*SUPPORTING INFORMATION*

# Carbon Dioxide Reduction and Carbon Monoxide Activation Employing a Reactive Uranium(III) Complex

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## Experimental Details

**General Procedures:** All experiments were performed under a dry nitrogen atmosphere using standard Schlenk techniques or MBraun inert-gas glove boxes. Solvents were purified using a two-column solid-state purification system (Glasscontour System, Irvine, CA), transferred to the glove box without exposure to air, and stored over activated molecular sieves and sodium metal (hydro carbons) or activated molecular sieves, only. NMR solvents were obtained from Cambridge Isotope Laboratories, degassed and stored over activated molecular sieves prior to use.

**Methods:** Unless otherwise noted, $^{1}H$ NMR spectra (300 MHz) were recorded at a probe temperature of 20$^{o}$C on a Varian Mercury 300 spectrometer in $C_6D_6$. Chemical shifts were referenced to *protio* solvent impurities ($\delta$ 7.15 ($C_6D_6$)) and are reported in ppm. Infrared spectra (400-4000 $cm^{-1}$) were obtained on a Thermo Nicolet Avatar 360 FT-IR spectrophotometer. The infrared spectra of gases and solid samples were recorded in a gas cell and as nujol mull, respectively. Elemental analyses were obtained from Kolbe Microanalytical Laboratory (Muelheim/Ruhr, Germany).

**Materials:** Uranium turnings were purchased from Alfa Aesar and activated according to literature proceedings. [$(THF)_4UI_3$] and [$U(N(SiMe_3)_2)_3$] were prepared as described by Clark et al.[1-3] [(L)U] and [(L)U($N_3$)] were prepared according to the previously reported procedures.[4] Azidotrimethylsilane (97%) was obtained from Acros, Organic and used as received.

**Carbon monoxide** (unlabeled, **Research Purity 99.998%**), see gas grade purity specifications below) was obtained from Matheson Tri Gas. **18O-labeled carbon dioxide** ($C^{18}O_2$, 95% 18-O enriched) was purchased from Cambridge Isotopes Laboratories. **13C-labeled carbon monoxide** ($^{13}CO_2$, 99% 13-C enriched) was purchased from Sigma-Aldrich, Matheson Tri-Gas, and Cambridge Isotope Laboratories. Sigma-Aldrich's Certificate of Analysis (see below) shows that commercially available sources of isotopically-labeled CO gas have only 99.9% maximum purity and contain, among other impurities, up to 20 ppm $CO_2$! Given the exceptional reactivity of trivalent [(L)U] (**1**) toward $CO_2$, as reported, isotope-labeled complexes of [{(L)U}$_2$($\mu$-CO)] (**4**) could not be synthesized; all attempts lead to rapid formation of $\mu$-oxo bridged [{(L)U}$_2$($\mu$-O)] (**2**).

# Carbon Monoxide

**Description**
Synonym: Carbon Oxide
Formula: CO

**Gas Data**
Molecular Weight: 28.01
Density: 1.16 kg/m³ @ 21.1°C, 101.325 kPa
0.0725 lb/ft³ @ 70°F, 14.696 psia
Specific Volume: 0.862 m³/kg @ 21.1°C, 101.325 kPa
13.8 ft³/lb @ 70°F, 14.696 psia

**Safety Information**
LFL in Air: 12.5%
UFL in Air: 74%
Permissible Exposure Limit (TLV): 25 ppm

**Shipping Information**
CAS Registry Number: 630-08-0
UN Number: UN 1016
DOT Proper Shipping Name: Carbon Monoxide, Compressed
DOT Classification: 2.3, Hazard Zone D (gas poisonous by inhalation)
DOT Label: INHALATION HAZARD 
FLAMMABLE GAS
TC Shipping Name: Carbon Monoxide, Compressed
TC Classification: 2.3, 2.1
TC Label: POISON GAS, FLAMMABLE GAS

**Cylinder Specifications**

| Cylinder Size | Valve Outlet CGA No. | Pressure psig @ 70°F | Pressure kPa @ 21.1°C | Approximate Ship Weight lb | kg |
|---|---|---|---|---|---|
| 1A | 350 | 1,650 | 11,376 | 145 | 66 |
| 1R | 350 | 1,650 | 11,376 | 58 | 26 |
| 2 | 350 | 1,650 | 11,376 | 69 | 31 |
| 2R | 350 | 1,650 | 11,376 | 36 | 16 |
| 3 | 350 | 1,650 | 11,376 | 31 | 14 |
| 3R | 350 | 1,650 | 11,376 | 18 | 8 |
| 6A | 180 | 1,500 | 10,342 | 2 | 1 |
| LB | 170 | 1,500 | 10,342 | 4 | 2 |

| Gas Grade Purity Specifications | | | Product Code | Cylinder Size | Content US | Content Metric | Equipment Recommendations | Model No. | Page No. |
|---|---|---|---|---|---|---|---|---|---|
| **Carbon Monoxide, Research Purity 99.998%** | | | G2119118 | 1R | 117 ft³ | 3.31 m³ | Dual Stage Reg. | Series 3810-350 | 311 |
| *Guaranteed Specifications* | | | G2119147 | 2R | 62 ft³ | 1.8 m³ | Single Stage Reg. | Series 3510-350 | 305 |
| Carbon Dioxide | < | 3 ppm | G2119159 | 3R | 23 ft³ | 0.7 m³ | Cross Purge | 4774-350 | 414 |
| Hydrogen | < | 1 ppm | G2119172 | 6A | 3.06 ft³ | 88 L | Personal Monitor | IQ250 | 388 |
| THC as Methane | < | 0.5 ppm | | | | | Leak Detector | 8081A | 394 |
| Iron Pentacarbonyl | < | 0.5 ppm | | | | | Detector Tube | 8014-106S | 381 |
| Nitrogen | < | 10 ppm | | | | | Cyl. Valve Wrench | TW-5 | 422 |
| Oxygen | < | 0.5 ppm | | | | | | | |
| Water | < | 1 ppm | | | | | | | |
| *A Certificate of Analysis is available on request.* | | | | | | | | | |
| **Carbon Monoxide, Matheson Purity 99.99%** | | | G1821101 | 1A | 175 ft³ | 4.96 m³ | Dual Stage Reg. | Series 3120-350 | 290 |
| *Guaranteed Specifications* | | | G1821140 | 2 | 66 ft³ | 1.87 m³ | Single Stage Reg. | Series 3530-350 | 306 |
| Carbon Dioxide | < | 20 ppm | G1821150 | 3 | 26 ft³ | 0.74 m³ | Tee Purge | 4753-350 | 415 |
| Hydrogen | < | 10 ppm | | | | | Personal Monitor | IQ250 | 388 |
| Nitrogen | < | 20 ppm | | | | | Leak Detector | 8081A | 394 |
| Oxygen | < | 5 ppm | | | | | Detector Tube | 8014-106S | 381 |
| THC as Methane | < | 5 ppm | | | | | Cyl. Valve Wrench | TW-5 | 422 |
| Water | < | 3 ppm | | | | | | | |
| *A Certificate of Analysis is available on request.* | | | | | | | | | |
| **Carbon Monoxide, Ultra High Purity 99.9%** | | | G1918701 | 1A | 175 ft³ | 4.96 m³ | Dual Stage Reg. | Series 3120-350 | 290 |
| *Guaranteed Specifications* | | | G1918740 | 2 | 66 ft³ | 1.87 m³ | Single Stage Reg. | Series 3530-350 | 306 |
| Carbon Dioxide | < | 50 ppm | G1918750 | 3 | 26 ft³ | 0.74 m³ | LB Regulator | Series 3550-170 | 307 |
| Nitrogen | < | 200 ppm | G1918775 | LB | 1.5 ft³ | 44 L | Tee Purge | 4753-350 | 415 |
| Oxygen | < | 10 ppm | | | | | Personal Monitor | IQ250 | 388 |
| THC as Methane | < | 10 ppm | | | | | Leak Detector | 8081A | 394 |
| Water | < | 10 ppm | | | | | Detector Tube | 8014-106S | 381 |
| | | | | | | | Cyl. Valve Wrench | TW-5 | 422 |
| *A Certificate of Conformance will be provided on request.* | | | | | | | | | |
| *A lot analysis is available at a nominal charge.* | | | | | | | | | |

# Carbon Monoxide *(continued)*

| Gas Grade Purity Specifications | Product Code | Cylinder Size | Content US | Content Metric | Equipment Recommendations | Model No. | Page No. |
|---|---|---|---|---|---|---|---|
| **Carbon Monoxide, CP Grade 99.5%** | G1132201 | 1A | 175 $ft^3$ | 4.96 $m^3$ | Dual Stage Reg. | Series 81-350 | 285 |
| | G1132240 | 2 | 66 $ft^3$ | 1.87 $m^3$ | Single Stage Reg. | Series 1-350 | 284 |
| | G1132250 | 3 | 26 $ft^3$ | 0.74 $m^3$ | LB Regulator | Series 3320-170 | 296 |
| | G1132275 | LB | 1.5 $ft^3$ | 44 L | Tee Purge | 4753-350 | 415 |
| | | | | | Personal Monitor | IQ250 | 388 |
| | | | | | Leak Detector | 8081A | 394 |
| | | | | | Detector Tube | 8014-106S | 381 |
| | | | | | Cyl. Valve Wrench | TW-5 | 422 |
| *A Certificate of Conformance will be provided on request.* | | | | | | | |
| **Carbon Monoxide, Technical Purity 99%** | G1132101 | 1A | 175 $ft^3$ | 4.96 $m^3$ | Dual Stage Reg. | Series 81-350 | 285 |
| | G1132140 | 2 | 66 $ft^3$ | 1.87 $m^3$ | Single Stage Reg. | Series 1-350 | 284 |
| | G1132150 | 3 | 26 $ft^3$ | 0.74 $m^3$ | LB Regulator | Series 3320-170 | 296 |
| | G1132175 | LB | 1.5 $ft^3$ | 44 L | Tee Purge | 4753-350 | 415 |
| | | | | | Personal Monitor | IQ250 | 388 |
| | | | | | Leak Detector | 8081A | 394 |
| | | | | | Detector Tube | 8014-106S | 381 |
| | | | | | Cyl. Valve Wrench | TW-5 | 422 |
| *A Certificate of Conformance will be provided on request.* | | | | | | | |

SIGMA-ALDRICH

# Certificate of Analysis

| | |
|---|---|
| **Product Name** | Carbon-$^{13}$C monoxide, 99 atom % 13C, <5 atom % 18O |
| **Product Number** | 38,850-5 |
| **Product Brand** | Aldrich |
| **CAS Number** | 1641-69-6 |
| **Molecular Formula** | $^{13}$CO |
| **Molecular Weight** | 29.00 |

| TEST | SPECIFICATION | LOT 10771AE RESULTS |
|---|---|---|
| **ISOTOPIC ASSAY** | 98.5 ATOM % 13C (MINIMUM) | 0.6 ATOM% 12C<br>99.4 ATOM% 13C<br>98.0 ATOM% 16O<br>0.5 ATOM% 17O<br>1.5 ATOM% 18O |
| **GAS LIQUID CHROMATOGRAPHY** | | N2 < 20 PPM<br>CO2 < 20 PPM<br>AR/O2<20 PPM<br>99.9% PURITY |
| **QUALITY CONTROL ACCEPTANCE DATE** | | MARCH 2005 |

Ronnie J. Martin, Supervisor
Quality Control
Milwaukee, Wisconsin USA

**Synthesis and characterization of [{(L)U}$_2$(μ-$^{16}$O)] and [{(L)U}$_2$(μ-$^{18}$O)] (2)[5]**

**Synthesis of [{(L)U}$_2$(μ-$^{16}$O)] and [{(L)U}$_2$(μ-$^{18}$O)]:** The following procedure is a general protocol for the synthesis of μ-oxo bridged diuranium complexes. A sealed vial containing 5 mL of a benzene or toluene solution of [(L)U] (0.100 g, 0.098 mmol) was sparged with carbon dioxide gas while stirring at room temperature. After 2 minutes a pale blue-green solid precipitate. This precipitate was collected, washed with pentane, and dried under vacuum (yield: 0.100 g, 0.050 mmol, >95%). For CHN elemental analysis see Ref. #5 below.

**$^1$H NMR (300 MHz, benzene-d$_6$, 20 °C)**: δ = 69.62 (s, 1H, $\Delta\nu_{1/2}$ = 12.82 Hz), 66.47 (s, 1H, $\Delta\nu_{1/2}$ = 10.99 Hz), 3.58 (s, 1H, $\Delta\nu_{1/2}$ = 16.09), 1.41 (s, 1H, $\Delta\nu_{1/2}$ = 20.58 Hz), 0.57 (s, 1H, $\Delta\nu_{1/2}$ = 8.49 Hz), –1.56 (s, 1H, $\Delta\nu_{1/2}$ = 27.90 Hz), –4.69 (s, 1H, $\Delta\nu_{1/2}$ = 8.69 Hz), –5.02 (s, 9H, $\Delta\nu_{1/2}$ = 11.57 Hz), –9.03 (s, 1H, $\Delta\nu_{1/2}$ = 17.23 Hz), –27.67 (s, 9H, $\Delta\nu_{1/2}$ = 21.97 Hz).

**IR spectrum (Nujol):** $\nu_{as}$(U–$^{16}$O—U) = 589 cm$^{-1}$, $\nu_{as}$(U–$^{18}$O—U) = 570 cm$^{-1}$.

**Figure S1. $^1$H NMR spectrum of [{(L)U}$_2$(μ-$^{16}$O)]:**

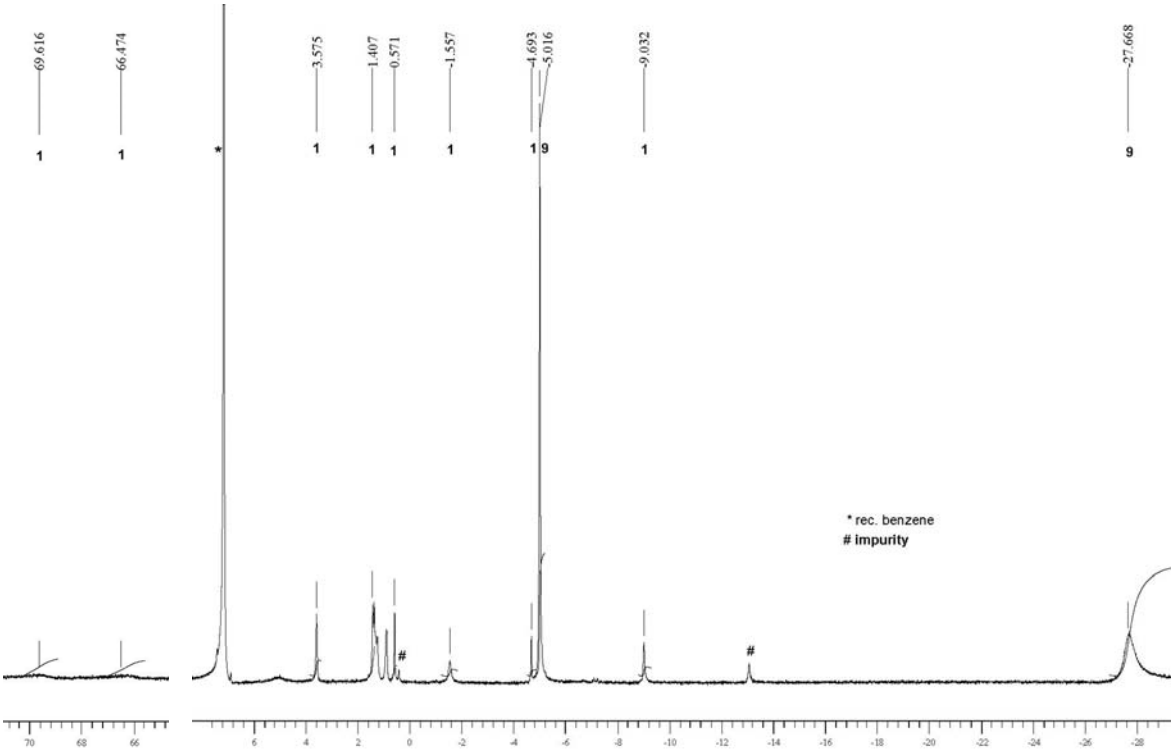


**Figure S2. IR spectra of [{(L)U}$_2$(μ-$^{16}$O)] and [{(L)U}$_2$(μ-$^{18}$O)]:**

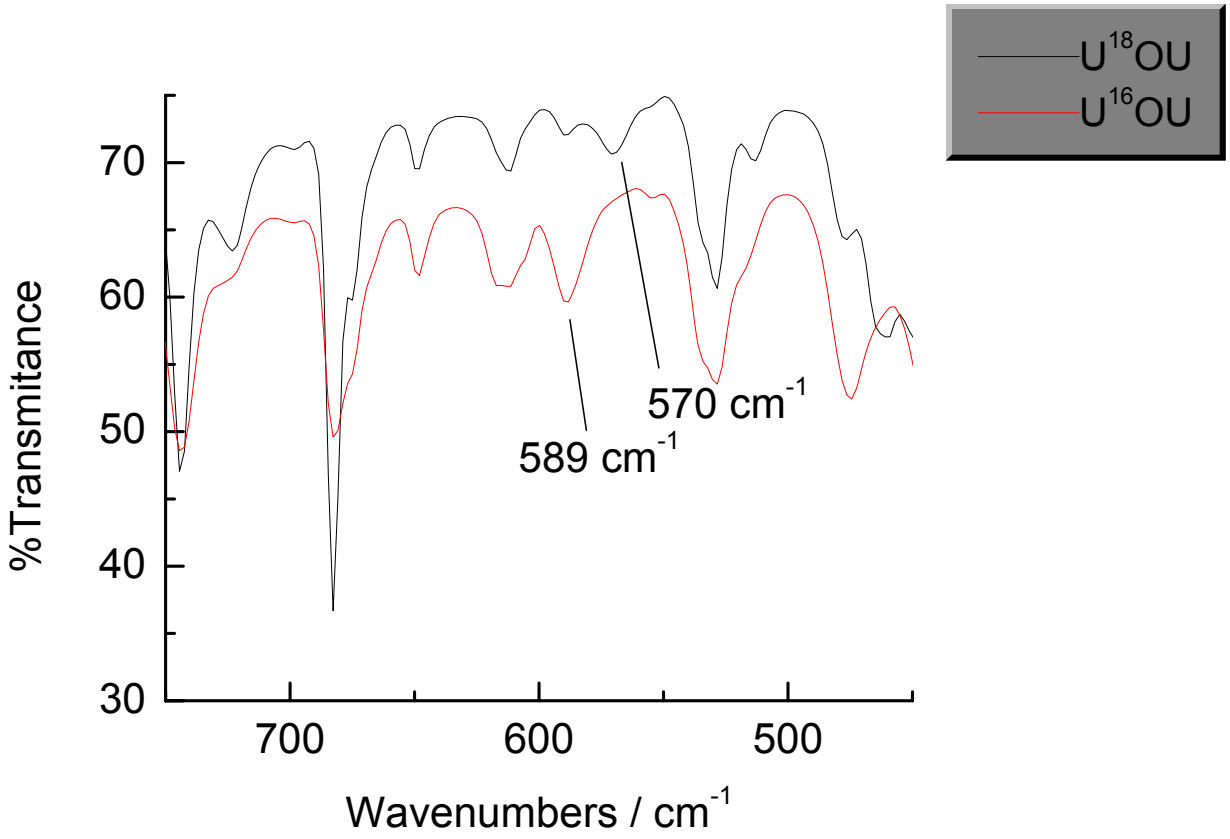
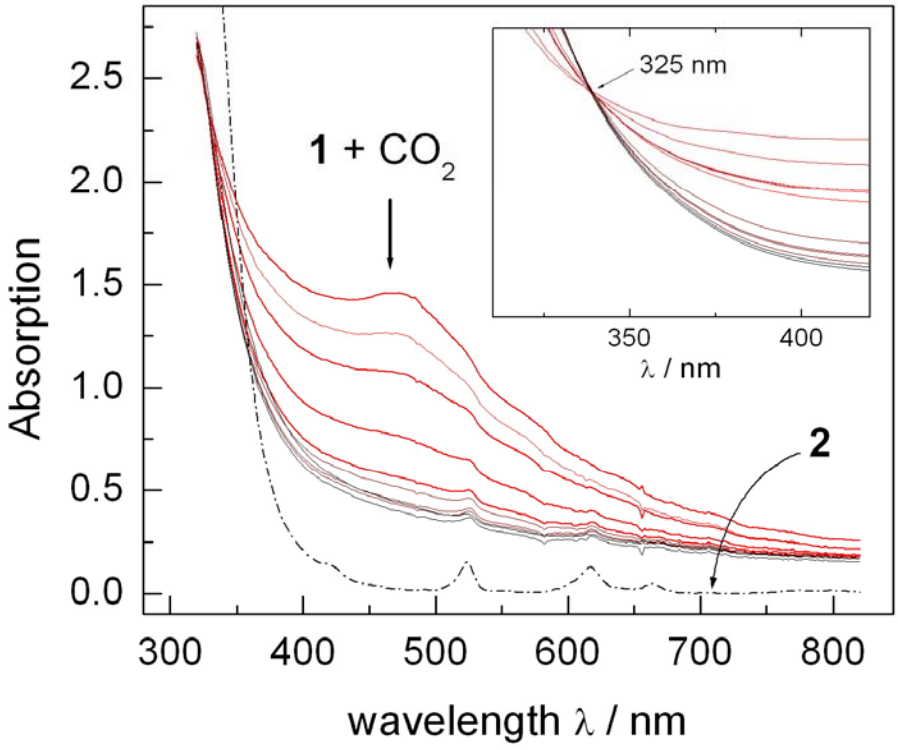


**Figure S3.** Electronic absorption spectrum of the reaction of **1** with $CO_2$ gas. The graph shows the gradual color change from deep-red **1** to colorless intermediate **3**. Upon extended reaction times, dinuclear μ-oxo bridged **2** (pale-blue) is formed (dashed line). Samples were measured in benzene. Sample conditions: 3 mg **1** in 3 mL $C_6H_6$ and 0.5 mL $CO_2$ at 25°C.



**Synthesis and characterization of [{(L)U}$_2$(μ-CO)] (4)**

**Synthesis of [{(L)U}$_2$(μ-CO)]:** A 20 mL vial was charged with [(L)U] (0.050 g, 0.049 mmol) and 5 mL of pentane. The opening of the vial was sealed with a ballon filled with carbon monoxide and the solution was stirred under the CO atmophere at room temperature. After ~10h all the solvent had evaporated and the resulting pale brown residue was disssolved in 2 mL of benzene and the solution was filtered. From this solution, brown hexagonal shaped crystals suitable for X-ray diffraction analysis formed within 4-6 weeks at room temperature (recrystallized yield: 0.005 g, 0.0024 mmol, 9.8 %).

We note that the corresponding dinitrogen-bridged species could not be synthesized, neither under ~1 atm nor an overpressure (80 psi) of $N_2$ gas.

**IR spectrum (Nujol):** $\nu_{as}(CO) = 2091\ cm^{-1}$

**CHN elemental analysis (%) calcd.:** C 58.81, H 7.70, N 4.06; found: C 59.63, H 7.65, N 4.09.

**Synthesis and characterization of [{(L)U}$_2$(μ-N$_3$)] (5)**

**Synthesis of [{(L)U}$_2$(μ-N$_3$)]:** A solution of [(L)U] (0.093 g, 0.091 mmol) in 5 mL benzene was added to a solution of [(L)U(N$_3$)] (0.096 g, 0.091 mmol) in 5 mL benzene and stirred for 18 h at room temperature. The resulting red/purple precipitate was filtered, washed with benzene and dried under vacuum (yield: 0.165 g, 0.079 mmol, 85 %). Recrystallization from benzene yielded red/brown crystals suitables for X-ray diffraction analysis.

**IR spectrum (Nujol):** $\nu_{as}(N_3) = 2074\ cm^{-1}$,

**CHN elemental analysis (%) calcd.:** C 58.89, H 7.56, N 6.06; found: C 58.97, H 7.48, N 6.01.

## References for Experimental Details:


(1) I. Castro-Rodriguez, K. Olsen, P. Gantzel, K. Meyer, *J. Am. Chem. Soc.* **2003**, *125*, 4565-4571.

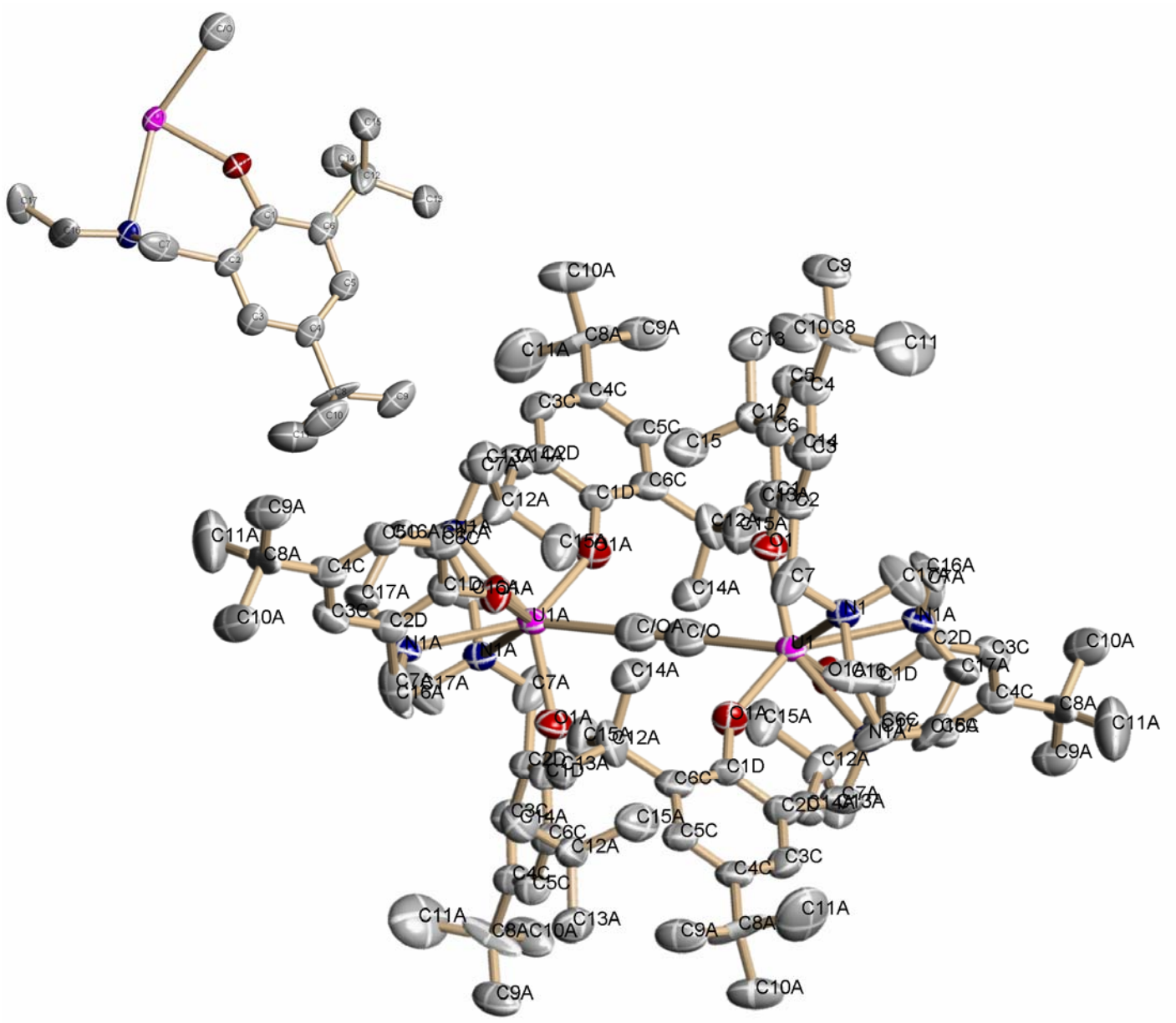
(2) Clark, D. L.; Sattelberger, A. P.; Andersen, R. A. *Inorg. Synth.*, **1997**, *31*, 307-315.

(3) Avens, L. R.; Bott, S. G.; Clark, D. L.; Sattelberger, A. P.; Watkin, Zwick, B. D. *Inorg. Chem.*, **1989**, *28*, 1771-1773.

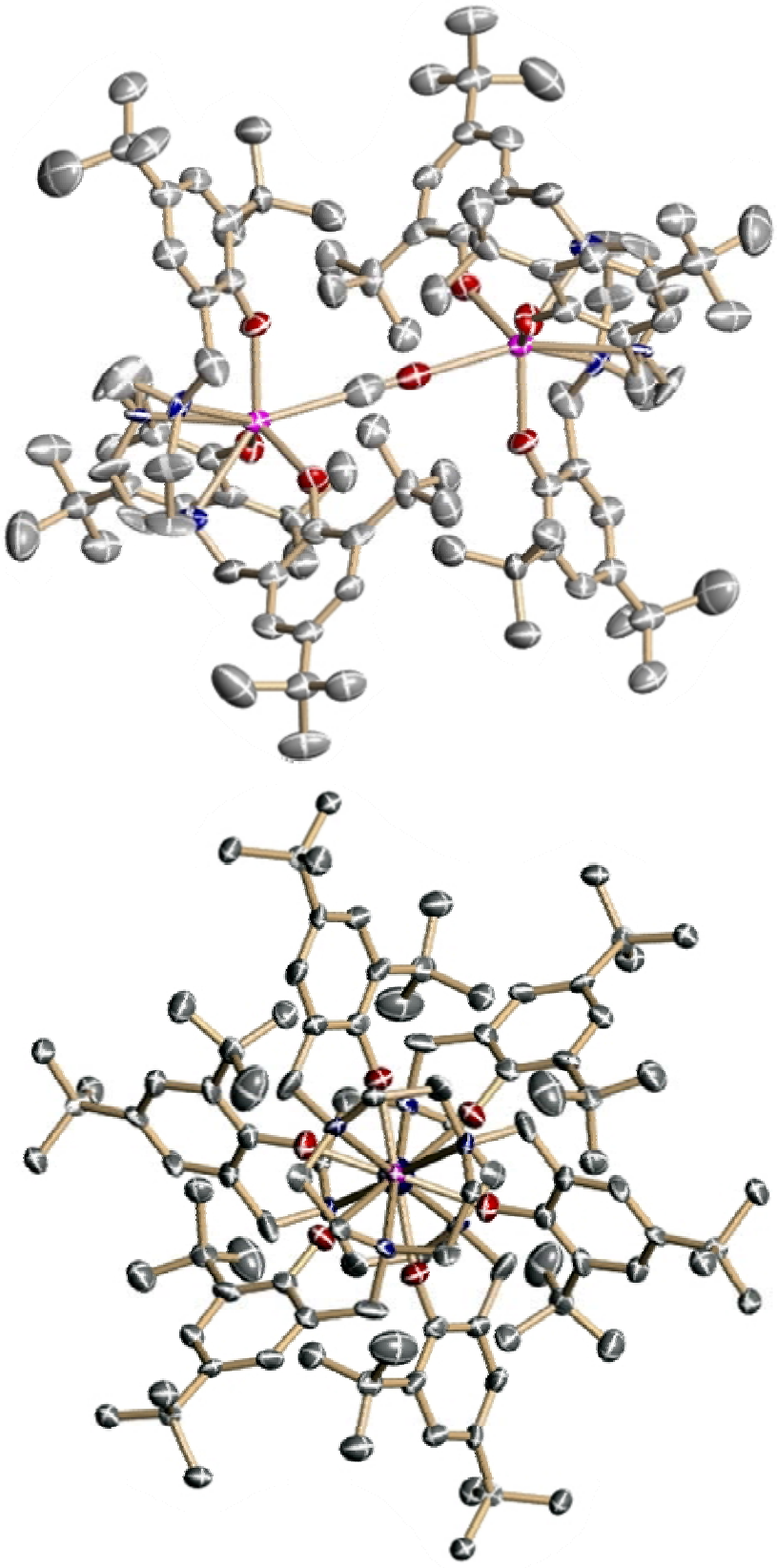
(4) D. L. Clark, A. P. Sattelberger, S. G. Bott, R. N. Vrtis, *Inorg. Chem.*, 1989, **28**, 1771-1773.

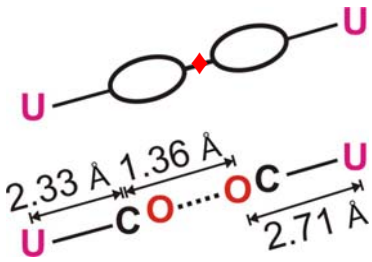
(5) I. Castro-Rodriguez, K. Olsen, P. Gantzel, K. Meyer *Chem. Commun.* **2002**, 2764-2765.

**Crystallographic Supporting Information for [{(L)U}$_2$(μ-CO)] • 3C$_6$H$_6$, (4) • 3C$_6$H$_6$**



**Additional graphic representations and comments regarding the employed model:**






The structure of **4** • 3 $C_6H_6$ is of limited resolution and thus, **no reliable bond distance data are available for the bridging CO entity**. However, for graphic representation only, a refinement was attempted that employed a model with an asymmetrically bridged U-CO—U ligand, comprising one short U-C bond and a longer U—O isocarbonyl interaction, disordered on two positions at the inversion center. Despite the fact that the metric parameters within the disordered U-CO-U entity in **4** are error prone, the U-C(CO) bond distance of 2.33(5) Å compares well to 2.383(6) Å found in $[(Me_4C_5H)_3U(CO)]$. The U—O isocarbonyl bond distance was determined to be 2.71(2) Å. The latter structural parameter is similar to previously observed values found for uranium complexes with oxygen donor ligands. The U-O bond distances in [(L)U(THF)] (**see below**), for instance, was found at 2.63 Å.

**Crystal data and structure refinements for 3 independently synthesized samples of [{(L)U}$_2$(μ-CO)]**

**1. Crystal data and structure refinement 4 • $3C_6H_6$ (discussed in the main manuscript)**

| | | |
|---|---|---|
| Identification code | Ingrid 34, **4 • $3C_6H_6$** | |
| Empirical formula | C84.50 H111 N3 O3.50 U | |
| Formula weight | 1462.79 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Rhombohedral | |
| Space group | R3 | |
| Unit cell dimensions | a = 16.0236(10) Å | α= 74.4450(10)°. |
| | b = 16.0236(10) Å | β= 74.4450(10)°. |
| | c = 16.0236(10) Å | γ = 74.4450(10)°. |
| Volume | 3732.0(4) Å$^3$ | |
| Z | 2 | |
| Density (calculated) | 1.302 Mg/m$^3$ | |
| Absorption coefficient | 2.225 mm$^{-1}$ | |
| F(000) | 1518 | |
| Crystal size | 0.15 x 0.15 x 0.05 mm$^3$ | |
| Theta range for data collection | 1.69 to 22.49°. | |
| Index ranges | -16<=h<=17, -17<=k<=17, -17<=l<=17 | |
| Reflections collected | 18143 | |
| Independent reflections | 3259 [R(int) = 0.0530] | |
| Completeness to theta = 22.49° | 100.0 % | |
| Absorption correction | None | |
| Max. and min. transmission | 0.8969 and 0.7314 | |
| Refinement method | Full-matrix least-squares on F$^2$ | |
| Data / restraints / parameters | 3259 / 0 / 230 | |
| Goodness-of-fit on F$^2$ | 1.163 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0435, wR2 = 0.1140 | |
| R indices (all data) | R1 = 0.0510, wR2 = 0.1202 | |
| Largest diff. peak and hole | 2.147 and -0.416 e.Å$^{-3}$ | |

## 2. Crystal data and structure refinement (additional information)

Table 1. Crystal data and structure refinement for INGRID20.

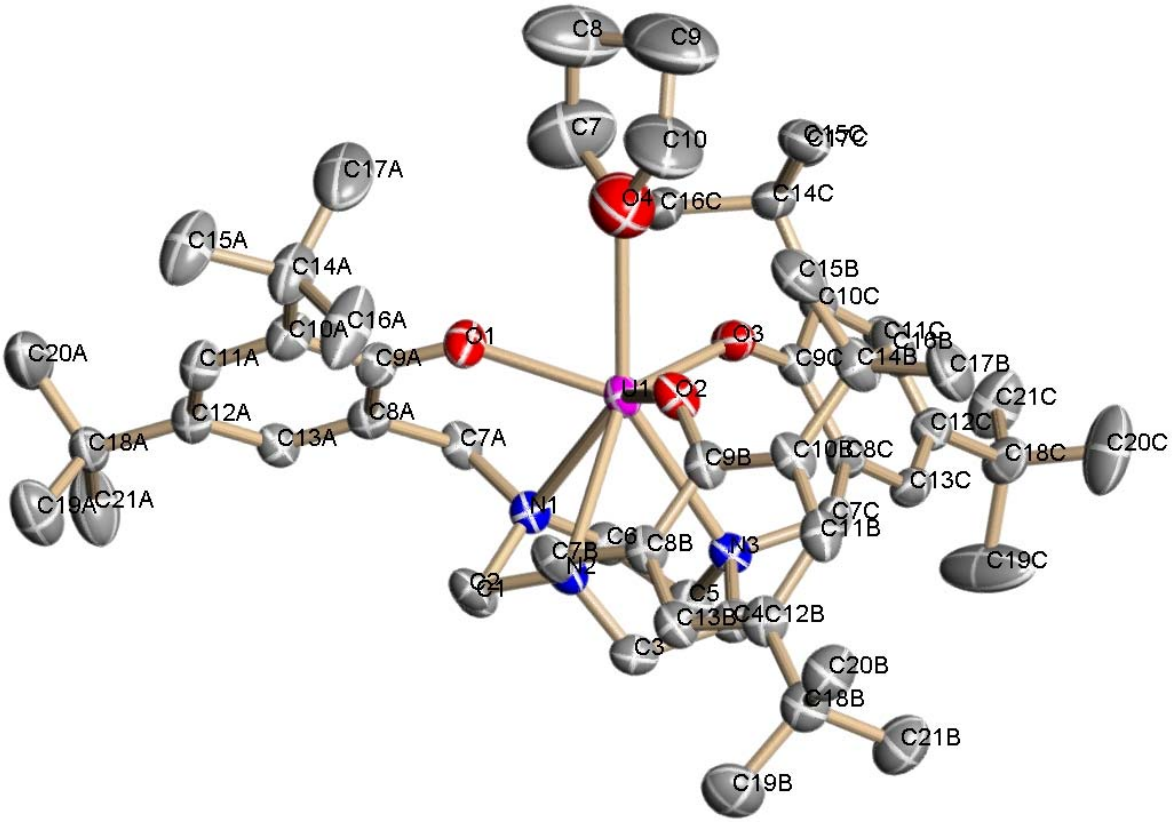
| | | |
|---|---|---|
| Identification code | Ingrid20 | |
| Empirical formula | C84 H111 N4 O3 U | |
| Formula weight | 1462.80 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Rhombohedral | |
| Space group | R-3 | |
| Unit cell dimensions | a = 16.0236(10) Å | α= 74.4450(10)°. |
| | b = 16.0236(10) Å | β= 74.4450(10)°. |
| | c = 16.0236(10) Å | γ = 74.4450(10)°. |
| Volume | 3732.0(4) $Å^3$ | |
| Z | 2 | |
| Density (calculated) | 1.302 $Mg/m^3$ | |
| Absorption coefficient | 2.225 $mm^{-1}$ | |
| F(000) | 1518 | |
| Crystal size | 0.15 x 0.15 x 0.05 $mm^3$ | |
| Theta range for data collection | 1.35 to 22.49°. | |
| Index ranges | -16<=h<=17, -17<=k<=17, -17<=l<=17 | |
| Reflections collected | 21738 | |
| Independent reflections | 3260 [R(int) = 0.0809] | |
| Completeness to theta = 22.49° | 100.0 % | |
| Absorption correction | None | |
| Max. and min. transmission | 0.8969 and 0.7314 | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 3260 / 0 / 89 | |
| Goodness-of-fit on $F^2$ | 1.225 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0477, wR2 = 0.1249 | |
| R indices (all data) | R1 = 0.0620, wR2 = 0.1388 | |
| Largest diff. peak and hole | 1.371 and -0.654 $e.Å^{-3}$ | |

### 3. Crystal data and structure refinement (additional information)

Table 1. Crystal data and structure refinement for hide24.

| | | |
|---|---|---|
| Identification code | Hide24 | |
| Empirical formula | C81 H108 N4 O3 U | |
| Formula weight | 1423.74 | |
| Temperature | 100(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Rhombohedral | |
| Space group | R-3c | |
| Unit cell dimensions | a = 19.3819(7) Å | α= 90°. |
| | b = 19.3819(7) Å | β= 90°. |
| | c = 68.664(5) Å | γ = 120°. |
| Volume | 22338.3(19) $Å^3$ | |
| Z | 12 | |
| Density (calculated) | 1.270 $Mg/m^3$ | |
| Absorption coefficient | 2.228 $mm^{-1}$ | |
| F(000) | 8856 | |
| Crystal size | 0.33 x 0.27 x 0.18 $mm^3$ | |
| Theta range for data collection | 1.35 to 27.53°. | |
| Index ranges | -15<=h<=25, -25<=k<=16, -88<=l<=89 | |
| Reflections collected | 43611 | |
| Independent reflections | 5608 [R(int) = 0.0462] | |
| Completeness to theta = 27.53° | 97.7 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 5608 / 0 / 268 | |
| Goodness-of-fit on $F^2$ | 1.065 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0358, wR2 = 0.1162 | |
| R indices (all data) | R1 = 0.0564, wR2 = 0.1241 | |
| Largest diff. peak and hole | 2.101 and -1.408 $e.Å^{-3}$ | |

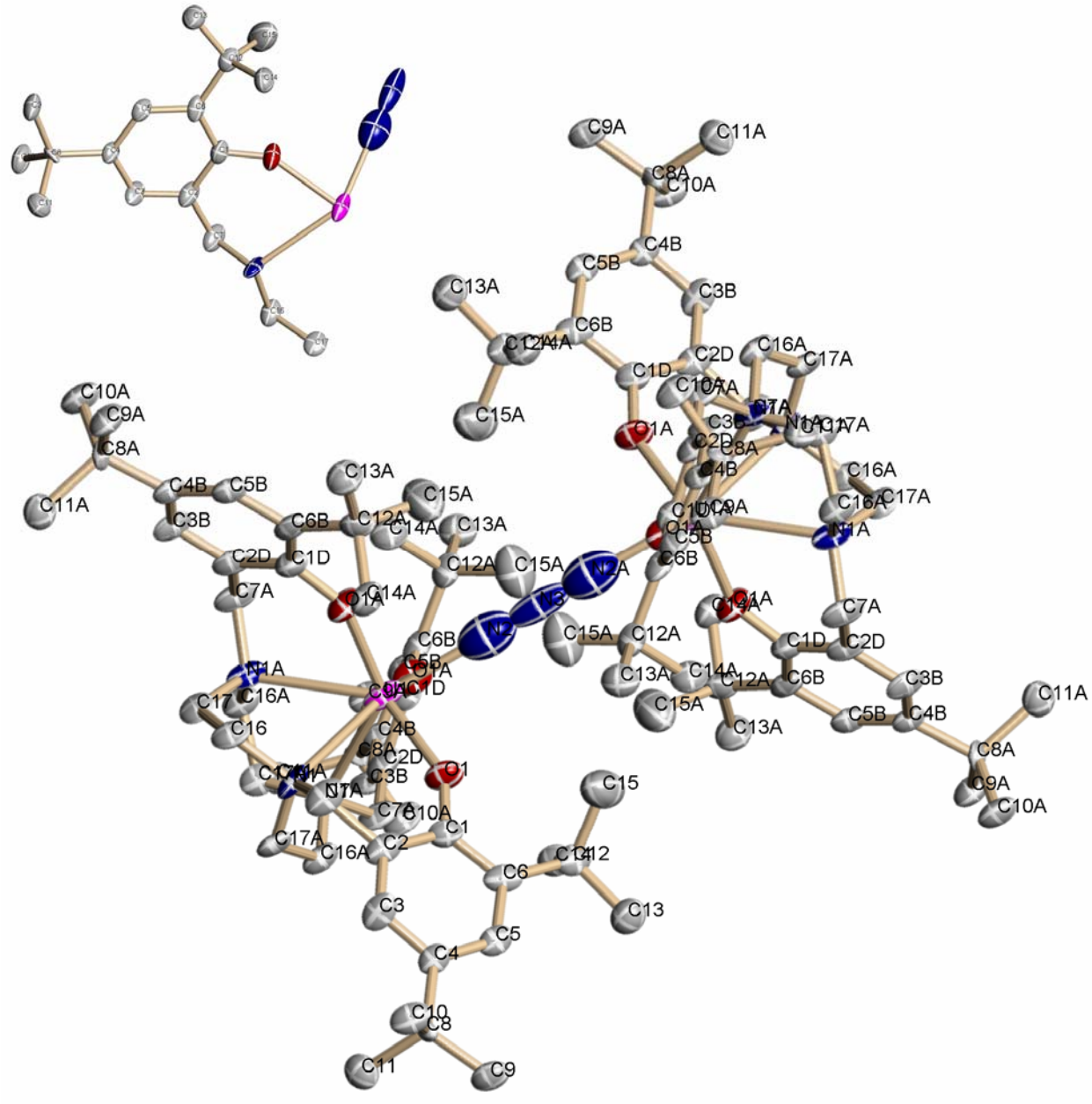
**Crystallographic Supporting Information for [(L)U(THF)] • $C_6H_{14}$**



**Crystal data and structure refinement for [(L)U(THF)] • $C_6H_{14}$**

| | | |
|---|---|---|
| Identification code | **[(L)U(THF)] • $C_6H_{14}$** | |
| Empirical formula | C3.73 H5.25 N0.18 O0.24 U0.06 | |
| Formula weight | 71.04 | |
| Temperature | 293(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Tetragonal | |
| Space group | I4(1)/a | |
| Unit cell dimensions | a = 27.6121(11) Å | $\alpha$= 90°. |
| | b = 27.6121(11) Å | $\beta$= 90°. |
| | c = 31.7727(18) Å | $\gamma$ = 90°. |
| Volume | 24224.4(19) $Å^3$ | |
| Z | 262 | |
| Density (calculated) | 1.276 $Mg/m^3$ | |
| Absorption coefficient | 2.724 $mm^{-1}$ | |
| F(000) | 9552 | |
| Crystal size | 0.15 x 0.2 x 02 mm | |
| Theta range for data collection | 1.47 to 27.58°. | |
| Index ranges | -35<=h<=29, -35<=k<=34, -41<=l<=41 | |
| Reflections collected | 76500 | |
| Independent reflections | 13957 [R(int) = 0.0687] | |
| Completeness to theta = 27.58° | 99.6 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 13957 / 0 / 600 | |
| Goodness-of-fit on $F^2$ | 0.982 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0385, wR2 = 0.1200 | |
| R indices (all data) | R1 = 0.0749, wR2 = 0.1258 | |
| Largest diff. peak and hole | 1.183 and -0.720 $e.Å^{-3}$ | |

**Crystallographic Supporting Information for [{(L)U}$_2$(μ-N$_3$)] • 3 C$_6$H$_6$, (5) • 3 C$_6$H$_6$**



**Crystal data and structure refinement for 5 • 3 $C_6H_6$**

| | | |
|---|---|---|
| Identification code | **5 • 3 $C_6H_6$** | |
| Empirical formula | C28 H34 N1.50 O U0.33 | |
| Formula weight | 486.91 | |
| Temperature | 293(2) K | |
| Wavelength | 0.71073 Å | |
| Crystal system | Rhombohedral | |
| Space group | R3 | |
| Unit cell dimensions | a = 16.1204(17) Å | $\alpha$= 73.850(2)°. |
| | b = 16.1204(17) Å | $\beta$= 73.850(2)°. |
| | c = 16.1204(17) Å | $\gamma$ = 73.850(2)°. |
| Volume | 3772.4(7) $Å^3$ | |
| Z | 6 | |
| Density (calculated) | 1.286 $Mg/m^3$ | |
| Absorption coefficient | 2.201 $mm^{-1}$ | |
| F(000) | 1507 | |
| Crystal size | 0.15 x 0.15 x 0.10 $mm^3$ | |
| Theta range for data collection | 1.35 to 22.49°. | |
| Index ranges | -17<=h<=17, -17<=k<=17, -17<=l<=17 | |
| Reflections collected | 22202 | |
| Independent reflections | 3298 [R(int) = 0.0871] | |
| Completeness to theta = 22.49° | 100.0 % | |
| Absorption correction | None | |
| Refinement method | Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 3298 / 0 / 193 | |
| Goodness-of-fit on $F^2$ | 1.119 | |
| Final R indices [I>2sigma(I)] | R1 = 0.0552, wR2 = 0.1221 | |
| R indices (all data) | R1 = 0.0596, wR2 = 0.1242 | |
| Largest diff. peak and hole | 1.281 and -1.885 $e.Å^{-3}$ | |