$MSO_3F$ is 1:1, the solids are best described as $LiBr(SO_3F)_2$ and $NaBr(SO_3F)_2$. The anion, $Br(SO_3F)_2^-$, is responsible for the yellow color of the salts.

When one attempts to obtain the corresponding salt of potassium or rubidium by evaporating $BrSO_3F$ from a solution containing dissolved potassium or rubidium fluorosulfate, the solid which crystallizes from the solution is $KSO_3F$ or $RbSO_3F$. This does not mean that the $Br(SO_3F)_2^-$ is absent from the liquid phase. The close similarity of Raman spectra for the liquid solutions of fluorosulfates of all the alkali metals in $BrSO_3F$ suggests that all contain similar species. Apparently the solubility relationships favor crystallization of the fluorosulfate in two cases and of the bis(fluorosulfato)-bromate(I) in three cases.

The relatively slow rate of removal of $BrSO_3F$ from solutions having a $BrSO_3F:MSO_3F$ molar ratio greater than 1 indicates some interaction. A comparison of the pumping rates indicates that cesium fluorosulfate causes the greatest reduction in volatility of the solvent while lithium fluorosulfate causes the least.

**Acknowledgment.** This work was supported in part through a fellowship from the IBM Co. to one of the authors, W. M. J. The nmr spectra were run with the assistance of B. J. Nist and the Raman spectra with the help of Dr. John W. Macklin.

**Registry No.** $NaSO_3F$, 14483-63-7; $BrSO_3F$, 13997-93-8; $LiSO_3F$, 13453-75-3; $LiBr(SO_3F)_2$, 40813-81-8; $NaBr(SO_3F)_2$, 40813-82-9.

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Contribution from the Department of Chemistry,
University of Utah, Salt Lake City, Utah 84112

# Preparation and Properties of Bromochlorofluorophosphine and Improved Syntheses for Dimethylaminochlorofluorophosphine and Dimethylaminobromofluorophosphine

R. G. Montemayor and R. W. Parry*

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Dimethylaminobromofluorophosphine, $(CH_3)_2NPFBr$, was first prepared in 1968 by Clune and Cohn[1] using the reaction between $F_2PNR_2$ and BrCN; the equation is

$$2(CH_3)_2NPF_2 + BrCN \rightarrow (CH_3)_2NPF_3CN + (CH_3)_2NPFBr$$

The two products could not be separated by distillation; chromatographic techniques were required. For this reason, the procedure cannot be recommended as an effective synthetic route to the compound $(CH_3)_2NPFBr$.

The related chloro compound, $(CH_3)_2NPFCl$, was described by Roesky in 1969[2] as one of the products of the reaction between $(CH_3)_2NPCl_2$ and $SbF_3$. Reported yields were low (6.3%). In recent studies carried out in this laboratory, the compounds $(CH_3)_2NPFX$ (X = Cl or Br) have been synthesized in good yields (above 80%) and in high purity by methods described herein. The ready availability of these compounds has made it possible to synthesize, isolate, and characterize the formerly unavailable compound PFClBr.[3]

**Synthesis of $(CH_3)_2NPFX$.** The compound $(CH_3)_2NPFX$, where X = Cl or Br, has been prepared by two general reactions. (a) The first synthesis is based on the equation

$$[(CH_3)_2N]_2PF + 2HX \rightarrow (CH_3)_2NPFX + [(CH_3)_2NH_2]^+X^-$$

In this process, the ratio of HX to $[(CH_3)_2N]_2PF$ is critical. With a ratio of four HX to one $[(CH_3)_2N]_2PF$, $PFX_2$ is the major product.[4] If, however, the ratio is held to two HX per fluorophosphine, the compound $(CH_3)_2NPFX$ (X = Cl or Br) can be prepared in yields of about 85%. The process is effective for the chloro compound; however, it is difficult to separate $(CH_3)_2NPF$*Br* from any unreacted $[(CH_3)_2N]_2PF$ by fractional distillation. (b) The second synthesis is based on the equation

$$2(CH_3)_2NH + PFX_2 \rightarrow (CH_3)_2NPFX + [(CH_3)_2NH_2]^+X^-$$

The $PFX_2$ needed as a reactant is easily prepared by the action of an excess of HX on $[(CH_3)_2N]_2PF$. Amines such as piperidine and alkylated piperidines have also been used in this process in place of dimethylamine.[5]

While the first process (a) is more direct and is somewhat simpler than the second one (b), it is more difficult to separate the products and reactants of (a) through vacuum line distillation. On the other hand, the reaction between $PFX_2$ and the secondary amine gives the desired product in an easily purified form and in high yield. If a pure product is desired, the second process involving $PFX_2$ is recommended.

**Synthesis and Properties of PFClBr.** The compound PFClBr can be made by the reaction

$$(CH_3)_2NPFCl + 2HBr \rightarrow PFClBr + [(CH_3)_2NH_2]^+Br^-$$

The new compound melts at −122° and appears as a liquid at −120°, but its vapor pressure and boiling point could not be determined accurately because of increasing disproportionation with an increase in temperature. The pertinent equation is

$$2PFClBr \rightarrow PFCl_2 + PFBr_2$$

The $^{19}F$ nmr spectrum of PFClBr (dissolved in $CFCl_3$ at −40°) shows the expected doublet ($J_{PF}$ = 1300 Hz, $\delta$ 64.6 ppm from $CFCl_3$). Small doublets for $PFCl_2$ ($J_{PF}$ = 1320 Hz, $\delta$ 57.5 ppm from $CFCl_3$) and $PFBr_2$ ($J_{PF}$ = 1300 Hz, $\delta$ 72.2 ppm from $CFCl_3$) also appeared as a result of the disproportionation process. The $^{31}P$ nmr spectrum of PFClBr dissolved in TMS also displayed the expected doublet ($J_{PF}$ = 1300 Hz, $\delta$ −233 ppm from 85% $H_3PO_4$) along with small doublets for $PFCl_2$ and $PFBr_2$. The infrared spectrum of PFClBr shows absorptions assigned to the P-F stretch at 840 $cm^{-1}$, to the P-Cl stretch at 515 $cm^{-1}$, and to the P-Br stretch at 421 $cm^{-1}$. Delwaulle and Francois[3] had assigned Raman lines in a mixture of $PFBr_2$ and $PFCl_2$ to PFClBr although no separation of the product was achieved. Their assignments are $\nu_{P-F}$ 822 $cm^{-1}$, $\nu_{P-Cl}$ 500 $cm^{-1}$, and $\nu_{P-Br}$ 415 $cm^{-1}$. The mass spectrum shows a very strong parent peak at $m/e$ 166. This is assigned to $PF^{37}Cl^{79}Br^+$ and to $PF^{35}Cl^{81}Br^+$. Other peaks are listed in the Experimental Section. The foregoing instrumental data leave no reasonable doubt as to the identity of PFClBr.

**Nmr Correlations. (a) Fluorine-19 Resonance.** The chemical shift values for $^{19}F$ nmr spectra in the series $(CH_3)_2NPFX$ are as follows: X = F, $\delta$ 66 ppm; X = Cl, $\delta$ 69.5 ppm; X = Br, $\delta$ 75.6 ppm (all measured from $CFCl_3$). The shield-

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[1] J. E. Clune and K. Cohn, *Inorg. Chem.*, 7, 2067 (1968).

[2] H. W. Roesky, *Inorg. Nucl. Chem. Lett.*, 5, 891 (1969).

[3] The compound PFClBr has never been isolated as a distinct species although M. F. Delwaulle and F. Francois, *Acad. Sci.*, 223, 796 (1946), reported seeing its Raman spectrum in a mixture of $PFBr_2$ and $PFCl_2$.

[4] J. G. Morse, Doctoral Dissertation, University of Michigan, 1966.

[5] R. G. Montemayor, Doctoral Dissertation, University of Michigan, 1970.

ing is greatest for X = Br and least for X = F, revealing a trend which is qualitatively consistent with the usual behavior of binary fluorides where the shielding of F atoms decreases with an increase in the electronegativity of the group attached to the fluorine.[6] The same trend is also indicated by the $^{19}F$ chemical shift values for the series $PFX_2$. Values are $PF_3$, δ 34.2 ppm;[7] $PFCl_2$, δ 55.9 ppm;[8] and $PFBr_2$, δ 70.4 ppm,[8] all measured from $CFCl_3$. Neither trend is linear with electronegativity values, but both are almost linear with the number of the chemical period of the halogen in the sequence F, Cl, Br.[9] On the basis of this simple correlation, it is possible to estimate the $^{19}F$ chemical shift expected for PFClBr. The calculated value, δ, is 63.2 ppm. The observed value is 64.6 ppm from $CFCl_3$, confirming the identity of the species.

**(b) Phosphorus-31 Resonance.** The shielding trends (chemical shifts) for $^{31}P$ are diametrically opposite to those noted for $^{19}F$. In the series $R_2NPFX$ the shielding is *greatest* for X = F and *smallest* for X = Br. The values are $(CH_3)_2NPF_2$, δ −143 ppm;[10] $(CH_3)_2NPFCl$, δ −172 ppm; $(CH_3)_2NPFBr$, δ −187 ppm (all measured from orthophosphoric acid). For the related series $PF_2X$ the $^{31}P$ shielding is also greatest for X = F and smallest for X = I. Values are $PF_3$, δ −97 ppm;[11] $PF_2Cl$, δ −178 ppm;[8] $PF_2Br$, δ −195 ppm;[4] and $PF_2I$, δ −242 ppm.[12] The trend is consistent, but not linear, a common observation in $^{31}P$ chemical shift values. The values for the series $PFX_2$ are $PF_3$, δ −97 ppm; $PFCl_2$, δ −224 ppm;[8] and $PFBr_2$, δ −255 ppm.[8] If these values are extrapolated parallel to the curves for $PF_2X$, one can estimate a value of −232 for PFClBr. The observed value is −233.

A number of authors have developed semiempirical methods for calculating chemical shift values of $^{31}P$ in the phosphorus halides and related species. Gutowsky and Larman[13] calculated the isotropic paramagnetic contribution to the shielding term of "A" in $AX_3$ molecules. According to their model, Br makes the greatest negative contribution to the $^{31}P$ chemical shift and F the least in the series $PF_3$, $PCl_3$, $PBr_3$. In the series $PCl_3$, $PCl_2Br$, $PClBr_2$, and $PBr_3$, the contribution of Br is greater than Cl. They estimated that changing one Cl for a Br would bring a change of about −6.2 ppm in the chemical shift for $^{31}P$. This would give an estimated value of −230 ppm for PFClBr as compared to the observed value of −233 ppm.

In 1966 Van Wazer and Letcher[14] proposed an equation for calculating $^{31}P$ chemical shift values. Using this equation[15] and the values summarized, one can calculate a $^{31}P$ chemical shift of −232 ppm for PFClBr which again compares well with the measured value of −233 ppm. Other calculated and observed values (ppm) are: $PFClN(CH_3)_2$, calcd −175, obsd −172; $PFBrN(CH_3)_2$, calcd −185, obsd −187. One can also estimate the value of $PFBrN(CH_3)_2$ from the measured value of $PFClN(CH_3)_2$ by using the empirical Gutowsky–Larman constant of 6 for replacement of a Cl by a Br. Using this technique, one estimates a value of −178 ppm for $PFBrN(CH_3)_2$. Agreement with experiment is only fair.

## Experimental Section

**(a) Equipment.** Standard high-vacuum techniques were used throughout. Mass spectra were recorded on a Perkin-Elmer Model 279 mass spectrometer. Infrared spectra were obtained either with a Beckman IR-20 or a Beckman IR-12 using a 70-mm path length gas cell with KBr windows. The nmr spectra were recorded on a Varian HA 56/60 instrument (56 MHz for $^{19}F$, 60 MHz for $^1H$) and on a Varian XL-100 operating at 40.5 MHz for $^{31}P$. X-Ray powder patterns were taken with a G.E. unit using a Cu target and operating at 35 kV and 25 mA.

**(b) Materials.** The $[(CH_3)_2N]_2PF$ was prepared by the method of Fleming and Parry.[16] $PFCl_2$ and $PFBr_2$ were prepared by the reaction between $[(CH_3)_2N]_2PF$ and either HCl or HBr as described by Morse.[4] $(CH_3)_2NH$, HCl, and HBr were obtained from the Matheson Co.

**(c) Synthesis of $(CH_3)_2NPFCl$. 1. From $[(CH_3)_2N]_2PF$ and HCl.** A 19.7-mmol sample of $[(CH_3)_2N]_2PF$ was mixed with a 40-mmol quantity of HCl in a 1-l. reaction bulb at room temperature. White clouds of $[(CH_3)_2NH_2]^+Cl^-$ began to form almost immediately. After the system had been standing about 30 min, the volatile products were fractionated through traps held at −45, −78, and −196°. A 2.13-g sample of $(CH_3)_2NPFCl$ (84% yield) was trapped at −78°; unreacted $[(CH_3)_2N]_2PF$ was retained at −45°; and HCl was stopped at −196°. The white solid remaining in the reaction flask was identified as $[(CH_3)_2NH_2]^+Cl^-$ by its X-ray powder pattern. The $(CH_3)_2NPFCl$ had an infrared spectrum identical with that reported by Roesky.[2] The mass spectrum of $(CH_3)_2NPFCl$ is given below as *m/e*, assignment, relative intensity: 131, $(CH_3)_2NPF^{37}Cl^+$, 8.5; 129, $(CH_3)_2NPF^{35}Cl^+$, 25.4; 94 $(CH_3)_2NPF^+$, 100; 87, $PF^{37}Cl^+$, 8.5; 85, $PF^{35}Cl^+$, 25.4; 50, $PF^+$, 27.1; 44, $(CH_3)_2N^+$, 98; 37, $^{37}Cl^+$, 1.7; 35, $^{35}Cl^+$, 8.5; 31, $^{31}P^+$, 13.6; 19, $^{19}F^+$, 5.1; 15, $CH_3^+$, 30.5; 14, $CH_2^+$ or $^{14}N^+$, 8.5. The $^{19}F$ nmr showed a 1:1 doublet at δ 69.5 ppm from $CFCl_3$ ($J_{PF}$ = 1165 Hz), each member of which is split into a septet ($J_{HCNPF}$ = 3.5 Hz). The proton nmr spectrum gave a 1:1 doublet at δ −2.7 ppm from TMS ($J_{PNCH}$ = 10.8 Hz) with each member being split into a further doublet ($J_{FPNCH}$ = 3.5 Hz). The $^{31}P$ spectrum showed a broad 1:1 doublet at δ −172 ppm from OPA ($J_{FP}$ = 1170 Hz). The melting point of $(CH_3)_2NPFCl$ was −85° as determined by the Stock plunger method.

**2. From $PFCl_2$ and $(CH_3)_2NH$.** A 15.9-mmol sample of $PFCl_2$ was condensed into a 1-l. reaction bulb, equipped with a stopcock. This was followed by a 30-mmol sample of $(CH_3)_2NH$. After 30 min at 25° the volatiles were fractioned as before. A 1.9-g sample of $(CH_3)_2NPFCl$ (95% yield) was recovered from the −78° trap. A small amount of unreacted $PFCl_2$ was found in the −196° trap.

**(d) Synthesis of $(CH_3)_2NPFBr$. 1. From $[(CH_3)_2N]_2PF$ and HBr.** The procedure was identical with that for $(CH_3)_2NPFCl$ except that in the fractionation process both $(CH_3)_2NPFBr$ and unreacted $[(CH_3)_2N]_2PF$ were retained in the −45° trap. Attempts to separate the two by trap-to-trap distillation were not successful. HBr was recovered in the −196° trap. The yield was estimated from the HBr used as about 80%. This procedure is not satisfactory for preparing pure $(CH_3)_2NPFBr$.

**2. From $PFBr_2$ and $(CH_3)_2NH$.** The conditions were essentially the same as those described for the preparation of $(CH_3)_2NPFCl$ except that the desired product was retained in the −45° trap in 85% yield. Its mass spectrum and infrared spectrum were identical with

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(6) J. W. Emsley, J. Feeney, and L. H. Sutcliffe, "High Resolution Nuclear Magnetic Resonance Spectroscopy," Vol, 3, Pergamon Press, New York, N. Y., 1966, p 874.

(7) E. L. Muetterties and W. D. Phillips, *J. Amer. Chem. Soc.*, **81**, 1084 (1959).

(8) A. Muller, E. Nicke, and O. Glemser, *Z. Anorg. Allg. Chem.*, **350**, 256 (1967).

(9) The paramagnetic term is the dominant one in fluorine chemical shifts, and absolute correlations are not accurately calculated now.[6] This simple correlation with period would appear to sum up combination effects in an acceptable manner at present.

(10) S. Fleming, Doctoral Dissertation, University of Michigan, 1963.

(11) H. S. Gutowsky, D. W. McCall, and C. P. Slichter, *J. Chem. Phys.*, 21, 279 (1953).

(12) R. W. Rudolph, J. G. Morse, and R. W. Parry, *Inorg. Chem.*, **5**, 1464 (1966).

(13) H. S. Gutowsky and J. Larman, *J. Amer. Chem. Soc.*, **87**, 3815 (1965).

(14) (a) J. H. Letcher and J. R. Van Wazer, *J. Chem. Phys.*, **44**, 815 (1966); (b) J. R. Van Wazer and J. H. Letcher, *Top. Phosphorus Chem.*, **5**, 216 (1967).

(15) The equation of Van Wazer and Letcher[14] is $\delta = 11{,}828.5 - 7940k_1\delta - 149k_2n_\pi$ where $k_1$ is a constant equal to 0.972 for triply connected phosphorus, $k_2$ is a constant equal to 2.80 for triply connected phosphorus, and $n_\pi$ is the total occupation of the phosphorus $d_\pi$ orbitals in electrons per P atom. Values used are: PFClBr = 0.217; $PFClNR_2$ = 0.100; $PFBrNR_2$ = 0.123. δ is a semiempirical chemical shift parameter for the contribution of the p orbitals. The value is a rather complex function of substituent electronegativity. Values from ref 14 are: PFClBr = 1.551; $PFClNR_2$ = 1.550; $PFBrNR_2$ = 1.550. Electronegativity values used are: F = 3.93; Cl = 3.15; Br = 2.94; $-NR_2$ = 3.10. Bond angles of 102° were used.

(16) S. Fleming and R. W. Parry, *Inorg. Chem.*, **11**, 1 (1972).

those reported by Clune and Cohn.[1] The $^{19}F$ nmr showed a 1:1 doublet at δ 75.6 ppm from $CFCl_3$ ($J_{PF} = 1170$ Hz), each member of which was split into a septet ($J_{HCNPF} = 3.5$ Hz). The proton nmr spectrum showed a doublet (δ −2.8 ppm from TMS; $J_{PNCH} = 13$ Hz), each member of which was split into a second doublet ($J_{FPNCH} = 3$ Hz). The $^{31}P$ spectrum showed a broad doublet ($J_{FP} = 1170$ Hz; δ = −187 ppm from OPA).

(e) **Synthesis of PFClBr.** A 6.7-mmol sample of $(CH_3)_2NPFCl$ was mixed with a 13.7-mmol sample of HBr in a 500-ml gas reaction flask equipped with a stopcock. The system was warmed gradually to room temperature. After the reaction mixture had been standing 15 min, the volatiles were fractionated through traps held at −78, −126, and −196°. The PFClBr was retained in the −126° trap in 41% yield, while unreacted $(CH_3)_2NPFCl$ was found in the trap at −78°. Some $PF_3$, HBr, HCl, and $SiF_4$ were found in the −196° trap. White solid, identified as $[(CH_3)_2NH_2]^+Br^-$ by its X-ray powder pattern, remained in the reaction vessel. All properties of PFClBr except its mass spectrum have been given in an earlier section. The mass spectrum is again reported as *m/e*, assignment, peak intensity. The values are 168, $PF^{37}Cl^{81}Br^+$, 3.7; 166, $PF^{37}Cl^{79}Br^+$ and $PF^{35}Cl^{81}Br^+$, 16.8; 164, $PF^{35}Cl^{79}Br^+$, 13.0; 131, $PF^{81}Br^+$, 9.2; 129, $PF^{79}Br^+$, 9.2; 112, $P^{81}Br^+$, 1.8; 110, $P^{79}Br^+$, 1.8; 87, $PF^{37}Cl^+$, 33.3; 85, $PF^{35}Cl^+$, 100; 81, $^{81}Br^+$, 36.1; 79, $^{79}Br^+$, 36.1; 68, $P^{37}Cl^+$, 1.9; 66, $P^{35}Cl^+$, 8.1; 50, $PF^+$, 16.7; 37, $^{37}Cl^+$, 2.5; 35, $^{35}Cl^+$, 7.8; 31, $^{31}P^+$, and 19, $^{19}F^+$, 1.4. In addition, peaks assignable to $PFCl_2$ and $PFBr_2$ were observed. When the same reaction was carried out at −78° in a sealed reaction tube for 16 hr, the yield as estimated from the recovered $(CH_3)_2NPFCl$ was close to 90%.

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**Registry No.** $(CH_3)_2NPFCl$, 29246-85-3; $[(CH_3)_2N]_2PF$, 1735-82-6; $PFCl_2$, 15597-63-4; $(CH_3)_2NH$, 124-40-3; $(CH_3)_2NPFBr$, 21475-62-7; $PFBr_2$, 15597-39-4; PFClBr, 20464-11-3.

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Contribution from the Department of Inorganic and Analytical Chemistry, The Hebrew University of Jerusalem, Jerusalem, Israel

## Molybdenum(V) in Aqueous Solution

M. Ardon* and A. Pernick

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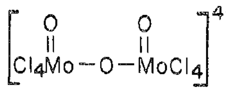
The role of molybdenum in the activity of various enzymes has renewed interest in the coordination chemistry of Mo(V). The structures of binuclear complexes of Mo(V) with oxalate, xanthate, and other chelating groups were elucidated;[1] yet, surprisingly little is known about the noncomplexed species of Mo(V) in aqueous solution.

Almost all the investigations concerned with the solution chemistry of Mo(V) were conducted in hydrochloric acid solutions. The well-characterized "Klason's salt"[2] $(NH_4)_2[MoOCl_5]$ dissolves in concentrated HCl (>10 *M*) without spectral change, and it is generally agreed[2] that the main species in this solution is the paramagnetic ion $MoOCl_5^{2-}$. Upon dilution of the solution to ~6 *M* HCl, another paramagnetic ion is formed for which structures I[2,3] and II[4] were suggested. Further dilution of this solution to <2 *M* HCl transforms the Mo(V) species into a diamagnetic ion. The two structures attributed to this diamagnetic ion are structure I by Colton and Rose[4] and a tetramer $(MoO_2^+)_4$ by Souchay.[5]



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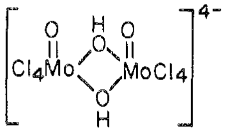

II

The results of the investigation reported here show that the main Mo(V) species in dilute HCl, as well as in $HClO_4$ and other acids, is a binuclear *cation* charged +2 which is not coordinated to chloride. This ion exists in all acidic aqueous solutions of Mo(V) in which complex formation does not occur.

Two different preparative methods were used to obtain Mo(V) solutions. (a) Klason's salt solution was prepared by dissolving $MoO(OH)_3$ in concentrated HCl. This solution was diluted with water to a HCl concentration of 1 *M*. (b) Molybdic(VI) acid dissolved in $HClO_4$ (1 *M*) was reduced electrolytically. Both solutions were yellow orange and the absorption spectra of each had maxima at 254 and 293 mμ. The perchloric acid solution was free of $Cl^-$ ions (no electrolytic reduction of $ClO_4^-$ took place). Each solution was absorbed on a cation exchange column (Dowex 50 X2 acid form). The yellow Mo(V) species formed a distinct band on each column and was eluted with 1 *M* $HClO_4$. This ion exchange behavior indicated a minimum charge of +2. The oxidation state of Mo in the eluted solutions was determined by oxidation to Mo(V) with excess $Fe^{3+}$ and back titration with permanganate. The 3:1 permanganate ratio for titration of an aliquot before and after reduction with a Jones reductor to Mo(III) confirmed the +5 oxidation state. The absorption spectra of the ion eluted from each column were identical with the spectra of the original solutions. The molar absorption was $\epsilon_{254} = 1999$ and $\epsilon_{293} = 1727$. No change occurred in the spectrum when the perchloric acid concentration was increased to 6 *M*, indicating the stability of the ion in strong acid media.[5] Cryoscopic measurements of Mo(V) were conducted in a eutectic perchloric acid solvent (5.27 *M*). The freezing point lowering of the solvent was approximately one-half of that expected for a mononuclear ion.[6] A solution containing 0.02 g-atom of Mo per kilogram of solvent lowered the freezing point by 0.052°. This corresponds to a molal depression of 2.6°, compared to 5.2° observed for mononuclear ions.

This result indicates that the Mo(V) ion is binuclear. This conclusion was confirmed by a determination of the charge per Mo atom and the charge per ion, according to the method of Cady and Connick.[7] Using acidified $La^{3+}$ solutions as eluent, the charge per Mo atom derived from six determinations was 1.0 ± 0.1. The charge per ion derived from three batch experiments was +1.73 ± 0.05. This ionic charge is well within the accuracy of the method for a +2 ion.[7]

No definite conclusion can be drawn from these data about the detailed structure of the binuclear dipositive Mo(V) ion but we favor structure III. This structure is expected to be diamagnetic, like the oxalato complex, which is structurally related to it[8] (structure IV).

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