An electrospray mass spectrometric study of methylmercury(II) nitrogen base derivatives and their ligand exchange reactions

Allan J. Canty
Chemistry Department, University of Tasmania, Hobart, Tas. 7001 (Australia)

Ray Colton*
Chemistry Department, La Trobe University, Bundoora, Vic 3083 (Australia)

(Received June 30, 1993; revised August 27, 1993)

Abstract

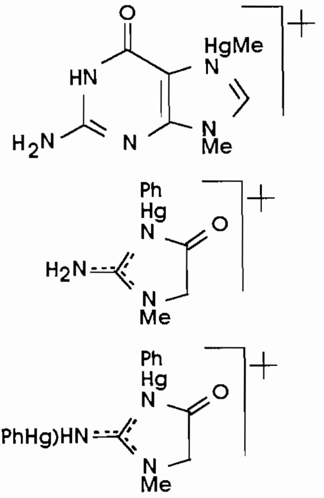
Electrospray mass spectra have been observed for solutions of a number of methylmercury(II) complexes of the type $[MeHgL]^+$ (L = neutral nitrogen base, e.g. *N*-methylpyrazoles, bis(pyridin-2-yl)methane, etc.) and PhHg(II) complexes of creatinine and in all cases the intact ion was observed. Electrospray mass spectrometry is a convenient technique with which to investigate ligand exchange reactions in solution, and the reactions of the *N*-methylpyrazole complex $[MeHg(C_4H_6N_2)]^+$ have been studied with a variety of reagents including other nitrogen bases, phosphines and phosphine sulfides and selenides.

Key words: Mass spectrometry; Mercury complexes; Alkyl complexes

Introduction

Methylmercury(II) is important in the environment since it is the end product of microbiological processes involving many forms of Hg(II) [1–3]. MeHg(II) prefers to bind to one other ligand to give linear complexes and examples of monodentate nitrogen base ligands which give such linear MeHg(II) complexes include *N*-methylpyrazoles and 3,5-dimethylpyridine [4, 5]. Polydentate nitrogen donor ligands often induce higher coordination numbers than the classic linear arrangement in the solid state, although the geometries at mercury are irregular and are based on only one strong Hg–N interaction. Ligands which are known from crystallographic studies to give high coordination numbers for MeHg(II) include bidentate bis(pyridin-2-yl)methane (py_2CH_2) [6], and the tridentate ligands 4,4′,4″-triethylterpyridyl (Et_3terpy) [6], and tris(pyridine-2-yl)methanol (py_3COH) [7]. Complexes of py_2CH_2, Me_3terpy, py_3COH and the related bidentate ligands 4,4′-diethylbipyridyl (Et_2bipy) [6] and bis(*N*-methylimidazol-1-yl)methanol (mim_2CHOH) [8] are included in this study, together with a 9-methylguanine complex [9] and PhHg(II) complexes of creatinine [10, 11]. Structures of the latter complexes are shown in Scheme 1.

Electrospray mass spectrometry (ESMS) provides a new method of transferring pre-existing ions from solution to the gas phase. At low ion source energies the transfer is very soft with minimal fragmentation. The ES technique has been developed largely by Fenn and co-workers [12–14] and its most impressive successes



Scheme 1. Structures of cationic MeHg(II) complexes of 9-methylguanine (9-MeGua), $[MeHg(9\text{-}MeGua)]NO_3$, and creatinine ($C_4H_7N_3O$, Hcreat), $[PhHg(Hcreat)]NO_3$ and $[(PhHg)_2(creat)]NO_3$.

---

*Author to whom correspondence should be addressed.

SSDI 0020-1693(93)03697-9

have been concerned with large bio-molecules such as proteins [15–17]. These are multiply protonated by the mobile phase (H_2O/MeOH/HOAc) to give species with accessible *m/z* values.

We have been investigating the application of ESMS to inorganic and organometallic systems, many of which are already ionic and for which observation of their ES mass spectra does not depend upon protonation by the mobile phase [18–26]. A feature of these studies is that the intact ion is almost always observed, often as the only significant peak in the mass spectrum.

In this paper we report an ESMS study of the methylmercury(II) complexes described above. A characteristic feature of the chemistry of methylmercury(II) is the ready exchange of ligands coordinated to it [27], so some exchange reactions of methylmercury(II) in solution have also been investigated by ESMS.

Experimental

The $[MeHgL]NO_3$ compounds were prepared as described previously [4–10] as were $PPh_3(E)$, $Ph_2P(E)CH_2PPh_2$ and $Ph_2P(E)CH_2P(E)Ph_2$ (E = S or Se) [28]. Solutions for ES mass spectrometry were prepared by dissolving about 1 mg of material in methanol (1 ml) and diluting this solution 1:10 with methanol. The diluted solution was injected directly into the spectrometer as described below. For exchange reactions, a few drops of a solution containing the new ligand were added before the dilution step. The nitrogen base ligands were generally soluble in methanol but the phosphine based ligands were usually added in dichloromethane solution.

Electrospray mass spectra were obtained with a VG Bio-Q triple quadrupole mass spectrometer (VG BioTech, Altrincham, Ches., UK) using a water/methanol/acetic acid (50:50:1%) mobile phase. The diluted solutions of the compounds, prepared as described above, were injected directly into the spectrometer via a Rheodyne injector equipped with a 10 μl loop. A Phoenix 20 micro LC syringe pump delivered the solution to the vaporisation nozzle of the electrospray ion source at a flow rate of 3 $\mu l\ min^{-1}$. Nitrogen was used as both a drying gas and for nebulisation with flow rates of approximately 3 $l\ min^{-1}$ and 100 $ml\ min^{-1}$, respectively. Pressure in the mass analyser region was usually about 3×10^{-5} torr. The compounds in this study give strong signals in their ES mass spectra and typically 8–10 signal averaged spectra were required to give a good signal to noise ratio. Increasing the voltage on the first skimmer electrode (B1) may induce fragmentation caused by collisions with solvent molecules in the region of the nozzle where the pressure is close to atmospheric. Measurements were made at a B1 voltage of 40 V unless stated otherwise.

Results and discussion

Peaks in the ES mass spectra are identified by the most intense *m/z* value in the isotopic mass distribution In all cases the experimental and calculated isotopic mass distributions were in excellent agreement ESMS data for all the methylmercury(II) complexes are summarised in Table 1.

ES mass spectra of nitrogen base complexes of $[MeHg]^+$

Figure 1(a) shows the ES mass spectrum at low ion source energy (B1 = 40 V) for a solution of $[MeHg(Me_3pz)]NO_3$ (Me_3pz = 3,5-dimethyl-*N*-methylpyrazole, $C_6H_{10}N_2$) and the peak at *m/z* 327 corresponds to the intact ion $[MeHg(Me_3pz)]^+$ Figure 1(b) shows the ES mass spectrum at a higher ion source energy (B1 = 70 V) and the daughter ion $[MeHg]^+$ (*m/z* 217) is formed by collisions with solvent molecules in the ion source, where the pressure is close to atmospheric A peak at *m/z* 111 is due to the protonated ligand formed in the ion source by reaction with the mobile phase after dissociation of the ligand. Figure 1(c) shows the comparison between the experimental and calculated isotopic mass distribution of the intact ion. The ES mass spectrum of $[MeHg(Me_3pz)]NO_3$ is remarkably simple, but this is typical for most of the compounds discussed in this paper, as demonstrated by the ES mass spectra for the other complexes containing monodentate nitrogen bases (Table 1). However, [MeHg(9-MeGua)]NO_3 did not give any $[MeHg]^+$ at higher ion source energies.

A correspondingly simple behaviour is observed for $[MeHg(py_2CH_2)]NO_3$ which contains a bidentate nitrogen base, but both $[MeHg(Et_2bipy)]NO_3$ and $[MeHg(mim_2CHOH)]NO_3$ failed to form $[MeHg]^+$ at higher ion source energies. In the case of $[MeHg(Et_2bipy)]NO_3$ a peak is observed at *m/z* 227 only at high ion source energies (B1 = 80 V) and this corresponds to the ion $[MeEt_2bipy]^+$ (corresponding to $[HEt_2bipy)]^+$. $[MeHg(mim_2CHOH)]NO_3$ gives a peak at *m/z* 299 (containing mercury) which is weak at low ion source energies, but becomes stronger relative to the peak due to the intact ion at higher ion source energies. It corresponds to loss of one ring and the CHOH group, followed by protonation, to give the ion $[MeHg(Hmim)]^+$. At low ion source energies there is a strong peak due to the protonated ligand $[Hmim_2CHOH]^+$ (*m/z* 193), but at higher ion source energies a stronger peak appears at *m/z* 175, presumably formed by loss of H_2O.

TABLE 1 ES mass spectrometric data for methylmercury(II) compounds

Compound	Ions at low ion source energy (B1 = 40 V) (*m/z*)	Ions at higher ion source energies (B1 > 40 V) (*m/z*)
$[MeHg(Me_3pz)]NO_3$	$[MeHg(Me_3pz)]^+$ (327)	$[MeHg]^+$ (217)
$[MeHg(Mepz)]NO_3$	$[MeHg(Mepz)]^+$ (299)	$[MeHg]^+$ (217); $[HMepz]^+$ (83)
$[MeHg(3,5\text{-}Me_2py)]NO_3$	$[MeHg(3,5\text{-}Me_2py)]^+$ (324)	$[MeHg]^+$ (217)
$[MeHg(9\text{-}MeGua)]NO_3$	$[MeHg(9\text{-}MeGua)]^+$ (382), $[H9\text{-}MeGua]^+$ (166)	
$[MeHg(py_2CH_2)]NO_3$	$[MeHg(py_2CH_2)]^+$ (387), $[Hpy_2CH_2]^+$ (171)	$[MeHg]^+$ (217)
$[MeHg(Et_2bipy)]NO_3$	$[MeHg(Et_2bipy)]^+$ (429); $(HEt_2bipy]^+$ (213)	$[MeEt_2bipy]^+$ (227)
$[MeHg(mim_2CHOH)]NO_3$	$[MeHg(mim_2CHOH)]^+$ (409), $[MeHg(Hmim)]^+$ (299), $[Hmim_2CHOH]^+$ (193)	$[Hmim_2C]^+$ (175)
$[MeHg(Me_3terpy)]NO_3$	$[MeHg(Me_3terpy)]^+$ (492), $[MeHg(Me_2bipy)]^+$(401), $[Hg(Me_3terpy)_2]^{2+}$ (376)	(307); $[HMe_3terpy]^+$ (276)
$[MeHg(py_3COH)]NO_3$	$[MeHg(py_3COH)]^+$ (480); $[Hpy_3COH]^+$ (264)	$[MeHg(py)]^+$ (296); $[py_3C]^+$ (246)
$[PhHg(Hcreat)]NO_3$	$[PhHg(Hcreat)]^+$ (392)	$[PhHg]^+$ (279)
$[(PhHg)_2(creat)]NO_3$	$[(PhHg)_2(creat)]^+$ (668); $[PhHg(Hcreat)]^+$ (392)	

The ES mass spectrum of $[MeHg(Me_3terpy)]NO_3$ is more complicated. At low ion source energy (B1 = 40 V) the base peak is at *m/z* 492 which is the intact ion $[MeHg(Me_3terpy)]^+$ but there are peaks of significant intensity at *m/z* 401 and 376 assigned to $[MeHg(Me_2bipy)]^+$ and $[Hg(Me_3terpy)_2]^{2+}$. The isotope pattern of the peak at *m/z* 376 confirms that it is due to a doubly charged cation. At higher ion source energies the intensity of the peak at *m/z* 401 increases relative to that of the intact ion, suggesting that $[MeHg(Me_2bipy)]^+$ is formed within the ion source by collisional activation. Under these conditions there is a peak at *m/z* 276 which is due to the protonated ligand but the base peak at *m/z* 307 (not containing mercury) is unidentified.

At low ion source energies the compound $[MeHg(py_3COH)]NO_3$ gives an ES mass spectrum in which the base peak is at *m/z* 264 which is due to $[Hpy_3COH)]^+$ together with a strong peak at *m/z* 480 due to the intact ion $[MeHg(py_3COH)]^+$. With B1 = 70 V the dominant peaks are at *m/z* 246 due to $[py_3C]^+$, which has been observed previously in other systems [25, 26], and *m/z* 296 which is assigned to $[MeHg(py)]^+$.

The compound $[PhHg(Hcreat)]NO_3$ gave the intact ion $[PhHg(Hcreat)]^+$ (*m/z* 392) as the only peak containing mercury at low ion source energies, but at higher energies $[PhHg]^+$ (*m/z* 279) was observed. The closely related complex $[(PhHg)_2(creat)]NO_3$ gave only a weak peak for the intact ion $[(PhHg)_2(creat)]^+$ (*m/z* 668), but a strong peak was observed for $[PhHg(Hcreat)]^+$ (*m/z* 392) formed by loss of one PhHg group followed by protonation within the ion source.

Exchange reactions

Most of the exchange reactions were carried out using an approximately 2 mM methanol solution of $[MeHg(Mepz)]NO_3$ (Mepz = *N*-methylpyrazole, $C_4H_6N_2$) and adding an excess of the extra ligand in methanol (nitrogen bases) or dichloromethane (phosphine based ligands). This mixed solution was then diluted 1:10 with methanol and injected into the spectrometer.

With the nitrogen bases *p*-toluidine, 3,5-Me_2pz and ethylenediamine the only mercury containing species observed in the ES mass spectrum in each case was $[MeHgL]^+$ (Table 2) with no remaining $[MeHg(Mepz)]^+$. These results confirm the known [27] lability of ligands bound to the $[MeHg]^+$ moiety.

The ES mass spectrum of an approximately equimolar mixture of $[MeHg(Mepz)]NO_3$ and PPh_3 gave a very strong peak at *m/z* 479 which is due to $[MeHg(PPh_3)]^+$. Addition of more PPh_3 did not result in the observation of a peak which could be assigned to $[MeHg(PPh_3)_2]^+$, which serves to emphasise that $[MeHg]^+$ strongly prefers to coordinate only one additional ligand. Reaction of triphenylphosphine selenide with $[MeHg(Mepz)]NO_3$ gave an ES mass spectrum with only one peak at *m/z* 557 due to $[MeHg(PPh_3Se)]^+$ (Fig. 2(a)).

Reaction between $[MeHg(Mcpz)]NO_3$ and the potentially bidentate ligand dpm (dpm = $Ph_2PCH_2PPh_2$) resulted in a ES mass spectrum containing only one peak at *m/z* 601 due to $[MeHg(dpm)]^+$. Interaction between $[MeHg(Mepz)]NO_3$ and dpmSe also results in only a single peak in the ES mass spectrum at *m/z* 679 due to $[MeHg(dpmSe)]^+$ (Fig. 2(b)) and es-

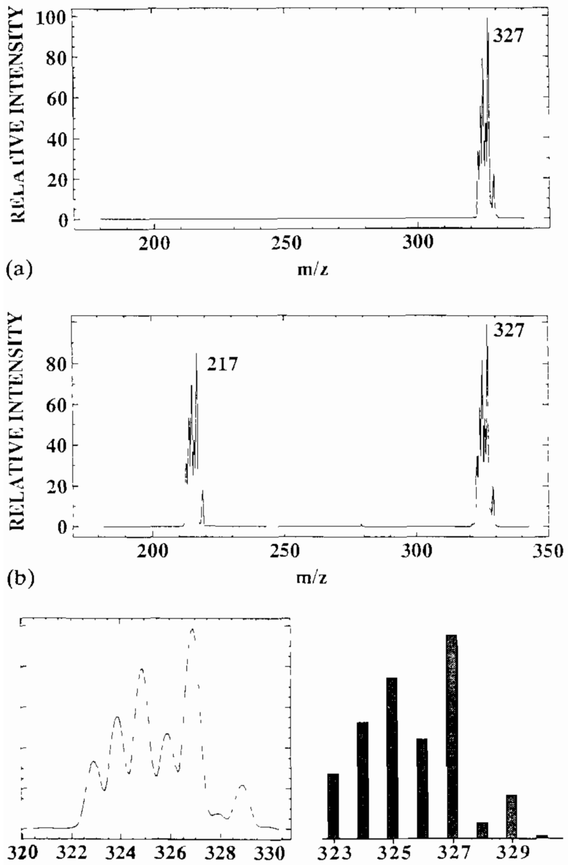


Fig 1. Positive ion ES mass spectrum of [MeHg(Me$_3$pz)]NO$_3$ (a) B1 = 40 V, (b) B1 = 70 V, (c) comparison between experimental and calculated isotopic mass distributions.

sentially similar results are obtained for a mixture of [MeHg(Mepz)]NO$_3$ and dpmSe$_2$ with a strong peak being observed at *m/z* 759 due to [MeHg(dpmSe$_2$)]$^+$, but there is also a weaker peak at *m/z* 643 which is assigned to [Hg(dpmSe$_2$)$_2$]$^+$. This cation has been characterised previously by multi-NMR methods from the reaction of Hg(ClO$_4$)$_2$ with dpmSe$_2$ [29]. When a small amount of PPh$_3$ was added to the mixture of [MeHg(Mepz)]NO$_3$ and dpmSe$_2$, the ES mass spectrum shown in Fig. 2(c) was obtained. It shows strong peaks assigned to [MeHg(dpmSe$_2$)]$^{2+}$ (*m/z* 759), [MeHg(dpmSe)]$^+$ (*m/z* 679) and [MeHg(PPh$_3$Se)]$^+$ (*m/z* 557) and no peak due to [MeHg(PPh$_3$)]$^+$ (*m/z* 479) These observations are entirely in accord with the known chemistry of phosphine selenides [30, 31]. Phosphorus–selenium bonds are labile in solution at room temperature and selenium transfer between phosphines is common. In particular, dpmSe$_2$ in which there is considerable steric crowding, very readily transfers one selenium atom to another phosphine to give dpmSe and the selenide of the second phosphine. Thus, in this case the small amount of added triphenylphosphine is converted to its selenide and methylmercury complexes are formed by PPh$_3$Se, dpmSe and the remaining dpmSe$_2$. All of the added PPh$_3$ was converted to PPh$_3$Se as shown by the absence of a peak due to [MeHg(PPh$_3$)]$^+$.

Phosphorus–sulfur bonds are known to be non-labile [30, 31] at room temperature although sulfur transfer does occur at a reasonable rate at about 120 °C. Therefore, somewhat different observations are expected in attempted sulfur exchange reactions on methylmercury. When PPh$_3$S is added to [MeHg(Mepz)]NO$_3$ the ES mass spectrum shows a single peak at *m/z* 511 due to [MeHg(PPh$_3$S)]$^+$ and corresponding results are obtained when dpmS and dpmS$_2$ are each added to [MeHg(Mepz)]NO$_3$ (Table 2). When PPh$_3$ is added to an approximately equimolar mixture of [MeHg(Mepz)]NO$_3$ and dpmS$_2$, the resulting ES mass spectrum is dominated by the peaks due to [MeHg(PPh$_3$)]$^+$ (*m/z* 479) and [MeHg(dpmS$_2$)]$^+$ (*m/z*

TABLE 2 Ions formed by exchange reactions

Mixture	Ions observed (*m/z*)
[MeHg(Mepz)]NO$_3$ + *p*-toluidine	[MeHg(*p*-tol)]$^+$ (324)
[MeHg(Mepz)]NO$_3$ + 3,5-Me$_2$pz	[MeHg(Me$_2$pz)]$^+$ (313)
[MeHg(Mepz)]NO$_3$ + en	[MeHg(en)]$^+$ (277)
[MeHg(Mepz)]NO$_3$ + PPh$_3$	[MeHg(PPh$_3$)]$^+$ (479)
[MeHg(Mepz)]NO$_3$ + PPh$_3$Se	[MeHg(PPh$_3$Se)]$^+$ (557)
[MeHg(Mepz)]NO$_3$ + dpm	[MeHg(dpm)]$^+$ (601)
[MeHg(Mepz)]NO$_3$ + dpmSe	[MeHg(dpmSe)]$^+$ (679)
[MeHg(Mepz)]NO$_3$ + dpmSe$_2$	[MeHg(dpmSe$_2$)]$^+$ (759); [Hg(dpmSe$_2$)$_2$]$^{2+}$ (643)
[MeHg(Mepz)]NO$_3$ + dpmSe$_2$ + PPh$_3$	[MeHg(dpmSe$_2$)]$^{2+}$ (759), [MeHg(dpmSe)]$^+$ (679), [Hg(dpmSe$_2$)$_2$]$^+$ (643); [MeHg(PPh$_3$Se)]$^+$ (557)
[MeHg(Mepz)]NO$_3$ + PPh$_3$S	[MeHg(PPh$_3$S)]$^+$ (511)
[MeHg(Me$_3$terpy)]NO$_3$ − dpmS	[MeHg(dpmS)]$^+$ (633); [Hg(dpmS)$_2$]$^{2+}$ (517)
[MeHg(Mepz)]NO$_3$ + dpmS$_2$	[MeHg(dpmS$_2$)]$^+$ (665)
[MeHg(Mepz)]NO$_3$ + dpmS$_2$ + PPh$_3$	[MeHg(dpmS$_2$)]$^+$ (665); [MeHg(PPh$_3$)]$^+$ (479)
[MeHg(Mepz)]NO$_3$ + dam	[MeHg(dam)]$^+$ (689)

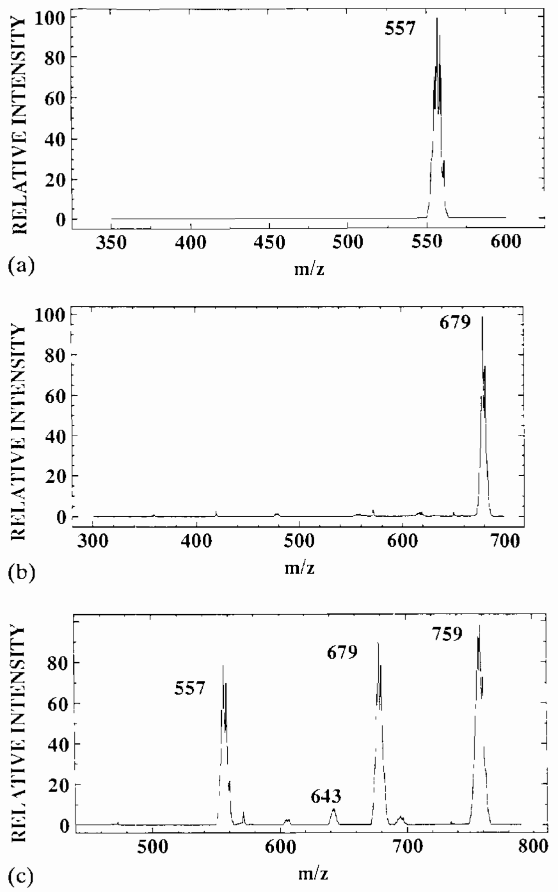


Fig 2. Positive ion ES mass spectra (B1 = 40 V) of (a) [MeHg(Me$_3$pz)]NO$_3$ + PPh$_3$Se, (b) [MeHg(Me$_3$pz)]NO$_3$ + dpmSe, (c) [MeHg(Me$_3$pz)]NO$_3$ + dpmSe$_2$ + small amount of PPh$_3$.

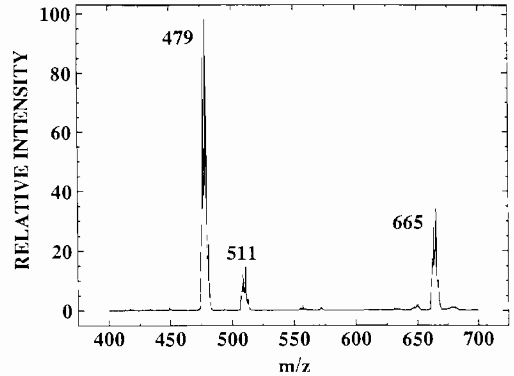


Fig 3. Positive ion ES mass spectrum of [MeHg(Me$_3$pz)]NO$_3$ + dpmS$_2$ + small amount of PPh$_3$

665) as shown in Fig. 3. There is a small peak at *m/z* 511 due to [MeHg(PPh$_3$S)]$^+$, but there is no corresponding peak due to [MeHg(dpmS)]$^+$ (*m/z* 633). Therefore, the PPh$_3$S cannot have been formed by sulfur transfer from dpmS$_2$, and its formation is best attributed to reaction within the ion source of PPh$_3$ with a small amount of elemental sulfur impurity in the dpmS$_2$, remaining from its preparation by reaction of dpm with sulfur. However, even with this impurity present it is obvious that most of the triphenylphosphine remains and sulfur transfer does not occur between dpmS$_2$ and PPh$_3$, in contrast to the selenides.

The potentially bidentate arsenic ligand dam (dam = Ph$_2$AsCH$_2$AsPh$_2$) displaces the nitrogen base from [MeHg(Mepz)]NO$_3$ to give [MeHg(dam)]$^+$ (*m/z* 689), but triphenylstibine failed to react with [MeHg(Mepz)]NO$_3$ and was the only ligand investigated which did not displace the pyrazole ligand.

Conclusions

In this paper we have demonstrated that ESMS is a powerful new technique with which to investigate the labile methylmercury complexes with nitrogen bases. The ES mass spectra of the complexes themselves serve to confirm that ESMS does indeed give a good indication of the ionic species present in solution, since in these cases the identities of the species were known from previous NMR studies. ESMS readily confirms that exchange between different nitrogen bases is facile. The exchange reactions between [MeHg(Mepz)]NO$_3$ and the phosphine based ligands reveal some new, but not unexpected, chemistry. It is important to note that ESMS does not give any information on the coordination number of mercury, nor on the mode of coordination of the ligand in an ion such as [MeHg(dpmSe)]$^+$ in solution, so detailed multi-NMR studies would be needed if this information was sought.

Acknowledgement

We thank La Trobe University for a SCAEF major equipment grant which assisted in the purchase of the ES mass spectrometer.

References

1 J.M Wood, F S. Kennedy and C G Rosen, *Nature (London), 220* (1968) 173
2 S Jensen, *Nature (London), 223* (1969) 753
3 R. DeSimone, M. Penley, L. Charbonneau, S Smith, J Wood, H. Hill, J Pratt, S. Ridsdale and R J P Williams, *Biochim Biophys Acta, 304* (1973) 851.
4 A J. Canty and C V Lee, *Organometallics, 1* (1982) 1063.
5 A.J Canty, P Barron and P C Healy, *Organomet Chem, 179* (1979) 447.
6 A.J. Canty, N Chaichit, B M Gatehouse, E E. George and G Hayhurst, *Inorg Chem, 20* (1981) 2414.
7 A J. Canty, N Chaichit, B M Gatehouse and E.E George, *Inorg Chem, 20* (1981) 4293
8 A J Canty, J M Patrick and A H White, *J Chem Soc, Dalton Trans*, (1983) 1873

9 A.J. Canty, R S Tobias, N Chaichit and B.M. Gatehouse, *J Chem Soc , Dalton Trans ,* (1980) 1693.

10 A.J. Canty, M. Fyfe and B.M Gatehouse, *Inorg Chem , 17* (1978) 1467

11 A J Canty, N. Chaichit and B.M. Gatehouse, *Acta Crystallogr , Sect B, 35* (1979) 592.

12 M. Yamashita and J B Fenn, *J Phys. Chem , 88* (1984) 4451.

13 M Yamashita and J B Fenn, *J Phys Chem., 88* (1984) 4671

14 C.M Whitehouse, R N. Dreyer, M Yamashita and J B Fenn, *Anal Chem , 57* (1985) 675

15 J B Fenn, M Mann, C K Meng, S F Wong and C.M. Whitehouse, *Science, 246* (1989) 64

16 R D. Smith, J.A. Loo, C G Edmonds, C.J Barinaga and H.R. Udseth, *Anal Chem , 62* (1990) 882.

17 R D. Smith, J.A Loo, R.R Ogorzalek Loo, M. Busman and H.R. Udseth, *Mass Spectrom Rev , 10* (1991) 359.

18 R. Colton and J.C. Traeger, *Inorg Chim Acta, 201* (1992) 153

19 R. Colton, J C Traeger and J Harvey, *Org Mass Spectrom , 27* (1992) 1030.

20 R Colton, J C Traeger and V Tedesco, *Inorg Chem , 31* (1992) 3865

21 R. Colton, B.D James, I.D Potter and J C Traeger, *Inorg Chem , 32* (1993) 2626.

22 R. Colton and D. Dakternieks, *Inorg Chim Acta, 208* (1993) 173.

23 T J Cardwell, R Colton, N Lambropoulos, J C Traeger and P J Marriott, *Anal Chim Acta, 280* (1993) 239

24 I Ahmed, A.M. Bond, R Colton, M Jurcevic, J.C Traeger and J N Walter, *J Organomet Chem , 447* (1993) 59

25 A.J Canty, R. Colton and I M Thomas, *J Organomet Chem , 455* (1993) 283.

26 A J Canty, P R Traill, R Colton and I.M Thomas, *Inorg Chim Acta, 210* (1993) 91

27 D L. Rabenstein, *Acc Chem Res , 11* (1978) 100.

28 A M. Bond, R Colton and P Panagiotidou, *Organometallics, 7* (1988) 1767

29 A M. Bond, R Colton and J Ebner, *Inorg Chem., 27* (1988) 1697

30 S O Grim and E D. Walton, *Inorg Chem , 19* (1980) 1982

31 S W Carr and R Colton, *Aust J Chem , 34* (1981) 35