Electronic Supplementary Information of paper entitled "Speciation of copper by using a new fullerene derivative as a mixed-mode sorbent". (Paper Ref. B605537K)

Following characterisation data (spectra of FT-IR, ^{1}H-NMR and MALDI-TOF-MS) give complementary structural information about the C_{60}–rubeanic acid derivative.

The FT-IR spectrum (Model MAGNA-IR-500, Nicolet) of the fullerene derivative is shown in Fig. 1. The spectra for C_{60} and rubeanic acid have also been taken for comparison, both of them being in good agreement with the spectra reported in the literature.[1,2] The IR spectrum of the adduct in comparison with the spectra of C_{60} and rubeanic acid indicates absence of pristine modes of the acid and presence of unreacted C_{60} (peaks at 576 and 527 cm^{-1}). Analysis of this spectrum shows that it exhibits peaks at 669, 685, 802, 1020, 1100, 1260 and 2960 cm^{-1}. It is clear that the C-N mode which occurs at 1203 cm^{-1} in rubeanic acid is shifter to 1100 cm^{-1} in this case of the adduct, as described for other fullerene derivatives;[3] this is another evidence for adduct formation. Similarly, other peaks at 840, 705 and 643 cm^{-1} are also shifted in the adduct compared to pristine rubeanic acid, as proposed in Fig. 2 (structure of C_{60}–rubeanic acid). New modes are observed for the adduct which appears at 2960, 1260 and 1020 cm^{-1} and which are absent in the reagents spectra, probably corresponding to the new bonds which are formed in the addition of the quelating agent to the fullerene core. However, the C-S and C-S-H stretching vibration tend to give rise to very weak absorption in the infrared spectrum,[4] and are not observed in the adduct spectrum; in addition, FT-IR neither provides information about the N-H mode in the region 3300-3100 cm^{-1}.

The spectrum of ^{1}H NMR (C_6D_6) of the C_{60}–rubeanic acid derivative (see Fig. 3) was recorder on a Bruker Advance 400-WB spectromer. As can be seen, it is rather strange and does not supply useful information to elucidate the chemical structural of this derivative.

The spectrum of MALDI-TOF–MS (Model 4700/TOF-TOF, Applied Biosystems) of the C_{60}–rubeanic acid derivative is depicted in Fig. 4. The sample was prepared in a mixture of acetonitrile:water (70:30), containing a final concentration of 0.1% trifluoroacetic acid and mixed with a matrix of α-cyano-4-hidroxycinnamic acid (3 mg mL^{-1}), being the sample pH lower than 1. However, at this pH the derivative is hydrolysed; therefore in the spectrum of MALDI-TOF-MS of the derivative is observed two peaks at *m/z* 122 and 720 corresponding to stable fragments of rubeanic acid and fullerene monocation C_{60}^{+}, respectively, and any peak at *m/z* 842 corresponding to molecular weight of the derivative fullerene. According to these results, it can assume that the fullerene derivative has a molecular weight of 842 (see Fig. 5).

Figures

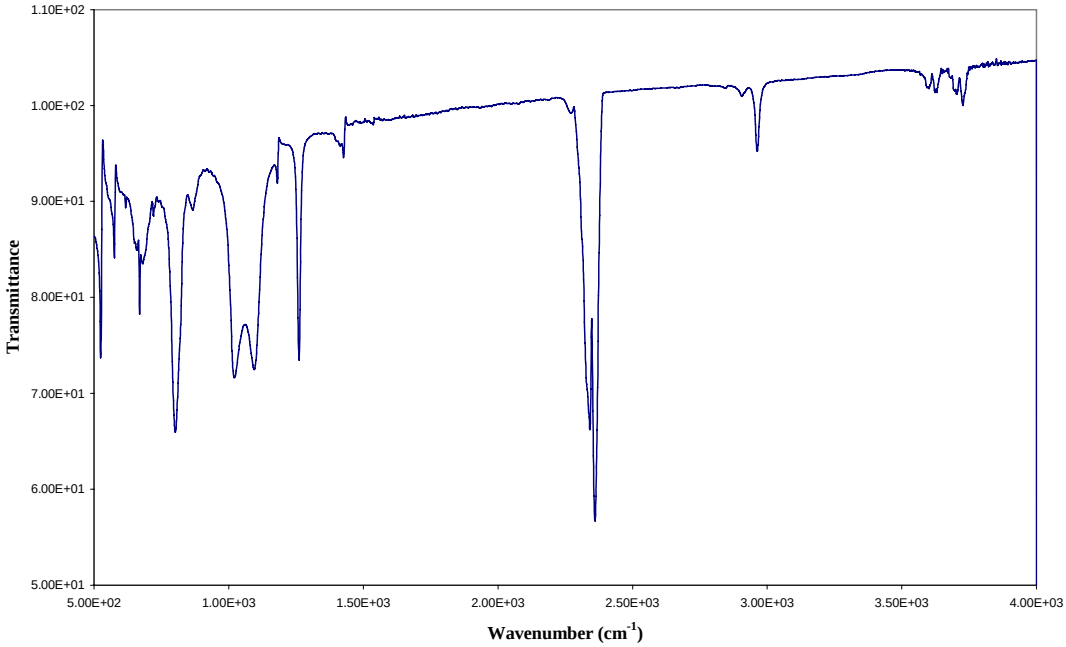

Fig. 1 FT-IR spectrum of C_{60}–rubeanic acid derivative.

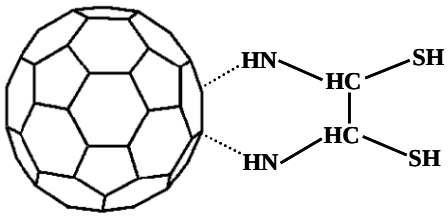

Fig. 2 Structure of the C_{60}–rubeanic acid derivative.

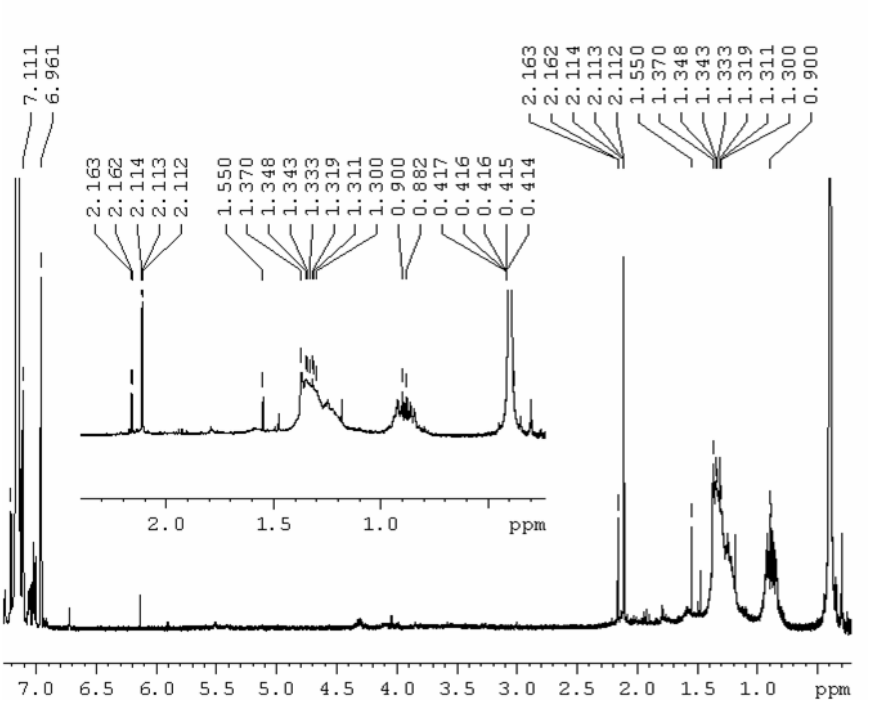

Fig. 3 ^{1}H NMR spectrum for the C_{60}–rubeanic acid derivative.

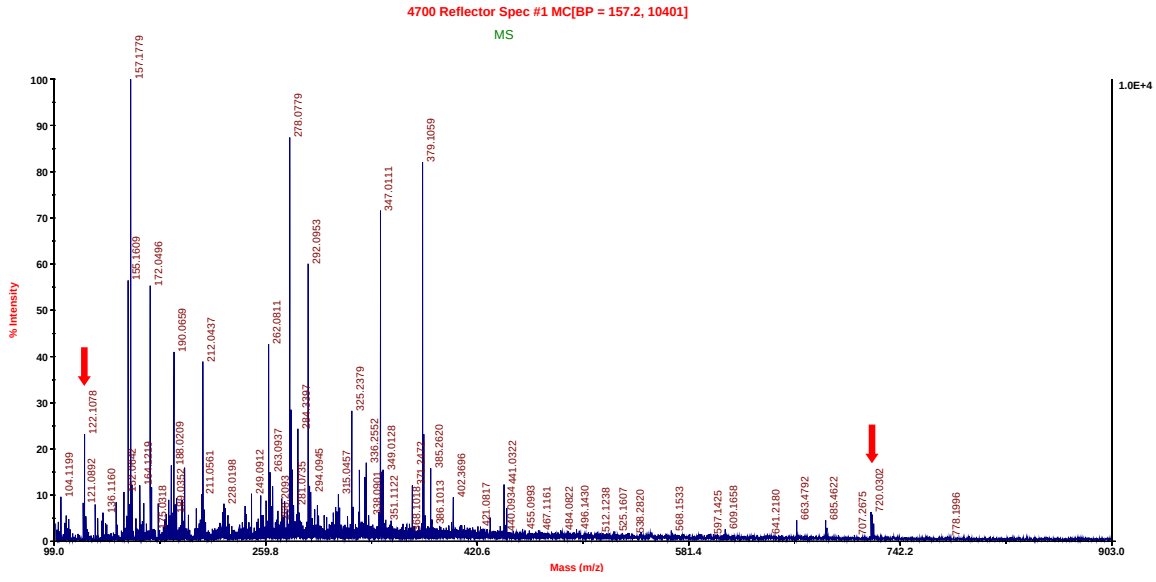

Fig. 4 MALDI-TOF-MS spectrum of C_{60}–rubeanic acid derivative.

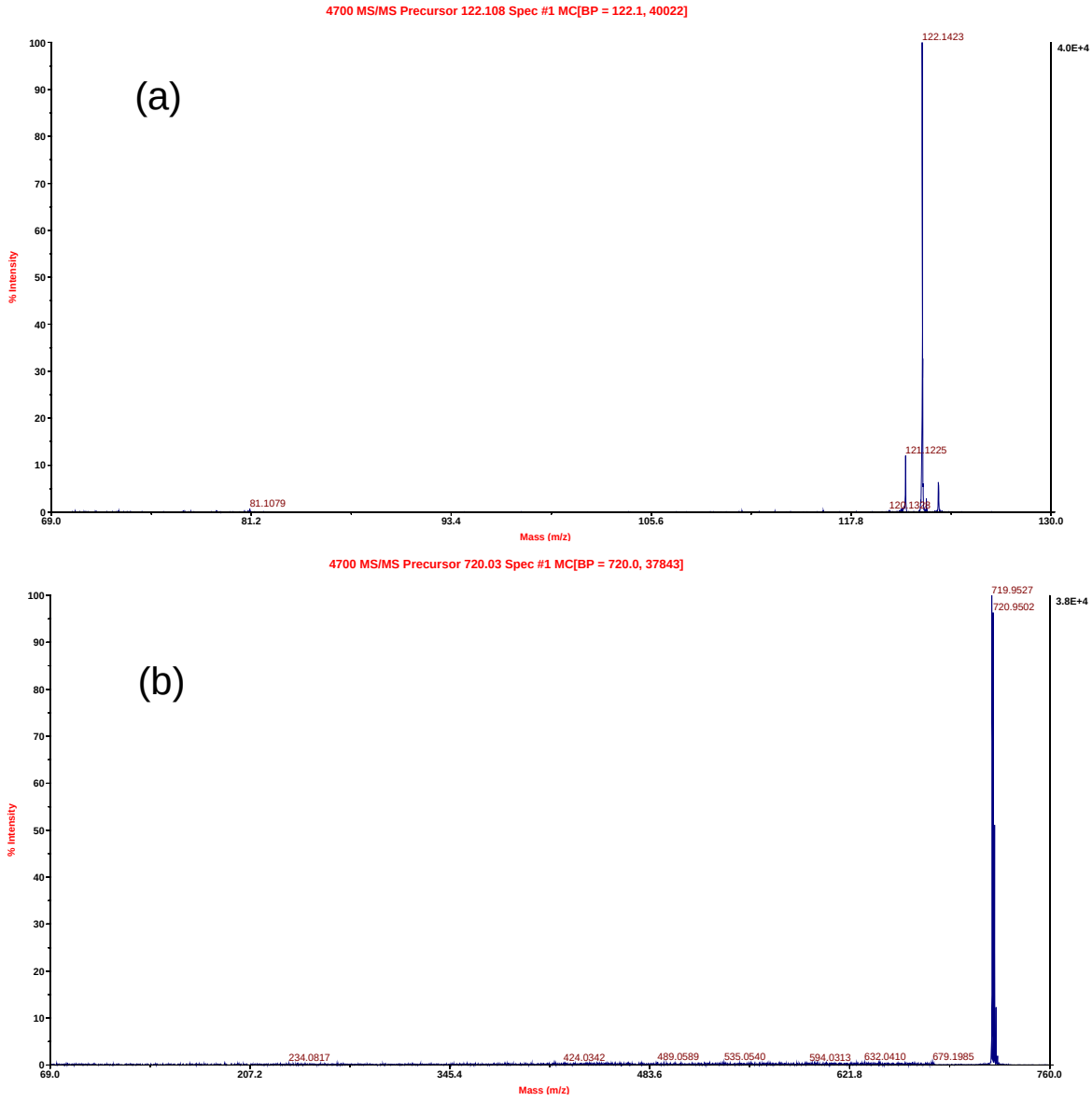

Fig. 5 MALDI-TOF-MS-MS spectrum of C_{60}−rubeanic acid derivative; (a) peak at *m/z* 122 and (b) peak at *m/z* 720.

References

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