Table III. Selected Angles (Deg)

O(1)-U-O(2)	179.4 (1)	O(8)-S-O(9)	112.0 (2)
O(1)-U-O(3)	82.4 (1)	O(7)-C(1)-N(1)	120.4 (4)
O(1)-U-O(4)	90.4 (1)	O(7)-C(1)-N(2)	119.8 (4)
O(1)-U-O(5)	90.5 (1)	N(1)-C(1)-N(2)	119.9 (4)
O(1)-U-O(6)	85.0 (1)	O(4)-C(2)-N(3)	121.8 (4)
O(1)-U-O(7)	93.5 (1)	O(4)-C(2)-N(4)	119.9 (4)
O(3)-U-O(4)	71.3 (1)	N(3)-C(2)-N(4)	118.3 (4)
O(4)-U-O(5)	70.0 (1)	O(5)-C(3)-N(5)	120.3 (5)
O(5)-U-O(6)	72.2 (1)	O(5)-C(3)-N(6)	121.2 (5)
O(6)-U-O(7)	74.2 (1)	N(5)-C(3)-N(6)	118.5 (5)
O(7)-U-O(3)	73.7 (1)	H(1)-N(1)-H(2)	121 (5)
O(3)-S-O(6)	105.0 (2)	H(3)-N(2)-H(4)	113 (5)
O(3)-S-O(8)	108.1 (2)	H(5)-N(3)-H(6)	119 (6)
O(3)-S-O(9)	111.7 (2)	H(7)-N(4)-H(8)	119 (6)
O(6)-S-O(8)	108.9 (2)	H(9)-N(5)-H(10)	104 (4)
O(6)-S-O(9)	110.9 (2)	H(11)-N(6)-H(12)	115 (5)

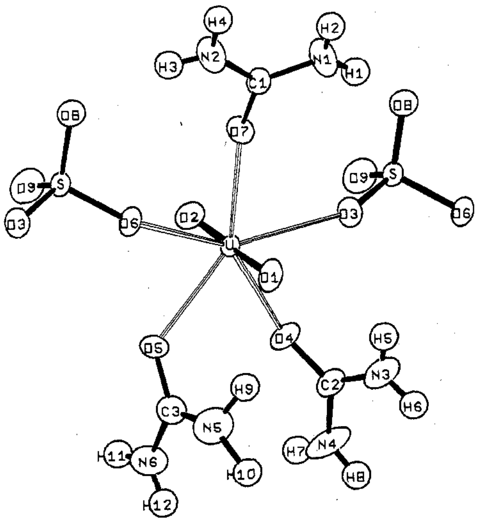

Figure 1. ORTEP drawing of the tris(urea)dioxouranium(VI) sulfate structure. An additional sulfate group is included to show the bridging. The sulfate on the right is related to the one on the left by a translation along *c* of one unit cell length.

less than 0.09σ. A difference Fourier calculation after the last cycle of refinement had a maximum electron density of 1.2 e Å^{-3}, at a location near that of uranium.

Results and Discussion

Atomic positions, distances, and angles are listed in Tables I–III. The structure (Figure 1) consists of chains of alternate tris(urea)dioxouranium(VI) ions and sulfate ions, with sulfate ions contributing two oxygen atoms to the pentagonal bipyramid of oxygen atoms coordinated to the uranium(VI) ion. The U–O(4) and U–O(5) bonds are perpendicular to the uranyl ion axis; the third urea oxygen atom (O(7)) is 0.16 Å from the equatorial plane defined by U, O(4), and O(5) while the two sulfate oxygen atoms are 0.30 Å (O(3)) and 0.20 Å (O(6)) from the equatorial plane on the side opposite to O(7). The urea molecules are planar. The H(9) and H(10) atoms appear to be out of the plane of their urea molecule, but we attribute this as an inaccuracy in the determination of these hydrogen-atom positions. One urea molecule is held approximately coplanar with the uranyl equatorial plane by hydrogen bonds to two of the sulfate oxygen atoms (N(1)–H(1)---O(8) and N(2)–H(3)---O(8) in Figure 1), while the other two urea molecules are approximately perpendicular to the uranyl equatorial plane. Bond distances and angles for urea and for the uranium(VI) coordination polyhedron are in close agreement with those in $[UO_2(OC(NH_2)_2)_5](NO_3)_2$[2] and $[UO_2(H_2O)(OC(NH_2)_2)_4](NO_3)_2$.[7] The present compound, with two sulfate oxygen atoms coordinated to the uranyl ion, is structurally similar to the nitrate salts mentioned above, in which the nitrate ions are not coordinated to the uranyl ion so that anion bridging between cation complexes cannot occur, and the two forms of $2UO_2SO_4{\cdot}7H_2O$,[8,9] in which three sulfate oxygen atoms are coordinated to the uranyl ion, resulting in complex anion bridging between cation complexes. Possible hydrogen bonds for which the N---O distance is less than 3.2 Å, the O---H distance is less than 2.4 Å, and the N–H---O angle is greater than 120° are listed in Table IV. The values in Table IV are comparable to the hydrogen-bond lengths (2.99, 3.03 Å) and angles (151, 167°) found in urea.[10]

Table IV. Possible Hydrogen Bond Distances (Å) and Angles (Deg)

atoms[a]	N–H dist	H—O dist	N···O dist	N–H–O angle
N(1)–H(1)--O(8)^{i}	0.92 (5)	2.09 (5)	2.980 (5)	162 (5)
N(1)–H(2)--O(6)^{ii}	0.77 (6)	2.30 (6)	3.070 (5)	174 (6)
N(2)–H(3)--O(8)	0.87 (5)	2.05 (6)	2.901 (5)	168 (5)
N(2)–H(4)--O(3)^{ii}	0.80 (6)	2.25 (6)	3.034 (5)	164 (6)
N(3)–H(6)--O(8)^{iii}	0.92 (5)	2.06 (5)	2.973 (5)	169 (5)
N(4)–H(8)--O(9)^{iii}	0.82 (5)	2.21 (5)	3.016 (5)	165 (6)
N(5)–H(9)--O(1)	1.11 (6)	2.14 (5)	3.090 (6)	141 (4)
N(5)–H(10)—O(7)^{iv}	1.16 (5)	2.08 (5)	3.090 (6)	126 (4)
N(6)–H(11)—O(5)^{v}	0.91 (5)	2.07 (5)	2.952 (6)	164 (5)

[a] Symmetry transformations: (i) $x, y, z-1$; (ii) $x-{}^1/_2, 1+{}^1/_2-y, {}^1/_2+z$; (iii) $1+x, y, z-1$; (iv) $1+x, y, z$; (v) $1-x, 1-y, 1-z$.

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Registry No. $UO_2(OC(NH_2)_2)_3SO_4$, 72318-07-1.

Supplementary Material Available: Listings of structure factor amplitudes and atomic thermal parameters (13 pages). Ordering information is given on any current masthead page.

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Contribution from the Central Research and Development
Department, E. I. du Pont de Nemours and Company,
Experimental Station, Wilmington, Delaware 19898

Some Bis(*tert*-butylimido) Complexes of the Group 6 Transition Metals and a Related Alkylamido Derivative

William A. Nugent* and Richard L. Harlow

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Recently we reported[1] the preparation of four-coordinate d^0 organoimido complexes of the group 4 and 5 transition metals and of rhenium. We now report the extension of our synthetic studies to the group 6 metals. A number of group 6 organoimido derivatives are known[2] although only one example of a four-coordinate d^0 complex has been reported.[3]

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(2) For a review of organoimido complexes and their chemistry see: Nugent, W. A.; Haymore, B. L. *Coord. Chem. Rev.* in press.

(3) One of the products of the reaction of lithium diethylamide with WCl_6 has been identified as $(EtN)_2W(NEt_2)_2$: Bradley, D. C.; Chisholm, M. H.; Extine, M. W. *Inorg. Chem.* **1977**, *16*, 1791.

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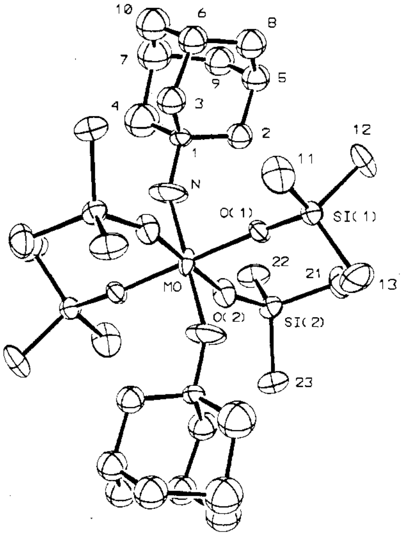

Figure 1. Structure of bis(1-adamantylamido)tetrakis((trimethylsilyl)oxo)molybdenum.

Tungsten hexachloride reacted violently with *tert*-butylamine in methylene chloride and no discrete products could be isolated. However, in hexane, where WCl_6 has limited solubility, its reaction with excess *tert*-butylamine proceeds according to eq 1. Hexane-soluble I is readily separated from

$$WCl_6 + 10t\text{-}BuNH_2 \rightarrow \underset{\text{I}}{(t\text{-}BuN)_2W(NH\text{-}t\text{-}Bu)_2} + 6t\text{-}BuNH_3Cl \quad (1)$$

the insoluble *tert*-butylammonium chloride. Recrystallization from toluene at −40 °C affords white cubes of I. Protonolysis of I as in eq 2 affords additionally IIa as a yellow distillable oil and IIb as colorless crystals.

$$I + 2ROH \rightarrow (t\text{-}BuN)_2W(OR)_2 + 2t\text{-}BuNH_2 \quad (2)$$
$$\text{IIa, R} = t\text{-Bu}$$
$$\text{IIb, R} = SiPh_3$$

When the dioxydichlorides of chromium or molybdenum were treated with *tert*-butyl(trimethylsilyl)amine[4] in refluxing hexane, the crystalline complexes III could be isolated in good yield. Although the insoluble products of this reaction were not characterized, the formation of products III can be rationalized by eq 3. Excess t-BuNH($SiMe_3$) appears to be

$$MCl_2O_2 + 4t\text{-}BuNH(SiMe_3) \rightarrow (t\text{-}BuN)_2M(OSiMe_3)_2 + 2t\text{-}BuNH_2(SiMe_3)Cl \quad (3)$$
$$\text{IIIa, M} = Cr$$
$$\text{IIIb, M} = Mo$$

required for complete reaction in the case of IIIa. The dioxydichlorides of tungsten or uranium do not react with t-BuNH($SiMe_3$).

The reaction of MoO_2Cl_2 with *N*-(1-adamantyl)(trimethylsilyl)amine did not afford the corresponding adamantylimido compound. Instead, it gave an alkylamido complex *trans*-$(AdNH)_2Mo(OSiMe_3)_4$, the structure of which was determined by X-ray crystallography (Figure 1). The molecule exhibits nearly perfect octahedral coordination with the molybdenum atom occupying a center of symmetry. Important bond distances and angles are shown in the Table I. It is noteworthy, that the Mo–N distances in this molecule are an average 0.15 Å longer than the Mo–O bonds. This contrasts with ca. 0.05 Å difference expected[5] when the bond orders to nitrogen and oxygen are the same. Another striking feature is the large Mo–N–C angle [150.8 (3)°] which may reflect steric factors or possibly hydrogen bonding of the amide hydrogen atom to oxygen.[6] Unfortunately, the twofold disorder in the adamantyl groups precludes further refinement of the structure to locate the amide hydrogen atoms.

Table I. Selected Bond Distances and Angles for Compound III with Estimated Standard Deviations

Bond Distances (Å)

Mo-O(1)	1.907 (2)	N-C(1)	1.446 (4)
Mo-O(2)	1.904 (2)	C(1)-C(2)	1.517 (6)
Mo-N	2.057 (3)	C(1)-C(3)	1.474 (6)
Si(1)-O(1)	1.630 (2)	C(1)-C(4)	1.584 (9)
Si(1)-C(11)	1.887 (4)	C(1)-C(2)P	1.528 (14)
Si(1)-C(12)	1.902 (4)	C(1)-C(3)P	1.490 (11)
Si(1)-C(13)	1.862 (4)	C(1)-C(4)P	1.621 (11)
Si(2)-O(2)	1.607 (2)		
Si(2)-C(21)	1.875 (4)		
Si(2)-C(22)	1.895 (4)		
Si(2)-C(23)	1.886 (3)		

Bond Angles (Deg)

O(1)-Mo-O(2)	90.1 (1)	O(1)-Si(1)-C(11)	109.8 (2)
O(1)-Mo-N	92.8 (1)	O(1)-Si(1)-C(12)	109.2 (2)
O(1)-Mo-O(2)	89.9 (1)	O(1)-Si(1)-C(13)	111.7 (2)
O(1)-Mo-N′	87.2 (1)	O(2)-Si(2)-C(21)	109.2 (1)
O(2)-Mo-N	89.9 (1)	O(2)-Si(2)-C(22)	110.5 (2)
O(2)-Mo-N′	90.2 (1)	O(2)-Si(2)-C(23)	109.5 (2)
Mo-O(1)-Si(1)	156.2 (1)	N-C(1)-C(2)	109.2 (3)
Mo-O(2)-Si(2)	161.0 (1)	N-C(1)-C(3)	106.1 (3)
Mo-N-C(1)	150.8 (3)	N-C(1)-C(4)	112.7 (4)
		N-C(1)-C(2)P	102.8 (6)
		N-C(1)-C(3)P	103.9 (5)
		N-C(1)-C(4)P	112.4 (5)

Experimental Section

Materials. Hexane, toluene, and *tert*-butylamine were sparged with nitrogen and dried over 5A molecular sieves prior to use. All reactions and subsequent manipulations were carried out under dry nitrogen.

Preparation of Complexes.[7] Bis(*tert*-butylimido)bis(*tert*-butylamido)tungsten, I, was prepared by suspending 10.0 g of WCl_6 in a solution of 30 mL of *tert*-butylamine and 200 mL of hexane. After 48 h,[8] the solution was filtered and the solvent and unreacted t-$BuNH_2$ were distilled off to afford 6.7 g (57%) of crude I as a yellow crystalline solid. Recrystallization from toluene (2 mL/g of I) at −40 °C gave white crystals: mp 89–90 °C; NMR (toluene-d_8) δ 1.25 (s, 18 H, N-t-Bu), 1.40 (s, 18 H, NH-t-Bu), 5.0–5.3 (br s, 2 H, NH).

Bis(*tert*-butylimido)bis(*tert*-butyloxo)tungsten, IIa, was prepared by stirring 3.69 g of I with 1.16 g of *tert*-butyl alcohol in 15 mL of hexane (1 h, 25 °C). After removal of the solvent, the resultant yellow oil was purified by fractional distillation (60–72 °C (0.1 torr)), a total of 1.9 g (51%) of product being collected. Lower boiling fractions were essentially pure IIa: NMR (toluene-d_8) δ 1.35 (s, 18 H, N-t-Bu), 1.42 (s, 18 H, O-t-Bu). Higher boiling fractions were contaminated by a second material, presumably (t-BuO)$_4$W(N-t-Bu), indicated by up to 10% of an additional NMR singlet at δ 1.50.

Bis(*tert*-butylimido)bis((triphenylsilyl)oxo)tungsten, IIb, was obtained by stirring 2.86 g of I and 3.37 g of triphenylsilanol in 50 mL of hexane overnight at room temperature. The solution was filtered and the solvent was distilled off, leaving a white solid which was washed by stirring with 10 mL of hexamethyldisiloxane. Crude IIb (1.62 g, 30%) was collected by filtration. This material was recrystallized from 20 mL of boiling hexamethyldisiloxane as feathers of colorless needles: mp 122–123 °C; NMR (toluene-d_8) δ 1.17 (s, 18 H, t-Bu), 6.9–7.9 (m, 30 H, aromatic).

Bis(*tert*-butylimido)bis((trimethylsilyl)oxo)chromium, IIIa, was prepared by refluxing a mixture of 3.75 g of CrO_2Cl_2, 18.0 g of t-BuNH($SiMe_3$), and 75 mL of hexane for 0.5 h. Filtration of the cooled solution followed by distillation of the solvent affords 7.1 g (79%) of crude IIIa. This material was dissolved in 25 mL of hexamethyldisiloxane and cooled to −40 °C to afford 4.83 g (54% overall yield) of deep red crystals: mp 74–76 °C; NMR (toluene-d_8) δ 0.23

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[7] All new complexes gave satisfactory elemental analysis (C, H, N).

[8] Yields were lower at shorter reaction times.

(s, 18 H, $SiMe_3$), 1.34 (s, 18 H, *t*-Bu).

Bis(*tert*-butylimido)bis((trimethylsilyl)oxo)molybdenum, IIIb, was prepared analogously from 7.75 g of MoO_2Cl_2 and 26.0 mL (20.2 g) of *t*-$BuNH(SiMe_3)$ in 150 mL of hexane. The IIIb compound obtained on removal of the solvent (15.06 g, 93%) is apparently analytically pure but can be additionally sublimed in vacuo or recrystallized (hexamethyldisiloxane, −40 °C): mp 42–43 °C; NMR (toluene-d_8) δ 0.20 (s, 18 H, $SiMe_3$), 1.37 (s, 18 H, *t*-Bu).

trans-Bis(1-adamantylamido)tetrakis(trimethylsilyl)oxo)molybdenum was prepared by refluxing 1.0 g of MoO_2Cl_2 with 4.5 g of (1-adamantyl)(trimethylsilyl)amine in 25 mL of hexane for 4.5 h. The solvent was removed to afford a waxy solid which was redissolved in 20 mL of toluene. Cooling this solution to −40 °C yielded 0.75 g of pale yellow cubes of the compound suitable for X-ray diffraction.

Structural Details. Data were collected on a Syntex P3 diffractometer with the crystal cooled to −55 °C (graphite monochromator; Mo Kα radiation; $\lambda = 0.71069$ Å). Preliminary examination indicated the crystal to be monoclinic with space group $P2_1/c$. The unit-cell dimensions were refined from the Bragg angles (as determined by the Syntex centering routine) of 48 reflections: $a = 10.533$ (2) Å, $b = 18.217$ (3) Å, $c = 10.817$ (1) Å, $\beta = 103.08$ (1)°. The cell volume, 2022 Å^3, yields a calculated density of 1.240 g cm^{-3} for $Z = 2$.

Intensity data for 3553 reflections were collected by using the ω-scan technique ($4° < 2\theta < 55°$; scan width of 0.8°; variable scan rate of 2.0–5.0° min^{-1}; background measurements at both ends of the scan; total background time equal to scan time). The intensities of four standard reflections were monitored after every 200 reflections; only statistical fluctuations were noted. The intensity of one reflection was measured in 10° increments about the diffraction vector; no variation in the intensity was observed. No absorption correction was applied ($\mu = 0.78$ cm^{-1}).

Registry No. I, 72207-45-5; IIa, 72207-46-6; IIb, 72207-47-7; IIIa, 71851-94-0; IIIb, 71851-95-1; *trans*-$(AdNH)_2Mo(OSiMe_3)_4$, 72207-48-8; WCl_6, 13283-01-7; $(t\text{-}BuO)_4W(N\text{-}t\text{-}Bu)$, 72207-49-9; CrO_2Cl_2, 7791-14-2; MoO_2Cl_2, 13637-68-8; *t*-$BuNH(SiMe_3)$, 5577-67-3; (1-adamantyl)(trimethylsilyl)amine, 36960-58-4.

Supplementary Material Available: Tables of final positional parameters (as fractional coordinates), thermal parameters, and structure factor amplitudes (observed and calculated) (29 pages). Ordering information is given on any current masthead page.

Contribution from the Department of Chemistry,
University of Missouri—St. Louis, St. Louis, Missouri 63121

Photosubstitution of Cyclopentadienylvanadium Tetracarbonyl

Donna G. Alway and Kenneth W. Barnett*[1]

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The lability of $CpV(CO)_4$ ($Cp = \eta^5\text{-}C_5H_5$) toward photosubstitution is well documented. A variety of diene,[2] acetylene,[3–5] and phosphine[5–10] derivatives have been prepared by irradiation of $CpV(CO)_4$ with the appropriate substrate. Photoreactions of $CpV(CO)_4$ with the monodentate phosphines PPh_3,[5,6] PBu_3,[7] or PH_3[8] (L) yield monosubstituted complexes $CpV(CO)_3(L)$, while reactions with $Ph_2P(CH_2)_nPPh_2$[9] (n = 1, 2, 4) or $PhP(CH_2CH_2PPh_2)_2$[10] (L_2) give the disubstituted complexes *cis*-$CpV(CO)_2L_2$. Photolysis of $CpV(CO)_4$ in tetrahydrofuran solutions containing X^- (X = Cl, Br, I) or BH_4^- is reported to yield $CpV(CO)_3X^-$ or mixtures of $CpV(CO)_3H^-$ and $CpV(CO)_2BH_4^-$, respectively.[6] Similarly, photolysis of tetrahydrofuran solutions containing $CpV(CO)_4$ and $CpV(CO)_3H^-$ gives the hydrogen-bridged dimeric complex, $[CpV(CO)_3]_2H^-$.[6]

The reactions cited above were promoted by irradiation of $CpV(CO)_4$ with unfiltered, high-intensity ultraviolet light. In some cases the photolyses were conducted for prolonged periods of time and/or at elevated temperatures. Although of synthetic value, such reactions provide little information about the excited-state decay processes available to the complex. We were prompted, therefore, to examine the photochemistry of $CpV(CO)_4$, using monochromatic irradiation.

Experimental Section

Materials. $CpV(CO)_4$ was purchased from Strem Chemicals, Inc., and recrystallized from chloroform/hexane prior to use. PPh_3 was obtained from Eastman Kodak Co. and used as received. High-purity carbon monoxide was purchased from Matheson Gas Products. Spectroscopic grade benzene was used after drying over molecular sieves. ^{13}CO (90%) was purchased from the Monsanto Chemical Co.

Spectra. Infrared spectra were recorded with Perkin-Elmer 337 or 521 grating spectrophotometers using matched 0.1- or 1.0-mm KBr solution cells. Proton NMR and UV–visible spectra were obtained with Varian T-60 and Beckman Acta MVI spectrophotometers, respectively.

Photolysis Procedures. All operations, except for the actual irradiations, were performed in the dark. Solutions of $CpV(CO)_4$ (5×10^{-4} M, 366 nm; 1×10^{-3} M, 436 nm) in 10-mL aliquots were placed in matched 10-mm i.d. Pyrex test tubes. The samples were deoxygenated by purging with prepurified argon for at least 10 min. The tubes were placed in a merry-go-round apparatus[11] of our own design. A 200-W Hanovia medium-pressure mercury-arc lamp was situated in an immersion well surrounded by two concentric cylindrical Pyrex jackets containing appropriate aqueous filter solutions to isolate the desired mercury emissions (336 nm: 0.125 M $NiSO_4$, 0.50 M $CoSO_4$ (l = 20 mm), and 6.4×10^{-4} M 2,7-dimethyl-3,6-diazacyclohepta-1,6-diene perchlorate (l = 10 mm); 436 nm: 5.4 M $NaNO_2$ (l = 20 mm) and 0.18 M $CuSO_4$, 2.7 M NH_3 (l = 10 mm)). Light intensities (366 nm, 4.4×10^{-8} einstein/s; 436 nm, 5.5×10^{-8} einstein/s) were measured by ferrioxalate actinometry.[12] The photoproduct, $CpV(CO)_3(PPh_3)$, was characterized by infrared, proton NMR, and UV–visible spectroscopy and was identified by comparison to the spectra of an authentic sample. Reactions of $CpV(CO)_4$ with PPh_3 were analyzed quantitatively by proton NMR spectroscopy. Following irradiation, the solvent was removed from the reaction solution by rotary evaporation at room temperature or below and the remaining materials were dissolved in deuteriochloroform for NMR analysis. The percentage yield of the product was determined by integration of the relative intensities of the cyclopentadienyl proton resonances of the complexes present. Quantum yields (corrected for incomplete absorption by $CpV(CO)_4$ samples) are reported as the average of triplicate determinations. Conversions were limited to 15% or less and were linear with irradiation time. Thermal dark reactions were monitored and were negligible within the times required for irradiation and analysis.

Synthesis of $CpV(CO)_3(PPh_3)$. $CpV(CO)_4$ (1.14 g, 5.0 mmol) and PPh_3 (1.31 g, 5.0 mmol) were dissolved in 450 mL of benzene. The orange solution was photolyzed with a water-cooled 200-W Hanovia medium-pressure mercury-arc lamp for 1 h. The volume of the solution was reduced to ~20 mL by rotary evaporation. Chromatography

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[1] To whom correspondence should be addressed at Ashland Chemical Co. Research and Development Department, P.O. Box 2219, Columbus, Ohio 43216.

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