

1. Single Step



plasma reaction products with
nitrogen

[Overview](#)

Steps/Stages

Notes

1.1 R:N₂ (plasma reaction products with methane), 8 h, 1 mbar

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

References

[Tholinomics - Chemical Analysis of Nitrogen-Rich Polymers](#)

By Pernot, Pascal et al

From Analytical Chemistry (Washington, DC, United States), 82(4), 1371-1380; 2010

[Experimental Procedure](#)

Tholin Production in a RF Dusty Plasma: The PAMPRE Experimental Setup. The apparatus used in this experiment was described in detail previously⁷ and is summarized here briefly. The plasma is a capacitively coupled plasma discharge produced at a frequency of 13.56 MHz in a N₂-CH₄ gaseous mixture. The plasma is confined in a cylindrical cage shaped by a metallic grid. In the plasma discharge, electrons dissociate and ionize N₂ and CH₄. This initiates chemical reactions and molecular growth, producing hydrocarbons and nitrogen-bearing molecules that eventually end up forming solid particles. The produced solid particles are negatively charged and maintained in levitation between the electrodes by electrostatic forces. The N₂-CH₄ gaseous mixture is injected continuously into the plasma reactor, as a neutral flow oriented downward. This produces a neutral drag force, which ejects the solid particles out of the plasma discharge. The ejected tholin is trapped in a glass vessel surrounding the metallic cage. When sufficient amounts of tholin are produced (i.e., typically after 8 h), the plasma is turned off; the reactor is thoroughly pumped to evacuate potential traces of HCN and other residual gases, then filled with N₂ up to atmospheric pressure and opened for solid sample collection. When operating under normal conditions, ~0.5 cm³ (~100 mg) of solid material is produced in an 8-h run. The tholins have the appearance of a very fine powder, orange to brown in color, deposited gently in the glass vessel without any interaction with the substrate. They are collected into microvials for ex situ analyses. Because of the plausible toxicity of tholin, gloves and nanofiltering masks are used during open air manipulation of the material. In this work, the samples were obtained under the following operating conditions: a total pressure of 1 mbar, a flow rate of 55 ± 0.1 sccm, and an absorbed radio frequency power of 30 ± 2 W. Two different samples were produced by adjusting the relative flux of N₂ vs CH₄. The first sample (SA98) was produced in a mixture containing 2.00 ± 0.06% of methane, while the second sample (SA90) was produced in a mixture containing 10.0 ± 0.2% of methane.

[Reaction Protocol](#)

Procedure

1. Carry out reaction of the plasma is a capacitively coupled plasma discharge produced at a frequency of 13.56 MHz in a N₂-CH₄ gaseous mixture.
2. The plasma is confined in a cylindrical cage shaped by a metallic grid.

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

2. Single Step

[Overview](#)**Steps/Stages**

1.1 R:

**Notes**

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

References

[Carbon-xenon bond strength in the methylxenonium cation determined from ion cyclotron resonance methyl cation exchange equilibria](#)

By Hovey, J. K. and McMahon, T. B.

From Journal of the American Chemical Society, 108(3), 528-9; 1986

CASREACT®: Copyright © 2020 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.

3. Single Step

hydrogenated

[Overview](#)**Steps/Stages**

1.1 R:

**Notes**

gas phase, silica wafers and aluminum coated glass substrates used, alternative preparation shown, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

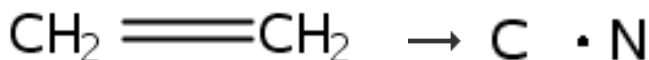
References

[Deposition and characterization of organic polymer thin films using a dielectric barrier discharge with different C₂H_m/N₂ \(m = 2, 4, 6\) gas mixtures](#)

By Chandrashekaraiiah, Thejaswini
Halethimmanahally et al

From European Physical Journal D: Atomic, Molecular, Optical and Plasma Physics, 69(6), 1-6; 2015

CASREACT®: Copyright © 2020 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.

4. Single Step

hydrogenated

[Overview](#)

Steps/Stages**Notes**

1.1 R:



gas phase, silica wafers and aluminum coated glass substrates used, alternative preparation shown, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

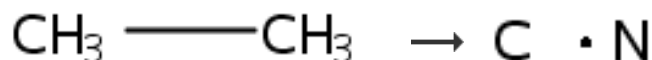
References

[Deposition and characterization of organic polymer thin films using a dielectric barrier discharge with different C₂H_m/N₂ \(m = 2, 4, 6\) gas mixtures](#)

By Chandrashekaraiah, Thejaswini
Halethimmanahally et al

From European Physical Journal D: Atomic, Molecular, Optical and Plasma Physics, 69(6), 1-6; 2015

CASREACT®: Copyright © 2020 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.

5. Single Step

hydrogenated

[Overview](#)**Steps/Stages****Notes**

1.1 R:



gas phase, silica wafers and aluminum coated glass substrates used, alternative preparation shown, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Deposition and characterization of organic polymer thin films using a dielectric barrier discharge with different C₂H_m/N₂ \(m = 2, 4, 6\) gas mixtures](#)

By Chandrashekaraiah, Thejaswini
Halethimmanahally et al

From European Physical Journal D: Atomic, Molecular, Optical and Plasma Physics, 69(6), 1-6; 2015

CASREACT®: Copyright © 2020 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.

6. Single Step[Overview](#)**Steps/Stages****Notes**

1.1 R:



rt

gas phase, flow-tube reactor used, alternative preparation shown, helium used as carrier gas, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Dissociation of Cyanogen Azide: An Alternative Route to Synthesis of Carbon Nitride](#)

By Benard, D. J. et al

From Journal of Physical Chemistry B, 102(31), 6010-6019; 1998

Experimental Procedure

General/Typical Procedure: **Flow Tube.** Figure 3 is a schematic illustration of the experimental apparatus used to grow carbon-nitrogen films at ambient temperature. The flow tube was constructed of 1.375 in. i.d. quartz tubing and was operated at pressures in the range 10-15 Torr. A converging/diverging nozzle made of glassfilled Teflon (area ratio ~ 8) was positioned typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate. The nozzle served to concentrate reactive gas flow along the centerline, thereby increasing interaction with the substrate and effectively eliminating wall losses. Assuming parabolic flow, an approximate centerline velocity of 65 m/s (upstream of the nozzle) was estimated from the cross sectional area, measured gas pressure, and a pressure versus flow curve of the vacuum system that was obtained separately by use of a calibrated electronic mass flowmeter. Pressure and velocity downstream of the nozzle depended on the position of the movable azide injector probe in relation to the nozzle throat. With the probe tip located upstream of the nozzle, upstream and downstream pressures varied less than 10%, indicating subsonic flow through the nozzle and centerline gas velocities downstream of the nozzle comparable to upstream values. Moving the 0.375 in. o.d. injector tube into or through the nozzle throat significantly restricted its open area and lead to pressure ratios as high as 3 to 1 (upstream to downstream) with corresponding acceleration of the gas to supersonic velocity at the nozzle exit plane.⁴⁸ Partial pressure of the donor species (NCN₃, NCCN, ClCN, or BrCN) in the flow tube was approximately 1 mTorr (upstream) based on total pressure measurements and mass flow rates. Rapid injection of donor molecules into the primary flow was optimized by using a conical probe tip (30° half-angle) with 12 0.035 in. diameter radial holes drilled around its circumference (normal to the reactor centerline) and by adding a variable flow of He to the secondary gas stream to allow penetration of resulting mixing jets into the surrounding stream of N₂*/He. Visually observing spatial dependence of resulting chemiluminescence demonstrated that mixing of primary and secondary flows was complete within 1 cm of the injector tip.

Reaction Protocol

Procedure

1. Construct the flow tube of 1.375 in. i.d. quartz tubing and operate at pressures in the range 10-15 Torr.
2. Position a converging/diverging nozzle made of glassfilled Teflon (area ratio ~ 8) typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate.

[View more...](#)

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 American Chemical Society. All Rights Reserved. CASREACT contains reactions from CAS and from: ZIC/INITI 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.

7. Single Step



[Overview](#)

Steps/Stages

Notes

1.1 R:



rt

gas phase, flow-tube reactor used, alternative preparation shown, helium used as carrier gas, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Dissociation of Cyanogen Azide: An Alternative Route to Synthesis of Carbon Nitride](#)

By Benard, D. J. et al

From Journal of Physical Chemistry B, 102(31), 6010-6019; 1998

Experimental Procedure

Flow Tube. Figure 3 is a schematic illustration of the experimental apparatus used to grow carbon-nitrogen films at ambient temperature. The flow tube was constructed of 1.375 in. i.d. quartz tubing and was operated at pressures in the range 10-15 Torr. A converging/diverging nozzle made of glassfilled Teflon (area ratio ~ 8) was positioned typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate. The nozzle served to concentrate reactive gas flow along the centerline, thereby increasing interaction with the substrate and effectively eliminating wall losses. Assuming parabolic flow, an approximate centerline velocity of 65 m/s (upstream of the nozzle) was estimated from the cross sectional area, measured gas pressure, and a pressure versus flow curve of the vacuum system that was obtained separately by use of a calibrated electronic mass flowmeter. Pressure and velocity downstream of the nozzle depended on the position of the movable azide injector probe in relation to the nozzle throat. With the probe tip located upstream of the nozzle, upstream and downstream pressures varied less than 10%, indicating subsonic flow through the nozzle and centerline gas velocities downstream of the nozzle comparable to upstream values. Moving the 0.375 in. o.d. injector tube into or through the nozzle throat significantly restricted its open area and lead to pressure ratios as high as 3 to 1 (upstream to downstream) with corresponding acceleration of the gas to supersonic velocity at the nozzle exit plane.⁴⁸ Partial pressure of the donor species (NCN₃, NCCN, ClCN, or BrCN) in the flow tube was approximately 1 mTorr (upstream) based on total pressure measurements and mass flow rates. Rapid injection of donor molecules into the primary flow was optimized by using a conical probe tip (30° half-angle) with 12 0.035 in. diameter radial holes drilled around its circumference (normal to the reactor centerline) and by adding a variable flow of He to the secondary gas stream to allow penetration of resulting mixing jets into the surrounding stream of N₂*/He. Visually observing spatial dependence of resulting chemiluminescence demonstrated that mixing of primary and secondary flows was complete within 1 cm of the injector tip.

Reaction Protocol

Procedure

1. Construct the flow tube of 1.375 in. i.d. quartz tubing and operate at pressures in the range 10-15 Torr.
2. Position a converging/diverging nozzle made of glassfilled Teflon (area ratio ~ 8) typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate.

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

8. 2 Steps



[Overview](#)

Steps/Stages

1.1 R:NaN₃, 2-3 h, 60°C, 80 psi

2.1 R:



rt

Notes

1) sealed cylinder used, alternative preparation shown, 2) gas phase, flow-tube reactor used, alternative preparation shown, helium used as carrier gas, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

[Dissociation of Cyanogen Azide: An Alternative Route to Synthesis of Carbon Nitride](#)

By Benard, D. J. et al

From Journal of Physical Chemistry B, 102(31), 6010-6019; 1998

Experimental Procedure

Step 1

General/Typical Procedure: Approximately 12.5 g of NaN₃ was added to 120 mL of solvent in the reactor, which also contained a 2 in. long Tefloncoated magnetic stirring bar. The stainless steel cylinder, with an absolute pressure gauge attached, was then quickly pumped down to vacuum and back-filled with C₁CN gas (to 1 atm pressure) with the magnetic stirrer turned off. The cylinder was then sealed off and stirring reactivated to absorb typically 90% of the chloride gas into the solvent in approximately 1-2 min. Subsequently, stirring was interrupted and the gas charging process repeated three more times, before finally adding He diluent to a net pressure of 80 psig. Stirring was then resumed for 2-3 h, and the lower third of the sealed-off cylinder warmed to 70 °C by means of external heating tapes. The azide diluted in He was finally withdrawn from the reservoir (still at 70 °C, with continued stirring) via a corrosion-resistant gas regulator adjusted to dispense at atmospheric pressure. This gas flow was in turn directed to a 14 cm long stainless steel FT-IR absorption cell with salt windows that evacuated to a downstream needle valve adjusted to bleed the cylinder pressure at 25 psig/h. The first hour of gas removal from the reaction cylinder was discarded to vacuum to ensure complete saturation of conductive tubing, after which the flow of dilute NCN₃ and He was redirected into the flow tube. The bromide method was carried out in the same reaction vessel using 1 g of BrCN and 2 g of NaN₃ with 80 psig of added He per batch, and reaction was complete after 2-3 h at 60 °C.

Step 2

Flow Tube. Figure 3 is a schematic illustration of the experimental apparatus used to grow carbon-nitrogen films at ambient temperature. The flow tube was constructed of 1.375 in. i.d. quartz tubing and was operated at pressures in the range 10-15 Torr. A converging/diverging nozzle made of glass-filled Teflon (area ratio ~ 8) was positioned typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate. The nozzle served to concentrate reactive gas flow along the centerline, thereby increasing interaction with the substrate and effectively eliminating wall losses. Assuming parabolic flow, an approximate centerline velocity of 65 m/s (upstream of the nozzle) was estimated from the cross sectional area, measured gas pressure, and a pressure versus flow curve of the vacuum system that was obtained separately by use of a calibrated electronic mass flowmeter. Pressure and velocity downstream of the nozzle depended on the position of the movable azide injector probe in relation to the nozzle throat. With the probe tip located upstream of the nozzle, upstream and downstream pressures varied less than 10%, indicating subsonic flow through the nozzle and centerline gas velocities downstream of the nozzle comparable to upstream values. Moving the 0.375 in. o.d. injector tube into or through the nozzle throat significantly restricted its open area and lead to pressure ratios as high as 3 to 1 (upstream to downstream) with corresponding acceleration of the gas to supersonic velocity at the nozzle exit plane.⁴⁸ Partial pressure of the donor species (NCN₃, NCCN, C₁CN, or BrCN) in the flow tube was approximately 1 mTorr (upstream) based on total pressure measurements and mass flow rates. Rapid injection of donor molecules into the primary flow was optimized by using a conical probe tip (30° half-angle) with 12 0.035 in. diameter radial holes drilled around its circumference (normal to the reactor centerline) and by adding a variable flow of He to the secondary gas stream to allow penetration of resulting mixing jets into the surrounding stream of N₂*/He. Visually observing spatial dependence of resulting chemiluminescence demonstrated that mixing of primary and secondary flows was complete within 1 cm of the injector tip.

Reaction Protocol

Procedure

1. Add approximately 12.5 g of NaN₃ to 120 mL of solvent in the reactor, contain a 2 in. long Tefloncoated magnetic stirring bar.
2. Pump down quickly the stainless steel cylinder, with an absolute pressure gauge attached, to vacuum and back-filled with C₁CN gas (to 1 atm pressure) with the magnetic stirrer turned off.

[View more...](#)

View with
MethodsNow

CASREACT®: Copyright © 2020 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.

9. 2 Steps



Overview

Steps/Stages

1.1 R:NaN₃, S:108-32-7, 2-3 h, 70°C, 80 psi

2.1 R:



rt

Notes

1) sealed cylinder used, alternative preparation shown, 2) gas phase, flow-tube reactor used, alternative preparation shown, helium used as carrier gas, Reactants: 1, Reagents: 2, Solvents: 1, Steps: 2, Stages: 2, Most stages in any one step: 1

References

[Dissociation of Cyanogen Azide: An Alternative Route to Synthesis of Carbon Nitride](#)

By Benard, D. J. et al

From Journal of Physical Chemistry B, 102(31), 6010-6019; 1998

Experimental Procedure

Step 1

Approximately 12.5 g of NaN₃ was added to 120 mL of solvent in the reactor, which also contained a 2 in. long Tefloncoated magnetic stirring bar. The stainless steel cylinder, with an absolute pressure gauge attached, was then quickly pumped down to vacuum and back-filled with ClCN gas (to 1 atm pressure) with the magnetic stirrer turned off. The cylinder was then sealed off and stirring reactivated to absorb typically 90% of the chloride gas into the solvent in approximately 1-2 min. Subsequently, stirring was interrupted and the gas charging process repeated three more times, before finally adding He diluent to a net pressure of 80 psig. Stirring was then resumed for 2-3 h, and the lower third of the sealed-off cylinder warmed to 70 °C by means of external heating tapes. The azide diluted in He was finally withdrawn from the reservoir (still at 70 °C, with continued stirring) via a corrosion-resistant gas regulator adjusted to dispense at atmospheric pressure. This gas flow was in turn directed to a 14 cm long stainless steel FT-IR absorption cell with salt windows that evacuated to a downstream needle valve adjusted to bleed the cylinder pressure at 25 psig/h. The first hour of gas removal from the reaction cylinder was discarded to vacuum to ensure complete saturation of conductive tubing, after which the flow of dilute NCN₃ and He was redirected into the flow tube.

Step 2

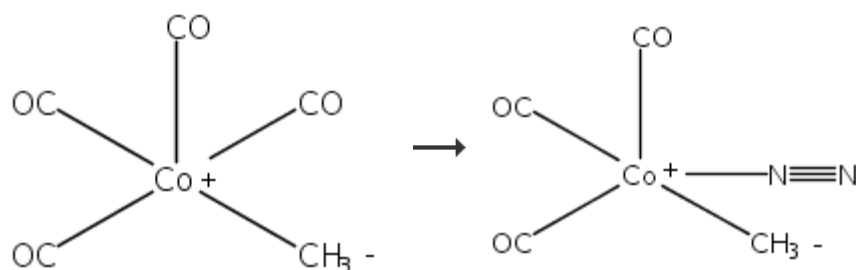
Flow Tube. Figure 3 is a schematic illustration of the experimental apparatus used to grow carbon-nitrogen films at ambient temperature. The flow tube was constructed of 1.375 in. i.d. quartz tubing and was operated at pressures in the range 10-15 Torr. A converging/diverging nozzle made of glassfilled Teflon (area ratio ~ 8) was positioned typically midway between the injector probe tip and a coaxially mounted 1 in. diameter substrate. The nozzle served to concentrate reactive gas flow along the centerline, thereby increasing interaction with the substrate and effectively eliminating wall losses. Assuming parabolic flow, an approximate centerline velocity of 65 m/s (upstream of the nozzle) was estimated from the cross sectional area, measured gas pressure, and a pressure versus flow curve of the vacuum system that was obtained separately by use of a calibrated electronic mass flowmeter. Pressure and velocity downstream of the nozzle depended on the position of the movable azide injector probe in relation to the nozzle throat. With the probe tip located upstream of the nozzle, upstream and downstream pressures varied less than 10%, indicating subsonic flow through the nozzle and centerline gas velocities downstream of the nozzle comparable to upstream values. Moving the 0.375 in. o.d. injector tube into or through the nozzle throat significantly restricted its open area and lead to pressure ratios as high as 3 to 1 (upstream to downstream) with corresponding acceleration of the gas to supersonic velocity at the nozzle exit plane.⁴⁸ Partial pressure of the donor species (NCN_3 , NCCN , ClCN , or BrCN) in the flow tube was approximately 1 mTorr (upstream) based on total pressure measurements and mass flow rates. Rapid injection of donor molecules into the primary flow was optimized by using a conical probe tip (30° half-angle) with 12 0.035 in. diameter radial holes drilled around its circumference (normal to the reactor centerline) and by adding a variable flow of He to the secondary gas stream to allow penetration of resulting mixing jets into the surrounding stream of N_2^*/He . Visually observing spatial dependence of resulting chemiluminescence demonstrated that mixing of primary and secondary flows was complete within 1 cm of the injector tip.

Reaction Protocol**Procedure**

1. Add approximately 12.5 g of NaN_3 to 120 mL of solvent in the reactor, contain a 2 in. long Tefloncoated magnetic stirring bar.
2. Pump down quickly the stainless steel cylinder, with an absolute pressure gauge attached, to vacuum and back-filled with ClCN gas (to 1 atm pressure) with the magnetic stirrer turned off.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

10. Single Step[Overview](#)**Steps/Stages****Notes**

1.1 R:



Photochem., in matrix, Reactants: 1,
Reagents: 1, Steps: 1, Stages: 1, Most stages
in any one step: 1

References

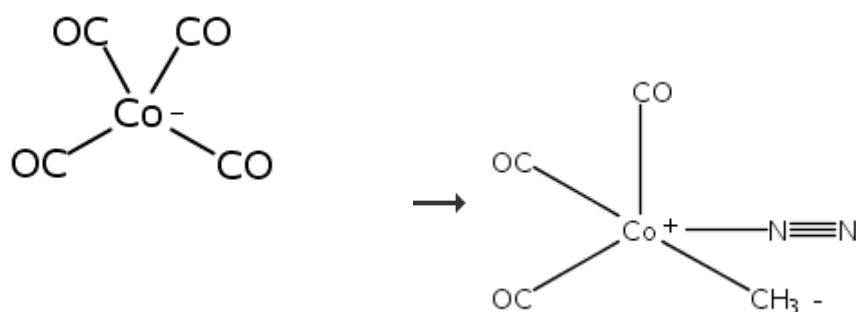
[Photolysis of tetracarbonylmethylcobalt\(I\) and tetracarbonylhydridocobalt\(I\) in inert-gas and hydrogen-containing matrixes: reaction of 16-electron, coordinatively unsaturated complexes with dihydrogen](#)

By Sweany, Ray L. and Russell, Franklin N.

From Organometallics, 7(3), 719-27; 1988

CASREACT®: Copyright © 2020 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.

11. 2 Steps



• Na +

Overview

Steps/Stages

1.1 R:Me₂SO₄

2.1 R:



Notes

2) Photochem., in matrix, Reactants: 1,
Reagents: 2, Steps: 2, Stages: 2, Most stages
in any one step: 1

References

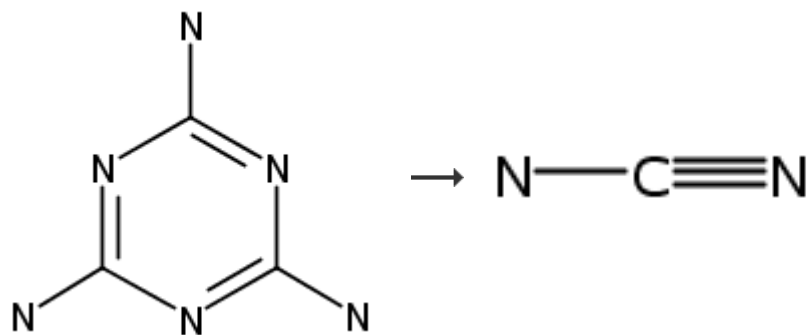
[Photolysis of tetracarbonylmethylcobalt\(I\) and tetracarbonylhydridocobalt\(I\) in inert-gas and hydrogen-containing matrixes: reaction of 16-electron, coordinatively unsaturated complexes with dihydrogen](#)

By Sweany, Ray L. and Russell, Franklin N.

From Organometallics, 7(3), 719-27; 1988

CASREACT®: Copyright © 2020 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.

12. Single Step



Overview

Steps/Stages

1.1 R:Csl

R:



20K

Notes

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

References

Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene

By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

Experimental Procedure

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

Reaction Protocol

Procedure

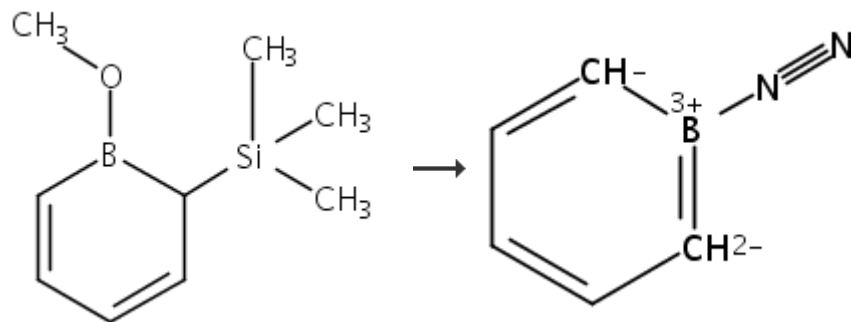
1. Vaporize crystallites of trinitrene at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated trinitrene by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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:



S:N₂

Notes

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

References

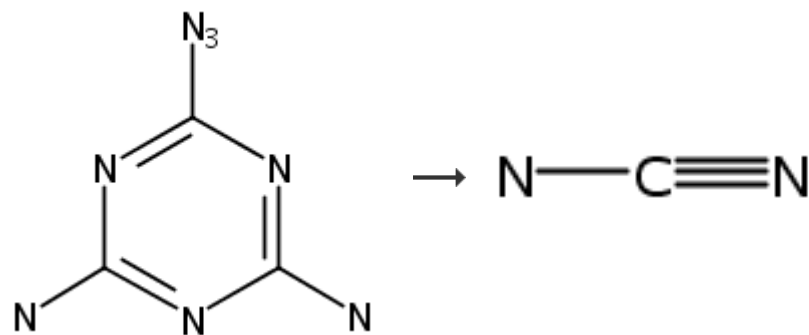
[Hetero-π-systems. 15. Nitrogen fixation by borabenzene](#)

By Maier, Guenther et al

From *Angewandte Chemie*, 100(2), 303; 1988

CASREACT®: Copyright © 2020 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.

14. 2 Steps



Overview

Steps/Stages

1.1 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K

Notes

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

References

[Generation and Photoreactions of 2,4,6-Trinitro-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadatake et al

From *Journal of the American Chemical Society*, 126(25), 7846-7852; 2004

Experimental Procedure

Step 1

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 2

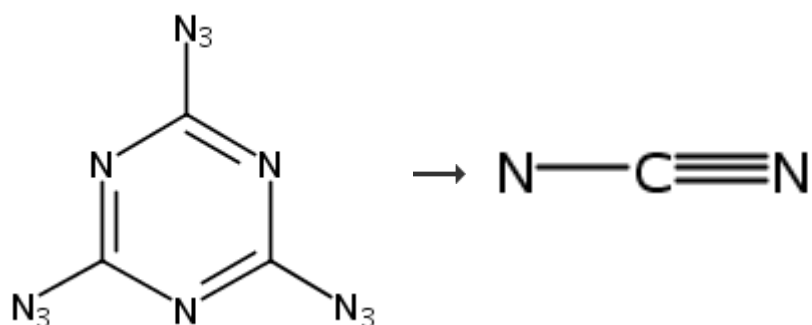
General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

Reaction Protocol**Procedure**

1. Vaporize crystallites of azide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a CsI substrate at 20 K.
2. Photolyze matrix-isolated azide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

15. 2 Steps[Overview](#)**Steps/Stages****Notes**

1.1 40-50°C

1.2 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotus LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotus LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

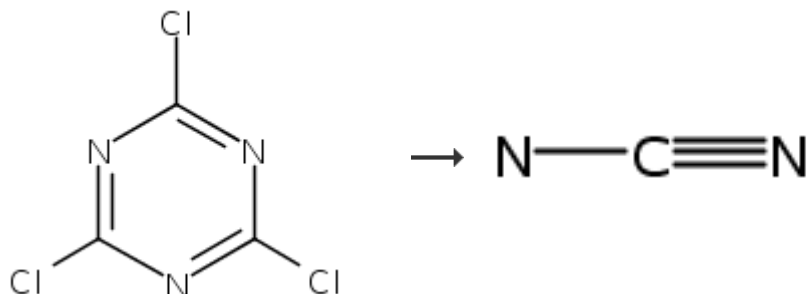
Reaction Protocol

Procedure

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotus LS-2125 equipped with YHG-34).

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

16. 3 Steps

[Overview](#)**Steps/Stages**

1.1 R:NaN₃, S:Me₂CO, S:H₂O

2.1 40-50°C

2.2 R:Csl

R:



20K

3.1 R:Csl, R:N₂, 20K

[Experimental Procedure](#)**Step 1**

2,4,6-Triazido-1,3,5-triazine (**1**) was synthesized by mixing an acetone solution of cyanuric chloride (2,4,6-trichloro-1,3,5-triazine) with an aqueous solution of sodium azide according to the reported procedure.¹³ The triazide **1** was photolyzed in a nitrogen matrix at 20 K.

Step 2

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

[Reaction Protocol](#)**Procedure**

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

17. 3 Steps**Notes**

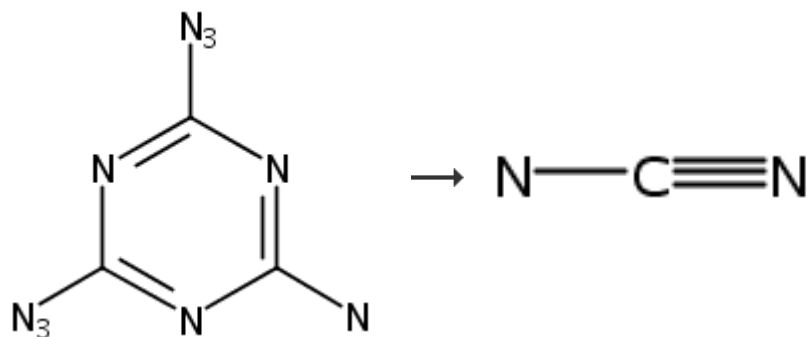
2) photochem., 3) photochem., Reactants: 1, Reagents: 3, Solvents: 2, Steps: 3, Stages: 4, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004



Overview

Steps/Stages

1.1 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K

3.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **3**

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

Reaction Protocol

Procedure

1. Vaporize crystallites of mononitrene at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated mononitrene by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

Notes

1) photochem., 2) photochem., 3) photochem.,
Reactants: 1, Reagents: 2, Steps: 3, Stages:
3, Most stages in any one step: 1

References

[Generation and Photoreactions of 2,4,6-Trinitro-1,3,5-triazine, a Septet Trinitrene](#)

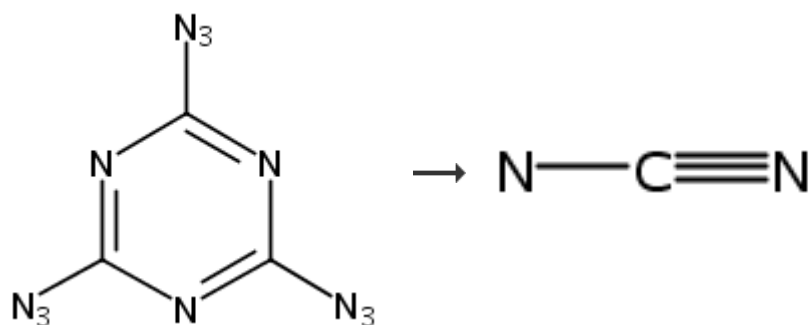
By Sato, Tadotake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

18. 3 Steps



Overview

Steps/Stages

1.1 40-50°C

1.2 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K

3.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Notes

1) photochem., 2) photochem., 3) photochem.,
Reactants: 1, Reagents: 2, Steps: 3, Stages:
4, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitro-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadataké et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, $0.5 \text{ mJ}\cdot\text{cm}^{-2} \text{ pulse}^{-1}$; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm^{-1} . UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

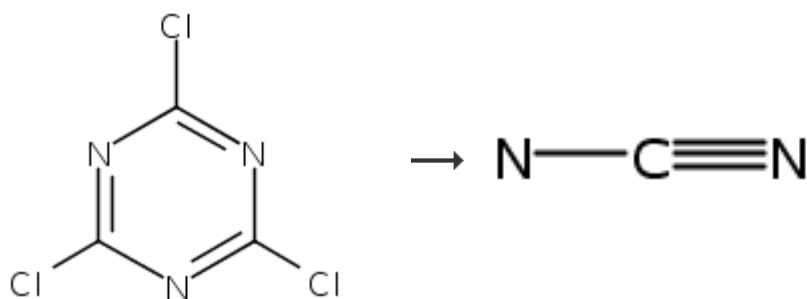
Reaction Protocol**Procedure**

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a CsI substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, $0.5 \text{ mJ}\cdot\text{cm}^{-2} \text{ pulse}^{-1}$; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

19. 4 Steps**Overview****Steps/Stages**

- 1.1 R:NaN₃, S:Me₂CO, S:H₂O
- 2.1 40-50°C

- 2.2 R:CsI

R:



20K

- 3.1 R:CsI, R:N₂, 20K

- 4.1 R:CsI, R:N₂, 20K

Experimental Procedure**Notes**

2) photochem., 3) photochem., 4) photochem.,
Reactants: 1, Reagents: 3, Solvents: 2, Steps:
4, Stages: 5, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

Step 1

2,4,6-Triazido-1,3,5-triazine (**1**) was synthesized by mixing an acetone solution of cyanuric chloride (2,4,6-trichloro-1,3,5-triazine) with an aqueous solution of sodium azide according to the reported procedure.¹³ The triazide **1** was photolyzed in a nitrogen matrix at 20 K.

Step 2

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 4

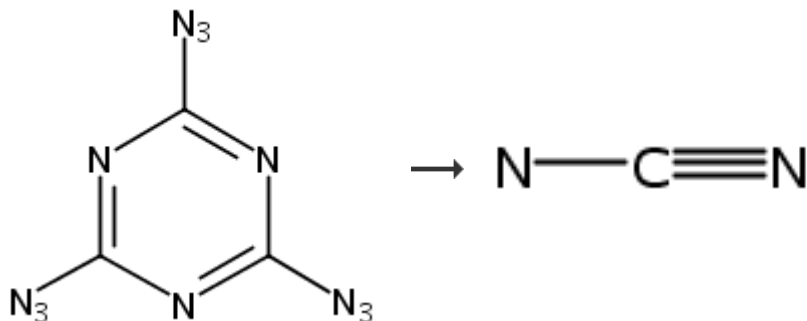
General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

[Reaction Protocol](#)**Procedure**

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a CsI substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

20. 4 Steps

Overview

Steps/Stages

1.1 40-50°C

1.2 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K3.1 R:Csl, R:N₂, 20K4.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GX1 spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **2**

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GX1 spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **3**

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GX1 spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 4

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GX1 spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

Reaction Protocol

Procedure

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

View more...

Notes

1) photochem., 2) photochem., 3) photochem., 4) photochem., Reactants: **1**, Reagents: **2**, Steps: 4, Stages: 5, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene](#)

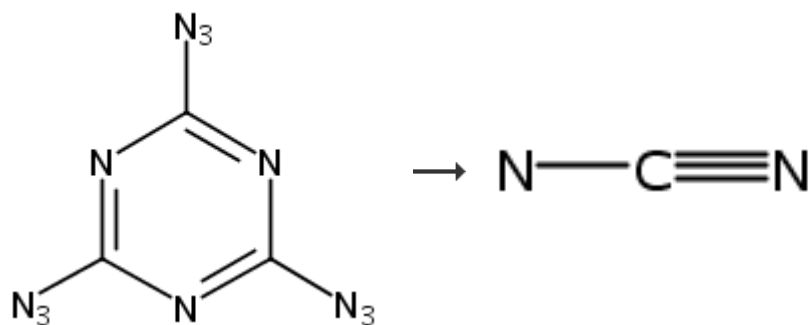
By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

View with
MethodsNow

CASREACT®: Copyright © 2020 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.

21. 4 Steps



Overview

Steps/Stages

1.1 40-50°C

1.2 R:Csl

R:



20K

2.1 R:Csl, R:N₂, 20K

3.1 R:Csl, R:N₂, 20K

4.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **3**

Notes

1) photochem., 2) photochem., 3) photochem., 4) photochem., Reactants: 1, Reagents: 2, Steps: 4, Stages: 5, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitro-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 4

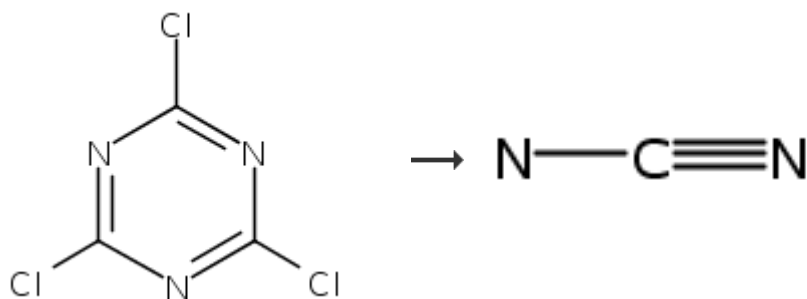
General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a CsI substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

[Reaction Protocol](#)**Procedure**

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a CsI substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

22. 5 Steps[Overview](#)**Steps/Stages****Notes**

1.1 R:NaN₃, S:Me₂CO, S:H₂O

2.1 40-50°C

2.2 R:Csl

R:



20K

3.1 R:Csl, R:N₂, 20K

4.1 R:Csl, R:N₂, 20K

5.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

2,4,6-Triazido-1,3,5-triazine (**1**) was synthesized by mixing an acetone solution of cyanuric chloride (2,4,6-trichloro-1,3,5-triazine) with an aqueous solution of sodium azide according to the reported procedure.¹³ The triazide **1** was photolyzed in a nitrogen matrix at 20 K.

Step 2

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **2**

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **3**

Step 4

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 5

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

Reaction Protocol

Procedure

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

2) photochem., 3) photochem., 4) photochem., 5) photochem., Reactants: 1, Reagents: 3, Solvents: 2, Steps: 5, Stages: 6, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene](#)

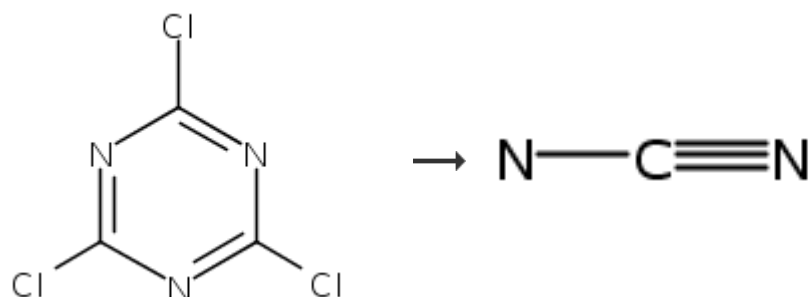
By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

23. 5 Steps



Overview

Steps/Stages

1.1 R:NaN₃, S:Me₂CO, S:H₂O
2.1 40-50°C

2.2 R:Csl

R:



20K

3.1 R:Csl, R:N₂, 20K

4.1 R:Csl, R:N₂, 20K

5.1 R:Csl, R:N₂, 20K

Experimental Procedure

Step 1

2,4,6-Triazido-1,3,5-triazine (**1**) was synthesized by mixing an acetone solution of cyanuric chloride (2,4,6-trichloro-1,3,5-triazine) with an aqueous solution of sodium azide according to the reported procedure.¹³ The triazide **1** was photolyzed in a nitrogen matrix at 20 K.

Step 2

Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotus LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer.

Notes

2) photochem., 3) photochem., 4) photochem., 5) photochem., Reactants: 1, Reagents: 3, Solvents: 2, Steps: 5, Stages: 6, Most stages in any one step: 2

References

[Generation and Photoreactions of 2,4,6-Trinitreno-1,3,5-triazine, a Septet Trinitrene](#)

By Sato, Tadatake et al

From Journal of the American Chemical Society, 126(25), 7846-7852; 2004

Step 3

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **3**

Step 4

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **4**

Step 5

General/Typical Procedure: Crystallites of **1** were vaporized at 40-45 °C and co-deposited with nitrogen (99.9999%) onto a Csl substrate at 20 K. Matrix-isolated **1** was photolyzed by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34). The sample chamber had a pair of quartz windows for UV-vis measurements and a pair of KBr windows for FTIR measurements, enabling us to monitor the photolysis by FTIR and UV-vis absorption spectroscopy simultaneously. The FTIR measurements were carried out on a PerkinElmer Spectrum GXI spectrometer with a resolution of 1 cm⁻¹. UV-vis absorption spectra were measured with a Shimadzu UV-3100 spectrometer. Product **5**

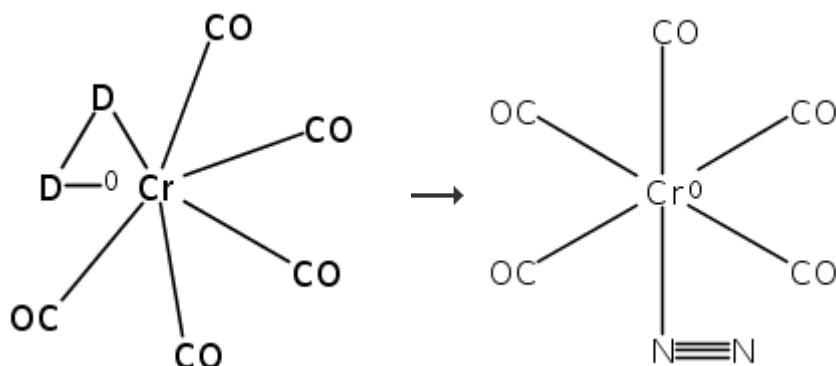
Reaction Protocol**Procedure**

1. Vaporize crystallites of cyanuric triazide at 40-45 °C and co-deposit with nitrogen (99.9999%) onto a Csl substrate at 20 K.
2. Photolyze matrix-isolated cyanuric triazide by using fourth-harmonic-generated (FHG) pulses from a Nd:YAG laser ($\lambda = 266$ nm, 10 Hz, 0.5 mJ·cm⁻² pulse⁻¹; Lotis LS-2125 equipped with YHG-34).

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

24. Single Step

[Overview](#)

Steps/Stages

Notes

1.1 R:



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

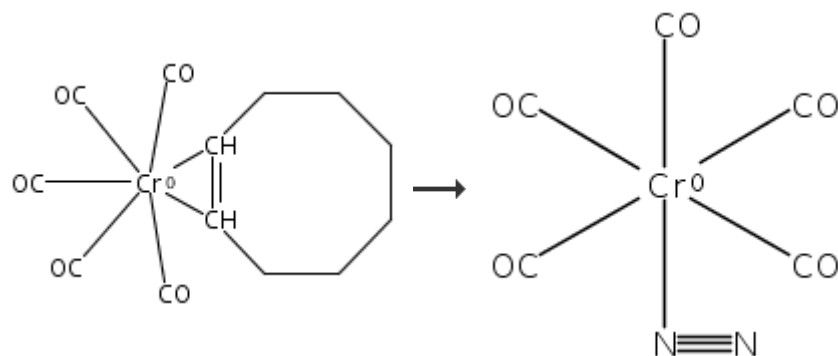
References

Photolysis of Group 6 metal carbonyl alkene complexes in liquefied noble gases. IR evidence for very labile dinitrogen and dihydrogen adducts

By Jackson, Sarah A. et al

From Journal of the Chemical Society, Chemical Communications, (9), 678-80; 1987

CASREACT®: Copyright © 2020 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.

25. 2 Steps**Overview****Steps/Stages**1.1 R:D₂, S:7439-90-9

2.1 R:

**Notes**

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

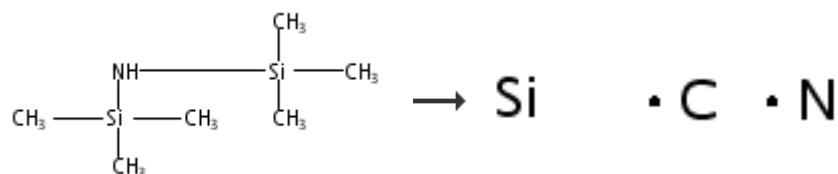
References

Photolysis of Group 6 metal carbonyl alkene complexes in liquefied noble gases. IR evidence for very labile dinitrogen and dihydrogen adducts

By Jackson, Sarah A. et al

From Journal of the Chemical Society, Chemical Communications, (9), 678-80; 1987

CASREACT®: Copyright © 2020 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.

26. Single Step

[Overview](#)**Steps/Stages**

1.1 R:



450°C

Notes

helium used as carrier gas, plasma enhanced chemical vapor deposition, alternative preparation shown, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Atmospheric-Pressure Plasma-Enhanced Chemical Vapor Deposition of a-SiCN:H Films: Role of Precursors on the Film Growth and Properties](#)

By Guruvenket, Srinivasan et al

From ACS Applied Materials & Interfaces, 4(10), 5293-5299; 2012

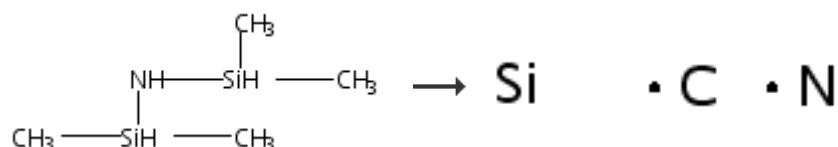
[Reaction Protocol](#)**Procedure**

1. Use Helium and nitrogen (N₂) as the plasma and reactive gases.
2. Maintain their flows at 30 and 0.5 lpm, respectively,.

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

27. Single Step[Overview](#)**Steps/Stages**

1.1 R:



450°C

Notes

helium used as carrier gas, plasma enhanced chemical vapor deposition, alternative preparation shown, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Atmospheric-Pressure Plasma-Enhanced Chemical Vapor Deposition of a-SiCN:H Films: Role of Precursors on the Film Growth and Properties](#)

By Guruvenket, Srinivasan et al

From ACS Applied Materials & Interfaces, 4(10), 5293-5299; 2012

[Reaction Protocol](#)

Procedure

1. Use Helium and nitrogen (N₂) as the plasma and reactive gases.
2. Maintain their flows at 30 and 0.5 lpm, respectively,.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

28. Single Step[Overview](#)**Steps/Stages**1.1 R:Ta₂O₅

R:



1600°C

Notes

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

References

[Emergence of Oxygen Reduction Activity in Partially Oxidized Tantalum Carbonitrides: Roles of Deposited Carbon for Oxygen-Reduction-Reaction-Site Creation and Surface Electron Conduction](#)

By Ishihara, Akimitsu et al

From Journal of Physical Chemistry C, 117(37), 18837-18844; 2013

[Experimental Procedure](#)

2.1. Sample Preparation. The starting material, tantalum carbonitride was prepared by heating the uniform mixture of Ta₂O₅ powder and carbon black at 1600 °C under flowing N₂. A composition of the tantalum carbonitride, namely, the ratio of C and N, was controlled by a quantity of the mixed carbon black. Atomic composition of the tantalum carbonitride used as a starting material. Tantalum carbonitride. Ta, 50.1 atom %; C, 24.7 atom %; N, 24.5 atom %; and O, 0.7 atom %. BET surface area of the Ta₂CN was 0.69 m² g⁻¹. The purity of Ta₂CN was about 99.94%.

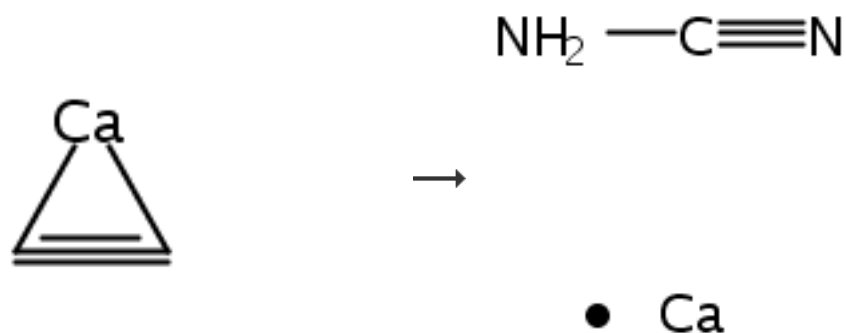
[Reaction Protocol](#)**Procedure**

1. Heat the uniform mixture of Ta₂O₅ powder and carbon black at 1600 °C under flowing N₂.
2. Control the reaction by a quantity of the mixed carbon black.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

29. Single Step


[Overview](#)

Steps/Stages

1.1 R:



Notes

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

References

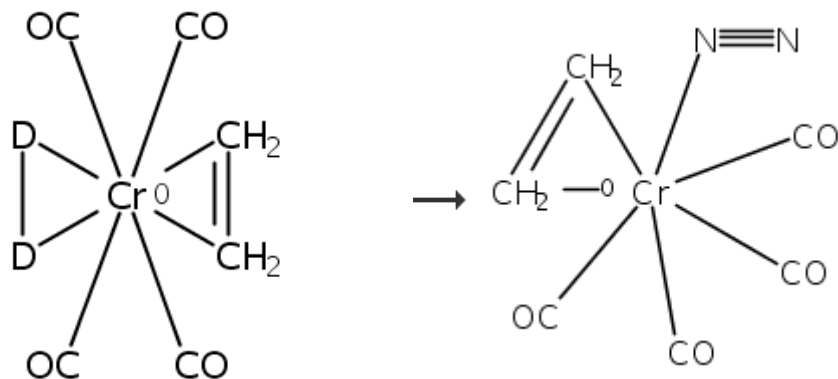
[Cyanamide](#)

By Schow, Steven

From e-EROS Encyclopedia of Reagents for Organic Synthesis, , 1-3; 2001

CASREACT®: Copyright © 2020 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.

30. Single Step


[Overview](#)

Steps/Stages

Notes

1.1 R:



thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 1, Steps: 1, Stages:
1, Most stages in any one step: 1

References

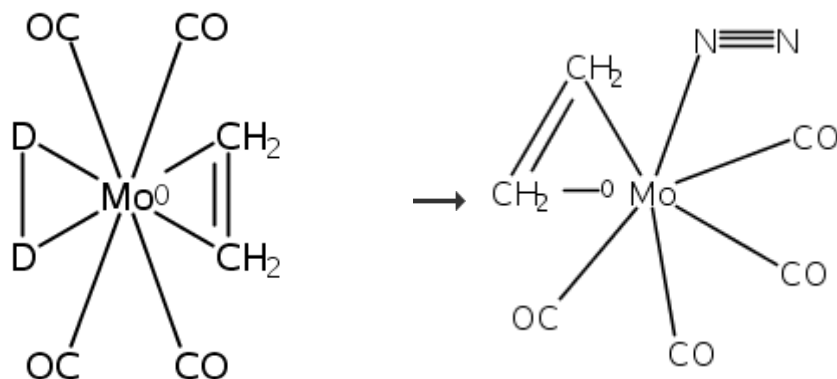
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

31. Single Step



Overview

Steps/Stages

1.1 R:



Notes

thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 1, Steps: 1, Stages:
1, Most stages in any one step: 1

References

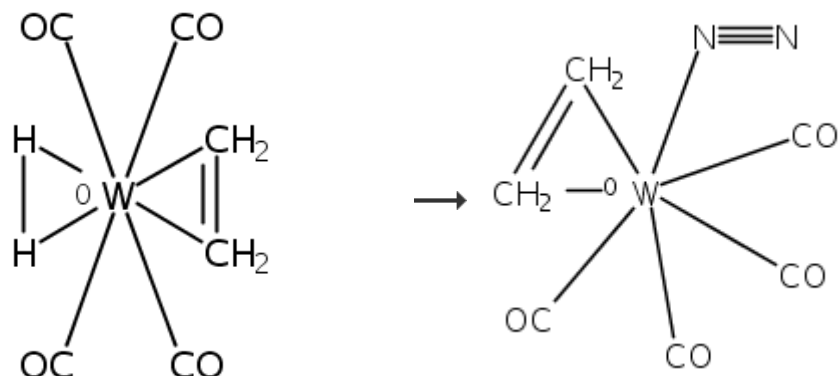
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

32. Single Step



Overview

Steps/Stages

1.1 R:



Notes

thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 1, Steps: 1, Stages:
1, Most stages in any one step: 1

References

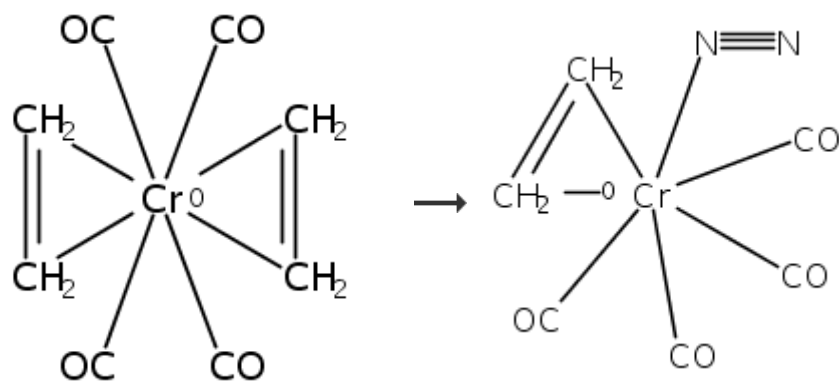
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

33. 2 Steps



Overview

Steps/Stages

Notes

1.1 R:

R:D₂2.1 R:N₂

1) photochem., supercritical Xenon medium, 2) thermal, supercritical Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

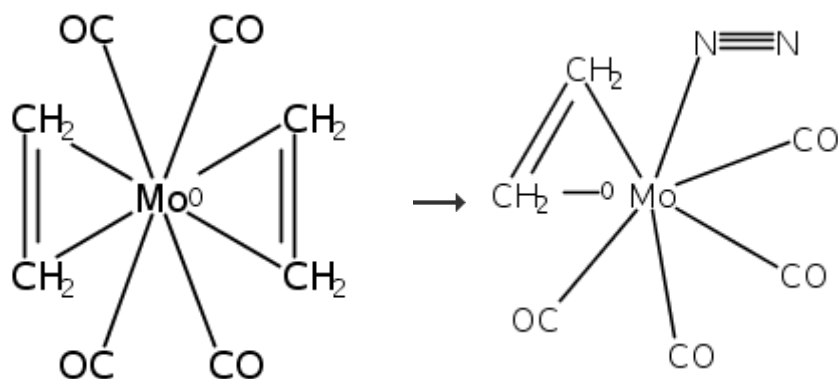
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

34. 2 Steps



Overview

Steps/Stages

1.1 R:D₂

2.1 R:



Notes

1) photochem., supercritical Xenon medium, 2) thermal, supercritical Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

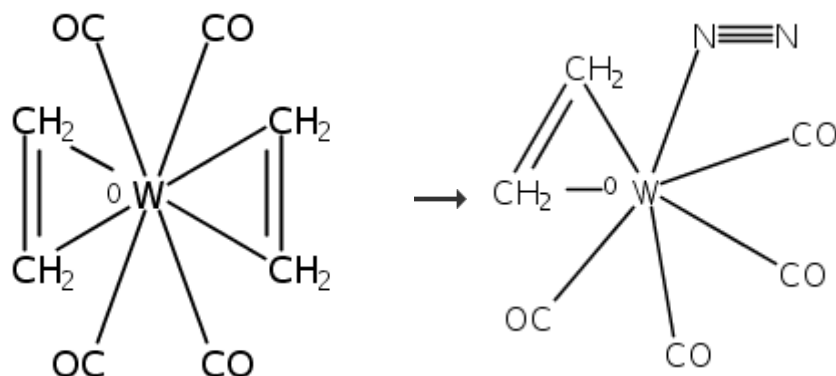
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

35. 2 Steps



Overview

Steps/Stages

1.1 R:H₂

2.1 R:



Notes

1) photochem., supercritical Xenon medium, 2) thermal, supercritical Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

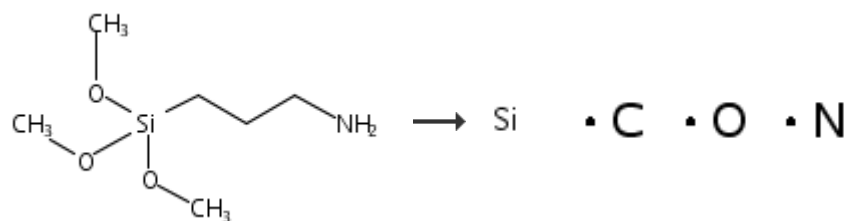
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η^4 -norbornadiene)M(CO)₃(η^2 -H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

36. Single Step



Overview

Steps/Stages

1.1 R:H₂

R:



300°C

Notes

plasma enhanced atomic layer deposition, Reactants: 1, Reagents: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

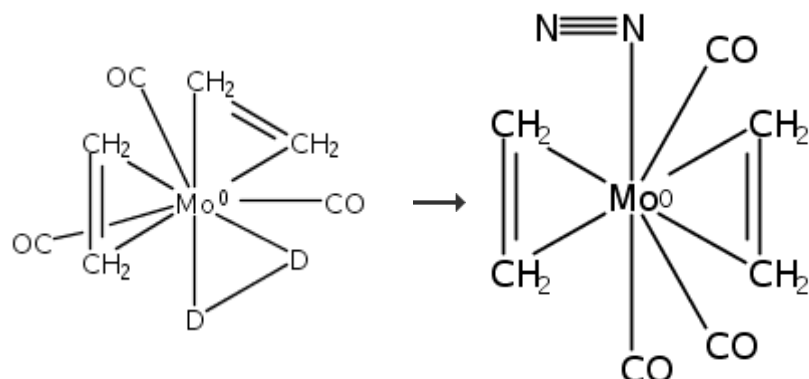
[Formation of SiOCN thin films](#)

By Suzuki, Toshiya and Pore, Viljami J.

From U.S. Pat. Appl. Publ., 20170140924, 18 May 2017

CASREACT®: Copyright © 2020 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.

37. Single Step



Overview

Steps/Stages

1.1 R:



Notes

thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 1, Steps: 1, Stages: 1,
Most stages in any one step: 1

References

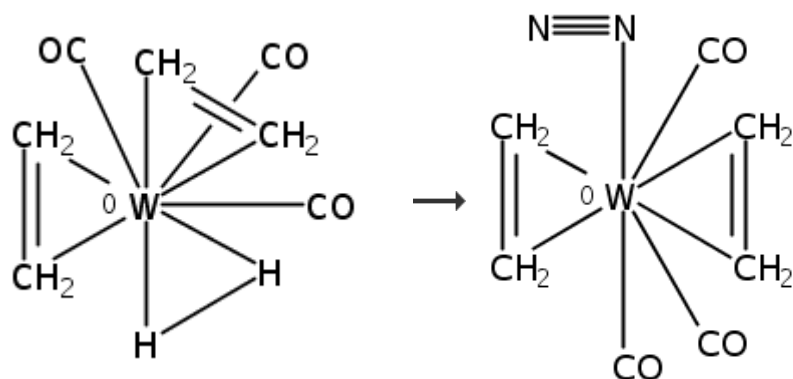
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

38. Single Step



Overview

Steps/Stages

Notes

1.1 R:



thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 1, Steps: 1, Stages:
1, Most stages in any one step: 1

References

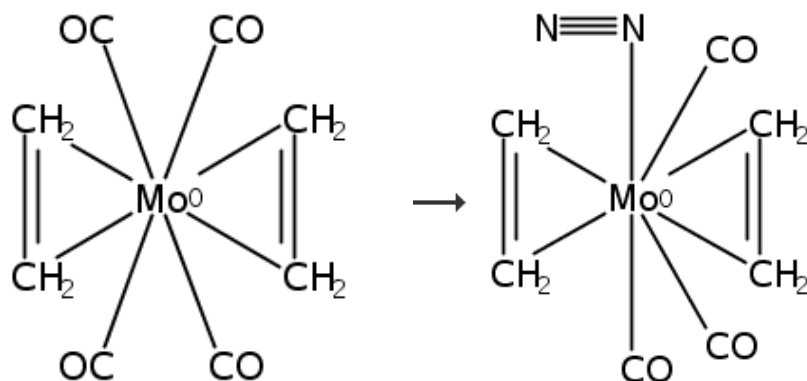
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

39. 2 Steps



Overview

Steps/Stages

1.1 R:D₂

2.1 R:



Notes

1) photochem., supercritical Xenon medium, 2) thermal, supercritical Xenon medium,
Reactants: 1, Reagents: 2, Steps: 2, Stages:
2, Most stages in any one step: 1

References

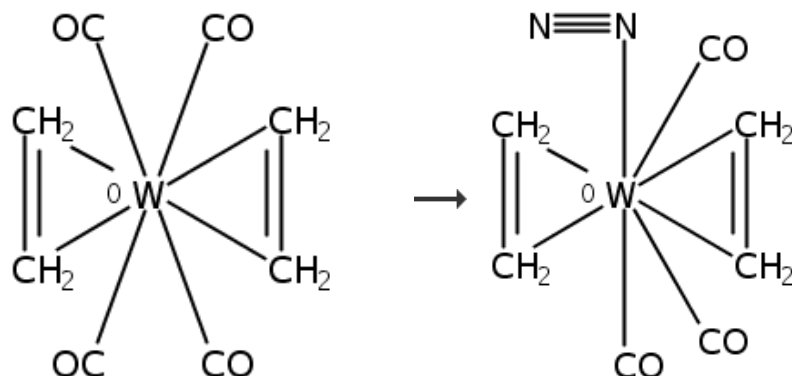
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

40. 2 Steps



Overview

Steps/Stages

1.1 R:H₂

2.1 R:



Notes

1) photochem., supercritical Xenon medium, 2) thermal, supercritical Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

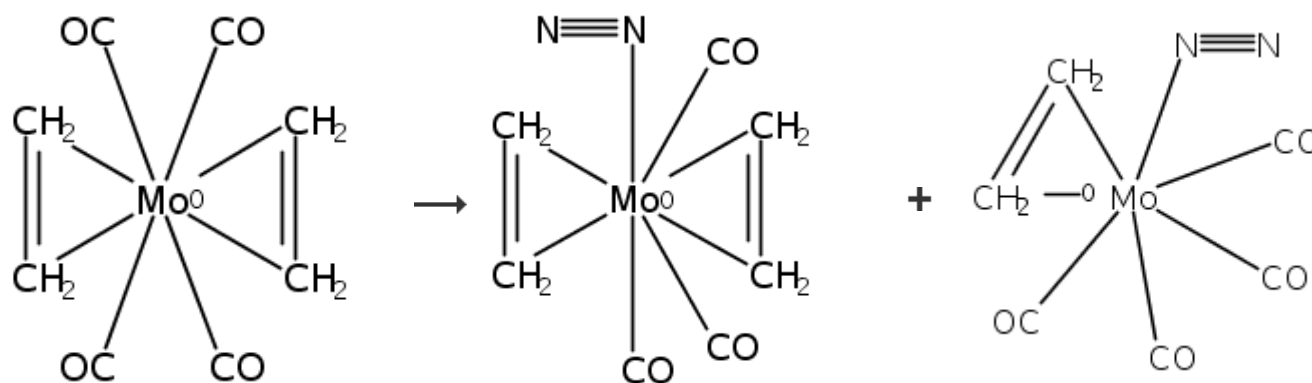
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

41. Single Step



Overview

Steps/Stages

Notes

1.1 R:



photochem., supercritical Xenon medium,
 Reactants: 1, Reagents: 1, Steps: 1, Stages:
 1, Most stages in any one step: 1

References

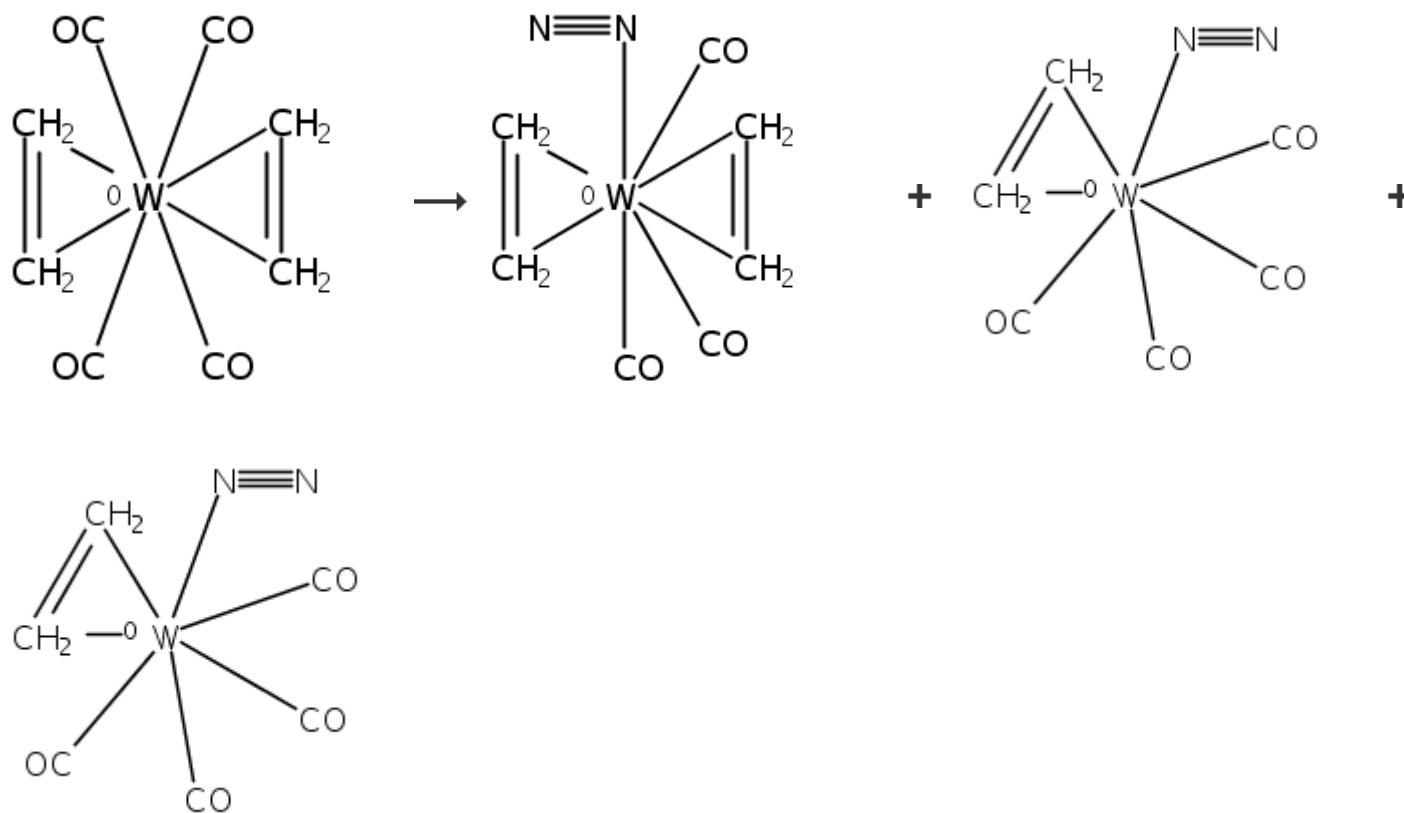
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
 1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

42. Single Step



[Overview](#)

Steps/Stages

Notes

1.1 R:



photochem., supercritical Xenon medium,
 Reactants: 1, Reagents: 1, Steps: 1, Stages:
 1, Most stages in any one step: 1

References

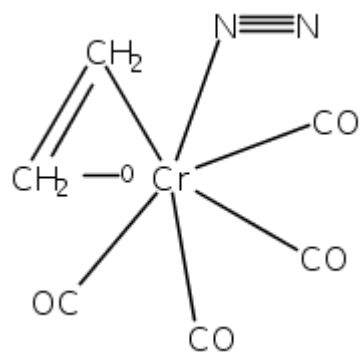
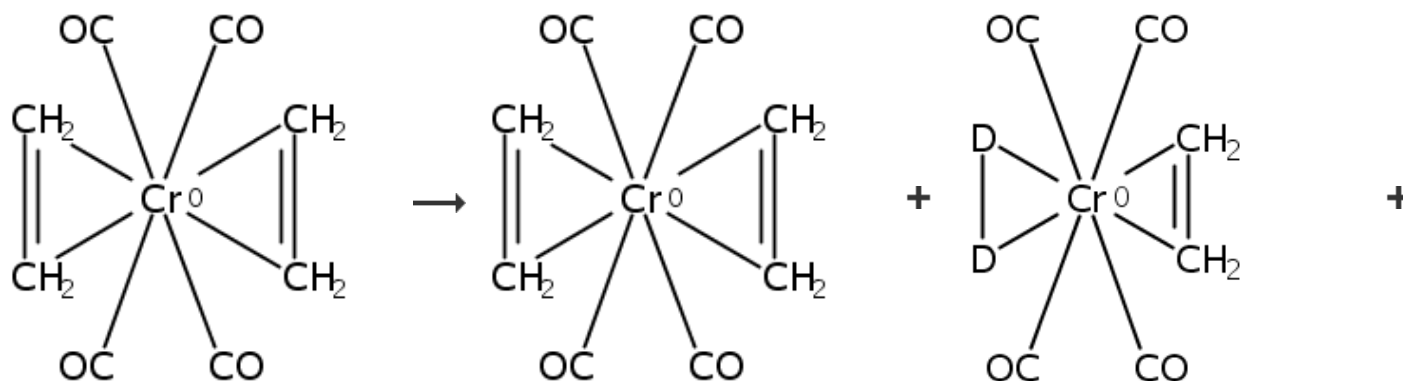
[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
 1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

43. Single Step



[Overview](#)

Steps/Stages

Notes

1.1 R:

R:D₂

photochem., supercritical Xenon medium,
Reactants: 1, Reagents: 2, Steps: 1, Stages:
1, Most stages in any one step: 1

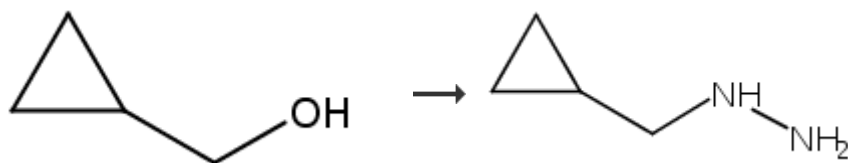
References

[Infrared spectroscopic studies on the photocatalytic hydrogenation of norbornadiene by Group 6 metal carbonyls.](#)
1. The role of H₂ and the characterization of nonclassical dihydrogen complexes, (η⁴-norbornadiene)M(CO)₃(η²-H₂)

By Jackson, Sarah A. et al

From Journal of the American Chemical Society, 112(3), 1221-33; 1990

CASREACT®: Copyright © 2020 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.

44. 2 Steps**Overview****Steps/Stages**1.1 R:*p*-Tosyl chloride

R:

R:C₅H₅N, S:C₅H₅N**Notes**

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

References

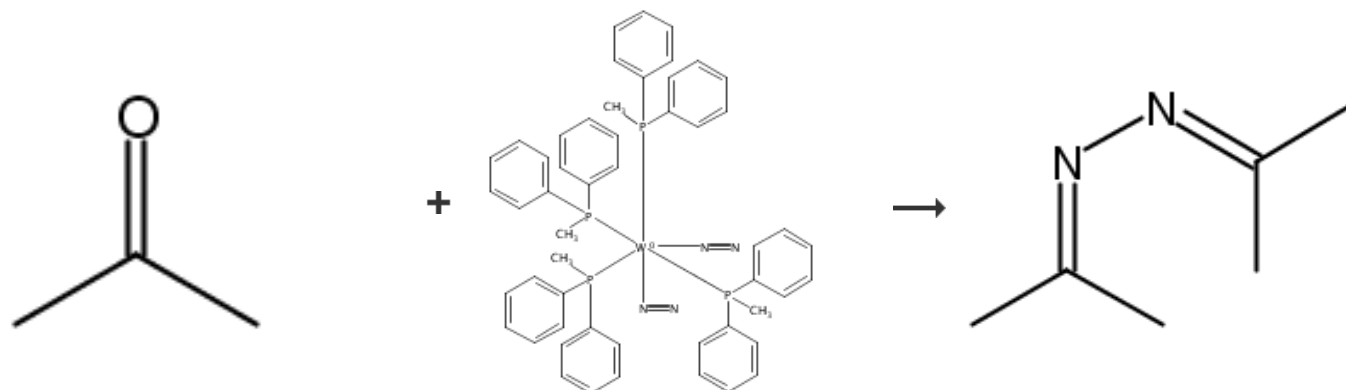
[The cyclopropylmethyl free-radical clock. Calibration for the 30-89° C range](#)

By Mathew, L. and Warkentin, J.

From Journal of the American Chemical Society, 108(25), 7981-4; 1986

2.1 R:N₂H₄, R:H₂O, S:EtOH

CASREACT®: Copyright © 2020 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.

45. Single Step

[Overview](#)**Steps/Stages**

1.1 R:MeOH

R:

S:MeOH, S:Me₂CO**Notes**

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

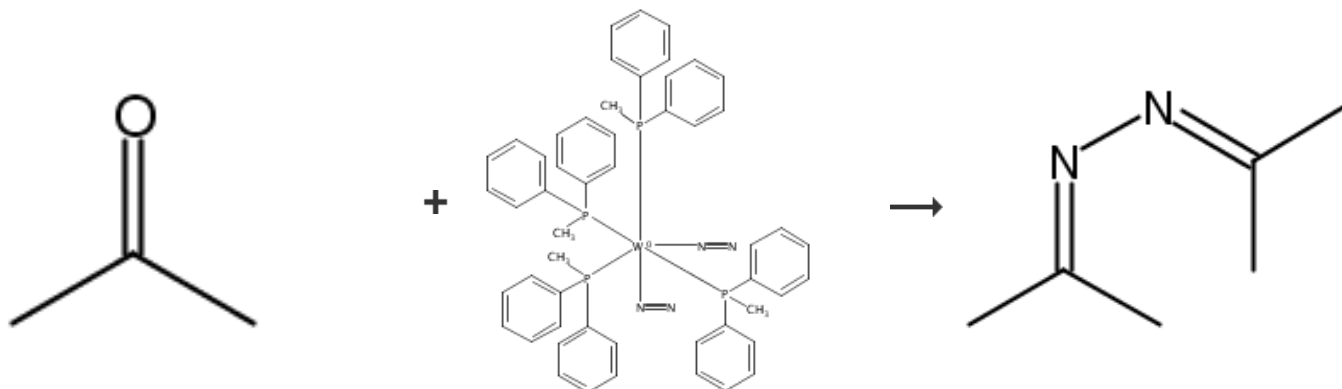
References

[Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten](#)

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

46. Single Step[Overview](#)**Steps/Stages**

1.1 R:MeOH

R:

R:NaOMe, S:MeOH, S:Me₂CO, S:THF**Notes**

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

References

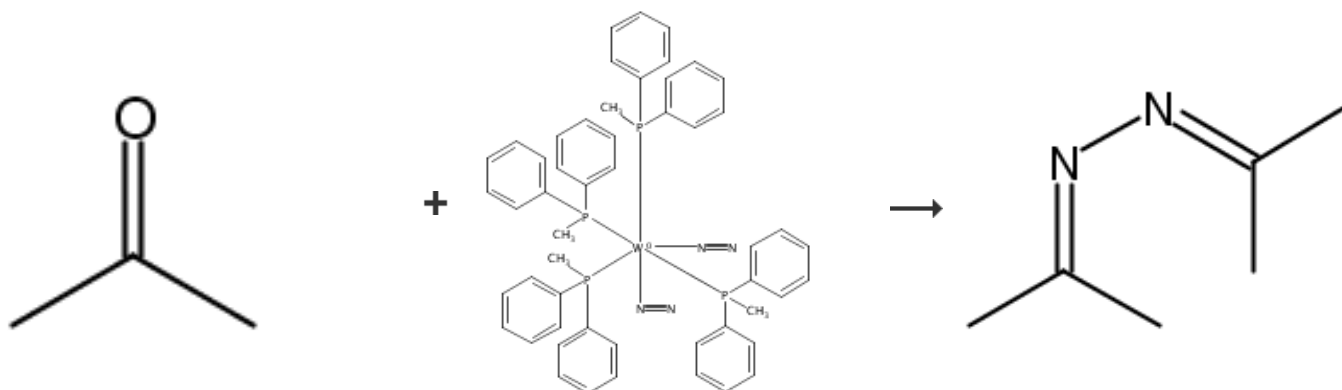
[Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten](#)

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

47. Single Step



Overview

Steps/Stages

1.1 R:MeOH

R:



S:MeOH, S:Me₂CO, S:THF

Notes

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

References

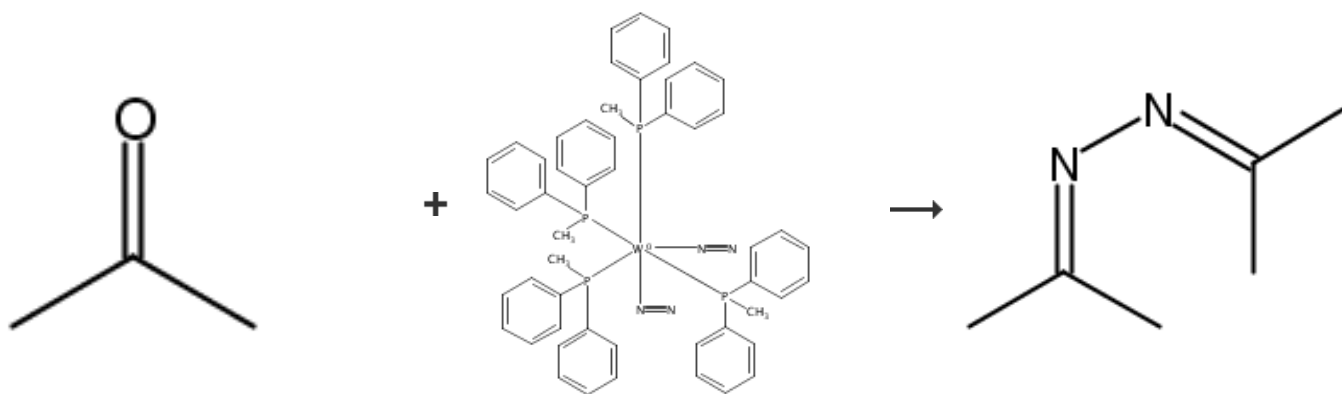
[Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten](#)

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

48. Single Step



Overview

Steps/Stages

Notes

1.1 R:MeOH

R:

R:NaOMe, S:MeOH, S:Me₂CO

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

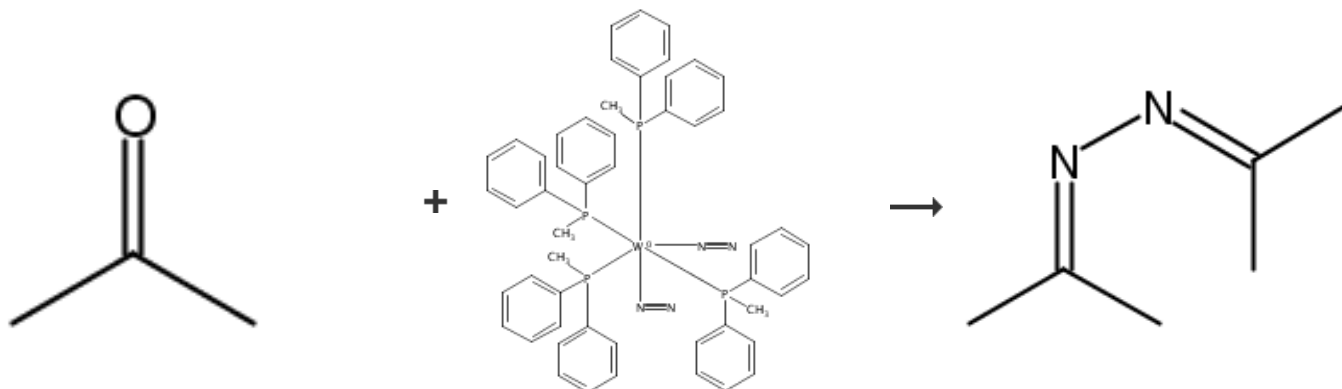
References

Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

49. Single Step**Overview****Steps/Stages**

1.1 R:MeOH

R:

S:MeOH, S:Me₂CO, S:PhMe**Notes**

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

References

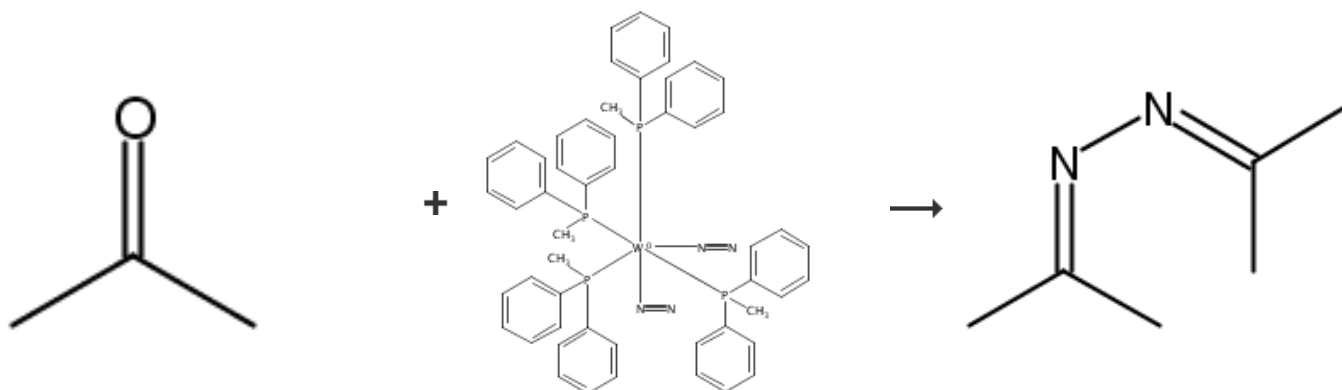
Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

50. Single Step



Overview

Steps/Stages

1.1 R:MeOH

R:



S:MeOH, S:Me₂CO

Notes

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

References

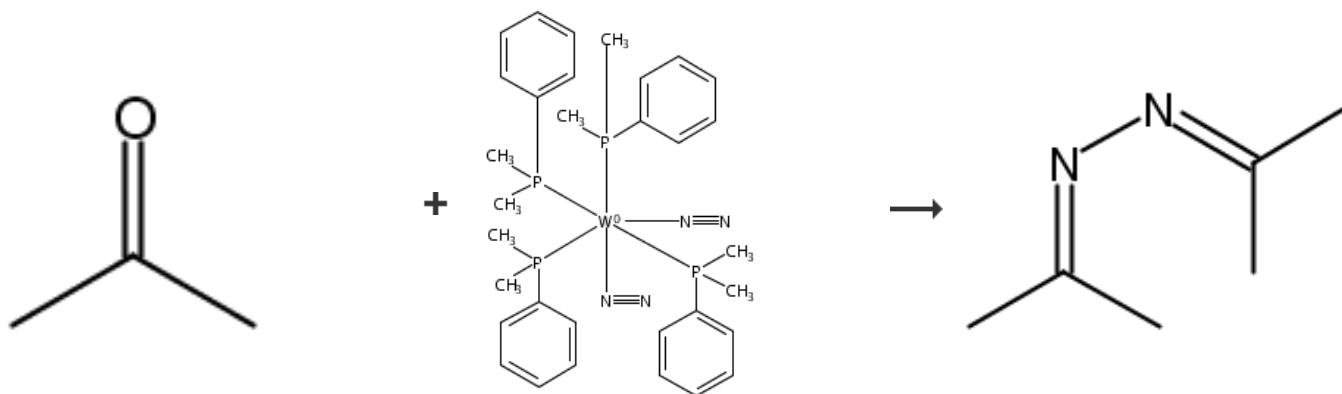
[Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten](#)

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

51. Single Step



Overview

Steps/Stages

Notes

1.1 R:EtOH

R:

S:MeOH, S:Me₂CO

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

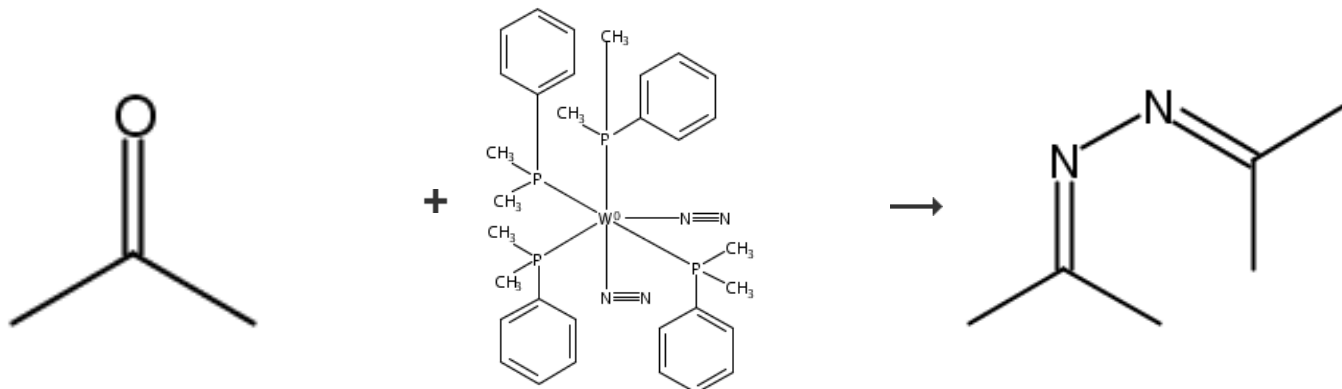
References

Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

52. Single Step**Overview****Steps/Stages**

1.1 R:MeOH

R:

S:MeOH, S:Me₂CO**Notes**

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

References

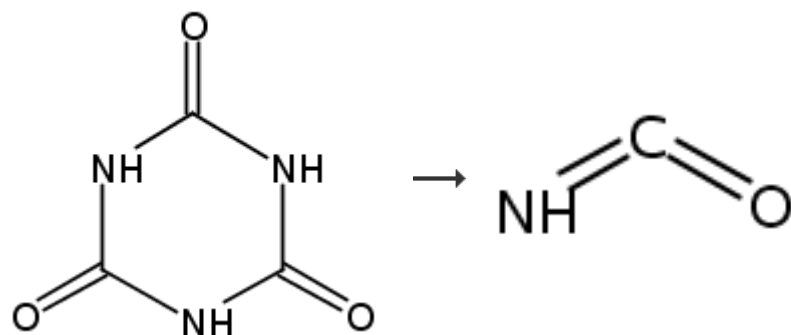
Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

53. Single Step



85%

[Overview](#)**Steps/Stages**

1.1 R:

**Notes**

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

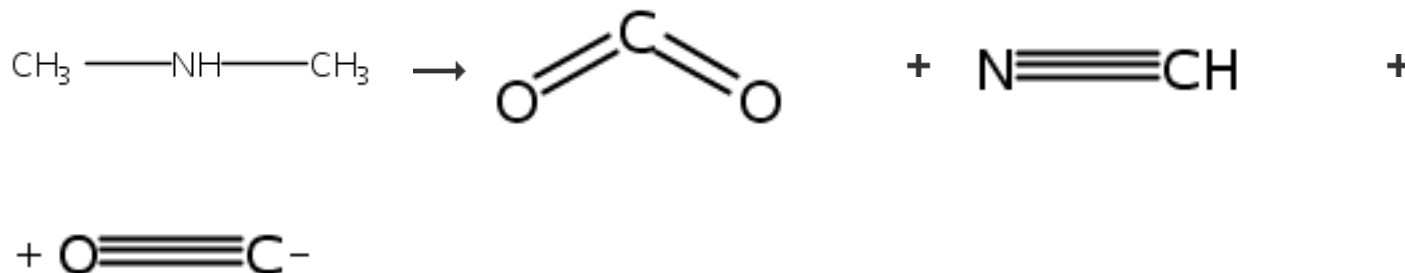
References

[Kinetics of isocyanuric acid pyrolysis](#)

By Mercadier, J. et al

From Journal of Analytical and Applied Pyrolysis, 28(1), 107-20; 1994

CASREACT®: Copyright © 2020 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.

54. Single Step[Overview](#)**Steps/Stages**

1.1 R:



R:O₂, C:TiO₂, 2.1 s, 573K

Notes

gas phase, thermal, green chemistry, in the dark, continuous flow tubular reactor used, reaction unsuccessful at lower temperature, kinetics studied, optimization study, optimized on reactant concentration, 12% conversion, mechanism studied, Reactants: 1, Reagents: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

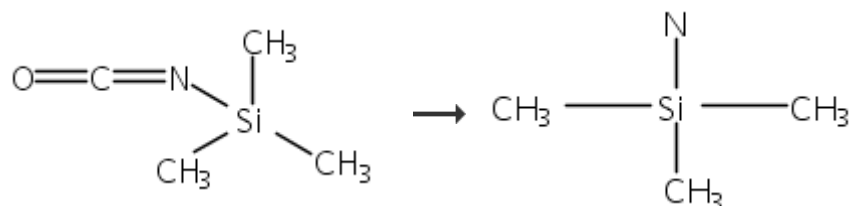
[Gas-phase photocatalytic oxidation of dimethylamine: the reaction pathway and kinetics](#)

By Kachina, Anna et al

From International Journal of Photoenergy, (2), 79847/1-79847/4; 2007

CASREACT®: Copyright © 2020 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.

55. Single Step



Overview

Steps/Stages

1.1 R:Ar
R:



Notes

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

References

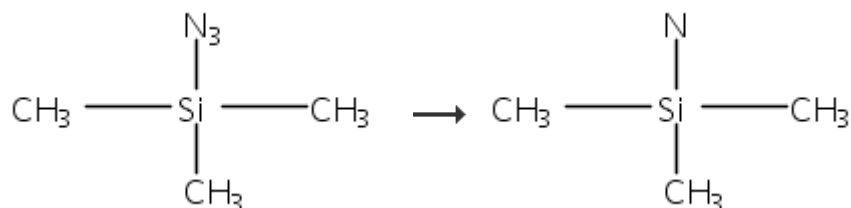
[Observation of discrete trimethylsilylnitrene by matrix-isolation spectroscopy](#)

By Ferrante, Robert F.

From Journal of Physical Chemistry, 94(9), 3502-8; 1990

CASREACT®: Copyright © 2020 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.

56. Single Step



Overview

Steps/Stages

1.1 R:Ar
R:



Notes

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

References

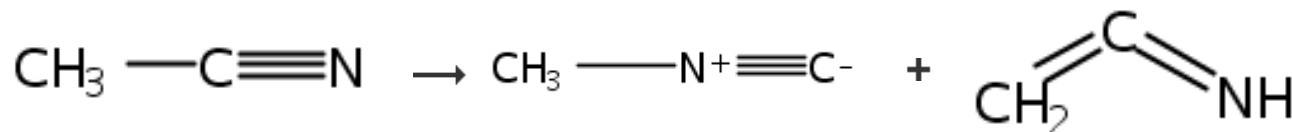
[Observation of discrete trimethylsilylnitrene by matrix-isolation spectroscopy](#)

By Ferrante, Robert F.

From Journal of Physical Chemistry, 94(9), 3502-8; 1990

CASREACT®: Copyright © 2020 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.

57. Single Step



Overview

Steps/Stages

1.1 R:



R:



10-40K

Notes

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

References

[Determination of \[OH3NC\]/\[H2C=C=NH\] Abundance Ratios from N + CH3CN Solid Phase Reaction in the Temperature Range from 10 to 40 K: Application to the Complex Chemistry in Star-Forming Regions](#)

By Krim, Lahouari and Mencos, Alejandro
From ACS Earth and Space Chemistry, 3(6), 973-979; 2019

CASREACT®: Copyright © 2020 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.

58. Single Step

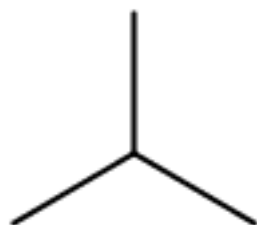


Overview

Steps/Stages

1.1 R:C₂H₆, R:CH₄, R:CH₃CH₂CH₃

R:



R:H₂

R:



C:Me(CH₂)₄Me, 35°C, 0.91 MPa

Notes

gas phase, Reactants: 1, Reagents: 6, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Polymerization process comprising treatment and recycling of effluent streams](#)

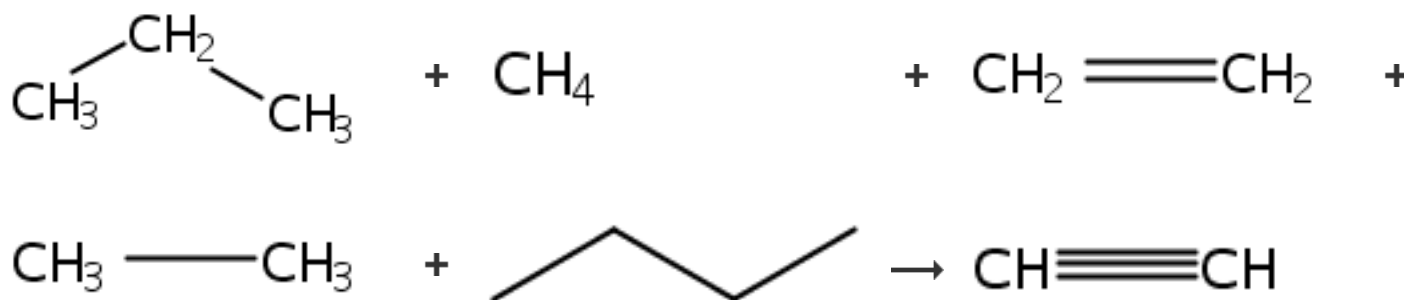
By Marissal, Daniel et al
From PCT Int. Appl., 2013135563, 19 Sep 2013

Experimental Procedure

General Process Ethylene is polymerised in two slurry loop reactors in series to produce a bimodal polyethylene with a density of 948 kg/m³ and a Melt Index (MI₅) of 0.31. In the first reactor ethylene is polymerised in the substantial absence of comonomer, but in the presence of hydrogen and with isobutane as diluent. Polymer from the first reactor is passed to a second reactor wherein further ethylene is polymerised in the presence of 1-hexene as comonomer and the substantial absence of hydrogen, again in the presence of isobutane. Polymer slurry is withdrawn from the first reactor and passed to a contacting vessel in the form of a stirred tank. Polymer slurry is recovered from the base of the contacting vessel and passed to the second reactor. Polymer slurry is withdrawn from the second reactor and passed via a slurry heater, in which the liquid components of the slurry are vaporised, to a flash tank at a pressure of 0.95 MPa. Polymer solids are withdrawn from the flash tank for further processing. The vapour recovered from the flash tank is passed as the first vapour to the contacting vessel where it is contacted with the slurry withdrawn from the first reactor. Vapour is withdrawn from the top of the contacting tank and passed to a fractionator in which it is contacted with a reflux stream. Liquids recovered from the of the fractionator are returned to the contacting tank. The combined "contacting tank/fractionator" is herein referred to as a "fractionation system". Vapour recovered overhead from the fractionator is cooled and partially condensed, then passed to a liquid/vapour (first) separator operating at a temperature of 35°C and a pressure of 0.91 MPa. A portion of the liquid recovered therefrom is utilised as the reflux stream to the fractionator. The vapour recovered from the liquid/vapour separator is further cooled and separated at -35°C and 0.91 MPa in a further separator in the high pressure recovery system. Condensed liquid is recycled to the liquid/vapour first separator, whilst overhead vapour is passed to flare. This stream is referred to as the high pressure flare stream. Polymer solids withdrawn from the flash tank are passed for further processing in a flush vessel at a pressure of 0.135 MPa. The flushing takes place by contact in two vertically orientated stages with polymer being introduced at the top and withdrawn from the base of the vessel, and with a recycled flush gas being introduced into the upper stage and nitrogen being introduced into the lower stage. A mixture of the flush gases and recovered diluent/monomer is recovered from the top of the flush vessel, cooled and passed to low pressure treatment steps. A portion is passed to a heavies separation unit to remove heavy components, but the majority (including a recycle stream from the heavies separation unit) is passed to a (second) separator at a pressure of 0.40 MPa and at a temperature of -30°C. From the base of the second separator is recovered a liquid stream which can be recompressed and recycled to the second reactor. Overhead from the second separator is recovered a vapour stream. The majority of this stream is recycled to the flush vessel, but a portion is passed to a flare. This stream is referred to as the low pressure flare stream.

CASREACT®: Copyright © 2020 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.

59. Single Step


[Overview](#)
[Steps/Stages](#)
[Notes](#)

1.1 R:

R:O₂

Reactants: 5, Reagents: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Production of commercial hydrogen and acetylene from propane-butane and liquid hydrocarbons in an electric-arc plasma reactor](#)

By Mosse, A. L. et al

From Journal of Engineering Physics and Thermophysics, 81(4), 652-658; 2008

Experimental Procedure

Experimental Technological Setup. This is represented by a plasma reactor (Fig. 1) consisting of a PDC-3D plasmatron, a sectioned reactor, and a quenching device manufactured in two variants: a quenching section ensuring the distributed injection of water jets perpendicularly to the motion of a plasma jet (the schematic diagram of the entire setup is shown in Fig. 2) and a quenching bunker (see Fig. 1) where pyrolysis gases were quenched by submerging them in the volume of the quenching medium-diesel fuel (the complete schematic diagram of the setup is presented in [9]). Mixtures of propane-butane and air were used as a plasma-generating gas for production of hydrogen and acetylene. The total flow rate of the gas mixture was 12-20 kg / h; its velocity at exit from the plasmatron was 40-95 m /sec and 20-50 m /sec at entry of the pyrogas into the quenching medium (diesel fuel). Both technological setups, as is seen in Fig. 2, also incorporated systems for feeding the plasma-generating gas-nitrogen-and the starting hydrocarbon raw material-propane-butane (liquefied gas), water supply of the plasmatron, the sections of the reactor and the quenching, bunker, and for sampling pyrolysis products for an analysis. A chemical analysis of the gas samples was made by the gas-adsorption-chromatography method. The gas samples for analysis were taken from the plasma reactor into gas pipets via a special filter with glass wool for separation of moisture and precipitation of solid soot particles using a water-cooled sampler and a vacuum pump [7]. A gas chromatographic analysis of hydrocarbon gases S1-S4 in the samples taken was made with a Tsvet-100M chromatograph (model 164) fitted with a plasma-ionization detector. An LKhM-8MD gas chromatograph fitted with a heat-conduction detector was used for measuring the concentrations of hydrogen, oxygen, nitrogen, and methane in the pyrogas samples.

CASREACT®: Copyright © 2020 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.

60. Single Step

11%

Overview**Steps/Stages**

1.1 R:

R:O₂, C:150703-91-6**Notes**

10.7% ethane conversion with 98% selectivity; 20 h at 510°, Reactants: 1, Reagents: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

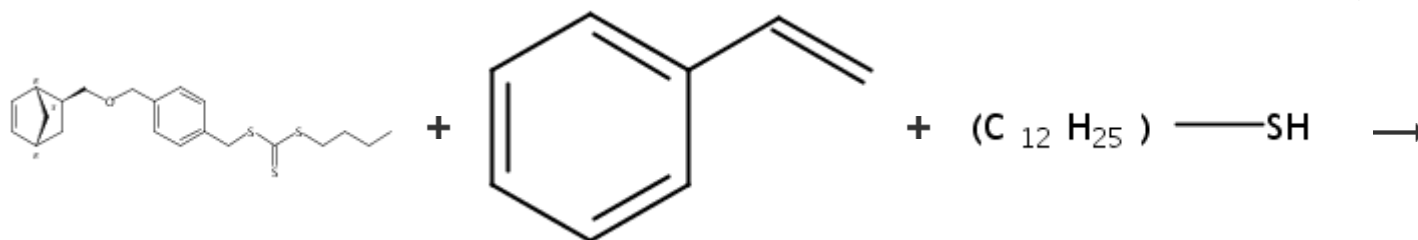
[Catalyst and process for oxidative dehydrogenation of ethane into ethylene](#)

By Kuttyrev, Michail et al

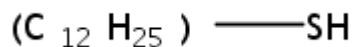
From Eur. Pat. Appl., 544372, 02 Jun 1993

CASREACT®: Copyright © 2020 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.

61. 2 Steps



[Step 2.1]



reaction product with norbornenyl
Bu thioc

Overview

Steps/Stages

1.1 C:AIBN, S:PhMe, 18 h, 70°C

1.2 R:



2.1 C:24650-42-8, S:THF, 10 min

Notes

1) optimization study, optimized on temperature, optimized on time, Schlenk tube used, polymer contains with Nb-TTC, liquid nitrogen used (stage 2), 2) photochemical, optimization study, optimized on time, stoichiometry of reactant, Schlenk tube used, UV lamp used (365nm), Click conditions used, thiol-ene modification of polystyrene with norbornene, Reactants: 3, Reagents: 1, Catalysts: 2, Solvents: 2, Steps: 2, Stages: 3, Most stages in any one step: 2

References

[Norbornenyl-Based RAFT Agents for the Preparation of Functional Polymers via Thiol-Ene Chemistry](#)

By Stamenovic, Milan M. et al

From *Macromolecules* (Washington, DC, United States), 44(14), 5619-5630; 2011

Experimental Procedure

Step 1

General Polymerization Procedures. All polymerizations were performed in a Schlenk tube and AIBN was employed as the thermal initiator. A typical polymerization procedure is as follows (Scheme 3): monomer, norbornenyl CTA, initiator, and solvent ($[M]_0/[CTA]_0/[AIBN]_0 = 200/1/0.1$) were placed in a Schlenk tube, which was degassed by three freeze-pump-thaw cycles, backfilled with nitrogen, sealed, and heated in an oil bath at the indicated temperature. The applied reaction time ranged from 2 to 48 h, depending on the targeted molecular weight and research purpose. The concentrations and reaction conditions for each specific reaction are given in Tables 1, 2 and 3. All reaction mixtures were quenched in liquid nitrogen. The polymerization kinetics using the various Nb-CTA were monitored by 1H NMR and SEC. For each single reaction, at specific time intervals during the polymerization, small aliquots were withdrawn from the polymerization solution and analyzed by 1H NMR spectroscopy for the determination of monomer conversions and by SEC for the determination of the molecular weights and polydispersities (PDI) of the resulting polymers. The purified polymers were obtained by repeated precipitation in 10-fold excess of corresponding nonsolvent (cold methanol for PS, 50/50 cold mixture of methanol and water for PMA/PEEA, cold hexane for PVAc). Alternatively, PEEA and PVAc were purified by removing monomer in vacuo. Polymers were collected either by decantation or filtration and dried at least overnight in vacuo. Entry 9.

Step 2

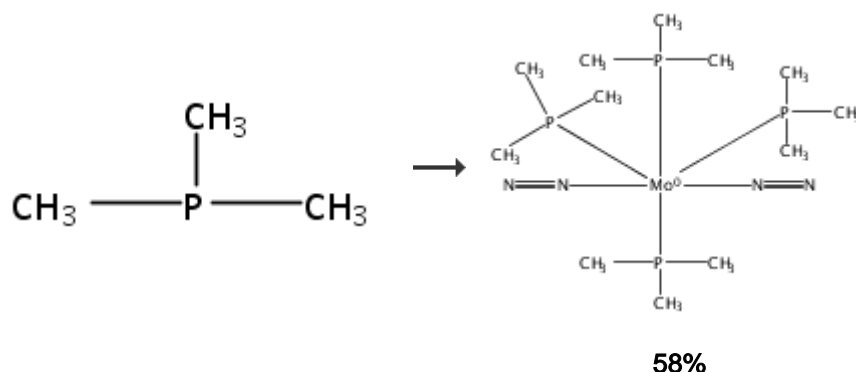
General Procedure for Thiol-Ene Reaction. A typical procedure is as follows (Table 4, entry 1): Nb-containing polymer (PS, 0.08 g, 1.6×10^{-5} mol, $M_n = 5000$ g 3 mol^{-1} , PDI = 1.27) and thiol-containing compound (dodecanethiol, benzyl mercaptan or 2-mercaptopropionic acid), and 0.2 equiv of photoinitiator 2,2-dimethoxy-2-phenylacetophenone (DMPA, 0.8 mg, 3.2×10^{-6} mol) were placed in a Schlenk tube and dissolved in minimal amounts of THF (0.9 mL) required to dissolve all components. The tube was sealed and the reaction mixture was degassed with three freeze-pump-thaw cycles, backfilled with nitrogen, and placed under an irradiation source. The flask was irradiated with a 365nm UV lamp, and was positioned at 45° toward the irradiation source, to ensure equal irradiation over the whole reacting mixture (distance between irradiation source and the flask ca. 20 cm). Irradiation time was typically 15 min except otherwise mentioned. The resulting functional polymers were precipitated three times in corresponding cold non solvent as stated above.

Reaction Protocol**Procedure**

1. Place monomer, norbornenyl CTA, initiator and solvent ($[M]_0/[CTA]_0/[AIBN]_0 = 200/1/0.1$) in a Schlenk tube.
2. Degas the mixture by three freeze-pump-thaw cycles.

[View more...](#)[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

62. Single Step**Overview****Steps/Stages**

1.1 R:



R:MoCl₅, R:Mg, S:THF, -78°C; 30 min, -78°C; -78°C → rt;
overnight, rt

Notes

stereoselective, under N₂ atmosphere, activated magnesium used, Reactants: 1, Reagents: 3, Solvents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[One-Pot Synthesis of Mo\(0\) Dinitrogen Complexes Possessing Monodentate and Multidentate Phosphine Ligands](#)

By Ning, Yalan et al

From Inorganic Chemistry, 51(5), 3051-3058; 2012

Experimental Procedure

Synthesis of *cis*-Mo(N₂)₂(PMe₃)₄ (1). Under a N₂ atmosphere, a 100-mL Schlenk flask containing MoCl₅ (1.0 g, 3.64 mmol, 1 equiv), and a Teflon-coated magnetic stir bar were cooled in an acetone/dry ice bath. A precooled (−78 °C) solution of PMe₃ (1.24 g, 16.1 mmol, 4.42 equiv) in THF (40 mL) and activated magnesium (3.0 g, excess) were added sequentially. The bath was removed after 30 min, and the resulting dark orange reaction mixture was allowed to stir overnight. The next day, the reaction mixture was evaporated to dryness under reduced pressure. The remaining brown solid was taken into the glovebox, extracted with hexanes (30 mL), and vacuum-filtered through a plug of Celite. The filtrate was evaporated to dryness under reduced pressure to remove the solvents and PMe₃. The remaining dark-yellow solid was redissolved in hexanes (15 mL) and filtered through a syringe filter (PFTE, 0.45 μm). The collected solution was concentrated to 10 mL and kept at −30 °C overnight to crystallize out a light yellow powder, which was isolated by filtration, washed with cold methanol (2–3 mL), and dried under reduced pressure. This recrystallization protocol was repeated two times to afford the pure product. *cis*-Mo(N₂)₂(PMe₃)₄ (1), light yellow powder (0.96 g, 58%) ¹H NMR (400.6 MHz, C₆D₆): δ 1.32 (t, 18H, *J* = 20.0 Hz, PMe₃), 1.08 (t, 18H, *J* = 20.0 Hz, PMe₃). ³¹P NMR (121.6 MHz, C₆D₆): δ 6.5 (t, 2P, *J* = 18.0 Hz, PMe₃), 4.4 (t, 2P, *J* = 18.0 Hz, PMe₃). ICP-OES: calc. = 25.24 wt % Mo, found = 26.30 wt %; calc. = 32.52 wt % P, found = 31.03 wt %.

Reaction Protocol

Procedure

1. Cool a 100-mL Schlenk flask containing MoCl₅ (1.0 g, 3.64 mmol, 1 equivalent) and a Teflon-coated magnetic stir bar in an acetone/dry ice bath under a N₂ atmosphere.
2. Add a precooled (−78 °C) solution of PMe₃ (1.24 g, 16.1 mmol, 4.42 equivalents) in THF (40 mL) and activated magnesium (3.0 g, excess) to the reaction mixture.

[View more...](#)

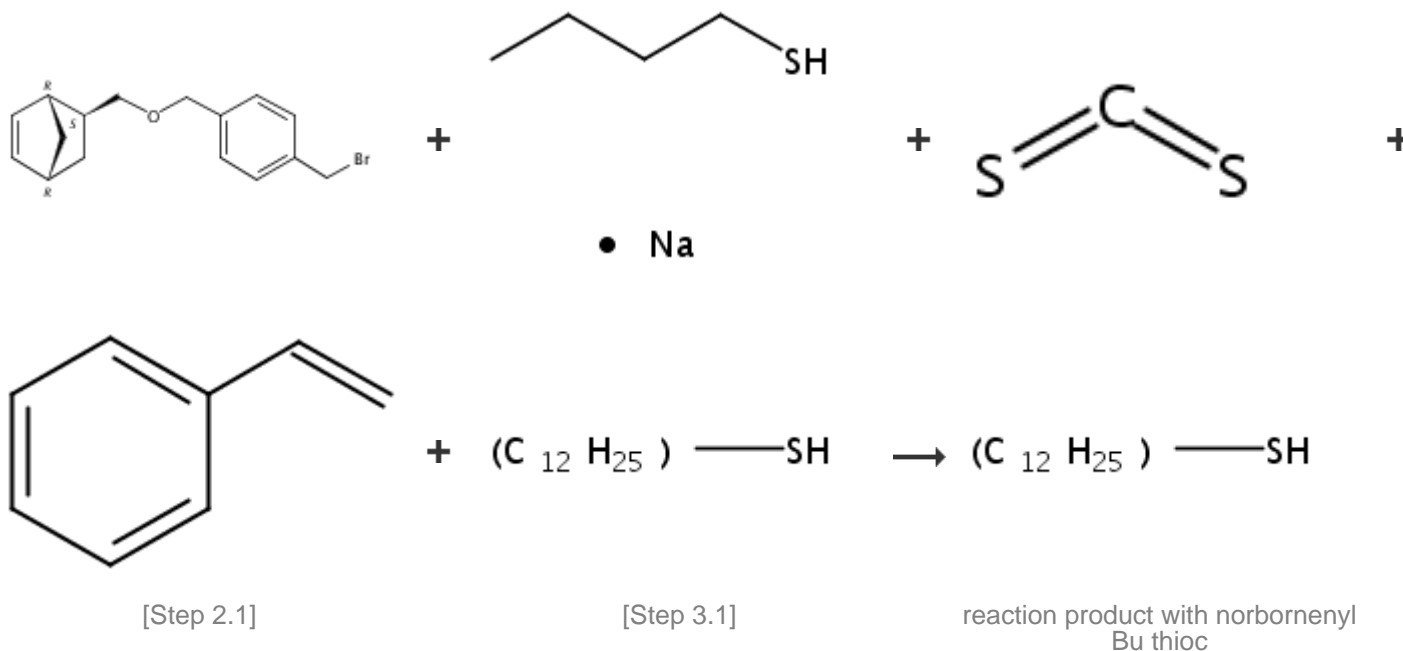
Available Experimental Data

¹H NMR, ³¹P NMR, Elemental Analysis

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

63. 3 Steps



[Overview](#)

Steps/Stages

Notes

- 1.1 S:THF, overnight, rt
 1.2 R:NaCl, S:H₂O, rt
 2.1 C:AIBN, S:PhMe, 18 h, 70°C
 2.2 R:



- 3.1 C:24650-42-8, S:THF, 10 min

2) optimization study, optimized on temperature, optimized on time, Schlenk tube used, polymer contains with Nb-TTC, liquid nitrogen used (stage 2), 3) photochemical, optimization study, optimized on time, stoichiometry of reactant, Schlenk tube used, UV lamp used (365nm), Click conditions used, thiol-ene modification of polystyrene with norbornene, Reactants: 5, Reagents: 2, Catalysts: 2, Solvents: 3, Steps: 3, Stages: 5, Most stages in any one step: 2

References

Norbornenyl-Based RAFT Agents for the Preparation of Functional Polymers via Thiol-Ene Chemistry

By Stamenovic, Milan M. et al

From Macromolecules (Washington, DC, United States), 44(14), 5619-5630; 2011

Experimental Procedure

Step 1

Synthesis of 4-((2-exo)-Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)-methyl)benzyl Butyl Carbonotrithioate (Norbornenyl-Functionalized CTA, Trithiocarbonate, Nb-TTC). A solution of benzylic bromide (**4**) (1.47 g, 4.785 mmol) in anhydrous THF (10 mL) was transferred (via cannulation) to a mixture of sodium 1-butanethiolate (805 mg, 7.177 mmol) and CS₂ (432 µL, 7.177 mmol) in anhydrous THF (15 mL). The resulting yellow mixture was stirred overnight at room temperature. The reaction was quenched with brine (100 mL) and the aqueous layer extracted with diethyl ether (3x150 mL). The combined organic extracts were dried (MgSO₄), filtered and evaporated *in vacuo*. The crude norbornenyl RAFT CTA (Nb-TTC) was purified by column chromatography (n-hexane/CH₂Cl₂: 8/2) and isolated as a yellow oil in 98% (1.846 g, 4.702 mmol) yield. C₂₁H₂₈OS₃ (392.64 g/mol). ¹HNMR (CDCl₃) δ (ppm): 7.34-7.28 (m, 4H), 6.10 (dd, 1H, 5.6, 3.0 Hz), 6.05 (dd, 1H, 5.6, 2.8 Hz), 4.61 (s, 2H), 4.50 (s, 2H), 3.52 (dd, 1H, 9.2, 6.2 Hz), 3.37 (m, 3H), 2.79 (m, 2H), 1.77-1.64 (m, 3H), 1.43 (m, 2H), 1.34-1.21 (m, 3H), 1.11 (app dt, 1H, 11.6, 3.9 Hz), 0.94 (t, 3H, 7.3 Hz). ¹³CNMR (CDCl₃) δ (ppm): 224.0, 138.6, 136.9, 136.8, 134.5, 129.5, 128.1, 75.3, 72.9, 45.2, 44.0, 41.8, 41.3, 39.1, 37.0, 30.3, 30.0, 22.3, 13.8. ESI-MS (*m/z*): 393 (38%) [M + H⁺], 269 (100%).

Step 2

General Polymerization Procedures. All polymerizations were performed in a Schlenk tube and AIBN was employed as the thermal initiator. A typical polymerization procedure is as follows (Scheme 3): monomer, norbornenyl CTA, initiator, and solvent ([M]₀/[CTA]₀/ [AIBN]₀ = 200/1/0.1) were placed in a Schlenk tube, which was degassed by three freeze-pump-thaw cycles, backfilled with nitrogen, sealed, and heated in an oil bath at the indicated temperature. The applied reaction time ranged from 2 to 48 h, depending on the targeted molecular weight and research purpose. The concentrations and reaction conditions for each specific reaction are given in Tables 1, 2 and 3. All reaction mixtures were quenched in liquid nitrogen. The polymerization kinetics using the various Nb-CTA were monitored by ¹HNMR and SEC. For each single reaction, at specific time intervals during the polymerization, small aliquots were withdrawn from the polymerization solution and analyzed by ¹H NMR spectroscopy for the determination of monomer conversions and by SEC for the determination of the molecular weights and polydispersities (PDI) of the resulting polymers. The purified polymers were obtained by repeated precipitation in 10-fold excess of corresponding nonsolvent (cold methanol for PS, 50/50 cold mixture of methanol and water for PMA/PEEA, cold hexane for PVAc). Alternatively, PEEA and PVAc were purified by removing monomer *in vacuo*. Polymers were collected either by decantation or filtration and dried at least overnight *in vacuo*. Entry 9.

Step 3

General Procedure for Thiol-Ene Reaction. A typical procedure is as follows (Table 4, entry 1): Nb-containing polymer (PS, 0.08 g, 1.6 × 10⁻⁵ mol, *M*_n = 5000 g mol⁻¹, PDI = 1.27) and thiol-containing compound (dodecanethiol, benzyl mercaptan or 2-mercaptopropionic acid), and 0.2 equiv of photoinitiator 2,2-dimethoxy-2-phenylacetophenone (DMPA, 0.8 mg, 3.2 × 10⁻⁶ mol) were placed in a Schlenk tube and dissolved in minimal amounts of THF (0.9 mL) required to dissolve all components. The tube was sealed and the reaction mixture was degassed with three freeze-pump-thaw cycles, backfilled with nitrogen, and placed under an irradiation source. The flask was irradiated with a 365nm UV lamp, and was positioned at 45° toward the irradiation source, to ensure equal irradiation over the whole reacting mixture (distance between irradiation source and the flask ca. 20 cm). Irradiation time was typically 15 min except otherwise mentioned. The resulting functional polymers were precipitated three times in corresponding cold non solvent as stated above.

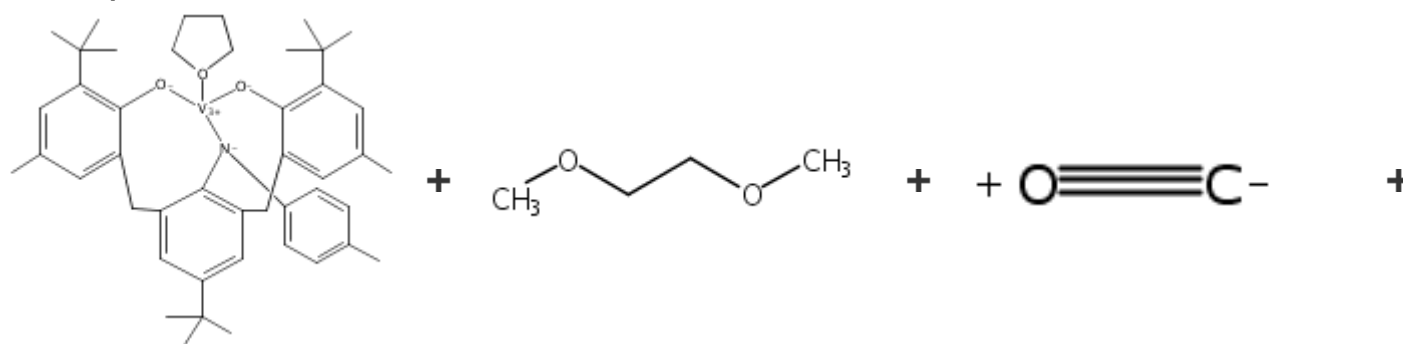
Reaction Protocol

Procedure

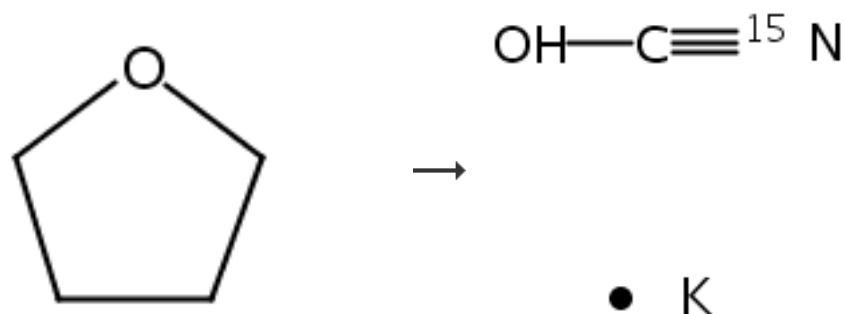
1. Transfer a solution of benzylic bromide (1.47 g, 4.785 mmol) in anhydrous THF (10 mL) (through cannulation) to a mixture of sodium 1-butanethiolate (7.177 mmol) and CS₂ (432 µL, 7.177 mmol) in anhydrous THF (15 mL).
2. Stir the resulting yellow mixture overnight at room temperature.

[View more...](#)**Available
Experimental
Data**¹H NMR, ¹³C NMR, Mass Spec, State[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

64. 4 Steps

[Step 3.1]



[Step 3.1]

[Overview](#)**Steps/Stages****Notes**

1.1 R:KH

R:



S:THF, 6 h, rt

1.2 S:PhMe

2.1 R:*p*-Benzoquinone, S:THF, overnight, rt

3.1 2 d, 2.2 MPa

4.1 R:MeC.tbd.CMe, S:THF, overnight, rt

3) high pressure, Reactants: 4, Reagents: 4,
Solvents: 2, Steps: 4, Stages: 5, Most stages
in any one step: 2

References

Nitrogen Atom Transfer from a Dinitrogen-Derived Vanadium Nitride Complex to Carbon Monoxide and Isocyanide

By Ishida, Yutaka and Kawaguchi, Hiroyuki

From Journal of the American Chemical Society, 136(49), 16990-16993; 2014

Experimental Procedure

Step 1

Synthesis of $[\{K(DME)\}_2\{(ONO)V(\mu-N)\}]_2$ (2). A suspension of KH (40.0 mg, 1.00 mmol) in THF (5 mL) was slowly added to a solution of $[(ONO)V(THF)]$ (1) (358 mg, 0.503 mmol) in THF (10 mL) under an atmosphere of dinitrogen, and then stirred for 6 h at room temperature, during which time the color of the solution gradually changed from forest green to dark brown. All volatiles were removed in vacuo to produce a dark brown residue. Extraction with toluene/DME, centrifugation, and removal of the solvent in vacuo afforded a brown solid, which was washed with DME. **Synthesis of $[\{K(DME)\}_2\{(ONO)V(\mu-N)\}]_2$ (2- ^{15}N).** The same procedure as used for 2 was followed. The reaction of 1 (370 mg, 0.520 mmol) with KH (64.0 mg, 1.60 mmol) in THF (15 mL) under $^{15}N_2$ gave the ^{15}N isotopologue 2- ^{15}N . Yield 234 mg, 49%.

Step 2

General/Typical Procedure: **Synthesis of $[\{K(DME)\}(ONO)VN\}]_2$ (3).** To a dark brown solution of 2 (171 mg, 93.7 μ mol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μ mol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. The synthesis of the ^{15}N isotopologue $[\{K(DME)\}(ONO)V(^{15}N)\}]_2$ (3- ^{15}N) was prepared following the same procedure above by using 2- ^{15}N . Data for 3- ^{15}N . IR (KBr; ν/cm^{-1}): 975 ($\nu^{15}N$). ^{15}N NMR (C_6D_6 , 131.6 MHz, δ/ppm): 958 ($\Delta\nu_{1/2} = 43$ Hz).

Step 3

General/Typical Procedure: **Reaction of 3 with CO.** A light yellow solution of 3 (20.2 mg, 13 μ mol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. The synthesis of $[\{K(DME)\}(ONO)V(^{15}NCO)\}]_2$ (4- ^{15}N) was prepared following the same procedure above by using 3- ^{15}N . Data for 4- ^{15}N . IR (KBr; ν/cm^{-1}): 2149 ($\nu^{15}NCO$).

Step 4

General/Typical Procedure: **Reaction of 7 with 2-Butyne.** To a solution of 7 (20.4 mg, 25.7 μ mol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The mixture of 7- ^{15}N and 2-butyne was analogously treated as described above, giving $K^{15}NCO$. $\nu^{15}NCO = 2149$ cm^{-1} , ^{15}N NMR (D_2O , 131.6 MHz, δ/ppm): 68.6.

Reaction Protocol

Procedure

1. Add a suspension of KH (64.0 mg, 1.60 mmol) in THF (15 mL) to a solution of [(ONO) V (THF)] (370 mg, 0.520 mmol) in THF (10 mL) under an atmosphere of dinitrogen.
2. Stir the mixture for 6 h at room temperature.

[View more...](#)

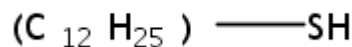
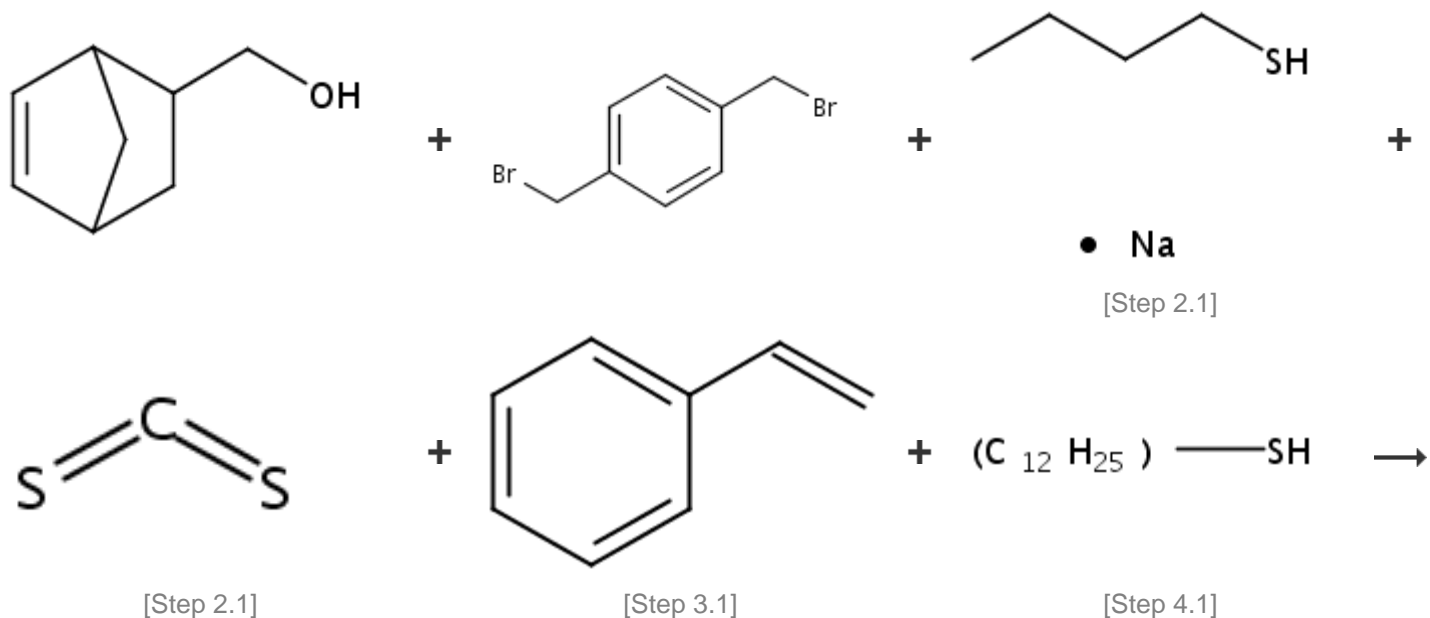
Available Experimental Data

IR, ^{15}N NMR, State

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

65. 4 Steps



reaction product with norbornenyl
Bu thiol

Overview

Steps/Stages

- 1.1 R:NaH, S:THF, overnight, 80°C
- 2.1 S:THF, overnight, rt
- 2.2 R:NaCl, S:H₂O, rt
- 3.1 C:AIBN, S:PhMe, 18 h, 70°C
- 3.2 R:



- 4.1 C:24650-42-8, S:THF, 10 min

Notes

1) literature preparation, 3) optimization study, optimized on temperature, optimized on time, Schlenk tube used, polymer contains with Nb-TTC, liquid nitrogen used (stage 2), 4) photochemical, optimization study, optimized on time, stoichiometry of reactant, Schlenk tube used, UV lamp used (365nm), Click conditions used, thiol-ene modification of polystyrene with norbornene, Reactants: 6, Reagents: 3, Catalysts: 2, Solvents: 3, Steps: 4, Stages: 6, Most stages in any one step: 2

References

[Norbornenyl-Based RAFT Agents for the Preparation of Functional Polymers via Thiol-Ene Chemistry](#)

By Stamenovic, Milan M. et al

From *Macromolecules* (Washington, DC, United States), 44(14), 5619-5630; 2011

Experimental Procedure

Step 1

Procedure Unavailable

Step 2

Synthesis of 4-([2-exo]-Bicyclo[2.2.1]hept-5-en-2-ylmethoxy)-methylbenzyl Butyl Carbonotrithioate (Norbornenyl-Functionalized CTA, Trithiocarbonate, Nb-TTC). A solution of benzylic bromide (**4**) (1.47 g, 4.785 mmol) in anhydrous THF (10 mL) was transferred (via cannulation) to a mixture of sodium 1-butanethiolate (805 mg, 7.177 mmol) and CS₂ (432 µL, 7.177 mmol) in anhydrous THF (15 mL). The resulting yellow mixture was stirred overnight at room temperature. The reaction was quenched with brine (100 mL) and the aqueous layer extracted with diethyl ether (3x150 mL). The combined organic extracts were dried (MgSO₄), filtered and evaporated *in vacuo*. The crude norbornenyl RAFT CTA (Nb-TTC) was purified by column chromatography (n-hexane/CH₂Cl₂: 8/2) and isolated as a yellow oil in 98% (1.846 g, 4.702 mmol) yield. C₂₁H₂₈OS₃ (392.64 g/mol). ¹HNMR (CDCl₃) δ (ppm): 7.34-7.28 (m, 4H), 6.10 (dd, 1H, 5.6, 3.0 Hz), 6.05 (dd, 1H, 5.6, 2.8 Hz), 4.61 (s, 2H), 4.50 (s, 2H), 3.52 (dd, 1H, 9.2, 6.2 Hz), 3.37 (m, 3H), 2.79 (m, 2H), 1.77-1.64 (m, 3H), 1.43 (m, 2H), 1.34-1.21 (m, 3H), 1.11 (app dt, 1H, 11.6, 3.9 Hz), 0.94 (t, 3H, 7.3 Hz). ¹³CNMR (CDCl₃) δ (ppm): 224.0, 138.6, 136.9, 136.8, 134.5, 129.5, 128.1, 75.3, 72.9, 45.2, 44.0, 41.8, 41.3, 39.1, 37.0, 30.3, 30.0, 22.3, 13.8. ESI-MS (*m/z*): 393 (38%) [M + H⁺], 269 (100%).

Step 3

General Polymerization Procedures. All polymerizations were performed in a Schlenk tube and AIBN was employed as the thermal initiator. A typical polymerization procedure is as follows (Scheme 3): monomer, norbornenyl CTA, initiator, and solvent ([M]₀/[CTA]₀/ [AIBN]₀ = 200/1/0.1) were placed in a Schlenk tube, which was degassed by three freeze-pump-thaw cycles, backfilled with nitrogen, sealed, and heated in an oil bath at the indicated temperature. The applied reaction time ranged from 2 to 48 h, depending on the targeted molecular weight and research purpose. The concentrations and reaction conditions for each specific reaction are given in Tables 1, 2 and 3. All reaction mixtures were quenched in liquid nitrogen. The polymerization kinetics using the various Nb-CTA were monitored by ¹HNMR and SEC. For each single reaction, at specific time intervals during the polymerization, small aliquots were withdrawn from the polymerization solution and analyzed by ¹H NMR spectroscopy for the determination of monomer conversions and by SEC for the determination of the molecular weights and polydispersities (PDI) of the resulting polymers. The purified polymers were obtained by repeated precipitation in 10-fold excess of corresponding nonsolvent (cold methanol for PS, 50/50 cold mixture of methanol and water for PMA/PEEA, cold hexane for PVAc). Alternatively, PEEA and PVAc were purified by removing monomer *in vacuo*. Polymers were collected either by decantation or filtration and dried at least overnight *in vacuo*. Entry 9.

Step 4

General Procedure for Thiol-Ene Reaction. A typical procedure is as follows (Table 4, entry 1): Nb-containing polymer (PS, 0.08 g, 1.6 x 10⁻⁵ mol, *M*_n = 5000 g 3 mol⁻¹, PDI = 1.27) and thiol-containing compound (dodecanethiol, benzyl mercaptan or 2-mercaptopropionic acid), and 0.2 equiv of photoinitiator 2,2-dimethoxy-2-phenylacetophenone (DMPA, 0.8 mg, 3.2 x 10⁻⁶ mol) were placed in a Schlenk tube and dissolved in minimal amounts of THF (0.9 mL) required to dissolve all components. The tube was sealed and the reaction mixture was degassed with three freeze-pump-thaw cycles, backfilled with nitrogen, and placed under an irradiation source. The flask was irradiated with a 365nm UV lamp, and was positioned at 45° toward the irradiation source, to ensure equal irradiation over the whole reacting mixture (distance between irradiation source and the flask ca. 20 cm). Irradiation time was typically 15 min except otherwise mentioned. The resulting functional polymers were precipitated three times in corresponding cold non solvent as stated above.

Reaction Protocol**Procedure**

1. Transfer a solution of benzylic bromide (1.47 g, 4.785 mmol) in anhydrous THF (10 mL) (through cannulation) to a mixture of sodium 1-butanethiolate (7.177 mmol) and CS₂ (432 µL, 7.177 mmol) in anhydrous THF (15 mL).
2. Stir the resulting yellow mixture overnight at room temperature.

[View more...](#)

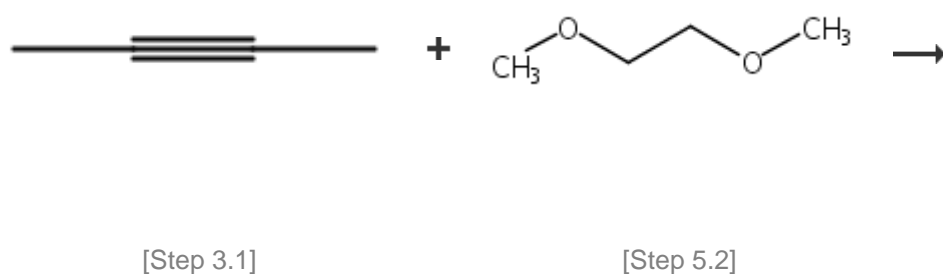
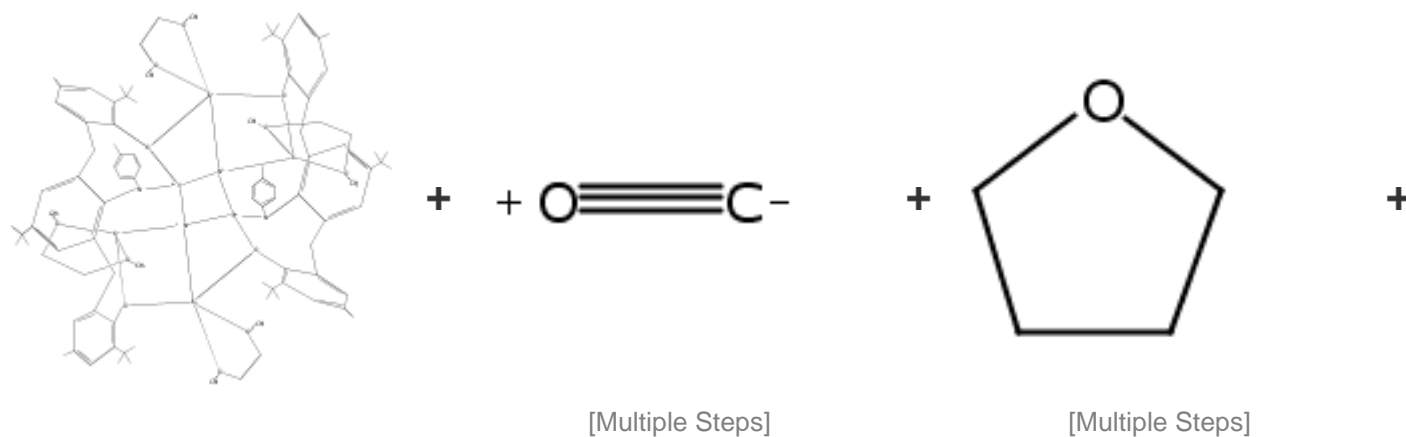
Available Experimental Data

¹H NMR, ¹³C NMR, Mass Spec, State

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

66. 8 Steps



Overview

Steps/Stages

- 1.1 R:*p*-Benzoquinone, S:THF, overnight, rt
- 2.1 2 d, 2.2 MPa
- 3.1 S:PhMe, overnight, rt
- 4.1

- 5.1 R:KH
- R:



- S:THF, 6 h, rt
- 5.2 S:PhMe
- 6.1 R:*p*-Benzoquinone, S:THF, overnight, rt
- 7.1 2 d, 2.2 MPa
- 8.1 R:MeC.tbd.CMe, S:THF, overnight, rt

Experimental Procedure

Notes

2) high pressure, 3) preparative scale, 4) no experimental detail, 7) high pressure, Reactants: 5, Reagents: 4, Solvents: 2, Steps: 8, Stages: 9, Most stages in any one step: 2

References

[Nitrogen Atom Transfer from a Dinitrogen-Derived Vanadium Nitride Complex to Carbon Monoxide and Isocyanide](#)

By Ishida, Yutaka and Kawaguchi, Hiroyuki

From Journal of the American Chemical Society, 136(49), 16990-16993; 2014

Step 1

Synthesis of $[\{K(DME)\}(\{ONO\}VN)]_2$ (3**).** To a dark brown solution of **2** (171 mg, 93.7 μ mol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μ mol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. **3** as light yellow powder (145 mg, 99%). Single crystals of **3** suitable for X-ray diffraction were grown from a saturated hexane solution at -35°C. 1H NMR (C_6D_6 , 500.1 MHz, 338 K, δ /ppm): 1.01 (s, 9H, *t*Bu), 1.69 (s, 18H, *t*Bu), 2.29 (s, 6H, ArO), 2.38 (s, 3H, *p*-Tol), 2.94 (brs, 10H, DME), 3.44 (d, J = 13.6 Hz, 2H, CH_2), 4.42 (d, J = 13.6 Hz, 2H, CH_2), 7.00 (d, J = 1.9 Hz, 2H, ArO), 7.13 (d, J = 1.9 Hz, 2H, ArO), 7.25 (d, J = 8.2 Hz, 2H, *p*-Tol), 7.30 (s, 2H, ArN), 7.82 (br, 2H, *p*-Tol). ^{13}C NMR (C_6D_6 , 125.8 MHz, δ /ppm): 20.9 (Me, *p*-Tol), 21.3 (Me, ArO), 31.4 (CMe_3 , ArN), 31.7 (CMe_3 , ArO), 34.5 (CMe_3 , ArN), 35.67 (CMe_3 , ArO), 35.70 (CH_2), 58.7 (DME), 72.2 (DME), 115.8 (CH), 125.3 (CH), 125.7 (CH), 126.3, 129.2 (CH), 129.9, 130.1 (CH), 138.0, 138.2, 138.3, 147.8, 150.2, 153.5, 162.7. ^{51}V NMR (C_6D_6 , 131.6 MHz, δ /ppm): -350 (br, $\Delta\nu_{1/2}$ = 2101 Hz). Anal. Calcd for $C_{90}H_{120}K_2N_4O_8V_2$: C, 69.03; H, 7.72; N, 3.58. Found: C, 69.07; H, 7.38; N, 3.59. UV-Vis (THF; λ_{max}/nm ($\epsilon/M^{-1} cm^{-1}$)): 387 (22700), 298 (30800). IR (KBr; ν/cm^{-1}): 1007 (VN). **Crystal data for **3**.** $C_{90}H_{120}K_2N_4O_8V_2$. *bu*. 0.5(C_6H_{14}), M = 1609.07, triclinic, space group P-1 (#2), a = 17.064(2) Å, b = 17.2356(15) Å, c = 18.934(2) Å, α = 116.880(9)°, β = 96.196(9)°, γ = 109.314(9)°, V = 4462.9(8) Å³, Z = 2, 56013 reflections measured, 20332 independent reflections (R_{int} = 0.0922). The final R_1 values was 0.0736 ($I > 2\sigma(I)$). The final $wR(F^2)$ value was 0.2237 (all data). The goodness-of-fit on F^2 was 1.135.

Step 2

Reaction of **3 with CO.** A light yellow solution of **3** (20.2 mg, 13 μ mol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. A brown powder of $[\{K(THF)\}(\{ONO\}V(NCO))]$ (**4**) in quantitative yield. Data for **4**. Anal. Calcd for $C_{46}H_{58}KN_2O_4V$: C, 69.67; H, 7.37; N, 3.53. Found: C 69.61; H, 7.51; N, 3.43%. UV-Vis (THF; λ_{max}/nm ($\epsilon/M^{-1} cm^{-1}$)): 732 (650), 585 (430). IR (KBr; ν/cm^{-1}): 2168 (NCO). μ_{eff} (Evans method, THF, 297 K): 2.6 μ_B . μ_{eff} (solid state, 297 K): 2.45 μ_B .

Step 3

Reaction of **7 with 2-Butyne.** To a solution of **7** (20.4 mg, 25.7 μ mol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The brown residue was extracted in hexane and centrifuged to remove an insoluble material, which was identified as KNCO (76% yield). Evaporation of the supernatant to dryness afforded a dark brown powder of $[(ONO)V(\mu^2-MeCCMe)]$ (**8**) (14.2 mg, 79%).¹

Step 4

Procedure Unavailable

Step 5

Synthesis of $[\{K(DME)\}_2(\{ONO\}V(\mu-N))]_2$ (2**).** A suspension of KH (40.0 mg, 1.00 mmol) in THF (5 mL) was slowly added to a solution of $[(ONO)V(THF)]$ (**1**) (358 mg, 0.503 mmol) in THF (10 mL) under an atmosphere of dinitrogen, and then stirred for 6 h at room temperature, during which time the color of the solution gradually changed from forest green to dark brown. All volatiles were removed in vacuo to produce a dark brown residue. Extraction μ_{eff} with toluene/DME, centrifugation, and removal of the solvent in vacuo afforded a brown solid, which was washed with DME. **Synthesis of $[\{K(DME)\}_2(\{ONO\}V(\mu-N))]_2$ (**2- ^{15}N**).** The same procedure as used for **2** was followed. The reaction of **1** (370 mg, 0.520 mmol) with KH (64.0 mg, 1.60 mmol) in THF (15 mL) under $^{15}N_2$ gave the ^{15}N isotopologue **2- ^{15}N** . Yield 234 mg, 49%.

Step 6

General/Typical Procedure: **Synthesis of $[\{K(DME)\}(\{ONO\}VN)]_2$ (**3**).** To a dark brown solution of **2** (171 mg, 93.7 μ mol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μ mol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. The synthesis of the ^{15}N isotopologue $[\{K(DME)\}(\{ONO\}V^{15}N)]_2$ (**3- ^{15}N**) was prepared following the same procedure above by using **2- ^{15}N** . Data for **3- ^{15}N** . IR (KBr; ν/cm^{-1}): 975 ($V^{15}N$). ^{15}N NMR (C_6D_6 , 131.6 MHz, δ /ppm): 958 ($\Delta\nu_{1/2}$ = 43 Hz).

Step 7

General/Typical Procedure: **Reaction of **3** with CO.** A light yellow solution of **3** (20.2 mg, 13 μ mol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. The synthesis of $[\{K(DME)\}(\{ONO\}V(^{15}NCO))]$ (**4- ^{15}N**) was prepared following the same procedure above by using **3- ^{15}N** . Data for **4- ^{15}N** . IR (KBr; ν/cm^{-1}): 2149 (^{15}NCO).

Step 8

General/Typical Procedure: **Reaction of 7 with 2-Butyne.** To a solution of **7** (20.4 mg, 25.7 μmol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The mixture of **7- ^{15}N** and 2-butyne was analogously treated as described above, giving K^{15}NCO . $\nu^{15}\text{NCO} = 2149\text{ cm}^{-1}$, ^{15}N NMR (D_2O , 131.6 MHz, δ/ppm): 68.6.

Reaction Protocol**Procedure**

1. Add a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μmol) to a dark brown solution of the reactant (171 mg, 93.7 μmol) in THF (20 mL) at room temperature.
2. Allow the reaction to proceed overnight, producing an orange suspension.

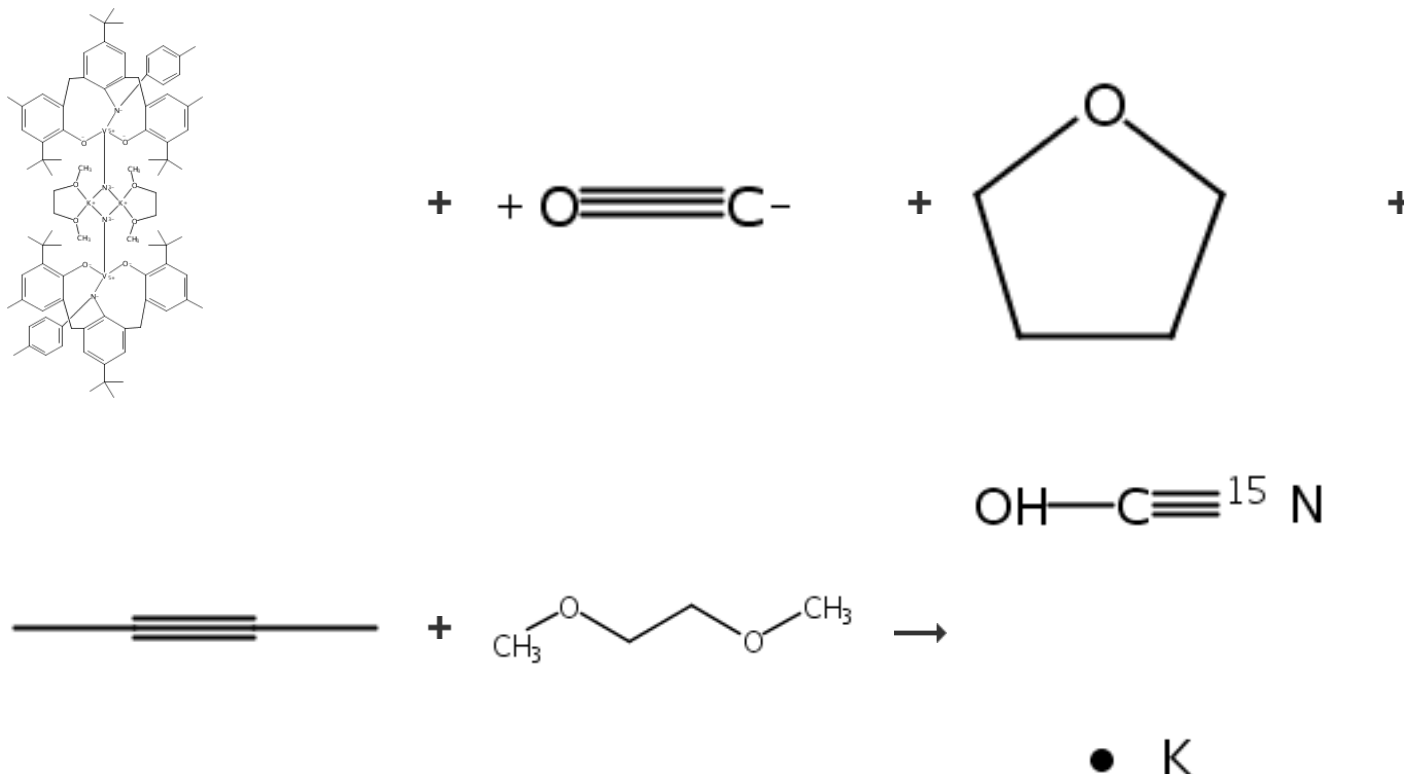
[View more...](#)

Available Experimental Data

^1H NMR, ^{13}C NMR, IR, ^{15}N NMR, Crystal Structure Data, Elemental Analysis, State, UV/Vis

[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

67. 7 Steps

[Step 2.1]

[Step 4.2]

[Overview](#)

Steps/Stages

Notes

- 1.1 2 d, 2.2 MPa
 2.1 S:PhMe, overnight, rt
 3.1
 4.1 R:KH

R:



- S:THF, 6 h, rt
 4.2 S:PhMe
 5.1 R:*p*-Benzoquinone, S:THF, overnight, rt
 6.1 2 d, 2.2 MPa
 7.1 R:MeC.tbd.CMe, S:THF, overnight, rt

1) high pressure, 2) preparative scale, 3) no experimental detail, 6) high pressure, Reactants: 5, Reagents: 4, Solvents: 2, Steps: 7, Stages: 8, Most stages in any one step: 2

References

[Nitrogen Atom Transfer from a Dinitrogen-Derived Vanadium Nitride Complex to Carbon Monoxide and Isocyanide](#)

By Ishida, Yutaka and Kawaguchi, Hiroyuki

From Journal of the American Chemical Society, 136(49), 16990-16993; 2014

Experimental Procedure

Step 1

Reaction of 3 with CO. A light yellow solution of **3** (20.2 mg, 13 μmol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. A brown powder of $[\{\text{K}(\text{THF})\}\{(\text{ONO})\text{V}(\text{NCO})\}]$ (**4**) in quantitative yield. Data for **4**. Anal. Calcd for $\text{C}_{46}\text{H}_{58}\text{KN}_2\text{O}_4\text{V}$: C, 69.67; H, 7.37; N, 3.53. Found: C 69.61; H, 7.51; N, 3.43%. UV-Vis (THF; $\lambda_{\text{max}}/\text{nm}$ ($\epsilon/\text{M}^{-1}\text{cm}^{-1}$)): 732 (650), 585 (430). IR (KBr; ν/cm^{-1}): 2168 (NCO). μ_{eff} (Evans method, THF, 297 K): 2.6 μ_{B} . μ_{eff} (solid state, 297 K): 2.45 μ_{B} .

Step 2

Reaction of 7 with 2-Butyne. To a solution of **7** (20.4 mg, 25.7 μmol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The brown residue was extracted in hexane and centrifuged to remove an insoluble material, which was identified as KNCO (76% yield). Evaporation of the supernatant to dryness afforded a dark brown powder of $[(\text{ONO})\text{V}(\mu^2\text{-MeCCMe})]$ (**8**) (14.2 mg, 79%).¹

Step 3

Procedure Unavailable

Step 4

Synthesis of $[\{\text{K}(\text{DME})\}_2\{(\text{ONO})\text{V}(\mu\text{-N})\}]_2$ (2**).** A suspension of KH (40.0 mg, 1.00 mmol) in THF (5 mL) was slowly added to a solution of $[(\text{ONO})\text{V}(\text{THF})]$ (**1**) (358 mg, 0.503 mmol) in THF (10 mL) under an atmosphere of dinitrogen, and then stirred for 6 h at room temperature, during which time the color of the solution gradually changed from forest green to dark brown. All volatiles were removed in vacuo to produce a dark brown residue. Extraction with toluene/DME, centrifugation, and removal of the solvent in vacuo afforded a brown solid, which was washed with DME. **Synthesis of $[\{\text{K}(\text{DME})\}_2\{(\text{ONO})\text{V}(\mu\text{-N})\}]_2$ (**2- ^{15}N**).** The same procedure as used for **2** was followed. The reaction of **1** (370 mg, 0.520 mmol) with KH (64.0 mg, 1.60 mmol) in THF (15 mL) under $^{15}\text{N}_2$ gave the ^{15}N isotopologue **2- ^{15}N** . Yield 234 mg, 49%.

Step 5

General/Typical Procedure: Synthesis of $[\{\text{K}(\text{DME})\}\{(\text{ONO})\text{VN}\}]_2$ (3**).** To a dark brown solution of **2** (171 mg, 93.7 μmol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μmol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. The synthesis of the ^{15}N isotopologue $[\{\text{K}(\text{DME})\}\{(\text{ONO})\text{V}^{15}\text{N}\}]_2$ (**3- ^{15}N**) was prepared following the same procedure above by using **2- ^{15}N** . Data for **3- ^{15}N** . IR (KBr; ν/cm^{-1}): 975 (V^{15}N). ^{15}N NMR (C_6D_6 , 131.6 MHz, δ/ppm): 958 ($\Delta\nu/2 = 43$ Hz).

Step 6

General/Typical Procedure: Reaction of 3 with CO. A light yellow solution of **3** (20.2 mg, 13 μmol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. The synthesis of $[\{\text{K}(\text{DME})\}\{(\text{ONO})\text{V}^{15}\text{NCO}\}]$ (**4- ^{15}N**) was prepared following the same procedure above by using **3- ^{15}N** . Data for **4- ^{15}N** . IR (KBr; ν/cm^{-1}): 2149 (^{15}NCO).

Step 7

General/Typical Procedure: Reaction of 7 with 2-Butyne. To a solution of **7** (20.4 mg, 25.7 μmol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The mixture of **7- ^{15}N** and 2-butyne was analogously treated as described above, giving K^{15}NCO . $\nu^{15}\text{NCO} = 2149\text{ cm}^{-1}$, ^{15}N NMR (D_2O , 131.6 MHz, δ/ppm): 68.6.

Reaction Protocol

Procedure

1. Stir a light yellow solution of the reactant (20.2 mg, 13 μmol) in THF (3 mL) under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turns brown.
2. Remove all volatiles in vacuo.

[View more...](#)

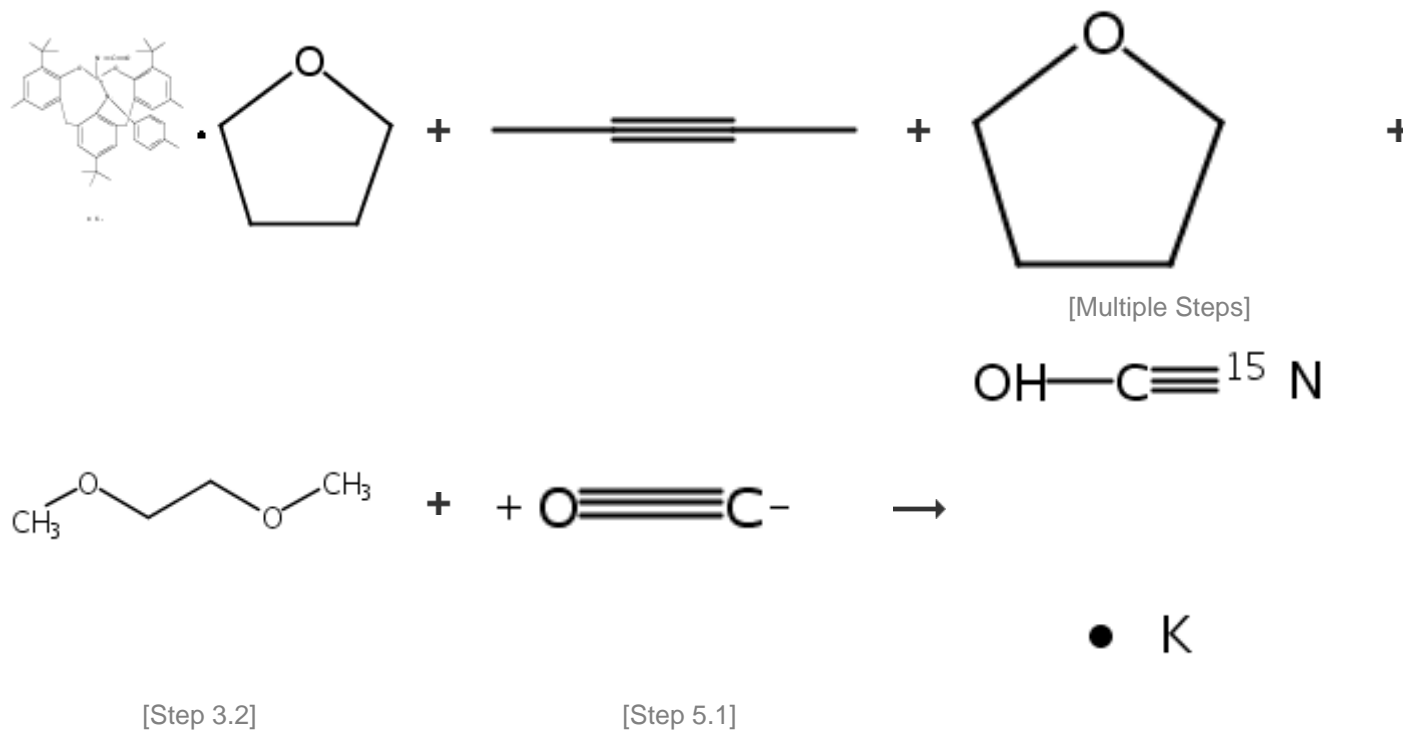
**Available
Experimental
Data**

IR, ^{15}N NMR, Elemental Analysis, State, UV/Vis

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

68. 6 Steps



[Overview](#)

Steps/Stages

Notes

1.1 S:PhMe, overnight, rt

2.1

3.1 R:KH

R:



S:THF, 6 h, rt

3.2 S:PhMe

4.1 R:*p*-Benzoquinone, S:THF, overnight, rt

5.1 2 d, 2.2 MPa

6.1 R:MeC.tbd.CMe, S:THF, overnight, rt

1) preparative scale, 2) no experimental detail, 5) high pressure, Reactants: 5, Reagents: 4, Solvents: 2, Steps: 6, Stages: 7, Most stages in any one step: 2

References

Nitrogen Atom Transfer from a Dinitrogen-Derived Vanadium Nitride Complex to Carbon Monoxide and Isocyanide

By Ishida, Yutaka and Kawaguchi, Hiroyuki

From Journal of the American Chemical Society, 136(49), 16990-16993; 2014

Experimental Procedure

Step 1

Reaction of 7 with 2-Butyne. To a solution of **7** (20.4 mg, 25.7 μmol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The brown residue was extracted in hexane and centrifuged to remove an insoluble material, which was identified as KNCO (76% yield). Evaporation of the supernatant to dryness afforded a dark brown powder of [(ONO)V(μ²-MeCCMe)] (**8**) (14.2 mg, 79%).¹

Step 2

Procedure Unavailable

Step 3

Synthesis of [(K(DME))₂[(ONO)V(μ-N)]]₂ (2**).** A suspension of KH (40.0 mg, 1.00 mmol) in THF (5 mL) was slowly added to a solution of [(ONO)V(THF)] (**1**) (358 mg, 0.503 mmol) in THF (10 mL) under an atmosphere of dinitrogen, and then stirred for 6 h at room temperature, during which time the color of the solution gradually changed from forest green to dark brown. All volatiles were removed in vacuo to produce a dark brown residue. Extraction_{μ_{eff}} with toluene/DME, centrifugation, and removal of the solvent in vacuo afforded a brown solid, which was washed with DME. **Synthesis of [(K(DME))₂[(ONO)V(μ-N)]]₂ (**2**-¹⁵N).** The same procedure as used for **2** was followed. The reaction of **1** (370 mg, 0.520 mmol) with KH (64.0 mg, 1.60 mmol) in THF (15 mL) under ¹⁵N₂ gave the ¹⁵N isotopologue **2**-¹⁵N. Yield 234 mg, 49%.

Step 4

General/Typical Procedure: **Synthesis of [(K(DME))[(ONO)VN]]₂ (**3**).** To a dark brown solution of **2** (171 mg, 93.7 μmol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μmol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. The synthesis of the ¹⁵N isotopologue [(K(DME))[(ONO)V¹⁵N]]₂ (**3**-¹⁵N) was prepared following the same procedure above by using **2**-¹⁵N. IR (KBr; ν/cm⁻¹): 975 (V¹⁵N). ¹⁵N NMR (C₆D₆, 131.6 MHz, δ/ppm): 958 (Δν_{1/2} = 43 Hz).

Step 5

General/Typical Procedure: **Reaction of 3 with CO.** A light yellow solution of **3** (20.2 mg, 13 μmol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. The synthesis of [(K(DME))[(ONO)V(¹⁵NCO)]] (**4**-¹⁵N) was prepared following the same procedure above by using **3**-¹⁵N. Data for **4**-¹⁵N. IR (KBr; ν/cm⁻¹): 2149 (¹⁵NCO).

Step 6

General/Typical Procedure: **Reaction of 7 with 2-Butyne.** To a solution of **7** (20.4 mg, 25.7 μmol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The mixture of **7**-¹⁵N and 2-butyne was analogously treated as described above, giving K¹⁵NCO. ν ¹⁵NCO = 2149 cm⁻¹, ¹⁵N NMR (D₂O, 131.6 MHz, δ/ppm): 68.6.

Reaction Protocol

Procedure

1. Add 2-butyne (0.1 mL) to a solution of the reactant (20.4 mg, 25.7 μmol) in toluene (5 mL) .
2. Stir the reaction mixture overnight at room temperature to produce a dark brown suspension.

[View more...](#)

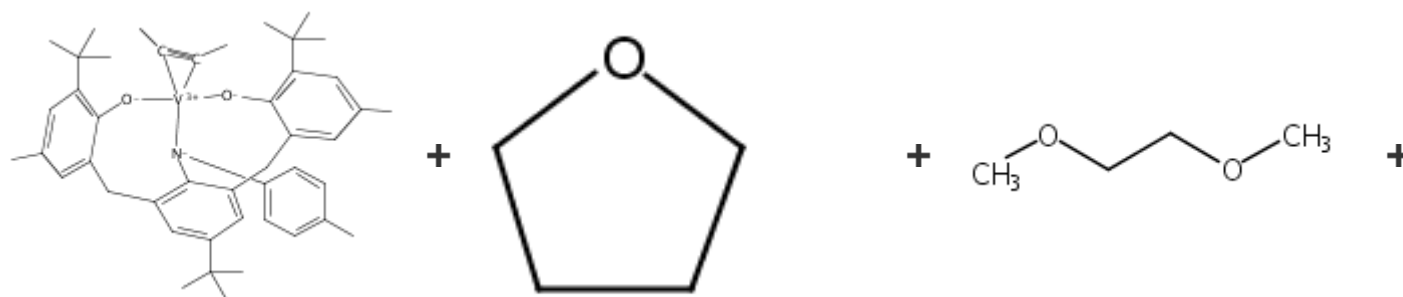
Available
Experimental
Data

IR, ¹⁵N NMR, State

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

69. 5 Steps



[Step 2.2]



[Step 4.1]

Overview

Steps/Stages

- 1.1
- 2.1 R:KH
R:
 $^{15}\text{N}\equiv\text{C}\equiv\text{C}^{15}\text{N}$
- S:THF, 6 h, rt
- 2.2 S:PhMe
- 3.1 R:*p*-Benzoquinone, S:THF, overnight, rt
- 4.1 2 d, 2.2 MPa
- 5.1 R:MeC.tbd.CMe, S:THF, overnight, rt

Experimental Procedure

Step 1

Procedure Unavailable

Notes

1) no experimental detail, 4) high pressure, Reactants: 4, Reagents: 4, Solvents: 2, Steps: 5, Stages: 6, Most stages in any one step: 2

References

[Nitrogen Atom Transfer from a Dinitrogen-Derived Vanadium Nitride Complex to Carbon Monoxide and Isocyanide](#)

By Ishida, Yutaka and Kawaguchi, Hiroyuki
From Journal of the American Chemical Society, 136(49), 16990-16993; 2014

Step 2

Synthesis of $[\{K(DME)\}_2\{(ONO)V(\mu-N)\}]_2$ (2**).** A suspension of KH (40.0 mg, 1.00 mmol) in THF (5 mL) was slowly added to a solution of $[(ONO)V(THF)]$ (**1**) (358 mg, 0.503 mmol) in THF (10 mL) under an atmosphere of dinitrogen, and then stirred for 6 h at room temperature, during which time the color of the solution gradually changed from forest green to dark brown. All volatiles were removed in vacuo to produce a dark brown residue. Extraction μ_{eff} with toluene/DME, centrifugation, and removal of the solvent in vacuo afforded a brown solid, which was washed with DME. **Synthesis of $[\{K(DME)\}_2\{(ONO)V(\mu-N)\}]_2$ (**2- ^{15}N**).** The same procedure as used for **2** was followed. The reaction of **1** (370 mg, 0.520 mmol) with KH (64.0 mg, 1.60 mmol) in THF (15 mL) under $^{15}N_2$ gave the ^{15}N isotopologue **2- ^{15}N** . Yield 234 mg, 49%.

Step 3

General/Typical Procedure: **Synthesis of $[\{K(DME)\}(ONO)VN\}]_2$ (**3**).** To a dark brown solution of **2** (171 mg, 93.7 μ mol) in THF (20 mL) at room temperature was added a THF (6 mL) solution of *p*-benzoquinone (10.1 mg, 93.4 μ mol). The reaction was allowed to proceed overnight, producing an orange suspension. After all volatiles were removed in vacuo, the residue was extracted in toluene/DME and centrifuged to remove an insoluble colorless material. Evaporation of the supernatant to dryness resulted in an orange powder, which was washed with pentane. The synthesis of the ^{15}N isotopologue $[\{K(DME)\}(ONO)V(^{15}N)\}]_2$ (**3- ^{15}N**) was prepared following the same procedure above by using **2- ^{15}N** . Data for **3- ^{15}N** . IR (KBr; ν/cm^{-1}): 975 ($\nu^{15}N$). ^{15}N NMR (C_6D_6 , 131.6 MHz, δ/ppm): 958 ($\Delta\nu_{1/2} = 43$ Hz).

Step 4

General/Typical Procedure: **Reaction of **3** with CO.** A light yellow solution of **3** (20.2 mg, 13 μ mol) in THF (3 mL) was stirred under an atmosphere of CO (2.2 MPa) for 2 days, during which the reaction mixture turned brown. All volatiles were removed in vacuo. The synthesis of $[\{K(DME)\}(ONO)V(^{15}NCO)\}]$ (**4- ^{15}N**) was prepared following the same procedure above by using **3- ^{15}N** . Data for **4- ^{15}N** . IR (KBr; ν/cm^{-1}): 2149 (^{15}NCO).

Step 5

General/Typical Procedure: **Reaction of **7** with 2-Butyne.** To a solution of **7** (20.4 mg, 25.7 μ mol) in toluene (5 mL) was added 2-butyne (0.1 mL). The reaction mixture was stirred overnight at room temperature to produce a dark brown suspension, and all volatiles were removed in vacuo. The mixture of **7- ^{15}N** and 2-butyne was analogously treated as described above, giving $K^{15}NCO$. $\nu^{15}NCO = 2149\text{ cm}^{-1}$, ^{15}N NMR (D_2O , 131.6 MHz, δ/ppm): 68.6.

Reaction Protocol**Procedure**

1. Add a suspension of KH (64.0 mg, 1.60 mmol) in THF (15 mL) to a solution of [(ONO) V (THF)] (370 mg, 0.520 mmol) in THF (10 mL) under an atmosphere of dinitrogen.
2. Stir the mixture for 6 h at room temperature.

[View more...](#)**Available Experimental Data**IR, ^{15}N NMR, State[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

70. Single Step

• 2 HCl

55%

[Overview](#)**Steps/Stages****Notes**

1.1 R: BH_3

R:



S: THF, 24 h, reflux; rt

1.2 R: HCl, S: EtOH, 10 min, rt

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

References

[Design and Synthesis of Visible Isotope-Coded Affinity Tags for the Absolute Quantification of Specific Proteins in Complex Mixtures](#)

By Bottari, Patricia et al

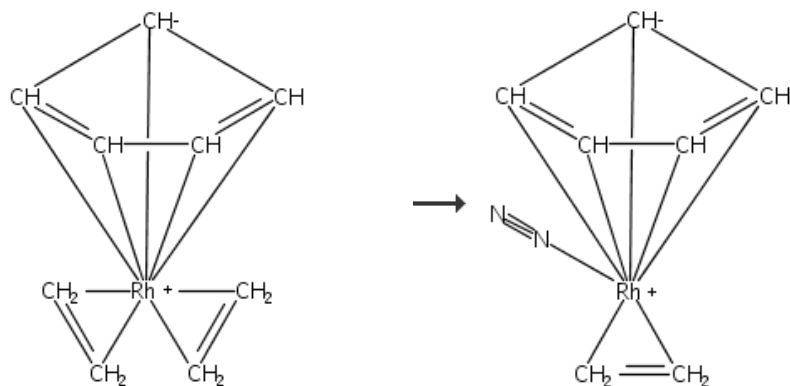
From Bioconjugate Chemistry, 15(2), 380-388; 2004

Experimental Procedure

$^{14}\text{N}_2$, $^{13}\text{C}_4$]-1,4-Butanediamine dihydrochloride (12). Compound 11 (137.1 mg, 1.59 mmol) and a 1 M solution of borane in dry THF (16 mL, 15.9 mmol) were refluxed for 24 h. After the reaction was cooled to room temperature, anhydrous ethanol (18 mL) was added. The reaction was stirred for 24 h, and HCl gas was bubbled through the solution for 10 min, or until saturation. The di-chloride salt was filtered, washed with anhydrous ethanol, and dried under vacuum to afford 146.5 mg (55.2%) of a white solid. ^1H NMR (300 MHz, D_2O) δ 3.5 (m, 2); 2.75 (m, 2H); 2.15 (m, 2); 1.75 (m, 2H).

CASREACT®: Copyright © 2020 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.

71. Single Step



Overview

Steps/Stages

1.1 R:



Notes

photochem., supercritical Xenon medium, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

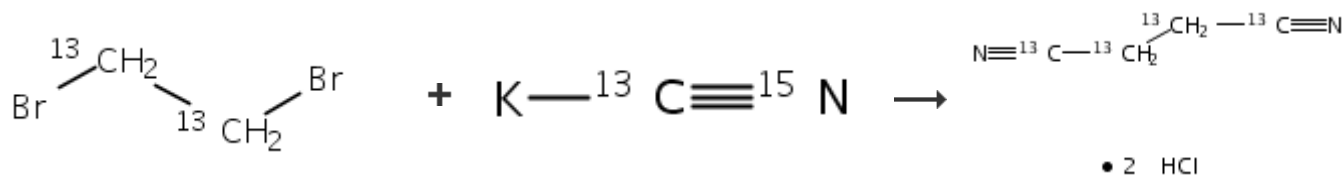
[Photochemical generation and reactions of rhodium \(\$\eta^5\$ -cyclopentadienyl\) \(ethene\) \(dinitrogen\) in liquid xenon](#)

By Haddleton, David M. et al

From Journal of Organometallic Chemistry, 311(1-2), C15-C20; 1986

CASREACT®: Copyright © 2020 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.

72. 2 Steps



Overview

Steps/Stages

1.1 S:H₂O, S:EtOH, 5 h, reflux; rt

2.1 R: BH₃

R:



S:THF, 24 h, reflux; rt

2.2 R:HCl, S:EtOH, 10 min, rt

Notes

2) nitrogen from air, Reactants: 2, Reagents: 3, Solvents: 3, Steps: 2, Stages: 3, Most stages in any one step: 2

References

[Design and Synthesis of Visible Isotope-Coded Affinity Tags for the Absolute Quantification of Specific Proteins in Complex Mixtures](#)

By Bottari, Patricia et al

From Bioconjugate Chemistry, 15(2), 380-388; 2004

Experimental Procedure

Step 1

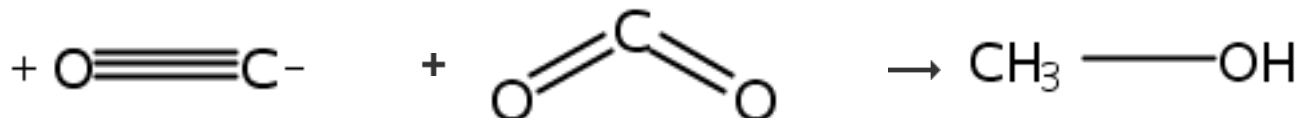
[¹⁵N₂, ¹³C₄]butanedinitrile (11). Potassium [¹³C, ¹⁵N] cyanide (176.3 mg, 2.63 mmol) and [1,2-¹³C₂]-dibromoethane (0.5 g, 2.63 mmol) were refluxed for 5 h in 70% ethanol (2 mL). The reaction mixture was cooled to room temperature and passed through a silica plug with benzene. The solvent was removed under reduced pressure to afford the product in 80% yield (90 mg). ¹H NMR (300 MHz, CDCl₃) δ 3.0 (m, 2); 2.55 (m, 2H).

Step 2

[¹⁴N₂, ¹³C₄]-1,4-Butanediamine dihydrochloride (12). Compound 11 (137.1 mg, 1.59 mmol) and a 1 M solution of borane in dry THF (16 mL, 15.9 mmol) were refluxed for 24 h. After the reaction was cooled to room temperature, anhydrous ethanol (18 mL) was added. The reaction was stirred for 24 h, and HCl gas was bubbled through the solution for 10 min, or until saturation. The di-chloride salt was filtered, washed with anhydrous ethanol, and dried under vacuum to afford 146.5 mg (55.2%) of a white solid. ¹H NMR (300 MHz, D₂O) δ 3.5 (m, 2); 2.75 (m, 2H); 2.15 (m, 2); 1.75 (m, 2H).

CASREACT®: Copyright © 2020 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.

73. Single Step



Overview

Steps/Stages

Notes

1.1 R:H₂

R:

C:CuO, C:ZnO, C:Al₂O₃, 250°C

catalyst prepared and used, alternative reaction conditions shown, Reactants: 2, Reagents: 2, Catalysts: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Method for preparation of heat-resistant copper/zinc-based catalyst for manufacture of methanol](#)

By Xu, Xuejun et al

From Faming Zhuanli Shenqing, 103372445, 30 Oct 2013

CASREACT®: Copyright © 2020 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.

74. Single Step[Overview](#)**Steps/Stages**1.1 R:H₂, R:CO₂

R:

C:CuO, C:ZnO, C:Al₂O₃, 250°C**Notes**

catalyst prepared and used, Reactants: 1, Reagents: 3, Catalysts: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Method for preparation of heat-resistant copper-containing catalyst for manufacture of methanol](#)

By Wang, Haitao et al

From Faming Zhuanli Shenqing, 103372438, 30 Oct 2013

CASREACT®: Copyright © 2020 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.

75. Single Step[Overview](#)**Steps/Stages****Notes**

1.1 R:



C:Disodium carbonate, 1 h, 773K; 773K → 673K

1.2 673K

other products also detected, Na₂CO₃/NaY catalyst prepared and used, Reactants: 1, Reagents: 1, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

[Formation and function of formate in the side-chain alkylation of toluene with methanol](#)

By Lin, Dan et al

From Cuihua Xuebao, 33(6), 1041-1047; 2012

CASREACT®: Copyright © 2020 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.

76. Single Step[Overview](#)**Steps/Stages**

1.1 R:



C:Disodium carbonate, 1 h, 773K; 773K → 673K

1.2 673K

Notes

other products also detected, Na₂CO₃/KX catalyst prepared and used, Reactants: 1, Reagents: 1, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

[Formation and function of formate in the side-chain alkylation of toluene with methanol](#)

By Lin, Dan et al

From Cuihua Xuebao, 33(6), 1041-1047; 2012

CASREACT®: Copyright © 2020 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.

77. Single Step[Overview](#)**Steps/Stages****Notes**

1.1 R:



other products also detected, K₂CO₃/NaY catalyst prepared and used, Reactants: 1, Reagents: 1, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

1.2 C:K₂CO₃, 1 h, 773K; 773K → 673K
673K

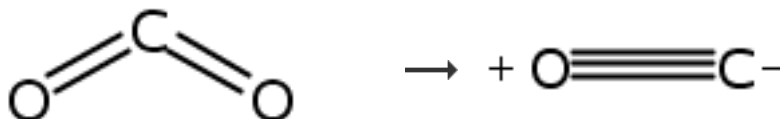
References

[Formation and function of formate in the side-chain alkylation of toluene with methanol](#)

By Lin, Dan et al

From Cuihua Xuebao, 33(6), 1041-1047; 2012

CASREACT®: Copyright © 2020 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.

78. Single Step[Overview](#)**Steps/Stages**1.1 R:H₂

R:



C:Pt, C:ZnO, 400°C

Notes

solid-supported catalyst, optimization study, optimized on catalyst, gas hourly space velocity, stoichiometry and reaction temperature, gas hourly space velocity of 28,145 per hour, fixed-bed quartz tube reactor used, 22% conversion, palladium-supported zinc oxide prepared and used as catalyst, Reactants: 1, Reagents: 2, Catalysts: 2, Steps: 1, Stages: 1, Most stages in any one step: 1

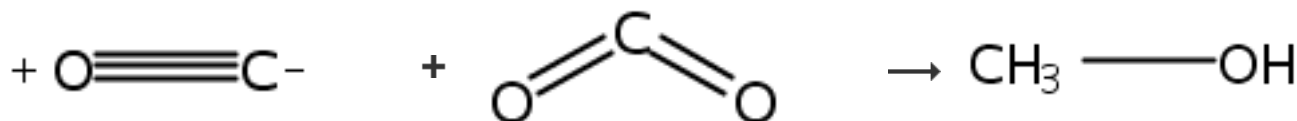
References

[The effect of PdZn particle size on reverse-water-gas-shift reaction](#)

By Lebarbier, V. et al

From Applied Catalysis, A: General, 379(1-2), 3-6; 2010

CASREACT®: Copyright © 2020 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.

79. Single Step[Overview](#)**Steps/Stages****Notes**

1.1 R:Disodium carbonate, C:Cu(NO₃)₂, C:Zn(NO₃)₂, C:13473-90-0,
S:H₂O, rt; rt, pH 7-8

1.2 R:H₂
R:

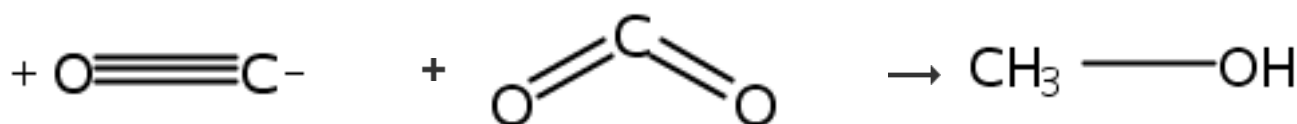


rt → 150°C; 180°C; 210°C; 210°C → 240°C; cooled

1.3 220-280°C, 3 MPa

CASREACT®: Copyright © 2020 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.

80. Single Step



aging for 2h in stage 1, reduction process lasted 16h in stage 2, high pressure in stage 3, Reactants: 2, Reagents: 3, Catalysts: 3, Solvents: 1, Steps: 1, Stages: 3, Most stages in any one step: 3

References

[Catalyst for synthesis for methanol and its preparation](#)

By Chang, Jie et al

From Faming Zhuanli Shenqing Gongkai Shuomingshu, 1810357, 02 Aug 2006

Overview

Steps/Stages

1.1 R:Disodium carbonate, C:Cu(NO₃)₂, C:Zn(NO₃)₂, C:13473-90-0,
C:LiNO₃, S:H₂O, rt; rt, pH 7-8

1.2 R:H₂
R:



rt → 150°C; 180°C; 210°C; 210°C → 240°C; cooled

1.3 220-280°C, 3 MPa

CASREACT®: Copyright © 2020 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.

81. Single Step



Notes

aging for 2h in stage 1, reduction process lasted 16h in stage 2, high pressure in stage 3, Reactants: 2, Reagents: 3, Catalysts: 4, Solvents: 1, Steps: 1, Stages: 3, Most stages in any one step: 3

References

[Catalyst for synthesis for methanol and its preparation](#)

By Chang, Jie et al

From Faming Zhuanli Shenqing Gongkai Shuomingshu, 1810357, 02 Aug 2006

Overview

Steps/Stages

Notes

1.1 R:



S:EtOH, 4 h, reflux

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

References

[Coenzyme B Induced Coordination of Coenzyme M via Its Thiol Group to Ni\(I\) of F430 in Active Methyl-Coenzyme M Reductase](#)

By Finazzo, Cinzia et al

From Journal of the American Chemical Society, 125(17), 4988-4989; 2003

CASREACT®: Copyright © 2020 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.

82. Single Step



Overview

Steps/Stages

Notes

1.1 R:H₂

R:

C:Cu(NO₃)₂, C:Zn(NO₃)₂, C:13473-90-0, 240°C, 5 MPa

other catalysts gave similar results, Reactants: 1, Reagents: 2, Catalysts: 3, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Low pressure methanol synthesis catalyst](#)

By Fang, De-ren et al

From Shiyong Huagong, 32(1), 5-8; 2003

CASREACT®: Copyright © 2020 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.

83. Single Step



10%

Overview

Steps/Stages

Notes

1.1 R:

C:K₂CO₃, 1 h, 773K; 773K → 673K

1.2 673K

other products also detected, K₂CO₃/KX catalyst prepared and used, Reactants: 1, Reagents: 1, Catalysts: 1, Steps: 1, Stages: 2, Most stages in any one step: 2

References

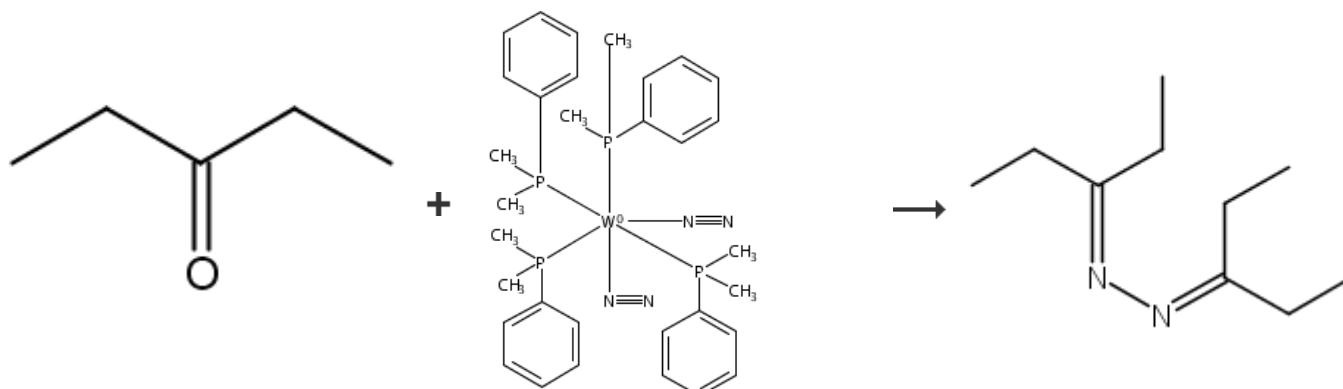
[Formation and function of formate in the side-chain alkylation of toluene with methanol](#)

By Lin, Dan et al

From Cuihua Xuebao, 33(6), 1041-1047; 2012

CASREACT®: Copyright © 2020 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.

84. Single Step



Overview

Steps/Stages

1.1 R:MeOH

R:



S:MeOH, S:Me₂CO

Notes

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

References

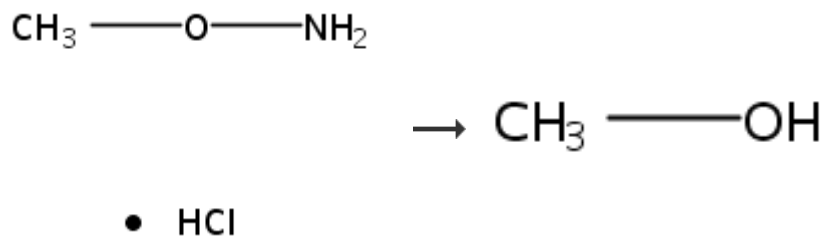
[Preparation and properties of molybdenum and tungsten dinitrogen complexes. 22. Synthesis of ketazines from dinitrogen coordinated to tungsten](#)

By Hidai, Masanobu et al

From Bulletin of the Chemical Society of Japan, 58(9), 2719-20; 1985

CASREACT®: Copyright © 2020 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.

85. Single Step



Overview

Steps/Stages

Notes

1.1 R:N₂O, R:H₂O

R:



R:N=O

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

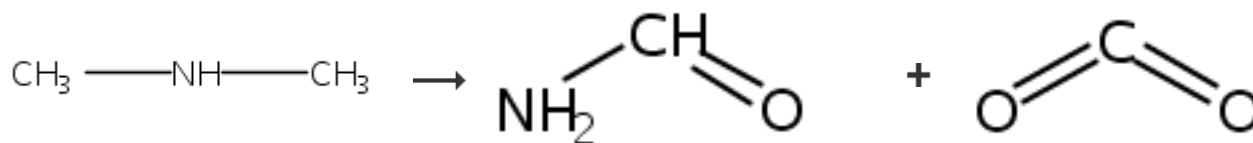
References

[Reduction of nitric oxide by hydroxylamine. 2. Reactions of the alkylated hydroxylamines](#)

By Wang, Nun Yii and Bonner, Francis T.

From Inorganic Chemistry, 25(11), 1863-6; 1986

CASREACT®: Copyright © 2020 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.

86. Single Step**Overview****Steps/Stages**

1.1 R:

R:O₂, C:TiO₂, 1.1 s, 413K**Notes**

gas phase, photochemical, thermal, green chemistry, mercury UV lamp (365 nm, 15 W) used, continuous flow tubular reactor used, UV-irradiated TiO₂ used as catalyst, reaction unsuccessful in absence of catalyst, kinetics studied, optimization study, optimized on reactant concentration, reaction temperature and time, 45% conversion, mechanism studied, catalyst recycled, other products also detected, Reactants: 1, Reagents: 2, Catalysts: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

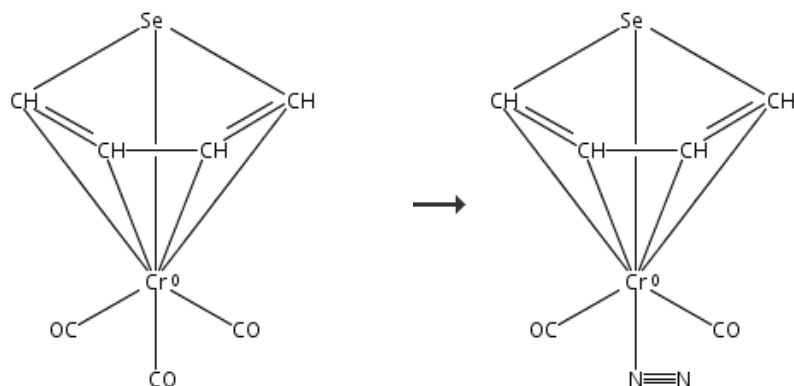
[Gas-phase photocatalytic oxidation of dimethylamine: the reaction pathway and kinetics](#)

By Kachina, Anna et al

From International Journal of Photoenergy, (2), 79847/1-79847/4; 2007

CASREACT®: Copyright © 2020 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.

87. Single Step



Overview

Steps/Stages

1.1 R:



20K

Notes

photochem., 365nm, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following Photolysis of \$\(\eta^5\text{-C}_4\text{H}_4\text{Se}\)\text{Cr}\(\text{CO}\)_3\$. A Picosecond and Nanosecond Time-Resolved Infrared, Matrix Isolation, and DFT Investigation](#)

By Brennan, Peter et al

From *Organometallics*, 27(15), 3671-3680; 2008

Experimental Procedure

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS_{202} closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. $(\eta^5\text{-C}_4\text{H}_4\text{Se})\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Reaction Protocol

Procedure

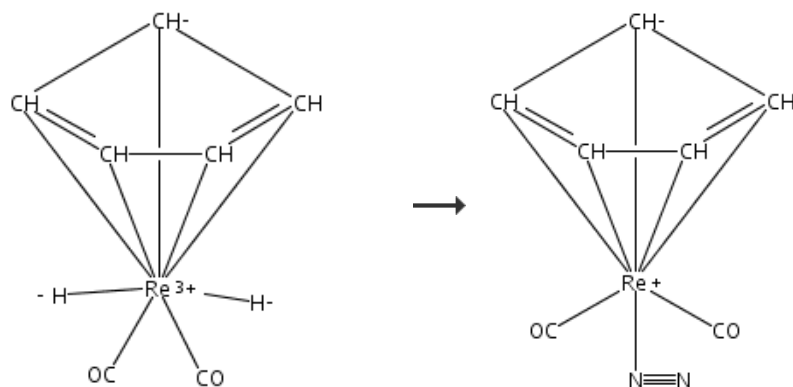
1. Isolate the matrix apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump and temperature control unit.
2. Deposit the matrixes onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud.

[View more...](#)

[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

88. Single Step



Overview

Steps/Stages

1.1 R:



Notes

supercrit. Xenon medium, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

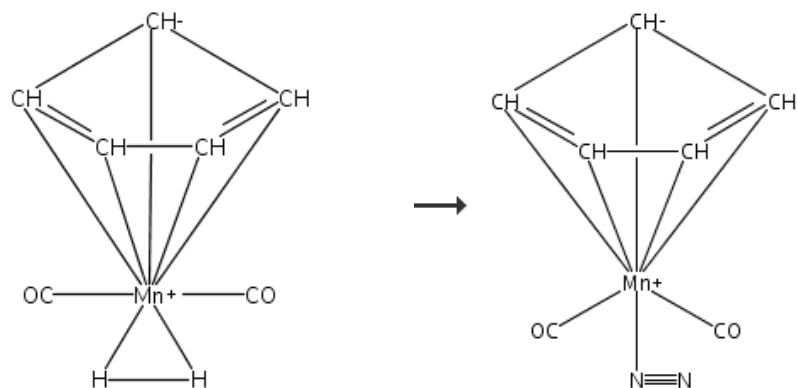
References

[Organometallic photochemistry in supercritical fluids: the reaction of hydrogen with \[\(η⁵-C₅H₅\)M\(CO\)₃\] \(M = rhenium and manganese\) and the formation of a non-classical dihydrogen complex of manganese\(I\)](#)

By Howdle, Steven M. and Poliakoff, Martyn
From Journal of the Chemical Society, Chemical Communications, (16), 1099-101; 1989

CASREACT®: Copyright © 2020 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.

89. Single Step



Overview

Steps/Stages

1.1 R:



Notes

supercrit. Xenon medium, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

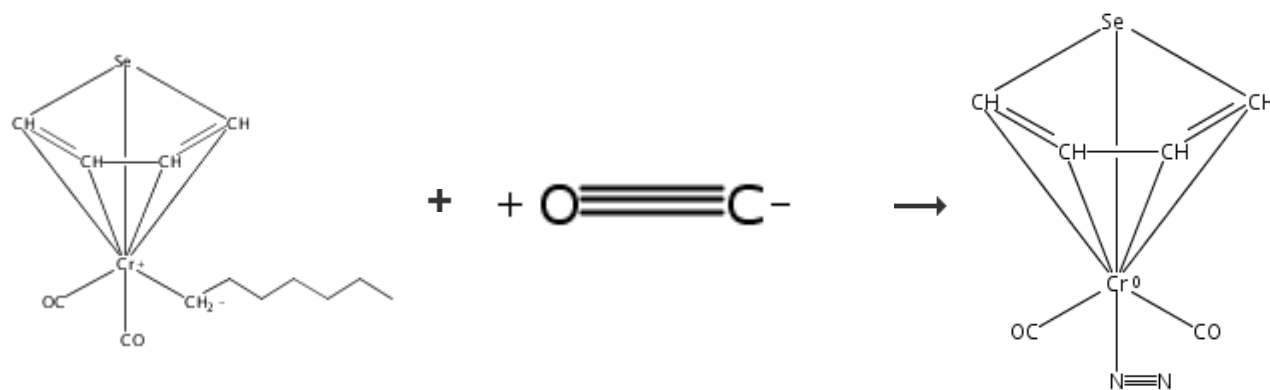
References

Organometallic photochemistry in supercritical fluids: the reaction of hydrogen with $[(\eta^5\text{-C}_5\text{H}_5)\text{M}(\text{CO})_3]$ (M = rhenium and manganese) and the formation of a non-classical dihydrogen complex of manganese(I)

By Howdle, Steven M. and Poliakoff, Martyn
From Journal of the Chemical Society, Chemical Communications, (16), 1099-101; 1989

CASREACT®: Copyright © 2020 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.

90. 2 Steps



Overview

Steps/Stages

Notes

1.1 298K

2.1 R:



20K

1) photochem., 365nm, 2) photochem., 365nm, Reactants: 2, Reagents: 1, Steps: 2, Stages: 2, Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following Photolysis of \(\$\eta^5\$ -C₄H₄Se\)Cr\(CO\)₃. A Picosecond and Nanosecond Time-Resolved Infrared, Matrix Isolation, and DFT Investigation](#)

By Brennan, Peter et al

From *Organometallics*, 27(15), 3671-3680; 2008

Experimental Procedure

Step 1

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF₂ window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF₂ window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η^5 -C₄H₄Se)Cr(CO)₃ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm⁻¹ resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Step 2

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF₂ window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF₂ window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η^5 -C₄H₄Se)Cr(CO)₃ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm⁻¹ resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Reaction Protocol

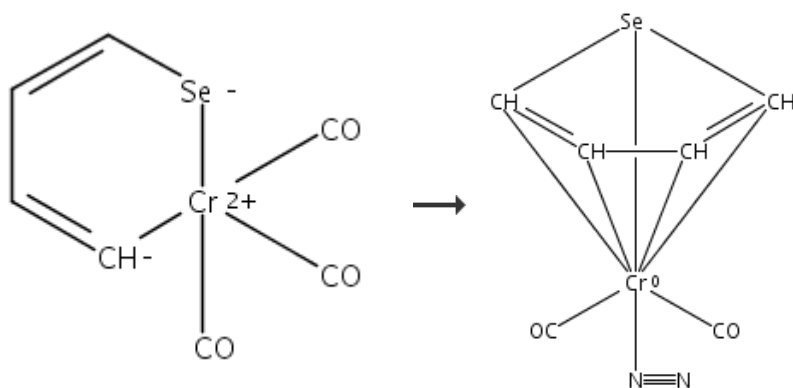
Procedure

1. Isolate the matrix apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump and temperature control unit.
2. Deposit the matrixes onto a CaF₂ window cooled to 20 K, with matching outer windows on the vacuum shroud.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

91. 2 Steps



Overview

Steps/Stages

1.1 20K

2.1 R:



20K

Notes

1) photochem., 500nm and higher, 2) photochem., 365nm, Reactants: 1, Reagents: 1, Steps: 2, Stages: 2, Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following Photolysis of \$\(\eta^5\text{-C}_4\text{H}_4\text{Se}\)\text{Cr}\(\text{CO}\)_3\$. A Picosecond and Nanosecond Time-Resolved Infrared, Matrix Isolation, and DFT Investigation](#)

By Brennan, Peter et al

From *Organometallics*, 27(15), 3671-3680; 2008

Experimental Procedure

Step 1

Matrix Isolation Apparatus. The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η 5- $\text{C}_4\text{H}_4\text{Se}$) $\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Step 2

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η 5- $\text{C}_4\text{H}_4\text{Se}$) $\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

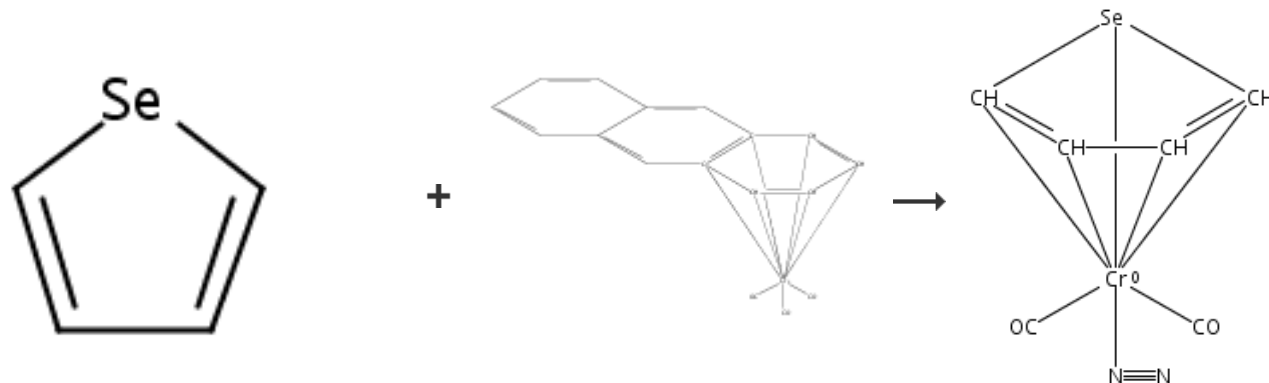
[Reaction Protocol](#)**Procedure**

1. Isolate the matrix apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump and temperature control unit.
2. Deposit the matrixes onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

92. 2 Steps



Overview

Steps/Stages

1.1 S:THF, 75 min, reflux

2.1 R:



20K

Notes

2) photochem., 365nm, Reactants: 2,
Reagents: 1, Solvents: 1, Steps: 2, Stages: 2,
Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following
Photolysis of \$\(\eta^5\text{-C}_4\text{H}_4\text{Se}\)\text{Cr}\(\text{CO}\)_3\$. A
Picosecond and Nanosecond Time-Resolved
Infrared, Matrix Isolation, and DFT
Investigation](#)

By Brennan, Peter et al

From *Organometallics*, 27(15), 3671-3680;
2008

Experimental Procedure

Step 1

Preparation of $(\eta^5\text{-C}_4\text{H}_4\text{Se})\text{Cr}(\text{CO})_3$. $(\eta^5\text{-C}_4\text{H}_4\text{Se})\text{Cr}(\text{CO})_3$ was prepared by thermal arene exchange of anthracene by selenophene from $(\eta^6\text{-C}_{14}\text{H}_{10})\text{Cr}(\text{CO})_3$ using a modification of the published method.²⁸ A solution of $(\eta^6\text{-C}_{14}\text{H}_{10})\text{Cr}(\text{CO})_3$ (400 mg, 1.3 mmol) and $\text{C}_4\text{H}_4\text{Se}$ (3 g, 23 mmol) in 2.5 mL of freshly distilled tetrahydrofuran (THF) was heated to reflux temperature under an argon atmosphere for 75 min. The solvent was then removed under reduced pressure at room temperature, yielding an orange solid. The crude material was chromatographed on neutral silica eluting with 3:1 petroleum ether/dichloromethane. Following elution of the first band of unreacted $(\eta^6\text{-C}_{14}\text{H}_{10})\text{Cr}(\text{CO})_3$ the mobile phase was changed to pure dichloromethane. The product eluted as the second band. Removal of the solvent under reduced pressure yielded a dark red powder. The crude product was recrystallized from toluene/n-pentane, yielding 197 mg of product, representing a 57% yield based on $(\eta^6\text{-C}_{14}\text{H}_{10})\text{Cr}(\text{CO})_3$. Spectroscopic data were in accordance with those previously reported.²⁸ $(\eta^5\text{-C}_4\text{H}_4\text{Se})\text{Cr}(\text{CO})_3$, yield 179 mg 57%

Step 2

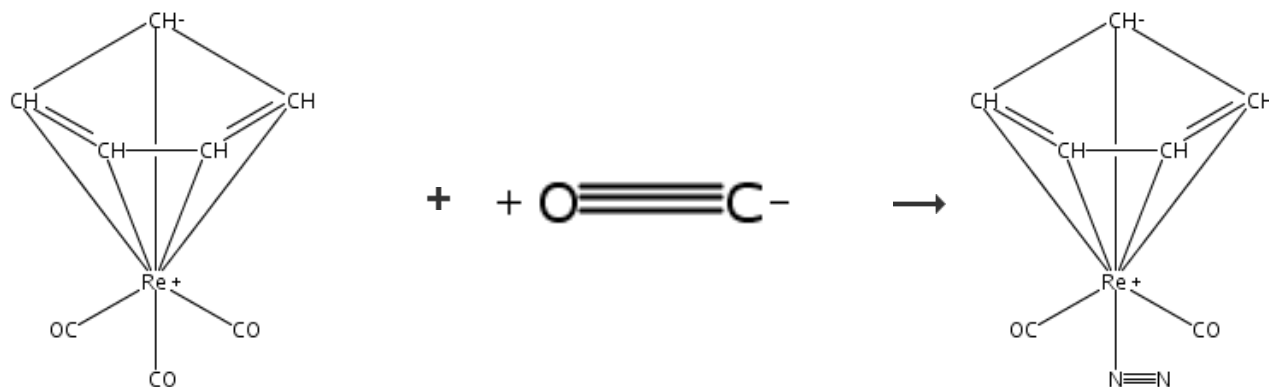
General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS_{202} closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η 5- $\text{C}_4\text{H}_4\text{Se}$) $\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or $> 500 \text{ nm}$) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Reaction Protocol**Procedure**

1. Prepare a compound by thermal arene exchange of anthracene by selenophene from (η $\text{C}_{14}\text{H}_{10}$) $\text{Cr}(\text{CO})_3$.
2. Heat a solution of ((η $\text{C}_{14}\text{H}_{10}$) $\text{Cr}(\text{CO})_3$ (1.3 mmol) and $\text{C}_4\text{H}_4\text{Se}$ (23 mmol) in 2.5 ml of freshly distilled tetrahydrofuran (THF) to reflux temperature under an argon atmosphere for 75 minutes.

[View more...](#)[View with MethodsNow](#)

CASREACT®: Copyright © 2020 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.

93. 2 Steps

[Step 2.1]

[Overview](#)[Steps/Stages](#)[Notes](#)

1.1 R:



2.1

1) photochem., supercrit. xenon medium,
Reactants: 2, Reagents: 1, Steps: 2, Stages:
2, Most stages in any one step: 1

References

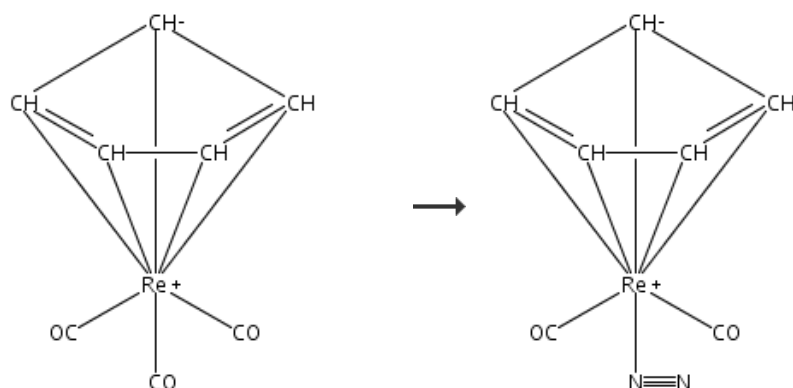
[The synthesis and spectroscopic identification of \$\[\(\eta^5\text{-C}_5\text{H}_5\)\text{Re}\(\text{N}_2\)_3\]\$ and \$\[\(\eta^5\text{-C}_5\text{H}_5\)\text{Re}\(\text{CO}\)\(\text{N}_2\)_2\]\$ in supercritical xenon at room temperature and in dinitrogen matrixes at 20 K](#)

By Howdle, Steven M. et al

From Journal of the Chemical Society, Chemical Communications, (20), 1517-19; 1989

CASREACT®: Copyright © 2020 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.

94. 2 Steps



Overview

Steps/Stages

1.1 R:H₂

2.1 R:



Notes

1) Photochem., supercrit. Xenon medium, 2) supercrit. Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

References

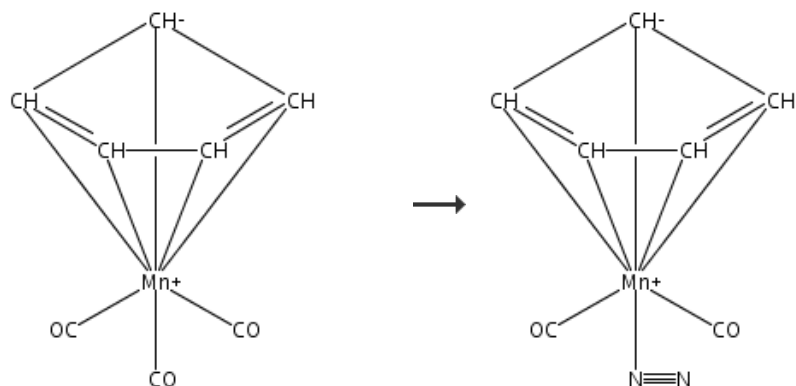
[Organometallic photochemistry in supercritical fluids: the reaction of hydrogen with \$\[\(\eta^5\text{-C}_5\text{H}_5\)\text{M}\(\text{CO}\)_3\]\$ \(M = rhenium and manganese\) and the formation of a non-classical dihydrogen complex of manganese\(I\)](#)

By Howdle, Steven M. and Poliakoff, Martyn

From Journal of the Chemical Society, Chemical Communications, (16), 1099-101; 1989

CASREACT®: Copyright © 2020 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.

95. 2 Steps



Overview

Steps/Stages

1.1 R:H₂

2.1 R:



Notes

1) Photochem., supercrit. Xenon medium, 2) supercrit. Xenon medium, Reactants: 1, Reagents: 2, Steps: 2, Stages: 2, Most stages in any one step: 1

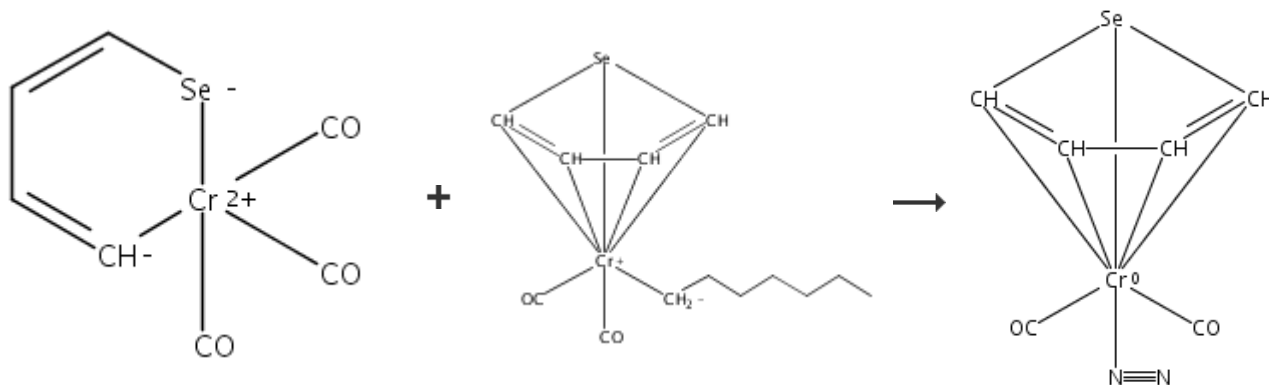
References

Organometallic photochemistry in supercritical fluids: the reaction of hydrogen with $[(\eta^5\text{-C}_5\text{H}_5)\text{M}(\text{CO})_3]$ (M = rhenium and manganese) and the formation of a non-classical dihydrogen complex of manganese(I)

By Howdle, Steven M. and Poliakoff, Martyn
From Journal of the Chemical Society, Chemical Communications, (16), 1099-101; 1989

CASREACT®: Copyright © 2020 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.

96. 3 Steps



[Step 2.1]

Overview

Steps/Stages

Notes

1.1 20K
2.1 298K

3.1 R:



20K

1) photochem., 365nm, 2) photochem., 365nm, 3) photochem., 365nm, Reactants: 2, Reagents: 1, Steps: 3, Stages: 3, Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following Photolysis of \(\$\eta^5\$ -C₄H₄Se\)Cr\(CO\)₃. A Picosecond and Nanosecond Time-Resolved Infrared, Matrix Isolation, and DFT Investigation](#)

By Brennan, Peter et al

From Organometallics, 27(15), 3671-3680; 2008

Experimental Procedure

Step 1

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF₂ window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF₂ window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10⁻⁷ mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η^5 -C₄H₄Se)Cr(CO)₃ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm⁻¹ resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Step 2

General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF₂ window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF₂ window cooled by an X CS₂₀₂ closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10⁻⁷ mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η^5 -C₄H₄Se)Cr(CO)₃ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm⁻¹ resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or >500 nm) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Step 3

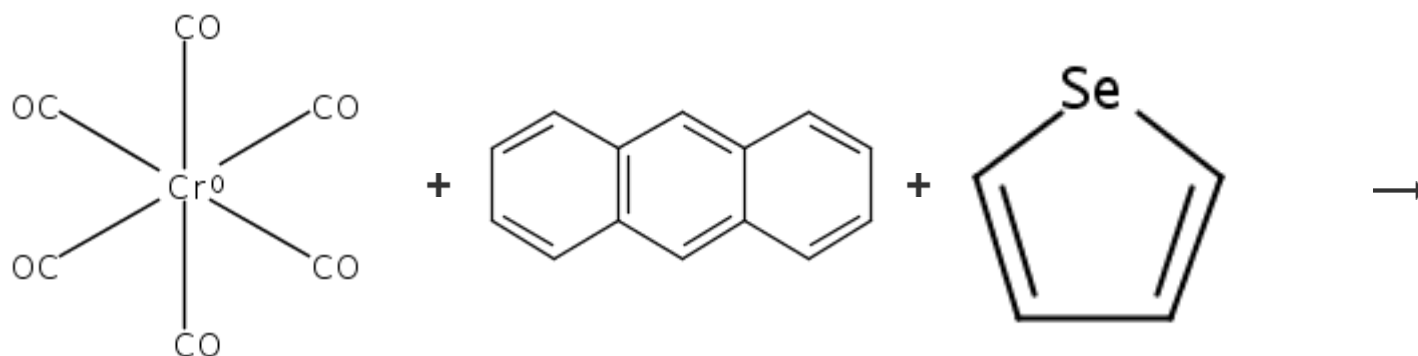
General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS_{202} closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η 5- $\text{C}_4\text{H}_4\text{Se}$) $\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or $> 500 \text{ nm}$) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Reaction Protocol**Procedure**

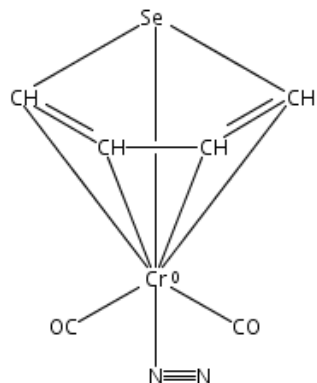
1. Isolate the matrix apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump and temperature control unit.
2. Deposit the matrixes onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

97. 3 Steps

[Step 2.1]



Overview

Steps/Stages

- 1.1 S:THF, S:Bu₂O, 6 h, reflux
- 2.1 S:THF, 75 min, reflux
- 3.1 R:



20K

Notes

3) photochem., 365nm, Reactants: 3,
Reagents: 1, Solvents: 2, Steps: 3, Stages: 3,
Most stages in any one step: 1

References

[Photoinduced Se-C Insertion Following Photolysis of \(η⁵-C₄H₄Se\)Cr\(CO\)₃. A Picosecond and Nanosecond Time-Resolved Infrared, Matrix Isolation, and DFT Investigation](#)

By Brennan, Peter et al

From *Organometallics*, 27(15), 3671-3680; 2008

Experimental Procedure

Step 1

Preparation of ((η⁶-C₁₄H₁₀)Cr(CO)₃). A solution of Cr(CO)₆ (400 mg, 1.8 mmol) and C₁₄H₁₀ (3.4 g, 19 mmol) in dibutyl ether (12 mL) containing THF (1 mL) was heated to its reflux temperature for 6 h with continual stirring. The resulting dark red solution was then cooled and filtered through Celite. The solvent was then removed under reduced pressure. The resulting crude product was chromatographed on silica using 2:1 petroleum ether/dichloromethane mobile, ((η⁶-C₁₄H₁₀)Cr(CO)₃ eluting as a red band. The product was then recrystallized from toluene/ n-pentane yielding 141 mg (0.44 mmol), representing a 41% yield based on Cr(CO)₆. Spectroscopic data were in accordance with those previously reported.²⁹ ((η⁶-C₁₄H₁₀)Cr(CO)₃, yield 141 mg 41%

Step 2

Preparation of (η⁵-C₄H₄Se)Cr(CO)₃. (η⁵-C₄H₄Se)Cr(CO)₃ was prepared by thermal arene exchange of anthracene by selenophene from (η⁶-C₁₄H₁₀)Cr(CO)₃ using a modification of the published method.²⁸ A solution of ((η⁶-C₁₄H₁₀)Cr(CO)₃ (400 mg, 1.3 mmol) and C₄H₄Se (3 g, 23 mmol) in 2.5 mL of freshly distilled tetrahydrofuran (THF) was heated to reflux temperature under an argon atmosphere for 75 min. The solvent was then removed under reduced pressure at room temperature, yielding an orange solid. The crude material was chromatographed on neutral silica eluting with 3:1 petroleum ether/dichloromethane. Following elution of the first band of unreacted (η⁶-C₁₄H₁₀)Cr(CO)₃ the mobile phase was changed to pure dichloromethane. The product eluted as the second band. Removal of the solvent under reduced pressure yielded a dark red powder. The crude product was recrystallized from toluene/n-pentane, yielding 197 mg of product, representing a 57% yield based on (η⁶-C₁₄H₁₀)Cr(CO)₃. Spectroscopic data were in accordance with those previously reported.²⁸ (η⁵-C₄H₄Se)Cr(CO)₃, yield 179 mg 57%

Step 3

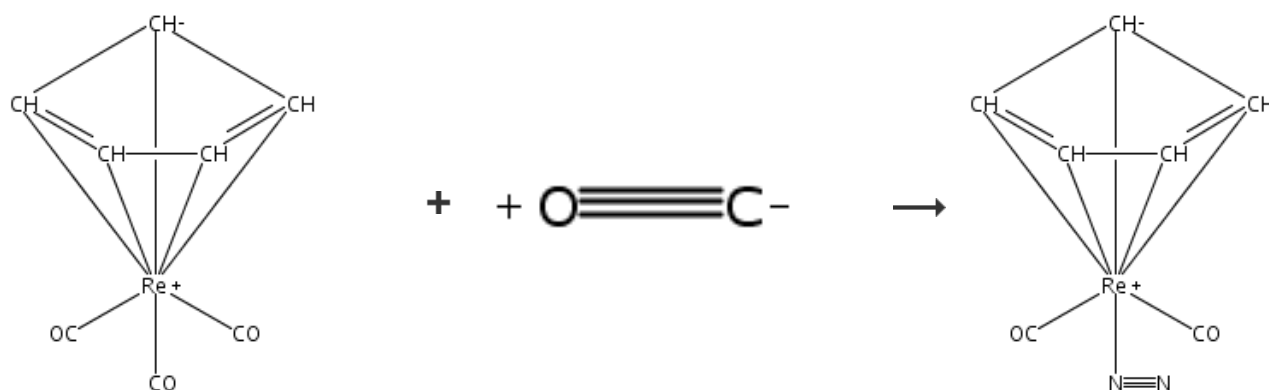
General/Typical Procedure: **Matrix Isolation Apparatus.** The matrix setup was described previously.³⁰ Briefly, the matrix isolation apparatus consists of a closed cycle helium refrigerator, sample window, shroud, deposition tube, gas mixing chamber, gas inlet, backing pump, diffusion pump, and temperature control unit. Matrixes were deposited onto a CaF_2 window cooled to 20 K, with matching outer windows on the vacuum shroud. Samples for infrared spectroscopy were deposited onto a CaF_2 window cooled by an X CS_{202} closed-cycle refrigerator to 20 K. A thermocouple embedded into a cavity beside the window and connected to the temperature control unit maintains the temperature. The window is cooled by a closed cycle helium refrigerator (Air Products), mounted via a double O-ring seal in a stainless steel vacuum shroud. The system pumps to 8×10^{-4} mbar prior to cooling and achieves 10^{-7} mbar upon cooling to 20 K. Host gases (Cryo Service) are deposited onto a window via a needle valve. For mixed gas matrixes, the gases are mixed in the correct ratio in the stainless steel mixing chamber prior to deposition. A ratio of sample molecule to host matrix in the region 1:2000 is desirable. Typically the rate of gas deposition of 0.6 Torr per minute achieves sufficient dilution of analyte in the matrix. All manipulations were carried out under an argon atmosphere. (η 5- $\text{C}_4\text{H}_4\text{Se}$) $\text{Cr}(\text{CO})_3$ was sublimed from a right-angled tube at 334 K as the gas stream entered the vacuum shroud. The sample was deposited onto the windows at 20 K and monitored in the infrared until the absorbance reading of one of the metal-carbonyl bands was approximately 1 AU. When the required absorbance was achieved, the samples were photolyzed and IR changes monitored on a Perkin-Elmer Spectrum One FTIR spectrophotometer (typically 16 scans at 1 cm^{-1} resolution). Photolysis wavelengths were selected with interference or cutoff filters (λ_{exc}) 405, 365, using a 250 W Xe/Hg lamp, > 410 or $> 500 \text{ nm}$) used in conjunction with a 250 W Xe lamp, both supplied by Oriel Scientific.

Reaction Protocol**Procedure**

1. Heat a solution of $\text{Cr}(\text{CO})_6$ (1.8 mmol) and $\text{C}_{14}\text{H}_{10}$ (19 mmol) in dibutyl ether (12 ml) containing THF (1 ml) to its reflux temperature for 6 hours with continual stirring.
2. Cool the resulting dark red solution.

[View more...](#)[View with
MethodsNow](#)

CASREACT®: Copyright © 2020 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.

98. 3 Steps

[Multiple Steps]

[Overview](#)**Steps/Stages****Notes**

1.1 R:



2.1

3.1

1) photochem., supercrit. xenon medium,
Reactants: 2, Reagents: 1, Steps: 3, Stages:
3, Most stages in any one step: 1

References

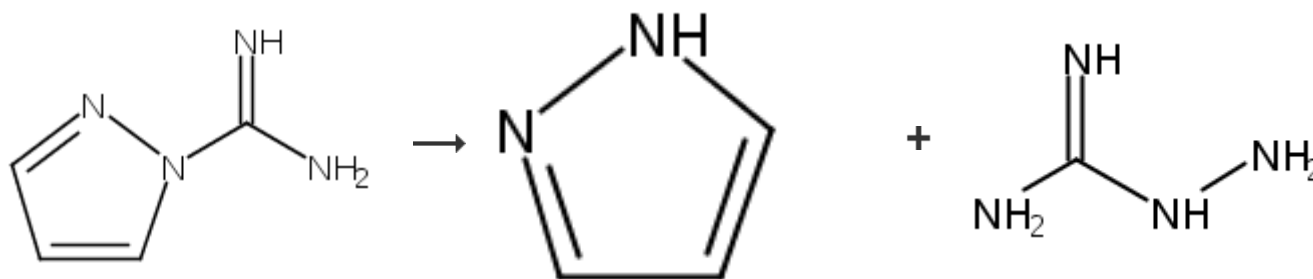
[The synthesis and spectroscopic identification of \$\[\(\eta^5\text{-C}_5\text{H}_5\)\text{Re}\(\text{N}_2\)_3\]\$ and \$\[\(\eta^5\text{-C}_5\text{H}_5\)\text{Re}\(\text{CO}\)\(\text{N}_2\)_2\]\$ in supercritical xenon at room temperature and in dinitrogen matrixes at 20 K](#)

By Howdle, Steven M. et al

From Journal of the Chemical Society, Chemical Communications, (20), 1517-19; 1989

CASREACT®: Copyright © 2020 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.

99. Single Step



Overview

Steps/Stages

1.1 R:



22°C

Notes

Synthesis of Substituted Guanidines, multistep transformation, Stage 1: Solvent, Base, Reactants: 1, Reagents: 1, Steps: 1, Stages: 1, Most stages in any one step: 1

References

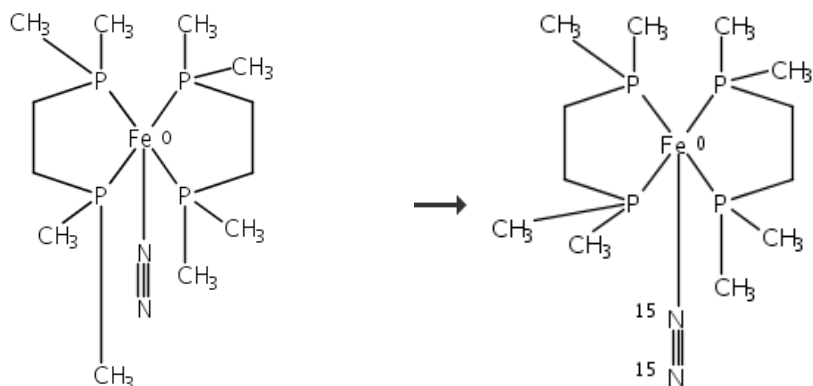
[1H-Pyrazole-1-carboxamidine Hydrochloride](#)

By Bernatowicz, Michael S.

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

CASREACT®: Copyright © 2020 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.

100. Single Step



Overview

Steps/Stages

1.1 R:



Notes

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

References

Cationic silyldiazenido complexes of the Fe(diphosphine)₂(N₂) platform: structural and electronic models for an elusive first intermediate in N₂ fixation

By Piascik, Adam D. et al

From Chemical Communications (Cambridge, United Kingdom), 53(54), 7657-7660; 2017

CASREACT®: Copyright © 2020 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.