1. Single Step

Overview

Steps/Stages

1.1 R:TMEDA, R:TiCl₃, S:THF, 4 h, 25°C

Notes

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

References

Reaction Pathway of Methylenation of Carbonyl Compounds with Bis(iodozincio)methane

By Sada, Mutsumi et al

From Journal of the American Chemical Society, 132(49), 17452-17458; 2010

90%

Experimental Procedure

General/Typical Procedure: **General procedure for methylenation of esters.** To β -TiCl $_3$ (4.0 mmol), THF (10 mL) was added at 0 °C. The mixture was stirred for 10 min at room temperature. To the obtained dispersion, bis(iodozincio)methane (1, 0.5 M in THF, 2.0 mmol), TMEDA (8.0 mmol), and ester (1.0 mmol) was added subsequently. The mixture was stirred for 4 h at room temperature. To the mixture, 20 mL of hexane was added. The resulting mixture was passed through celite® column. The product was purified with a short alumina column chromatography. **2-(Methoxy)-2-phenylethene (16d):** Yield 89%. ¹H-NMR (300 MHz, CDCl $_3$): δ 7.3-7.1 (m, 5H), 3.98 (s, 1H), 3.91 (s, 1H), 3.1 (s, 3H). The spectra was identified with the authentic sample.

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2. Single Step

Overview

Steps/Stages Notes

Se Br

<AMD>(polystyrene-supported)</AMD>, S:THF, 30 min, rt

1.2 R:LiBH₄, 1 h, rt

S:THF, 10 min, rt; 12 h, rt S:THF, 30 min, rt 1.3

1.4

1.5 R:NaH, 1 h, rt

1.6 S:THF, 15 min, rt; 2 h, reflux; reflux \rightarrow rt

1.7 R:H₂O₂, S:H₂O, S:THF, 30 min, rt selenium bromide bound to polystyrene support, green chemistry-process simplification, solid-supported reaction, Reactants: 2, Reagents: 4, Solvents: 2, Steps: 1, Stages: 7, Most stages in any one step: 7

Page 2

References

A facile solid-phase synthesis of vinyl ethers using a selenium traceless linker

By Liu, Xiao-Ling et al

From Journal of Chemical Research, (2), 118-120; 2006

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3. Single Step

Overview

Steps/Stages

1.1 R:NaH, S:THF, 1 h, rt

1.2 S:THF, rt; 1 h, reflux; reflux \rightarrow 0°C

1.3 R:H₂O₂, S:H₂O, 10 min, 0°C; 20 min, rt

Notes

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

References

Convenient one-pot synthesis of vinyl ethers from phenyl 2-hydroxyalkyl selenides

By Sheng, Shou-Ri et al

From Synthetic Communications, 35(22), 2839-2845; 2005

Experimental Procedure

General/Typical Procedure: General Procedure for the Preparation of Vinyl Ethers To a solution of phenyl 2-hydroxyalkyl selenide (10 mmol) in dry THF (20 mL) at room temperature was added sodium hydride (0.04 g, 60% dispersion, 1.0 mmol). The resulting mixture was stirred for ca. 1 h at room temperature. A solution of the organic halide (1.0 mmol) in dry THF (2 mL) was then added dropwise, and the reaction flask was placed in an oil bath preheated to 80°C. The mixture was refluxed for 1 h and then cooled gradually to 0°C. The 30% hydrogen peroxide (1.0 mL, 11.6 mmol) was added over 10 min. After an additional stirring for 20 min at room temperature, water (20 mL) was added and the solution was extracted with ether (20 x 3 mL). The combined organic phase was washed with saturated NaHCO₃ solution, brine, and water (twice) and then dried over magnesium sulfate. The CH₂Cl₂/hexane, 10:90) to give the pure product. **1-Methoxy-l-phenylethene (4c):** [9b] Colorless oil, yield 88%. ¹H NMR: δ =7.65-7.52 (m, 2H), 7.18-7.04 (m, 3H), 4.58 (d, J= 2.0Hz, 1H), 4.21 (d, J= 2.0 Hz, 1H), 3.71 (s, 3H); IR (film): ν = 3057, 2974, 2855, 1634, 1593, 1495, 1380, 1243, 1094, 1045, 988, 812 cm⁻¹.

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4. Single Step

Overview

Steps/Stages

- 1.1 R:NaH, S:THF, 1 h, 80°C
- 1.2 R:H₂O₂, S:H₂O, S:THF, 0°C; .5 h, rt

Notes

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

References

A facile one-pot method for the synthesis of vinyl ethers from 2-hydroxyalkyl phenyl selenides

By Sheng, Shou Ri et al From Chinese Chemical Letters, 16(11), 1421-1423; 2005

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5. Single Step

Overview

Steps/Stages

1.1 R:

CI —— Ti —— CI

R:TMEDA, S:THF

Notes

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

References

Methylenation of esters with bis(iodozincio)methane-TiCl2-TMEDA system

By Matsubara, Seijiro et al

From Chemistry Letters, (8), 825-826; 1999

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93%

Overview

Steps/Stages

1.1 R:Me₃SiSO₃CF₃, R:EtN(Pr-i)₂, S:CH₂Cl₂

Notes

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

References

Synthesis of cyclic and acyclic enol ethers (vinyl ethers)

By Gassman, Paul G. et al From Journal of Organic Chemistry, 58(6), 1449-57: 1993

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7. Single Step

92%

Overview

Steps/Stages

1.1 R:NaH

Notes

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

References

An efficient synthesis of ketone enol ethers mediated by N-(1-alkoxyalkyl)benzotriazoles

By Katritzky, Alan R. et al

From Synthesis, (4), 279-83; 1991

Experimental Procedure

Alkyl 1-Alkenyl Ethers 7; General Procedure: A mixture of compound 5 (30 mmol) and NaH (60 mmol) is slowly heated (oil bath) under reduced pressure (0.1-5 Torr, depending on the molecular weight of the desired ether) in a distillation appara tus equipped with a receiver cooled in a dry ice/acetone bath. The 1-alkenyl ether distilled off in the range of 140-160°C. The crude product (usually of purity > 90 % is further purified by fractional distillation. **7e**, yield 92%. bp (°C)/ mbar 100-104/ 60; Molecular Formula 8 or Lit. bp (°C)/mbar 86-89/18 22; 1 H-NMR (CDCl $_3$ /TMS) 5 5 , J(Hz) 3.73 (s, 3H, OCH $_3$), 4.21 (d, 1H, J= 2.2, =CH $_2$), 4.66 (d, 1H, J= 2.6, =CH $_2$), 7.33 (m, 5H, Ph); 1 3C-NMR (CDCl $_3$ /TMS) 5 5 55.2 (OCH $_3$), 81.7 (CH $_2$), 82.1 (=CO), 125.3 (2C, Ph), 125.4 (Ph), 128.1 (2C, Ph), 128.4 (Ph); MS (7 eV) $^\circ$ m/z (%) 134 (100, M+), 103 (51), 91 (53), 78 (80), 65 (24), 51 (69).

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93%

8. Single Step

Overview

Steps/Stages

1.1

Notes

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

References

Organic transformations via metal silane reagents: syntheses of vinyl ethers from dimethyl ketals and pentacarbonyl(trimethylsilyl)manganese

By Marsi, Marianne and Gladysz, J. A. From Tetrahedron Letters, 23(6), 631-4; 1982

79%

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9. Single Step

Overview

Steps/Stages Notes

1.1 R:HgO, R:I₂, S:Et₂O, 3 h, rt

1.2 R:t-BuOK, S:THF, overnight, rt workup, Reactants: 2, Reagents: 3, Solvents: 2, Steps: 1, Stages: 2, Most stages in any one

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step: 2

References

Carbon-carbon bond formation by radical addition-fragmentation reactions of Oalkylated enols

By Cai, Yudong et al

From Organic & Biomolecular Chemistry, 2(17), 2517-2529; 2004

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10. Single Step

$$CH_3$$
 CH_3
 CH_3

70%

Overview

Steps/Stages

1.1 R:

S:THF, 60 min, 0°C

Notes

in-situ generated reagent (dianion), Reactants: 1, Reagents: 2, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

A new and highly effective organometallic approach to 1,2-dehalogenations and related reactions

By Azzena, Ugo et al

From Journal of Organometallic Chemistry, 692(18), 3892-3900; 2007

1.2 R:H₂O, 0°C

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Overview

Steps/Stages

- 1.1 R:Me₃SiCl, C:PhCO₂H, S:C₅H₅N, 3 h, 70°C; 70°C \rightarrow 0°C
- 1.2 R:NaOH, S:H₂O, 0°C

Notes

slow addition of aq. NaOH stage 2, Reactants: 1, Reagents: 2, Catalysts: 1, Solvents: 2, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Gallium tribromide catalyzed coupling reaction of alkenyl ethers with ketene silyl acetals

By Nishimoto, Yoshihiro et al

From Angewandte Chemie, International Edition, 51(32), 8073-8076, S8073/1-S8073/68; 2012

Reaction Protocol

Procedure

- 1. Add benzoic acid (3.0 mmol, 0.37 g), followed by trimethylsilyl chloride (90 mmol, 9.7 g) to the dimethyl acetal of acetophenone (30 mmol, 4.8 g) in pyridine (30 mL).
- 2. Stir the reaction mixture at 70 °C for 3 h.

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12. Single Step

Overview

Steps/Stages

- 1.1 R:NaH, S:THF, 1 h, reflux; reflux → rt
- 1.2 1 h, rt

Notes

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

References

Some uses of trifyl in organic synthesis

By Judelson, Deborah Aaronson From null, , 120 pp.; 1982 CASREACT ®: Copyright © 2016 American Chemical Society. All Rights Reserved. CASREACT contains reactions from CAS and from: ZIC/VINITI database (1974-1999) provided by InfoChem; INPI data prior to 1986; Biotransformations database compiled under the direction of Professor Dr. Klaus Kieslich; organic reactions, portions copyright 1996-2006 John Wiley & Sons, Ltd., John Wiley and Sons, Inc., Organic Reactions Inc., and Organic Syntheses Inc. Reproduced under license. All Rights Reserved.

13. Single Step

Overview

Steps/Stages

1.1 R:Et₃N, C:HgCl₂, S:MeOH

Notes

2:1.5:3 alkyne-HgCl2-NEt3, reflux 1 h, Reactants: 2, Reagents: 1, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

60%

References

Catalytic and oxidative methoxymercuration of terminal alkynes: syntheses of 2-methoxy-1-alkenes and 2-methoxyacrolein acetals

By Barluenga, Jose et al From Synthesis, (2), 144-6; 1988

Experimental Procedure

General/Typical Procedure: **2-Methoxy-1-alkenes 2; General Procedure:** Mercury(II) chloride (4.07 g. 15 mmol) is added under argon to a stirred solution of the appropriate 1-alkyne or propargyl ether **1** (20 mmol) and anhydrous Et₃N (4.2 mL, 30 mmol) in anhydrous CH₃OH (50 mL), and the resultant suspension is refluxed for) h (**2g,** h require 2 h). The suspension is filtered under argon and the liquid phase is evaporated at 0.05 Torr. The residue is triturated with anhydrous Et₂O (3 x 30 mL), the suspension filtered under argon, and the filtrate concentrated at 0.05 Torr. The crude product is a nearly pure, pale yellow oil, which is subsequently bull-to-bull distilled at 0.05 Torr. **2g.** Yield 60%. bpc (°C)/Torr: 60-70/0.05; Lit. bp (°C)/Torr: 85-87/13¹¹. ¹H-NMR (CDCl₃/TMS),^a δ , J (Hz): 3.7 (s, 3H); 4.2 (d, 1H, J = 3); 4.6 (d, 1 H, J = 3); 7.1- 7.8 (m, 5H); ¹³C-NMR (neat/TMS),^a δ : 55.8 (q); 82.6 (t); 126.4 (d); 129.1 (d); 129.4 (d); 137.6 (s); 161.8 (s); MS (70 eV),^b m/z: 134 (M+).

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Steps/Stages

1.1 R:Me₃SiCl, R:C₅H₅N, C:PhCO₂H

Notes

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

References

Diels-Alder Reactions of α -Substituted Styrenes with p-Benzoquinone

By Willmore, Nikolaos D. et al

From Journal of Organic Chemistry, 59(7), 1889-91; 1994

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15. Single Step

$$CH_3$$
 CH_3

37%

Overview

Steps/Stages

- 1.1 C:p-MeC₆H₄SO₃H, S:PhCl, 2 h, reflux; reflux \rightarrow rt
- 1.2 R:N(CH₂CH₂OH)₃, rt, neutralized

Notes

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

References

A Marcus Treatment of Rate Constants for Protonation of Ring-Substituted α -Methoxystyrenes: Intrinsic Reaction Barriers and the Shape of the Reaction Coordinate

By Richard, John P. and Williams, Kathleen B.

From Journal of the American Chemical Society, 129(21), 6952-6961; 2007

Experimental Procedure

α-Methoxystyrene was prepared by refluxing p-toluenesulfonic acid (50 mg) and acetophenone dimethyl ketal (10 g, 0.07 mol) in 150 mL of chlorobenzene (150 mL) for 2 hours in a flask equipped with a Dean Stark trap to remove water. The solution was then cooled to room temperature, the acid neutralized with 100 mg of triethanolamine and the solvent was removed. Vacuum distillation provided the desired alkene in 37% which contained significant amounts of the starting keton. The final purification was by column chromatography over silica gel, eluting with 20/80 ether/hexanes (v/v) and 0.5% triethylamine. α-**Methoxystyrene**, yield 37% 1 H NMR (400 MHz, CDCl₃): δ 3.75 (3H, s, OCH₃), 4.22 (1H, d, J = 2.9 Hz, R₂ = CH₂), 4.66 (1H, d, J = 2.9 Hz, R₂=CH₂), 7.31-7.63 (5H, m, Ar).

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→ CH₃

Na

Overview

Steps/Stages

1.1 S:DMF

Notes

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

References

Competition between vinylic substitution and conjugate addition in the reactions of vinyl selenoxides and vinyl selenones with nucleophiles in DMF

By Tiecco, Marcello et al

From Tetrahedron, 42(17), 4889-96; 1986

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17. Single Step

Overview

Steps/Stages

1.1 R:CH₂=CH(CH₂)₅Me, S:THF

Notes

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

References

Pyridylseleno group in organic synthesis. Part 4. Oxyseleniation of olefins using pyridine-2-selenenyl bromide as a selenium reagent and its utilization in the synthesis of 2-pyridyl vinylic selenides

By Toshimitsu, Akio et al

From Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), (2), 373-8; 1985

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18. Single Step

Overview

Steps/Stages

- 1.1 S:THF, S:Benzene
- 1.2 R:NaOH, S:Et₂O, S:H₂O

Notes

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

References

Carbonyl methylenation using a titaniumaluminum (Tebbe) complex

By Pine, Stanley H. et al From Journal of Organic Chemistry, 50(8), 1212-16; 1985

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19. Single Step

Overview

Steps/Stages Notes

1.1 S:THF, S:PhMe

1.2 R:NaOH, S:Et₂O, S:H₂O

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

References

Carbonyl methylenation using a titaniumaluminum (Tebbe) complex

By Pine, Stanley H. et al From Journal of Organic Chemistry, 50(8), 1212-16; 1985

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20. Single Step

Overview

Steps/Stages

1.1 R:HCIO₄, S:MeOH

Notes

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

References

Conversion of aromatic ketones into π -arylalkanoic acids. Oxidation by thallium(III) and by halogens

By Higgins, Stanley D. and Thomas, C. Barry From Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), (1), 235-42; 1982

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Overview

Steps/Stages

1.1 R:C₅H₅N, S:THF, S:PhMe

Notes

Classification: Olefination; # Conditions: pyridine toluene THF; -40 deg 30mn; 1h30mn warm to 20 deg, Reactants: 2, Reagents: 1, Solvents: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Tunneling effects in the hydrogen atom transfer reaction of tetrasubstituted cyclopropenes

By Padwa, Albert and Chou, Chuen S. From Journal of the American Chemical Society, 102(10), 3619-20; 1980

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22. Single Step

Overview

Steps/Stages

1.1 C:Et₂NH

Notes

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

References

Arene substitution reactions and reduction by sodium in suspension in amines

By David, L. et al

From Bulletin de la Societe Chimique de France, (11-12, Pt. 2), 587-9; 1978

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23. 2 Steps

$$CI$$
 CH_2
 CI
 CH_3
 CH_3
 CH_3
 CH_3
 CH_3
 CH_3

Overview

Steps/Stages

- 1.1 S:THF, 1 h, rt; 15 min, 0°C; 2 h, 0°C
- 2.1 R:TMEDA, R:TiCl₃, S:THF, 4 h, 25°C

Notes

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

References

Reaction Pathway of Methylenation of Carbonyl Compounds with Bis(iodozincio)methane

By Sada, Mutsumi et al

From Journal of the American Chemical Society, 132(49), 17452-17458; 2010

Experimental Procedure

Step 1

Bis(iodozincio)methane (1): A mixture of Zn (25 mmol), diiodomethane (1.0 mmol), and PbCl₂ (0.01 mmol) in THF (2.0 mL) was sonicated for 1 h in an ultrasonic cleaner bath under Ar. To the mixture, diiodomethane (10 mmol) in THF (20 mL) was added dropwise over 15 min at 0 °C with vigorous stirring. The mixture was stirred for 2 h at 0 °C. After the stirring was stopped, the reaction vessel was stood undisturbed for several hours. Excess zinc was separated by sedimentation. ¹H NMR spectra of the obtained supernatant showed a broad singlet at -1.1 ppm at 0 °C, which corresponded to the methylene proton of 1. The concentration of the obtained solution was determined by ¹H NMR using 2,2,3,3-tetramethylenebutane as an internal standard. The supernatant was used for the further reaction as a solution of 1 in THF (0.45-0.6 M). Bis(Iodozincio)methane in THF can be kept unchanged for at least two months in the sealed reaction vessel at room temperature. **Bis(iodozincio)methane (1).**

Step 2

General/Typical Procedure: General procedure for methylenation of esters. To β -TiCl $_3$ (4.0 mmol), THF (10 mL) was added at 0 °C. The mixture was stirred for 10 min at room temperature. To the obtained dispersion, bis(iodozincio)methane (1, 0.5 M in THF, 2.0 mmol), TMEDA (8.0 mmol), and ester (1.0 mmol) was added subsequently. The mixture was stirred for 4 h at room temperature. To the mixture, 20 mL of hexane was added. The resulting mixture was passed through celite® column. The product was purified with a short alumina column chromatography. **2-(Methoxy)-2-phenylethene (16d):** Yield 89%. ¹H-NMR (300 MHz, CDCl $_3$): δ 7.3-7.1 (m, 5H), 3.98 (s, 1H), 3.91 (s, 1H), 3.1 (s, 3H). The spectra was identified with the authentic sample.²⁷

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24. 2 Steps

Overview

Steps/Stages

S:MeOH

2.1 R:CH₂=CH(CH₂)₅Me, S:THF

Notes

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

References

Pyridylseleno group in organic synthesis. Part 4. Oxyseleniation of olefins using pyridine-2-selenenyl bromide as a selenium reagent and its utilization in the synthesis of 2-pyridyl vinylic selenides

By Toshimitsu, Akio et al

From Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), (2), 373-8; 1985

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25. 2 Steps

[Step 2.1]

Overview

Steps/Stages

C:CH(OMe)₃ 1.1

2.1 R:HCIO₄, S:MeOH

Notes

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

References

Conversion of aromatic ketones into π arylalkanoic acids. Oxidation by thallium(III) and by halogens

By Higgins, Stanley D. and Thomas, C. Barry From Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), (1), 235-42; 1982

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26. 3 Steps

[Step 2.1]

Overview

Steps/Stages

- 1.1 R:Br₂, S:MeOH
- 2.1 S:MeOH
- 3.1 R:CH₂=CH(CH₂)₅Me, S:THF

Notes

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

References

Pyridylseleno group in organic synthesis. Part 4. Oxyseleniation of olefins using pyridine-2-selenenyl bromide as a selenium reagent and its utilization in the synthesis of 2-pyridyl vinylic selenides

By Toshimitsu, Akio et al

From Journal of the Chemical Society, Perkin Transactions 1: Organic and Bio-Organic Chemistry (1972-1999), (2), 373-8; 1985

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Steps/Stages

1.1 S:MeOH

Notes

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

References

Photochemical Generation of a Primary Vinyl Cation from (E)-Bromostyrene: Mechanisms of Formation and Reaction

By Gronheid, Roel et al

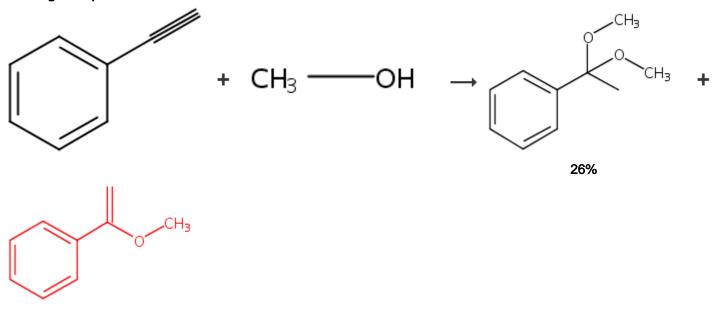
From Journal of Organic Chemistry, 68(8), 3205-3215; 2003

Experimental Procedure

Reactive Intermediates. Product Composition. In a wide range of solvents (methanol, acetonitrile, acetic acid, 2,2,2-trifluoroethanol, 1,4-dioxane, n-pentane, and hexane), irradiation of (E)-bromostyrene (1E) at 254 nm under argon yields three primary photoproducts: (Z)-bromostyrene (1Z), phenylacetylene (2), and styrene (3).

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28. Single Step



Overview

Steps/Stages

1.1 C:159123-30-5, 90 min, reflux

64%

Notes

chemoselective, product distribution depends on catalyst, Reactants: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Organometallic Gold(III) Compounds as Catalysts for the Addition of Water and Methanol to Terminal Alkynes

By Casado, Raquel et al

From Journal of the American Chemical Society, 125(39), 11925-11935; 2003

General Procedure for the Addition of Methanol to Terminal Alkynes. To a solution of 1 mmol of phenylacetylene (1) (0.110 mL) or *n*-heptyne (15) (0.130 mL) in 5 mL of anhydrous MeOH was added the amount of gold compound as specified in Tables 3 and 5, and the mixture was heated at reflux under Ar for 90 min. The reaction mixture was analyzed by GCMS (after addition of 0.5 mL of triethylamine and elimination of metallic gold). If the conversion was higher than 90%, we follow the previously described workup to obtain compounds 12 and 17 as pure colorless oils or mixtures of 2/12 and 16/17 (see Tables 3 and 5 and the Results and Discussion section) as characterized by ¹H NMR (see Supporting Information). Entry 1 compounds 12 and 13.

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29. Single Step

Overview

Steps/Stages

1.1 R:MeSO₃H, C:23108-72-7, S:MeOH

Notes

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

References

Cationic gold(I) complexes: highly efficient catalysts for the addition of alcohols to alkynes

By Teles, J. Henrique et al From Angewandte Chemie, International Edition, 37(10), 1415-1418; 1998

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Overview

Steps/Stages

1.1 C:NaOH, rt; rt \rightarrow 260°C; 5 h, 260°C; 260°C \rightarrow rt

Notes

thermal, 64% conversion, 68% selectivity to Methyl benzoate, optimized on catalyst, MgO gave higher conversion with lower selectivity, alternative catalysts gave lower selectivity, optimization study, other products also detected, Reactants: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Synthesis of methyl benzoate by methoxycarbonylation of acetophenone with dimethyl carbonate over solid base catalysts

By Wu, Dudu et al

From Fuel Processing Technology, 89(8), 803-807; 2008

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34%

Page 20

48%

Overview

Steps/Stages

1.1 S:MeOH

Notes

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

References

Formation and reactivity of the addition products of alkoxides and thiolate anions to vinyl selenones

By Tiecco, Marcello et al From Tetrahedron, 42(17), 4897-906; 1986

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32. 2 Steps

Overview

Steps/Stages Notes

1.1 R:NaOMe, S:MeOH

2.1 S:MeOH

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

References

Formation and reactivity of the addition products of alkoxides and thiolate anions to vinyl selenones

By Tiecco, Marcello et al

From Tetrahedron, 42(17), 4897-906; 1986

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33. Single Step

Overview

Steps/Stages

1.1 S:MeCN

Notes

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

References

Activation of hydrogen by cationic cyclopentadienylmolybdenum dimers with sulfido ligands. 1. Cationic complexes derived from protonation of 1,2-alkenedithiolate ligands

By Laurie, J. C. V. et al

From Journal of the American Chemical Society, 108(20), 6234-41; 1986

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34. 2 Steps

Overview

Steps/Stages

- 1.1 R:HBF₄, S:CHCl₃
- 2.1 S:MeCN

Notes

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

References

Activation of hydrogen by cationic cyclopentadienylmolybdenum dimers with sulfido ligands. 1. Cationic complexes derived from protonation of 1,2-alkenedithiolate ligands

By Laurie, J. C. V. et al

From Journal of the American Chemical Society, 108(20), 6234-41; 1986

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40%

Overview

Steps/Stages

1.1 S:Et₂O

Notes

Classification: Exchange; # Conditions: Et2O 20 deg, Reactants: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Reaction of metal-carbene complexes with Wittig reagents. New vinyl ether synthesis By Casey, Charles P. and Burkhardt, Terry J. From Journal of the American Chemical Society, 94(18), 6543-4; 1972

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Overview

Steps/Stages

1.1 S:MeOH

1.2 R:I₂, S:MeOH

Notes

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

References

The reaction of ethynylferrocene with mercuric acetate

By Bassetti, M. et al From Organometallics, 4(4), 617-23; 1985

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37. Single Step

Overview

Steps/Stages Notes

Page 25

1.1 S:Benzene

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

References

Cyclopropanations and cycloadditions of transition metal carbene complexes

By Wulff, William D. et al

From Pure and Applied Chemistry, 60(1), 137-44: 1988

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38. Single Step

Overview

Steps/Stages

1.1 S:PhMe

Notes

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

References

Metal complexes of biologically important ligands, Part CXVIII. Metathesis of dehydro amino acids with Fischer carbene complexes: synthesis of complexes of amino acid- and peptide- α -carbenes and of isoindoles

By Dialer, Harald et al

From Journal of Organometallic Chemistry, 589(1), 21-28; 1999

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36%

Overview

Steps/Stages

1.1 S:Benzene

Notes

100% overall, Reactants: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Cyclopropanations and cycloadditions of transition metal carbene complexes

By Wulff, William D. et al From Pure and Applied Chemistry, 60(1), 137-44; 1988

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36%

Overview

Steps/Stages

1.1 S:Benzene

Notes

100% overall, Reactants: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[2 + 1]- Versus [4 + 2]-cycloadditions of Fischer carbene complexes with 1,3-dienes. Evidence for a zwitterionic intermediate in a cyclopropanation reaction

By Wulff, William D. et al

From Journal of the American Chemical Society, 110(8), 2653-5; 1988

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Overview

Steps/Stages

1.1 S:PhMe

Notes

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

23%

References

Metal complexes of biologically important ligands, Part CXVIII. Metathesis of dehydro amino acids with Fischer carbene complexes: synthesis of complexes of amino acid- and peptide- α -carbenes and of isoindoles

By Dialer, Harald et al

From Journal of Organometallic Chemistry, 589(1), 21-28; 1999

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