with both H^+ and PCl_5 (path a in Scheme I).

Tensimetric Titration. A tensimetric titration was performed on py·PCl_5 under conditions identical with those used for pz·PCl_5. (Compare parts a and b of Figure 1.) The failure of py·PCl_5 to react with HCl indicates that P–N bond rupture is probably not the mechanism by which pz·PCl_5 reacts with the first mole of HCl under heterogeneous conditions.

Raman Spectroscopy. The Raman spectrum of pz·PCl_5·HCl, recorded between 100 and 700 cm^{-1} is shown in Figure 3. Bands at 183, 362, and 460 cm^{-1} are pertinent to this discussion when compared to the spectra of pz·PCl_5[2] and py·PCl_5.[8] The a_1 ν(P–N) bands of these two compounds appear at 189 and 190 cm^{-1}, respectively. Because of similar relative intensity, we assign the 183 cm^{-1} band to the P–N mode of pz·PCl_5·HCl. Both pz·PCl_5 and py·PCl_5 display Raman bands at 351 (strongest band, a_1 $\nu(P\text{–}Cl_{eq})$) and 455 cm^{-1} (a_1 $\nu(P\text{–}Cl_{ax})$). Pz·PCl_5·HCl has two Raman bands of similar relative intensities at 362 (strongest band) and 460 cm^{-1}, which we assign to a_1 $\nu(P\text{–}Cl_{eq})$ and a_1 $\nu(P\text{–}Cl_{ax})$. The presence of these modes, and the fact that neither PCl_6^- [9] nor pyrazinium ion[10] display the same Raman pattern, argues in favor of part a in Scheme I.

Infrared Spectroscopy. Comparison of the spectra of pz·PCl_5·HCl and pz·PCl_5·DCl shows the following. pz·PCl_5·HCl has a band at 2110 cm^{-1} (broad), which is shifted to 1590 cm^{-1} in its deuteriated analogue. The frequency ratio ν(N–D)/ν(N–H) of 0.754 compares favorably with 0.768 for the deuterium-shifted N–H(D) band observed for pz·$H(D)^+Cl^-$ by Foglizzo and Novak.[10] Their infrared absorptions are at 2800 (N–H) and 2150 cm^{-1} (N–D). Our lower frequencies can be attributed to the decreased basicity of the second ring nitrogen. In the adduct, $[(D)H\text{–}NC_4H_4N\cdot PCl_5]^+$, the N–H(D) bond para to the N–P bond would be expected to exhibit a reduced N–H(D) stretching frequency compared to that of monoprotonated pyrazine. This is consistent with the pK_a values of 0.65 and –5.78 for the mono and diacid forms of pyrazine[11] and supports the contention that the species $[H\text{–}NC_4H_4N\cdot PCl_5]^+$ is present as postulated in the first step of path a of Scheme I.

(2) Reaction of HBr(g) with pz·PCl_5. Tensimetric titrations performed with HBr(g) yielded results parallel to the HCl reactions discussed above. (Figure 2b) The diprotonated adduct is not stable at room temperature, similar to the HCl addition product. However, a change in the color of the reaction mix occurred upon adding HBr(g) to pz·PCl_5. The chemistry responsible for the color change did not affect the tensimetric titration result, except that recovery of gas, added beyond a 2:1 mole ratio of HBr/pz·PCl_5, showed a quantitative excess of HCl, not HBr. A metathesis reaction occurs, which yields PBr_5 and HCl and is the subject of current investigation.

Conclusions

Under the conditions employed in these experiments (heterogeneous reaction between undissolved pz·PCl_5 and HX in CCl_4 and CH_2Cl_2), protonation of the para nitrogen atom of the pyrazine ring takes place. The attack of HX occurs in this manner, rather than to displace PCl_5 from the complex. The analogous complex, py·PCl_5, was found not to react with HX under conditions identical with those employed with pz·PCl_5. The P–N bond, although labile in these complexes when in solution, is inert to attack of the first mole of HX under the conditions reported herein.

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Comparative Matrix Isolation and Time-Resolved Infrared Studies on the Photochemistry of $MnRe(CO)_{10}$ and $Re_2(CO)_{10}$: Evidence for CO-Bridged $MnRe(CO)_9$

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We wish to report the results of an investigation into the photochemistry of $MnRe(CO)_{10}$ and $Re_2(CO)_{10}$ using both matrix isolation at 12 K and time-resolved IR spectroscopy of solutions at room temperature. For both molecules we have detected primary photoproducts involving loss of CO from the parent decacarbonyl. $MnRe(CO)_9$ has a bridging CO group while $Re_2(CO)_9$ does not. In hydrocarbon solution, $MnRe(CO)_9$ reacts with CO substantially faster than does $Mn_2(CO)_9$.

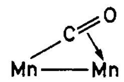
The photochemistry of organometallic compounds[1] has been extensively studied, and at present considerable interest is centered on dinuclear systems possessing metal–metal bonds.[2] However, only recently has it been recognized that there are two primary photochemical pathways for such compounds: (i) homolytic cleavage of the metal–metal bond to give radicals or (ii) dissociative loss of CO without rupture of the metal–metal bond. Evidence for these processes came originally from UV flash photolysis kinetic studies, which did not, however, provide much structural information about the intermediates. Definitive identification of these intermediates has required time-resolved IR spectroscopy, which in turn has made extensive use of IR data from matrix isolation experiments for interpretation of the spectra.[3]

A particularly well-studied problem is the photolysis of $Mn_2(CO)_{10}$, where conventional flash photolysis,[4] matrix isolation,[5,6] and time-resolved IR spectroscopy[7,8] have all been used. The two primary photochemical processes are

$$Mn_2(CO)_{10} \rightarrow 2Mn(CO)_5$$

$$Mn_2(CO)_{10} \rightarrow Mn_2(CO)_9 + CO$$

Detailed matrix isolation experiments[5,6] have shown that the CO-loss product $Mn_2(CO)_9$ has a novel structure involving an η^1, η^2 semibridging CO group (**1**).



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By comparison with $Mn_2(CO)_{10}$, relatively less is known about the photochemistry of $Re_2(CO)_{10}$. Two recent flash photolysis studies, one with UV detection[9] and the other with IR spectroscopy,[10] have shown that the $Re_2(CO)_{10}$ system is more complex than that of $Mn_2(CO)_{10}$ and that there is no evidence for a CO-bridged $Re_2(CO)_9$ species in solution.[10] Even less is known about the photochemistry of the mixed-metal carbonyl $MnRe(CO)_{10}$.

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Here we report new results on the photochemistry of both $MnRe(CO)_{10}$ and $Re_2(CO)_{10}$.

Experimental Section

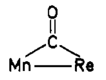
The matrix apparatus[11] and the photolysis equipment[12] have been described previously. All IR spectra of matrix-isolated species were obtained with a Nicolet MX-3600 FTIR interferometer and 1280 data system (32K data collection, 256K Fourier transform, i.e. ~0.7-cm^{-1} resolution). Matrices were prepared by slow deposition.[13] The time-resolved IR apparatus, (based on a UV flashlamp (15 μs fwhm) and a CW CO (IR) laser) has been described previously.[3,14] The UV lamp is flashed repeatedly, the transient IR signal is monitored at a series of discrete wavelengths, and an IR spectrum is constructed "point-by-point". The solution in the IR cell was changed after each flash of the UV lamp. $Re_2(CO)_{10}$ (Aldrich Chemicals), $Mn_2(CO)_{10}$ (Strem Chemicals Inc.), cyclohexane (BDH Aristar Grade), matrix gases (Messer Greisheim), and $MnRe(CO)_{10}$ (a gift from Professor J. A. Connor) were all used without further purification.

Results and Discussion

The UV irradiation (either broad band or $\lambda = 290 \pm 10$ nm) of the mixed dimer $MnRe(CO)_{10}$ isolated in argon matrices led to the formation of free CO (2140 cm^{-1}). At the same time, there was a decrease in the intensity of the IR bands of $MnRe(CO)_{10}$ and a corresponding growth of several absorptions due to *terminal* CO groups and of a single absorption, 1759.8 cm^{-1}, in the bridging-CO region (see Figure 1). Subsequent irradiation of the matrix with visible light ($\lambda > 400$ nm) caused a decrease in these new absorption bands and regeneration of the bands due to $MnRe(CO)_{10}$. The growth and decay behavior of the bands suggested that they were due to a *single* photoproduct, almost certainly $MnRe(CO)_9$.

$$MnRe(CO)_{10} \underset{\lambda > 400\ \text{nm}}{\overset{\text{UV}}{\rightleftharpoons}} MnRe(CO)_9 + CO$$

$MnRe(CO)_9$ presumably contains a bridging CO group, because of the IR band in the "bridging" region, but it is not clear whether the band is due to a "classical" (**2**) or a semibridging (**3**) CO



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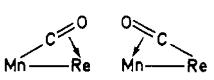

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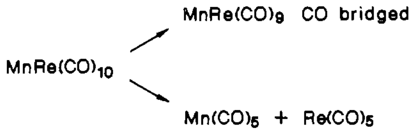
bridge. However, considering the similarity in wavenumber and intensity of the bands of $Mn_2(CO)_9$ and $MnRe(CO)_9$, it is probable that the CO bridges are also similar, i.e. **1** and **3**. Unfortunately, our attempts to confirm the presence of a semibridging CO group by polarization experiments on $MnRe(CO)_9$, analogous to those carried out[6] on $Mn_2(CO)_9$, were inconclusive.

Table I. Wavenumbers (cm^{-1}) of ν(CO) Bands of $MM'(CO)_9$ Species in Argon Matrices at 12 K

	$Mn_2(CO)_9$				
	a	*b*	*c*	$MnRe(CO)_9$	$Re_2(CO)_9$^{d}
ν(CO) terminal			2108.6	2116.2	
	2058	2058	2056.9		2065.9
	2037		2040.6 2036.2	2040.7 2036.6	
		2020	2024.7	2017.6	2014.7
		2006	2008.1	2010.8	1998.8
	1993	1994	1993.2	1989.7	1986.9
	1977	1977	1977.5	1973.0	
				1961.9	1955.1
				1944.2	1942.6
ν(CO) bridging	1764	1760	1765.1	1759.8	

[a] Reference 6. [b] Cyclohexane solution at room temperature.[7] [c] This work. [d] First photoproduct.

Time-resolved IR spectroscopy has already been used[7] to show that $Mn_2(CO)_9$ has the same structure in solution at room temperature as in low-temperature matrices.[5,6] We have performed similar time-resolved IR experiments on $MnRe(CO)_{10}$. Flash photolysis of $MnRe(CO)_{10}$ (5×10^{-4} M) in argon-saturated cyclohexane generated a species with an IR band at ~1765 cm^{-1} (see Figure 2a). The band maximum is at a slightly higher wavenumber than that of $Mn_2(CO)_9$ generated from $Mn_2(CO)_{10}$ under similar conditions (Figure 2b), but given the low resolution of time-resolved IR spectra, the difference is not large. However, the species generated from $MnRe(CO)_{10}$ is very much shorter lived[15] than $Mn_2(CO)_9$ (compare parts c and d of Figure 2) and its lifetime is further reduced in CO-saturated solution. By comparison with our matrix data (Figure 1) the IR band in solution is reasonably assigned to $MnRe(CO)_9$ with the same CO-bridged structure as in the matrix. In addition, we observed IR bands at 1985 cm^{-1} ($t_{1/2} \sim 50$ μs) and 1990 cm^{-1} ($t_{1/2} \sim 10$ μs), which were consistent with the $Mn(CO)_5$ and $Re(CO)_5$ radicals previously observed in the photolysis of $Mn_2(CO)_{10}$[7] and $Re_2(CO)_{10}$.[10] Thus, $MnRe(CO)_{10}$ also possesses two primary photochemical pathways.



Surprisingly, the photolysis of matrix-isolated $Re_2(CO)_{10}$ has not been studied previously. As with $MnRe(CO)_{10}$ the photolysis of $Re_2(CO)_{10}$ in argon matrices either with unfiltered UV or with a 290-nm filter leads to the ejection of CO, disappearance of parent $Re_2(CO)_{10}$, and the appearance of several new absorptions in the terminal CO region (see Table I). However, at no stage were any bands observed in the bridging CO region (in direct contrast to the photolysis of both $Mn_2(CO)_{10}$ and $MnRe(CO)_{10}$ in rigid matrices). These observations are in agreement with time-resolved IR studies of the photolysis of $Re_2(CO)_{10}$ in both solution and gas phases, which also failed to produce any evidence for transient products with bridging carbonyl groups. As with $Mn_2(CO)_{10}$ and $MnRe(CO)_{10}$, the photolysis of matrix-isolated $Re_2(CO)_{10}$ can be reversed by visible radiation ($\lambda > 400$ nm). We therefore assign the primary photoproduct as $Re_2(CO)_9$ with a structure consisting of one saturated and one unsaturated rhenium center (i.e. of the type $(CO)_5Re{-}Re(CO)_4$). Because of the unsymmetrical nature of such a molecule and hence the large number of CO infrared absorptions, it is not possible at present to assign a definitive structure to $Re_2(CO)_9$. It is nevertheless clear that $Re_2(CO)_9$ has a structure radically different from those proposed for $Mn_2(CO)_9$ and $MnRe(CO)_9$. The matrix photochemistry of $Re_2(CO)_{10}$ is complicated by a further process[16] occurring on irradiation with visible light that appears to promote the conversion of $Re_2(CO)_9$ into a second species, possibly a different isomer of $Re_2(CO)_9$ but still without a bridging CO group. These complications will be discussed in more detail elsewhere.[17]

Conclusion

These experiments again underline the complementary nature of matrix isolation and time-resolved IR spectroscopy. The presence of bridging CO groups in $Mn_2(CO)_9$ and $MnRe(CO)_9$[18] and the absence of such groups in $Re_2(CO)_9$ reflect the usual structure trends within a periodic group. What is perhaps rather

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(15) The decay rate of $Mn_2(CO)_9$ in our experiment is close to that previously reported by Church et al., under similar conditions. The decay of $MnRe(CO)_9$ was approximately 10 times faster than that of $Mn_2(CO)_9$.

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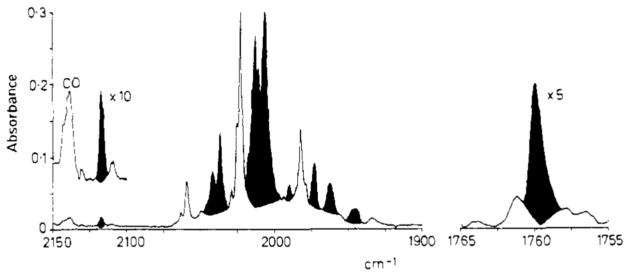

Figure 1. IR spectrum in the ν(CO) region after UV photolysis (λ = 290 ± 10 nm) of $MnRe(CO)_{10}$ in an argon matrix at 12 K (matrix ratio ~1:5000). Bands due to $MnRe(CO)_9$ are shown in black. Note that the low-wavenumber ν(CO) region is shown with expanded wavenumber and absorbance scales to emphasize the presence of a bridging CO group in $MnRe(CO)_9$.

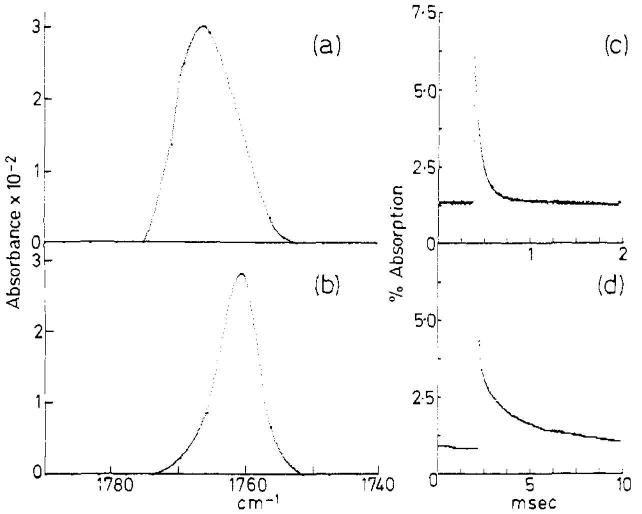

Figure 2. Time-resolved IR spectra in the ν(CO) bridging region (1790–1740 cm^{-1}) obtained 15 μs after UV flash photolysis (5 × 10^{-4} M cyclohexane solution, 25 °C): (a) $MnRe(CO)_{10}$; (b) $Mn_2(CO)_{10}$. Kinetic traces showing formation and decay: (c) $MnRe(CO)_9$; (d) $Mn_2(CO)_9$. These traces were measured at 1764.1 cm^{-1} for $MnRe(CO)_9$ and 1761.3 cm^{-1} for $Mn_2(CO)_9$. Note that the time scale for (d) is expanded with respect to that of (c).

less expected is the much greater reactivity of $MnRe(CO)_9$ than $Mn_2(CO)_9$. We are now investigating the photosubstitution reactions of these compounds both in solid matrices and in liquid xenon solution in an attempt to understand these differences in reactivity.

Acknowledgment. We thank the SERC, the Paul Instrument Fund of the Royal Society, NATO (Grant No. 591/83), and Applied Photophysics Ltd. for support. We are grateful to Dr. M. A. Healy, A. J. Dixon, J. G. Gamble, and J. W. Whalley for their help, to Professor J. A. Connor for a gift of $MnRe(CO)_{10}$, and to Dr. S. P. Church, Professor G. R. Dobson, Dr. I. R. Dunkin, Dr. C. J. Shields, and Professor E. Weitz for helpful discussions.

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Kinetics of Acid Hydrolysis of the Nitridotrisulfate Ion

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A number of nitrogen sulfonates are produced by reactions between bisulfite ions and nitrite ions in aqueous solution.[1] Thus, their chemistry is of interest in studies of aqueous aerosols in polluted air and in wet flue gas scrubbing systems. The hydrolysis reactions of nitridotrisulfate (NTS) and hydroxysulfamate (HSA) have not been thoroughly investigated. Sisler and Audrieth[2] observed the hydrolysis reaction of NTS at several temperatures:

$$N(SO_3)_3^{3-} + H_2O \rightarrow HN(SO_3)_2^{3-} + H^+ + SO_4^{2-} \quad (1)$$

They found that the NTS hydrolysis reaction occurred much more quickly than the iminodisulfate hydrolysis reaction but did not obtain any quantitative results. We have studied the hydrolysis of NTS over a temperature range of 283–333 K at solution pHs ranging from 4.1 to 6.7. The influence of ionic strength was determined over a range of μ = 0.03–0.5 M.

Experimental Section

The potassium salt of NTS was prepared by a modification of the method of Sisler and Audrieth.[2] A 12.5-g sample of potassium nitrite and 107 g of potassium sulfite were added to 100 mL of hot water, and sufficient concentrated HCl was added to adjust the solution to pH 7. The rest of the prepn. was carried out in the manner outlined by Sisler and Audrieth. The potassium salt of IDS was prepared for use as a reference by the hydrolysis of NTS, again using the method of Sisler and Audrieth. The compounds were stored in an evacuated desciccator at 0 °C. No noticeable deterioration of the compounds was observed over a period of several months.

Ion chromatography was used to monitor the hydrolysis reaction. A Dionex 2010i ion chromatograph with a conductivity detector was used in the measurements. Both hydrolysis reaction products, IDS and SO_4^{2-}, could be determined with this system. The procedure is described elsewhere in more detail.[3] With the system used, the response to SO_4^{2-} was linear up to 4 × 10^{-4} M and the response to IDS was linear up to 1 × 10^{-4} M. In the experiments done, SO_4^{2-} was usually the only reaction product monitored because of its greater range of linearity. When IDS measurements were made, they agreed well with the SO_4^{2-} measurements.

Solutions for the hydrolysis runs were prepared by dissolving the NTS salt in deionized water made alkaline with a small amount of NaOH. These were then mixed with buffer solutions, also prepared from deionized water, to initiate the hydrolysis reaction. The potassium salt of NTS has limited solubility at room temperature (<0.02 M). The concentrations of NTS in the reacting solutions ranged from 1 × 10^{-3} to 10 × 10^{-3} M. Most runs were done with a NTS concentration of 2.5 × 10^{-3} M to allow the ionic strength to be kept at a fairly low value. Buffers used included acetate, oxalate, phosphate, phthalate, malonate, and succinate salts. The choice of buffer did not appear to influence the hydrolysis rate. Most runs were done with a buffer concentration of 0.025 M.

The pH values of the reaction mixtures at 25 °C were measured with a pH meter that was calibrated daily with standard buffer solutions. For pH measurements at other temperatures, correction factors for the pH meter calibrated at 25 °C were obtained. This was done by measuring acetate and oxalate buffer solutions and adjusting for changes in the ionization constants with temperature.[4]

After the NTS and buffer solutions were mixed to initiate the hydrolysis process, samples were withdrawn periodically for analysis. The samples were diluted 10:1 with water or 5 × 10^{-3} M sodium carbonate solution and injected into the ion chromatograph. The samples were diluted to ensure that the concentration of the reaction product to be measured was within the range of linearity of the instrument. The sodium carbonate solution was used as a diluting agent when there was concern that a significant amount of hydrolysis would occur if the sample was diluted with water. Injections of 1 × 10^{-4} M solutions of SO_4^{2-} or IDS were interspersed with injections of the diluted hydrolysis run samples to allow quantitative measurement of the reaction products. The initial concentration of the NTS was checked by allowing the hydrolysis reaction to go to completion and measuring the final concentrations of the products.

Results and Discussion

Generally, the first few measurements of the reacting solutions were used in calculating the hydrolysis reaction rate. The pH decrease in the solutions was sufficient to cause a slightly larger hydrolysis rate at the end of the run than at the beginning of the run. The hydrolysis rate dependence on the concentrations of NTS and H$^+$ were investigated to determine if the assumed first-order

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