



3 July 2014, 10:00 - 11:00 CET

MINUTES

1. Background

- Diammonium hexachlororuthenate is scheduled for a range of tests, among others, a repeated dose test (OECD 422 or OCED 407/421, depending on the outcome of the acute tox test OECD 425).
- The testing lab informed us that the substance received did not match the substance description on the MSDS. They received a slimy paste, while the substance should have been a brown powder. We went back to the sample provider to confirm substance identity.
- The sample provider (Heraeus) has carried out a range of analytical measurements. Spectra were circulated to the Expert Group before the call.
- The sample provider concludes from the spectra that the substance is not 'Diammonium hexachlororuthenate' but 'Tetraammonium decachloro mu-oxo-bis(pentachlororuthenate(IV))', CAS 85392-65-0, the oxo-bridged dimer. Also, the substance is a mono-constituent substance, rather than a UVCB.
- The oxo-bridged dimer is on the PGM indicative list already as a mono-constituent substance (with Vale as LR), so this could be handled. However, re-assigning the above material to this substance would likely change the tonnage bands and require PMC to re-assess the testing requirements.
- In parallel, Vale has done some analysis on Tetraammonium decachloro-mu-oxodiruthenate, for which they are LR. This was also circulated before the call.

2. XRD spectra

- Heraeus concluded their substance is the oxo-bridged dimer by comparing their XRD spectrum to a literature reference spectrum of the potassium-salt of the oxo-bridged dimer.
- No evidence of any other Ru-Cl species was found in the XRD spectra measured by Heraeus.
- The XRD pattern of the ammonium oxo-bridged salt agrees with the theoretically adjusted pattern reported for the potassium oxo-bridged salt.
- AHCOs and AHClr should exhibit similar XRD spectra as their Ru analogue, however the published data do show very different spectra, further indicating that our substance is different from the hexachloro monomer.
- It is proposed to produce an XRD spectrum of the Johnson Matthey sample at Heraeus, so 3 XRD spectra produced under the same conditions can be compared (spectra of Vale and Heraeus samples are already available). XRD spectra can be produced ca. one week after receipt of the samples.

Actions:

- Heraeus to circulate reference paper with XRD spectrum of the potassium-salt of the oxo-bridged dimer.
- Expert Group to confirm if reference paper is correct in their identification.
- Johnson Matthey to send sample to Heraeus (2g per sample material required, wet sample, Heraeus will dry the sample using their drying process for consistency).
- Heraeus to produce XRD spectrum of the Johnson Matthey sample.
- Expert Group to reconvene once we have XRD spectra and elemental analysis (see below).

3. IR spectra

- IR spectra are in general less useful than XRD spectra here. Nevertheless these should be carried out by the three members as it is the PMC preferred method of sameness comparison



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for solids.

- Reference IR spectra have been published by Clarks et al. of oxo-bridged dimers: characteristic peak at 860-880 (oxo-bridge Ru-O).

Action: Dave Boyd to circulate reference paper with IR spectrum (Done - see below).

Reference for IR r (also contains Raman and electronic spectral data):

RJH Clark, ML Franks and PC Turtle, J Am Chem Soc (99:8) April 13 1977 pages 2473-2480

(Comment from Dave: Note particularly the synthesis method and analyses given in the 'Experimental Section-preparation of samples' at the top of page 2474, and the infrared band assignment in Table1.)

4. Elemental analysis

- Measured values were reported verbally by participants and compared.
- High variation was observed (from ± 25 to 37% Ru), probably due to contamination with other substances (e.g., chlorides). The elemental analysis needs to be repeated with purified substances. It was agreed to conduct all elemental analyses at the same CRO for consistency. The analysis will take a couple of weeks.

Actions:

- Registrants to produce purified substance to carry out elemental analysis for Ru and Cl.
- PMC to propose laboratory where elemental analysis should be conducted

5. Substance ID

- All samples measured so far are derived from the actual process.
- Heraeus used dried substances (at 105°C) which showed the same spectrum as moist compound = no water
- 'Water of crystallization': Heraeus stated that both the K monomer and the oxo-bridged dimer contain one molecule of water (mono-hydrate). DAB expressed some doubt of this.
- Mono-constituent substance (= well-defined substance) versus UVCB:
 - It is currently suggested (cf. PMC indicative list) to register the oxo-bridged dimer as a mono-constituent substance. It was noted that there is a requirement to demonstrate > 80% purity for mono-constituent substance. This might be possible via XRD.
- Diammonium hexachlororuthenate is currently listed as a UVCB substance
- Substance versus intermediate:
 - Both Diammonium hexachlororuthenate and the oxo-bridge dimer are currently listed as non-SCC intermediates
 - It was noted that ECHA recently issued a new practical guidance on intermediates and strictly controlled conditions (SCC)
(http://echa.europa.eu/documents/10162/13655/pg16_intermediate_registration_en.pdf).

Action: PMC to re-confirm SCC status of all PGM intermediates in light of new guidance