

Synthesis and Molecular Structure of a Novel Monocarbon Hydridorhodacarborane: *closo*-2,7-(PPh₃)₂-2-H-2-Cl-1-(NMe₃)-2,1-RhCB₁₀H₉

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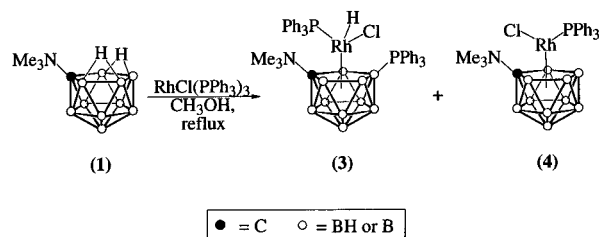
Introduction

Since the first demonstration of the exceptional efficacy of hydridometallacarboranes derived from the 7,8- and 7,9-isomers of *nido*-C₂B₉H₁₁²⁻ as homogeneous catalyst precursors,² significant advances have been achieved in the development of their unique chemistry.³ In addition, many related hydrido-metallacarboranes of the platinum group metals based upon a variety of carborane derivatives have been obtained.^{3c,4} We here report the synthesis and single-crystal X-ray diffraction study of a novel monocarbon hydridometallacarborane derived from *nido*-B₁₀H₁₂CNMe₃ (**1**), viz. *closo*-2,7-(PPh₃)₂-2-H-2-Cl-1-(NMe₃)-2,1-RhCB₁₀H₉ (**3**), which is the first structurally characterized mononuclear hydride complex with an icosahedral monocarbon carborane ligand.

Results and Discussion

The reaction of **1**⁵ with 1 molar equiv of Rh(PPh₃)₃Cl (**2**) in hot methanol produced **3** as a yellow precipitate in 72% yield, together with a small amount of a 16-electron rhodacarborane, *closo*-2-(PPh₃)₂-2-Cl-1-(NMe₃)-2,1-RhCB₁₀H₁₀ (**4**), which was isolated from the reduced mother liquor by column chromatography on silica gel (Scheme 1). The structure of **4** was

Scheme 1



deduced from spectroscopic and analytical data. The ³¹P{¹H} NMR spectrum of **4** exhibited one doublet at 37.3 ppm with *J*_{P-Rh} = 152 Hz due to the PPh₃ group attached to rhodium. Both the chemical shift and coupling constant values associated with this signal proved to be very similar to those observed in the ³¹P{¹H} NMR spectra of the previously described 16-electron Rh(III) mononuclear monocarbon *closo*-rhodacarboranes.⁶ The ¹H NMR spectrum of **4** revealed phenyl multiplets centered at 7.45 ppm as well as a singlet at 3.69 ppm, as expected for equivalent methyl groups in the N(CH₃)₃ cage substituent. The latter complex was obtained in reasonable yield when the reaction was terminated and the products were isolated after 3.5 h.⁷ The latter observation suggests **4** as one of the possible intermediates leading to the formation of **3**.

Complex **3** was obtained as an air-stable crystalline material, and its structure was elucidated as described below. The ³¹P{¹H} NMR spectrum of **3** in CH₂Cl₂ contained two resonances, a sharp doublet (*δ*_{Pa} = 26.2 ppm, *J*_{P-Rh} = 104.5 Hz) and a broadened signal (*δ*_{Pb} = 11.0 ppm, *J*_{P-B} ≈ 120 Hz) which resembled a quadruplet. These resonances were attributed to PPh₃ groups at rhodium and boron atoms, respectively. This was confirmed by the ¹¹B{¹H} NMR spectrum of **3** which revealed a unique sharp doublet at -7.8 ppm (*J*_{B-P} ≈ 127 Hz) originating from the boron atom substituted by a PPh₃ group within a set of broad resonances derived from the BH vertices of the carborane cage. The ¹H NMR spectrum of **3** in CD₂Cl₂ exhibited complex overlapping multiplets originating from both of the PPh₃ groups, a singlet at 2.92 ppm from equivalent methyl groups at the N(CH₃)₃ substituent, and a doublet of doublets of doublets at -9.63 ppm due to the terminal hydride at the rhodium atom. The multiplicity of the hydride resonance observed is consistent with the coupling of hydrogen to two nonequivalent PPh₃ groups (³¹P_a and ³¹P_b) and a ¹⁰³Rh nucleus. Selective decoupling of ³¹P_b nuclei caused a collapse of the hydride signal to a doublet of doublets with *J*_{H-Rh} = 11.0 Hz and *J*_{H-Pa} = 46 Hz. The small coupling constant of the ³¹P_b nucleus with the hydride at the Rh center (*J*_{H-P} ≈ 7 Hz) was elucidated from a second decoupling ¹H{³¹P_a} NMR experiment and gave additional confirmation of the nature of this PPh₃ group.

The solid state structure of complex **3** was determined by an X-ray diffraction study which proved this species to be a *closo*-hydridorhodacarborane (Figure 1) with quaternary nitrogen and phosphorus groups attached to a monocarbon carborane ligand

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- (2) (a) Paxson, T. E.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1974**, *96*, 4674. (b) Belmont, J. A.; Soto, J.; King, R. E., III; Donaldson, A. J.; Hewes, J. D.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1989**, *111*, 7475 and references therein.
- (3) (a) Kalb, W. C.; Demidowicz, Z.; Speckman, D. M.; Knobler, C. B.; Teller, R. G.; Hawthorne, M. F. *Inorg. Chem.* **1982**, *21*, 4027. (b) Long, J. A.; Marder, T. B.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1984**, *106*, 3004. (c) Doi, J. A.; Mizusawa, E. A.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1984**, *23*, 1482. (d) Long, J. A.; Marder, T. B.; Behnken, P. E.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1984**, *106*, 2979. (e) Walker, J. A.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1985**, *24*, 2688. (g) Zakharkin, L. I.; Chizhevsky, I. T.; Zhigareva, G. G.; Petrovskii, P. V.; Polyakov, A. V.; Yanovsky, A. I.; Struchkov, Yu. T. *J. Organomet. Chem.* **1988**, *358*, 449. (f) Chizhevsky, I. T. *Metalloorg. Khim.* **1991**, *4*, 1416.
- (4) (a) Wong, E. H. S.; Hawthorne, M. F. *Inorg. Chem.* **1978**, *17*, 2863. (b) Hewes, J. D.; Knobler, C. B.; Hawthorne, M. F. *J. Chem. Soc., Chem. Commun.* **1981**, 206. (c) Busby, D. C.; Hawthorne, M. F. *Inorg. Chem.* **1982**, *21*, 4101. (d) Delaney, M. S.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1981**, *20*, 1341. (e) Walker, J. A.; O'Con, C. A.; Zheng, L.; Knobler, C. B.; Hawthorne, M. F. *J. Chem. Soc., Chem. Commun.* **1983**, 803. (g) Hewes, J. D.; Thompson, M.; Hawthorne, M. F. *Organometallics* **1985**, *4*, 13. (f) Alcock, N. W.; Taylor, J. G.; Wallbridge, M. G. H. *J. Chem. Soc., Dalton Trans.* **1987**, 1805. (h) Nestor, K.; Fontaine, X. L. R.; Greenwood, N. N.; Kennedy, J. D.; Plecek, J.; Stibr, B.; Thornton-Pett, M. *Inorg. Chem.* **1989**, *28*, 2219.
- (5) Knoth, W. H.; Little, J. L.; Lawrence, J. R.; Scholer, F. R.; Todd, L. *J. Inorg. Synth.* **1968**, *11*, 35.

- (6) Chizhevsky, I. T.; Pisareva, I. V.; Petrovskii, P. V.; Bregadze, V. I.; Yanovsky, A. I.; Struchkov, Yu. T.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1993**, *32*, 3393.

- (7) In this case another new crystalline product was isolated from the reaction mixture by the procedure given in the Experimental Section. Although this compound was not available in sufficient quantities to allow complete characterization from spectroscopic data, it was concluded that the species can be regarded as a 12-vertex monocarbon rhodacarborane with PPh₃ (*δ*_P = 30.9 ppm, *J*_{P-Rh} = 164.2 Hz in CD₂-Cl₂) and NMe₃ (*δ*_H = 3.49 ppm in CD₂Cl₂) ligands at the rhodium and at the carbon cage atoms, respectively. No other attempts, however, were made to characterize this compound.

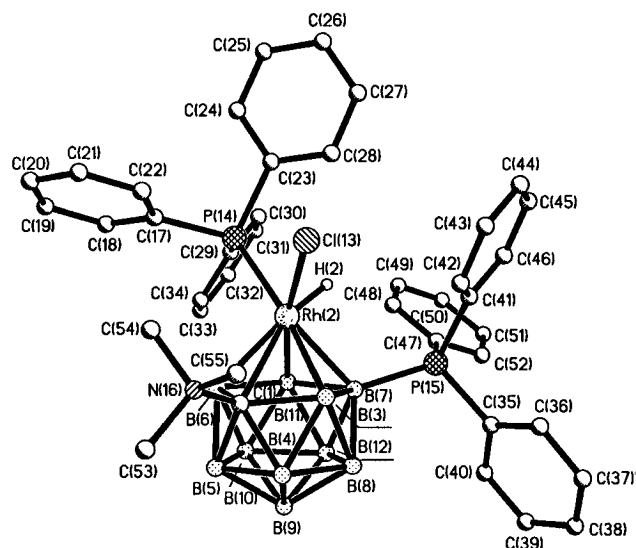


Figure 1. Molecular structure of complex 3. All hydrogen atoms except H(Rh) have been omitted for clarity. Selected interatomic distances (Å) and angles (deg): Rh(2)–B(3) = 2.195(4), Rh(2)–B(6) = 2.189(4), Rh(2)–B(7) = 2.209(4), Rh(2)–B(11) = 2.178(4), Rh(2)–C(1) = 2.343(3), Rh(2)–P(14) = 2.355(1), Rh(2)–Cl = 2.449(1), Rh(2)–H(2) = 1.41(7), C(1)–N(16) = 1.561(4), B(17)–P(15) = 1.958(4); P(14)–Rh(2)–Cl(13) = 88.49(4), P(14)–Rh(2)–H(2) = 70(3), Cl(13)–Rh(2)–H(2) = 93(3).

Table 1. Crystallographic Data for Complex 3

formula	C ₄₁ H ₅₁ B ₁₀ Cl ₃ NP ₂ Rh	V, Å ³	2225.2(7)
fw	937.11	Z	2
cryst syst	triclinic	d _{calcd} , g/cm ³	1.398
space group	P $\bar{1}$ (No. 2)	λ (Mo K α), Å	0.71073
a, Å	11.460(2)	μ (Mo K α), cm ⁻¹	6.7
b, Å	11.649(2)	temp, °C	20
c, Å	17.836(4)	R ₁ ^a	0.0352
α , deg	108.94(3)	wR ₂ ^b	0.1044
β , deg	94.57(3)	independent reflectn	7819
γ , deg	95.69(3)	obsd reflectn	5888 ($I > 2\sigma$)

^a $R_1 = \sum |F_o - |F_c|| / \sum (F_o)$ for observed reflections. ^b $wR_2 = (\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2])^{1/2}$ for all reflections.

and an 18-electron Rh center. The Rh(2)–C(1) distance of 2.343(3) Å, as in the previously studied monocarbon *closo*-2-Br-2-(PPh₃)-1-[(CH₂CH=CHCH₃)₂NH]-2,1-RhCB₁₀H₁₀,⁶ is somewhat longer than the Rh–B bonds [2.178(4)–2.209(4) Å]. The Rh(2)–P(14) and the Rh(2)–Cl bond lengths of 2.355(1) and 2.449(1) Å, respectively, are in the range of the distances which are usually observed for (phosphine)rhodacarboranes containing chlorine at the metal vertex.^{6,8} The B(7)–P(15) distance of 1.958(4) Å proved to be considerably longer than those observed in uncoordinated *nido*-9-PPh₃-7,8-C₂B₉H₁₁ [1.912(2) Å]⁹ or monocarbon *nido*-carborane 9-(PPh₃)-7-[(CH₃)₃Si]₂CH]CB₁₀H₁₁ [1.928(5) Å]¹⁰ with a PPh₃ substituent at the boron cage atoms.

One may assume that the attachment of the PPh₃ ligand to a carborane cage proceeds via an electronically unsaturated complex 4 which plays the role of an intermediate in the formation of 3 from 1 and 2. The same mechanism has previously been suggested for the formation of some boron-substituted (phosphine)rhoda-,⁸ (phosphine)ruthena-,¹¹ and (phos-

Table 2. Selected Bond Lengths (Å) and Angles (deg) for Complex 3

C(1)–N(16)	1.561(4)	B(6)–B(11)	1.884(6)
C(1)–B(4)	1.697(5)	B(7)–B(12)	1.767(6)
C(1)–B(6)	1.711(5)	B(7)–B(8)	1.777(6)
C(1)–B(5)	1.721(5)	B(7)–B(11)	1.817(6)
C(1)–B(3)	1.785(5)	B(7)–P(15)	1.958(4)
C(1)–Rh(2)	2.343(3)	B(8)–B(12)	1.784(6)
Rh(2)–B(11)	2.178(4)	B(8)–B(9)	1.784(6)
Rh(2)–B(6)	2.189(4)	B(9)–B(12)	1.765(6)
Rh(2)–B(3)	2.195(4)	B(9)–B(10)	1.792(6)
Rh(2)–B(7)	2.209(4)	B(10)–B(11)	1.777(6)
Rh(2)–P(14)	2.355(1)	B(10)–B(12)	1.781(6)
Rh(2)–Cl(13)	2.449(1)	B(11)–B(12)	1.786(6)
B(3)–B(8)	1.795(6)	P(14)–C(29)	1.836(4)
B(3)–B(7)	1.813(5)	P(14)–C(17)	1.838(4)
B(3)–B(4)	1.821(6)	P(14)–C(23)	1.842(4)
B(4)–B(5)	1.771(6)	P(15)–C(35)	1.817(4)
B(4)–B(9)	1.772(6)	P(15)–C(41)	1.817(4)
B(4)–B(8)	1.772(6)	P(15)–C(47)	1.824(4)
B(5)–B(9)	1.746(6)	N(16)–C(55)	1.500(5)
B(5)–B(10)	1.748(6)	N(16)–C(54)	1.501(5)
B(5)–B(6)	1.812(6)	N(16)–C(53)	1.517(5)
B(6)–B(10)	1.786(6)		
N(16)–C(1)–B(4)	112.7(3)	P(14)–Rh(2)–Cl(13)	88.49(4)
N(16)–C(1)–B(6)	123.0(3)	B(12)–B(7)–B(8)	60.5(2)
B(4)–C(1)–B(6)	113.9(3)	B(12)–B(7)–B(3)	107.3(3)
N(16)–C(1)–B(5)	114.8(3)	B(8)–B(7)–B(3)	60.0(2)
B(4)–C(1)–B(5)	62.4(2)	B(12)–B(7)–B(11)	59.8(2)
B(6)–C(1)–B(5)	63.7(2)	B(8)–B(7)–B(11)	108.4(3)
N(16)–C(1)–B(3)	121.2(3)	B(3)–B(7)–B(11)	106.7(3)
B(4)–C(1)–B(3)	63.0(2)	B(12)–B(7)–P(15)	113.5(2)
B(6)–C(1)–B(3)	108.4(3)	B(8)–B(7)–P(15)	114.6(2)
B(5)–C(1)–B(3)	112.3(3)	B(3)–B(7)–P(15)	126.8(2)
N(16)–C(1)–Rh(2)	116.4(2)	B(11)–B(7)–P(15)	122.8(2)
B(4)–C(1)–Rh(2)	119.6(2)	B(12)–B(7)–Rh(2)	118.2(2)
B(6)–C(1)–Rh(2)	63.1(2)	B(8)–B(7)–Rh(2)	119.4(2)
B(5)–C(1)–Rh(2)	119.9(2)	B(3)–B(7)–Rh(2)	65.3(2)
B(3)–C(1)–Rh(2)	62.5(2)	B(11)–B(7)–Rh(2)	64.6(2)
B(11)–Rh(2)–B(6)	51.1(2)	P(15)–B(7)–Rh(2)	118.0(2)
B(11)–Rh(2)–B(3)	83.5(2)	C(29)–P(14)–C(17)	103.4(2)
B(6)–Rh(2)–B(3)	80.6(2)	C(29)–P(14)–C(23)	102.8(2)
B(11)–Rh(2)–B(7)	48.9(2)	C(17)–P(14)–C(23)	100.0(2)
B(6)–Rh(2)–B(7)	83.9(2)	C(29)–P(14)–Rh(2)	113.80(12)
B(3)–Rh(2)–B(7)	48.62(14)	C(17)–P(14)–Rh(2)	120.17(12)
B(11)–Rh(2)–C(1)	80.87(14)	C(23)–P(14)–Rh(2)	114.33(13)
B(6)–Rh(2)–C(1)	44.21(14)	C(35)–P(15)–C(41)	107.2(2)
B(3)–Rh(2)–C(1)	46.19(13)	C(35)–P(15)–C(47)	104.8(2)
B(7)–Rh(2)–C(1)	80.24(13)	C(41)–P(15)–C(47)	102.8(2)
B(11)–Rh(2)–P(14)	98.67(12)	C(35)–P(15)–B(7)	111.5(2)
B(6)–Rh(2)–P(14)	94.28(12)	C(41)–P(15)–B(7)	114.8(2)
B(3)–Rh(2)–P(14)	171.42(10)	C(47)–P(15)–B(7)	114.8(2)
B(7)–Rh(2)–P(14)	138.10(10)	C(55)–N(16)–C(54)	107.9(3)
C(1)–Rh(2)–P(14)	125.79(9)	C(55)–N(16)–C(53)	106.7(3)
B(11)–Rh(2)–Cl(13)	170.03(11)	C(54)–N(16)–C(53)	106.9(4)
B(6)–Rh(2)–Cl(13)	135.66(11)	C(55)–N(16)–C(1)	110.7(3)
B(3)–Rh(2)–Cl(13)	90.39(11)	C(54)–N(16)–C(1)	110.3(3)
B(7)–Rh(2)–Cl(13)	121.37(11)	C(53)–N(16)–C(1)	114.1(3)
C(1)–Rh(2)–Cl(13)	100.61(9)		

phine)nickelacarboranes.¹² The reaction of 4 with 1 molar equiv of PPh₃ was carried out at reflux in methanol for 2 h to produce 3, whose structure was confirmed by its characteristic ¹H and ³¹P NMR spectra, in 68% yield. This result lends strong support to the assumption that 3 arises from the reaction of 4 with PPh₃.

Experimental Section

All experiments were performed under an atmosphere of dry argon in standard Schlenk equipment. Solvents were dried according to standard literature procedures¹³ prior to use. The ¹H, ³¹P, and ¹¹B NMR

(8) Baker, R. T.; Delaney, M. S.; King, R. E., III; Knobler, C. B.; Long, J. A.; Marder, T. B.; Paxson, T. E.; Teller, R. G.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1984**, *106*, 2965.

(9) Kim, K. M.; Do, Y.; Knobler, C. B.; Hawthorne, M. F. *Bull. Korean Chem. Soc.* **1989**, *10*, 321.

(10) Quintana, W.; Ernest, R. L.; Carroll, P. J.; Sneddon, L. G. *Organometallics* **1988**, *7*, 166.

(11) Jung, C. W.; Hawthorne, M. F. *J. Am. Chem. Soc.* **1980**, *102*, 3024.

(12) (a) Miller, S. B.; Hawthorne, M. F. *J. Chem. Soc., Chem. Commun.* **1976**, 786. (b) King, R. E., III; Miller, S. B.; Knobler, C. B.; Hawthorne, M. F. *Inorg. Chem.* **1983**, *22*, 3548.

spectra were obtained by using a Bruker WP-200-SY spectrometer operating at 200.13, 81.02, and 64.2 MHz, respectively, and using SiMe_4 , H_3PO_4 , and $\text{BF}_3 \cdot \text{Et}_2\text{O}$ as external standards with high-field resonances being quoted as negative values. The IR spectra were obtained as KBr pellets and recorded on a Specord M-82 spectrometer. Microanalyses were carried out in the analytical laboratory of the Institute of Organoelement Compounds of the Russian Academy of Sciences.

Preparation of *closo*-2,7-(PPh₃)₂-2-H-2-Cl-1-(NMe₃)-2,1-RhCB₁₀H₉ (3) and *closo*-2-(PPh₃)₂-2-Cl-1-(NMe₃)-2,1-RhCB₁₀H₁₀ (4). (A) A suspension of **1** (0.10 g, 0.52 mmol) and $\text{Rh}(\text{PPh}_3)_3\text{Cl}$ (0.48 g, 0.52 mmol) in methanol (30 mL) was stirred under reflux for 16 h, and the yellow precipitate thus formed was collected by filtration, washed with methanol, and dried under vacuum to give an analytically pure complex **3** (0.32 g, 72.4%). Anal. Calcd for $\text{C}_{40}\text{H}_{49}\text{B}_{10}\text{ClINP}_2\text{Rh}$: C, 56.36; H, 5.79; B, 12.69; Cl, 4.15; P, 7.26. Found: C, 56.58; H, 5.48; B, 12.29; Cl, 4.00; P, 7.29. IR (cm^{-1}): $\nu(\text{B}-\text{H})$ 2524. ^1H NMR (CD_2Cl_2 , 22 °C): δ 7.61–7.52 (m, 30H, Ph), 2.92 (s, 9H, CH_3), –9.63 (ddd, 1H, Rh–H). $^{31}\text{P}\{^1\text{H}\}$ NMR (CH_2Cl_2 , 22 °C): δ 26.2 (d, $J_{\text{P}-\text{Rh}}$ = 104.5 Hz), 11.0 (q-like, $J_{\text{P}-\text{B}}$ $\approx$ 120 Hz). $^{11}\text{B}\{^1\text{H}\}$ NMR (CH_2Cl_2 , 22 °C): –2.9 (s), –7.8 (d, $J_{\text{P}-\text{B}}$ = 127 Hz), –8.9 (s), –19.1 (s), –22.3 (s). The reduced mother liquor was chromatographed on silica gel with CH_2Cl_2 as the eluent. The deep red fraction eluted as the first band was evaporated, and the solid residue was recrystallized from a mixture of $\text{CH}_2\text{Cl}_2/n$ -hexane to afford red microcrystalline **4** (0.022 g, 7.2%). Anal. Calcd for $\text{C}_{22}\text{H}_{34}\text{B}_{10}\text{ClINPRh}$: C, 44.78; H, 5.77; B, 18.32; Cl, 6.02. Found: C, 44.77; H, 5.35; B, 18.20; Cl, 6.89. IR (cm^{-1}): $\nu(\text{B}-\text{H})$ 2528. ^1H NMR (CD_2Cl_2 , 22 °C): δ 7.63–7.32 (m, 30H, Ph), 3.69 (s, 9H, CH_3). $^{31}\text{P}\{^1\text{H}\}$ NMR (CH_2Cl_2 , 22 °C): δ 37.3 (d, $J_{\text{P}-\text{Rh}}$ = 152 Hz). $^{11}\text{B}\{^1\text{H}\}$ NMR (CD_2Cl_2 , 22 °C): 8.4 (1B), 6.7 (2B), 2.9 (2B), –6.3 (1B), –18.0 (4B).

(B) The treatment of **1** with $\text{Rh}(\text{PPh}_3)_3\text{Cl}$ under exactly the same conditions, but interrupted after 3.5 h and worked up by an analogous procedure followed by separation of the reduced mother liquor on

preparative TLC plates using CH_2Cl_2 as the eluent, afforded **3** (0.17 g, 38%), **4** (0.073 g, 24%), and a small quantity of an unidentified pink crystalline species (from $\text{CH}_2\text{Cl}_2/n$ -hexane) which was characterized by ^1H and $^{31}\text{P}\{^1\text{H}\}$ NMR spectra only.⁷

(C) Freshly recrystallized PPh_3 (0.050 g, 0.19 mmol) was added to a stirred suspension of **4** (0.050 g, 0.085 mmol) in methanol (10 mL) which was allowed to reflux for 2 h. The yellow precipitate was filtered off and dried under vacuum to afford **3** (0.050 g, 68%).

X-ray Diffraction Study of 3. The crystal data are summarized in Table 1, and selected bond lengths and angles are given in Table 2. X-ray quality crystals of **3** were obtained by slow crystallization from $\text{CH}_2\text{Cl}_2/n$ -hexane at –5 °C. Experimental data were collected on a parallelepiped, $0.3 \times 0.3 \times 0.2$ mm, on a CAD4 Enraf-Nonius diffractometer (T = 293 K, graphite-monochromated $\text{Mo K}\alpha$ radiation, $(\theta - 5)/3\theta$ scan technique, $2\theta < 50^\circ$). The structure was solved by direct methods using SHELXTL PLUS programs (PC Version) and refined by the SHELXL-93 program. The carbon atom of the CH_2Cl_2 solvating molecule is disordered over two positions with occupancies of 0.8 and 0.2.

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Supporting Information Available: Tables of crystallographic data collection parameters, atom coordinates, bond distances and angles, and anisotropic parameters and a figure for **3** (11 pages). Ordering information is given on any current masthead page.

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(13) Perrin, D. D.; Armarego, W. L. F.; Perrin, D. R. *Purification of Laboratory Chemicals*, 2nd ed.; Pergamon Press: Oxford, U.K., 1980.