program.[16] With the assumption that the activation volume ΔV^* is independent of pressure,[17a] the conditional first-order rate constant is expressed as eq 13, where p^0 and q^0 are con-

$$k_0 = \frac{p^0 \exp(-P\Delta V_p^*/RT)[\mathrm{SCN^-}]}{q^0 \exp(-P\Delta V_q^*/RT) + [\mathrm{SCN^-}]} \qquad (13)$$

stants at zero pressure. Values of p^0, q^0, ΔV_p^*, and ΔV_q^*[18] were obtained by a nonlinear least-squares fit to all six sets of data (132 data points) simultaneously. Values of p^0 and q^0 were revealed to be the same as corresponding values obtained at atmospheric pressure. All the parameters obtained are tabulated in Table I.

Discussion

The p and q values correspond to k_1 and k_{-1}/k_2 in Mechanism I and k and K^{-1} in Mechanism II, respectively. In Mechanism I, for

$$\mathrm{MP(H_2O)_2^{5+}} \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} \mathrm{MP(H_2O)^{5+}} + \mathrm{H_2O} \qquad (14)$$

$$\mathrm{MP(H_2O)^{5+}} + \mathrm{SCN^-} \xrightarrow{k_2} \mathrm{MP(H_2O)(SCN)^{4+}} \xrightarrow[\text{fast}]{\mathrm{SCN^-}} \mathrm{MP(SCN)_2^{3+}} + \mathrm{H_2O} \qquad (15)$$

the value of ΔV_p^* for k_1 should be positive and almost equal to or a little lower than the partial molal volume of water (+18 cm^3 mol^{-1}), since the partial molal volume of the five-coordinated metalloporphyrin does not differ much from that of the six-coordinated metalloporphyrin. The ΔV_q^* value for k_{-1}/k_2 seems to be small: the effect of the partial charge neutralization in the transition state of the k_2 path should be small, since the positive charge on the metalloporphyrin is largely delocalized over the whole porphyrin nucleus. In Mechanism II for

$$\mathrm{MP(H_2O)_2^{5+}} + \mathrm{SCN^-} \overset{K}{\rightleftharpoons} \mathrm{MP(H_2O)_2^{5+}\cdots SCN^-} \qquad (16)$$

$$\xrightarrow{k} \mathrm{MP(H_2O)(SCN)^{4+}} + \mathrm{H_2O} \xrightarrow[\text{fast}]{\mathrm{SCN^-}} \mathrm{MP(SCN)_2^{3+}} + 2\mathrm{H_2O} \qquad (17)$$

the ΔV value for K should be positive because of partial relieving of electrostriction by ion-pair formation[19-21] and the ΔV_p^* value for k should be small. From these considerations together with the results given in Table I, we conclude that the substitution of $\mathrm{CoP(H_2O)_2^{5+}}$ with $\mathrm{SCN^-}$ occurs by a dissociative mechanism. Negative pressure effect on the reaction makes Mechanism III unlikely.

Pasternack et al.[5] studied the reaction of $\mathrm{CoP(H_2O)_2^{5+}}$ with $\mathrm{SCN^-}$ to produce the monothiocyanato complex $\mathrm{CoP(H_2O)(SCN)^{4+}}$ and obtained the activation parameters ΔH^* = 82.8 kJ mol^{-1} and ΔS^* = 44.8 J mol^{-1} K^{-1}. In the present case, these values correspond to $\Delta H_p^* - \Delta H_q^*$ = 77 kJ mol^{-1} and $\Delta S_p^* - \Delta S_q^*$ = 24 J mol^{-1} K^{-1}, respectively. The agreement of their results with ours is good, when we take into account the experimental errors involved. Available evidence suggests that the similar reactions proceed via an $\mathrm{S_N1}$ type mechanism in which the rate-determining step involves the breaking of a cobalt(III)-water bond.[6,22]

Acknowledgment. The present work was partially supported by Grant-in-Aid for Scientific Research No. 584023 from the Ministry of Education, Science and Culture.

Registry No. $\mathrm{CoP(H_2O)_2^{5+}}$, 51405-04-0; $\mathrm{SCN^-}$, 302-04-5.

Supplementary Material Available: Table SI, a listing of the conditional first-order rate constants obtained under the various conditions (3 pages). Ordering information is given on any current masthead page.

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[17] (a) Stranks[17b] indicated the low compressibilities of octahedral complexes. Taking for granted that the intrinsic contribution is incompressible, the pressure dependence of the volume of activation can be correlated with the compressibility of the solvent. Changes of the molar volume of water in pressures up to 1250 kg cm^{-2} are small, within the accepted error limits for volumes of activation. In fact, the present data were satisfied with the assumption of the pressure independence of the activation volume. (b) Stranks, D. R. *Pure Appl. Chem.* **1974**, *38*, 303.

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^{31}P NMR Spin-Lattice Relaxation Time Measurements on Platinum(II)- and Ruthenium(II)-Substituted Phosphine, Diphenylphosphinito, and Dimethyl Phosphito Complexes

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In a continuing synthetic effort on the coordination chemistry of diphenylphosphinous acid and dimethyl phosphite, we have observed considerable intensity differences between nonequivalent phosphorus nuclei in the ^{31}P NMR spectra of the metal complexes with these ligands. These intensity variations could have their origin in spin-lattice time (T_1) differences between the separate phosphorus nuclei. In view of the very small amount of published work on the ^{31}P spin-lattice relaxation time in transition-metal phosphine complexes,[2] we have carried out a series of T_1 measurements to try and learn more about the factors influencing line intensities in complexes of platinum(II) and ruthenium(II) with diphenylphosphinito and dimethyl phosphito ligands.

Results and Discussion

The measured T_1 data for a series of platinum(II) and ruthenium(II) complexes are shown in Table I. For comparison T_1 values for $\mathrm{[NH_4][S_2PPh_2]}$, $\mathrm{Ph_2PH(O)}$, and $\mathrm{(MeO)_2PH(O)}$ are also given. The compound $\mathrm{[NH_4][S_2PPh_2]}$ has a long relaxation time of 30.0 s, a value comparable with that reported for triphenylphosphine.[3] For the methyl compound $\mathrm{Na[S_2PMe_2]}$, the corresponding T_1 value is 20.0 s. The compound $\mathrm{Ph_2PH(O)}$ has a short relaxation time of 2.5 s. Such a short time arises because of the dipole-dipole relaxation contribution from the vicinal hydrogen directly bonded to the phosphorus. The efficiency of the dipole mechanism is proportional to the sixth power of the phosphorus-hydrogen separation,[4,5] and hence, a directly bonded proton will make a dominant dipole-dipole contribution to the relaxation. The value found

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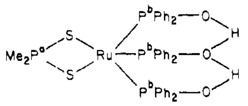
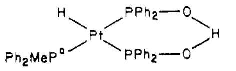
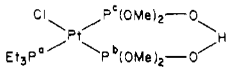
Table I. T_1 Values for ^{31}P Nuclei in Pt and Ru Compounds

compd	T_1, s
$Ph_2PH(O)$	2.5^a
$Na[S_2PMe_2]$	$20.0^{b,c}$
$[NH_4][S_2PPh_2]$	$30.0^{b,c}$
	7.4 (P^a), 6.0 (P^b)[b]
	5.9 (P^a), 6.6 (P^b), 6.7 (P^c)[a]
$(MeO)_2PH(O)$	7.5^a
	7.0 (P^a), 10.4 (P^b), 10.4 (P^c)[a]
$Pt[(OP(OMe)_2)_4H_2]$	10.4^a

[a] Measured by inversion recovery method on a proton-coupled sample. [b] Measured by progressive saturation method on a proton proton-decoupled sample. [c] D_2O solvent.

for dimethyl phosphite has the significantly longer value of 5.5 s, even though again there is a P–H bond in the molecule which should dominate the dipole–dipole relaxation.[6]

Coordination to a metal through phosphorus leads to complexes having no P–H bonds, and hence the relaxation time T_1 is longer in the Pt(II) and Ru(II) complexes. The values of T_1 for the diphenylphosphinito or dimethyl phosphito ligands in the complexes show little variation. The phosphite T_1 values remain longer than the phosphine ones. The T_1 value appears to be independent of molecular geometry. Furthermore, the diphenylphosphinite group in the platinum hydride complex $PtH((OPPh_2)_2H)PMePh_2$ does not appear to have a shortened T_1 due to any dipole–dipole relaxation from the hydride ligand. If a major dipole–dipole relaxation contribution was being made by the hydride ligand, we would expect the T_1 values to be significantly less than found for the (diphenylphosphinito)ruthenium complex. This hydride compound does, however, show a concentration-dependent ^{31}P spectrum with the lines P_a and P_c, but not P_b, showing reversible broadening at high solute concentrations. This observation can be explained by the proposal of a monomer–dimer equilibrium occurring in solutions of the complex. The broadening of certain resonances may be indicative of the particular site of the association, although we cannot decide the specific alternative on the basis of present data.

$$2PtH((OPPh_2)_2H)PMePh_2 \rightleftharpoons [PtH((OPPh_2)_2H)PMePh_2]_2$$

Coordination of the phosphinito ligand to the metal at one end and a hydrogen-bonded structure at the other end, as shown for the compounds in Table I, will likely reduce any contribution from spin rotation relaxation. Nevertheless, this structure arrangement will create a more rigid framework to lengthen the correlation time T_c. Since T_1 is inversely proportional to T_c, this effect will contribute to the relatively short T_1 values in the diphenylphosphinito and dimethyl phosphito ligands in the complexes. This increased correlation time may explain why the T_1 values for the coordinated dimethyl phosphito ligand are only slightly longer than that of the free compound with its directly bonded hydrogen atom.

Table II. T_1 Values for ^{31}P Nuclei in Ru Complexes with Changing Phosphine Ligands Obtained from the Progressive Saturation Technique

compd	T_1, s	$\delta(S_2PMe_2)^c$
cis-$[Ru(S_2PMe_2)_2(PPh_2OEt)_2]$	$3.8,^a$ 4.3^b	86.5
cis-$[Ru(S_2PMe_2)_2(PPh_2H)_2]$	$5.3,^a$ 2.3^b	89.3
cis-$[Ru(S_2PMe_2)_2(PPhCl_2)_2]$	$7.2,^a$ 8.7^b	92.1
cis-$[Ru(S_2PMe_2)_2(PCl_3)_2]$	$13.6,^a$ 14.4^b	95.5

[a] S_2PMe_2 group. [b] PR_3 group. [c] Chemical shifts in ppm from H_3PO_4.

In Table II are shown the T_1 values found for a series of complexes $Ru(S_2PMe_2)_2(PR_3)_2$ (PR_3 = PPh_2H, PPh_2OEt, $PPhCl_2$, PCl_3). The T_1 values cover a range of 4–14 s for the S_2PMe_2 and 2–14 s for the PR_3 ligands. The T_1 for the ligand PR_3 is shortest for PPh_2H and then becomes progressively longer as the number of phenyl groups on the phosphorus decreases and hence also the number of protons in close proximity for dipole–dipole relaxation. The T_1 values for the ruthenium complexes show little shortening from the values in $PtH((OPPh_2)_2H)PMePh_2$, indicating any quadrupole relaxation contribution from ^{99}Ru and ^{101}Ru is small. The T_1 values for the S_2PMe_2 ligand show a trend parallel to that of the PR_3 ligand, and in the four complexes the T_1 values for the inequivalent phosphorus nuclei are similar. Thus, the differing intensities in the $^{31}P\{^1H\}$ spectra of these complexes is not due to differences in the T_1 values. In fact, preliminary studies reveal that proton-coupled spectra of these compounds show *no* significant intensity differences for the different phosphorus nuclei. Thus, the fundamental conclusion of our work is that the variation in line intensities is not a consequence of T_1 differences.

From Table II it is apparent that there is a rough correspondence between the chemical shift (δ) and the T_1 value for the S_2PMe_2 ligand. This correlation implies that there is communication between the ligands. The change in T_1 for the S_2PMe_2 ligand may be partly due to dipole–dipole coupling with PR_3, which is enhanced when PR_3 has a large number of phenyl rings. This relationship between T_1 and δ would not necessarily be anticipated for a dipole–dipole mechanism but *may* be indicative of a contribution from a spin rotation mechanism where both the chemical shift and relaxation time are related in the tensor.[7] Alternately, the relaxation mechanism can be completely dipole–dipole and changes in T_1 due to differences in correlation times.

Experimental Section

Dimethyl phosphite was a commercial sample that was purified by distillation.

Diphenylphosphinous acid and the metal complexes were prepared by literature methods.[8] T_1 values were measured in $CDCl_3$ solvent except for those of $[NH_4][S_2PPh_2]$ and $Na[S_2PMe_2]$, which were measured in D_2O because of their low solubility in $CDCl_3$. Measurements were made at 303 K. Samples were not degassed. Spectra were recorded on either a Varian SL-100 spectrometer operating at 40.51 MHz (progressive saturation method) or a Bruker WH-90 spectrometer operating at 36.44 MHz (inversion recovery method). Relaxation time measurements were made by using the progressive saturation method $(90°\text{-AT-t})_n$ or the inversion recovery method $(\text{PD-}180°\text{-t-}90°\text{-AT})_n$. Quick estimates of T_1 values were obtained with the inversion recovery null signal method, whence $T_1 = t/\ln 2$,

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where *t* is the pulse interval required for a null signal.

For each compound measured, the observed spectral width was chosen to be the narrowest which would encompass all the signals from the compound. For the progressive saturation method, the length for each measurement was 8192 points, the maximum available. This led to the digital resolution varying from 0.08 Hz/point to 1.25 Hz/point (for $Ru(S_2PMe_2)(PPh_2OEt)_2$). A sensitivity-enhancement weighting function was applied to all FID's before transformation, ensuring that sufficient points defined all peaks to be measured. Whenever possible, peak integrals were used. Analysis of the results was done by the classical semilog linearization followed by least-squares analysis of the straight line obtained. Comparison with the three-parameter nonlinear least-squares method of Gerhards[9] showed identical trends, with slight numerical differences in T_1 values.

For the inversion recovery method, a sweep width of 3012 Hz was used incorporating 4096 data points. An acquisition time/scan of 1.36 s was used and recovery times were chosen that were a minimum of 5 times the T_1 values. Delay times were set and data acquisition was performed by the computer T_1 program. Analysis of the data from T_1 measurements was by least-squares fitting of the semilog plot ln $(S_\infty - S_t)$ against *t*, whence the slope provides the T_1 value (S_∞ = equilibrium signal intensity, S_t = signal intensity at time *t*). A minimum of four time *t* values were used, and up to 1000 scans had to be collected to obtain acceptable signal-to-noise ratios in the signals. For each method we estimate our values to be good to ±6%.

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Registry No. $Ph_2PH(O)$, 4559-70-0; $Na[S_2PMe_2]$, 34669-04-0; $[NH_4][S_2PPh_2]$, 56152-33-1; $Ru(S_2PMe_2)[(OPPh_2)_3H_2]$, 79466-66-3; $PtH(PPh_2Me)[(OPPh_2)_2H]$, 68547-04-6; $(MeO)_2PH(O)$, 868-85-9; $PtCl(PEt_3)[(OP(OMe)_2)_2H]$, 63264-38-0; $Pt[(OP(OMe)_2)_4H_2]$, 30053-63-5; *cis*-$[Ru(S_2PMe_2)_2(PPh_2OEt)_2]$, 79547-84-5; *cis*-$[Ru(S_2PMe_2)_2(PPh_2H)_2]$, 79482-69-2; *cis*-$[Ru(S_2PMe_2)_2(PPhCl_2)_2]$, 79482-70-5; *cis*-$[Ru(S_2PMe_2)_2(PCl_3)_2]$, 79482-71-6.

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A Heteronuclear (Pt₄,Ag) Complex of 1-Methyluracil and Its Conversion into a Crystalline Platinum Blue

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Metal binding to the N3 position of the 1-methylthymine or 1-methyluracil monoanion (deprotonated at N3) facilitates binding of additional metals or of a proton through exocyclic oxygens of these ligands. This has been demonstrated for di-[1-3] and heteronuclear (Ag,[4] Mn,[5] H[6]) complexes of *cis*-$Pt(NH_3)_2^{2+}$ and a complex of Ag.[7]

We herewith report the synthesis and structure of another heteronuclear complex of 1-methyluracil containing four *cis*-$Pt(NH_3)_2^{2+}$ moieties and one Ag^+, $Ag[Pt(NH_3)_2(1\text{-}MeU)]_4(NO_3)_5\cdot 4H_2O$ (**1**),[8] and its conversion into a crystalline "platinum blue" of composition $[Pt(NH_3)_2(1\text{-}MeU)]_4(NO_3)_5\cdot 5H_2O$ (**2**). Although the insufficient crystal size of **2** so far has prevented the X-ray structure determination,[9] there is considerable evidence (vide infra) that it is the analogue of the "α-pyridone blue".[10]

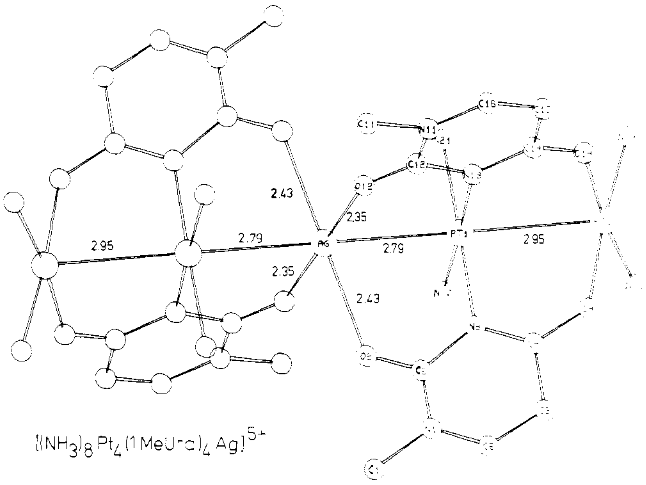

Figure 1. Molecular cation of **1**, $[(NH_3)_4Pt_2(C_5H_5N_2O_2)_2Ag(C_5H_5N_2O_2)_2Pt_2(NH_3)_4]^{5+}$.

Crystals of **1** were isolated from a solution of the head-to-head dimer *cis*-$[Pt_2(NH_3)_4(C_5H_5N_2O_2)_2](NO_3)_2\cdot H_2O$[11] (425 mg in 15 mL of H_2O), to which $AgNO_3$ (338 mg) had been added, upon slow evaporation (pH 4–5). The yield was 100 mg of **1** within 1–2 days; deep yellow crystals, somewhat photosensitive (gradual darkening on exposure to light). Characterization was accomplished by X-ray analysis[12] and elemental analysis.[13] Among other products, that will be reported on later, varying amounts (5–30 mg) of a second crystalline compound **2** were isolated. The identical compound **2** was also obtained when **1** was dissolved in water (O_2 free, saturated with N_2) and kept in a sealed tube. The color of the solution (0.1 M Ag, pH 2–3) rapidly changed from yellow immediately after being dissolved to turquois or, if briefly (5–15 min) warmed to 80 °C, to blue.[14] Formation of the blue color was observed regardless of whether the sample was exposed to daylight or not. Upon cooling, crystals of **2** precipitated from the blue solution along with other products.[15] Separation was accomplished by hand under a microscope, and recrystallization was from water. With the warmed sample, occasional formation of a metallic mirror (Ag) was observed, wheras with samples kept at 3–22 °C, formation of some black

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(8) 1-MeU is the monoanion of 1-methyluracil.

(9) Crystal size ca. 1 × 0.02 × 0.02 mm.

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(11) Preparation analogous to that of the corresponding 1-methylthymine dimer (ref 1). Elemental analysis was satisfactory for C, H, N, and Pt.

(12) The crystal data of **1** are a = 14.418 (3) Å, b = 12.894 (4) Å, c = 13.527 (3) Å, β = 99.86 (2)°, V = 2477.6 Å^3, space group $P2_1/c$, Z = 2, ρ_{calcd} = 2.55 g cm^{-3}, and ρ_{obsd} = 2.53 g cm^{-3}. The 2566 observed reflections (Syntex P2$_1$ diffractometer, λ = 0.71069 Å, $2° \leq 2\theta \leq 48°$, $F_o > 4.7\sigma_{F_o}$, empirical absorption correction) gave a final $R = \sum||F_o| - |F_c||/\sum|F_o|$ = 0.072. The structure was solved by MULTAN 80 and difference Fourier syntheses. The heavy atoms were refined anisotropically and all light atoms, except the hydrogens, isotropically. One of the anions is positioned at a center of inversion and is disordered.

(13) Anal. Calcd for $AgPt_4C_{20}H_{48}N_{21}O_{27}$: C, 12.62; H, 2.55; N, 15.46; Ag, 5.67. Found: C, 12.52; H, 2.72; N, 15.04; Ag, 5.58.

(14) With diluted samples, formation of an olive green color was observed on heating.

(15) Other identified products were: $[Pt_2(NH_3)_2(1\text{-}MeU)_2](NO_3)_2$ (head-to-head dimer) and $Pt(NH_3)_2(1\text{-}MeU)_2\cdot 0.5AgNO_3$. No attempts were made to optimize the yield of **2**.

0020-1669/82/1321-0451$01.25/0