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CHARACTERIZATION OF THE GOLD MERCAPTOTRIAZOLE COMPLEX USING THE TANDEM QUADRUPOLE MASS SPECTROMETRY (TQD)

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Abstract

This work is aimed to characterize the gold complexes using the electron spray ionization (ESI), and atmospheric solids analysis probe (ASAP), tandem quadrupole mass spectrometer (TQD). It is demonstrated here that the use of the ASAP is more suitable than the ESI for a rapid analysis of the gold coatings in the solid and liquid state. The mass spectra indicate that mercaptotriazole and glycine can coordinate with the gold ion and to build complexes such as [Au-2MT-Gly] and [Au-2MT-2Gly].

Keywords*: gold complexes, mercaptoriazole, tandem quadrupole mass spectrometer*

1 INTRODUCTION

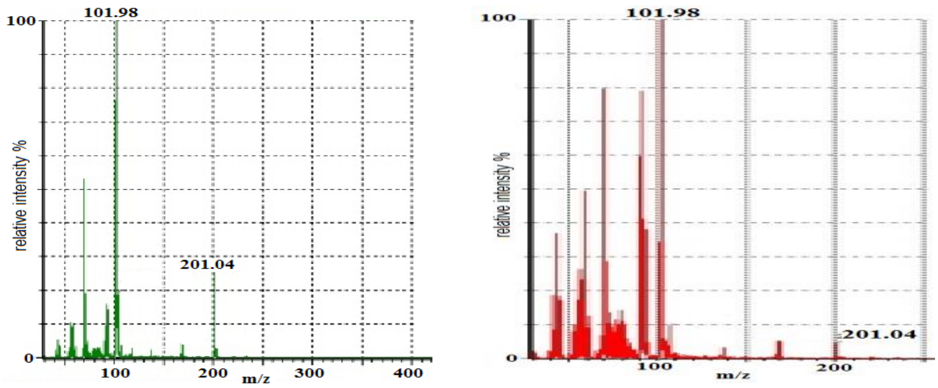
An electrolyte based on a gold complex with mercaptotriazole (Au-MT) was synthesized in a wide range of pH values from acidic to alkaline (pH = 2-12) [1]. After synthesizing the electrolyte, a detailed characterization of the complex in liquid and solid-state was performed in the entire range of complex stability in previous research [1-3]. The Au-MT solution was evaporated to dryness at room temperature to obtain and characterize the compound in crystalline form. Optical microscopy of these crystals showed that the crystals obtained at different pH values differ in color, size, and homogeneity. The most homogeneous (in color and size) and smallest crystals were obtained from electrolytes at pH=9. Raman spectroscopy provided a definitive confirmation of the established bond between metal ions and sulfur atoms [4]. Also, these techniques have indicated that the nitrogen atom in the ring of the award-winning compound Au-MT remains protonated, which does not support the original assumption derived from the UV spectrum analysis that at pH = 9, coordination of not only sulfur atoms but also nitrogen atoms with gold. The mass spectrometry method provided information helpful in resolving the structure of the Au-MT complex [1,4].

2 EXPERIMENTAL

In this work, the mass spectra of the gold complex crystals, obtained at pH=9 and the gold complex solution at the same pH value (Figure 1), were recorded on a tandem quadrupole mass spectrometer (TQD) (Acquity Tandem Quadrupole Detector, Waters, USA). This instrument has several ionization methods, such as the Electron Spray Ionization (ESI), Atmospheric Pressure Chemical Ionization (APCI), and Atmospheric Solids Analysis Probe (ASAP). The positive mode ESI and ASAP technique was used to record the spectrum of samples at pH=9 (solid and liquid). The results of the experiments were processed using the MassLynx 4.1 software package (Waters Corporation, Manchester, UK) [5-6].

3 RESULTS AND DISSCUSION

The pH value of nine was chosen because previous research has shown that the best gold coatings are obtained from electrolytes at this pH value [1, 7-10]. Figure 1 shows the ESI mass spectrum of this gold coating.

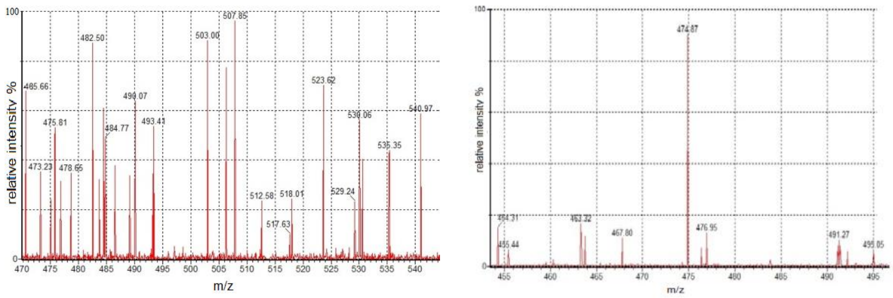


a) *b)*

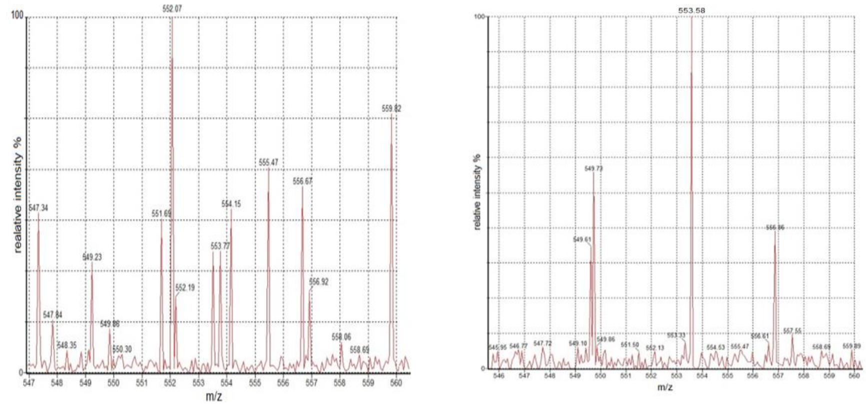
Figure 1 *The ESI mass spectra of the old complex at pH = 9 for: a) complex in the crystalline state, b) solution of gold complex*

The ESI MS is recorded at a corona voltage of 4.4 kV and cone voltage of 35 V for the complex in the crystalline state and solution state. These mass spectra contain peaks at *m/z* 101.98 and 201.04 which correspond to the molecular ion and dimer of mercaptotriazole, respectively.

Figures 2 presents two regions of the ASAP mass spectra of same sample: *m/z* 470-540 and *m/z* 540-560.



a) *b)*



c) *d)*

Figure 2 *The ASAP mass spectra of the gold complex at pH=9 for: complex in the crystalline state (a, c), and solution of gold complex (b, d)*

The important peaks are in the ASAP MS at *m/z* 473.23 for the solid sample (Figure 2a) and *m/z* 474.87 for the liquid sample (Figure 2b), which could correspond to the combination $[Au\text{-}2MT\text{-}Gly]^+$ and $[Au\text{-}2MT\text{-}Gly+H]^+$, respectively. The peaks at *m/z* 552.07 for the solid sample (Figure 2c) and *m/z* 553.58 for the liquid sample (Figure 2d), indicate the formation ion of $[Au\text{-}2MT\text{-}2Gly]^+$ and their protonated ion. In addition, for both solid and liquid samples (Figures 2c and 2d) the mass spectra contain *m/z* 549 that corresponds to $[Au\text{-}2MT\text{-}2Gly]^+$.

4 CONCLUSION

The ASAP MS unlike the ESI MS indicate the possibility that not only mercaptotriazole molecules but also glycine can coordinate with the gold ion. This is especially indicated by the intense peaks of molecular ion complexes in the crystalline state, which appear at *m/z* 473.23 and 549 which correspond to $[Au\text{-}2MT\text{-}Gly]^+$ and $[Au\text{-}2MT\text{-}2Gly]^+$, respectively. These peaks also appear in the mass spectra of the ASAP analysis of liquid samples of the complex synthesized at pH = 9, although at a much lower intensity ("noise" region). The ASAP-MS analysis also revealed the low-intensity peaks that could correspond to a fragment whose mass comes from the combination: Au-2MT-Gly. One of the dominant peaks in all mass spectra of the ASAP and ESI analysis is the peak that can originate from two molecules of mercaptotriazole (2MT), which further suggests the possibility of forming a disulfide bridge between these two molecules.

ACKNOWLEDGEMENTS

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