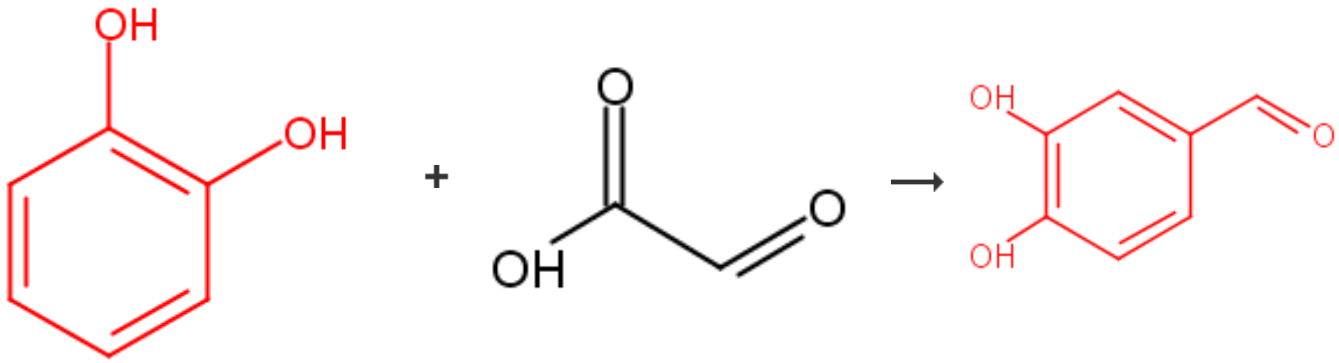
1. Single Step

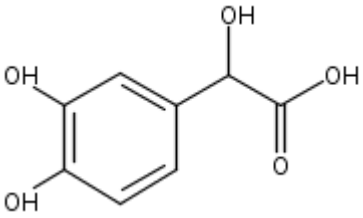


96%

Overview

Steps/Stages

1.1 R:



R:CuO, R:NaOH, S:H_2O, rt → 100°C; 7.5 h, 100°C

1.2 R:H_2SO_4, S:H_2O, pH 1.5-2

Notes

optimization study, optimized on temperature and reaction time, Reactants: 2, Reagents: 4, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

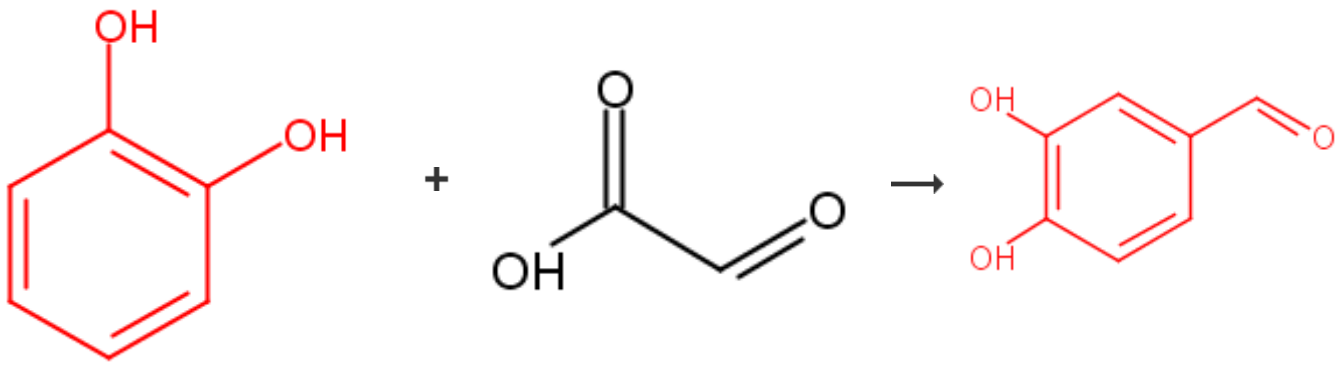
References

Process for preparation of protocatechuic aldehyde via oxidation with the lattice oxygen in copper oxide

By Liu, Yu et al

From Faming Zhuanli Shenqing, 103012091, 03 Apr 2013

2. Single Step



91%

Overview

Steps/Stages

Notes

1.1

1.2 R:NaOH, R:O_2, C:Mo, C:Mn, C:Al, C:Zn, C:Na, C:Cu, C:Fe, S:H_2O, 7 h, 90°C

optimization study (optimized on catalyst reactant ratio, reaction time and air flow), Reactants: 2, Reagents: 2, Catalysts: 7, Solvents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

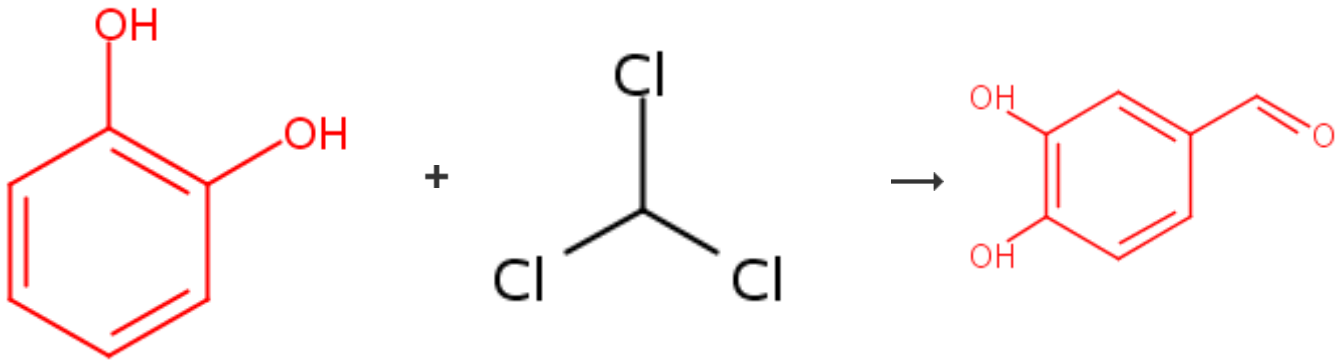
References

Preparation of 3,4-dihydroxybenzaldehyde by air catalytic oxidation method

By Li, Yaoxian et al

From Faming Zhuanli Shenqing Gongkai Shuomingshu, 101676253, 24 Mar 2010

3. Single Step



96%

Overview

Steps/Stages

1.1 C:107745-73-3, S:H_2O, S:MeOH

Notes

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

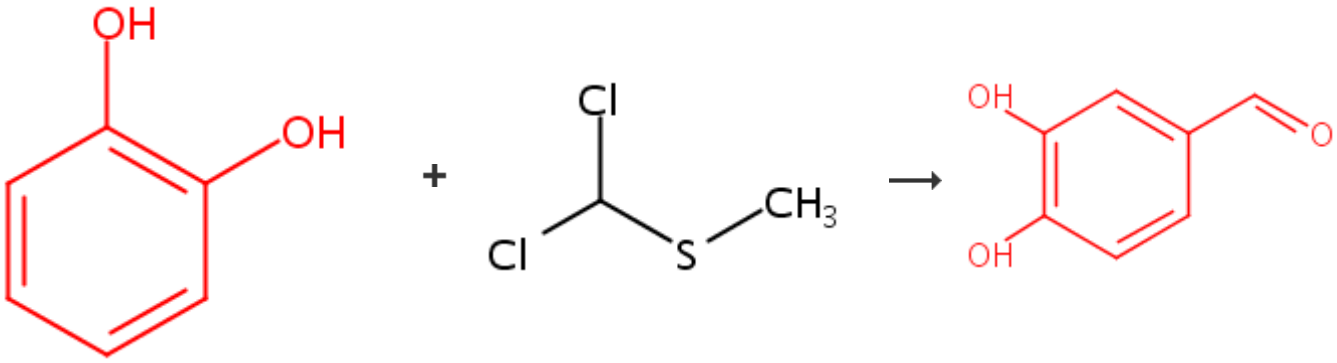
References

β-Cyclodextrin mediated regioselective photo-Reimer-Tiemann reaction of phenols

By Ravichandran, Ramaswamy

From Journal of Molecular Catalysis A: Chemical, 130(3), L205-L207; 1998

4. Single Step



83%

Overview

Steps/Stages

1.1 C:$SnCl_4$, S:CH_2Cl_2, 1 s, -20°C; 1 min, -20°C

1.2 R:HCl, S:H_2O, -20°C; -20°C → rt; 30 min, rt

Notes

regioselective, 4A molecular sieves added in first stage, Friedel-Crafts formylation, conversion = 88%, radiochemical purity = 99%, optimized on solvent, reaction temperature and addition time of catalyst, Reactants: 2, Reagents: 1, Catalysts: 1, Solvents: 2, Steps: 1, Stages: 2, Most stages in any one step: 2

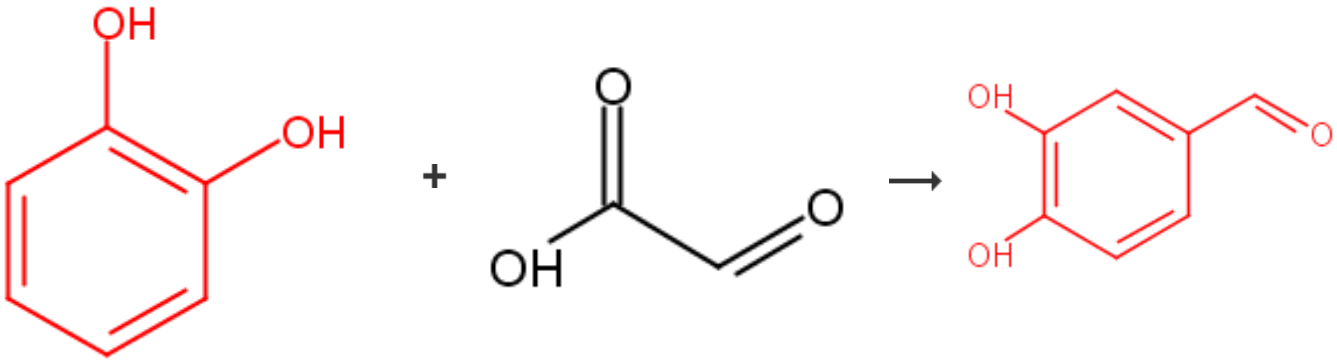
References

Synthesis of [uniformly ring-14C]-labelled 4-hydroxybenzaldehyde, vanillin, and protocatechualdehyde

By Ji, Rong and Schaeffer, Andreas

From Journal of Labelled Compounds & Radiopharmaceuticals, 47(4), 209-216; 2004

5. Single Step



76%

Overview

Steps/Stages

Notes

1.1 R:NaOH, S:H_2O, rt → 40°C; 40°C; 10 h, 40°C
1.2 acidify
1.3 R:NaOH, R:O_2, S:H_2O, 7 h, reflux; reflux → rt

unspecified reagent used to adjust pH in stage 2, base assumed in stage 3, optimization study, optimized on temperature, catalyst amount and time, unspecified catalyst used in stage 3, Reactants: 2, Reagents: 2, Solvents: 1, Steps: 1, Stages: 3, Most stages in any one step: 3

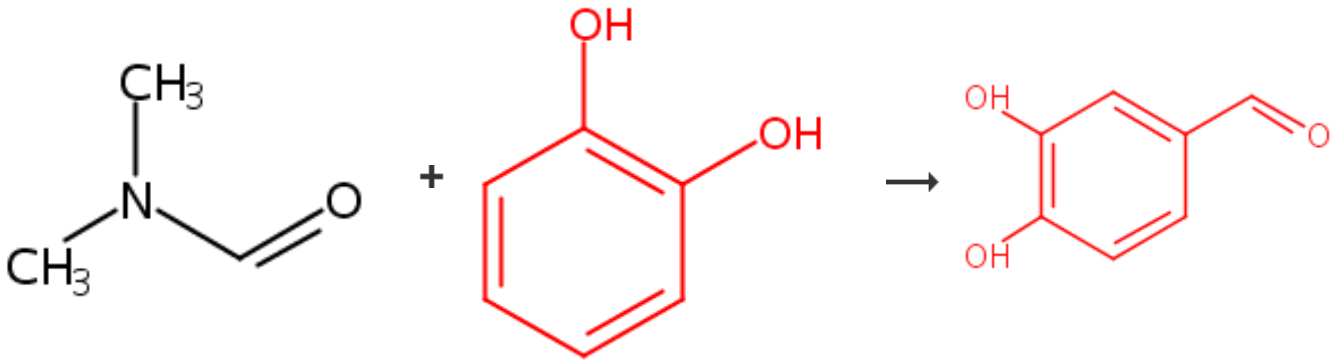
References

Synthesis of 3,4-dihydroxybenzaldehyde by air-catalytic oxidation

By Wang, Hengguo et al

From Yingyong Huagong, 37(11), 1278-1280; 2008

6. Single Step



80%

Overview

Steps/Stages

1.1 R:$POCl_3$

Notes

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

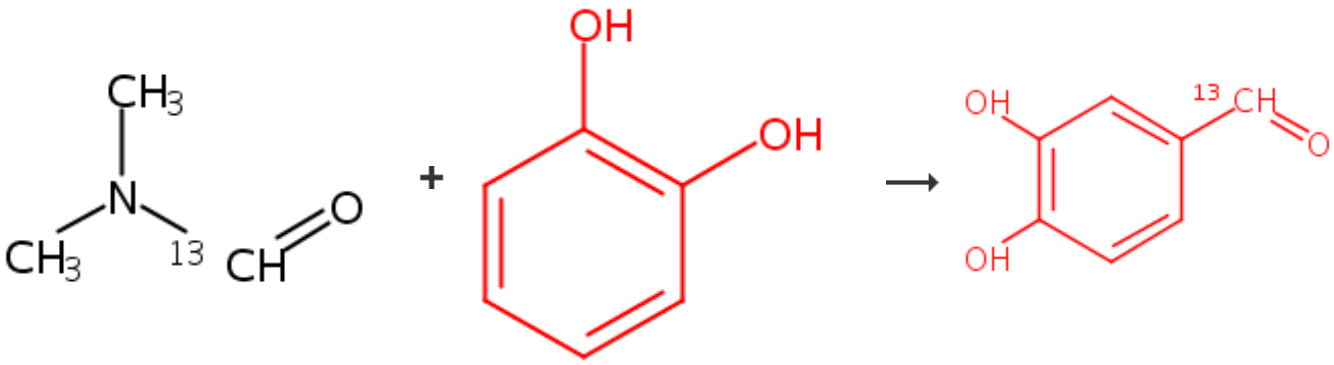
References

Preparation of 3,4-dihydroxybenzaldehyde or 3-alkoxy-4-hydroxybenzaldehyde by Vilsmeier reaction

By Kawarazaki, Naoki et al

From Jpn. Kokai Tokkyo Koho, 10001451, 06 Jan 1998

7. Single Step



49%

Overview

Steps/Stages

1.1 R:$POCl_3$, cooled; 30 min, rt
1.2 4 h, 120°C; 120°C → rt
1.3 S:H_2O, 1 h, rt

Notes

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

References

1H NMR Probe for in Situ Monitoring of Dopamine Metabolism and Its Application to Inhibitor Screening

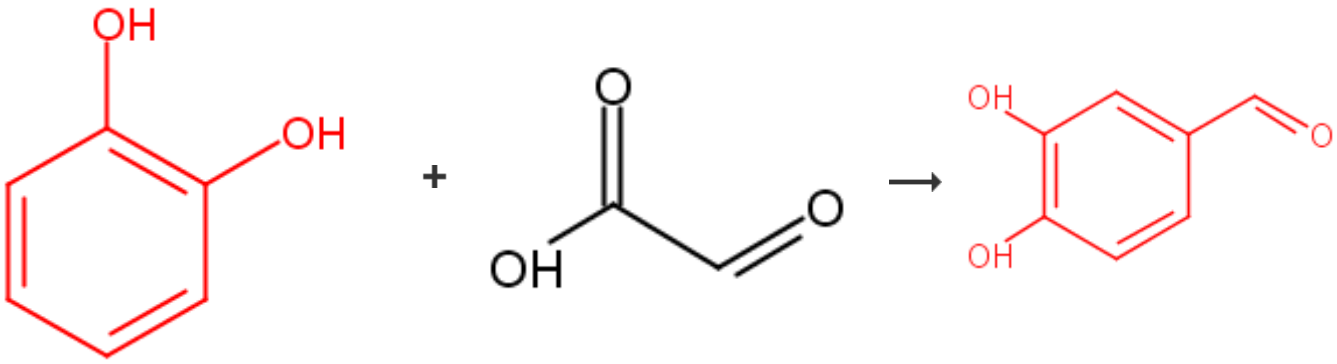
By Ueki, Ryosuke et al

From Journal of the American Chemical Society, 134(30), 12398-12401; 2012

Experimental Procedure

Synthesis of 3,4-dihydroxybenzaldehyde-^{13}C. Phosphoryl chloride (2.10 g, 13.6 mmol) was added dropwise to dimethylformamide-carbonyl-13C (1.00 g, 13.5 mmol) on ice bath and the mixture was stirred at room temperature. After 30 min, catechol (1.00 g, 9.1 mmol) was added to the reaction solution, and the solution was stirred at 120 °C for 4 h. After cooling to the room temperature, water (20 mL) was added and the mixture was further stirred for 1 h. This was evaporated under vacuum, resulting in a dark oily residue. The residue was purified using silica gel column chromatography (eluent: chloroform:methanol = 20:1) to give 3,4-dihydroxybenzaldehyde-^{13}C. Yield (614 mg, 49%). ^{1}H NMR (CD_3OD, 400 Hz) δ = 6.90 (d, *J* = 8.0 Hz, 1H, aromatic), 7.26-7.30 (m, 2H, aromatics), 9.66 (d, *J* = 172.0 Hz, 1H); ^{13}C NMR (CD_3OD, 100 MHz) δ = 193.1; HRMS(FAB): *m/z* calc. for $C_{613}CH_7O_3^+$ $[M+H]^+$ = 140.0423, found = 140.0411.

8. 2 Steps



Overview

Steps/Stages

Notes

1.1 R:NaOH, S:H_2O

2.1 R:$Cu(OH)_2$

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

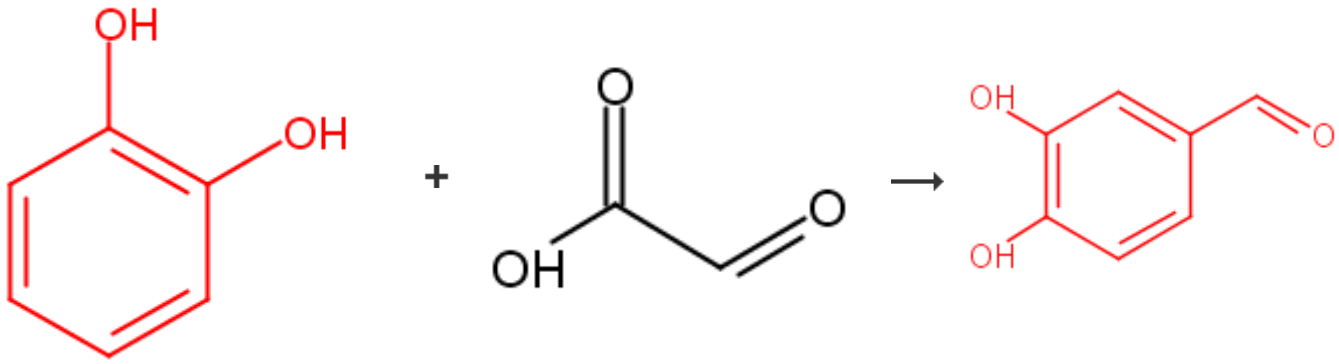
References

Process for the preparation of 3,4-dihydroxybenzaldehyde

By Wang, Junhua

From Faming Zhuanli Shenqing Gongkai Shuomingshu, 1508112, 30 Jun 2004

9. 2 Steps



Overview

Steps/Stages

1.1 R:NaOH, R:Al_2O_3, S:H_2O

1.2

1.3 R:HCl

1.4 S:AcOEt

2.1 R:$CuCl_2$, S:H_2O, S:AcOEt

Notes

1) chemoselective, Reactants: 2, Reagents: 4, Solvents: 2, Steps: 2, Stages: 5, Most stages in any one step: 4

References

High Selectivity in the Oxidation of Mandelic Acid Derivatives and in O-Methylation of Protocatechualdehyde: New Processes for Synthesis of Vanillin, iso-Vanillin, and Heliotropin

By Bjorsvik, Hans-Rene et al

From Organic Process Research & Development, 4(6), 534-543; 2000

Experimental Procedure

Step 1

3,4-Dihydroxy Mandelic Acid 1 (Optimised Procedure). Catechol (5.00 g, 45.41 mmol) was dissolved in aqueous NaOH (3.21 g, 80.3 mmol in 55.0 mL of water) followed by addition of Al_2O_3 (2.04 g, 20 mmol). After 5 min glyoxylic acid (7.10 g of 50% aqueous solution, 48.0 mmol) was added to the reaction mixture, and the mixture was heated at 60 °C for 24 h under vigorous stirring. The reaction mixture was then allowed to precipitate for 10 min. and filtered to remove Al_2O_3. The obtained filter cake was washed with 1 M NaOH (20 mL). The basic washing water was combined with the water solution, and this was acidified to pH 3-4 with 6.0 mL of 37% HCl and extracted with ethyl acetate to recover the unreacted catechol (1.2 g). The aqueous solution was further acidified to pH 1 by 2 mL of concentrated HCl and extracted with ethyl acetate to isolate the mandelic acid derivative (5.1 g, 28.08 mmol). Conversion 77.5%, selectivity 90.5%.

Step 2
Protocatechualdehyde 13. 3,4-Dihydroxy mandelic acid (2 g, 10.86 mmol) was dissolved in 140 mL of ethyl acetate, and 11.11 g of $CuCl_2 \cdot 2H_2O$ was dissolved in 30 mL of water. The two-phase system was vigorously stirred and heated at 60 °C for 5 h under nitrogen atmosphere. The organic phase was separated, and the solvent was removed. The HPLC analysis revealed a complete conversion of the mandelic acid derivative and the yield of protocatechualdehyde of 96%. The copper salt aqueous solution/suspension was recycled by oxidising Cu(I) to Cu(II) by air after the removal of the organic phase; the results were substantially unchanged.

Reaction Protocol

Procedure

1. Dissolve catechol (5.00 g, 45.41 mmol) in aqueous NaOH (3.21 g, 80.3 mmol in 55.0 mL of water) followed by addition of Al_2O_3 (2.04 g, 20 mmol).
2. After 5 minutes, add glyoxylic acid (7.10 g of 50% aqueous solution, 48.0 mmol) to the reaction mixture.

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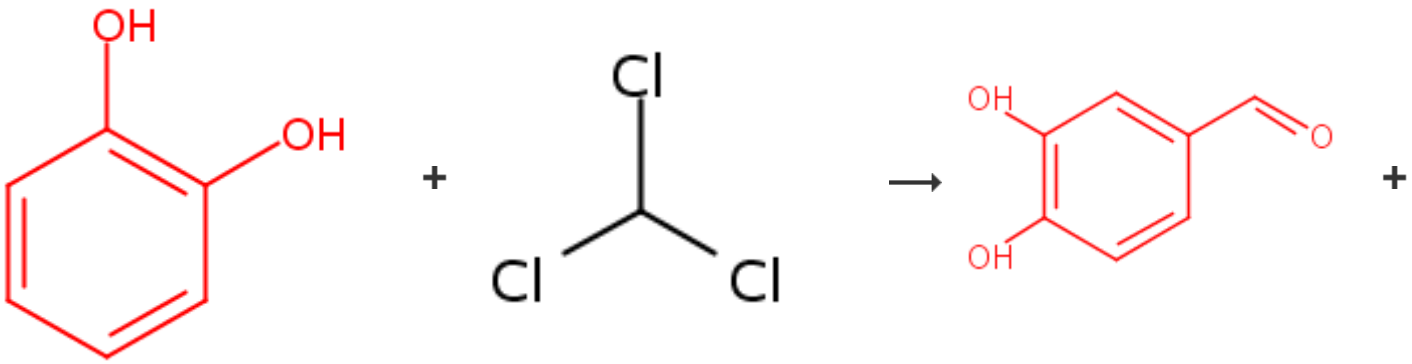
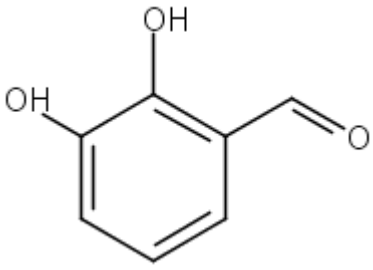
10. Single Step





Overview

Steps/Stages **Notes**

1.1 S:$CHCl_3$, S:H_2O

regioselective, Alkali, Aq. Hydroxide, CHCl3, Yield 18%, Alkylation, C-Alkylation, C-Formylation, Carbene intermediate, Cleavage, Hydrolysis, Selective, Reactants: 2, Solvents: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

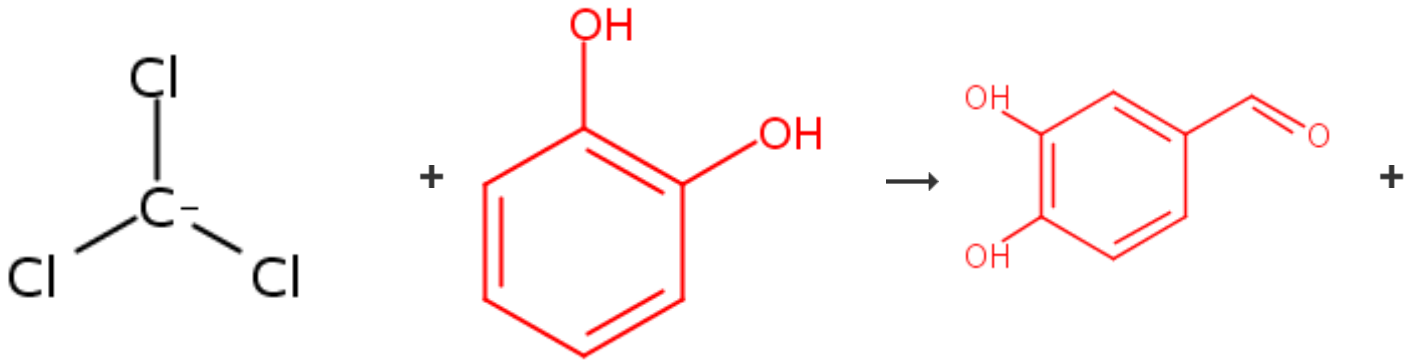
References

The Reimer-Tiemann reaction

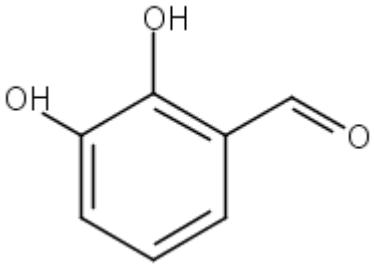
By Wynberg, Hans and Meijer, Egbert W.

From Organic Reactions (Hoboken, NJ, United States), 28, No pp. given; 1982

11. 2 Steps



[Step 2.1]



Overview

Steps/Stages

1.1 R:H_2O

2.1 S:$CHCl_3$, S:H_2O

Notes

1) H2O, 2) regioselective, Alkali, Aq. Hydroxide, CHCl3, Yield 18%, Alkylation, C-Alkylation, C-Formylation, Carbene intermediate, Cleavage, Hydrolysis, Selective, Reactants: 2, Reagents: 1, Solvents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

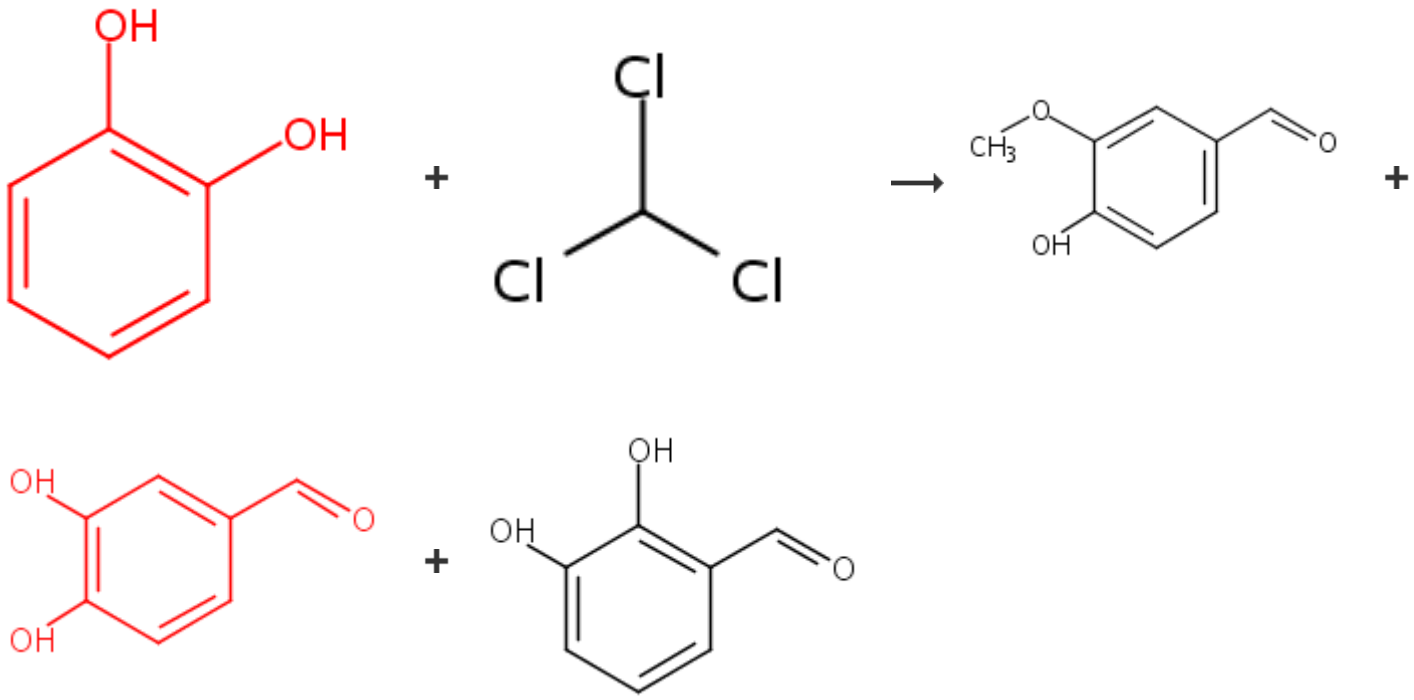
References

The Reimer-Tiemann reaction

By Wynberg, Hans and Meijer, Egbert W.

From Organic Reactions (Hoboken, NJ, United States), 28, No pp. given; 1982

12. Single Step



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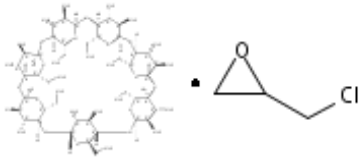
Overview

Steps/Stages

1.1 R:KOH, R:beta-Cyclodextrin

R:



S:H_2O

Notes

REACTANT MIXTURE CONTAINING CATECHOL AND GUAIACOL, REGIOSELECTIVE, Reactants: 2, Reagents: 3, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

Reimer-Tiemann reactions of guaiacol and catechol in the presence of β-cyclodextrin

By Divakar, S. et al

From Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry, 31B(8), 543-6; 1992