sample of $Re_2(^{13}CO)_{10}$ containing 99.9% ^{13}C was prepared and converted to $Re_2(^{13}CO)_9(MeCN)$ by treatment with Me_3NO in MeCN solvent.[12] This compound was mixed with an approximately equimolar amount of unlabeled **1**, and the mixture was then converted to **2a** by reaction with $HC{\equiv}CCO_2Me$ in the above-described manner. An analysis of the product **2a** by mass spectrometry showed ions due only to the presence of **2a** without ^{13}C enrichment and **2a** having 99.9% ^{13}C. There were no significant amounts of the ions attributable to the crossover products $Re(^{13}CO)_4[\mu\text{-}HC{=}C(CO_2Me)]Re(CO)_5$ and $Re(CO)_4[\mu\text{-}HC{=}C(CO_2Me)]Re(^{13}CO)_5$. Also, no inhibition in the reaction rate or yield was found when the reaction was performed in the presence of the radical scavengers O_2 (air), 2,6-di-*tert*-butylphenol, and duroquinone in a 30-fold excess. The combination of evidence is most consistent with an *intramolecular* insertion mechanism.[12]

Theoretical studies of dimetalated olefins have indicated that the cis/trans isomerization process (C to D) has a high activation energy and thus would occur very slowly under the conditions that we have used.[4] Accordingly, we propose the mechanism shown in Scheme I, which does not require traversing an intermediate, such as C. The first step is a simple substitution of the MeCN ligand and coordination of the C–C triple bond to one of the rhenium atoms (E). At some point an interaction must develop between the second rhenium atom and at least one of the acetylenic carbon atoms of the ligand. We have chosen the carboxylate-substituted carbon atom for this interaction. As this interaction develops F, the rhenium–rhenium bond will develop a heteropolar (donor/acceptor) character, and in the limit (G), a μ-$\|$ alkyne ligand of the type B would bridge a full heteropolar rhenium–rhenium bond. At some point the metal–metal bond must be cleaved. From G this could be as simple as withdrawal of the shared electron pair to the donating $Re(CO)_5$ grouping. To maintain an 18-electron configuration, the $Re(CO)_4$ grouping might then engage two additional electrons from the alkynyl group. This could be achieved through a 90° twist of the MeO_2C–C–$Re(CO)_5$ grouping to form an η^2-alkenyl ligand serving as a three-electron donor (H). Numerous examples of η^2-alkenyl ligands acting as three-electron donors have been characterized recently, although we are not aware of any examples having one of the substituents as a metal-containing group.[13] The formation of **2** is completed by coordination of one of the lone pairs of electrons of the ketonic oxygen atom of the carboxylate group to the $Re(CO)_4$ grouping, a release of two electrons to the η^2-alkenyl group, and a further rotation of the MeO_2C–C–$Re(CO)_5$ group to produce the trans stereochemistry. In all of the intermediates 18-electron configurations are maintained at both metal atoms.

This reaction appears to be the first example of an intramolecular insertion reaction of an alkyne into a metal–metal bond that produces a trans stereochemistry. The proposed mechanism which does not traverse the intermediate C, having cis stereochemistry, could also be operative for the insertion of alkynes into metal–ligand bonds where trans stereochemistry has been observed.[14]

Acknowledgment. This research was supported by the Office of Basic Energy Science of the U.S. Department of Energy.

Supplementary Material Available: Text giving the procedure for the synthesis of $Re_2(^{13}CO)_{10}$, the mass spectrum of the $Re_2(^{13}CO)_{10}$ so obtained, the mass spectrum of the parent ion of **2a** obtained from the crossover experiment, and tables of crystal data, positional parameters, bond distances and angles, and anisotropic thermal parameters for **2a** (15 pages). Ordering information is given on any current masthead page.

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(12) No inhibition in the reaction rate or yield was found when the reaction was performed in the presence of the radical scavengers, duroquinone and 2,6-di-*tert*-butylphenol, but it must be noted that this fact and the lack of crossover do not rule out the possibility of dissociative mechanisms in which the fragments remain proximate to one another in tight solvent cages.

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Metal Transfer and Double Acyl Formation in the Reaction of ((Trimethylsilyl)ethynyl)(Fischer carbene)metal Complexes with $Co_2(CO)_8$

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Summary: A new cobalt carbene complex was obtained and fully characterized in the reaction of ((trimethylsilyl)ethynyl)((diallylamino)carbene)pentacarbonyltungsten with dicobalt octacarbonyl. This complex may be regarded as the consequence of a different evolution of the Pauson–Khand reaction.

Pentacarbonyl(carbene)metal complexes (Fischer complexes) undergo efficient metal-induced cycloadditions.[1] Recently, we reported that alkynyl (allylamino)carbene

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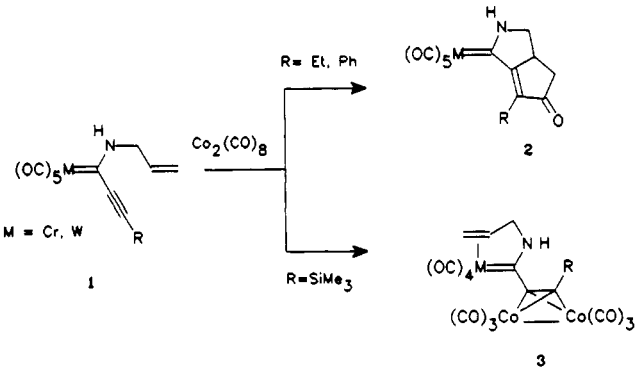
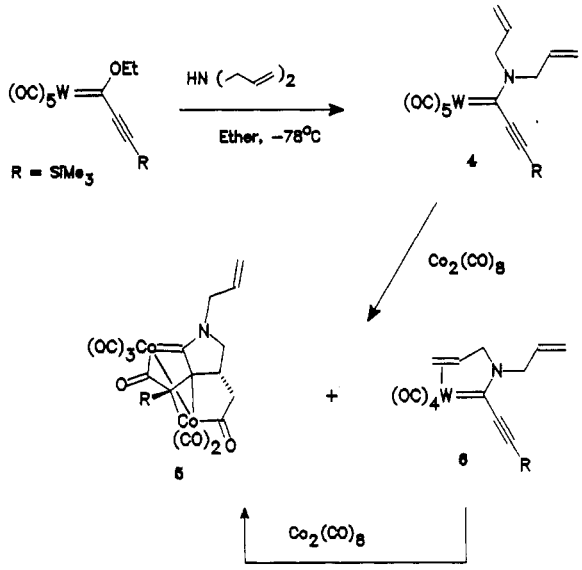
Scheme I



Scheme II



complexes undergo the Pauson–Khand reaction under very mild conditions.[2] However, the reaction failed to give the internal cycloadduct when a large substituent such as trimethylsilyl was attached to the alkyne moiety. In this case, due probably to steric reasons, the interaction of the olefin with tungsten proved to compete advantageously with that of the coordinated alkyne. As a result, complex **3** was obtained in high yields instead of **2**. All simple attempts (change of solvent, higher temperatures, further addition of $Co_2(CO)_8$, ...) to force **3** to undergo carbonylative cycloaddition were unsuccessful.

We envisaged the circumvention of this drawback by placement of an additional allyl group on the heteroatom, as in complex **4**. Thus, although coordination of the triple bond to $Co_2(CO)_8$ would lead one of the allyl groups to coordinate to the tungsten, the remaining one might still interact with the coordinated alkyne to accomplish the Pauson–Khand reaction similarly to that reported for other type of cycloadditions.[3]

When complex **4**[4] was treated with $Co_2(CO)_8$ (1.1 equiv) in THF at room temperature for 2 days, the new complex

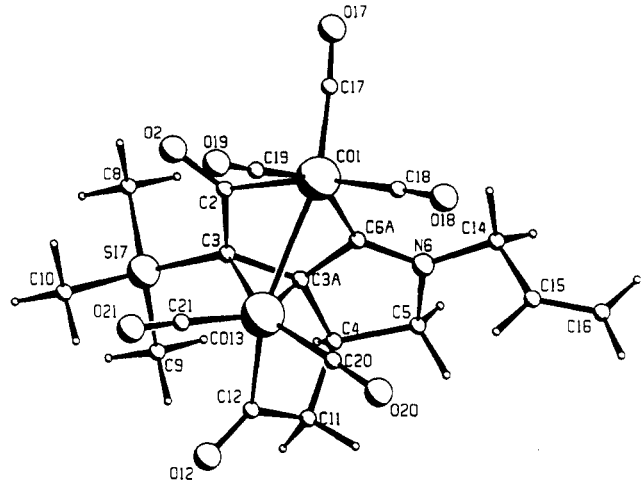


Figure 1. Molecular structure of **5**. Selected bond lengths (Å): Co(1)–Co(13), 2.645 (8); Co(1)–C(2), 2.037 (4); C(2)–C(3), 1.472 (5); C(3)–C(3a), 1.419 (5); C(3a)–C(6a), 1.435 (5); C(6a)–Co(1), 1.929 (4); C(6a)–N(6), 1.331 (5); C(3)–Co(13), 2.079 (4); C(3a)–Co(13), 2.018 (4).

5 could be isolated after flash chromatography (hexane/*tert*-butyl methyl ether, 3/2) in 48% yield.

A second product was also isolated, although it was too unstable to be characterized (no reliable ^{1}H NMR spectrum could be recorded). This product, after it stood in hexane solution for 1 day and was filtered and separated by flash chromatography, afforded **5** (corresponding to a 58% overall yield) and the tetracarbonyl complex **6**.[5] In an independent experiment, **6** was allowed to react with $Co_2(CO)_8$ in THF. After 24 h the solvent was evaporated and the residue chromatographed on silica, affording more **5** (48%).

From the spectroscopic data of **5**[6] it was concluded that the tungsten had been replaced by cobalt at the carbene center and one of the allyl groups had been inserted into the alkyne. However, since no satisfactory structure could be worked out from the data, a single-crystal X-ray diffractometry structure determination was performed. The results are shown in Figure 1.[7]

The structure of this complex consisted of a metallacyclopentene unit sharing an edge with an azacyclopentane unit, in an almost planar fashion. The C(4) atom was

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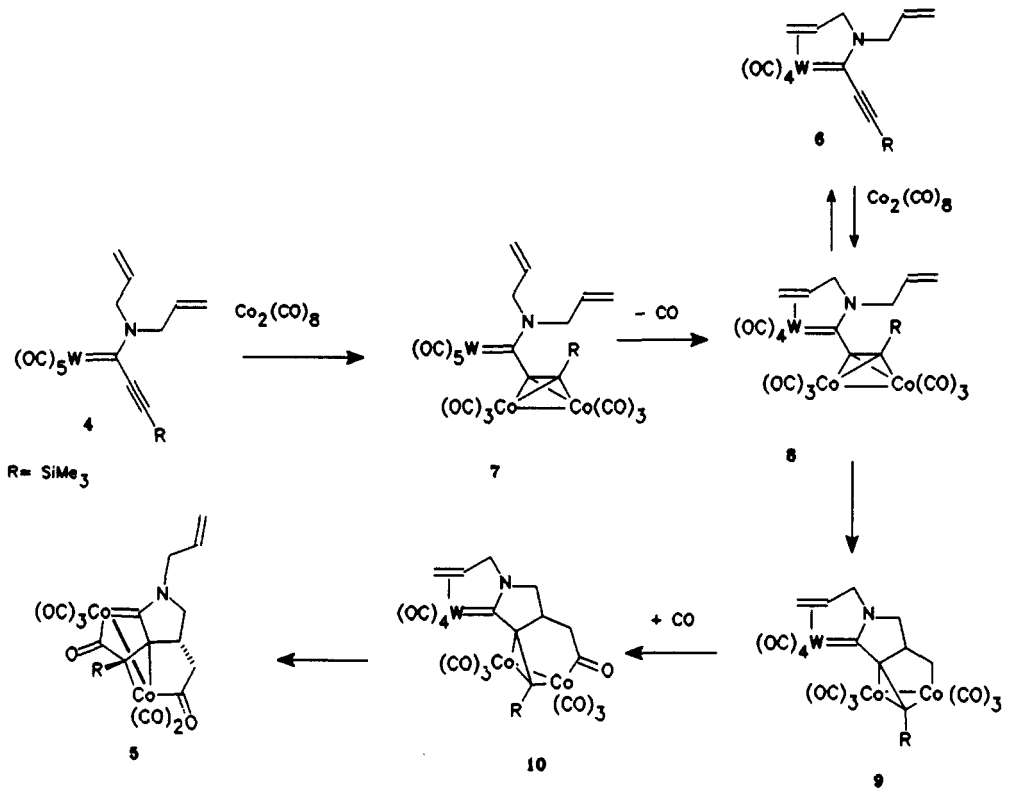
(4) Although the (diallylamino)carbene complexes of this type are usually not available by aminolysis of the corresponding alkoxy complexes because of the preferred conjugate addition (Fischer, E. O.; Kalder, H. J. *J. Organomet. Chem.* **1977**, *131*, 57), we have been able to prepare **4** by this method since, probably due to steric protection of the triple bond by the Me_3Si group, attack at the carbene center becomes preferred.

(5) Anal. Calcd for **6** ($C_{19}H_{19}NO_4SiW$): C, 38.33; H, 3.83; N, 2.79. Found: C, 38.48; H, 3.81; N, 2.87. IR (CCl_4): ν 2125, 2020, 1945, 1915, 1892 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.25 (s, 9 H), 3.18 (bd, 1 H, J = 12.3 Hz), 3.27 (bd, 1 H, J = 9.3 Hz), 3.82 (m, 1 H), 4.30 (AB s dt, 2 H, J_{AB} = 14.7 Hz, J_d = 6.3 Hz, J_t = 1.2 Hz), 4.45 (m, 2 H), 5.28 (dd, 1 H, J = 16.8, 1.2 Hz), 5.29 (dd, 1 H, J = 10.2, 1.2 Hz), 5.75 (ddt, 1 H, J = 16.8, 10.2, 6.3 Hz). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ 0.5 (q), 58.8 (t), 59.05 (t), 61.3 (t), 70.9 (d), 104.5 (s), 120.3 (t), 130.8 (d), 131.6 (s), 203.2 (s), 203.5 (s), 210.5 (s), 211.8 (s), carbene carbon not found.

(6) Anal. Calcd for **5** ($C_{19}Co_2H_{19}NO_7Si$): C, 43.91; H, 3.70; N, 2.70. Found: C, 43.48; H, 3.70; N, 2.66. IR ($CHCl_3$): ν 2080, 2040, 2020, 1700, 1630 cm^{-1}. ^{1}H NMR ($CDCl_3$, 300 MHz): δ 0.20 (s, 9 H), 2.85 (dd, 1 H, J = 16.8, 3.3 Hz), 3.12 (m, 1 H), 3.45 (dd, 1 H, J = 16.8, 8.0 Hz), 3.66 (d, 1 H, J = 11.1 Hz), 4.20 (d, 2 H, J = 5.7 Hz), 4.59 (dd, 1 H, J = 11.1, 7.2 Hz), 5.41 (m, 2 H), 5.93 (m, 1 H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ 0.5 (q), 30.8 (d), 56.0 (t), 72.1 (t), 74.0 (t), 74.7 (s), 120.7 (t), 121.5 (s), 131.2 (d), 200.0 (bs), 218.0 (s), 243.8 (s). MS (FAB$^+$, matrix NBA): m/e 520 (M$^+$), 491, 463, 435, 407, 379, 351, 323.

(7) Crystal data: $C_{19}Co_2H_{19}NO_7Si$; M_r = 519.32, space group $P2_1/a$ (No. 14); a = 13.547 (6) Å, b = 8.990 (3) Å, c = 18.101 (6) Å, β = 95.99 (4)°; V = 2192 (9) Å^3, Z = 4; D_c = 1.573 g cm^{-3}; radiation Mo Kα (λ = 0.71073 Å); μ = 16.0 cm^{-1}; $F(000)$ = 1056; T = 21 ± 1 °C; final R = 0.062; number of unique reflections 3344. A single crystal was mounted at the end of a glass fiber and centered in the Enraf-Nonius CAD4 diffractometer geometry. Cell parameters were determined by least-squares fitting of 25 high-angle reflections. Lorentz and polarization effects were corrected, but not absorption. The structure was solved by application of an automated Patterson search (ROTSEARCH). Refinement was carried out using full-matrix least-squares methods (SHELX-76).

Scheme III



linked to a CH_2–C(O)–$Co(CO)_2$ unit situated below the bicyclic moiety. The two cobalt atoms, directly bonded to each other, had distorted-octahedral environments. The Co–Co distance of 2.654 Å was rather long but was within the range observed for other nonbridged dinuclear cobalt complexes.[8] The olefinic part of the metallacycle was η^2-bonded to the $Co(CO)_2$ unit with Co(13)–C(3) and Co(13)–C(3a) distances of 2.079 and 2.015 Å, respectively. The Co(1)–C(6a) bond was 1.929 Å long, a value consistent with a Co–C(carbene) distance.[9] Furthermore, the C(6a)–N(6) distance was 1.331 (5) Å, a characteristic value for aminocarbene complexes.[10]

The formal electron count on the metal atoms gave 19 electrons for Co(1) and 17 for Co(13). The diamagnetism observed in complex 3 may be accounted for by assuming the existence of back-donation from Co(1) to Co(13), as has been proposed for other dinuclear cobalt complexes.[11]

Although, as mentioned, the second product obtained in the reaction was too unstable to be characterized, from its behavior we ascribed structure 8 to it.

To our knowledge, the $Co_2(CO)_6$ unit can be easily coordinated to the triple bond of alkynyl(carbene)metal complexes without any apparent loss of thermal stability;[12] only the presence of two allyl groups seems to confer instability to the resulting complex 8, which evolves to either the complex 5 or, alternatively, back to 6. Since no significant electronic differences between the very stable 3 and 8 may be anticipated, we conclude that steric factors are important in this reaction.

The formation of 5 may be regarded as a consequence of a different evolution of the Pauson–Khand reaction prior to reductive elimination of the first cobalt atom[13] (see Scheme III). In fact, the steric demands of the Me_3Si group may be thought to be the origin for the cobalt carbonyl moiety replacing the tungsten carbonyl at the carbene center. This change in steric characters would thwart the final reductive elimination required in the conventional cycloaddition (see Scheme III) since (a) the *tert*-butylethynyl analog behaves similarly (with lower yields)[14] and (b) similar complexes with less hindered substituents on the alkyne (either (allylamino)- or (diallylamino)carbene complexes) all give the carbonylative cycloaddition.[15]

Steps from intermediate complex 8 to the final product 5 are tentative, and certainly they represent a steric relief since one of the bulky metal carbonyls ($W(CO)_4$) has been lost. However, there is no evidence indicating at which precise point this may happen. Studies to clarify the possible mechanism are under way.

Although several cobalt carbene complexes are known,[9,16] to our knowledge this is the first example in which a carbene ligand is coordinated with a dicobalt diacyl unit, thus improving the stability of the carbene bond.

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and J.M.V. the CIRIT (Generalitat de Catalunya) for fellowships.

Supplementary Material Available: Tables of crystal data and refinement details, bond distances and angles, positional and thermal parameters, and torsion angles and packing diagrams for 5 (15 pages). Ordering information is given on any current masthead page.

OM920406N

Ring Opening of Strained-Ring Heterocycles Containing Nitrogen by an Osmium Cluster Complex

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Received December 23, 1991

Summary: The osmium cluster complex $Os_3(CO)_{10}(\mu\text{-}NCH_2CH_2CH_2)(\mu\text{-}H)$ (1) was obtained in 34% yield from the reaction of $Os_3(CO)_{10}(NCMe)_2$ with azetidine. Complex 1 was characterized crystallographically and shown to contain a four-membered $NCH_2CH_2CH_2$ ring bridging two of the metal atoms via the nitrogen atom. When heated to 125 °C, 1 was transformed to the new complex $Os_3(CO)_{10}[\mu\text{-}N{=}C(H)Et](\mu\text{-}H)$ (2; 27% yield) by an opening of the four-membered ring and a hydrogen shift. Crystal data: for 1, space group $P2_1/c$, a = 8.793 (2) Å, b = 16.265 (2) Å, c = 13.766 (4) Å, β = 110.56 (1)°, Z = 4, 1872 reflections, R = 0.039; for 2, space group $P\bar{1}$, a = 14.626 (3) Å, b = 22.961 (6) Å, c = 9.089 (2) Å, α = 92.73 (2)°, β = 99.03 (2)°, γ = 81.52 (2)°, Z = 6, 4862 reflections, R = 0.024.

The ring opening of nitrogen-containing heterocycles is an integral step in the process of hydrodenitrogenation.[1] The mechanism by which this step occurs is poorly understood, and a considerable amount of research has been devoted to modeling the heterogeneous reaction by using homogeneous catalysts.[2,3] Although it has considerable ring strain, the four-membered ring of azetidine, HNC_3H_6, is opened thermally only at temperatures in excess of 400 °C.[4] Recently, we have shown that the four-membered sulfur-containing heterocycles thietanes exhibit an enhanced tendency to ring-open when the sulfur atom is coordinated to certain metal cluster complexes in a bridging position.[5] We have now prepared the cluster complex $Os_3(CO)_{10}(\mu\text{-}NCH_2CH_2CH_2)(\mu\text{-}H)$ (1), which contains a four-membered heterocycle that bridges two of the metal atoms through its nitrogen atom, and have found that this heterocycle opens when the complex is heated to only 125 °C.

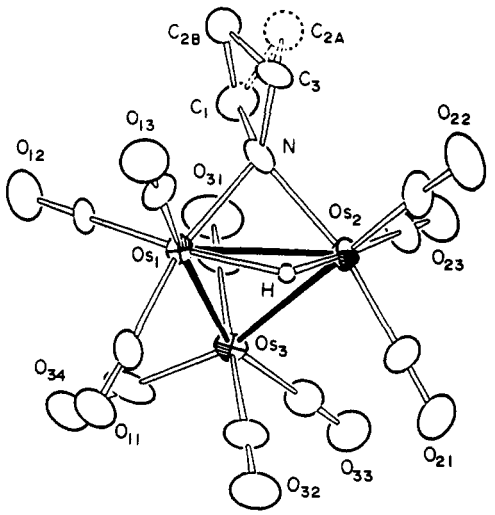

Figure 1. ORTEP diagram of $Os_3(CO)_{10}(\mu\text{-}NC_3H_6)(\mu\text{-}H)$ (1). Selected interatomic distances (Å) and angles (deg): Os(1)–N = 2.12 (2), Os(2)–N = 2.11 (2), N–C(1) = 1.59 (3), N–C(3) = 1.50 (2); C(1)–N–C(3) = 85 (2).

Complex 1 was prepared in 34% yield from the reaction of $Os_3(CO)_{10}(NCMe)_2$ with azetidine in CH_2Cl_2 solvent at reflux for 6 h.[6] It was characterized by a single-crystal X-ray diffraction analysis and was found to contain an azetidine ring system bridging two of the osmium atoms of a triangular cluster through its nitrogen atom (see Figure 1).[7,8] The azetidine ring is slightly puckered, the dihedral angle equals 24.5°, and the methylene group at the 3-position is disordered between two sites. As a result, the C–C bond lengths span a wide range (1.35 (6)–1.71 (5) Å) with large errors, but it is felt that the deviations from

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(6) A solution consisting of 100 mg of $Os_3(CO)_{10}(NCMe)_2$ (0.107 mmol) and 20 μL of azetidine in 80 mL of CH_2Cl_2 was heated to reflux for 6 h. The product was separated by TLC on silica gel using a CH_2Cl_2/hexane (1/9) solvent mixture to yield 32.5 mg of yellow $Os_3(CO)_{10}(\mu\text{-}NCH_2CH_2CH_2)(\mu\text{-}H)$ (1; 34%). IR (ν(CO), cm^{-1}; in hexane): 2103 (w), 2062 (vs), 2050 (m), 2019 (s), 2007 (m), 1997 (vw), 1986 (s), 1976 (w). ^{1}H NMR (ppm; in $CDCl_3$ at 25 °C): 4.43 (2 H, t, ^3J = 6.9 Hz), 3.99 (2 H, t, $^3J_{H-H}$ = 6.9 Hz), 2.53 (2 H, q, $^3J_{H-H}$ = 6.9 Hz), −14.00 (1 H, s). Anal. Calcd (found): C, 17.18 (17.19); H, 0.77 (0.67); N, 1.54 (1.50).

(7) Crystal data for 1: space group $P2_1/c$, a = 8.793 (2) Å, b = 16.265 (2) Å, c = 13.766 (4) Å, β = 110.56 (1)°, Z = 4, 1872 reflections, R = 0.039.

(8) Diffraction measurements at 20 °C were made on a Rigaku AFC6S four-circle diffractometer using Mo Kα radiation. The structure solution and refinement was made by using the TEXSAN structure-solving program library (v5.0) of the Molecular Structure Corp., The Woodlands, TX. An absorption correction was applied to the data.

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