



A Publication
of Reliable Methods
for the Preparation
of Organic Compounds

Working with Hazardous Chemicals

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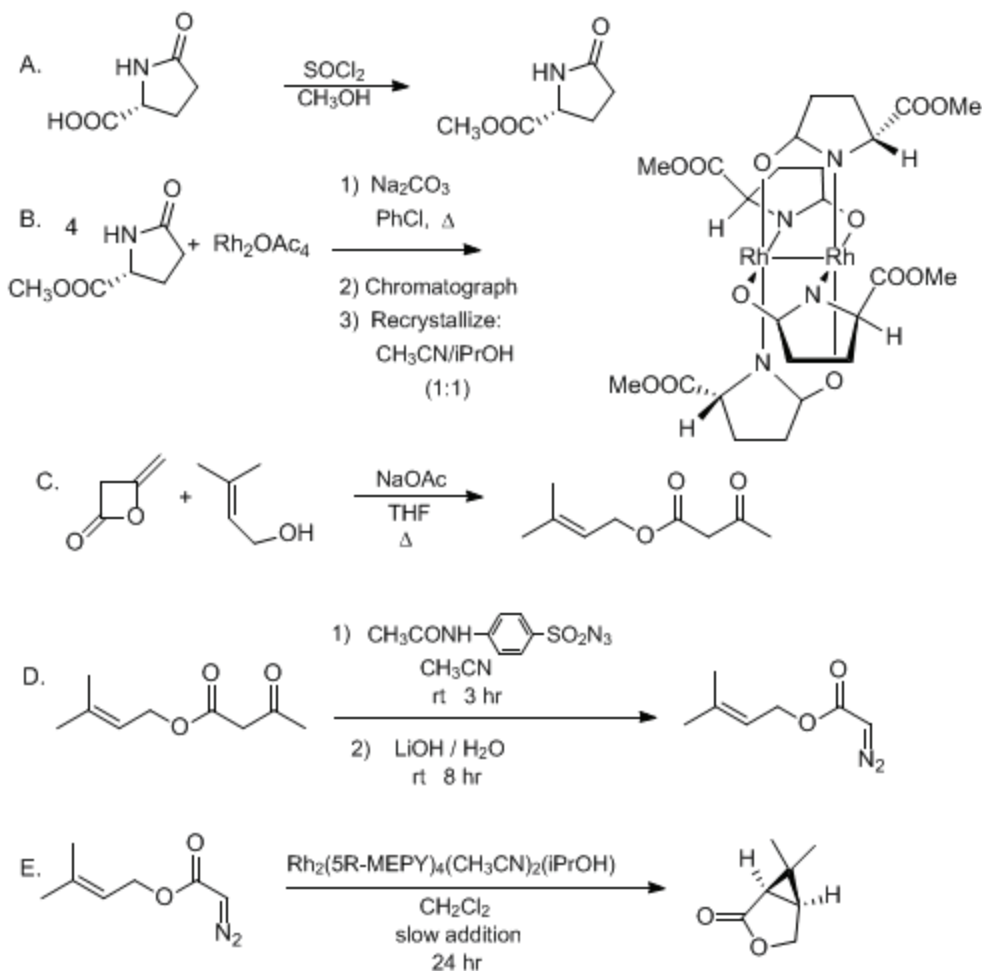
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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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**(1R,5S)-(-)-6,6-DIMETHYL-3-OXABICYCLO[3.1.0]HEXAN-2-ONE.
HIGHLY ENANTIOSELECTIVE INTRAMOLECULAR
CYCLOPROPANATION CATALYZED BY DIRHODIUM(II)
TETRAKIS[METHYL 2-PYRROLIDONE-5(R)-CARBOXYLATE]**

[3-Oxabicyclo[3.1.0]hexan-2-one, 6,6-dimethyl-, (1R-cis)-]



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1. Procedure

Caution! Thionyl chloride is a reactive substance that must be handled in a fume hood.

A. *(-)-Methyl 2-pyrrolidone-5(R)-carboxylate.* A 500-mL, single-necked, round-bottomed flask, equipped with a Teflon-coated magnetic stirring bar, loose fitting rubber septum, and a needle for nitrogen inlet which is vented through a mineral oil bubbler, is flushed with nitrogen and charged with 6.60 g (51.1 mmol) of *(R)-(+)-2-pyrrolidone-5-carboxylic acid* (Note 1) and 200 mL of *methanol* (Note 2). *Thionyl chloride* (0.50 mL, 6.8 mmol) is added cautiously by syringe over a 0.5-min period to the rapidly stirring solution at room temperature. The reaction flask is sealed with the septum and the

nitrogen inlet removed after addition is complete, and the resulting solution is stirred continuously at room temperature for 24 hr. The flask is uncapped, and aqueous, saturated sodium carbonate is added with stirring until the reaction solution is pH 7 to litmus paper (Note 3). After removal of the methanol at room temperature under reduced pressure by rotary evaporator, the residue is dissolved in 50 mL of dichloromethane and washed with 10 mL of aqueous, saturated sodium chloride solution, dried over anhydrous magnesium sulfate, filtered, and concentrated under reduced pressure. The residue is distilled through a short-path distillation apparatus to afford 5.60 g (75% yield) of colorless methyl (R)-2-pyrrolidone-5-carboxylate, bp 83°C (0.25 mm), $[\alpha]_D^{24}$ -8.6° (EtOH, c 1.13) (Note 4).

B. (+)-Dirhodium(II) tetrakis[methyl 2-pyrrolidone-5(R)-carboxylate] acetonitrile-isopropanol solvate, (+)-Rh₂ [(5R)-MEPY]₄ (CH₃CN)₂ (i-PrOH). A 50-mL, single-necked, round-bottomed flask, equipped with a Teflon-coated stirring bar and nitrogen inlet, is flushed with nitrogen and charged with 200 mg (0.452 mmol) of rhodium(II) acetate (Note 5), 1.02 g (7.13 mmol) of methyl 2-pyrrolidone-5 (R)-carboxylate, and 20 mL of chlorobenzene (Note 6). The flask is fitted with a Soxhlet extraction apparatus (Note 7) into which is placed a thimble containing 5 g of an oven-dried mixture of 2 parts sodium carbonate and 1 part of sand (Note 8). The initially green rhodium acetate, upon mixing with the pyrrolidonecarboxylate forms a blue solution, which is heated at reflux under nitrogen for 6 hr (Note 9); removal of the solvent under reduced pressure using a rotary evaporator yields a blue, glass-like solid. The solid is dissolved in a minimal volume of methanol and purified by chromatography on a column containing J.T. Baker BAKERBOND Cyano 40 μm prep LC pack (10 g) in methanol. The dirhodium(II) product forms a broad blue band across the top of the column, which moves imperceptively with methanol. However, the excess ligand moves rapidly as a yellow/brown band in methanol and may be collected with the first 100 mL of eluent (Note 10). Subsequent elution with 1.0 vol % acetonitrile in methanol causes an instantaneous color change from blue to red and rapid elution. The entire red band is collected as one fraction, and the solvent is removed under reduced pressure (Note 11), (Note 12). The resulting blue, glass-like solid (380 mg) is recrystallized by dissolving the solid in 1.0 mL of acetonitrile per 100 mg of solid and then adding an equivalent amount of isopropyl alcohol. Bright red crystals of Rh₂ (5R-MEPY)₄ (CH₃CN)₂ (i-PrOH) form overnight, and are collected by suction, washed with isopropyl alcohol and air-dried to yield 240 mg (0.262 mmol, 58% yield) (Note 13).

C. 3-Methyl-2-buten-1-yl acetoacetate. A 500-mL, three-necked, round-bottomed flask, equipped with a Teflon-coated magnetic stirring bar, stoppered pressure-equalizing addition funnel and a reflux condenser bearing a drying tube, is charged with a solution of 21.5 g (0.250 mol) of 3-methyl-2-buten-1-ol in 70 mL of anhydrous tetrahydrofuran and 1.20 g (0.015 mol) of sodium acetate (Note 14). The reaction mixture is heated at reflux with continuous magnetic stirring, and a solution of 23.5 g (0.280 mol) of freshly distilled diketene in 30 mL of tetrahydrofuran is added dropwise via the addition funnel over 1 hr (Note 15). The reaction mixture is heated at reflux for an additional 30 min upon completion of the addition. The brown reaction mixture is cooled to room temperature, transferred to a separatory funnel containing 300 mL of ether and 50 mL of saturated aqueous sodium chloride solution, mixed thoroughly, and the aqueous layer withdrawn. The aqueous layer is extracted twice with 50-mL portions of ether, and the combined ether solution is washed twice with 50-mL portions of saturated aqueous sodium chloride solution. After the organic layer is dried over anhydrous magnesium sulfate, the solvent is removed under reduced pressure, and the brown residue is distilled (bp 85°C, 0.15 mm) to afford 28.9 g (68% yield) of 3-methyl-2-buten-1-yl acetoacetate as a colorless liquid (Note 16).

D. 3-Methyl-2-buten-1-yl diazoacetate. A 1-L, three-necked, round-bottomed flask, equipped with a Teflon-coated magnetic stirring bar and a stoppered, pressure-equalizing addition funnel is charged with a solution of 30.5 g (0.179 mmol) of 3-methyl-2-buten-1-yl acetoacetate and 23.3 g (0.231 mol) of triethylamine in 150 mL of anhydrous acetonitrile (Note 16). A solution of 55.85 g (0.233 mol) of p-acetamidobenzenesulfonyl azide in 150 mL of acetonitrile is added dropwise over 30 min with continuous magnetic stirring (Note 17). A white precipitate of p-acetamidobenzenesulfonamide is observed after ~30 min (additional acetonitrile is added if necessary to facilitate stirring). The resulting heterogeneous yellow mixture is maintained at room temperature for an additional 2.5–3 hr (Note 18). A solution of 24.6 g (0.586 mol) of lithium hydroxide (LiOH·H₂O) in 200 mL of water is added to the reaction mixture (the precipitate dissolves), and the resulting light brown mixture is stirred for 8 hr (Note 19). The reaction mixture is extracted with two 50-mL portions of 2:1 ether:ethyl acetate. The

combined organic phases are washed with one 25-mL portion of saturated aqueous sodium chloride solution, dried over anhydrous magnesium sulfate, and the solvent is removed under reduced pressure. The resulting orange liquid, containing some solid sulfonamide, is purified by flash column chromatography on silica (200 g) with 1:10 ethyl acetate:hexane as the eluent. Collection of the yellow band and concentration under reduced pressure affords 22.39 g (81% yield) of 3-methyl-2-butenyl diazoacetate as a yellow oil (Note 20).

E. (1*R*,5*S*)-(-)-6,6-Dimethyl-3-oxabicyclo[3.1.0]hexan-2-one. A 1-L, three-necked, round-bottomed flask, equipped with a stoppered pressure-equalizing addition funnel and a reflux condenser bearing a drying tube and a nitrogen inlet vented through a mineral oil bubbler, is charged with a solution of 0.203 g (0.221 mmol) of Rh₂ (5*R*-MEPY)₄ (CH₃CN)₂ (i-PrOH) in 150 mL of freshly distilled, anhydrous dichloromethane (Note 21). The apparatus is flushed with nitrogen and the reaction mixture is heated at reflux during the dropwise addition of a solution of 14.9 g (96.7 mmol) of 3-methyl-2-buten-1-yl diazoacetate in 450 mL of anhydrous dichloromethane over a 30-hr period. The slow flow of external inert gas is terminated after 1 hr, and the evolution of nitrogen from the reaction solution is used to monitor the progress of the reaction. After the addition is complete, the blue reaction mixture is heated at reflux for an additional hour, cooled to room temperature, and the solvent is removed from the green reaction mixture under reduced pressure. Kugelrohr distillation of the residue (80°C, 0.15 mm) affords 12.1 g of a nearly colorless liquid that upon chromatographic purification on silica-alumina (100 g) with hexane:ethyl acetate (80:20 to 70:30) affords 10.2–10.7 g (84–88% yield) of >99% pure (1*R*,5*S*)-(-)-6,6-dimethyl-2-oxabicyclo- [3.1.0]hexan-2-one, bp 70°C (0.15 mm), [α]_D²⁴ -81.2° (CHCl₃, *c* 2.4) (Note 22), whose optical purity was determined on a Chiraldex B-PH capillary column to be 92–93% ee (Note 23). The solid residue remaining after Kugelrohr distillation is dissolved in a minimal volume of methanol and purified by chromatography, as described in Part B. Isolation of the red band affords 0.098–0.099 g (~49%) of recovered catalyst (Note 24).

2. Notes

1. (R)-(+)-2-Pyrrolidone-5-carboxylic acid [Fluka Chemika-BioChemika, D-pyroglutamic acid, [α]_D²² +9±1° (H₂O, *c* 5)] was recrystallized from ethanol/ether (2/1), [α]_D²² +10° (H₂O, *c* 0.97), prior to use.
2. HPLC grade methanol (Aldrich Chemical Company, Inc.) was used without further purification.
3. This titration generally requires the addition of 8 mL of aqueous, saturated sodium carbonate solution.
4. The literature value for this ester is reported to be [α]_D -8.7° (EtOH, *c* 1.13).² The NMR spectra are as follows: ¹H NMR (300 MHz, CDCl₃) δ: 2.18–2.56 (m, 4 H), 3.78 (s, 3 H), 4.26 (dd, 1 H, *J* = 8.5, 5.1), 6.25 (s(br), 1 H); ¹³C NMR (75 MHz, CDCl₃) δ: 25.3, 29.9, 53.1, 56.1, 173.3, 179.1.
5. Rhodium(II) acetate is obtained commercially (Degussa Corporation, Aldrich Chemical Company, Inc., AESAR/Johnson Matthey, or Johnson Matthey/Alfa Products) or prepared from rhodium(III) chloride hydrate (Johnson Matthey) by the standard literature procedure.³
6. Chlorobenzene was distilled under nitrogen from calcium hydride (-4 to +40 mesh) prior to use.
7. A micro Soxhlet extraction apparatus (Ace Glass, Inc.) consisting of the extractor, a 50-mL round-bottomed flask, and an Allihn condenser fitted with a 10 × 50 mm thimble is used.
8. Sodium carbonate is used to trap acetic acid liberated in the ligand exchange reaction. Sand is included to maintain the porosity of the solid. A layer of sand at the top of the carbonate mixture prevents sodium carbonate from entering the reaction flask.
9. When the reaction is followed by HPLC using a μ-Bondapak-CN column with 2% acetonitrile in methanol as the eluent, two bands are observed initially: a broad band eluting with the solvent front [chlorobenzene and excess methyl 2-pyrrolidone-5(R)-carboxylate] and a second band at 1.6 min when the flow rate is 1.5 mL/min [rhodium(II) acetate]. As the reaction progresses, the rhodium(II) acetate band diminishes and is replaced by several bands with longer retention volumes until one major band, in addition to that for chlorobenzene and ligand, is observed at about 4 min. Only minor impurities elute at intermediate times. A brown-black material, insoluble in all common solvents, is observed in some preparations. The origin of this material is unknown, but its presence decreases product yield by 25%.
10. Unreacted methyl 2-pyrrolidone-5(R)-carboxylate may be reisolated by distillation (70% recovery) without loss of optical purity.
11. This material is >95% pure by HPLC analysis.
12. Direct recrystallization is an alternative to chromatography, but decreased yields result because of

the large amount of unreacted ligand present. In this procedure 1 mL of [acetonitrile](#) is added to the crude, blue glass-like solid. The resulting solution is filtered through glass wool and 5 mL of [isopropyl alcohol](#) is added. Overnight refrigeration gives a 38% yield of red crystals that are identical with those described in the procedure.

13. This procedure produced analytically pure crystals with the following physical properties: Anal. Calcd for $C_{31}H_{46}N_6O_{13}Rh_2$: C, 40.63; H, 5.06; N, 9.17. Found: C, 40.37, H, 5.11; N, 9.12; 1H NMR (300 MHz, $CDCl_3$) δ : 1.21 (d, 6 H, $J = 6.1$), 1.35 (d, 1 H, $J = 4.4$), 1.8–2.4 (m, 12 H), 2.26 (s, 6 H), 2.55–2.70 (m, 4 H), 3.68 (s, 6 H), 3.70 (s, 6 H), 3.95 (dd, 2 H, $J = 8.6, 2.1$), 3.96–4.08 (m, 1 H), 4.32 (dd, 2 H, $J = 8.8, 3.0$); ^{13}C NMR (75 MHz, $CDCl_3$) δ : 2.9, 25.0, 25.2, 26.0, 31.2, 31.4, 51.6, 51.9, 64.0, 66.4, 66.6, 114.9, 175.5, 175.7, 188.3, 188.5. $[\alpha]_D^{24} +270.6^\circ$ (CH_3CN , c 0.112).

14. [Tetrahydrofuran](#) was distilled from [lithium aluminum hydride](#).

15. This reaction has an induction period. If too much [diketene](#) is added before condensation begins, an exotherm may result. Reaction onset is evident by a color change and a significant increase in temperature.

16. The NMR spectra are as follows: 1H NMR (300 MHz, $CDCl_3$) δ : 1.72 (s, 3 H), 1.79 (s, 3 H), 2.27 (s, 3 H), 3.45 (s, 2 H), 4.64 (d, 2 H, $J = 7.3$), 5.32–5.40 (m, 1 H); enol form at 1.96 (s) and 4.99 (s). ^{13}C NMR (75 MHz, $CDCl_3$) δ : 18.5, 26.2, 30.5, 50.5, 62.6, 118.6, 140.2, 167.7, 201.2 (minor amounts of the enol tautomer may also be present).

17. Commercially available [p-acetamidobenzenesulfonyl azide](#) (Aldrich Chemical Company, Inc.) was preferred over [p-dodecylbenzenesulfonyl azide](#) (Danheiser, R. L.; Miller, R. F.; Brisbois, R. G. *Org. Synth., Coll. Vol. IX 1998*, 197, Note 9; Ref. 4a), or [methanesulfonyl azide](#)⁴ for reasons of safety, yield, and ease of manipulation. See also Davies, H. M. L.; Cantrell, Jr., W. R.; Romines, K. R.; Baum, J. S. *Org. Synth., Coll. Vol. IX 1998*, 422.

18. After this time, no starting β -keto ester is observed upon analysis by TLC.

19. The principal reaction competing with deacylation is ester hydrolysis.

20. The spectral properties are as follows: 1H NMR (300 MHz, $CDCl_3$) δ : 1.72 (s, 3 H), 1.77 (s, 3 H), 4.66 (d, 2 H, $J = 7.3$), 4.74 (s, 1 H), 5.32–5.39 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ : 18.4, 26.2, 46.6, 62.2, 119.2, 139.7, 167.4; IR (thin film) cm^{-1} : 2109 ($C=N_2$), and 1694 ($C=O$).

21. [Dichloromethane](#) was distilled from [phosphorus pentoxide](#) and stored under [nitrogen](#). All glassware was oven dried.

22. The NMR spectra are as follows: 1H NMR (300 MHz, $CDCl_3$) δ : 1.17 (s, 3 H), 1.18 (s, 3 H), 1.93 (d, 1 H, $J = 6.2$), 2.04 (dd, 1 H, $J = 6.2, 5.5$), 4.13 (d, 1 H, $J = 9.8$), 4.35 (dd, 1 H, $J = 9.8, 5.5$); ^{13}C NMR (75 MHz, $CDCl_3$) δ : 14.2, 25.0, 29.9, 30.3, 66.4, 174.9. Enantiomerically pure (1R,5S)-(–)-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one is reported to have $[\alpha]_D^{25} -89.9^\circ$ ($CHCl_3$, c 1.4) (calculated).⁵

23. A 30 m \times 0.32 mm ID Chiraldex B-PH (β -cyclodextrin) column was used under isothermal conditions at 120°C. Retention time of the (1R,5S)-enantiomer was 16.9 min, while the (1S,5R)-enantiomer eluted at 17.9 min. The submitters report that separation also occurred on a 30-m Chiraldex γ -cyclodextrin trifluoroacetate column operated at 140°C; retention times were 14.2 min (1R,5S-enantiomer) and 18.9 min (1S,5R-enantiomer).

24. The submitters report that the recovered catalyst can be reused in the same reaction; (1R,5S)-(–)-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one was formed in 83% yield and 88% ee.

Waste Disposal Information

All toxic materials were disposed of in accordance with "Prudent Practices in the Laboratory"; National Academy Press; Washington, DC, 1995.

3. Discussion

This is the first detailed procedure for the synthesis of a chiral dirhodium(II) carboxamide catalyst and its application to intramolecular cyclopropanation. The preparation of the ligand, [methyl 2-pyrrolidone-5\(R\)-carboxylate](#), is adapted from the procedure of Ackermann, Matthes, and Tamm.² The method for ligand displacement from [dirhodium\(II\) tetraacetate](#) is an extension of that reported for the synthesis of dirhodium(II) tetraacetamide.⁶ The title compound, (1R,5S)-(–)-6,6-dimethyl-3-oxabicyclo[3.1.0]hexan-2-one, is a synthetic precursor to (1R,3S)-(+)-cis-chrysanthemic acid.⁵

Dirhodium(II) tetrakis[methyl 2-pyrrolidone-5(R)-carboxylate], Rh, (5R-MEPY)₄, and its

enantiomer, Rh₂ (5S-MEPY)₄, which is prepared by the same procedure, are highly enantioselective catalysts for intramolecular cyclopropanation of allylic diazoacetates (65–94% ee) and homoallylic diazoacetates (71–90% ee),^{7,8} intermolecular carbon-hydrogen insertion reactions of 2-alkoxyethyl diazoacetates (57–91% ee)⁹ and N-alkyl-N-(tert-butyl)diazoacetamides (58–73% ee),¹⁰ intermolecular cyclopropanation of alkynes with ethyl diazoacetate (54–69% ee) or menthyl diazoacetates (77–98% diastereomeric excess, de),¹¹ and intermolecular cyclopropanation of alkenes with menthyl diazoacetate (60–91% de for the cis isomer, 47–65% de for the trans isomer).¹² Their use in ≤1.0 mol % in dichloromethane solvent effects complete reaction of the diazo ester and provides the carbenoid product in 43–88% yield. The same general method used for the preparation of Rh₂ (5R-MEPY)₄ was employed for the synthesis of their isopropyl⁷ and neopentyl⁹ ester analogs.

References and Notes

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Appendix

Chemical Abstracts Nomenclature (Collective Index Number); (Registry Number)

silica-alumina

(1R,5S)-(-)-6,6-DIMETHYL-3-OXABICYCLO[3.1.0]HEXAN-2-ONE

DIRHODIUM(II) TETRAKIS[METHYL 2-PYRROLIDONE-5(R)-CARBOXYLATE

(-)-Methyl 2-pyrrolidone-5(R)-carboxylate

(+)-Dirhodium(II) tetrakis[methyl 2-pyrrolidone-5(R)-carboxylate]

(1R,5S)-(-)-6,6-dimethyl-2-oxabicyclo- [3.1.0]hexan-2-one

dirhodium(II) tetraacetamide

ethanol (64-17-5)

acetic acid (64-19-7)

ethyl acetate (141-78-6)

methanol (67-56-1)

ether (60-29-7)

sodium acetate (127-09-3)

acetonitrile (75-05-8)

thionyl chloride (7719-09-7)

sodium chloride (7647-14-5)

sodium carbonate (497-19-8)

nitrogen (7727-37-9)

diketene (674-82-8)

chlorobenzene (108-90-7)

isopropyl alcohol (67-63-0)

dichloromethane (75-09-2)

magnesium sulfate (7487-88-9)

ethyl diazoacetate (623-73-4)

Tetrahydrofuran (109-99-9)

lithium aluminum hydride (16853-85-3)

hexane (110-54-3)

triethylamine (121-44-8)

calcium hydride (7789-78-8)

lithium hydroxide (1310-65-2)

phosphorus pentoxide (1314-56-3)

rhodium(II) acetate (5503-41-3)

methanesulfonyl azide

p-Acetamidobenzenesulfonyl azide (2158-14-7)

p-acetamidobenzenesulfonamide (121-61-9)

p-dodecylbenzenesulfonyl azide (79791-38-1)

Menthyl

3-Oxabicyclo[3.1.0]hexan-2-one, 6,6-dimethyl-, (1R-cis)- (71565-25-8)

Methyl (R)-2-pyrrolidone-5-carboxylate,
Methyl 2-pyrrolidone-5(R)-carboxylate (64700-65-8)

(R)-(+)-2-pyrrolidone-5-carboxylic acid,
D-pyroglutamic acid (4042-36-8)

rhodium acetate

3-Methyl-2-buten-1-yl acetoacetate (21597-32-0)

3-methyl-2-buten-1-ol (556-82-1)

3-Methyl-2-buten-1-yl diazoacetate,
3-Methyl-2-butenyl diazoacetate (72800-60-3)

rhodium(III) chloride hydrate

dirhodium(II) tetraacetate (15956-28-2)

(1R,3S)-(+)-cis-chrysanthemic acid

menthyl diazoacetate