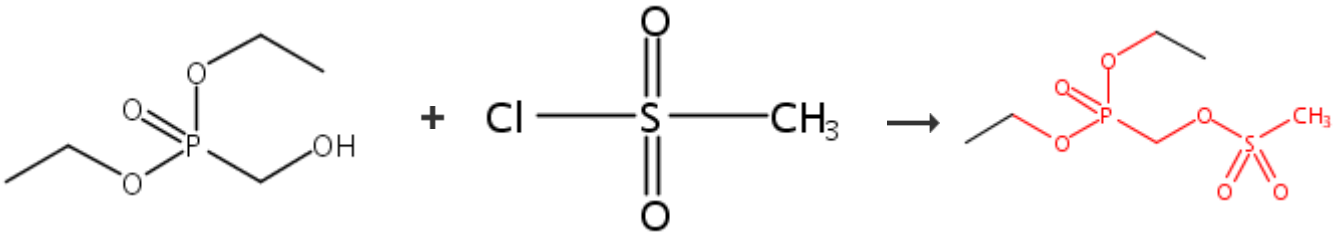
1. Single Step



90%

Overview

Steps/Stages

1.1 R:Et_3N, S:CH_2Cl_2, -10°C; 2 h, rt

Notes

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

References

Truncated phosphonated C-1'-branched N,O-nucleosides: A new class of antiviral agents

By Romeo, Roberto et al

From Bioorganic & Medicinal Chemistry, 20(11), 3652-3657; 2012

Reaction Protocol

Procedure 1. Add triethylamine (2.4 mL, 17 mmol) and mesyl chloride (1.45 g, 12 mmol) to a solution of diethyl hydroxymethyl phosphonate (500 mg, 2.97 mmol) in dry dichloromethane (20 mL) at -10 °C.
2. Stir the reaction mixture at room temperature for 2 hours.

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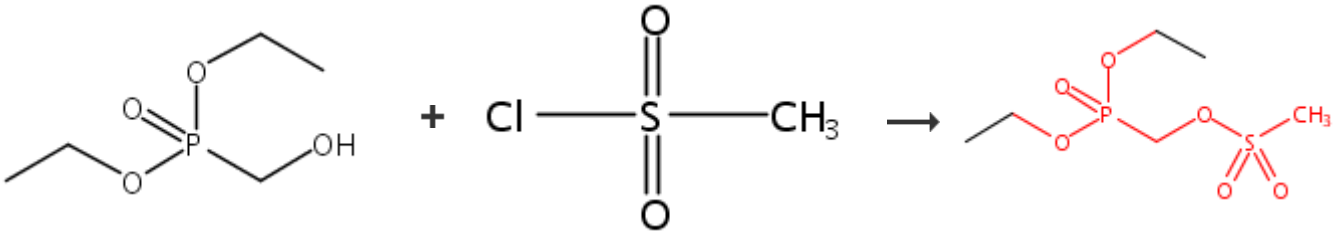
2. Single Step



80%

Overview

Steps/Stages

Notes

1.1 R:Et_3N, S:CH_2Cl_2, 0°C; 0°C → rt; 4 h, rt
1.2 S:H_2O, rt

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

References

An efficient synthesis of phosphonate derivatives of 1,2-disubstituted carbocyclic purine nucleosides with a cyclopentane ring

By Besada, Pedro et al

From Synthesis, (15), 2363-2368; 2008

Experimental Procedure

Diethyl Methanesulfonyloxymethanephosphonate (14b) To a solution of diethyl hydroxymethylphosphonate15 (100 mg, 0.60 mmol) in CH_2Cl_2 (3 mL) was added dropwise MsCl (74 mg, 50 mL, 0.65 mmol) and Et_3N (0.13 mL) at 0 °C. The mixture was stirred and the temperature was allowed to reach r.t. gradually and then the stirring was continued for an additional 4 h. After adding H_2O (3 mL), the product was extracted with CH_2Cl_2 (2 × 3 mL). The combined organic layers were washed with sat. aq $NaHCO_3$ (9 mL), brine (9 mL), and dried (Na_2SO_4). The solvent was removed in vacuo to obtain **14b** which was used directly for reactions in Procedure A without further purification. Colorless oil, yield 117 mg, 80%. R_f = 0.41 (CH_2Cl_2-MeOH, 95:5). ^{1}H NMR ($CDCl_3$): δ = 4.42 (d, J = 8.9 Hz, 2 H, CH_2P), 4.22 (m, 4 H, 2 × CH_2CH$_3$), 3.13 (s, 3 H, CH_3), 1.38 (t, J = 7.1 Hz, 3 H, 2 × CH$_2$CH_3). ^{13}C NMR ($CDCl_3$): δ = 63.8 (d, J = 6.4 Hz, CH_2CH_3), 61.5 (d, J = 169.7 Hz, CH_2P), 38.4 (CH_3), 16.8 (d, J = 5.7 Hz, CH_2CH_3). MS (EI): m/z (%) = 247 ([M + 1]$^+$, 6), 219 (M$^+$ - C_2H_3, 32), 201 (M$^+$ - C_2H_5O, 10), 191 (M$^+$ - C_4H_7, 18), 188 (17), 173 (21), 167 (M$^+$ - CH_3SO_2, 18), 160 (34), 137 (M$^+$ - $C_2H_5SO_3$, 63), 109 (M$^+$ - $C_4H_9SO_3$, 100), 81 (23), 79 (25), 65 (14).

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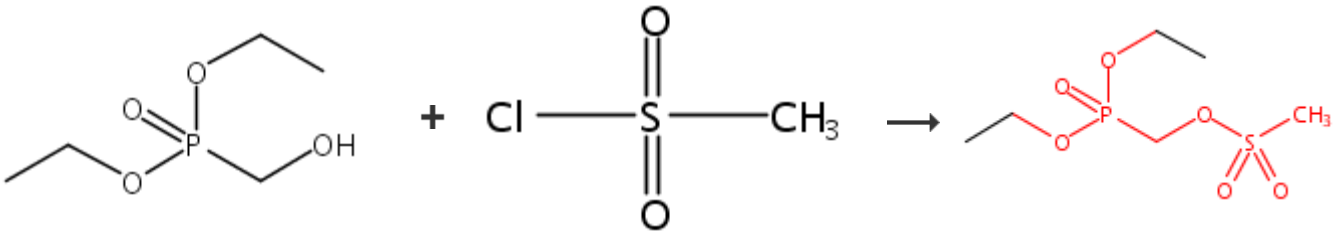
3. Single Step



Overview

Steps/Stages

1.1 R:Et_3N, S:CH_2Cl_2

Notes

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

References

Synthesis and screening of novel inositol phosphonate derivatives for anticancer functions in vitro

By Chen, Wen-Bin et al

From Chinese Chemical Letters, 26(3), 329-333; 2015

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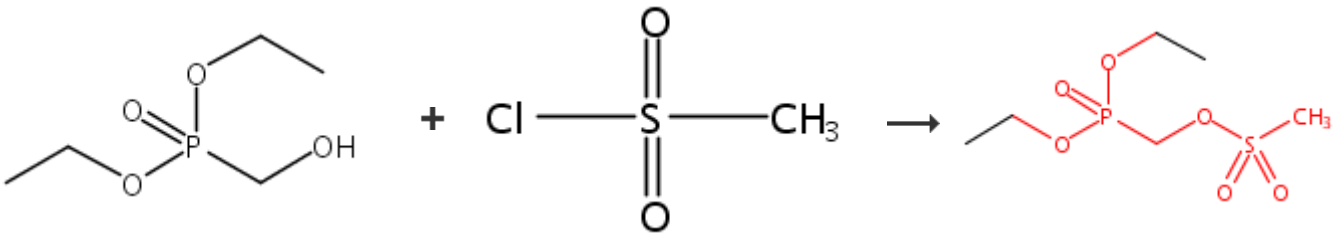
4. Single Step



Overview

Steps/Stages

1.1 R:Et_3N, S:CH_2Cl_2, < 10°C; 5 h, rt

Notes

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

References

Method for preparing tenofovir

By Jiao, Qunfang et al

From Faming Zhuanli Shenqing, 103848868, 11 Jun 2014

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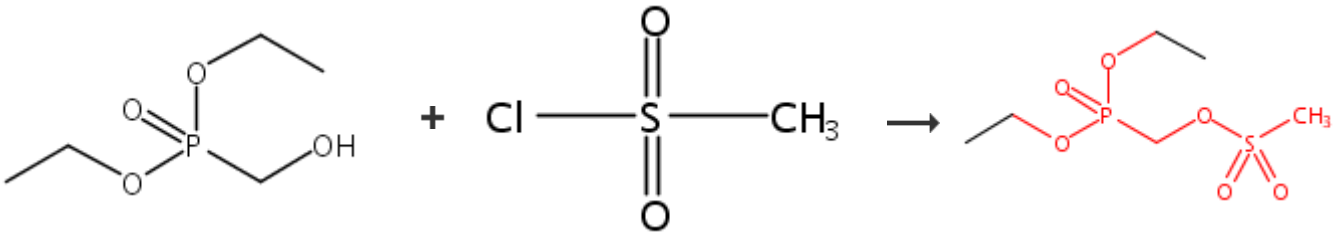
5. Single Step



Overview

Steps/Stages

1.1 S:CH_2Cl_2, 1°C

1.2 R:Et_3N, < 10°C; 5 h, rt

Notes

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

References

Method for preparing diethyl sulfonyloxy methylphosphonate compound

By Jiao, Qunfang et al

From Faming Zhuanli Shenqing, 103848866, 11 Jun 2014

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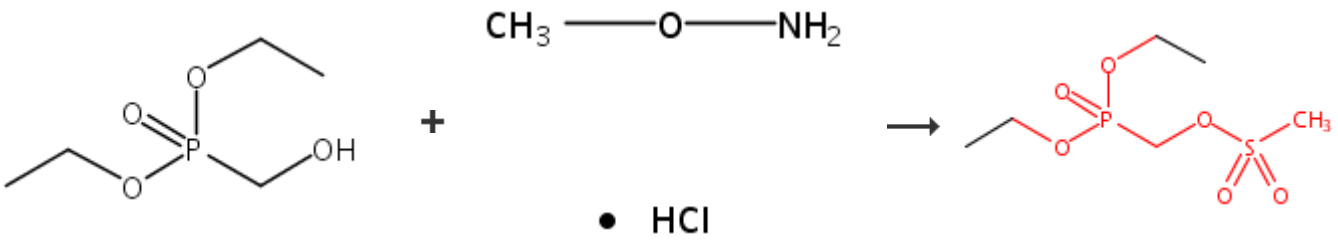
6. Single Step



Overview

Steps/Stages

1.1

1.2 R:Et_3N, -78°C

Notes

literature preparation, Swern oxidation in stage 1, Reactants: 2, Reagents: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

Truncated phosphonated C-1'-branched N,O-nucleosides: A new class of antiviral agents

By Romeo, Roberto et al

From Bioorganic & Medicinal Chemistry, 20(11), 3652-3657; 2012

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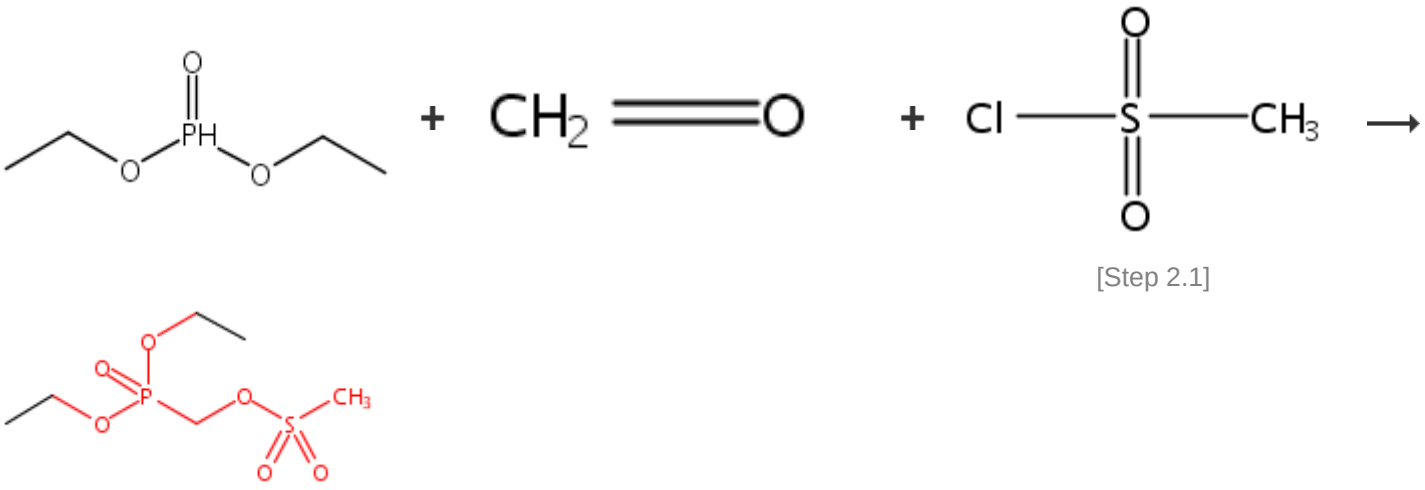
7. 2 Steps



[Step 2.1]

Overview

Steps/Stages

Notes

1.1

2.1 R:Et_3N, S:CH_2Cl_2

1) no experimental detail, literature preparation, Reactants: 3, Reagents: 1, Solvents: 1, Steps: 2, Stages: 2, Most stages in any one step: 1

References

Synthesis and screening of novel inositol phosphonate derivatives for anticancer functions in vitro

By Chen, Wen-Bin et al

From Chinese Chemical Letters, 26(3), 329-333; 2015

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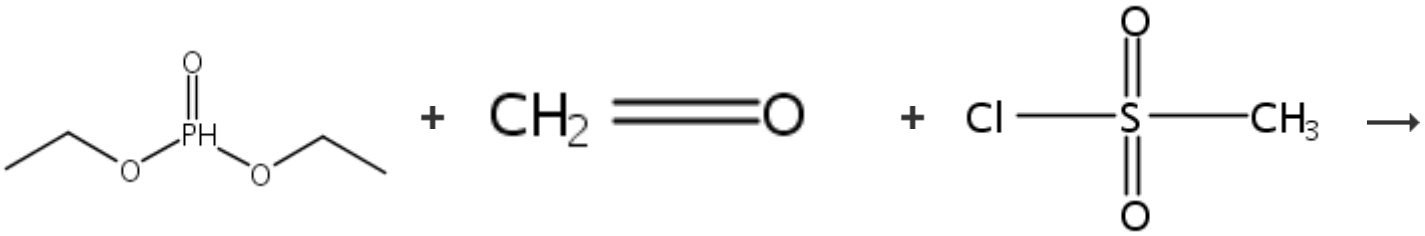
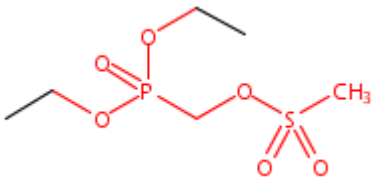
8. 2 Steps



[Step 2.1]



Overview

Steps/Stages

1.1 R:Et_3N, 8 h, 90°C

2.1 R:Et_3N, S:CH_2Cl_2, < 10°C; 5 h, rt

Notes

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

References

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By Jiao, Qunfang et al

From Faming Zhuanli Shenqing, 103848868, 11 Jun 2014

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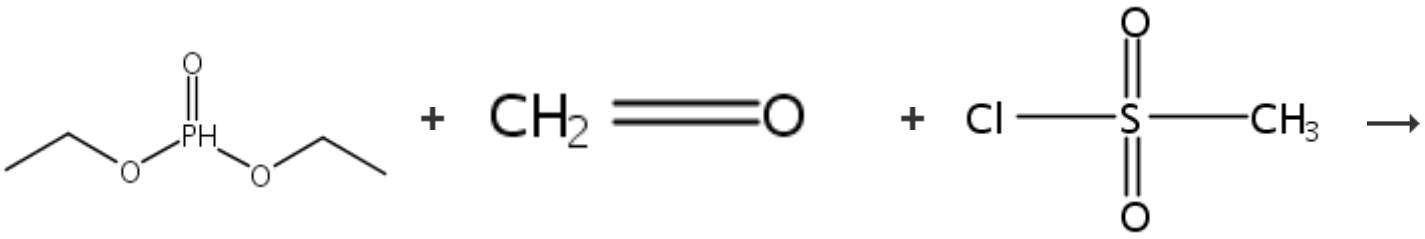
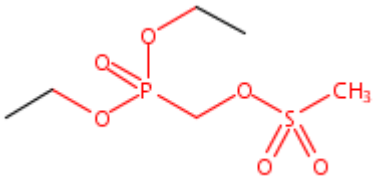
9. 2 Steps



[Step 2.1]



Overview

Steps/Stages

1.1 R:Et_3N, 8 h, 90°C

2.1 S:CH_2Cl_2, 1°C

2.2 R:Et_3N, < 10°C; 5 h, rt

Notes

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

References

Method for preparing diethyl sulfonyloxy methylphosphonate compound

By Jiao, Qunfang et al

From Faming Zhuanli Shenqing, 103848866, 11 Jun 2014

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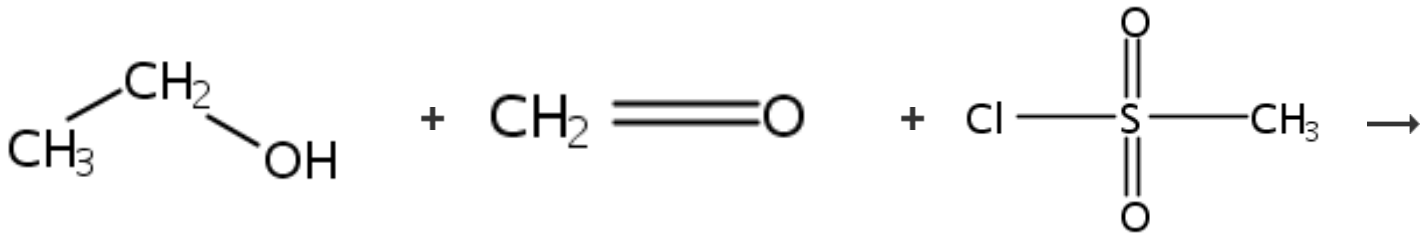
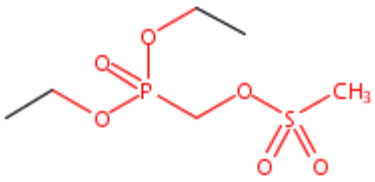
10. 3 Steps



[Step 2.1]

[Step 3.1]



Overview

Steps/Stages

Notes

1.1 $R:PCl_3$

2.1

3.1 $R:Et_3N$, $S:CH_2Cl_2$

1) literature preparation, 2) no experimental detail, literature preparation, Reactants: 3, Reagents: 2, Solvents: 1, Steps: 3, Stages: 3, Most stages in any one step: 1

References

Synthesis and screening of novel inositol phosphonate derivatives for anticancer functions in vitro

By Chen, Wen-Bin et al

From Chinese Chemical Letters, 26(3), 329-333; 2015

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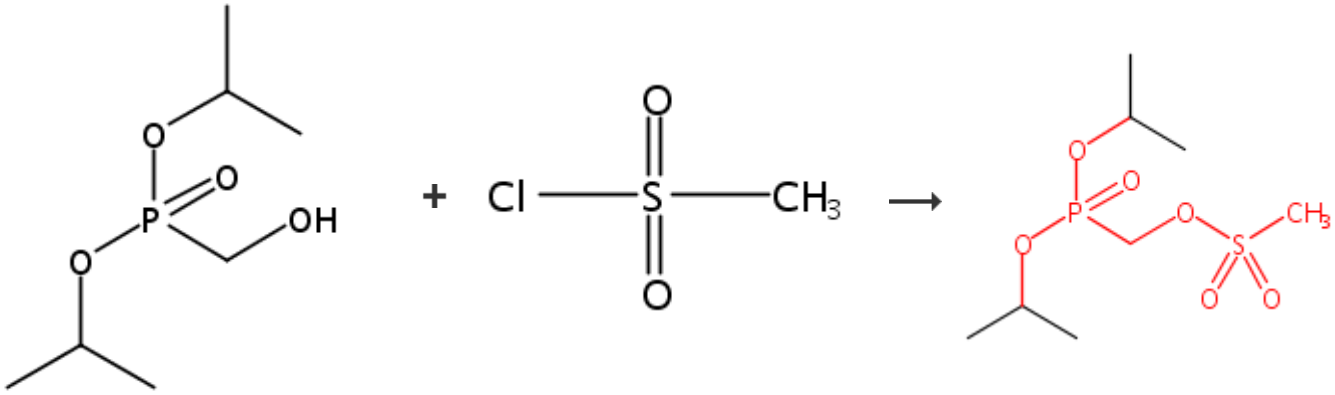
11. Single Step



95%

Overview

Steps/Stages

1.1 $R:Et_3N$, $S:CH_2Cl_2$, 0°C; 0°C → rt; 20 h, rt; rt → 0°C

1.2 $R:H_2CO_3$-Et_3N (1:1), $S:H_2O$, 2 h, 0°C

Notes

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

References

Sulfonyloxymethylphosphonate derivative, and method for the preparation of diisopropyl [[1-(trityloxymethyl)-cyclopropyl]oxy]methyphosphonate therefrom

By Lee, Sang Hu et al

From Repub. Korean Kongkae Taeho Kongbo, 2011133913, 14 Dec 2011

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