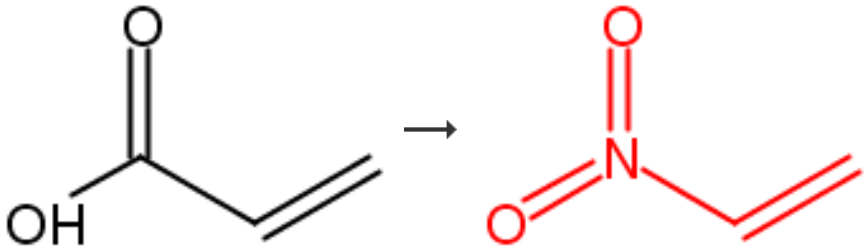
1. Single Step



92%

Overview

Steps/Stages

1.1 C:15025-74-8, S:$HOCH_2CH_2OH$ polymer, S:$ClCH_2CH_2Cl$, S:MeCN, 1.5 h, reflux; cooled

1.2 R:Disodium carbonate, S:H_2O

Notes

optimization study, PEG-300 used as solvent, optimized on solvent (PEG), Hunsdiecker reaction, Reactants: 1, Reagents: 1, Catalysts: 1, Solvents: 4, Steps: 1, Stages: 2, Most stages in any one step: 2

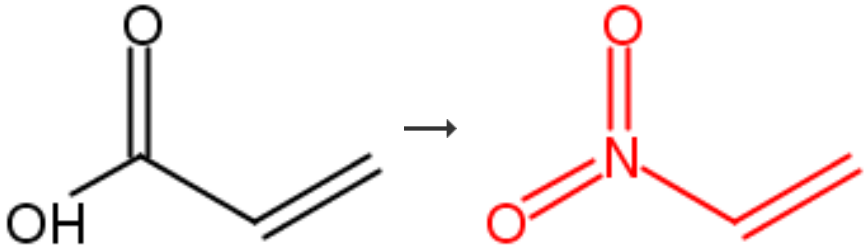
References

Polyethylene glycol mediated kinetic study of nitro decarboxylation of α,β-unsaturated acids by Blau's Fe(III) Phen complex

By Ramesh, K. et al

From Journal of Chemistry, , 703271/1-703271/11, 11 pp.; 2013

2. Single Step



92%

Overview

Steps/Stages

Notes

1.1 R:$Fe(NO_3)_3$, R:2,2'-$(C_5H_4N)_2$, S:MeCN, rt
1.2 S:$HOCH_2CH_2OH$ polymer, 1.5 h, rt

1.3 R:Disodium carbonate, S:H_2O, rt

green chemistry-process simplification, in-situ generated reagent [(Fe(III)-Bipy) complex] formed in stage 1, see experimental details, kinetics studied, mechanism studied, PEG 300 used as solvent in stage 2, optimization study in stage 2, optimized on type of PEG, green chemistry-solvent (PEG), Nitro Hunsdiecker reaction, alternatively reaction carried out in absence of PEG in stage 2 decreased yield and increased reaction time, solvent recyclable (PEG), Reactants: 1, Reagents: 3, Solvents: 3, Steps: 1, Stages: 3, Most stages in any one step: 3

References

Polyethylene Glycol-Mediated Kinetic Study of Nitrodecarboxylation of α,β-Unsaturated Acids by Blau Fe(III) Bipy Complex

By Ramesh, K. et al

From International Journal of Chemical Kinetics, 46(2), 126-137; 2014

Reaction Protocol

Procedure

1. Suspend the AA (0.001 mol) dissolved in MeCN and 0.001 moles of Blau's[Fe (III)-Bipy] complex in a three-necked round-bottomed flask, equiped with a mechanical stirrer and condenser.
2. Reflux the reaction mixture for 1.5 hours at room temperature until completion of the reaction.

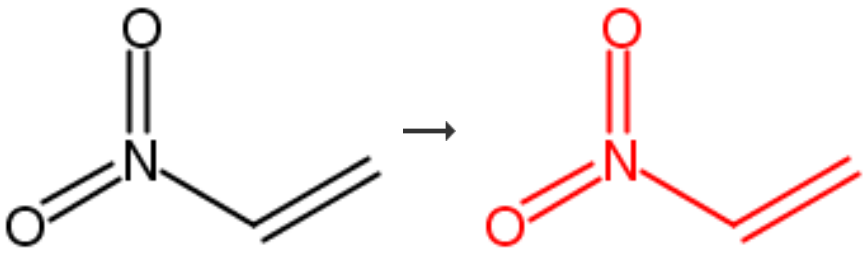
View more...

Available Experimental Data

^{1}H NMR, Mass Spec

View with MethodsNow

3. Single Step



95%

Overview

Steps/Stages **Notes**

1.1 R:NaOEt, S:EtOH, 14 h, rt

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

References

Silicon-catalyzed conversion of nitro compounds into ketones and poly(1,3-diketones)

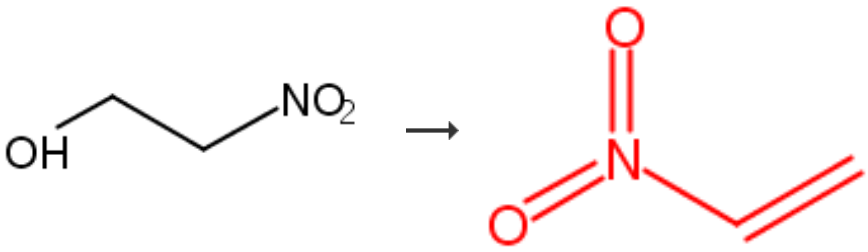
By Hwu, Jih Ru et al

From Synthesis, (19), 3305-3308; 2006

Experimental Procedure

Poly(nitroethylene) (6)[15b]. To a stirred soln of nitroethene (**4**; 5.03 g, 68.9 mmol, 1.0 equiv) in EtOH (690 mL) was added NaOEt (234 mg, 3.44 mmol, 0.050 equiv). The mixture was stirred at r.t. for 14 h, and after this time a white solid was obtained. The solid was purified by washing with H_2O (1 ×), MeOH (2 ×), Et_2O (2 ×) and then dried under reduced pressure over P_2O_5 to give polymer **6**. Yield 4.78 g, 95%. soluble in DMSO, THF, and DMF; MW 7.12 × 104. IR (KBr): 3000 (m), 2907 (m), 1559 (br s, N-O), 1436 (m), 1363 (br m, N-O), 849 (s, =CH) cm^{-1}. ^1H NMR (80 MHz, $CDCl_3$): δ = 2.49-2.65 (br d, 2 H, CH_2), 4.59- 4.75 (br m, 1 H, $CHNO_2$). Anal. Calcd for $(C_2H_3NO_2)_n$: C, 32.88; H, 4.14; N, 19.17. Found: C, 33.01; H, 4.15; N, 19.22.

4. Single Step



90%

Overview

Steps/Stages

1.1 R:Phthalic anhydride, 110-140°C, 60 mmHg

Notes

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

References

Aminoethylation of aldehydes in synthesis of γ-amino acids

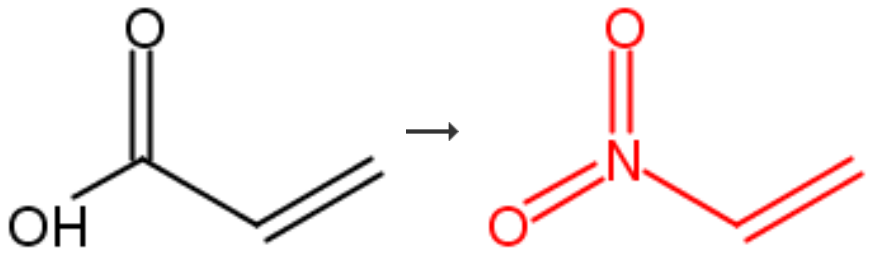
By Gellman, Samuel Helmer et al

From U.S. Pat. Appl. Publ., 20090264676, 22 Oct 2009

Experimental Procedure

Nitroethylene This compound was prepared using a modification of a literature procedure (see Kunetsky et al., *Tetrahedron Lett.* 2005, 46, 5203; Ranganathan et al, *Tetrahedron Lett.* 1987, 28, 2893). 2-Nitroethanol (100 g) and phthalic anhydride (210 g) were mixed in a 500 ml round bottom flask equipped with a magnetic stir bar. The flask was then equipped with a vacuum distillation setup with a fractional distillation column and a -78°C. bath-cooled receiver. The apparatus was evacuated to about 60 mm Hg, and the oil bath was heated to and maintained at 110-140°C. The starting materials turned to a homogeneous solution (solid material may exist depending on the temperature), and the distillate was collected until the distillation ceased to give a pale yellow solid at -78°C. The solid, containing a mixture of nitroethylene and water (~90 g, 90% yield), was warmed in an icewater bath to give a pale yellow heterogeneous mixture. The mixture was mixed with toluene, dried over anhydrous $CaCl_2$ (anhydrous), and filtered through a pad of anhydrous $CaCl_2$. The filtrate was collected as a pale yellow stock solution of nitroethylene in toluene, and stored at -10°C. for future use. The concentration of nitroethylene of the stock solution can be estimated via ^1H NMR analysis in benzene-d6 (with toluene as internal standard). Concentration estimated from the ^1H NMR analysis agreed with that calculated from mass of the crude nitroethylene product (the impurity is water) and toluene used in preparation of the stock solution. Nitroethylene as a solution in dry benzene was found to be stable (no change in NMR) for at least 6 months when stored in a refrigerator. Nitroethylene, yield (~90 g, 90% yield). ^1H NMR of stock solution in toulene (300 MHz, $CDCl_3$) δ 7.11 (dd, J=7.2,15 Hz), 6.62 (dd, J=14.7,2.1 Hz), 5.86 (br d, J=7 Hz); ^{13}C NMR of stock solution in toluene (75 MHz, $CDCl_3$) δ 145.56, 122.36.

5. Single Step

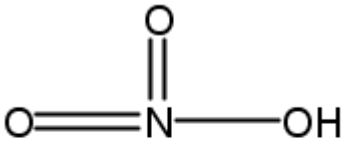


88%

Overview

Steps/Stages

1.1 R:



• 1/3 Al

C:$HOCH_2CH_2OH$ polymer, S:MeCN, 1.5 h

Notes

regioselective, optimized on metal nitrate,type of PEG and method, PEG-600 used as catalyst, Hunsdiecker-Borodin reaction, green chem.-catalyst, optimization study, Reactants: 1, Reagents: 1, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

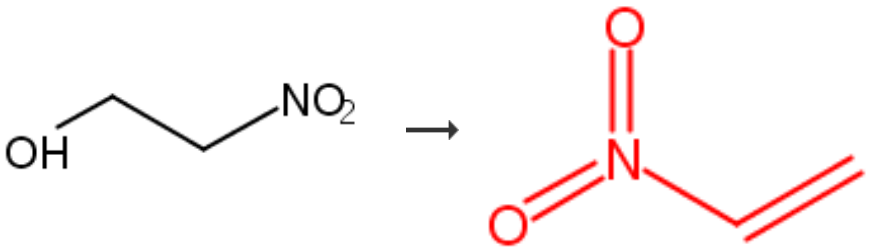
References

Poly ethylene glycols as efficient media for the synthesis of β-nitro styrenes from α, β-unsaturated carboxylic acids and metal nitrates under conventional and non-conventional conditions

By Rajanna, Kamatala Chinna et al

From Green and Sustainable Chemistry, 1(4), 132-148; 2011

6. Single Step



90%

Overview

Steps/Stages

1.1 R:Phthalic anhydride, rt → 110°C; 110-140°C

Notes

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

References

Enantioselective organocatalytic Michael addition of aldehydes to nitroethylene: efficient access to γ2-amino acids

By Chi, Yonggui et al

From Journal of the American Chemical Society, 130(17), 5608-5609; 2008

Experimental Procedure

Nitroethylene: This compound was prepared using a modification of a literature procedure. 7, 8 2-Nitroethanol (100 g) and phthalic anhydride (210 g) were mixed in a 500 ml round bottom flask equipped with a magnetic stir bar. The flask was then equipped with a vacuum distillation setup with a fractional distillation column and a -78 0 C bath-cooled receiver. The apparatus was evacuated to about 60 mmHg, and the oil bath was heated to and maintained at 110-140 °C. The starting materials turned to a homogeneous solution (solid material may exist depending on the temperature), and the distillate was collected until the distillation ceased to give a pale yellow solid at -78 °C. The solid, containing a mixture of nitroethylene and water (~90g, 90% yield), was warmed in an ice-water bath to give a pale yellow heterogeneous mixture. The mixture was mixed with toluene, dried over anhydrous $CaCl_2$ (anhydrous), and filtered through a pad of anhydrous $CaCl_2$. The filtrate was collected as a pale yellow stock solution of nitroethylene in toluene, and stored at -10 °C for future use. The concentration of nitroethylene of the stock solution can be estimated via ^{1}H NMR analysis in benzene-d_6 (with toluene as internal standard). Concentration estimated from the ^{1}H NMR analysis agreed with that calculated from mass of the crude nitroethylene product (the impurity is water) and toluene used in preparation of the stock solution. Nitroethylene as a solution in dry benzene was found to be stable (no change in NMR) for at least 6 months when stored in a refrigerator. **Nitroethylene:**Yield (~90g, 90%). ^{1}H NMR of stock solution in toulene (300 MHz, $CDCl_3$) δ 7.11 (dd, *J* = 7.2, 15 Hz), 6.62 (dd, *J* = 14.7, 2.1 Hz), 5.86 (br d, *J* = 7 Hz); ^{13}C NMR of stock solution in toluene (75 MHz, $CDCl_3$) δ 145.56, 122.36.

Reaction Protocol

Procedure

1. Mix 7, 8 2-nitroethanol (100 g) and phthalic anhydride (210 g) in a 500 ml round bottomed flask equipped with a magnetic stir bar.
2. Equip the flask with a vacuum distillation setup with a fractional distillation column.

View more...

Available Experimental Data

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

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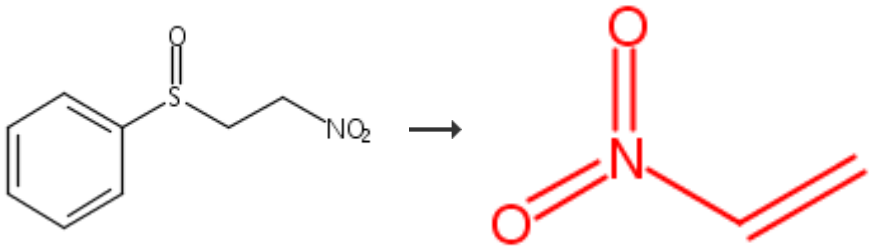
7. Single Step



100%

Overview

Steps/Stages

1.1 S:Benzene

Notes

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

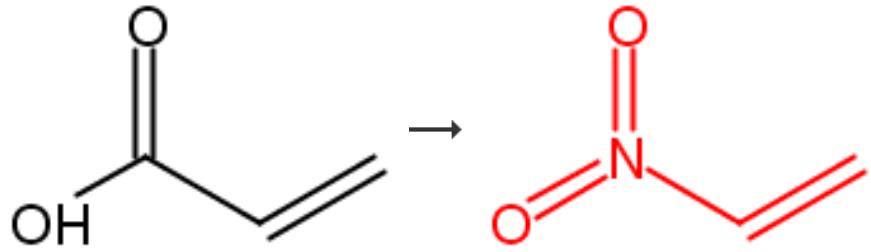
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

8. Single Step



80%

Overview

Steps/Stages

Notes

1.1 R:*t*-BuONO, S:MeCN, 4-5 min, 100°C, 3 bar

microwave irradiation, silica gel used, alternative conditions (conventional, sonication) gave lower yield, small amount of solvent used (solvent-free conditions-author emphasis), Reactants: 1, Reagents: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Ultrasonic and microwave-assisted synthesis of β-nitro styrenes and nitro phenols with tertiary butyl nitrite under acid-free conditions

By Kumar, M. Satish et al

From Synthetic Communications, 43(19), 2672-2677; 2013

Experimental Procedure

General/Typical Procedure: **General Procedure for Microwave-Assisted Synthesis of Nitro Compounds Under Solvent-Free Conditions** Organic substrate (cinnamic acids, phenols), TBN and solvent (small amount of acetonitrile) were mixed with silica gel and heated in a controlled microwave synthesizer (Biotage Initiator+SP Wave model 0.200W at 2.45 GHz, capped at 60W during steady state) for 4-5 min (attainted temperature of 100 °C and 3 bar pressure). Progress of the reaction was monitored by TLC. After completion, the reaction mixture was further processed for the isolation of product as described previously. ***1-Nitro Ethene***, yield 80%. Mp 98-102 °C (98.5 °C) ^{1}HNMR (300MHz, $CDCl_3$): δ 5.92 (dd, 1H, Hb, J=11.3 Hz, J=1.5Hz); 6.65 (dd, 1H, Ha, J=15.5 Hz, J=11.3 Hz); 7.25 (dd, 1H, Hc, J=15.5Hz, J=1.5 Hz), m/z = 73.

Reaction Protocol

Procedure

1. Mix acid derivative, tert-butyl nitrite and acetonitrile with silica gel.
2. Heat the mixture in a controlled microwave synthesizer (Biotage Initiator+SP Wave model 0.200W at 2.45 GHz, capped at 60W during steady state) for 4-5 minutes (attainted temperature of 100 °C and 3 bar pressure).

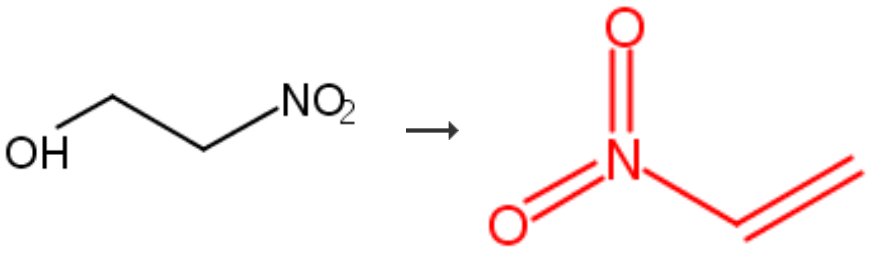
View more...

Available Experimental Data

^{1}H NMR, Mass Spec, MP

View with MethodsNow

9. Single Step



80%

Overview

Steps/Stages **Notes**

1.1 R:Phthalic anhydride, 140-150°C, 80 mmHg; 175-180°C

thermal, no solvent, low pressure, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Preparation of N-pyridinyl carboxamide derivatives as modulators of ATP-binding cassette transporters

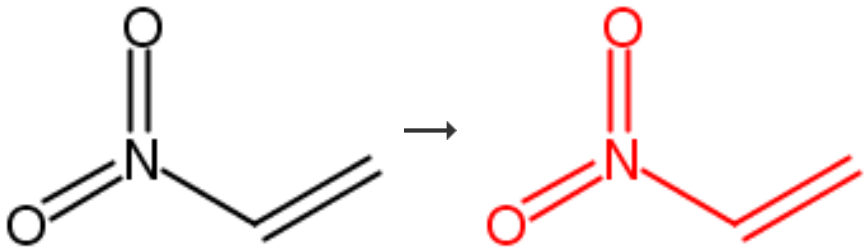
By Hadida-Ruah, Sara et al

From U.S. Pat. Appl. Publ., 20080019915, 24 Jan 2008

Experimental Procedure

Nitroethylene 2-Nitroethanol (3.5 g, 39 mmol) and sublimed phthalic anhydride (7.5 g, 58 mmol) were mixed in a distillation unit with a short fractional column and an ice-cooled receiver. The apparatus was evacuated to 80 mm of Hg, and the bath temperature was maintained at 140-150° C. until the mixture was homogeneous. The temperature was increased and held at 175-180° C. until distillation ceased. The distillate was dried over anhydrous $CaCl_2$ to give nitroethylene (2.3 g, 80%). ^1H NMR ($CDCl_3$, 400 MHz) δ 7.16-7.10 (m, 1H), 6.66-6.62 (m, 1H), 5.91-5.90 (m, 1H).

10. Single Step



oxidized

80%

Overview

Steps/Stages

1.1 R:NaOEt, S:Dioxane, 24 h, rt

1.2 R:KH, S:Dioxane, 1.5 h, 10°C

1.3 R:Me_3SiCl, 2 h, rt; 24 h, reflux

Notes

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

References

Silicon-catalyzed conversion of nitro compounds into ketones and poly(1,3-diketones)

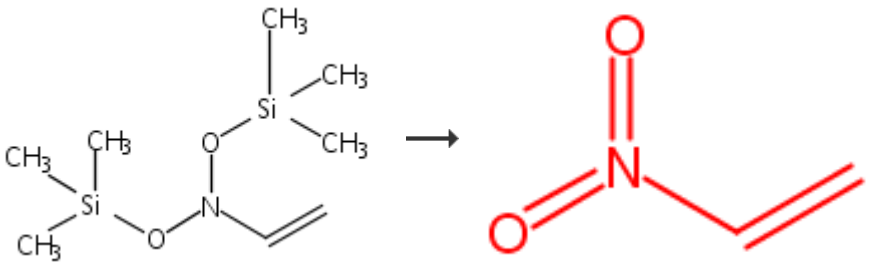
By Hwu, Jih Ru et al

From Synthesis, (19), 3305-3308; 2006

Experimental Procedure

Poly(oxoethylene) (2); Method B in One Flask. NaOEt (149 mg, 0.050 equiv) was added to a soln of nitroethene (**4**; 3.20 g, 43.8 mmol, 1.0 equiv) in 1,4-dioxane (439 mL) and the mixture was stirred at r.t. for 24 h. Then a 1,4-dioxane soln of KH (1.94 g, 1.0 equiv) was added to the mixture and stirring was continued at 10 °C for 1.5 h. Me_3SiCl (477 mg, 4.39 mmol, 0.10 equiv) was injected into the soln and this was stirred at r.t. for 2.0 h and then heated at reflux for 24 h. The cooled mixture was diluted with hexanes and a brown solid was precipitated. The solid was washed with hexanes and CCl_4 and then was dried under reduced pressure over P_2O_5 to give polymer **2.** Yield 1.47 g, 80%.

11. Single Step



82%

Overview

Steps/Stages

1.1 R:Bu_4N^+ •-OAc, R:Br_2, S:CH_2Cl_2, -78°C; 15 min, -78°C

Notes

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

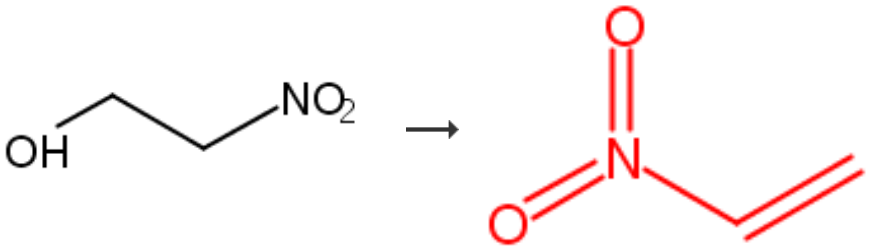
References

Novel synthesis of α-nitroalkenes from nitroalkanes via halogenation of intermediate N,N-bis(silyloxy)enamines

By Kunetsky, Roman A. et al

From Tetrahedron Letters, 46(31), 5203-5205; 2005

12. Single Step



89%

Overview

Steps/Stages

1.1 R:Phthalic anhydride

Notes

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

References

Synthesis of Tricyclic Nitrogen-Containing Systems

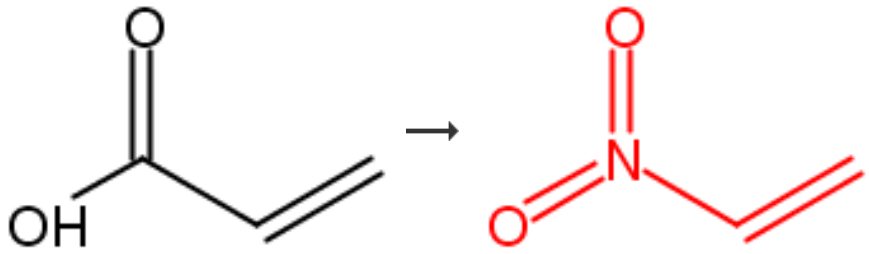
By Grohmann, Franz

From null, , No pp.; 1991

Experimental Procedure

Nitroethene (L5): 5.0 g (55 mmol) 2-nitroethanol and 10.4 g (84 mmol) of phthalic acid anhydride were heated at 100 mbar in a distillation apparatus to 140-150 degC, until the solution was clear. Then the bath temperature was increased to 180 degC, while the nitroethene was distilled collected in a receiver (cooled to -30 degC). Yield: 3.6 g of pale yellow liquid (89 % of theory). bp 30-34 degC/110 mbar.

13. Single Step



75%

Overview

Steps/Stages

1.1 R:Cl(O=)CC(=O)Cl, R:DMF, S:MeCN, -5°C

1.2 R:KNO_3, S:MeCN, 40-60 min, rt

Notes

optimization study, optimized on reagent and methods (conventional and microwave), selective nitration, in-situ generated reagent (iminium salt) (stage 1), ultrasound (stage 2), Reactants: 1, Reagents: 3, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Oxalyl chloride/DMF as an Efficient Reagent for Nitration of Aromatic Compounds and Nitro Decarboxylation of Cinnamic Acids in Presence of KNO3 or NaNO2 Under Conventional and Nonconventional Conditions

By Kumar, M. Satish et al

From Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry, 43(8), 977-983; 2013

Experimental Procedure

General/Typical Procedure: **General Procedure for Synthesis of Nitro Arenes and β-Nitro Styrenes Using $(COCl)_2$+DMF Iminium Salt (Under Sonication)** Organic substrate, KNO_3 (or $NaNO_2$), [$(COCl)_2$+DMF] iminium salt and solvent (MeCN) were taken in a clean conical flask at room temperature and immersed in a sonicator and progress of the reaction monitored by TLC. After completion, the reaction mixture is further processed for the isolation of product as detailed in earlier section. ***β-Nitro styrene 1-Nitro Ethene*** Yield 75%. δ5.92 (dd, 1H, Hb, J=11.3 Hz, J=1.5Hz); 6.65 (dd, 1H, Ha, J=15.5 Hz, J=11.3 Hz); 7.25 (dd, 1H, Hc, J=15.5Hz, J=1.5 Hz). m/z -73

Reaction Protocol

Procedure

1. Take acrylic acid, KNO_3, [$(COCl)_2$+DMF] iminium salt and solvent (MeCN) in a clean conical flask at room temperature.
2. Immerse the mixture in a sonicator.

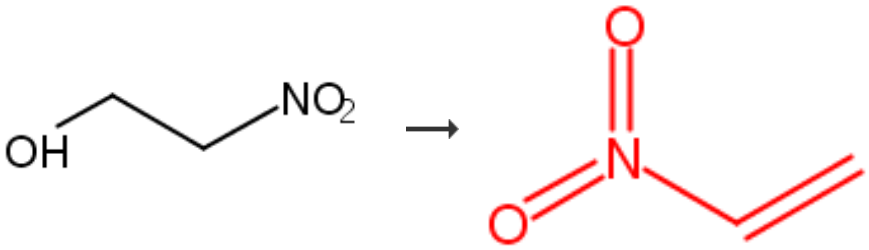
View more...

Available Experimental Data

^{1}H NMR, Mass Spec

View with MethodsNow

14. Single Step



69%

Overview

Steps/Stages

1.1 R:Phthalic anhydride, cooled; 130°C; 1 h, 130°C; 2 h, 130°C → 180°C

Notes

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

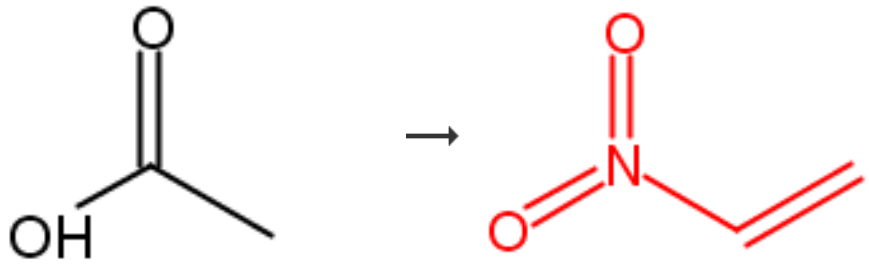
References

Preparation of arylcyclopentenones as herbicides

By Hachisu, Shuji et al

From PCT Int. Appl., 2016062585, 28 Apr 2016

15. Single Step



72%

Overview

Steps/Stages

Notes

1.1 R:KNO_3, R:DMF, R:$POCl_3$, rt

green chemistry-process simplification, Vilsmeier-Haack conditions used, no solvent, microwave irradiation (300 W), mechanism studied, optimization study, optimized on reaction conditions, Reactants: 1, Reagents: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

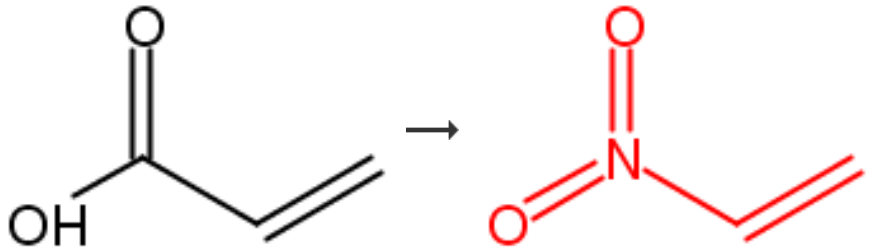
References

Iminium salt-mediated nitro decarboxylation of α, β-unsaturated acids for the synthesis of β-nitro styrenes under non-conventional conditions

By Mukka, Satish Kumar et al

From Organic Communications, 5(2), 42-49; 2012

16. Single Step



66%

Overview

Steps/Stages

1.1 R:HNO_3, S:MeCN, 180 s, rt

Notes

stereoselective, microwave irradiation, green chemistry-catalyst, reusable catalyst, zeolite Y used as catalyst, alternatively conventional heating and sonication method shown, Reactants: 1, Reagents: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

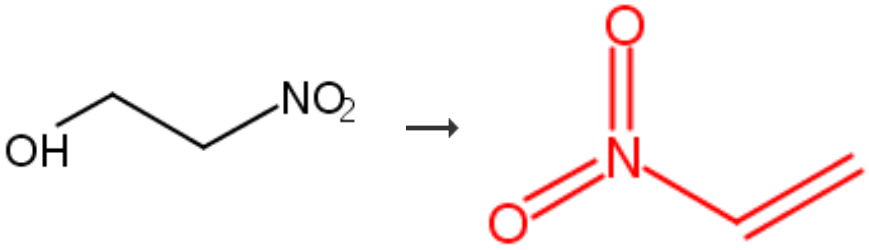
References

Zeolite Y-assisted nitration of aromatic and heterocyclic compounds and decarboxylative nitration of α,β-unsaturated acids under non-conventional conditions

By Sudhakar Chary, V. et al

From Catalysis Science & Technology, 6(5), 1430-1434; 2016

17. Single Step



69%

Overview

Steps/Stages

1.1 R:Phthalic anhydride, -78°C → 130°C; 1 h, 130°C; 2 h, 130°C → 180°C

Notes

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

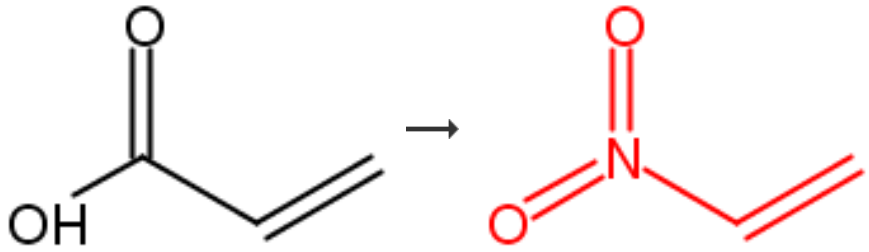
References

Preparation of herbicidally active 2-(substituted-phenyl)-cyclopentane-1,3-dione compounds and derivatives thereof

By Avery, Alaric James et al

From PCT Int. Appl., 2014170413, 23 Oct 2014

18. Single Step



65%

Overview

Steps/Stages

1.1 R:Isocyanuric chloride, R:DMF, R:$NaNO_2$, S:CH_2Cl_2, 85 min, rt

Notes

ultrasound, green chemistry, alternative reaction conditions gave lower yield (Conventional method), other solvent may also used (acetonitrile), Reactants: 1, Reagents: 3, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Ultrasonically Assisted Rate Enhancements in Trichloroisocyanuric Acid/DMF/NaNO2 Triggered Nitration of Aromatic Compounds and Decarboxylative Nitration of α,β-Unsaturated Acids

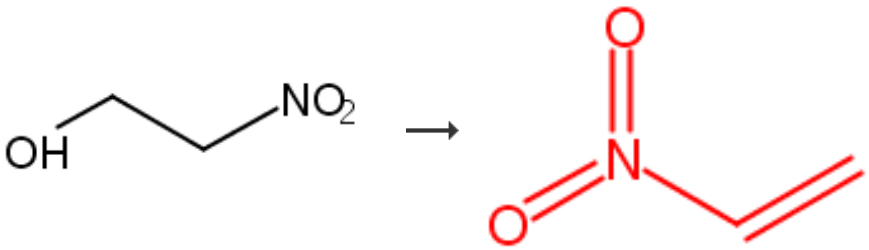
By Satish Kumar, Mukka et al

From Synthetic Communications, 45(19), 2251-2258; 2015

Experimental Procedure

General/Typical Procedure: **Ultrasonically Assisted Synthesis of Nitro Compounds Using TCICA=DMF Reagent with Sodium Nitrite** The reaction mixture containing centimolar molar organic substrate, [TCICA=DMF] reagent, and sodium nitrite were added to the CH_2Cl_2 or acetonitrile in a clean round-bottomed flask clamped in an ultrasonic bath with a frequency of 33 kHz and 100 W electric power rating and stirred for about 1.0 to 1.5 h. After completion, the reaction mixture was treated with $NaHCO_3$ solution, followed by the addition of ethyl acetate. The separation and purification procedure is almost the same as the previous procedure. ***1-Nitro Ethene.*** [1] HNMR (300MHz, $CDCl_3$): δ 5.92 (dd, 1H, H_b, *J*=11.3 Hz, J = 1.5Hz), 6.65 (dd, 1H, H_a, *J*=15.5 Hz, *J*=11.3 Hz), 7.25 (dd, 1H, H_c, *J*=15.5Hz, *J*=1.5 Hz); m/z = 73.

19. Single Step



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Overview

Steps/Stages

1.1 R:Phthalic anhydride, -20 - -5°C, 0.1 MPa; -5°C → 150°C; 1 h, 150°C; 2 h, 150°C → 180°C

Notes

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

References

Asymmetric dearomatization of β-naphthols through a bifunctional-thiourea-catalyzed Michael reaction

By Wang, Shou-Guo et al

From Angewandte Chemie, International Edition, 54(49), 14929-14932; 2015

Reaction Protocol

Procedure

1. Add nitroethanol (13.65 g) and phthalic anhydride (28.86 g, 0.195 mol) to a flask equipped with distillation apparatus.
2. Evacuate the flask to -0.1 mPa.

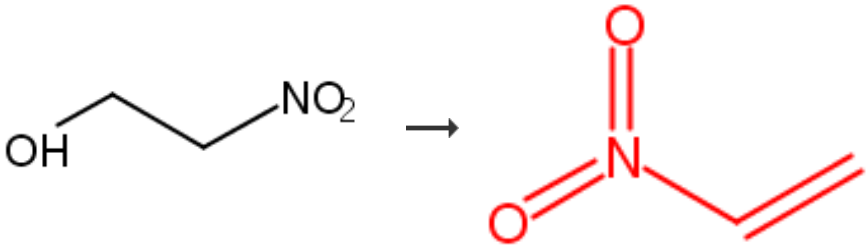
View more...

Available Experimental Data

^{1}H NMR

View with MethodsNow

20. Single Step



Overview

Steps/Stages

1.1 R:EtN(Pr-*i*)$_2$, R:MeSO$_2$Cl, S:PhMe

Notes

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

References

Asymmetric Organocatalysis in Flow

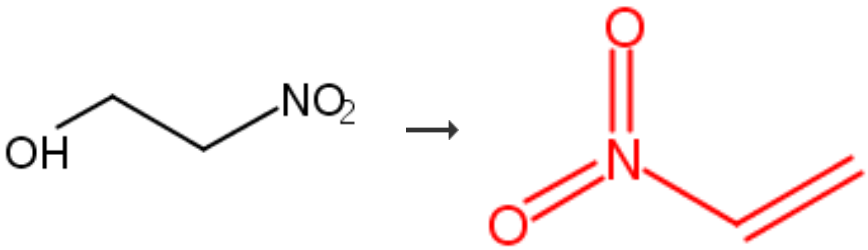
By Jin, Xiaoliang

From null, , No pp.; 2010

Experimental Procedure

Preparation of nitroethylene: B) Procedure in flow: A stock solution of 2-nitroethanol (35.7 microL, 0.5 mmol) and mesyl chloride (38.6 microL, 0.5 mmol) in toluene (1 mL) was prepared and injected into one of the sample loops; Another stock solution of Hunig's base (175 microL, 1 mmol) in toluene (1 mL) was also prepared and injected into a separate sample loops of the R2+ unit of the Vapourtec system. The resulting streams were mixed in a T-piece and then directly into a flow coil (10 mL volume, 1.0 mm i.d.) mounted on the R4 unit to give residence time of 5 min with a total flow rate of 2 mL/min. After leaving the coil the reaction mixture was directed into a glass column containing the scavenging resin A15 (polymer-supported acid) (1.1 g, 1.1 mmol). The purified product 50 was then used directly for the Michael addition reaction.

21. Single Step



Overview

Steps/Stages

1.1 R:Phthalic anhydride

Notes

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

References

Asymmetric Organocatalysis in Flow

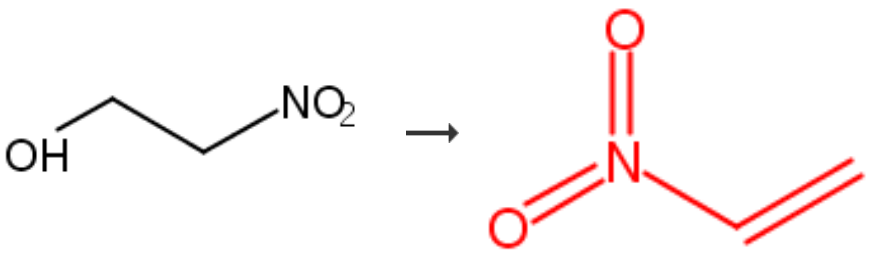
By Jin, Xiaoliang

From null, , No pp.; 2010

Experimental Procedure

Preparation of nitroethylene: A) Procedure in batch: 2-Nitroethanol (0.36 mL, 5 mmol) and phthalic anhydride (0.96 g, 6.5 mmol) were mixed in a 50 mL round bottom flask equipped with a magnetic stir bar. The flask was then equipped with a vacuum distillation setup with a fractional distillation column and a -78 degC bath-cooled receiver. The apparatus was evacuated to 60 mm Hg, and heated to and maintained at 140 degC. The starting materials turned into a homogeneous solution, and the distillate was collected until the distillation ceased to give a pale yellow solid at -78 degC. The solid, containing a mixture of nitroethylene and water, was warmed in an ice-water bath to give a pale yellow heterogeneous mixture. The mixture was mixed with toluene, dried over anhydrous CaCl2 and filtered through a pad of anhydrous CaCl2. The filtrate was collected as a pale yellow stock solution of nitroethylene in toluene, and stored at -20 degC for future use. The concentration of nitroethylene of the stock solution can be estimated via NMR analysis in chloroform (with toluene as internal standard). Nitroethylene as a solution in dry toluene was found to be stable (no change in NMR) for at least 3 months when stored in a freezer.

22. Single Step



Overview

Steps/Stages

1.1

Notes

no exptl. detail, literature prepn., Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

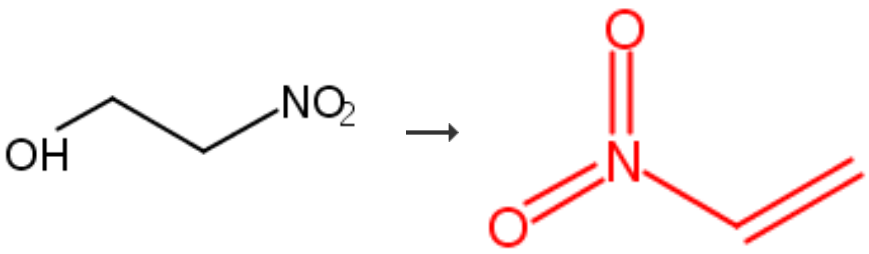
References

Dioxindole in asymmetric catalytic synthesis: direct access to 3-substituted 3-hydroxy-2-oxindoles via 1,4-additions to nitroalkenes

By Retini, Michele et al

From Chemical Communications (Cambridge, United Kingdom), 48(27), 3336-3338; 2012

23. Single Step



Overview

Steps/Stages

1.1 C:Phthalic anhydride, 140-180°C, 80 mmHg

Notes

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

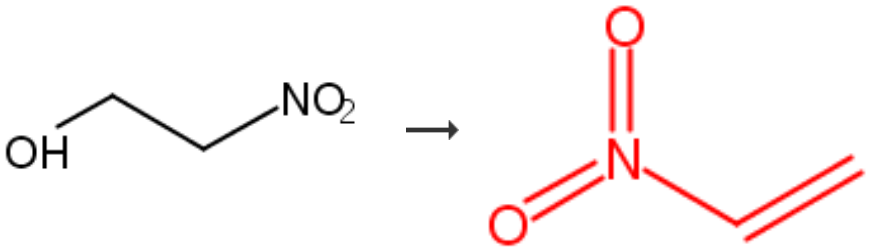
References

Nitroethylene

By Singleton, Daniel A.

From e-EROS Encyclopedia of Reagents for Organic Synthesis, , No pp. given; 2001

24. Single Step



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Overview

Steps/Stages

1.1

Notes

Classification: Elimination; Dehydration; # Conditions: phthalic anhydride; 140-150 deg /80mm; 175-180 deg vac distil, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

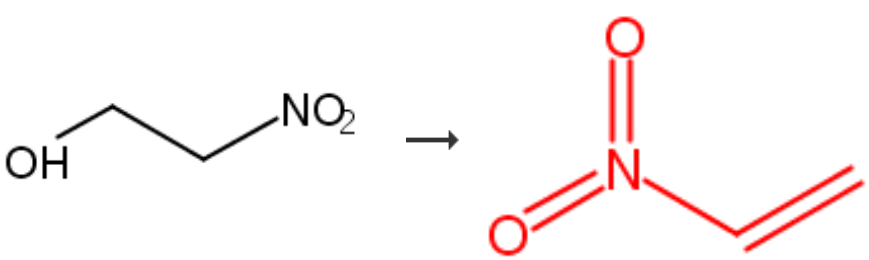
References

Aliphatic nitro compounds. I. Preparation of nitro olefins by dehydration of 2-nitro alcohols

By Buckley, G. D. and Scaife, C. W.

From Journal of the Chemical Society, , 1471-2; 1947

25. Single Step



36%

Overview

Steps/Stages

1.1 R:Phthalic anhydride

Notes

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

References

Asymmetric synthesis of aphanorphine and synthetic approaches towards dietyoxetane

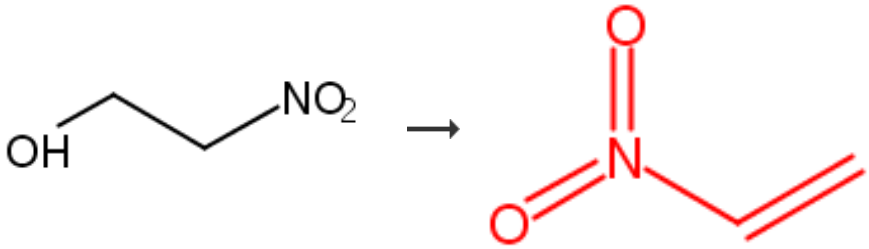
By Welsh, Emma Jane

From null, , No pp.; 2007

Experimental Procedure

Nitroethene 534: A mixture of nitroethanol 533 (518 was author's error) (18.96 g, 208 mmol) and phthalic anhydride (34.25 g, 231 mmol) was heated at reflux at 180 degC for 45 min. The mixture was allowed to cool to rt before replacing the condenser with a distillation apparatus. Nitroethene was distilled from the crude reaction mixture under vacuum into a receiving flask containing CaCl2 and then redistilled from CaCl2 to provide the title compound (5.50 g, 36 %). (LACHRYMATOR!) Nitroethene was stored over CaCl2 at -18 degC. bp 97-99 degC.

26. Single Step



38%

Overview

Steps/Stages

1.1 R:Phthalic anhydride

Notes

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

References

Studies towards the total synthesis of (-)mitragynine using solid-supported reagents

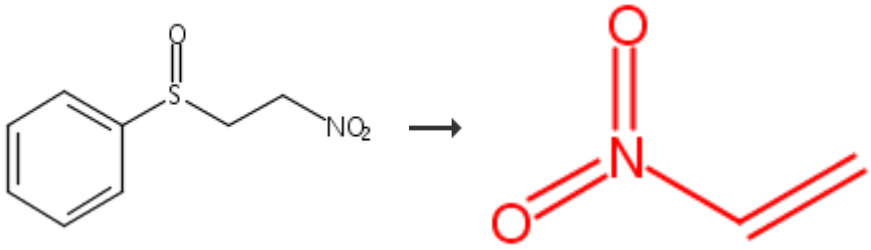
By Henry, D. J.

From null, , No pp.; 2003

Experimental Procedure

Nitroethylene 86: 2-Nitroethanol 90 (3.96 g, 43.5 mmol) was added to phthalic anhydride (9.7 g, 65 mmol) and the mixture heated to 150 degC at approx. 80 mm Hg until the mixture had homogenised. The product was then distilled at 180 degC, and the resultant yellow liquid was dried over calcium chloride and filtered to afford nitroethylene 86 (1.22 g, 38 %).

27. Single Step



Overview

Steps/Stages

1.1 S:Benzene

Notes

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

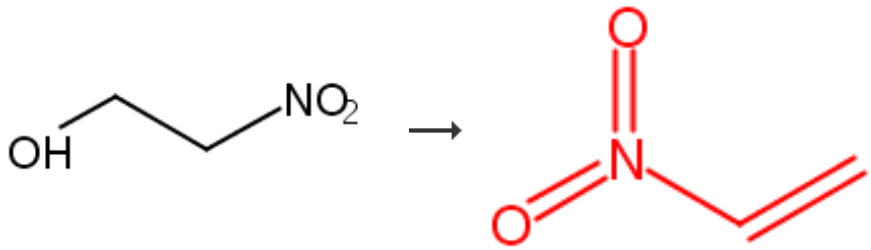
References

2-Nitroethyl phenyl sulfoxide. A novel reagent for facile nitroethylene transfer

By Ranganathan, Darshan et al

From Journal of Chemical Research, Synopses, (3), 78-9; 1983

28. Single Step



Overview

Steps/Stages

1.1 C:Phthalic anhydride

Notes

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

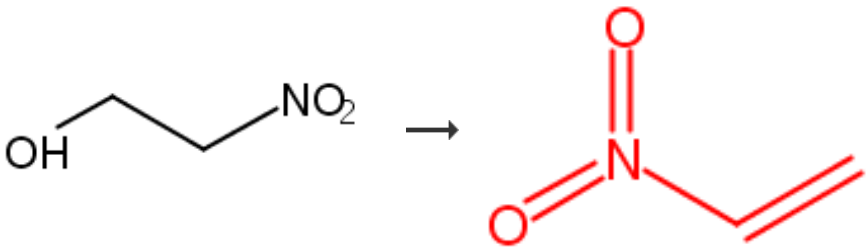
References

Nitroethylene: a stable, clean, and reactive agent for organic synthesis

By Ranganathan, Darshan et al

From Journal of Organic Chemistry, 45(7), 1185-9; 1980

29. Single Step



Overview

Steps/Stages

1.1

Notes

Classification: Elimination; Dehydration; # Conditions: phthalic anhydride, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

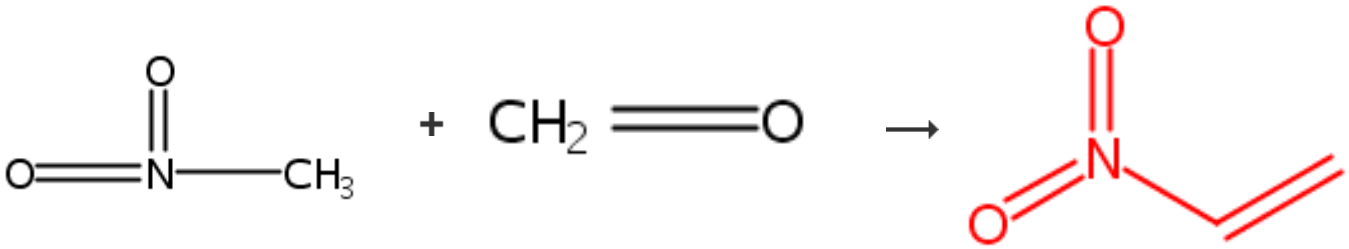
References

Polycyclic compounds containing nitrogen. III. Diels-Alder reaction of nitroethylene

By Drake, Nathan L. and Kraebel, Charlotte M.

From Journal of Organic Chemistry, 26, 41-5; 1961

30. 2 Steps



Overview

Steps/Stages

1.1 R:KOH, S:MeOH

2.1 R:Phthalic anhydride

Notes

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

References

Asymmetric synthesis of aphanorphine and synthetic approaches towards dietyoxetane

By Welsh, Emma Jane

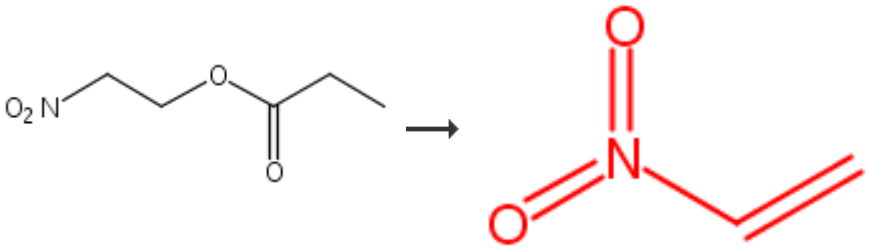
From null, , No pp.; 2007

Experimental Procedure

Step 1
The crude nitroethanol 533 (518 was author's error): A three necked round bottomed flask was equipped with a dropping funnel, thermometer and a condenser. Paraformaldehyde (18.0 g, 0.6 mol) and freshly distilled nitromethane (340 mL, 6.7 mol) were added to the flask and the mixture was stirred vigorously. KOH (3 M in MeOH, Ca. 1.5 mL) was added dropwise through the dropping funnel until pH 8 was reached (litmus paper). The mixture became homogenous after 20 min and the solution was stirred at rt for 1 hr. Conc. H2SO4 was added until the solution reached pH 4 and the solution was stirred at rt overnight. The reaction mixture was subjected to vacuum filtration and the filtrate (66 g) was added to a flask containing diphenyl ether (66 g). The solution was distilled under reduced pressure (0.8 mbar). The first fraction to distil contained unreacted nitromethane (bp 24 degC), the second fraction contained nitroethanol and diphenyl ether (bp 84 degC). The second fraction was biphasic and the lower layer was removed and washed with hexane (30 mL). The lower layer was separated to afford nitroethanol (23.88 g, 44 %) and was used without further purification.

Step 2
Nitroethene 534: A mixture of nitroethanol 533 (518 was author's error) (18.96 g, 208 mmol) and phthalic anhydride (34.25 g, 231 mmol) was heated at reflux at 180 degC for 45 min. The mixture was allowed to cool to rt before replacing the condenser with a distillation apparatus. Nitroethene was distilled from the crude reaction mixture under vacuum into a receiving flask containing CaCl2 and then redistilled from CaCl2 to provide the title compound (5.50 g, 36 %). (LACHRYMATOR!) Nitroethene was stored over CaCl2 at -18 degC. bp 97-99 degC.

31. Single Step



43%

Overview

Steps/Stages

1.1 R:$CaSO_4$

Notes

Classification: Elimination; # Conditions: CaSO4 heat; 240-260 deg, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

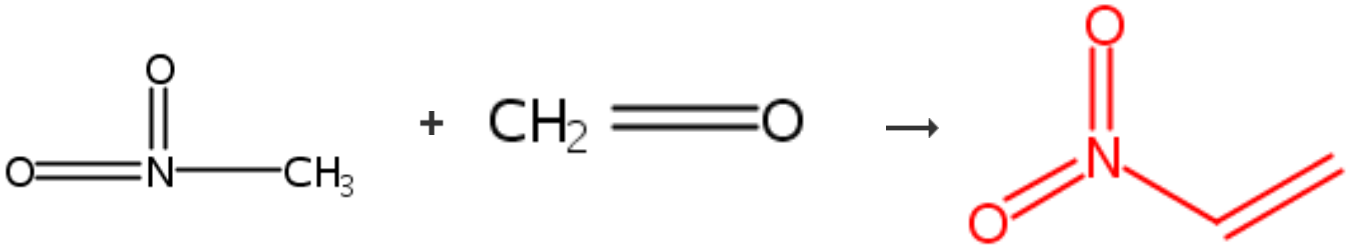
References

Nitroethylene

By Hopff, H. and Capaul, M.

From Helvetica Chimica Acta, 43, 1898-910; 1960

32. 2 Steps



Overview

Steps/Stages

1.1 S:H_2O

2.1 R:Phthalic anhydride

Notes

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

References

Studies towards the total synthesis of (-)mitragynine using solid-supported reagents

By Henry, D. J.

From null, , No pp.; 2003

Experimental Procedure

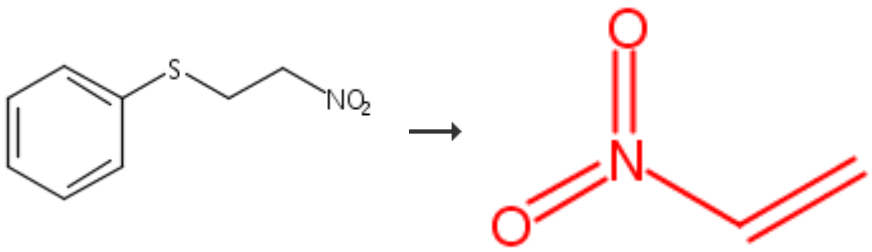
Step 1

2-Nitroethanol 90: Formaldehyde (67 % aqueous solution) (10 mL, 193 mmol) was added to a suspension of polymer-supported hydroxide (95 g, 285 mmol) in nitromethane (200 mL) and the reaction mixture stirred at rt for 10 min. The mixture was filtered, the beads washed extensively with nitromethane (3 x 100 mL) and CH2Cl2 (5 x 100 mL) and the solvent then removed in vacuo. The resultant yellow oil was redissolved in CH2Cl2 (100 mL), dried (MgSO4) and the solvent removed in vacuo to afford 2-nitroethanol 90 as a yellow oil (3.96 g, 23 %).

Step 2

Nitroethylene 86: 2-Nitroethanol 90 (3.96 g, 43.5 mmol) was added to phthalic anhydride (9.7 g, 65 mmol) and the mixture heated to 150 degC at approx. 80 mm Hg until the mixture had homogenised. The product was then distilled at 180 degC, and the resultant yellow liquid was dried over calcium chloride and filtered to afford nitroethylene 86 (1.22 g, 38 %).

33. 2 Steps

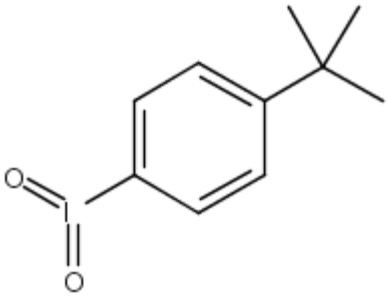


Overview

Steps/Stages

Notes

1.1 R:



R:F_3CCO_2H, S:Benzene

2.1 S:Benzene

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

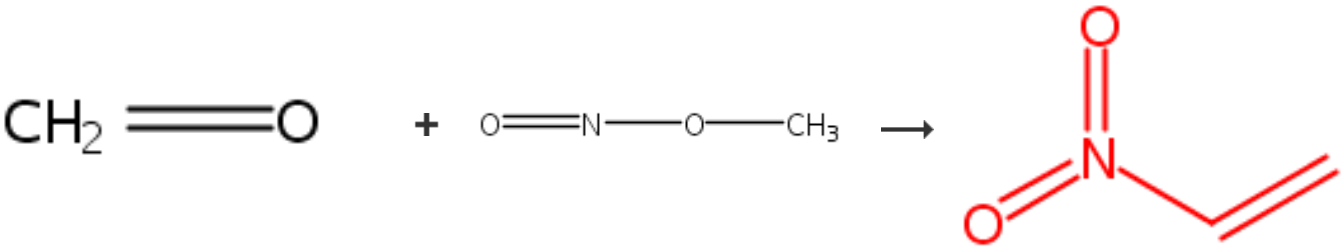
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

34. 2 Steps



Overview

Steps/Stages

1.1

2.1 C:Phthalic anhydride

Notes

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

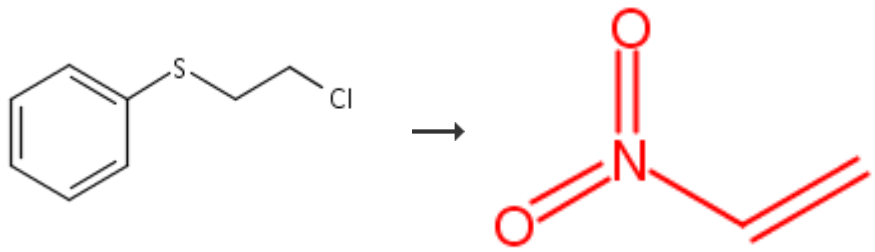
References

Nitroethylene: a stable, clean, and reactive agent for organic synthesis

By Ranganathan, Darshan et al

From Journal of Organic Chemistry, 45(7), 1185-9; 1980

35. 3 Steps

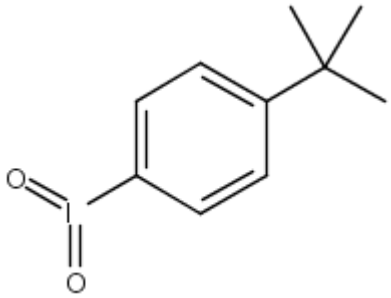


Overview

Steps/Stages

1.1 R:$AgNO_2$, S:Et_2O

2.1 R:



R:F_3CCO_2H, S:Benzene

3.1 S:Benzene

Notes

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

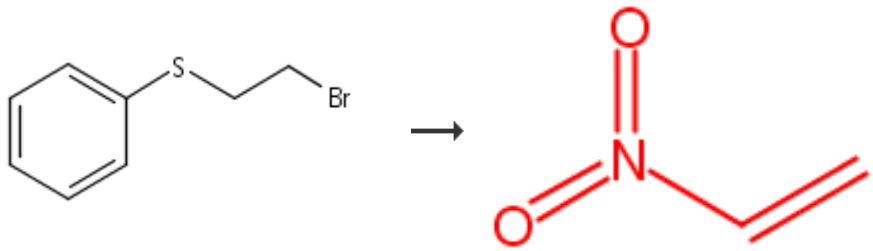
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

36. 3 Steps

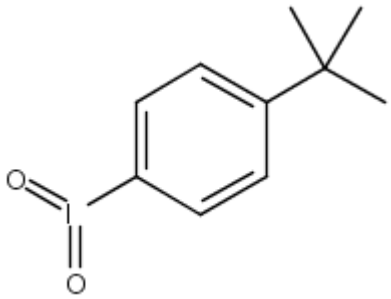


Overview

Steps/Stages

1.1 R:$NaNO_2$, S:DMSO

2.1 R:



R:F_3CCO_2H, S:Benzene

3.1 S:Benzene

Notes

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

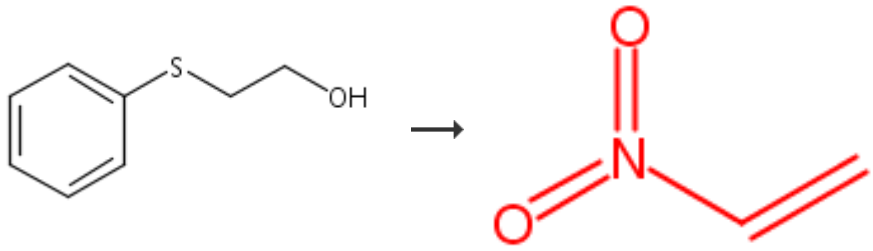
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

37. 4 Steps



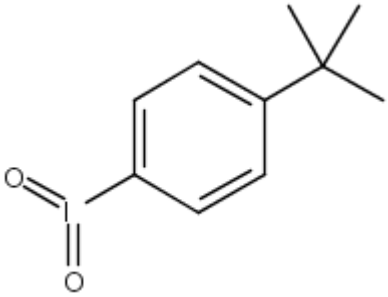
Overview

Steps/Stages

1.1 R:$SOCl_2$

2.1 R:$AgNO_2$, S:Et_2O

3.1 R:



R:F_3CCO_2H, S:Benzene

4.1 S:Benzene

Notes

Reactants: 1, Reagents: 4, Solvents: 2, Steps: 4, Stages: 4, Most stages in any one step: 1

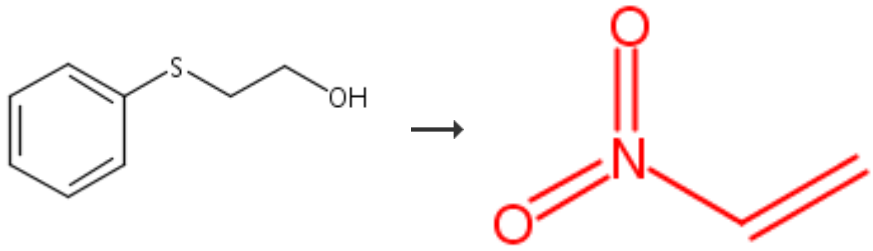
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

38. 4 Steps



Overview

Steps/Stages

Notes

1.1 R:PBr_3

Reactants: 1, Reagents: 4, Solvents: 2, Steps: 4, Stages: 4, Most stages in any one step: 1

2.1 R:$NaNO_2$, S:DMSO

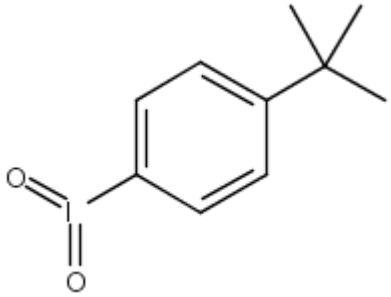
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

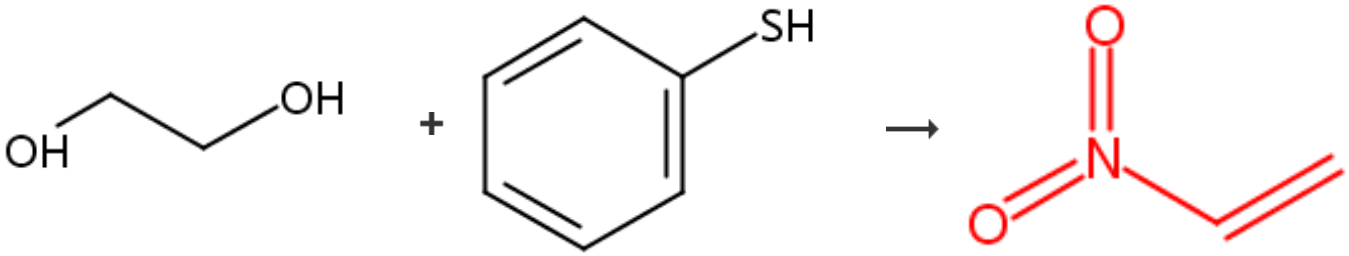
3.1 R:



R:F_3CCO_2H, S:Benzene

4.1 S:Benzene

39. 6 Steps



[Step 2.1]

Overview

Steps/Stages

Notes

1.1 R:HBr, S:H_2O

Reactants: 2, Reagents: 6, Solvents: 3, Steps: 6, Stages: 6, Most stages in any one step: 1

2.1 R:NaOH, S:H_2O

References

3.1 R:$SOCl_2$

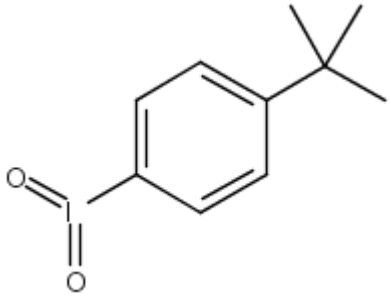
A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

4.1 R:$AgNO_2$, S:Et_2O

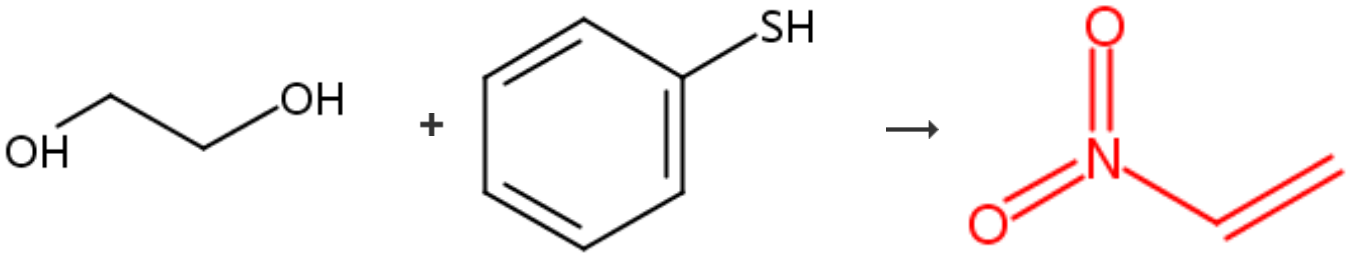
5.1 R:



R:F_3CCO_2H, S:Benzene

6.1 S:Benzene

40. 6 Steps



[Step 2.1]

Overview

Steps/Stages

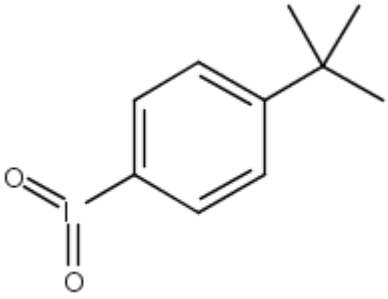
1.1 R:HBr, S:H_2O

2.1 R:NaOH, S:H_2O

3.1 R:PBr_3

4.1 R:$NaNO_2$, S:DMSO

5.1 R:



R:F_3CCO_2H, S:Benzene

6.1 S:Benzene

Notes

Reactants: 2, Reagents: 6, Solvents: 3, Steps: 6, Stages: 6, Most stages in any one step: 1

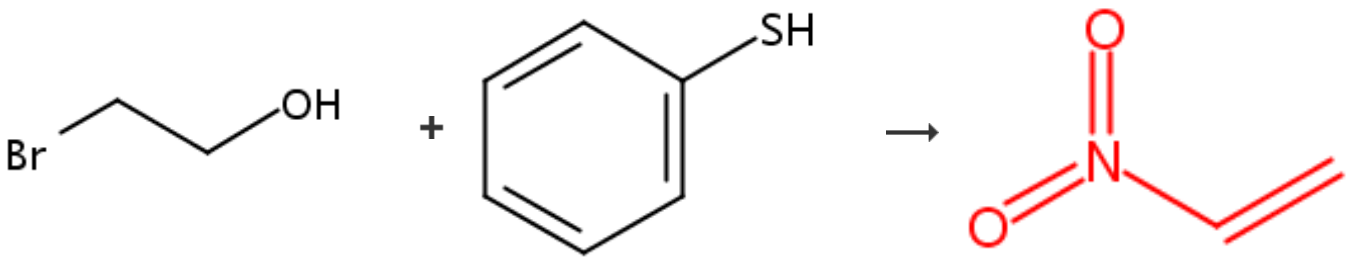
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

41. 5 Steps



Overview

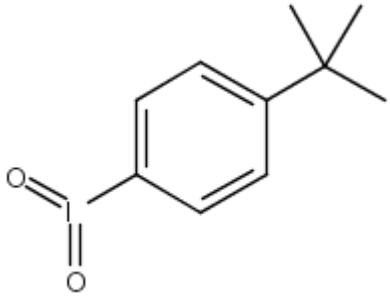
Steps/Stages

1.1 R:NaOH, S:H_2O

2.1 R:$SOCl_2$

3.1 R:$AgNO_2$, S:Et_2O

4.1 R:



R:F_3CCO_2H, S:Benzene

5.1 S:Benzene

Notes

Reactants: 2, Reagents: 5, Solvents: 3, Steps: 5, Stages: 5, Most stages in any one step: 1

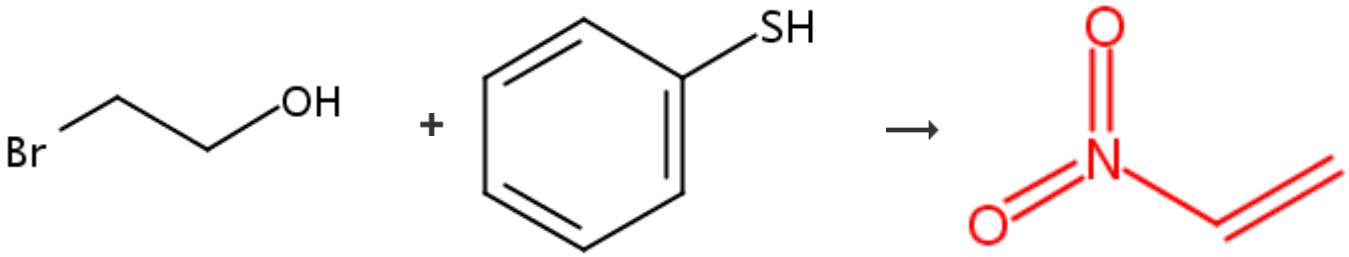
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

42. 5 Steps



Overview

Steps/Stages

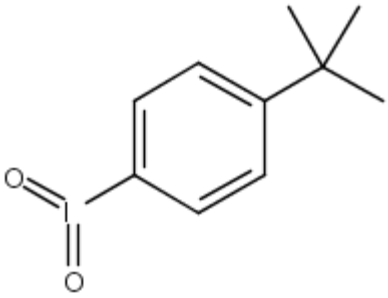
Notes

1.1 R:NaOH, S:H_2O

2.1 R:PBr_3

3.1 R:$NaNO_2$, S:DMSO

4.1 R:



R:F_3CCO_2H, S:Benzene

5.1 S:Benzene

Reactants: 2, Reagents: 5, Solvents: 3, Steps: 5, Stages: 5, Most stages in any one step: 1

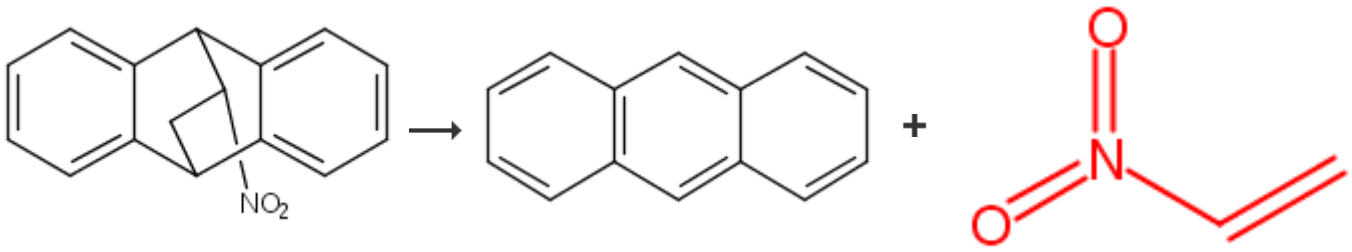
References

A practical and convenient synthesis of the nitroethylene transfer reagent, 2-nitroethyl phenyl sulfoxide

By Ranganathan, S. et al

From Tetrahedron Letters, 28(25), 2893-4; 1987

43. Single Step



Overview

Steps/Stages

1.1 250°C

Notes

Aromatization, Cleavage, Ring cleavage, thermal, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

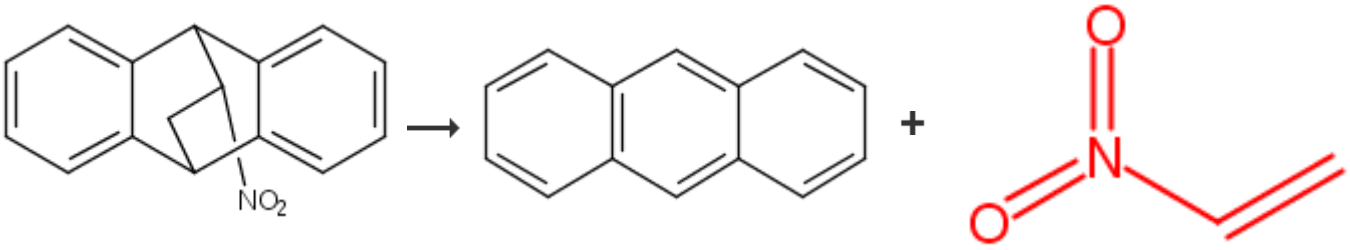
References

The retro-Diels-Alder reaction. Part I. C-C dienophiles

By Rickborn, Bruce

From Organic Reactions (Hoboken, NJ, United States), 52, No pp. given; 1998

44. Single Step



Overview

Steps/Stages

1.1 250°C

Notes

Cleavage, Ring cleavage, thermal, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

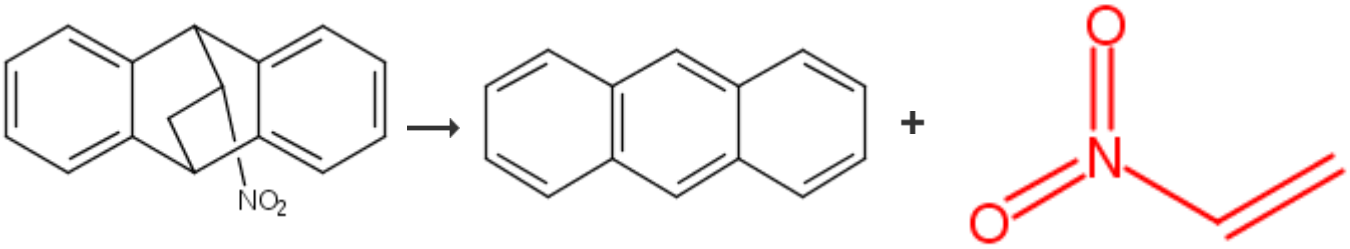
References

The retro-Diels-Alder reaction. Part I. C-C dienophiles

By Rickborn, Bruce

From Organic Reactions (Hoboken, NJ, United States), 52, No pp. given; 1998

45. Single Step



Overview

Steps/Stages

1.1 S:PhOPh, 250°C

Notes

Cleavage, Ring cleavage, thermal, Reactants: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

The retro-Diels-Alder reaction. Part I. C-C dienophiles

By Rickborn, Bruce

From Organic Reactions (Hoboken, NJ, United States), 52, No pp. given; 1998