Supporting information for “Hands-on Inquiry-Based Qualitative Identification of Metals in Coins Utilizing Atmospheric Pressure Chemical Ionization Mass Spectrometry”

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1. Mass Spectrometric Studies

This practical introduces first-year undergraduates to mass spectrometry in a research setting and, consequently, we have made efforts to simplify the fragmentation patterns to make the spectra more accessible. In the practical the low fragmentation setting is used exclusively, again to simplify the procedure; however, work has been carried out showing fine-tuning of the temperature at which the ionization is carried out can further improve the spectra (**Table 1**, **2**).

**Table S1. Source parameters for the nine preset fragmentation modes on the Advion mass spectrometer. The current of the corona discharge is 5 μA for all entries.**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Entry | Fragmentation setting | Capillary T / °C | Capillary voltage / V | Ion source T / °C | Source voltage offset / V | Source voltage range / V |
| 1 | Low T/Low fragmentation | 135 | 120 | 250 | 25 | 0 |
| 2 | Low T/Med fragmentation | 135 | 160 | 250 | 30 | 5 |
| 3 | Low T/High fragmentation | 135 | 180 | 250 | 55 | 20 |
| 4 | Med T/Low fragmentation | 200 | 120 | 350 | 25 | 0 |
| 5 | Med T/Med fragmentation | 200 | 160 | 350 | 30 | 5 |
| 6 | Med T/High fragmentation | 200 | 180 | 350 | 55 | 20 |
| 7 | High T/Low temperature | 250 | 120 | 400 | 25 | 0 |
| 8 | High T/Med temperature | 250 | 160 | 400 | 30 | 5 |
| 9 | High T/High temperature | 250 | 180 | 400 | 55 | 20 |

Table S2. Mass spectrometric data for 10 pure samples of transition metal acetylacetonates including their method of acquisition and the relevant ion formed.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Entry | Complex | Frag.  method | Temperature | Mass expected*a* | Mass found*a* | Ion*b* |
| 1 | Co(acac)2 | Low | Low | 258.0 | 257.9 | [59Co(acac)2+H]+ |
| 2 | Ni(acac)2 | Low | Medium | 257.0 | 257.0 | [58Ni(acac)2+H]+ |
| 3 | Cu(acac)2 | Low | High | 262.0 | 261.9 | [63Cu(acac)2+H]+ |
| 4 | Zn(acac)2 | Low | Medium | 263.0 | 262.9 | [64Zn(acac)2+H]+ |
| 5 | V(acac)3 | Low | Low | 348.1 | 349.0 | [51V(acac)3+H]+ |
| 6 | Cr(acac)3 | Low | Low | 350.1 | 350.0 | [52Cr(acac)3+H]+ |
| 7 | Mn(acac)3 | Low | Low | 253.0 | 252.9 | [55Mn(acac)2]+ |
| 8 | Fe(acac)3 | Low | Medium | 254.1 | 254.0 | [56Fe(acac)2]+ |
| 9 | Co(acac)3 | Low | High | 257.0 | 256.9 | [59Co(acac)2]+ |
| 10 | V(O)(acac)2 | Low | Medium | 266.0 | 265.9 | [51V(O)(acac)2+H]+ |
| *a*Mass for most naturally abundant isotope shown; *b*positive ionization allowed more reliable characterization of the ion peaks than negative ionization. | | | | | | |

1. Example mass spectrometry data

Below are instructor generated data for all typical coinage metal acac complexes, for reference.

Table S3. Observed isotopic abundances using ASAP-MS.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Entry | Ion*a* | Expected Mass*c* | Expected abundance*c* | Observed Mass | Observed abundance |
| 1 | [*x*Ni(acac)2+H]+ | 257.0  258.0  259.0  260.0  261.0  263.0 | 100.0%  10.8%  38.5%  5.9%  5.3%  1.4% | 257.0  258.0  259.0  260.0  261.0  263.0 | 100.0%  12.5%  34.5%  5.2%  5.1%  1.2% |
| 2 | [*x*Cu(acac)2+H]+ | 262.0  263.0  264.0  265.0 | 100.0%  10.8%  44.6%  4.8% | 261.9  263.0  263.9  265.0 | 100%  8.5%  47.4%  5.1% |
| 3 | [*x*Zn(acac)2+H]+ | 263.0  264.0  265.0  266.0  267.0  268.0  269.0 | 100.0%  10.8%  57.4%  14.6%  38.6%  4.2%  1.3% | 262.9  264.0  264.9  265.9  266.9  267.9  -*b* | 100.0%  11.3%  64.0%  13.6%  39.4%  3.8%  -b |
| 4 | [*x*Cr(acac)3+H]+ | 348.1  -*b*  350.1  351.1  352.1 | 5.2%  -*b*  100.0%  27.5%  7.0% | 348.0  349.0  350.0  351.0  352.0 | 5.8%  17.8%  100.0%  32.1%  7.8% |
| 5 | [*x*Fe(acac)2]+ | 252.0  254.0  255.0 | 6.4%  100.0%  10.8% | 7.1%  100.0%  12.8% | 7.1%  100.0%  12.8% |

*a* Co and V were not included in this analysis due to the very high natural abundance of their major isotopes; *b* peak too faint for accurate relative abundance analysis; *c* obtained from ChemDraw prediction.

Figure S1. Mass spectrum of Co(acac)2.

A picture containing timeline

Description automatically generated

Figure S2. Mass spectrum of Ni(acac)2.

Chart

Description automatically generated

Figure S3. Mass spectrum of Cu(acac)2.

Timeline

Description automatically generated

Figure S4. Mass spectrum of Zn(acac)2.

A picture containing timeline

Description automatically generated

Figure S5. Mass spectrum of V(acac)3.

A screenshot of a computer

Description automatically generated with low confidence

Figure S6. Mass spectrum of Cr(acac)3.

Timeline

Description automatically generated

Figure S7. Mass spectrum of Mn(acac)3.

Graphical user interface

Description automatically generated

Figure S8. Mass spectrum of Fe(acac)3.

Chart, histogram

Description automatically generated

Figure S9. Mass spectrum of Co(acac)3.

A screenshot of a computer

Description automatically generated with low confidence

Figure S10. Mass spectrum of V(O)(acac)2.

Timeline

Description automatically generated