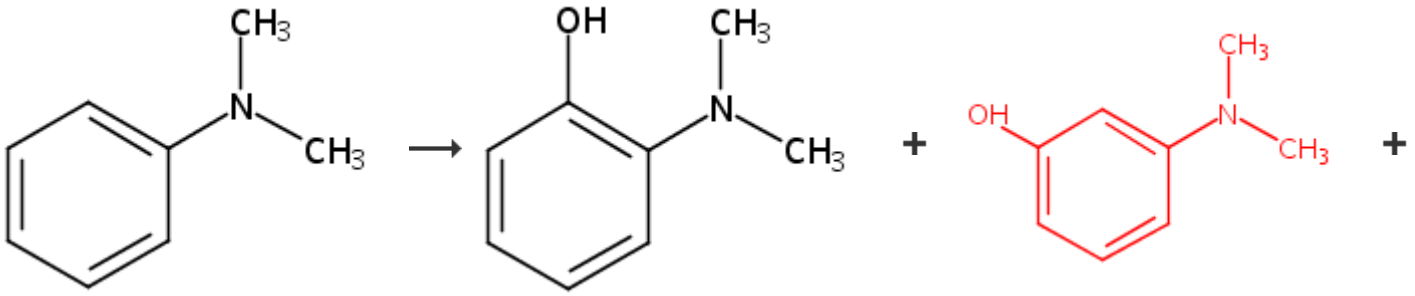
References

Direct spectroscopic detection and EPR investigation of a ground state triplet phenyl oxenium ion

By Li, Ming-De et al

From Journal of the American Chemical Society, 137(32), 10391-10398; 2015

26. Single Step



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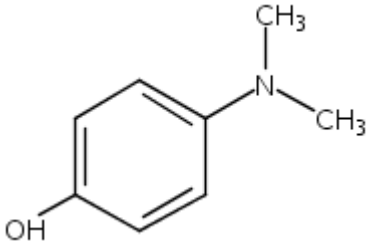


26%

Overview

Steps/Stages

1.1 R:HF, R:SbF_5, R:H_2O_2, S:H_2O

Notes

Reactants: 1, Reagents: 3, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

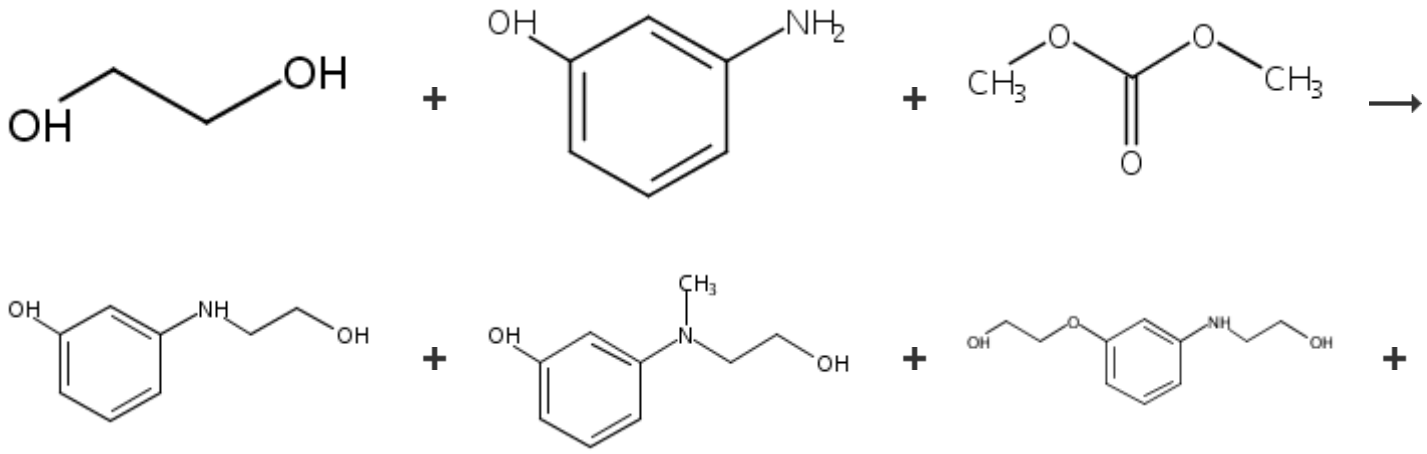
References

Direct hydroxylation of anilines and aminophenols

By Jacquesy, Jean Claude et al

From Bulletin de la Societe Chimique de France, (4), 625-9; 1986

27. Single Step



36%

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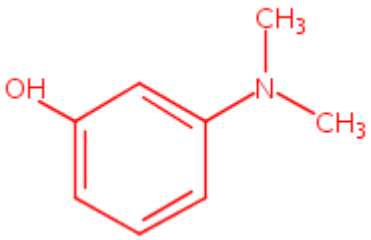
16%



9%

Overview

Steps/Stages

1.1 S:$(CH_2OH)_2$, rt → 443K, 34 bar; 2 h, 443K, 34 bar; 443K → rt

Notes

chemoselective, Zeolite Na-Y used as catalyst, autoclave used, other product also detected, selective N-alkylation via transesterification, zeolite activated prior to use (air,773K,6 h), high pressure, Reactants: 3, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Tandem Synthesis of β-Amino Alcohols from Aniline, Dialkyl Carbonate, and Ethylene Glycol

By Shivarkar, Anandkumar B. et al

From Industrial & Engineering Chemistry Research, 47(8), 2484-2494; 2008

Experimental Procedure

General/Typical Procedure: **2.1. General Experimental Procedure for High-Pressure Reaction.** High-pressure reactions were carried out in a parr autoclave (50 mL, SS-316). In a typical experiment, aniline (10.7 mmol), dimethyl carbonate (33 mmol), ethylene glycol (286 mmol), and the solid catalyst (0.25 g) were charged into the reactor. The reactor was flushed with nitrogen and then pressurized with nitrogen up to 3.4 MPa. Then the contents were heated up to 443 K under well mixed conditions and the progress of the reaction was monitored by withdrawing the intermediate samples which were quantitatively analyzed by GC for reactants and products. The reaction was continued for specified time, the contents cooled to room temperature and gas vented off. Liquid phase analysis was carried out by GC using HP-1 capillary column (30 m length × 0.32 mm i.d. × 0.25μm film thickness). **2-[(3-Hydroxyphenyl)amino]ethanol (5g).** IR (film): ν_{max} = 3332 (OH, NH), 2947 (CH), 1606 (C=C, Ar), 1496 (C=C, Ar), 1338 (C-N, Ar), 1055, 767 cm^{-1}. ^1H NMR (200 MHz, acetone-d_6): δ = 2.96 (brs, 2H; NH and OH); 3.19 (t, *J* = 5.6 Hz, 2H; -NCH_2-); 3.72 (t, *J* = 5.6 Hz, 2H; -OCH_2-); 4.77 (brs, 1H; OH,Ar); 6.08-6.17 (m, 3H; -CH, Ar); 6.90 (dd, *J* = 7.8 and 8.5 Hz, 1H; -CH, Ar) ^{13}C NMR (100 MHz, acetone-d_6): δ = 46.75 (-NCH_2); 61.20 (-OCH_2); 100.33 (-CH, Ar); 104.64 (-CH, Ar); 105.37 (-CH, Ar); 130.43 (-CH, Ar); 151.33 (-C, Ar); 159.17 (-C, Ar). GC-MS (70 eV, EI) *m/z* (%): 153 (34) $[M]^+$, 122 (100) [*m*-OH-$C_6H_4NH{=}CH_2]^+$, 109 (3), 94 (13), 77 (5), 65 (9), 53 (2).

Reaction Protocol

Procedure

1. Carry out high-pressure reactions in a parr autoclave (50 mL, SS-316).
2. Add aniline (10.7 mmol), dimethyl carbonate (33 mmol), ethylene glycol (286 mmol) and the solid catalyst (0.25 g) into the reactor.

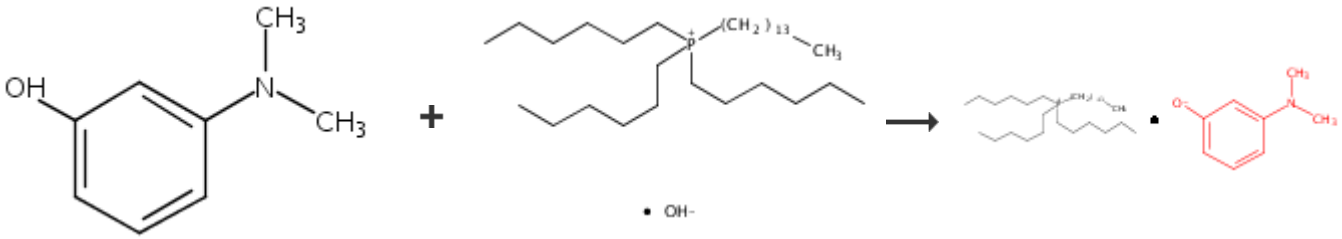
View more...

Available Experimental Data

^1H NMR, ^{13}C NMR, IR, Mass Spec

View with MethodsNow

28. Single Step



Overview

Steps/Stages

1.1 S:EtOH, 16 h, rt

Notes

green chemistry, ionic liquid formed, mechanism studied, Reactants: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

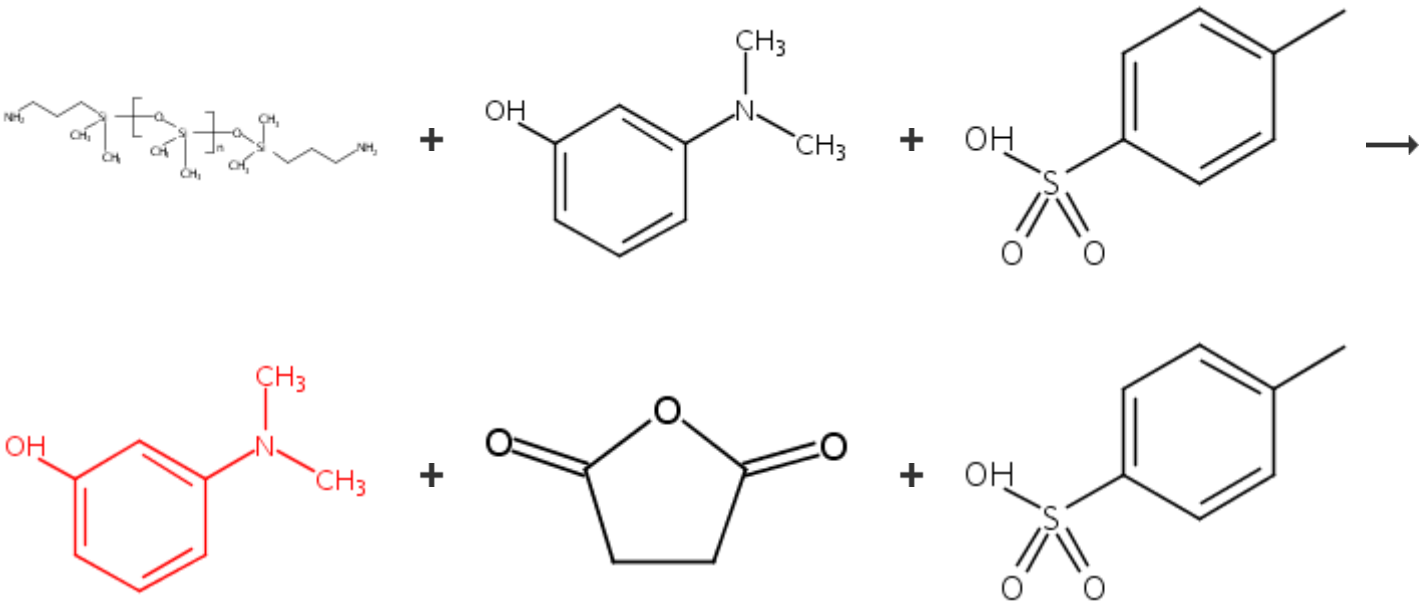
References

Tuning the Physicochemical Properties of Diverse Phenolic Ionic Liquids for Equimolar CO2 Capture by the Substituent on the Anion

By Wang, Congmin et al

From Chemistry - A European Journal, 18(7), 2153-2160, S2153/1-S2153/20; 2012

29. Single Step



reaction products with succinic anhydride-

polysiloxane derivs., reaction products wi

salts with adducts of succinic anhydride-p

Overview

Steps/Stages

1.1 1 h, 90°C; 90°C → 150°C
1.2 1 h, 150°C → 90°C; 90°C → rt

Notes

Reactants: 3, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Colored polysiloxanes

By Banning, Jeffery H. et al

From U.S. Pat. Appl. Publ., 20130150571, 13 Jun 2013

Experimental Procedure

To a 500 mL 24/40 3-necked round-bottom flask with TEFLON-coated stir magnet and condenser is added about 45.25 g 3-(dimethylamino)phenol (available from Aidrich Chemical Co., Milwaukee, Wis.) and about 100 g 75-100 centastoke succinic anhydride-terminated polydimethylsul foxide (Mw about 800 g/mole; available as DMS-Al 1 from Gelest, Inc., Morrisve, Pa.). The flask is placed in a 90°C. oil bath and allowed to stir to dissolve all reactants. After 1 h the mixture is allowed to heat to 150°C. until a magenta color is formed and consistent throughout. Then about 21.5 g of p-toluenesulfonic acid (Aldrich Chemical Co.) is added and the reaction is allowed to cool to 90°C. and held for 1 h. The reaction mixture is cooled to room temperature and poured into a jar. . Final product.