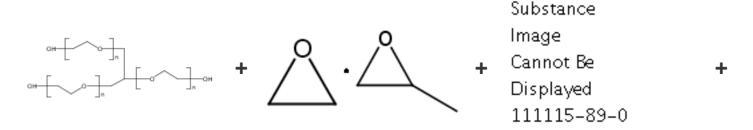
1. Single Step



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1469370-41-9

Overview

Steps/Stages

1.1 C:Bu₂Sn dilaurate, C:947-19-3, C:7473-98-5, 1 h, 60°C

Notes

photochemical, UV irradiation (1000mJ/cm2) prior to heat treatment, Reactants: 4, Catalysts: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

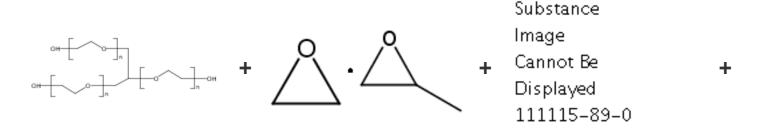
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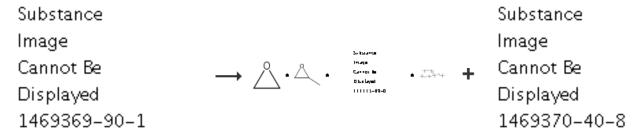
Adhesive compositions for optical parts and cured products thereof

By Nakamura, Satoshi and Morozumi, Yasutaka

From Jpn. Kokai Tokkyo Koho, 2013213175, 17 Oct 2013

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Overview

Steps/Stages

1.1 C:Bu₂Sn dilaurate, C:947-19-3, C:7473-98-5, 1 h, 60°C

Notes

photochemical, UV irradiation (1000mJ/cm2) prior to heat treatment, Reactants: 4, Catalysts: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

References

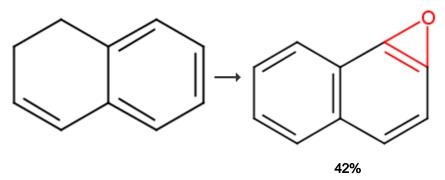
Adhesive compositions for optical parts and cured products thereof

By Nakamura, Satoshi and Morozumi, Yasutaka

From Jpn. Kokai Tokkyo Koho, 2013213175, 17 Oct 2013

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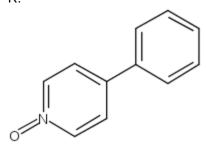
3. Single Step



Overview

Steps/Stages

1.1 R:



R:NaOCI, C:216241-44-0 polymer-supported, S:CH₂CI₂

Notes

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

References

Synthesis and Characterization of a Polymer-Supported Salen Ligand for Enantioselective Epoxidation

By Angelino, Mark D. and Laibinis, Paul E. From Macromolecules, 31(22), 7581-7587; 1998

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4. Single Step

$$F \xrightarrow{F} F \xrightarrow{F} F$$

Overview

Steps/Stages

1.1

Notes

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

References

Argon-matrix isolation of bis(trifluoromethyl)oxirene, perfluoromethylethyloxirene, and their isomeric ketocarbenes

By Torres, M. et al

From Journal of the American Chemical Society, 105(6), 1698-700; 1983

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46%

5. Single Step

Overview

Steps/Stages

- 1.1 R:Polysorb 1 (2-oxopropylated, 2-hydroxy-2-methylpropyla),S:THF, 12 h, rt
- 1.2 R:H₂SO₄, S:H₂O, 0°C; 10 min, 0°C
- 1.3 R: H_2O_2 , S: H_2O , 10 h, rt
- 1.4 S:Dioxane, 36 h, 70°C

Notes

solid-supported reagent, Reactants: 1, Reagents: 3, Solvents: 3, Steps: 1, Stages: 4, Most stages in any one step: 4

References

Epoxidation and oxidation reactions using divinylbenzene crosslinked polystyrene supported tert-butyl hydroperoxide

By Sheela, M. S. and Sreekumar, K.

From Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry, 45B(4), 943-950; 2006

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6. Single Step

Overview

Steps/Stages

1.1 R:mCPBA, S:CH₂Cl₂

Notes

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

References

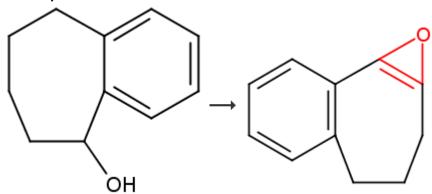
Structural effects on the rates of formation and the stability of enols of cyclic benzyl ketones

By Eldin, Sherif et al

From Journal of the American Chemical Society, 113(4), 1344-9; 1991

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7. 2 Steps



Overview

Steps/Stages

Notes

1.1 R:EtOH, S:Benzene

2.1 R:mCPBA, S:CH₂Cl₂

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

References

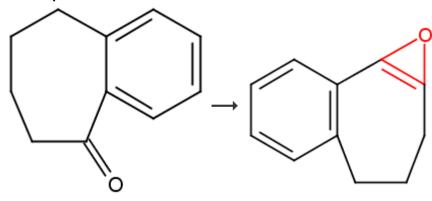
Structural effects on the rates of formation and the stability of enols of cyclic benzyl ketones

By Eldin, Sherif et al

From Journal of the American Chemical Society, 113(4), 1344-9; 1991

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8.3 Steps



Overview

Steps/Stages

1.1 R:NaBH₄, S:EtOH

2.1 R:EtOH, S:Benzene

3.1 R:mCPBA, S:CH₂Cl₂

Notes

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

References

Structural effects on the rates of formation and the stability of enols of cyclic benzyl ketones

By Eldin, Sherif et al

From Journal of the American Chemical Society, 113(4), 1344-9; 1991

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Overview

Steps/Stages

1.1 R:NaOCI, R:Bu₄N+ •Br-, S:CHCl₃, pH 8-9

Notes

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

References

Sodium Hypochlorite

By Galvin, Jennifer M. et al From e-EROS Encyclopedia of Reagents for Organic Synthesis, , No pp. given; 2001

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10. Single Step

Overview

Steps/Stages

1.1 R:H₂O₂, C:Lipase nanoconjugates with polymers, C:691397-13-4 nanoconjugates with lipase, S:H₂O, S:PhMe, 24 h, 50°C

Notes

enzymic, biotransformation, optimization study, optimized on solvent, Reactants: 1, Reagents: 1, Catalysts: 2, Solvents: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Epoxidation of Fatty Acids by Pluronic-Conjugated Lipase in Organic Media

By Yuki, Oda et al

From Catalysis Letters, 146(6), 1073-1078; 2016

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Overview

Steps/Stages

1.1 R:HCO₂H, R:H₂O₂, S:H₂O, 4°C; 2-5 h, rt

Notes

agitation (900 rpm), Reactants: 1, Reagents: 2, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Synthesis, characterization and physicochemical properties of oleic acid ether derivatives as biolubricant basestocks

By Salimon, Jumat et al

From Journal of Oleo Science, 60(12), 613-618; 2011

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12. Single Step

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Notes

photochemical, LED lamp (2000mJ/cm2) used, silica used as filler, Reactants: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Connected structure with improved electric connection reliability, and its manufacture

By Mahara, Shigeo

From Jpn. Kokai Tokkyo Koho, 2014207224, 30 Oct 2014

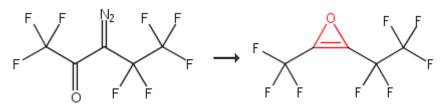
Overview

Steps/Stages

1.1 C:75980-60-8, 5 min

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13. Single Step



Overview

Steps/Stages

1.1

Notes

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

References

Argon-matrix isolation of bis(trifluoromethyl)oxirene, perfluoromethylethyloxirene, and their isomeric ketocarbenes

By Torres, M. et al

From Journal of the American Chemical Society, 105(6), 1698-700; 1983

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14. Single Step

Overview

Steps/Stages

1.1

Notes

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

References

Argon-matrix isolation of bis(trifluoromethyl)oxirene, perfluoromethylethyloxirene, and their isomeric ketocarbenes

By Torres, M. et al

From Journal of the American Chemical Society, 105(6), 1698-700; 1983

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15. Single Step

$$\bigcap_{\mathsf{NO}_2}$$

reaction products with Cy3 and histidine,

Overview

Steps/Stages

1.1

Notes

transition metal alkykidene complexes used as catalyst, limited experimental detail, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Preparation of fluorescent probe-based reactome array for detection, immobilization and isolation of enzymes

By Golyshin, Peter N. et al From PCT Int. Appl., 2010105851, 23 Sep 2010

Experimental Procedure

1-Nitronaphthalene-5,6-oxide. FAB-HR[M+ H]+ found: 1268,467, 59Co-NMR(300 MHZ, CD₃CN): 9610

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16. Single Step

reaction products with Cy3 and histidine,

Overview

Steps/Stages

1.1

Notes

transition metal alkykidene complexes used as catalyst, limited experimental detail, Reactants: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Preparation of fluorescent probe-based reactome array for detection, immobilization and isolation of enzymes

By Golyshin, Peter N. et al From PCT Int. Appl., 2010105851, 23 Sep 2010

Experimental Procedure

1-Nitronaphthalene-7,8-oxide. FAB-HR[M+ H]+ found: 1268,467, 59Co-NMR(300 MHZ, CD₃CN): 6210

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17. Single Step

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+ CI + NH₂ -

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Overview

Steps/Stages

Notes

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1.1 R:

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C:162881-26-7, S:1,3-Dioxolane, 10 min, 100°C

1.2 R:

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C:162881-26-7, S:1,3-Dioxolane

Experimental Procedure

coated on PET film in stage 1, 405nm used in stage 2, photochemical in stage 2, Reactants: 3, Reagents: 3, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

Page 11

References

Photosensitive multilayer dry-resist films with excellent processability and fire resistance, printed circuit boards using them, and their manufacture

By Yamanaka, Toshio et al From Jpn. Kokai Tokkyo Koho, 2009048170, 05 Mar 2009

[Example] Hereinafter, the present invention will be described more specifically with reference to Examples and Comparative Examples. The present invention was not limited thereto. Preparation of the photosensitive resin composition, specific production of the photosensitive dry film resist and the evaluation of physical properties were carried out as follows. Also, the binder polymer used in the following Examples and Comparative Examples were prepared by the method shown in Synthesis Examples 1~10 below. <Preparation of photosensitive resin composition> First photosensitive layer resin composition, (A1) a binder polymer, (B1) (meth) acrylic compound, (C1) light reaction initiator, (D1) flame retardant, (É1) dyes and/or pigments, if necessary (F1) other components were mixed at a predetermined ratio and manufactured. Dioxolane with a solid content weight% (Sc) = 40% was added, the solution was uniformly dissolved and prepared as an organic solvent solution of the first photosensitive layer resin composition. Similarly, second photosensitive layer resin composition, (A2) a binder polymer, (B2) (meth) acrylic compound and preferably (C2) light reaction initiator, if necessary (F2) other components were mixed at a predetermined ratio and manufactured. Dioxolane with a solid content weight% (Sc) = 30% was added, the uniformly dissolved solution was prepared as an organic solvent solution of the second photosensitive layer resin composition. Here, the solid weight of a material other than an organic solvent, the total weight of (A), (B), (C), (D), (E) and the component (F) was shown. For example, in the case of the first photosensitive layer resin composition of (A1), (B1) (C1), (D1), and (E1) and (F1) component was the weight of the liquid materials other than the organic solvent. The liquid was assumed to be included in the weight as a solid content. < Production of the photosensitive dry film resist> The organic solvent solution of the first photosensitive layer resin composition mentioned above was applied to the support film so that the thickness after drying (photosensitive dry film resist having a thickness) was 20Mm. PET film (manufactured by Toray Industries, Inc. LUMIRROR, thickness 25Mm) was used as the support film. Then, the coating layer on the support film was dried under conditions of 100°C for 10 minutes. The organic solvent was removed. As a result, the sheets comprising a first photosensitive layer/PET film was obtained. Incidentally, the first photosensitive layer was in a B stage state. Then, the second photosensitive layer was allowed to form on the first surface of the photosensitive layer, forming a second photosensitive layer. The following direct coating method was carried out in the methods of two types of the transfer method. (Example 1) < Production of the photosensitive dry film resist> Components shown below were mixed, dioxolane in the solid weight (Sc) = as 40% was added and dissolved uniformly. The organic solvent of the first photosensitive layer solution and an organic solvent solution of the second photosensitive layer were prepared. <Organic solvent solution of the first photosensitive layer> (A1) Binder polymer Vinyl polymer containing the carboxyl group (Daicel Cytec Co., Ltd., product name ACA320 weight average molecular weight of 25,000) 100 parts by weight (B1) (Meth)acrylic compound Bisphenol AEO-modified di(meth)acrylate (Daicel Cytec Co., Ltd., product name EB150) 20 parts by weight, bisphenol AEO-modified di(meth)acrylate (Hitachi Chemical Co., Ltd., product name FA321M) 20 parts by weight (C1) Light reaction initiator Bis(2,4,6-trimethylbenzoyl)phenyl phosphine oxide (manufactured by Ciba Specialty Chemicals Co., Ltd. Irgacure 819) 1 part by weight (D1) Flame retardants Resorcinol bis(di-2,6-xylenyl)phosphate (Daihachi Chemical Co., Ltd., product name PX-20030 parts by weight (E1) Dyes and/or pigments ORASOLBLUEGN (manufactured by Ciba Specialty Chemicals Co., Ltd.) 2 parts by weight < Organic solvent solution of the second photosensitive layer> (A2) Binder polymer Vinyl polymer containing carboxyl group (Daicel Cytec Co., Ltd., product name ACÁ320 weight average molecular weight of 25,000) 100 parts by weight (B2) (Meth)acrylic compound Pentaerythritol acrylate (Toagosei Co., Ltd., product name M305) 40 parts by weight (C2) Light reaction initiator An organic solvent solution of bis(2,4,6-trimethylbenzoyl)phenylphosphine oxide (manufactured by Ciba Specialty Chemicals Co., Ltd. Irgacure 819) 1 part by weight of the photosensitive resin composition of the above composition was prepared. Photosensitive dry film resist in a B-stage state of first photosensitive layer 20M thick, second photosensitive layer 5M thick was prepared by the transfer method. Results of evaluation of the physical properties of the resultant photosensitive dry film resist was as follows. Alkali solubility: Dissolved in 30 seconds in 1 wt% aqueous solution of sodium carbonate. [Example 4] < Production of the photosensitive dry film resist> Components shown below were mixed. Dioxolan was added so as to make the solid weight% (Sc) =40% and then uniformly dissolved. The organic solvent solution of the first photosensitive layer and the organic solvent solution of the second photosensitive layer was prepared. <Organic solvent solution of the first photosensitive layer> (A1) Binder polymer Soluble polyimide having carboxyl groups synthesized in Synthesis Example 4 100 parts by weight (B1) (Meth)acrylic compound Epoxy acrylate of modified bisphenol A type (Daicel Cytec Co., Ltd., product name Ebecryl3708) 50 parts by weight (C1) Light reaction initiator Bis(2,4,6-trimethylbenzoyl)phenyl phosphine oxide (Ciba Specialty Chemicals Co., Ltd., product name Irgacure 819) 2 parts by weight (D1) Flame retardants Phosphazene compound (Otsuka Chemical Co., Ltd., product name SPE-100) 15 parts by weight parts by weight (E1) Dyes and/or pigments IRGALITE BLUE GLVO (manufactured by Ciba Specialty Chemicals Co., Ltd.) 2 parts by weight <Organic solvent solution of the second photosensitive layer> (A2) Binder polymer Soluble polyimide having the carboxyl group synthesized in Synthesis Example 4 100 parts by weight (B2) (Meth)acrylic compound Epoxy acrylate of modified bisphenol A type (Daicel Cytec Co., Ltd., product name Ebecryl3708) 50 parts by weight (C2) Light reaction initiator Bis(2,4,6trimethylbenzoyl)phenyl phosphine oxide (Ciba Specialty Chemicals Co., Ltd., product name Irgacure 819) 2 parts by weight (F2) Other components Bisphenol A type epoxy resin (Japan Epoxy Resins Co., Ltd., product name Epikote 828) 10 parts by weight as a epoxy resin, 4,4'-diaminodiphenylmethane (DDM) 1 part by weight as a curing agent. The organic solvent solution of the photosensitive resin composition of the above composition was prepared. Photosensitive dry film resist in a B-stage state first photosensitive layer 20M thick, second photosensitive layer 5M thick was prepared by direct

coating method. <Evaluation results of physical properties> Results of evaluation of the physical properties of the resultant photosensitive dry film resist was as follows. Alkali solubility: Not soluble in 1 wt% aqueous solution of sodium carbonate in 180 seconds. Dissolved in 30 seconds in an aqueous solution of sodium hydroxide. Developing property: hole of $100 \text{Mm} \times 100 \text{Mm}$ square, both are residue without developing holes of $200 \text{Mm} \times 200 \text{Mm}$ angle and are pass. Resolution: 90 Mm adhesion: Pass. Flame retardant: Pass. Electrical Reliability: pass. (Line/space = 100/100 Mm: $5.7 \times 108 \Omega$, Line/space = 25/25 Mm: $3.4 \times 106 \Omega$) solder heat resistance: solder heat resistance pass at $300 \,^{\circ}$ C. Tackiness of the B stage state: Pass in tack-free. Warp: Pass in 3mm.

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18. Single Step

Overview

Steps/Stages

1.1

Notes

Synthesis of Arene Oxides, Reactants: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

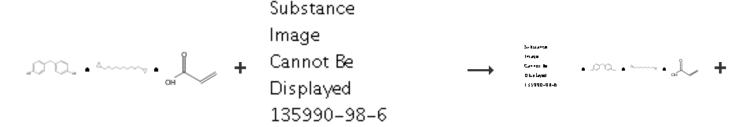
References

Hexamethylphosphorous Triamide

By Harvey, Ronald G.

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

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Overview

Steps/Stages

1.1 R:

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R:Al₂O₃, C:75980-60-8, C:931-36-2

Notes

photochemical, UV irradiation, conductive particle containing nickel and gold coated onto divinylbenzene resin used, Reactants: 2, Reagents: 2, Catalysts: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Anisotropically conductive pastes with controlled viscosity, bonded structures, and their manufacture using them

By Ishizawa, Eisuke et al

From Jpn. Kokai Tokkyo Koho, 2013033734, 14 Feb 2013

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20. 2 Steps



[Step 2.1]

Overview

Steps/Stages

1.1

1.2

2.1 R:

Substance

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Notes

1) no experimental detail, 2) photochemical, UV irradiation, conductive particle containing nickel and gold coated onto divinylbenzene resin used, Reactants: 4, Reagents: 2, Catalysts: 2, Steps: 2, Stages: 3, Most stages in any one step: 2

References

Anisotropically conductive pastes with controlled viscosity, bonded structures, and their manufacture using them

By Ishizawa, Eisuke et al

From Jpn. Kokai Tokkyo Koho, 2013033734, 14 Feb 2013

R:Al₂O₃, C:75980-60-8, C:931-36-2

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Overview

Steps/Stages

1.1 C:330-54-1, 2 h, 135°C

Notes

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

References

Manufacture of pressure tank from fiberreinforced plastic

By Okamoto, Satoshi et al From Jpn. Kokai Tokkyo Koho, 2016017110, 01 Feb 2016

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22. Single Step

Overview

Steps/Stages

1.1 2 h, 70°C 1.2 C:104558-95-4

Notes

photochemical (stage 2), ultraviolet irradiation (stage 2), UV-lamp used, 120W (stage 2), Reactants: 3, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Radiation-curable coating compositions containing epoxy resins, cationic photopolymerization catalysts, and (meth)acrylates

By Yamazaki, Tetsuya et al From Jpn. Kokai Tokkyo Koho, 2013194120, 30 Sep 2013

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23. Single Step

Overview

Steps/Stages

1.1 R:mCPBA, S:CICH₂CH₂CI, 3 h, reflux

Notes

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

References

Substituted aryl sulfone derivatives as calcium channel blockers

By Chakravarty, Prasun K. and Shao, Pengcheng Patrick

From PCT Int. Appl., 2010036596, 01 Apr 2010

Experimental Procedure

Step 3 : 2-(1-methyl-1-{[3-(trifluoromethyl)phenyl]sulfonyl}ethyl)oxirane To a solution of 1,1-dimethylprop-2-en-1-yl 3-(trifluoromethyl)phenyl sulfone (0.278 g, 1 mmol.) in 1,2-dichloroethane (1 mL) was added *m*-CPBA (85% purity, 0.404 g, 2 mmol). The resulting reaction mixture was refluxed for 3 h until TLC indicated the starting material was completely consumed. It was cooled to 0 °C, treated with Na₂S₂O₃ (saturated solution, 10 mL, to remove excess *m*-CPBA) and extracted with DCM (20 mLx3). Organics were washed with saturated NaHCO₃, dried and concentrated. Crude product was purified by column chromatography (PE:EA-10:1-3:1) to afford title compound. ^1H NMR (CDCl₃) δ : 8.10(s, 1H), 8.04(d, J = 8.0 Hz, 1H), 7.87(d, J = 8.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H)3.30-3.32 (m, 1 H), 2.64 (t, J = 4.0 Hz, 1H), 2.36-2.38 (m, 1 H), 1.27 (s, 3 H), 1.18 (s, 3 H).

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24. 2 Steps

Overview

Steps/Stages Notes

- 1.1 R:(Me₃Si)₂NH •Na, S:THF, -78°C; 30 min, -78°C
- 1.2 2 h, -78°C \rightarrow rt; rt \rightarrow -78°C; 30 min, -78°C; -78°C \rightarrow rt; overnight, rt
- 2.1 R:mCPBA, S:CICH₂CH₂CI, 3 h, reflux

1) incremental addition of reactant and agent(methyl iodide sodium hexamethyldisilazide), Reactants: 2, Reagents: 2, Solvents: 2, Steps: 2, Stages: 3, Most stages in any one step: 2

References

Substituted aryl sulfone derivatives as calcium channel blockers

By Chakravarty, Prasun K. and Shao, Pengcheng Patrick

From PCT Int. Appl., 2010036596, 01 Apr 2010

Experimental Procedure

Step 1

Step 2: 1,1-dimethylprop-2-en-1-yl 3-(trifiuoromethyl)phenyl sulfone To a stirred solution of 1-(allylsulfonyl)-3-(trifluoromethyl)benzene (1.25 g, 5 mmol) in THF (20 mL) was added NaHMDS dropwise (2 mol / L in THF, 2.6 mL, 5.05 mmol) at -78 °C . The resulting reaction mixture was stirred for 30 min. Mel (0.8 g, 5.05 mmol) was added. Reaction mixture was allowed to warm up to ambient temperature for 2 hrs. Then it was cooled to -78 °C. NaHMDS (2 mol / L in THF, 2.6 mL, 5.05 mmol) was added and the resulting reaction mixture was stirred for 30 min. Mel (0.8 g, 5.05 mmol) was added. Reaction mixture was allowed to warm up to ambient temperature and stirred overnight. Brine (10 mL) was added. Reaction mixture was extracted with ethyl acetate (30 mLx3). Organics were dried over anhydrous Na₂SO₄, concentrated. Crude product was purified by column chromatography (PE:EA=10:1-3:1) to afford title compound . ¹H NMR (CDCl₃) δ : 8.06(s, 1H), 8.00(d, J = 1.6 Hz, 1H), 7.87(d, J = 8.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H), 6.02 (dd, J = 16.8 Hz, 11.2 Hz, 1H), 5.28(d, J = 11.2 Hz, 1H), 5.05(d, J = 16.8 Hz, 1H), 1.44 (s, 6 H).

Step 2

Step 3 : 2-(1-methyl-1-{[3-(trifluoromethyl)phenyl]sulfonyl}ethyl)oxirane To a solution of 1,1-dimethylprop-2-en-1-yl 3-(trifluoromethyl)phenyl sulfone (0.278 g, 1 mmol.) in 1,2-dichloroethane (1 mL) was added m-CPBA (85% purity, 0.404 g, 2 mmol). The resulting reaction mixture was refluxed for 3 h until TLC indicated the starting material was completely consumed. It was cooled to 0 °C, treated with Na₂S₂O₃ (saturated solution, 10 mL, to remove excess m-CPBA) and extracted with DCM (20 mLx3). Organics were washed with saturated NaHCO₃, dried and concentrated. Crude product was purified by column chromatography (PE:EA-10:1-3:1) to afford title compound. 1 H NMR (CDCl₃) δ : 8.10(s, 1H), 8.04(d, J = 8.0 Hz, 1H), 7.87(d, J = 8.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H)3.30-3.32 (m, 1 H), 2.64 (t, J = 4.0 Hz, 1H), 2.36-2.38 (m, 1 H), 1.27 (s, 3 H), 1.18 (s, 3 H).

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Overview

Steps/Stages

1.1

Notes

Notes

Synthesis of Arene Oxides, multistep transformation, Reactants: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Hexamethylphosphorous Triamide

By Harvey, Ronald G.

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

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26. 3 Steps

Overview

Steps/Stages

- 1.1 R:BuLi, S:THF, S:Me(CH₂)₄Me, -78°C; 2 h, -78°C
- 1.2 R:SO₂, -78°C
- 1.3 S:H₂O, -78°C
- 1.4 S:DMF, 2 h, 50°C; overnight, rt
- 2.1 R:(Me₃Si)₂NH •Na, S:THF, -78°C; 30 min, -78°C
- 2.2 2 h, -78°C \rightarrow rt; rt \rightarrow -78°C; 30 min, -78°C; -78°C \rightarrow rt; overnight, rt
- 3.1 R:mCPBA, S:CICH₂CH₂Cl, 3 h, reflux

2) incremental addition of reactant and agent(methyl iodide sodium hexamethyldisilazide), Reactants: 3, Reagents: 4, Solvents: 5, Steps: 3, Stages: 7, Most stages in any one step: 4

References

Substituted aryl sulfone derivatives as calcium channel blockers

By Chakravarty, Prasun K. and Shao, Pengcheng Patrick

From PCT Int. Appl., 2010036596, 01 Apr 2010

Experimental Procedure

Step 1

Step 1 : 1-(allylsulfonyl)-3-(trifluoromethyl)benzene To a stirred solution of 1-bromo-3-(trifluoromethyl)benzene (22.4 g, 0.10 mol) in THF (200 mL) was added dropwise BuLi (2.5 M in hexane, 60 mL, 0.15 mol) at -78 °C with dry ice bath. After additional, the reaction mixture was stirred at -78 °C for 2 hrs. Then, SO₂ was purged into the reaction mixture until the green color turned to yellow color. H_2O (10 mL) was added to quench the reaction. Volatiles were evaporated under vacuum and DMF (250 mL) was added, followed by allyl bromide (18 g, 0.15 mol). The resulting mixture was heated at 50°C for 2 hrs, and then stirred at ambient temperature overnight The reaction mixture was poured into water (200 mL), extracted with ether (100 mL x3), dried over anhydrous Na_2SO_4 , evaporated to afford the crude product. It was further purified by column chromatography (Petroleum ether: EtOAcl 0: 1 to 3: 1) to afford title compound. ¹H NMR (CDCl₃) δ : 8.09(s, 1H), 8.03(d, J = 7.6 Hz, 1H), 7.87(d, J = 8Hz, 1H), 7.67-7.71 (m, 1H), 5.68-5.81 (m, 1H), 5.31(d, J = 10 Hz, 1H), 5.11(d, J = 17.2 Hz, 1H), 3.82(d, J = 7.2 Hz, 2H).

Step 2

Step 2: 1,1-dimethylprop-2-en-1-yl 3-(trifiuoromethyl)phenyl sulfone To a stirred solution of 1-(allylsulfonyl)-3-(trifluoromethyl)benzene (1.25 g, 5 mmol) in THF (20 mL) was added NaHMDS dropwise (2 mol / L in THF, 2.6 mL, 5.05 mmol) at -78 °C . The resulting reaction mixture was stirred for 30 min. Mel (0.8 g, 5.05 mmol) was added. Reaction mixture was allowed to warm up to ambient temperature for 2 hrs. Then it was cooled to -78 °C. NaHMDS (2 mol / L in THF, 2.6 mL, 5.05 mmol) was added and the resulting reaction mixture was stirred for 30 min. Mel (0.8 g, 5.05 mmol) was added. Reaction mixture was allowed to warm up to ambient temperature and stirred overnight. Brine (10 mL) was added. Reaction mixture was extracted with ethyl acetate (30 mLx3). Organics were dried over anhydrous Na₂SO₄, concentrated. Crude product was purified by column chromatography (PE:EA=10:1-3:1) to afford title compound . ¹H NMR (CDCl₃) δ : 8.06(s, 1H), 8.00(d, J = 1.6 Hz, 1H), 7.87(d, J = 8.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H), 6.02 (dd, J = 16.8 Hz, 11.2 Hz, 1H), 5.28(d, J = 11.2 Hz, 1H), 5.05(d, J = 16.8 Hz, 1H), 1.44 (s, 6 H).

Sten 3

Step 3 : 2-(1-methyl-1-{[3-(trifluoromethyl)phenyl]sulfonyl}ethyl)oxirane To a solution of 1,1-dimethylprop-2-en-1-yl 3-(trifluoromethyl)phenyl sulfone (0.278 g, 1 mmol.) in 1,2-dichloroethane (1 mL) was added *m*-CPBA (85% purity, 0.404 g, 2 mmol). The resulting reaction mixture was refluxed for 3 h until TLC indicated the starting material was completely consumed. It was cooled to 0 °C, treated with Na₂S₂O₃ (saturated solution, 10 mL, to remove excess *m*-CPBA) and extracted with DCM (20 mLx3). Organics were washed with saturated NaHCO₃, dried and concentrated. Crude product was purified by column chromatography (PE:EA-10:1-3:1) to afford title compound. ¹H NMR (CDCl₃) δ : 8.10(s, 1H), 8.04(d, J = 8.0 Hz, 1H), 7.87(d, J = 8.0 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H)3.30-3.32 (m, 1 H), 2.64 (t, J = 4.0 Hz, 1H), 2.36-2.38 (m, 1 H), 1.27 (s, 3 H), 1.18 (s, 3 H).

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Overview

Steps/Stages

1.1 C:24650-42-8, S:PhMe

Notes

photochemical, UV irradiation, high pressure mercury lamp used, coated onto silicone release agent treated polyethylene terephthalate film, Reactants: 3, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Adhesive sheet

By Hirayama, Takamasa and Kitayama, Kazuhiro

From PCT Int. Appl., 2014142194, 18 Sep 2014

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28. 2 Steps

[Step 2.1]

[Step 2.1]

Substance

Cannot Be

Image

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1627110-39-7

Overview

Steps/Stages

- $C:(PhCO_2)_2$, S:PhMe, heated C:24650-42-8, S:PhMe
- 2.1

Notes

2) photochemical, UV irradiation, high pressure mercury lamp used, coated onto silicone release agent treated polyethylene terephthalate film, Reactants: 6, Ćatalysts: 2, Solvents: 1, Steps: 2, Stages: 2, Most stages in any one step: 1

References

Adhesive sheet

By Hirayama, Takamasa and Kitayama, Kazuhiro

From PCT Int. Appl., 2014142194, 18 Sep 2014

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29. Single Step

polymers

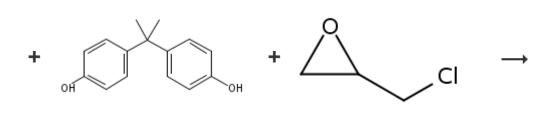
Substance

Image

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Displayed

1243541-61-8



Overview

Steps/Stages

1.1 R:

C:Dabco, S:EtC(=O)Me, rt; 6 h, 180°C

Notes

photochemical, ultraviolet irradiation, Reactants: 6, Reagents: 1, Catalysts: 1, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Adhesive phenoxy resin composition for electronic component and adhesive tape

By Hong, Seung U. et al From Repub. Korea, 1481710, 21 Jan 2015

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Substance

Steps/Stages

Notes

1.1 R:H₂PtCl₆, S:Me₂CHOH, 2 h, 80°C

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

Page 24

References

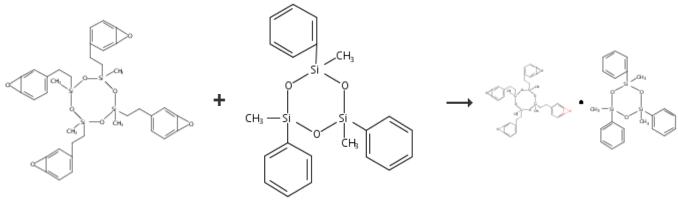
Epoxy-containing silicone resin with high light transmittance and high refractive index and manufacture method

By Zhang, Yingqiang et al

From Faming Zhuanli Shenqing, 104672457, 03 Jun 2015

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31. Single Step



polysiloxanes

trimethylsilyl-terminated

Overview

Steps/Stages

1.1 R:Me₃SiOSiMe₃ (reaction products with polysiloxane), $40^{\circ}C \rightarrow 90^{\circ}C$; 10 h, $90^{\circ}C$

Notes

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

References

Epoxy-containing silicone resin with high light transmittance and high refractive index and manufacture method

By Zhang, Yingqiang et al

From Faming Zhuanli Shenqing, 104672457, 03 Jun 2015

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polysiloxanes

trimethylsilyl-terminated

Overview

Steps/Stages

1.1 R:Me₃SiOSiMe₃ (reaction products with polysiloxane), 40°C → 80°C; 4 h, 80°C

Notes

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

References

Epoxy-containing silicone resin with high light transmittance and high refractive index and manufacture method

By Zhang, Yingqiang et al

From Faming Zhuanli Shenqing, 104672457, 03 Jun 2015

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33. 2 Steps

trimethylsilyl-terminated

Overview

Steps/Stages Notes

1.1 R:H₂PtCl₆, S:Me₂CHOH, 2 h, 80°C

2.1 R:Me₃SiOSiMe₃ (reaction products with polysiloxane), $40^{\circ}C \rightarrow 90^{\circ}C$; 10 h, $90^{\circ}C$

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

References

Epoxy-containing silicone resin with high light transmittance and high refractive index and manufacture method

By Zhang, Yingqiang et al

From Faming Zhuanli Shenqing, 104672457, 03 Jun 2015

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34. 2 Steps

trimethylsilyl-terminated

Overview

Steps/Stages

- 1.1 R:H₂PtCl₆, S:Me₂CHOH, 2 h, 80°C
- 2.1 R:Me₃SiOSiMe₃ (reaction products with polysiloxane), $40^{\circ}C \rightarrow 80^{\circ}C$; 4 h, $80^{\circ}C$

Notes

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

References

Epoxy-containing silicone resin with high light transmittance and high refractive index and manufacture method

By Zhang, Yingqiang et al

From Faming Zhuanli Shenqing, 104672457, 03 Jun 2015

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Overview

Steps/Stages

1.1 S:PhMe, 12 h, rt

Notes

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

89%

References

Structure and photophysics of near-infrared emissive ytterbium(III) monoporphyrinate acetate complexes having neutral bidentate ligands

By He, Hongshan et al

From Dalton Transactions, (36), 7454-7461; 2009

Reaction Protocol

Procedure

- 1. Add an excess of 5,6'-epoxy-5,6-dihydroxy-1,10-phenanthroline (24.5 mg, 0.12 mmol) to a toluene solution (20 mL) of the reactant (24.6 mg, 0.027 mmol).
- 2. Stir the solution magnetically at room temperature for 12 hours.

View more...

Available Experimental Data Crystal Structure Data, Elemental Analysis, State

View with MethodsNow

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