as O(7) has sulfur atom neighbors at 3.24 (1) and 3.33 (1) Å. These distances are within the range of O-H···S hydrogen bond distances which have been verified by nmr[17,18] and neutron diffraction[16] studies for sodium thiosulfate pentahydrate and magnesium thiosulfate hexahydrate, and the angles to these two sulfur neighbors and the two sodium neighbors are roughly tetrahedral. On this basis we assign the two hydrogen atoms of O(7) to O-H···S bonds. Water molecule O(8) has a neighbor O(5) at 2.75 (1) Å and a normal distance for an O-H···O hydrogen bond, and again the angles defined by this oxygen neighbor and the two sodium neighbors are reasonable for hydrogen bonding. The position of the other hydrogen on O(8) must complete a more or less tetrahedral coordination for this oxygen atom, but no neighbor is available close enough in a suitable direction for a hydrogen bond. Thus we conclude that O(8) makes only one hydrogen bond.

The nearest neighbors of the sodium atoms are all oxygen atoms. Na(1) is coordinated by six oxygen atoms at distances of 2.36-2.63 Å; the two nearest neighbors are two oxygen atoms of water molecules. Na(2) is also coordinated by six oxygen atoms at distances from 2.35 to 2.54 Å, the nearest of which comes from a water molecule. Na(3) is coordinated to five oxygen atoms ranging in distance from 2.34 to 2.53 Å, the closest of which is from a water molecule. The stereogram in Figure 3 shows some of the coordination about the sodium ions.

The next nearest neighbor to the gold atom, besides S(3) and S(4), is another gold atom at 3.302 (1) Å. This distance may be compared with 2.884 Å in metallic gold[19] and values ranging from 2.67 to 3.27 Å in various gold cluster complexes.[20] If metallic bonding is present in our complex, it must be relatively weak.

Registry No. $Na_3Au(S_2O_3)_2 \cdot 2H_2O$, 33847-99-3.

Supplementary Material Available. The table of observed and calculated 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 Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-74-1836.

(17) C. R. K. Murty and Z. M. El Saffar, *Acta Crystallogr.*, 15, 536 (1962).
(18) Z. M. El Saffar, *Acta Crystallogr., Sect. B*, 25, 1708 (1969).
(19) D. N. Batchelder and R. O. Simmons, *J. Appl. Phys.*, 36, 2864 (1965).
(20) S. L. Lawton, W. J. Rohrbaugh, and G. T. Kokotailo, *Inorg. Chem.*, 11, 2227 (1972), and references cited therein.

Contribution from the Department of Chemistry and Laboratory for Research on the Structure of Matter, University of Pennsylvania, Philadelphia, Pennsylvania 19174

Crystal and Molecular Structure of μ-Carbonyl-bis-μ-[bis(bis(trifluoromethyl)phosphino)sulfur-*P,P'*]-bis(carbonylnickel)[1]

HOWARD EINSPAHR and JERRY DONOHUE*

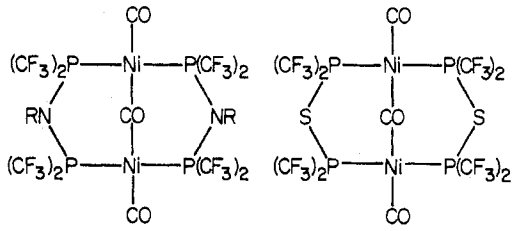
Received November 5, 1973 AIC40156M

The crystal structure of $Ni_2[(CF_3)_2PSP(CF_3)_2]_2(CO)_3$ was determined from visually estimated intensity data from single crystals. The unit cell is orthorhombic, space group *Pbcn*, with $a = 10.49 \pm 0.01$ Å, $b = 14.26 \pm 0.01$ Å, and $c = 18.77 \pm 0.01$ Å, and contains four molecules. A trial structure was obtained by direct methods and Fourier techniques and refined by full-matrix least squares. The final value of the R index is 0.087. The molecular structure includes a nickel-nickel bond of length 2.577 (5) Å and five-membered nickel-phosphorus-sulfur rings.

Introduction

The synthesis of $Ni_2[(CF_3)_2PSP(CF_3)_2]_2(CO)_3$ was first reported as one of a series of syntheses of nickel carbonyl complexes with bifunctional phosphine ligands.[2,3] With the exception of the thiobisphosphine product, all were fairly well characterized by standard physicochemical methods. The infrared spectra of two of the products, $Ni_2[(CF_3)_2PN(R)P(CF_3)_2]_2(CO)_3$, R = H, CH_3, suggested the presence of both terminal and bridging carbonyl groups. On the basis of this and other evidence (molecular weights, decomposition products), bicyclic structures (I and II) were proposed for both. ^{19}F nmr spectra of both amine products[3] precluded the presence of significant paramagnetic behavior in either compound.

Attempts to measure the molecular weight of the thiobisphosphine product were without success. Its infrared spectrum, however, also gave evidence of bridging carbonyl groups and a structure similar to those of the aminobisphosphines was postulated. The thiobisphosphine product is assumed to exhibit magnetic properties similar to those of aminobisphosphines.



I II

R = H, CH_3

The presence of a bridging carbonyl group suggested the possible presence of metal-metal bonding. Among the population of known metal-metal bonds, examples of bonds between zerovalent nickel atoms are relatively rare. At the time this investigation was initiated only one such example

(1) Supported by the National Science Foundation and the Advanced Research Projects Agency, Office of the Secretary of Defense.
(2) A. B. Burg and R. A. Sinclair, *J. Amer. Chem. Soc.*, 88, 5354 (1966).
(3) R. A. Sinclair and A. B. Burg, *Inorg. Chem.*, 7, 2160 (1968).

Table I. Atomic Parameters ($\times 10^4$ except Those at $^1/_4$)[a]

Atom	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C_5	0	3057 (18)	$^1/_4$	124 (27)	50 (14)	41 (13)	0	−12 (35)	0
O_5	0	3807 (11)	$^1/_4$	231 (30)	30 (7)	41 (9)	0	45 (29)	0
Ni	89 (2)	2000 (2)	1815 (1)	117 (3)	62 (1)	36 (1)	−10 (4)	−9 (3)	3 (3)
P_1	2010 (4)	1479 (3)	1796 (2)	81 (3)	49 (2)	21 (1)	−7 (5)	8 (4)	0 (5)
P_2	1394 (4)	973 (3)	3370 (3)	81 (3)	44 (2)	22 (1)	13 (5)	12 (4)	−1 (5)
S	2801 (4)	662 (3)	2610 (3)	102 (5)	73 (3)	33 (2)	72 (6)	20 (5)	22 (6)
C_6	31 (21)	2788 (13)	3934 (12)	166 (24)	48 (9)	42 (8)	88 (30)	−65 (26)	−74 (23)
O_6	42 (18)	3246 (15)	4410 (10)	243 (26)	122 (14)	59 (8)	35 (34)	−38 (26)	−90 (23)
C_1	3228 (17)	2477 (18)	1769 (16)	86 (17)	93 (16)	51 (9)	−75 (30)	−17 (25)	−12 (32)
F_{11}	3051 (16)	2965 (12)	1206 (10)	232 (21)	114 (11)	75 (8)	−175 (26)	−37 (21)	73 (20)
F_{12}	3079 (13)	3007 (9)	2299 (9)	167 (15)	81 (8)	65 (6)	−71 (19)	20 (17)	−75 (17)
F_{13}	4431 (10)	2195 (9)	1754 (7)	104 (11)	100 (8)	64 (6)	−77 (17)	4 (14)	7 (15)
C_2	2620 (20)	772 (20)	1001 (13)	125 (21)	102 (19)	35 (7)	−4 (36)	86 (25)	5 (32)
F_{21}	3860 (11)	590 (12)	1048 (9)	118 (13)	191 (15)	78 (7)	30 (25)	31 (17)	−124 (21)
F_{22}	2496 (18)	1239 (14)	457 (8)	360 (33)	117 (14)	37 (5)	2 (32)	63 (22)	−32 (16)
F_{23}	2031 (13)	−17 (10)	974 (10)	199 (18)	94 (9)	85 (9)	−121 (23)	141 (22)	−93 (18)
C_3	1108 (19)	−269 (15)	3654 (12)	123 (21)	66 (12)	34 (7)	−14 (29)	7 (22)	42 (24)
F_{31}	553 (12)	−260 (8)	4334 (6)	191 (15)	72 (7)	29 (3)	6 (19)	21 (14)	3 (13)
F_{32}	2093 (12)	−794 (8)	3685 (8)	163 (14)	70 (7)	61 (6)	51 (18)	26 (16)	39 (14)
F_{33}	233 (11)	−667 (8)	3231 (6)	187 (16)	68 (6)	35 (4)	−73 (18)	−10 (14)	13 (12)
C_4	2508 (19)	1261 (16)	4132 (12)	119 (18)	67 (13)	36 (7)	−41 (30)	−20 (23)	0 (28)
F_{41}	3027 (13)	2112 (10)	3996 (8)	172 (15)	114 (10)	64 (7)	−70 (21)	−105 (19)	22 (17)
F_{42}	1830 (13)	1416 (10)	4725 (6)	214 (18)	115 (10)	30 (4)	31 (25)	−29 (14)	−10 (14)
F_{43}	3422 (11)	711 (9)	4248 (7)	144 (13)	112 (10)	55 (6)	69 (22)	−93 (15)	−25 (16)

[a] Estimated standard deviations are in parentheses. Temperature factors are expressed as $\exp[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + hl\beta_{23})]$.

had been reported, that being the trinuclear paramagnetic species $Ni_3(C_5H_5)_3(CO)_2$.[4] Further interest was generated by the thiobisphosphine ligand and the unusual ring system in which it was postulated to participate. For these reasons, an X-ray investigation was undertaken.

Experimental Section

Samples of the thiobisphosphine product were provided by Professor A. B. Burg of the University of Southern California. The samples had been recrystallized by sublimation and contained a number of well-formed single crystals. The crystals were observed to be rhombic prisms. They were transparent and yellow; however, after several days of exposure to the atmosphere, they acquired a brassy cast due to surface decomposition.

Preliminary Weissenberg photographs indicated an orthorhombic lattice. Systematic absences ($hk0$, $h + k = 2n + 1$; $0kl$, $k = 2n + 1$; $h0l$, $l = 2n + 1$) uniquely determine the space group as $Pbcn$-D_{2h}^{14} (No. 60). Lattice constants at room temperature, obtained using $h0l$ and $0kl$ precession photographs, are $a = 10.49 \pm 0.01$ Å, $b = 14.26 \pm 0.01$ Å, and $c = 18.77 \pm 0.01$ Å. A density of 2.2 g cm^{-3} was obtained by flotation.[5] The calculated density, assuming four formula weights per unit cell, is 2.23 g cm^{-3}.

Two crystals were used to collect the intensity data. A prism 0.50 mm in length with a cross section of about 0.25 × 0.25 mm was used to record the data of layers $hk0$ through $hk8$, and a second crystal with dimensions of about 0.25 × 0.25 × 0.25 mm was used to collect the data of layers $h0l$ through $h5l$. All data were collected using the multiple-film equiinclination Weissenberg technique with Cu Kα radiation. Intensities were estimated by visual comparison with a standard intensity strip. Lorentz–polarization corrections were applied and structure factor magnitudes calculated. No absorption corrections were made ($\mu = 68.5$ cm^{-1}). The 289 observed reflections common to both axial settings were used to place all observations on a common scale employing the method of Heilbronner.[6] The agreement index of the common data after scaling, $\Sigma|F_b^2 - F_c^2|/\Sigma(F_b^2 + F_c^2)$, was evaluated as 0.085. A total of 948 unique reflections was observed.

A three-dimensional Patterson map was calculated which determined the coordinates of the nickel and sulfur atoms as (0.00, 0.20, 0.18) and (0.28, 0.07, 0.25). Successive electron density maps failed to produce a trial model owing to the creation by these coordinates of a mirror plane perpendicular to and at $^1/_4$ in z which prohibited phasing of $h + k$ odd structure factors. Although a sharpened Patterson map revealed the positions of the phosphorus atoms at (0.20, 0.15, 0.175) and (0.14, 0.10, 0.325), their contributions provided a violation of the pseudosymmetry insufficient to phase a significant number of the $h + k$ odd reflections.

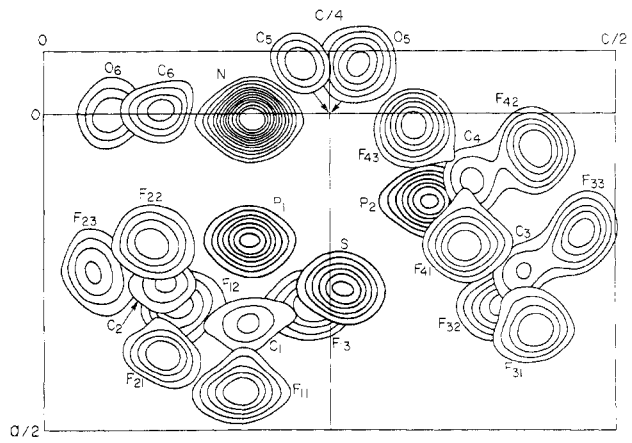


Figure 1. Composite of the final electron density map viewed down the b axis. Contours of the Ni, P, and S peaks are shown at intervals of 2 e/Å^3 beginning at 4 e/Å^3; all other peaks are contoured at intervals of 1 e/Å^3 beginning at 3 e/Å^3.

The data were then converted to normalized structure factors, and the Σ_2 relation[7] was applied.[8] The phases of the origin-determining reflections, two of which had $h + k$ odd indices, were chosen to be consistent with the origin from the Patterson map. Symbols were assigned, where necessary, to enhance the phasing of $h + k$ odd reflections. The phase information obtained in this manner was compared with that from structure factor calculations and, after several iterations, a trial structure was obtained from an electron density map. A contoured representation of the final map is shown in Figure 1.

Full-matrix least-squares refinement of this trial structure was made using program UCLALS4[9] which minimizes the function $\Sigma w \cdot [|F_o| - (1/k)|F_c|]^2$. The weighting scheme was that of Hughes[10]

(4) A. Hock and O. S. Mills, "Advances in the Chemistry of Coordination Compounds," Macmillan, New York, N. Y., 1961, pp 640–648.

(5) R. A. Sinclair, unpublished work, 1965.

(6) E. Heilbronner, unpublished work, 1963.

(7) H. Hauptman and J. Karle, "Solution of the Phase Problem. I. The Centrosymmetric Crystal," American Crystallographic Association Monograph No. 3, Polycrystal Book Service, Pittsburgh, Pa., 1953.

(8) R. E. Long, Doctoral Dissertation, University of California, Los Angeles, Calif., 1956.

(9) P. K. Gantzel, R. A. Sparks, R. E. Long, and K. N. Trueblood, unpublished work, 1964.

(10) E. W. Hughes, *J. Amer. Chem. Soc.*, 63, 1737 (1941).

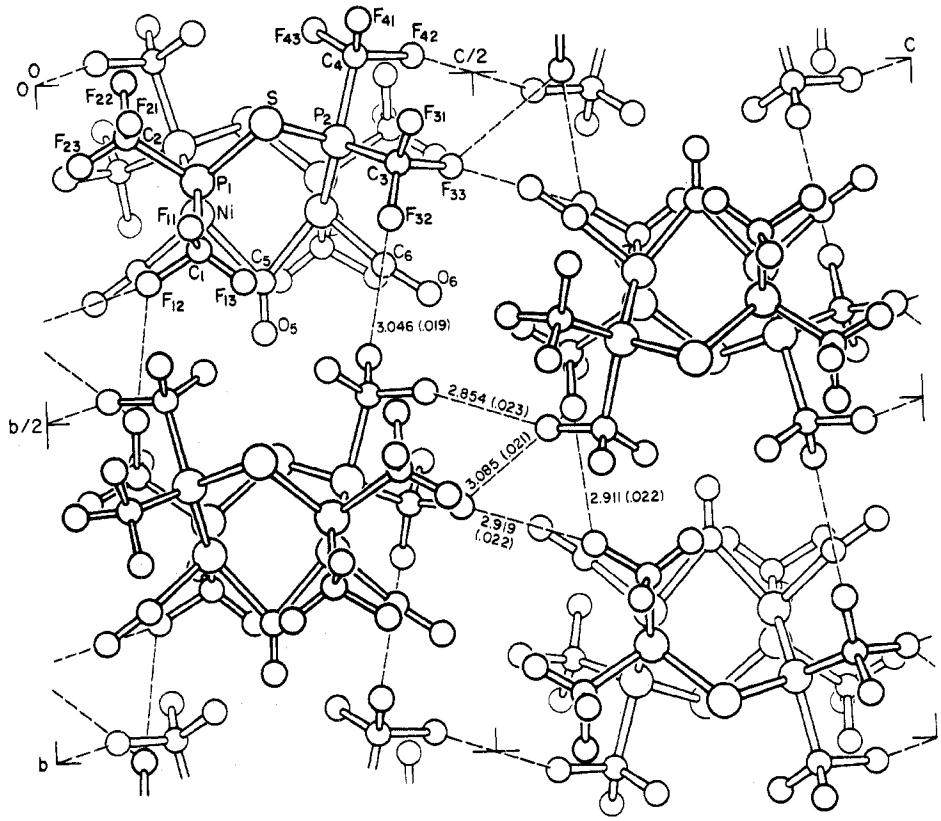


Figure 2. Representation of the structure viewed down the *a* axis. Fluorine–fluorine contacts are indicated by dashed lines. Distances (esd's) are in angstroms.

with $4F_{min} = 25$. Isotropic refinement continued until convergence with an R index, $\Sigma||F_o| - |F_c||/\Sigma|F_o|$, of 0.13. The data were then edited for indexing errors and refinement was continued with anisotropic thermal parameters for all atoms. Convergence was achieved in eight cycles with a final R index of 0.087. A structure factor calculation for the unobserved reflections revealed no serious discrepancies. The large fraction of unobserved reflections in the Cu sphere is attributed in part to absorption and large thermal motion. A final difference map contained no peak heights with magnitudes greater than 1 e/Å^3. The atomic scattering factors from ref 11 were used exclusively.

Results

Final values of the atomic parameters are shown in Table I. Table II is a list of observed and calculated structure factors.[12]

There are no unusually short intermolecular distances in this structure. The closest approaches of atoms in adjacent molecules are F···F van der Waals contacts ranging from 2.85 to 3.09 Å ($\sigma = 0.02$ Å). Five of these are shown in Figure 2, a representation of the structure viewed down *a*. Not shown are $F_{11}\cdots F_{11}'$ of 3.043 (25) Å, $F_{11}\cdots F_{32}'$ of 3.018 (18) Å, and $F_{21}\cdots F_{31}'$ of 2.910 (17) Å to molecules in the unit cell above this one in *a*. The prime indicates that the atom so specified occupies the position $(1 - x, y, {}^1/_2 - z)$.

Bond distances and angles for the molecule are shown in Figure 3 and are further elaborated for the trifluoromethyl groups in Table III.

Discussion

The intramolecular distance between Ni atoms of 2.577 Å strongly suggests the presence of a metal–metal bond. The Ni–Ni distance in the metal is 2.487 Å at 18°.[13] Numerous measurements of metal–metal bonds in analogous compounds of iron and cobalt indicate that the metal–metal distance found in this compound lies within the range of distances expected for Ni(0)–Ni(0) bond. In $Ni_4(CO)_6[P(C_2H_4CN)_3]_4$[14] the Ni(0)–Ni(0) bonds average 2.508 (4) Å.

Further evidence for the existence of a nickel–nickel bond

Table III. Distances (Å) and Angles (deg) for the Trifluoromethyl Groups[a]

	Distances		
C_1–F_{11}	1.33	C_3–F_{31}	1.26
C_1–F_{12}	1.28	C_3–F_{32}	1.35
C_1–F_{13}	1.26	C_3–F_{33}	1.34
C_2–F_{21}	1.33	C_4–F_{41}	1.28
C_2–F_{22}	1.29	C_4–F_{42}	1.41
C_2–F_{23}	1.23	C_4–F_{43}	1.34
	Angles		
P_1–C_1–F_{11}	114	F_{11}–C_1–F_{12}	107
P_1–C_1–F_{12}	109	F_{11}–C_1–F_{13}	108
P_1–C_1–F_{13}	110	F_{12}–C_1–F_{13}	108
P_1–C_2–F_{21}	112	F_{21}–C_2–F_{22}	108
P_1–C_2–F_{22}	109	F_{21}–C_2–F_{23}	105
P_1–C_2–F_{23}	109	F_{22}–C_2–F_{23}	113
P_2–C_3–F_{31}	118	F_{31}–C_3–F_{32}	107
P_2–C_3–F_{32}	107	F_{31}–C_3–F_{33}	111
P_2–C_3–F_{33}	110	F_{32}–C_3–F_{33}	103
P_2–C_4–F_{41}	116	F_{41}–C_4–F_{42}	107
P_2–C_4–F_{42}	108	F_{41}–C_4–F_{43}	110
P_2–C_4–F_{43}	110	F_{42}–C_4–F_{43}	105

[a] Estimated standard deviations are 0.03 Å for distances and 2° for angles.

(11) "International Tables for X-Ray Crystallography," Vol. III, Kynoch Press, Birmingham, 1968, pp 202–209.

(12) See paragraph at end of paper regarding supplementary material.

(13) W. B. Pearson, "Lattice Spacing and Structures of Metals and Alloys," Pergamon Press, London, 1958.

(14) M. J. Bennett, F. A. Cotton, and B. H. C. Winquist, *J. Amer. Chem. Soc.*, 89, 5366 (1967).

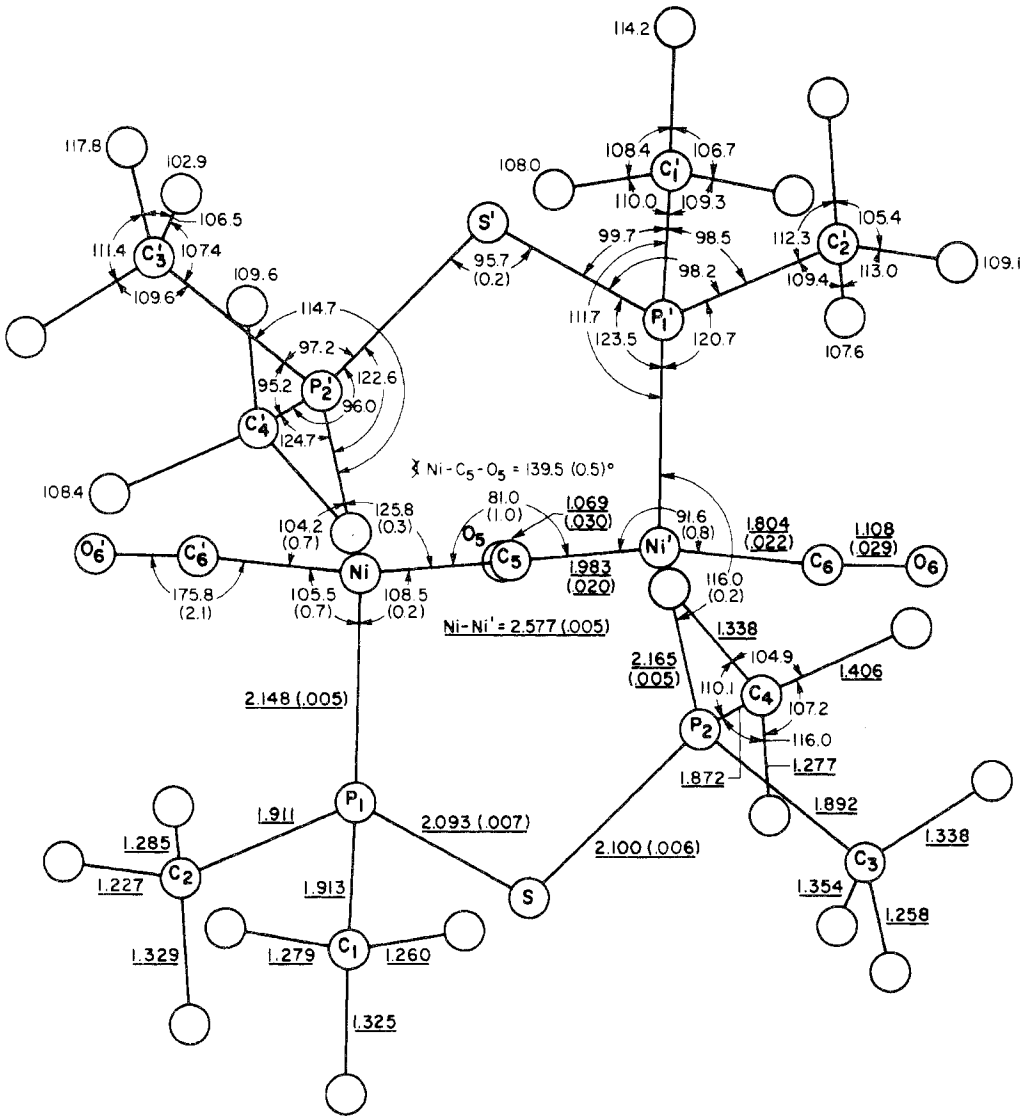


Figure 3. View of the molecule down the *b* axis. Fluorine atoms are not labeled. Distances are in angstroms and angles are in degrees. Primed atoms are the twofold equivalents of the corresponding unprimed atoms. Esd's are 0.3° for Ni-P-S, 0.7° for Ni-P-C, 0.8° for S-P-C, and 1° for C-P-C angles and 0.02 Å for P-C distances.

in this compound is found in the valence angles involving the nickel atoms, notably the bridging Ni-C-Ni angle and Ni-Ni-P angles. In fact, the sum of the angles about the Ni atom (involving C_5, P_1, and P_2 only) is 350.30°, the Ni atom being within 0.4 Å of the plane of these atoms, 0.3 Å closer to this plane (and toward the other Ni atom) than would be expected for the same bond distances and tetrahedral angles.

Once the existence of Ni-Ni bond has been established, an unusual five-membered Ni_2P_2S ring presents itself. The small P-S-P angle may thus be the result of the strain of ring closure. The configuration of the ring is nonplanar, P_1 being 0.4 Å above and P_2 0.5 Å below the Ni_2S plane. Table IV contains values for the internal torsion angles of the ring. The torsion angle between the two Ni_2S planes in the molecules is 114.1°. The two P_2S planes are twisted with respect to each other in a manner which is consistent with the difference in C_5-Ni-P angles.

The Ni-P bond lengths average 2.16 (1) Å. This may be compared with an average of 2.162 (6) Å for two such bonds reported[14] in $Ni_4(CO)_6[P(C_2H_4CN)_3]_4$. The trifluoromethyl groups assume staggered conformations about the P-C bonds. Average values of bond distances and angles for this group agree well with previously reported values, as do average values of the P-C and P-S bond lengths.

Table IV. Torsion Angles[a] (deg) for the Nickel-Phosphorus-Sulfur Ring[b]

Ni-P_1-S-P_2	+15.6
P_1-S-P_2-Ni'	−6.7
S-P_2-Ni'-Ni	+20.0
P_2-Ni'-Ni-P_1	−22.6
Ni'-Ni-P_1-S	−26.1

[a] W. Klyne and V. Prelog, *Experientia*, 16, 521 (1960). [b] The average standard deviation is estimated as 0.4°. Primed atoms occupy the equivalent position ($\bar{x}$, y, $^1/_2 - z$).

The distances and angles associated with the carbonyl groups are comparable with previously reported values. While the C_5-O_5 distance is unusually short, it is unlikely that this anomaly is real. Since this portion of the molecule exhibits a great deal of thermal motion, it is more likely that a sizable thermal correction is associated with this bond length. The fact that the C_5-Ni bond is somewhat longer than expected suggests a center of libration for the bridging carbonyl intermediate between the C_5 and O_5 positions.

Acknowledgments. We thank Drs. A. B. Burg and R. A. Sinclair for preparation of the crystals used in this study and

Drs. H. L. Carrell and C.-C. Tsai for helpful suggestions during the course of this investigation. The figures were prepared by the late Mrs. Maryellin Reinecke.

Registry No. $Ni_2[(CF_3)_2PSP(CF_3)_2]_2(CO)_3$, 51391-40-3.

Supplementary Material Available. Table II, 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 Journals Department, American Chemical Society, 1155 16th St., N.W., Washington, D. C. 20036. Remit check or money order for $3.00 for photocopy or $2.00 for microfiche, referring to code number INORG-74-1839.

Contribution from the Department of Chemistry, University of Western Ontario, London, Ontario, N6A, 3K7, Canada

Studies on Metal-Acetylene Complexes. III. Crystal and Molecular Structure of Methyl(hydrotris(1-pyrazolyl)borato)hexafluorobut-2-yneplatinum(II)

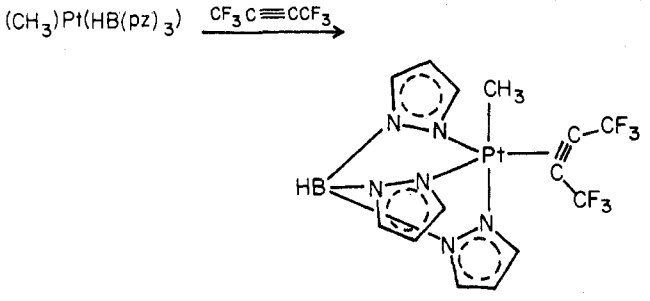
BRIAN WILLIAM DAVIES and NICHOLAS C. PAYNE*

Received October 15, 1973 AIC30754L

The crystal and molecular structure of methyl(hydrotris(1-pyrazolyl)borato)hexafluorobut-2-yneplatinum(II), $(CH_3)(HB(N_2C_3H_3)_3)(F_3CC{\equiv}CCF_3)Pt^{II}$, has been determined by X-ray diffractometric methods. Crystals are orthorhombic with $a = 18.424$ (5), $b = 13.385$ (3), and $c = 7.568$ (2) Å. Refinement has been achieved in the centric space group *Pnma*. A crystallographic mirror plane is imposed on the molecule such that the platinum atom, the methyl carbon atom, the boron atom, and one pyrazole ring lie on the mirror plane. Full-matrix least-squares refinement on F utilizing 1917 observations for which $I > 3\sigma(I)$ has led to residuals $R_1 = 0.035$ and $R_2 = 0.050$. The coordination about the platinum atom is essentially trigonal bipyramidal, with the acetylene considered to be a monodentate ligand. The acetylenic triple bond is lengthened on coordination to 1.292 (12) Å. The acetylene bend-back angle is 34.4 (4)°, consistent with a reasonably perturbed acetylene, $\Delta\nu(C{\equiv}C)$ being 457 cm^{-1}. The polypyrazolylborate ligand has essentially C_{3v} symmetry, the mean angle between the pyrazolyl rings being 120.1 (2)°. Two equatorial and one axial sites of the trigonal bipyramid about the Pt atom are occupied by the tridentate ligand.

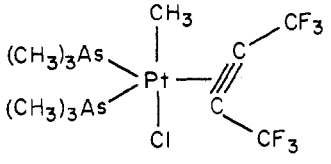
Introduction

Recently Clark and Manzer prepared a series of Pt(II) complexes containing polypyrazolylborate ligands.[1] The complexes were prepared by cleavage of the polymeric complex $(CH_3)Pt[HB(pz)_3]$ with a variety of olefins, acetylenes, and allenes, where $(HB(pz)_3)^-$ is the hydrotris(1-pyrazolyl)borate anion. Thus with hexafluorobut-2-yne



I

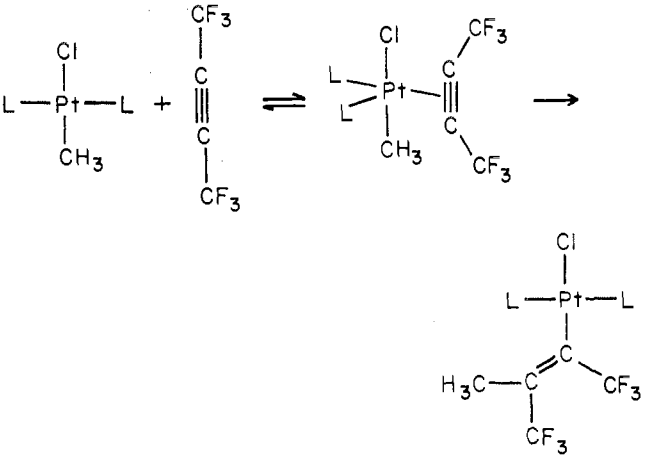
Nmr evidence suggested that the product was best formulated as I. This formulation is similar to that of *trans*-chloromethylbis(trimethylarsine)hexafluorobut-2-yneplatinum(II), $Cl(CH_3)((CH_3)_3As)_2(C_4F_6)Pt$, II, the structure of which has



II

been determined in this laboratory.[2]

Of particular interest to us is the difference in reactivities of I and II toward "insertion" reactions. Thus, II was postulated as a possible intermediate in the reaction



(where L = $As(CH_3)_3$) which occurs readily under mild conditions.[3]

In direct contrast, I does not "insert" and is remarkably unreactive. It is stable in solution up to 100° and the acetylene is resistant to nucleophilic attack.[1] Both complexes have similar reductions in C≡C stretching frequencies upon coordination, $\Delta\nu(C{\equiv}C)$ being 457 cm^{-1} for I and 462 cm^{-1} for II. We investigated the structure of I as part of our continuing studies of metal-acetylene complexes and for the purpose of seeing whether any structural feature could explain

(1) L. E. Manzer and H. C. Clark, *J. Amer. Chem. Soc.*, 95, 3812 (1973); *Inorg. Chem.*, 13, 1291, 1996 (1974).

(2) Part I: B. W. Davies, R. J. Puddephat, and N. C. Payne, *Can. J. Chem.*, 50, 2276 (1972); part II: B. W. Davies and N. C. Payne, *ibid.*, 51, 3477 (1973).

(3) R. J. Puddephat and H. C. Clark, *Inorg. Chem.*, 10, 18 (1971).