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*International Journal of Mass Spectrometry and Ion Processes*, 112 (1992) 79–91
Elsevier Science Publishers B.V., Amsterdam

# Cationization of organometallo carbonyl compounds by fast ion bombardment

Gary Siuzdak[1], Sebastian V. Wendeborn[2] and K.C. Nicolaou[2]
*The Scripps Research Institute, Department of Chemistry, La Jolla, CA 92037 (USA)*

(Received 24 June 1991)

## ABSTRACT

Organodicobalt, organochromium, and organomolybdenum carbonyl complexes have been studied using fast ion bombardment mass spectrometry. It has been found that the addition of cesium iodide to the liquid matrix, *m*-NBA, can significantly enhance the ability to observe the precursor ions of these organometallics through charge localization. In most cases the $[M + Cs]^+$ ions were more abundant than the radical cations $M^{\cdot +}$, the protonated molecules $[M + H]^+$, or the sodium cationized molecules $[M + Na]^+$ which were either unobservable or less intense than those treated with the cesium iodide salt solution. The decomposition of the compounds took place primarily through the successive loss of carbonyls from the radical cation with some carbonyl loss observed through the protonated and cationized species. The FAB matrix ions produced when cesium iodide was added to *m*-NBA also allowed for internal calibration.

*Keywords*: cationization; organometallo carbonyl compounds; fast ion bombardment.

## INTRODUCTION

The mass analysis of organometallic compounds has benefited from the use of particle beam desorption techniques; specifically, liquid matrix fast atom bombardment (FAB) has proven to be a valuable tool in the examination of organometallics [1] through its capacity to induce molecular ionization as well as fragmentation. For organometallics, ionization induced by FAB desorption can result in cation addition or in the formation of the radical cation. Similar to radical cation formation for organic compounds [2], organometallics often exhibit only nominal formation of the precursor ion. While protonation can produce significantly more stable ions, organometallics often do not exhibit significant yields of the protonated molecule in FAB ionization. This behavior is especially apparent for organometallo carbonyls.

The desorption ionization of organometallo carbonyl compounds commonly

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[1] Author to whom correspondence should be addressed.

[2] Also at: Department of Chemistry, University of California at San Diego, La Jolla, CA 92093, USA.

results in the formation of the radical cation with fragmentation through the successive loss of carbonyls, a consequence of the relatively weak metal–carbonyl bond. For example, Group VI organometallo carbonyls including chromium, molybdenum, and tungsten pentacarbonyls [3] and manganese, iron, and nickel complexes [4] have shown fragmentation patterns corresponding to carbonyl loss. However, in many cases the precursor radical cation can only be weakly observed [5] or not at all [6]—a characteristic also observed in the spectra of cobalt acylium compounds [7]. The same softness that the FAB ionization technique has exhibited with organic compounds has made it useful for organometallic compounds as well [1]; yet in many cases significant precursor ion formation can still not be observed.

The inability to observe significant precursor ion formation was addressed in field ionization and field desorption studies in the mid-1970s by Rollgen and Schulten [2] and by Schulten and Games [8] with a variety of organic compounds. In these studies the addition of the lithium cation proved to stabilize precursor ion formation through charge localization. The increased abundance of the molecular ions was attributed to the localization of the positive charge at the alkali atom and to a high activation energy for a charge shift within the ion [2,9]. This stabilization effect has been well established [10] and applied to molecular ion detection in fission fragment ionization [11], laser desorption [12], chemical ionization [13], electron impact [14], and FAB [15,16], for a variety of organic compounds including alkanes [17], oligosaccharides [18], organosilicates [19], and alcohols [2].

The relative difficulty in observing molecular ion formation for some organometallo compounds has prompted this fast ion bombardment study. Eight organodicobalt complexes (recently synthesized derivatives [20] related to dynemicin A) and an analogous organic compound not containing the dicobalt carbonyl complexation were examined. In addition, the ionization behavior of an organochromium carbonyl compound and an organomolybdenum carbonyl compound were compared with the organodicobalt carbonyl compounds. Cesium iodide and sodium iodide salts were added to the matrix to determine their effect on ion formation through cationization and also to examine the ability of *m*-NBA + cesium iodide and sodium iodide in the production of reference ions for accurate mass measurement. The mass spectra were also examined for evidence of carbonyl site-specific cationization in order to determine if attachment of the cation was occurring at the metallo carbonyl as previously suggested [3] for the Group VI metal pentacarbonyls.

## EXPERIMENTAL

The fast ion bombardment positive ion mass spectra were obtained on a VG ZAB-VSE double focusing mass spectrometer equipped with a cesium ion gun. The mass spectrometer was manually tuned to a resolution of 2000 (10% valley definition) with amplifier and multiplier gains of $10^6$ (at 300 V). A 35 keV cesium ion source was used to produce the fast ion beam and the accelerating voltage for the desorbed ions was 8 kV. The mass spectra were acquired by scanning the mass range from 100 to 1250 Da using cesium iodide for initial calibration and an *m*-NBA/cesium iodide mixture as an internal standard; typically 10 spectra were accumulated and averaged. Spectra were recorded with a Digital VAXstation 3100, and the peaks were automatically centroided. The data were later transferred to a Macintosh II cx microcomputer to undergo further processing and analysis. The *m*-NBA matrix compound as well as glyerol, cesium iodide, and sodium iodide were used as obtained from Aldrich at 98%, 99.5+%, 99.9%, and 99+% purities respectively. The 2-nitrophenyl octyl ether (NPOE) matrix was also used as obtained from Johnson Matthey Electronics at 98% purity. In a representative experiment 10.0 $\mu$g of the sample in chloroform was applied to 2.0 $\mu$l of the matrix and the solvent was allowed to evaporate. The probe was inserted and the spectra were accumulated and averaged. To introduce the salts 0.5 $\mu$l of a saturated aqueous solution of either cesium iodide or sodium iodide was added to the matrix solution. The sample was then reintroduced into the ionization chamber for analysis and the results from the matrix not containing the salt were compared with the salt-prepared matrix. In addition to these mass spectrometric studies, detailed information on the synthesis of these compounds has been reported elsewhere [20,21], as well as the structural confirmation deduced through spectroscopic methods and X-ray crystallographic studies.

## RESULTS AND DISCUSSION

### *Matrix*

*m*-NBA was used as the matrix in these experiments since it proved to be the most effective medium for solvating these samples and introducing the salt solutions. The addition of cesium iodide to *m*-NBA gave a characteristic mass spectrum which was used for internal calibration and could be characterized by the ions $[m\text{-NBA}_x + \text{Cs}_y\text{I}_z]^+$ to form ions up to $m/z = 2211$ or $[\text{Cs}_9\text{I}_8]^+$ in the presence of the sample (Table 1); yet, more typical was the observation of reference ions up to $[\text{Cs}_5\text{I}_4]^+$ or $m/z = 1172$. The sodium iodide + *m*-NBA mixture formed ions analogous to cesium iodide + *m*-NBA ions and gave significant formation up to $m/z$ 800. The durations of the matrix ions were typically 2–5 min long and were used in conjunction with the cationization of the cobalt carbonyl complexes for accurate mass measurement. Cesium iodide has previously been added to glycerol matrices [5] as well as potassium

TABLE 1

Ions resulting from the fast ion bombardment of the *m*-NBA/CsI matrix

| Ion | Exact mass (calculated) | Exact mass (experimental) | Relative intensity (%) |
|---|---|---|---|
| $[Cs]^+$ | 132.90543 | 132.90441 | 100 |
| $[m\text{-NBA} + H]^+$ | 154.05042 | 154.05108 | 20 |
| $[Cs_2 + H_2O - H]^+$ | 282.81361 | 282.81369 | 0.1 |
| $[m\text{-NBA} + Cs]^+$ | 285.94803 | 285.94890 | 51 |
| $[m\text{-NBA}_2 + H - H_2O]^+$ | 289.08245 | 289.08318 | 5 |
| $[m\text{-NBA}_2 + H]^+$ | 307.09301 | 307.09563 | 7 |
| $[NO_2 + Cs_2]^+$ | 311.80377 | 311.80411 | 35 |
| $[Cs_2I]^+$ | 392.71534 | 392.71533 | 11 |
| $[m\text{-NBA} + Cs_2]^+$ | 418.85346 | 418.85346 | 6 |
| $[m\text{-NBA}_2 + Cs]^+$ | 438.99062 | 438.99400 | 6 |
| $[m\text{-NBA}_3 + H]^+$ | 460.13561 | 460.13620 | 0.1 |
| $[m\text{-NBA} + Cs_2I]^+$ | 545.75794 | 545.75811 | 26 |
| $[m\text{-NBA}_2 + Cs_2]^+$ | 571.89605 | 571.89988 | 1 |
| $[m\text{-NBA}_3 + Cs]^+$ | 592.03321 | 592.03354 | 4 |
| $[Cs_3I_2]^+$ | 652.52525 | 652.52589 | 8 |
| $[m\text{-NBA} + Cs_3I]^+$ | 678.66337 | 678.66619 | 6 |
| $[m\text{-NBA}_2 + Cs_2I]^+$ | 698.80053 | 698.80090 | 6 |
| $[m\text{-NBA}_3 + Cs_2]^+$ | 724.93865 | 724.93580 | 0.5 |
| $[m\text{-NBA} + Cs_3I_2]^+$ | 805.56785 | 805.56189 | 3 |
| $[m\text{-NBA}_2 + Cs_3I]^+$ | 831.70596 | 831.70555 | 1 |
| $[Cs_4I_3]^+$ | 912.33516 | 912.33856 | 2 |
| $[m\text{-NBA} + Cs_4I_2]^+$ | 938.47328 | 938.47675 | 2 |
| $[m\text{-NBA}_2 + Cs_3I_2]^+$ | 958.61044 | 958.61116 | 1 |
| $[m\text{-NBA} + Cs_4I_3]^+$ | 1065.37776 | 1065.37432 | 0.2 |
| $[m\text{-NBA}_2 + Cs_4I_2]^+$ | 1091.51587 | 1091.51146 | 0.2 |
| $[Cs_5I_4]^+$ | 1172.14507 | 1172.14677 | 1 |
| $[m\text{-NBA} + Cs_5I_3]^+$ | 1198.28319 | 1198.28900 | 0.5 |
| $[Cs_6I_5]^+$ | 1431.95498 | 1431.95555 | 0.5 |
| $[Cs_7I_6]^+$ | 1691.76489 | 1691.77002 | 0.4 |
| $[Cs_8I_7]^+$ | 1951.57480 | 1951.57234 | 0.3 |
| $[Cs_9I_8]^+$ | 2211.38471 | 2211.38989 | 0.3 |

chloride to *m*-NBA [22] to form reference ions, but to the authors' knowledge this is the first example of cesium iodide in *m*-NBA to form reference ions and enhance molecular ionization of organometallics. The addition of the salts through an aqueous salt solution also proved to be an effective means of introduction rather than adding the salt directly to the matrix. Mass spectra were recorded in the *m*-NBA matrix with and without water as a medium for the salt; no significant variation was observed except an increase in the ion abundance when the salt was introduced in the aqueous form. Glycerol and NPOE were also used as matrices for these compounds; however, glycerol was

TABLE 2

Abundances of the precursor ions relative to the cesium-cationized molecule

| Compound | Matrix | | | |
|---|---|---|---|---|
| | NBA/CsI | NBA/NaI | NBA | |
| | Precursor ion | | | |
| | $[M + Cs]^+$ | $[M + Na]^+$ | $[M + H]^+$ | $M^{\cdot +}$ |
| **1** | 1.00 | 0.94 | 0.66 | – |
| **2** | 1.00 | 0.56 | – | – |
| **3** | 1.00 | 0.14 | – | 1.00 |
| **4** | – | – | – | – |
| **5** | – | – | – | – |
| **6** | 1.00 | < .01 | 0.05 | – |
| **7** | 1.00 | – | – | < .01 |
| **8** | 1.00 | < .01 | 0.58 | – |
| **9** | 1.00 | 0.38 | 3.31 | – |
| **10** | 1.00 | 0.20 | – | 0.20 |
| **11** | 1.00 | 0.10 | – | 0.18 |

ineffective at solvating the samples. NPOE effectively solvated the samples and gave FAB mass spectra equivalent to those obtained with the neat *m*-NBA matrix. However, NPOE proved ineffective as a medium for cationization with the alkali salts; no cationization could be observed in this matrix independent of the amount of salt introduced to the NPOE/sample solution.

### *Ionization of organodicobalt carbonyl compounds*

A principal aim of this study was to determine whether cationization would facilitate molecular ion formation for these organometallo carbonyl compounds. Initially, to ensure that proton and cation attachment in the FAB experiments would be observed for the organic analog of the organodicobalt carbonyl compounds, FAB analyses were performed on compound **1**. It was found that cesium and sodium cation addition produced higher abundances of precursor ions for compound **1** than protonation (Table 2); the cesium cation produced the largest formation of precursor ions. Compounds **2** and **3**, the acetylene hexacarbonyl dicobalt complexes of **1**, displayed markedly different ionization behavior upon FAB analysis. It was found that compound **3**, when analyzed in neat *m*-NBA, generated significant ion yield of the precursor radical cation; however, the protonated precursor ions of both **2** and **3** were no longer detectable. This was in sharp contrast to the mass spectra of the samples analyzed in the salinized matrix where the addition of cesium or sodium iodide resulted in the clear observation of molecular cationization.

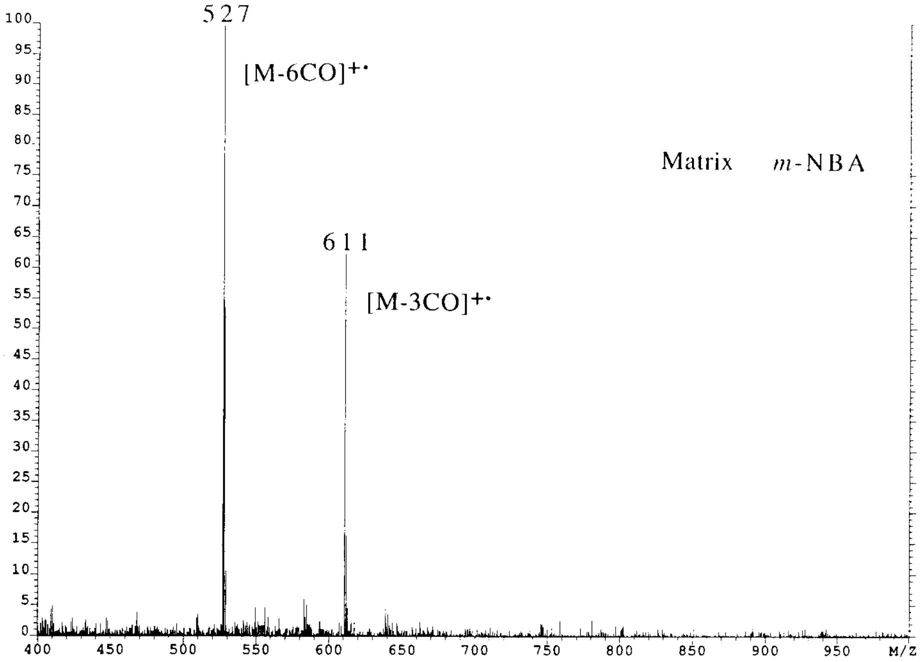


Fig. 1. Mass spectrum of **2** in *m*-NBA matrix.

The mass spectrum of **2** in neat *m*-NBA and the background corrected mass spectrum of **2** in *m*-NBA + cesium iodide are shown in Figs. 1 and 2 respectively, the relative intensities of the precursor ions for each matrix are also reproduced in Table 2.

The analysis of compounds **4**–**9** resulted in molecular ion yields that might be anticipated [2,10,23,24] by the relative polarities of these molecules and the functionalities available for cationization. The comparatively non-polar compounds, **4** and **5**, produced no precursor ions under any of the matrix conditions described. Compounds **6** and **7**, having more polar functionalities available for cationization, underwent cationization with precursor ion abundances greater for compound **6** than for compound **7**. This polarity dependence was especially apparent for compounds **8** and **9** where the removal of the carbamate increased the basicity of each of the molecules by exposing the free nitrogen. Overall the greater abundance of precursor ions was observed when the hydroxyl (**2**, **3** and **6**) or free nitrogen functionality (**8** and **9**) were available, which suggested that these functionalitites were the primary sources of cation addition.

The inability to observe precursor ions for compounds **4** and **5** indicated that appreciable cationization was not taking place at the cobalt carbonyl complex or carbamate functionality. Specifically, with the results obtained on

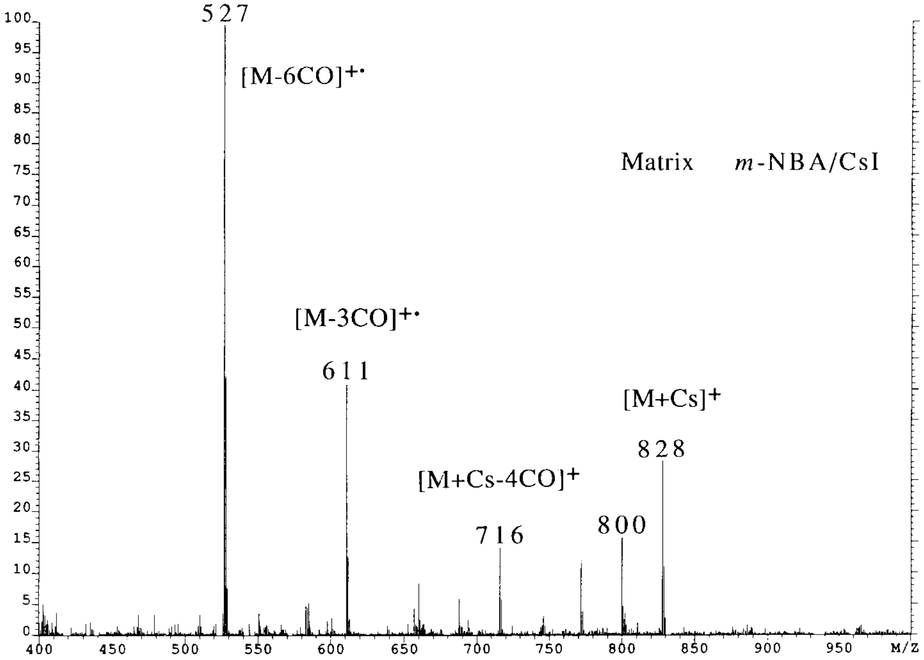
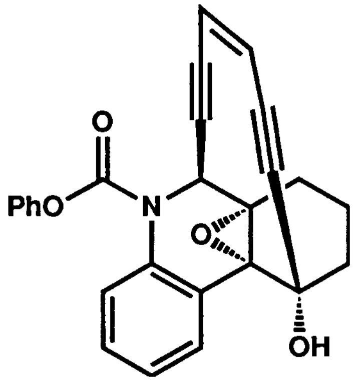


Fig. 2. Background-subtracted mass spectrum of **2** in *m*-NBA/CsI matrix.

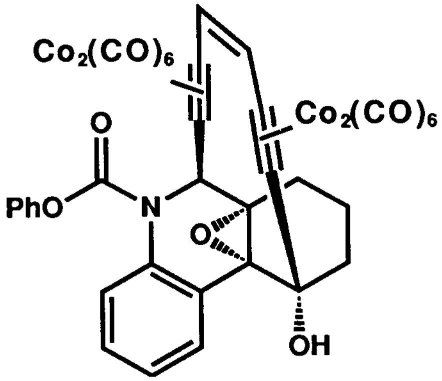
compounds **1** and **5**, we were also able to further address the question of whether significant cation attachment was occurring at the cobalt carbonyl functionality [3]. The mass spectra that resulted from the fast ion bombardment of **1** in *m*-NBA and cesium or sodium iodide displayed abundant ion formation corresponding to the cationized molecule; however, when cesium or sodium iodide was added to **5** in *m*-NBA no precursor ion formation corresponding to $[M + Cs]^+$ or $[M + Na]^+$ was observed. This evidence further suggests that cation attachment was not occurring at the metallo carbonyl site.

### *Precursor ion abundances of CsI and NaI*

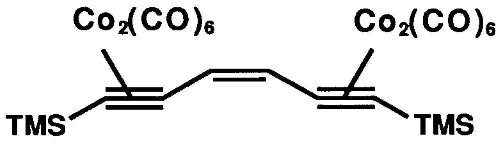
The different precursor ion abundances observed for the cesium iodide and sodium iodide doped matrices (noted in Table 2), indicate that the cesium cation is a more efficient source of cationization. This result is initially counterintuitive considering the relative binding energies of the alkali cations [23]; however, the relative size of the two cations may be consequential to their cationization efficiency. For example, multidentate bonding has recently been suggested for peptides and alkaline earth and alkali metal ions [25]. It is possible that the larger cesium cation can chelate these compounds more


PhO
O
N
O
OH

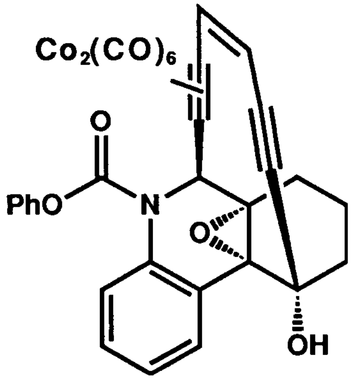
1


Co$_2$(CO)$_6$
Co$_2$(CO)$_6$
PhO
O
N
O
OH

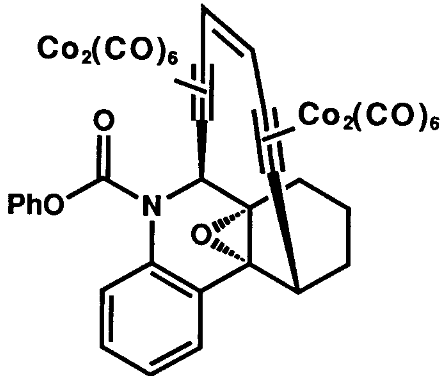
3


Co$_2$(CO)$_6$
Co$_2$(CO)$_6$
TMS
TMS

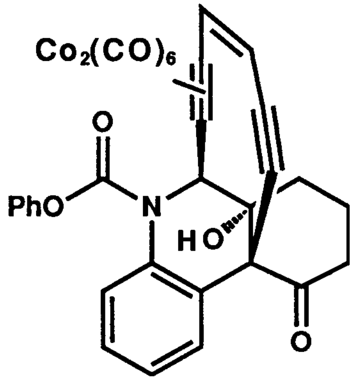
5


Co$_2$(CO)$_6$
PhO
O
N
O
OH

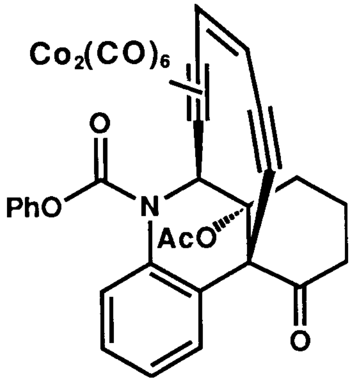
2


Co$_2$(CO)$_6$
Co$_2$(CO)$_6$
PhO
O
N
O

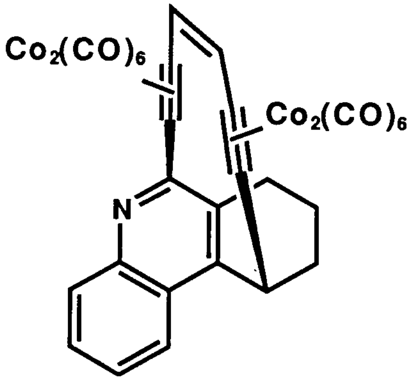
4


Co$_2$(CO)$_6$
PhO
O
N
HO
O

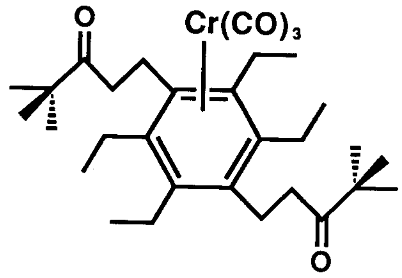
6


Co₂(CO)₆
PhO
O
N
AcO
O

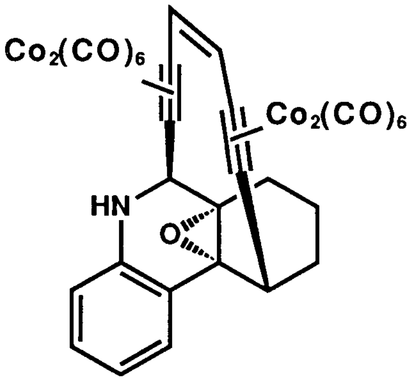
**7**


Co₂(CO)₆
Co₂(CO)₆
N

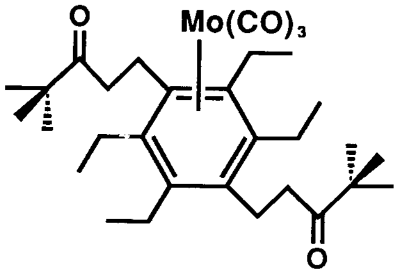
**9**


O
Cr(CO)₃
O

**10**


Co₂(CO)₆
Co₂(CO)₆
HN
O

**8**


O
Mo(CO)₃
O

**11**

effectively than the smaller sodium cation owing to the spatial organization of the polar functional groups in these molecules.

### *Decomposition*

The alkali ion affinities for organic molecules are often smaller ($< 2$eV) [26] than the bond energies in neutral molecules. This fact suggests that the most probable decomposition reaction of a cationized molecule would initially occur through the elimination of the alkali ion. However, the collisional activation analysis of cationized peptides has shown that in some cases molecular decomposition is more energetically favorable than loss of the alkali cation [23,27]. This is evidence that the alkali ion affinity may be similar to the bonding energy within these peptides.

One of the distinguishing features of these data was that decomposition resulted predominantly in carbonyl loss from the radical cation even in the presence of the alkali salt. Possibly the radical cation was formed in addition to the cationized molecule and thus the spectra exhibited carbonyl losses from both the radical cation and the cationized molecule. In these results (Table 3 and Figs. 1 and 2) fragmentation from the cationized molecule was also observed; however, in most cases carbonyl losses from the radical cation dominated the mass spectra. The loss of carbonyls from the cesium-cationized molecules (Table 3) does suggest that the cesium cation affinity for some of these molecules (compounds **2–4**, **6** and **7**) was at least on the order of 1.0–1.5 eV, the cobalt carbonyl bond strength [28,29].

### *Comparison to chromium and molydbenum carbonyl compounds*

The FAB analysis of an organochromium carbonyl compound, **10**, and an organomolybdenum compound, **11**, has yielded results consistent with those obtained with cobalt (Table 2). In the first case, **10**, a significant increase in the precursor ion abundance was noted upon the addition of cesium iodide; this increase was associated with cation attachment to the moderately polar ketone. This ionization behavior was also observed with the organomolybdenum carbonyl compound, **11**, which exhibited an increased precursor ion abundance upon addition of cesium iodide (Table 2).

## CONCLUSION

Cationization has proven to be a useful means of observing molecular ions for a variety of compounds; in this study its utility has been found to be applicable toward the analysis of transition metal organometallo carbonyl complexes. The use of cesium iodide in the *m*-NBA matrix to initiate cation-

TABLE 3

Carbonyl loss [a] observed in the *m*-NBA and *m*-NBA/CsI matrices

| Compound | Matrix | Carbonyl losses observed from these precursor ions | | |
|---|---|---|---|---|
| | | $[M + Cs]^+$ | $[M + H]^+$ | $M^{\cdot +}$ |
| **2** | *m*-NBA | – | – | (3&6) |
| | *m*-NBA/CsI | (1–6) | – | (3&6) |
| **3** | *m*-NBA | – | – | (1–12) |
| | *m*-NBA/CsI | (1–4, 6–10) | – | (1–12) |
| **4** | *m*-NBA | – | – | (1–12) |
| | *m*-NBA/CsI | (1–2) | – | (1–12) |
| **5** | *m*-NBA | – | – | (2–12) |
| | *m*-NBA/CsI | – | – | (2–12) |
| **6** | *m*-NBA | – | – | (2,5,6) |
| | *m*-NBA/CsI | (1&5) | – | (2,5,6) |
| **7** | *m*-NBA | – | – | (2,5,6) |
| | *m*-NBA/CsI | (1&5) | – | (2,5,6) |
| **8** | *m*-NBA | – | – | (2,4–9,11–12) |
| | *m*-NBA/CsI | – | – | (2,4–9,11–12) |
| **9** | *m*-NBA | – | (1) | (2–12) |
| | *m*-NBA/CsI | – | – | (2–12) |
| **10** | *m*-NBA | – | – | (1&3) |
| | *m*-NBA/CsI | – | – | (1&3) |
| **11** | *m*-NBA | – | – | (2&3) |
| | *m*-NBA/CsI | – | – | (2&3) |

[a] No carbonyl loss observed from the $[M + Na]^+$ compounds.

ization significantly enhanced the ability to observe precursor ionization; in fact, of the 10 organometallics examined seven exhibited the most intense precursor ions upon addition of the cesium salt. In three of these cases the molecular ion was not observable prior to the addition of the salt. This increase in precursor ion abundance was attributed to charge localization on the cesium cation. As expected, the ability for cation addition appeared to be affected by the presence of the hydroxyl or nitrogen (compound **8** and **9**) functionalities; however, these functionalities were not necessary to obtain reasonable precursor ion abundance.

Cesium cation addition also appears, for these compounds, to be a more effective source of cationization than sodium cation addition or protonation. This greater affinity for the cesium cation attachment over other alkali ions for some systems has been previously reported [15a,30]. In addition the abundant precursor ion formation through cesium cationization along with the formation of reference ions $[m\text{-NBA}_x + \mathrm{Cs}_y\mathrm{I}_z]^+$ also made this method useful for accurate mass measurement. The matrix *m*-NBA was more useful than NPOE which also solvated these samples yet would not promote cationization regardless of the quantity of salt introduced into the matrix. The results obtained with the organodicobalt, organochromium, and organomolybdenum carbonyl compounds suggest that cesium cation addition may also be useful for other organometallic compounds.

## ACKNOWLEDGMENTS

The authors are indebted to Jay S. Siegel for his donation of the organochromium and organomolybdenum compounds. The authors are also grateful to C.-K. Hwang and Yasuhiro Torisawa for helpful discussions and also to the second reviewer for many worthwhile suggestions.

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