standard deviation this distance is not significantly longer than that observed in [Co(PnAO-H)$(NO_2)_2$]$^6$ of 2.432 (3) Å or in bis(2-amino-2-methyl-butanone oximato)nickel(II) ion$^2$ of 2.420 (3) Å. In the latter, neutron diffraction revealed a nearly symmetrical hydrogen bond O1–H (1.187 (5) Å) and O2–H (1.242 (5) Å). In the present compound the space group symmetry places the hydrogen atom on a mirror with exactly equal O–H distances or randomly disordered on either side of the mirror. From the difference Fourier the hydrogen atom appears to be on the mirror or very close to it, but because of the rhenium atom, hydrogen atom positions are not well defined.

The infrared frequency associated with the O---H---O vibration is a broad band peaking at 1790 $cm^{-1}$ with a shoulder at about 1855 $cm^{-1}$. In [Cu(PnAO-H)]$NO_3$ this band also occurs at 1800 $cm^{-1}$ but without the shoulder. The position of these bands is similar to that reported for other α-amine oxime complexes.$^{21}$

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**Registry No.** [Cu(PnAO-H)($ReO_4$)], 56804-74-1.

**Supplementary Material Available.** A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Business Office, Books and Journals Division, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for \$4.00 for photocopy or \$2.50 for microfiche, referring to code number AIC503226-12-75.

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# Crystal and Molecular Structure of Chlorobis(3-butenyldiphenylphosphino)rhodium(I)[1]

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The structure of chlorobis(3-butenyldiphenylphosphino)rhodium(I) has been determined by three-dimensional X-ray diffractometer techniques. The compound crystallizes in the space group $P2_1/c$ with cell constants of $a$ = 10.697 (5) Å, $b$ = 9.832 (5) Å, $c$ = 36.44 (2) Å, and $\beta$ = 96.42 (3)°; $Z$ = 4. The structure, solved by heavy-atom techniques, was refined by full-matrix least-squares methods using the 4218 reflections which had intensities 3σ above background to an unweighted $R$ value of 5.1%. The coordination around the Rh(I) atom is best described as trigonal bipyramidal with one phosphorus and the midpoints of the two olefinic groups forming the equatorial plane which lies 0.13 Å toward the Cl atom from Rh(I). In spite of the different geometric constraint imposed by the butenyl chains, both olefinic groups lie in the equatorial plane (within 8.5°). This phenomenon and the placement of the best π acceptor in the equatorial plane are interpreted in terms of a synergetic cooperation between the π and σ bonding in this plane.

## Introduction

Several transition metal complexes containing bidentate ligands in which one donor is a group 5 or 6 atom and the other an olefin have been prepared and a few reactions involving the olefin have been reported.[2,3] The chelate effect offers the opportunity to study weak metal–olefin interactions and the significance of these interactions in the mechanism of catalytic hydrogenation.

During studies concerning rhodium(I) species as homogeneous catalysts, the reaction of $(C_6H_5)_2PCH_2CH_2CH{=}CH_2$ with $[Rh(C_2H_4)_2Cl]_2$ in diethyl ether at room temperature yielded yellow-orange, air-stable crystals of $RhCl[(C_6H_5)_2PCH_2CH_2CH{=}CH_2]_2$. The infrared spectrum of this compound in methylene chloride has an absorption corresponding to unbonded olefin ($\nu_{C=C}$ 1642 $cm^{-1}$) in addition to an absorption at 1505 $cm^{-1}$, assigned as a C–H bending frequency, typical of metal–olefin bonding. In the solid state, however, the spectrum suggests that all olefins are coordinated. The complex is monomeric in chloroform and was thought to be four-coordinate in solution and five-coordinate in the solid state, with each olefin occupying a coordinate site.

The steric requirements of the bidentate ligand $(C_6H_5)_2PCH_2CH_2CH{=}CH_2$ are best met with a 90° angle, P–

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**Table I.** Fractional Coordinates ($\times 10^5$ for Atoms Heavier than C and $\times 10^4$ for the Remainder) and Thermal Parameters[a]

| ATM | X | Y | Z | B11 | B22 | B33 | B12 | B13 | B23 |
|---|---|---|---|---|---|---|---|---|---|
| RH | 14793( 5) | 20195( 5) | 34810( 1) | 667( 5) | 820( 5) | 64( 0) | -116(12) | 128( 2) | 25( 3) |
| CL | 32931(17) | 14653(20) | 85292( 5) | 925(20) | 1512(26) | 139( 2) | 797(39) | 238(11) | 5(12) |
| P1 | 968(16) | 3412(17) | 34590( 4) | 780(18) | 768(19) | 68( 1) | -57(33) | 100( 8) | -5( 9) |
| P2 | 7267(16) | 15633(16) | 89361( 4) | 708(17) | 777(20) | 71( 1) | 132(30) | 77( 8) | -0( 8) |

| ATM | X | Y | Z | B | ATM | X | Y | Z | B |
|---|---|---|---|---|---|---|---|---|---|
| C1 | 9229( 7) | 1070( 8) | 6793( 2) | 4.51(16) | C26 | 6639( 7) | 2036( 9) | 9213( 2) | 5.18(16) |
| C2 | 8162( 8) | 499( 9) | 6996( 2) | 5.36(18) | C27 | 1988( 8) | 210( 8) | 9981( 2) | 5.16(17) |
| C3 | 2610( 7) | 557( 7) | 3234( 2) | 4.50(16) | C28 | 1236( 7) | 407( 7) | 9641( 2) | 4.50(15) |
| C4 | 2855( 7) | 441( 8) | 3619( 2) | 4.69(17) | C29 | 1615( 6) | 1337( 6) | 9388( 2) | 3.51(13) |
| H1 | 9920(56) | 1402(59) | 6949(15) | 5.00( 0) | C30 | 2711( 7) | 2046( 8) | 9477( 2) | 4.81(16) |
| H2 | 8810(53) | 1653(57) | 6581(15) | 5.00( 0) | C31 | 3439( 8) | 1831( 9) | 9820( 2) | 5.89(20) |
| H3 | 7618(54) | 1375(59) | 7063(15) | 5.00( 0) | C32 | 3054( 8) | 899( 8) | 60( 2) | 5.37(18) |
| H4 | 8530(53) | 118(60) | 7215(15) | 5.00( 0) | C33 | 7548( 7) | 1077( 7) | 3394( 2) | 4.05(15) |
| H5 | 3002(55) | 1133(59) | 3105(15) | 5.00( 0) | C34 | 8564( 6) | 681( 6) | 3216( 2) | 3.42(13) |
| H6 | 7463(54) | 236(61) | 6254(15) | 5.00( 0) | C35 | 8418( 7) | 675( 7) | 2826( 2) | 4.64(16) |
| H7 | 3504(55) | 998(61) | 3734(15) | 5.00( 0) | C36 | 7284( 8) | 1069( 8) | 2633( 2) | 5.66(19) |
| C5 | 9231( 7) | 161( 7) | 1262( 2) | 4.22(16) | C37 | 6299( 8) | 1438( 8) | 2813( 2) | 5.82(19) |
| C6 | 9442( 8) | 81( 8) | 1689( 2) | 4.86(17) | C38 | 6404( 7) | 1448( 8) | 3193( 2) | 5.05(17) |
| C7 | 201( 7) | 1332( 7) | 8158( 2) | 3.86(14) | C39 | 9476( 7) | 259( 7) | 5790( 2) | 4.34(15) |
| C8 | 865( 7) | 2006( 8) | 7919( 2) | 4.61(15) | C40 | 236( 6) | 506( 6) | 6113( 2) | 3.44(13) |
| H8 | 8384(57) | 485(59) | 1219(15) | 5.00( 0) | C41 | 1193( 7) | 1479( 7) | 6119( 2) | 4.44(15) |
| H9 | 9807(54) | 657(61) | 1162(15) | 5.00( 0) | C42 | 1352( 8) | 2155( 9) | 5779( 2) | 6.06(21) |
| H10 | 8640(55) | 269(59) | 1796(14) | 5.00( 0) | C43 | 629( 7) | 1875( 8) | 5471( 2) | 5.47(17) |
| H11 | 185(55) | 575(58) | 1792(15) | 5.00( 0) | C44 | 9674( 7) | 942( 8) | 5465( 2) | 5.19(17) |
| H12 | 9331(55) | 1556(57) | 8183(15) | 5.00( 0) | H15 | 6332(55) | 678(59) | 8780(15) | 5.00( 0) |
| H13 | 479(53) | 2210(60) | 2792(15) | 5.00( 0) | H16 | 8201(52) | 376(58) | 8653(15) | 5.00( 0) |
| H14 | 1673(56) | 1727(59) | 7876(15) | 5.00( 0) | H17 | 9482(54) | 1959(58) | 4464(15) | 5.00( 0) |
| C9 | 5034( 7) | 1261( 9) | 1840( 2) | 6.03(19) | H18 | 7566(54) | 3431(57) | 9582(15) | 5.00( 0) |
| C10 | 4466( 8) | 146( 9) | 1963( 2) | 6.33(19) | H19 | 5726(56) | 2099(60) | 9235(15) | 5.00( 0) |
| C11 | 3328( 8) | 239( 9) | 2134( 2) | 7.18(22) | H20 | 8237(52) | 473(59) | 9831(15) | 5.00( 0) |
| C12 | 2859( 8) | 1550( 9) | 2180( 2) | 6.73(21) | H21 | 9585(54) | 136(58) | 428(14) | 5.00( 0) |
| C13 | 3470( 9) | 2311(10) | 7072( 2) | 7.84(23) | H22 | 2968(53) | 2340(59) | 4318(15) | 5.00( 0) |
| C14 | 4576( 9) | 2580( 9) | 1895( 2) | 7.44(23) | H23 | 4042(56) | 2338(60) | 9846(16) | 5.00( 0) |
| C15 | 6520( 9) | 2349(10) | 5480( 3) | 8.10(24) | H24 | 3607(53) | 711(58) | 275(15) | 5.00( 0) |
| C16 | 5976(10) | 1920(10) | 744( 3) | 8.82(26) | H25 | 7585(52) | 968(58) | 3637(16) | 5.00( 0) |
| C17 | 4976( 9) | 2430(10) | 5910( 3) | 8.08(24) | H26 | 9206(52) | 380(57) | 2675(14) | 5.00( 0) |
| C18 | 4601( 9) | 1092(10) | 5800( 2) | 7.69(23) | H27 | 7279(52) | 1009(58) | 2373(16) | 5.00( 0) |
| C19 | 5216( 8) | 464( 9) | 5539( 2) | 6.69(20) | H28 | 5461(55) | 1772(56) | 2696(15) | 5.00( 0) |
| C20 | 6187( 8) | 1047( 9) | 5386( 2) | 7.14(22) | H29 | 5765(54) | 1672(57) | 3340(15) | 5.00( 0) |
| C21 | 6881( 7) | 1148( 8) | 8946( 2) | 5.11(18) | H30 | 1206(53) | 377(58) | 4215(14) | 5.00( 0) |
| C22 | 8124( 7) | 955( 7) | 8861( 2) | 4.26(15) | H31 | 1777(52) | 1648(55) | 6382(15) | 5.00( 0) |
| C23 | 9101( 6) | 1679( 6) | 9055( 2) | 3.13(12) | H32 | 1823(58) | 2513(64) | 897(17) | 5.00( 0) |
| C24 | 8828( 6) | 2441( 7) | 4332( 2) | 3.76(14) | H33 | 724(52) | 2427(55) | 5225(15) | 5.00( 0) |
| C25 | 7580( 7) | 2269( 7) | 4409( 2) | 4.60(16) | H34 | -817(52) | 587(56) | 5230(15) | 5.00( 0) |

[a] Anisotropic thermal parameters are defined by $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + 2B_{12}hk + 2B_{13}kl + B_{23}kl)]$ and are multiplied by $10^4$.

Rh–(C=C), and the olefin requires a coordination site and orientation favorable to back-bonding from metal d orbitals. Solution studies indicate that energy differences between various pentacoordinate structures are quite small[4] and the barrier to rotation about a metal–monoolefin bond is in the range 10–15 kcal mol$^{-1}$.[5,6]

The crystal structure determination was undertaken to substantiate the formulation as a monomeric five-coordinate complex of rhodium(I) and to determine which five-coordinate structure could meet both the steric requirements of two bidentate ligands and the back-bonding requirements of the two olefins.

## Experimental Section

The best formed crystals available appeared spontaneously from a $C_6D_6$ solution used for NMR studies. Precession photographs established the space group to be $P2_1/c$. The crystal was mounted on a Picker diffractometer automated with a PDP-8 computer and accurate cell constants were obtained by least-squares refinement of 12 reflections ($a$ = 10.697 (5) Å, $b$ = 9.832 (5) Å, $c$ = 36.44 (2) Å, $\beta$ = 96.42 (3)°, $Z$ = 4). One unique data set was collected with Cu K$\alpha$ radiation up to 120° in $2\theta$ [5658 intensities measured; 4218 with $I > 3\sigma(I)$].

Intensities were measured using a 2° $\theta$–$2\theta$ step-scan in 0.05° increments with a 2-sec count taken at each step, and a 20-sec background count was measured at each end of the scan. A standard reflection measured after every 50 data points showed a decrease of approximately 20% over the period of data collection and was corrected for by fitting a fourth degree polynomial to the standard reflection curve. Absorption was corrected for using Burnham's program[7] as modified by Larson, Cromer, and Roof. The linear absorption coefficient for Cu K$\alpha$ radiation is 52.4 cm$^{-1}$ and the calculated transmission varied from 0.60 to 0.38.

The Rh atom was located by the Patterson method and all remaining atoms (other than hydrogen) were found in successive Fourier electron density maps. Two molecules of $C_6D_6$ of crystallization appeared during the analysis. Most of the hydrogen atoms (including all 14 in the butene side chains) were located in a difference synthesis. Missing hydrogens on the phenyl rings were included in the refinement in their idealized position. Peaks which appeared in the proper location to be deuterium atoms were not included in the refinements.

Full-matrix least-squares refinements were carried out on all parameters listed in Table I except for the hydrogen thermal pa-

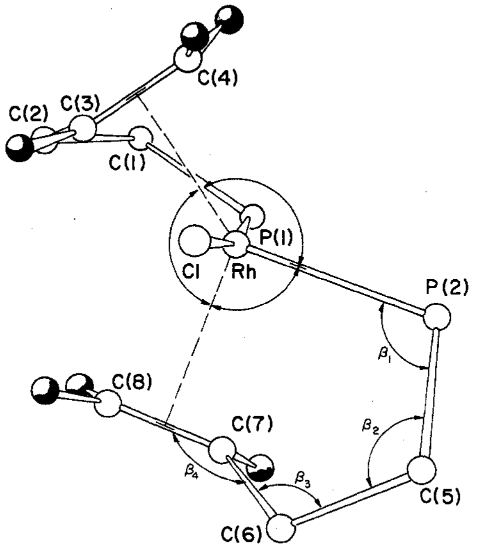

**Figure 1.** Projection of the coordination about Rh.

rameters. The function minimized was $\Sigma w(F_o - F_c)^2$ where $w = 1/\sigma_F^2$ and $\sigma_F$ is computed from $\sigma_I^2 = T + B + [0.015(T - B)]^2$, $T$ being the total count and $B$ the estimated background. Neutral scattering factors were used for Rh, Cl, P, and C with real and imaginary anomalous terms included.[8] The scattering factor curve of Stewart et al.[9] was used for hydrogen. A final difference map showed residuals such that $|\rho(x)| < 0.5$ e/Å$^3$ and the maximum and minimum values were centered about the heavy atoms except as noted above. The final $R$ value ($R = \Sigma|F_o| - |F_c|/\Sigma|F_o|$) was 0.051 with all atoms heavier than carbon assigned anisotropic temperature factors and all phenyl and butene hydrogen positions refined.

## Discussion

The coordination about the Rh atom, displayed in Figures 1 and 2, can be considered to be a distorted trigonal bipyramid with an equatorial plane defined by P(2) and the centers of the two olefin groups. The axial angle defined by P(1)–Rh–Cl is 169.3 (1)° and the Rh is displaced 0.13 Å toward P(1) from the equatorial plane. In both olefinic groups the C=C bond is slightly tipped with respect to the equatorial plane, C(3)–C(4) by 1.5° and C(7)–C(8) by 8.5°. (See Table II for selected distances and angles.)

One of the most interesting features of the structure is the striking difference between two butene groups. Olefin C(3)–C(4) has a long C–C distance and short Rh–C distances, while the C(7)–C(8) olefin exhibits a short C–C distance and a long Rh–C distance, and P(1) has a shorter Rh–P distance than does P(2). Examination of the Rh–P–C and $\beta_3$ angles shows clear evidence for considerably more strain in butenyl 2 [C(7) through C(8)] than in butenyl 1. Although the strain may be partially relieved by a more favorable torsional angle about the C(6)–C(7) bond in butenyl 2, it is apparent that the molecule has sacrificed considerable conformational energy in butenyl 2 in order to orient the C(7)–C(8) bond in the equatorial plane of the idealized trigonal bipyramid.

**Table II.** Selected Distances (Å) and Angles (deg)

(a) Angles in the Butenyl Side Chains

| | $\beta_1$ | $\beta_2$ | $\beta_3$ | $\beta_4$ | $\tau^a$ |
|---|---|---|---|---|---|
| Butene 1 | 106.3 (2) | 108.5 (5) | 111.8 (7) | 121.7 (7) | 35.7 |
| Butene 2 | 103.9 (2) | 109.9 (5) | 114.6 (6) | 123.4 (7) | 123.0 |

(b) Distances in the Butenyl Side Chains

| | Rh–P | P–$C_n$ | $C_n$–$C_{n+1}$ | $C_{n+1}$–$C_{n+2}$ | $C_{n+2}$–$C_{n+3}$ |
|---|---|---|---|---|---|
| Butene 1 | 2.211 (2) | 1.853 (7) | 1.535 (10) | 1.519 | 1.402 (9) |
| Butene 2 | 2.375 (2) | 1.846 (7) | 1.547 (9) | 1.530 (9) | 1.358 (9) |

(c) Distances and Angles about Rh(I)

| | | | |
|---|---|---|---|
| Rh–Cl | 2.437 | Cl–Rh–P(1) | 169.3 (1) |
| Rh–P(1) | 2.211 (2) | Cl–Rh–P(2) | 85.4 (1) |
| Rh–P(2) | 2.375 (2) | P(1)–Rh–P(2) | 100.7 (1) |
| Rh–C(3) | 2.14 (1) | Cl–Rh–MP34 | 85.9 |
| Rh–C(4) | 2.16 (1) | Cl–Rh–MP78 | 89.5 |
| Rh–MP34[b] | 2.03 | P(1)–Rh–MP34 | 83.9 |
| Rh–C(7) | 2.35 (1) | P(1)–Rh–MP78 | 99.1 |
| Rh–C(8) | 2.29 (1) | P(2)–Rh–MP34 | 141.2 |
| Rh–MP78 | 2.22 | P(2)–Rh–MP78 | 90.2 |
| | | MP34–Rh–MP78 | 127.4 |

[a] $\tau$ is the dihedral angle about the $C_{n+1}$–$C_{n+2}$ bond. [b] Denotes the midpoint of the C(3)–C(4) bond.

Convincing proof of the preference of Rh(I), in a trigonal-bipyramidal environment, for olefinic bonds lying in the equatorial plane is afforded by the structure of $[Rh(C_2H_4)_3(CH_3CN)_2]BF_4$,[10] and to a lesser extent (because of possible constraints imposed by the butenyl group) by $RhCl[P(CH_2CH_2CH{=}CH_2)_3]$.[11]

A simple qualitative argument has been advanced by Rösch and Hoffmann[12] to account for this behavior. In a molecule with $D_{3h}$ symmetry (for example, $Ni(C_2H_4)_3$), the $\sigma$ orbitals for the olefinic groups contain the irreducible representation e' as do $d_{xy}$ and $d_{x^2-y^2}$. The $\pi^*$ orbitals transform as $a_2' + e'$ if they lie in the equatorial plane and as $a_2'' + e''$ if the C–C bonds are perpendicular to the plane. Therefore if the $\pi^*$ orbitals are higher in energy than the d orbitals of the isolated metal atom, the $\sigma$ interaction provides a perturbation which tends to favor orientation of the olefinic $\pi^*$ orbitals in the equatorial plane, for systems with more than six d electrons. Since such a synergistic effect is prohibited by symmetry for a ligand in an axial position, it has been pointed out that the best $\pi$ acceptors are expected to be in the equatorial plane for trigonal-bipyramidal geometry.[12-14] The structure presented here provides a convincing illustration of this principle; i.e., the Cl⁻ and one of the phosphorus atoms reside at the axial positions while the remaining phosphorus atom and olefinic ligands take equatorial positions.

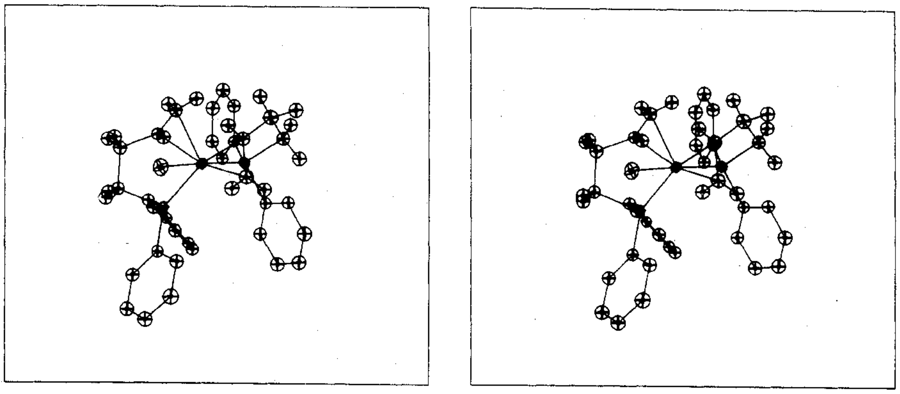

**Figure 2.** Stereoview of the molecule. View vector is from the $-a$ axis.

**Acknowledgment.** The authors are indebted to A. C. Larson for the use of his crystal codes.

**Registry No.** $RhCl[(C_6H_5)_2PCH_2CH_2CH{=}CH_2]_2$, 56629-85-7.

**Supplementary Material Available.** A listing of structure factor amplitudes will appear following these pages in the microfilm edition of this volume of the journal. Photocopies of the supplementary material from this paper only or microfiche (105 × 148 mm, 24× reduction, negatives) containing all of the supplementary material for the papers in this issue may be obtained from the Business Office, Books and Journals Division, American Chemical Society, 1155 16th St., N.W., Washington, D.C. 20036. Remit check or money order for \$4.50 for photocopy or \$2.50 for microfiche, referring to code number AIC50395P-12-75.

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(1) This work was sponsored in part by the Energy Research and Development Administration.
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(15) Supplementary material.

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# Crystal and Molecular Structure of Iron(II) Bis(diethyldithiocarbamate)

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The molecular structure of $[Fe(S_2CN(C_2H_5)_2)_2]_2$ has been determined from single-crystal X-ray diffraction data obtained by counter methods. The compound crystallizes in space group $C_{2h}^5$-$P2_1/c$ with four monomers per unit cell of dimensions $a = 10.0540$ (69) Å, $b = 10.6937$ (40) Å, $c = 16.5824$ (42) Å, and $\beta = 112.6$ (1)°. The calculated and observed densities are 1.470 and 1.457 (5) g cm$^{-3}$, respectively. Full-matrix least-squares refinement using data having $F_o^2 > 3\sigma(F_o^2)$ gave $R_1 = 0.058$ for the 2786 data. The compound is dimeric with distorted trigonal-bipyramidal geometry around each iron atom. Thus it is similar in structure to the isomorphous $[Zn(S_2CN(C_2H_5)_2)_2]_2$. The two dithiocarbamate ligands each bridge one axial and one equatorial position of the trigonal bipyramid. The distorted trigonal bipyramid is achieved by dimerization through sulfur bridges with a resultant Fe–Fe distance of 3.350 Å.

## Introduction

Iron(II) dithiocarbamates are a class of chemically reactive compounds with some striking physical properties.[1] We have been studying the chemical reactions of iron(II) dithiocarbamates with NO, CO, and $NO^+$.[2,3] The magnetic susceptibility and Mössbauer[4] spectra of $Fe^{II}(S_2CNEt_2)_2$ indicate that it is dimeric. The magnetic susceptibility shows antiferromagnetic coupling between pairs of iron atoms and a Neel temperature of 110 K.

This class of compounds also has properties which may be related to those of iron–sulfur proteins.[5–7] Recently Holm[8] has prepared iron–sulfur clusters containing the $Fe_2S_2$ and $Fe_4S_4$ units. The structures of the $Fe_4S_4$ cluster and the $Fe_2S_2$ cluster containing **Fe(III)** have been determined by Holm, Ibers, and their coworkers.[9,10] However, as Lippard indicated in his recent review of this subject,[11] there appear to be no structural studies of $Fe_2S_2$ complexes in which **Fe(II)** is exclusively coordinated to sulfur ligands. While a preliminary investigation of $Fe^{II}(S_2CNEt_2)_2$ by X-ray powder patterns[12] showed it to be isomorphous with the corresponding dinuclear Zn(II) complex[13] and Cu(II) complex,[14] the Cu(II) and Zn(II) complexes have different coordination geometries. Thus, the coordination geometry around Fe(II) remained unknown. The crystal structure determination described below was undertaken to provide a detailed picture of the coordination geometry around the iron atom in $Fe^{II}(S_2CNEt_2)_2$.

## Experimental Section

Crystals of the compound $Fe(S_2CNEt_2)_2$ were obtained by the method published elsewhere.[15] Large dark red crystals were obtained by this procedure. The crystals obtained are extremely sensitive toward air and therefore were stored in sealed ampoules under nitrogen. Crystals for X-ray investigation were sealed in glass capillaries under nitrogen. Preliminary precession photographs taken with Mo K$\alpha$ radiation indicated that the crystals are monoclinic. The systematic absences of $h0l$ when $l \neq 2n$ and $0k0$ when $k \neq 2n$ are consistent with the space group $C_{2h}^5$-$P2_1/c$. At 23 ± 2°C the unit cell parameters are $a = 10.0540$ (69) Å, $b = 10.6937$ (40) Å, $c = 16.5824$ (42) Å, and $\beta = 112.6$ (1)°. These parameters were determined from a least-squares refinement of the setting angles of 12 reflections that were centered on a Picker FACS-I computer-controlled four-circle X-ray diffractometer. The values of $2\theta$ for these reflections were 30–42°. In the refinement the wavelength of Mo K$\bar{\alpha}$ radiation was taken as 0.71069 Å. The crystal density of 1.457 (5) g cm$^{-3}$ determined by flotation in bromobenzene–chlorobenzene is in good agreement with the value of 1.470 g cm$^{-3}$ calculated for four molecules per unit cell.

For data collection a crystal of approximate dimensions 0.4 × 0.4 × 0.3 mm was mounted with its $b$ axis approximately collinear with the $\phi$ axis of the diffractometer. The mosaicity of the crystal was checked by $\omega$ scans at a takeoff angle of 0.7°. All reflections had peak widths of 0.15° or less. An incident beam monochromator equipped with a graphite crystal was used to obtain Mo K$\alpha$ radiation. The $\theta$–$2\theta$ axis of the monochromator is perpendicular to the $\theta$–$2\theta$ axis of the diffractometer. Data were collected at a takeoff angle of 2.0° using the $\theta$–$2\theta$ scan technique and an asymmetric scan range from $2\theta_{MoK\alpha_1} - 0.85°$ to $2\theta_{MoK\alpha_2} + 0.85°$. The pulse height analyzer was set to admit ~90% of Mo K$\alpha$ peak and the scintillation counter was 24 cm away from the crystal. Cu foils having attenuation factors of ~1.7 were inserted into the diffracted beam whenever the counting rate exceeded ~$10^3$ counts/sec. During data collection the intensities of three reflections in diverse regions of reciprocal space were monitored after every 100 reflections. The maximum variation of any standard from its mean value was ~5%. A total of 4893 unique reflections having $2\theta \leq 50°$ were obtained. The data were reduced to $F^2$ and