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Report:

Platinum blues are a class of variously coloured, amorphous and biologically active Pt-complexes [1]. Pt-uridine green is an example of the antitumor active products which are formed when cis-$[Pt(NH_3)_2(H_2O)_2]^{2+}$ reacts with pyrimidine derivatives. Pt-pyrimidine species can be synthesized with several counter anions.

Pt-uridine blue [2] and green sulfate have been studied earlier by AWAXS. In both cases, the Pt-Pt coordination number, determined from the experimental Pt–Pt partial radial distribution function, was found out to be one or lower. This indicates that their dominating Pt-structures are mono- and dinuclear. To obtain evidence for the suspected ligand exchange Pt-uridine blue and green selenate were synthesized to obtain information on the Pt-Se distances.

Experimental

The syntheses of Pt-uridine blue and green were carried out according to those of the corresponding sulfates [3,4]. Microanalysis are by H. Kolbe, Mikroanalytisches Laboratorium, Germany.

Elem. anal. of Pt-uridine blue selenate: C, 18.73; H, 3.43; N, 8.34; Se, 13.52; Pt, 33.85 and Pt-uridine green selenate: C, 12.84; H, 3.14; N, 8.32; Se, 15.28; Pt, 38.31.

The samples were pressed pellets and the symmetrical reflection geometry was used. Pt-uridine green selenate was measured in the solid state by AWAXS with 3 energies below the PtLIII absorption edge and 2 energies below the SeK absorption edge. Pt-uridine blue selenate was measured with two energies below PtLIII absorption edge.

The scattered intensity was measured with a scintillation counter. To reduce the fluorescence of Pt at SeK edge an analyser crystal was installed but it was not used because the counting rate was too low. Another and more serious problem was that the colour and the structure of the sample changed during the experiments at SeK edge.

Results

The AWAXS data of Pt-uridine blue selenate were analysed with the DAS-technique. The results for Pt-uridine blue selenate are compared with those of Pt-uridine blue sulfate [2] in Fig. 1. The RDFs resemble each other closely at larger r, which suggests that the dominating Pt-structures are the same for both these products. According to model calculations, the differences at small r are caused by the contribution of the Se–O distances of the selenate counter ion and not differences in the first coordination sphere of platinum.

The preliminary analysis of AWAXS data of Pt-uridine green selenate suggests that the product is mononuclear while in the case of Pt-uridine green sulfate dinuclear Pt-units did form. The experimental results thus indicate that that the counter ion may affect the Pt-nuclearity of the product. No unambiguous Pt–Se distances can be identified in Pt-blue or Pt-green, but the model calculations exclude a distance smaller than 3 Å.

References

[1] J. P. Davidson, P. J. Faber, R. G. Fischer, S. Mansy, H. J. Peresie, B. Rosenberg and L. VanCamp, Cancer Chemother. Rep., Part 1, 1975, 59, 287.

[2] R. Serimaa, V. Eteläniemi, O. Serimaa, T. Laitalainen and A. Bienenstock. J. Appl. Cryst. in press.

[3] R. Serimaa, S. Vahvaselkä, T. Paakkari, T. Laitalainen, and J. Am. Chem. Sot. 115 (1993) 10036-10041.

[4] J. Pitkänen, T. Laitalainen, R. Serimaa, V. Eteläniemi, and M. Torkkeli. To be published.

Figure 1. The ΔRDF (thick line) and ΔDDF (dots) of Pt-uridine blue selenate and ΔRDF of Pt-uridine blue sulfate (thin line).