4d orbitals in these compounds. In Table I we have summarized the relevant data for a number of molybdenum compounds. Consider, for example, molybdenum hexacarbonyl. Here the ionization potential of the t_{2g} electrons (8.50 eV) is 2.78 eV higher than the LOIP (the value that the ionization potential would have if the t_{2g} electrons were strictly nonbonding, i.e. 5.72 eV). Thus for the first time we have an experimental value for the stabilization of the t_{2g} orbitals in $Mo(CO)_6$ caused by back-bonding to the carbonyl groups.

Table I includes two ionization potentials for each of six compounds of the type $Mo(CO)_5PX_3$. In each case the lower ionization potential corresponds to the e(Mo) orbital, which has the same symmetry as the π orbitals of the PX_3 group, and the higher ionization potential corresponds to the b_2(Mo) orbital, which is orthogonal to the π orbitals of the PX_3 group. All the b_2 stabilization energies fall in the range 2.78–2.90 eV, essentially equal to the stabilization energy of $Mo(CO)_6$. All the e stabilization energies fall in the range 2.51–2.70 eV, slightly lower than the stabilization energy of $Mo(CO)_6$. It is not surprising that all the ionization potentials of the b_2 orbitals, which can interact with four CO groups, are greater than the ionization potentials of the e orbitals, each of which can interact with three CO groups and the PX_3 group. This observation, which has been previously discussed by Yarbrough and Hall,[13] is consistent with the assumption that all of the PX_3 groups are poorer π acceptors than CO. The fact, revealed by the present data, that all the stabilization energies of the b_2 orbitals are very similar in magnitude to the stabilization energy of the t_2 orbitals of $Mo(CO)_6$ is also not surprising. This fact merely serves as another confirmation of the validity of the general LOIP method. However, we were surprised by the fact that, in all the $Mo(CO)_5PX_3$ compounds, the *percentage* reduction in the stabilization energy as one goes from the b_2 to the e orbital (i.e., on replacement of one of four CO groups by a PX_3 group) is only about 10% or less. The data clearly show that, when one of the four CO groups that interact with a $d\pi$ orbital is replaced by a PX_3 group, the three remaining CO groups *almost completely* take up the slack in back-bonding. Although this qualitative behavior has long been assumed in rationalizations of the trans effect, it has not been recognized that CO ligands are so resilient that they can almost completely compensate for the reduction in ligand π acceptance when a CO ligand is replaced by a relatively poor π-acceptor ligand such as $P(CH_3)_3$.

Table I also contains data for *cis*- and *trans*-$Mo(CO)_4[P(CH_3)_3]_2$ and *fac*-$Mo(CO)_3[P(CH_3)_3]_3$. As expected, the stabilization energy of the d_{xy} orbital of *trans*-$Mo(CO)_4[P(CH_3)_3]_2$, 2.82 eV, is essentially the same as the t_{2g} stabilization energy of $Mo(CO)_6$. The stabilization energies of all the other d orbitals (which could conceivably engage in π interactions with the $P(CH_3)_3$ ligands) are 2.30, 2.60, and 2.33 for *cis*- and *trans*-$Mo(CO)_4[P(CH_3)_3]_2$ and 2.37 eV for *fac*-$Mo(CO)_3[P(CH_3)_3]_3$. These values are significantly lower than the t_{2g} stabilization energy of $Mo(CO)_6$. Thus the data are consistent with a steady decrease in the overall back-bonding of the molybdenum $d\pi$ orbitals as CO ligands are stepwise replaced with $P(CH_3)_3$ ligands.

Next consider the dimolybdenum tetracarboxylates $Mo_2(O_2CH)_4$, $Mo_2(O_2CCH_3)_4$, and $Mo_2(mhp)_4$. The Mo 4d ionization potentials listed for these molecules correspond to the Mo–Mo δ-bonding orbitals. The stabilization energies, as measured by the IP – LOIP values, are all around 1 eV, indicating that the δ contributions to these Mo–Mo quadruple bonds are quite substantial.

Experimental Section

Tetrakis(dimethylamido)molybdenum(IV) was prepared from $LiN(CH_3)_2$ and $MoCl_5$.[14] The product was purified by two sublimations (10^{-3} torr) at room temperature onto a −20 °C cold finger. The infrared and mass spectra agreed with those in the literature.[14] Vapor-phase X-ray photoelectron spectra were obtained with a GCA/McPherson ESCA-36 spectrometer using a Mg anode. The vapor from a sample held at 10 °C diffused directly into the spectrometer gas cell through large diameter (1.5-cm) tubing. The spectra were calibrated against the N 1s line of admixed N_2 gas, with a correction for spectrometer nonlinearity determined by Ne 1s, N_2 1s, and Ne 2s calibration lines at 870.37, 409.93, and 48.47 eV, respectively. The binding energies and full width at half-maximum (fwhm) values (eV) were 234.04 (3) and 1.11 (12) for Mo $3d_{5/2}$, 403.14 (4) and 1.08 (14) for N 1s, and 290.66 (3) and 1.16 (12) for C 1s. (The 2σ values of least-squares fits of the data are indicated parenthetically.)

Acknowledgment. This work was supported by the Director, Office of Basic Energy Sciences, Chemical Sciences Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

Registry No. $Mo[N(CH_3)_2]_4$, 100207-68-9; $Mo(CO)_6$, 13939-06-5; $Mo(CO)_5P(CH_3)_3$, 16917-96-7; $Mo(CO)_5P[N(CH_3)_2]_3$, 14971-43-8; $Mo(CO)_5P(OC_2H_5)_3$, 15603-75-5; $Mo(CO)_5P(OCH_3)_3$, 15631-20-6; $Mo(CO)_5PCl_3$, 19212-18-1; $Mo(CO)_5PF_3$, 15322-05-1; *cis*-$Mo(CO)_4[P(CH_3)_3]_2$, 16027-45-5; *trans*-$Mo(CO)_4[P(CH_3)_3]_2$, 30513-03-2; *fac*-$Mo(CO)_3[P(CH_3)_3]_3$, 19195-94-9; $Mo_2(O_2CH)_4$, 51329-49-8; $Mo_2(O_2CCH_3)_4$, 14221-06-8; $Mo_2(mhp)_4$, 67634-80-4.

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The ^{18}O Isotope Shift in ^{15}N NMR Spectroscopy. 2. Synthesis of $^{15}N,^{18}O$-Labeled Hydroxylamine Hydrochloride

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Received December 26, 1985

Substitution of oxygen-18 for oxygen-16 results in detectable upfield shifts on the NMR signals of many nuclei including ^{13}C, ^{31}P, and ^{15}N, when they are directly bonded to oxygen.[1-3] These upfield shifts provide a convenient and direct method of following enzymatic and nonenzymatic oxygen-exchange reactions occurring at carbon,[4,5] phosphorus,[2,6] and nitrogen.[3] The ^{18}O-induced isotope shift in ^{15}N NMR has been utilized in studies of the oxidation of ammonium ion to nitrite by a *Nitrosomonas* species[7] and nitrite to nitrate by *Nitrobacter agilis*,[8] but otherwise the technique has not yet found wide applications. This is due in part to the lack of a systematic study of the characteristics of the ^{18}O isotope shift in ^{15}N NMR spectra of various compounds. Consequently, we have undertaken the synthesis of various types of compounds containing N–O groupings with both ^{15}N and ^{18}O isotopic enrichment, as well as examination of their ^{15}N NMR spectral properties.

Studies on the synthesis of $^{15}N,^{18}O$ dual-labeled compounds are limited to sodium and silver nitrites, sodium nitrate, some oxides of nitrogen, and alkyl nitrites and nitrates.[9-11] Experiments

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0020-1669/86/1325-0876$01.50/0

on the exchange of oxygen between aliphatic/aromatic nitro compounds and water under acidic and alkaline conditions and at elevated temperatures revealed no detectable oxygen exchange under the conditions tested.[12] We employed a similar range of conditions in attempts to cause an exchange of oxygen between hydroxylamine and [^{18}O]water but observed no ^{18}O enrichment of the hydroxylamine. In contrast to the facile oxygen exchange of nitrite,[3] many such compounds having N–O bonds exhibit little or no oxygen exchange. Consequently, they must usually be synthesized by starting from ^{15}N,^{18}O-labeled precursors. Hydroxylamine can serve as a key intermediate in the synthesis of a variety of compounds, including many important heterocycles that have N–O groupings. Therefore, the synthesis of [^{15}N,^{18}O]hydroxylamine hydrochloride was undertaken.

Published procedures for the synthesis of hydroxylamine were examined for the synthesis of the dual-labeled compound. The reductions of aqueous $NaNO_2$ or KNO_2 by SO_2 to NH_2OH·HCl were examined for their utility in the synthesis of $^{15}NH_2{}^{18}OH$·HCl.[13,14] Although the chemical yields were high, the percentage of ^{18}O in the product was much less than expected. Under the conditions necessary for the reduction of nitrite by SO_2, the oxygen-18 label in nitrite exchanged with [^{16}O]water at a competitive rate. The use of [^{18}O]water as a reaction medium was prohibitively expensive. The reduction of aqueous $NaNO_2$ by zinc dust[15,16] in the presence of acetone, NH_4Cl, CO_2, or NH_3 and at different temperatures gave only 30–40% yields of acetoxime. The nitrosation of an active methylene-containing compound[17] and its subsequent hydrolysis to yield NH_2OH·HCl was tested with use of ethyl acetoacetate and acetylacetone. Although the yield of the initial oxime and the retention of the ^{18}O label were good, the method was not satisfactory because the yield of hydroxylamine hydrochloride following hydrolysis was only about 10%. Another scheme involving Victor Meyer reactions was examined.[18,19] Although complete retention of ^{18}O label was observed, the overall yield of $^{15}NH_2{}^{18}OH$·HCl based on $Na^{15}N^{18}O_2$ was only 52%. Considering the cost of ^{18}O and ^{15}N isotopes, the moderate overall yield of $^{15}NH_2{}^{18}OH$·HCl, as well as the number of steps involved in the synthesis, this approach was also judged unsatisfactory. Thus, among the various methods reported in the literature for the synthesis of NH_2OH·HCl, some procedures afforded high chemical yields with low percentages of ^{18}O, whereas other methods gave low to moderate yields, albeit with little or no loss of the ^{18}O label.

A major source of difficulty appeared to be the fact that the oxygen label of nitrite exchanged when the reduction was performed in aqueous solution. In order to prevent such exchange, we sought to develop a method employing nonaqueous solvents in order to achieve an efficient method of synthesizing $^{15}NH_2{}^{18}OH$·HCl of high isotopic purity. Borane in THF has been employed to reduce oximes[20] and alkyl nitronates[21] to the corresponding alkylhydroxylamines. More recently, it has been demonstrated that the same reagent in the presence of a catalytic amount of $NaBH_4$ reduces α,β-unsaturated nitro compounds to alkylhydroxylamines.[22] We therefore explored the possibility of reducing nitrite ion by using boron hydrides. Sodium borohydride was found to be ineffective in reducing $NaNO_2$. Tetramethylammonium nitrite was then prepared, stirred in dry THF, and treated with 1 M BH_3·THF. Although some complex unidentified products were present when the reaction mixture was worked up by hydrolysis and reaction with acetone, analyses by NMR and mass spectrometry provided no evidence for the formation of acetoxime.

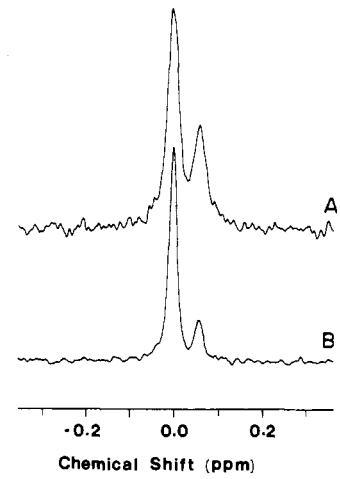

Figure 1. ^{15}N NMR spectra of ^{15}N-enriched acetoxime containing different percentages of oxygen-18: (A) 32% ^{18}O; (B) 17% ^{18}O. An upfield shift of 0.061 ± 0.001 ppm is observed upon replacement of ^{16}O by ^{18}O.

However, we found that an exothermic reaction ensued when $NaNO_2$, suspended in dry THF, was treated with borane–methyl sulfide (BMS). We examined the course of this reaction using ^{11}B NMR spectroscopy, with nitrite:BMS ratios of 1:0.67, 1:1, and 1:2 and measured the yield of NH_2OH (as the acetoxime) in each reaction. It was found that the reaction required 2 mol of BMS/mol of $NaNO_2$ reduced and that 1 mol of BH_4^- was formed (as evidenced by the quintet in the ^{11}B NMR spectra). With use of this method, the yield of $^{15}NH_2{}^{18}OH$·HCl was 74% (based on $Na^{15}N^{18}O_2$), with no loss of the ^{18}O label. The ^{18}O enrichment was demonstrated both by mass spectrometry and by ^{15}N NMR spectroscopy of the isolated acetoxime (Figure 1).

The ready availability of dual-labeled hydroxylamine, a useful inorganic ligand and key intermediate in the synthesis of organic complexing agents and heterocycles, as well as the further demonstration of the generality of the ^{18}O isotope shift in ^{15}N NMR spectroscopy, should be useful in numerous areas of chemistry and biochemistry.

Experimental Section

Materials. Sodium [^{15}N]nitrite (95 atom % ^{15}N, MSD Isotopes), [^{18}O]water (95+ atom % ^{18}O, MSD Isotopes), borane–methyl sulfide complex (BMS) (9.2 and 2 M, Aldrich), borane–tetrahydrofuran complex (1.0 M in THF, Aldrich), deuteriochloroform (99.8 atom % ^{2}H containing 1% Me_4Si, Aldrich), and deuterium oxide (99.8 atom % ^{2}H, Aldrich) were used. All other reagents were analytical grade. The ^{18}O isotopic enrichment was quantitated by ^{15}N NMR and by mass spectrometry using a Finnigan 4000 instrument equipped with a 9610 gas chromatograph and a Nova 4 data system.

NMR Spectra. A Varian XL-200 NMR spectrometer operating at 20.28 MHz was fitted with a 10-mm probe equilibrated at 24 °C for ^{15}N NMR analysis. A sweep width of ±250 Hz (quadrature phase detection), a 60° pulse angle, an 8K data block, and Waltz broad-band proton decoupling[23] were used. A resolution enhancement factor was applied to FID. A Varian FT-80 NMR spectrometer was used for ^{11}B NMR analysis at 25.517 MHz and for ^{13}C NMR analysis at 20 MHz. The instrument was fitted with a 10-mm probe equilibrated at 24 °C.

Preparation of Sodium [^{15}N,^{18}O]Nitrite. Sodium [^{15}N]nitrite (1 g; 95 atom % ^{15}N, Merck) and $NaNO_2$ (3 g) were dissolved in 8 mL of [^{18}O]water (40 atom % ^{18}O). The solution was cooled in an ice–salt mixture and was acidified by carefully adding 0.2 mL of concentrated HCl. The flask was stoppered and allowed to stand overnight at room temperature. After the mixture was cooled to −5 °C, the flask was opened, the solution was neutralized with solid NaOH (98 mg), residual [^{18}O]water was recovered under vacuum, and $Na^{15}N^{18}O_2$ was dried in the air.

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Synthesis of [^{15}N,^{18}O]NH_2OH·HCl. A magnetic stirring bar and finely powdered $Na^{15}N^{18}O_2$ (24% ^{15}N, 32% ^{18}O; 3.45 g, 50 mmol) were placed in an oven-dried 200-mL flask. This was flushed with nitrogen, and a rubber septum and a reflux condenser were attached. After 75 mL of dry THF was introduced, the addition of BMS (9.2 M, 11.2 mL, 103 mmol) was started at room temperature. The addition of BMS was stopped after every 5–10 drops in order to allow the reaction to proceed. The progress of the reaction was apparent from the smooth evolution of bubbles and a gentle refluxing of methyl sulfide on the walls of the flask. (It is important to add BMS slowly. In one of the trial experiments, violent frothing occurred when the addition was more rapid.) The addition of BMS was then continued dropwise to maintain a gentle refluxing of methyl sulfide. Stirring was continued overnight under a nitrogen atmosphere. The reaction mixture was cooled in an ice–salt mixture and hydrolyzed carefully with 20 mL of water, and the borohydride was decomposed by the addition of 20 mL of 6 N HCl, with care taken not to allow the temperature to rise above 5 °C. After the mixture was stirred for 20 min, acetone (5 mL) was added and stirring was continued for 10 min. The pH of the mixture was then brought to 8–9 by the careful addition of NaOH solution, and the reaction mixture was saturated with NaCl. The organic layer was separated, and the aqueous layer was washed three times with diethyl ether. The ether extract and the organic layer were mixed together and dried with $CaCl_2$, and the solvent was evaporated to obtain [^{15}N,^{18}O]acetoxime (3.03 g, 83%; 24% ^{15}N, 32% ^{18}O).

The labeled acetoxime (3 g) in a 50-mL flask was refluxed gently with 30 mL of 2 N HCl for 1 h, and the mixture was then distilled at atmospheric pressure. When the distillate was free of acetone, distillation was continued at reduced pressure until the volume was reduced to 3 mL. This solution was cooled in an ice–salt mixture, whereupon colorless crystals of hydroxylamine hydrochloride appeared. This was then lyophilized to obtain dry, crystalline $^{15}NH_2{}^{18}OH$·HCl (2.78 g; 24% ^{15}N, 32% ^{18}O), which was then recrystallized from absolute alcohol: yield 2.55 g (74%); mp 155 °C.

In separate experiments, the reduction of sodium nitrite suspended in THF was performed with use of BH_3·THF (1 M) as well as BMS (2 M) in THF; the yield of acetoxime was about the same in all the cases. However, it was necessary to conduct reductions with BH_3·THF at 0 °C because lower yields resulted when the reaction was done at room temperature.

Acknowledgment. This research was supported by DHHS NIH Grant GM 22933 from the National Institute of General Medical Sciences and by NSF Chemical Instrumentation Program Grant CHE-8010832.

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Correlations of ^{195}Pt–^{31}P Coupling Constants with Platinum–Ligand and Platinum–Platinum Bond Lengths in Platinum(I) Dimers and in Related Platinum(II) Complexes[1]

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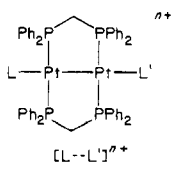
Received July 5, 1985

Dimeric platinum(I) complexes with bridging bis(diphenylphosphino)methane ligands, $[Pt_2(\mu\text{-dppm})_2(L)L']^{n+}$, or, as abbreviated here, $[L\text{--}L']^{n+}$, are now quite numerous.[2-7] Their



availability presents a rare opportunity to study the physical properties and reactivity of a metal–metal bond as a function of the ligands trans to it with a minimum of competing variables. For example, the reactions of diazomethane with various $[L\text{--}L']^{n+}$ complexes, forming methylene-bridged A-frames, vary considerably in rate[8] and even change mechanism[9] as a function of the terminal ligands. Similarly, the reactions of *cyclo*-octasulfur with various $[L\text{--}L']^{n+}$ complexes, forming sulfido-bridged A-frames, have been studied systematically.[9,10] We now wish to report several interesting trends of Pt–P coupling constants with Pt–L and Pt–Pt bond lengths in Pt(I) dimers, $[L\text{--}L']^{n+}$, and structurally related Pt(II) complexes, *trans*-$Pt(PR_3)_2(L)L'$. Some of these trends were suggested earlier, and we merely provide further supportive evidence from more recent data.

† Henry Gilman Fellow, Iowa State University, 1983–1984.

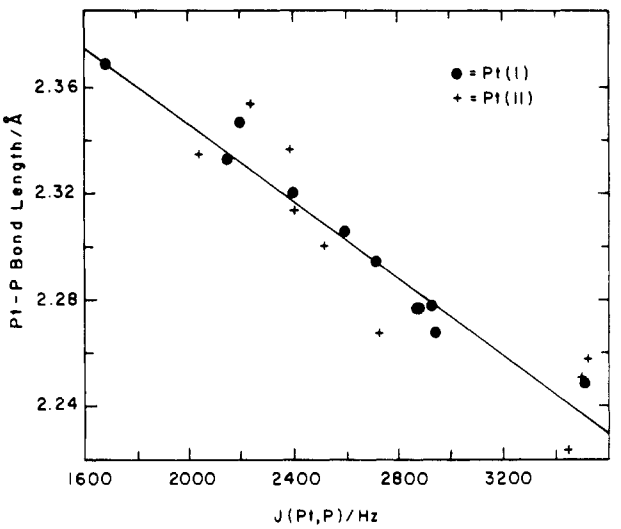


Figure 1. Pt–P bond length vs. ^1J(Pt,P) for dppm-bridged Pt(I) dimers (●) and Pt(II) compleces (+). The least-squares slope is extrapolated to $^1J(Pt_A,P_T)$ = 1680 Hz to estimate the bond distance in $[Pt_2(\mu\text{-dppm})_2(PPh_3)_2]^{2+}$.

Table I. Selected Pt–Cl Bond Lengths in *trans*-$[Pt(Y)Cl(PR_3)_2]^{n+}$

complex	Y–	Pt–Cl/Å	ref
trans-$[PtH(PEtPh_2)_2Cl]$	H–	2.422	12
$[Pt_2(\mu\text{-dppm})_2Cl_2]$	Cl–Pt–	2.405	13
$[Pt_2(\mu\text{-dppm})_2Cl(PPh_3)]^+$	Ph_3P–Pt–	2.403	7
$[Pt_2(\mu\text{-dppm})_2(CO)Cl]^+$	OC–Pt–	2.384	15
$[Pt(PEt_3)_3Cl]^+$	Et_3P–	2.366	16
trans-$[Pt(PEt_3)_2Cl_2]$	Cl–	2.249	17

Results and Discussion

Trans Influence of the dppm-Bridged Pt–Pt Bond. The influence[11] of trans ligands on ground-state properties can extend to the trans metal–ligand bond distance, the vibrational frequency or force constant, the NMR coupling constant between the metal and the trans-ligand donor atom, and a host of other parameters. As the trans influence of a ligand increases, the $M\text{–}L_{trans}$ bond

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