



Precious Metals and Rhenium Consortium Re WG meeting

Brussels, 23 April 2010
Metals Conference Centre
Rue du Duc 100, B-1150 Brussels



1. Introduction and welcome



Proposed Agenda

1. Introduction and welcome
 - Including pending actions from last meeting
2. Re project progress
 - Phases I - V (with focus on III)
 - Overall timeline
3. AOB, next meeting and closing remarks
 - Next meeting, preparation for Assembly meeting and preparation for IUCLID



Actions from last meeting (Brussels, 4 Dec 2009)

	What?	Who?	When?
1	To confirm with Powmet (and BASF in due course) whether these companies agree to have their names mentioned as data holders in the PMC's dossiers.	CB	21 Dec 2009
2	To revert to the PMC with the adjusted timeline of tier 1 confirmed by AQura (and confirm whether first draft report is likely to be finalised in March 2010 – include original laboratory reports).	WCA	Jan 2010
3	To obtain a confirmation from the UK HSE about their interpretation on animal testing and testing proposals requirements	WCA	21 Dec 2010
	To consult Member State representatives to capture their views on animal testing.	Re WG	Jan 2010
4	To prepare one ID Card per substance and intermediate in scope.	CB	Jan 2010
	To revise ID Cards before they are used to address sameness and Lead Registrant election with each pre-SIEF.	WCA and Re WG	Jan 2010
5	To organise an <i>ad hoc</i> discussion on the Nickel alloy scrap case with the concerned companies after the next PMC Refiners WG meeting.	CB + HW	Week 20 Jan
6	To block 2010 Re WG meeting dates with WCA and BIBRA.	CB	21 Dec 2010



2. PGM Project development and progress



Phase I



Phase I: Literature search



- Addition of K perrhenate as intermediate in 1-10 band to scope agreed by Re WG in Jan 2010
- Literature search performed for K perrhenate:
 - 1966 “*Pharmacology and toxicology of potassium perrhenate and rhenium trichloride*” identified but not possible to use as key study
 - Invoice will be sent to relevant company as agreed



Phase II





Phase II: Category building

- Categories formed as ‘families’ within which read-across is scientifically sound and relevant

- Preliminarily based on:
 - Ion oxidation state
 - Counter-ion (e.g.: ammonium)

- Preliminary categories:

- Re0: Rhenium metal
- Re7:
 - Perrhenic acid
 - Sodium rhenate
 - Dirhenium heptasulphide
 - Potassium perrhenate
- Re7A: Ammonium perrhenate

Reasonable read-across source

Overall, worst case read-across source



Phase III



Phase III: Testing programme - Tier 1



- Testing recommendations discussed at Sep 2009 in Sonning

- Agreed to test in tiers

- Tier 1 scope:

- UV vis, T/D, melting point
- Algae, *Daphnia*, fish
- Respiration inhibition

- Tier 1 original timeline:

- Start in Jan, reporting in March 2010



Tier 1



Phase III: Testing programme - Tier 1



• UV Visible and pH:



- Tests conducted on Re, Na perrhenate, APR, perrhenic acid



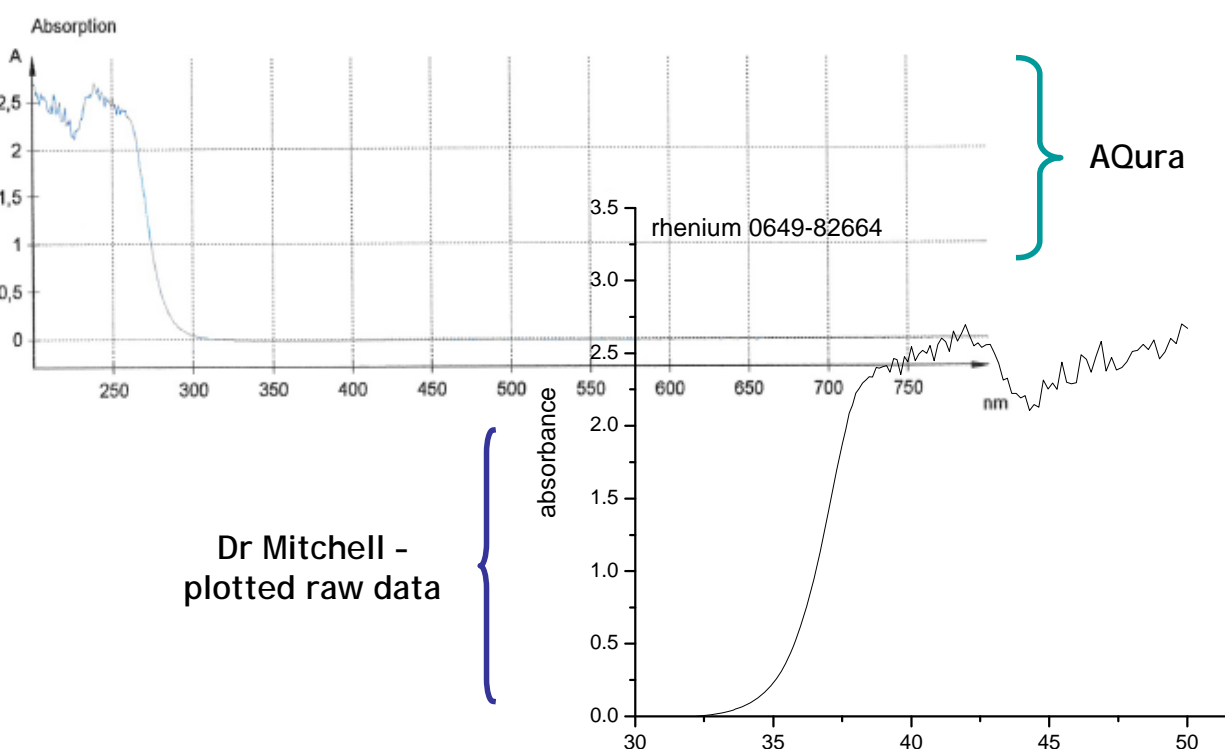
- Results received on 1 March



- Forwarded to Dr P. Mitchell (University of Reading) for interpretation/assessment



E.g.: UV vis spectrum Re metal (pH = 2.1)





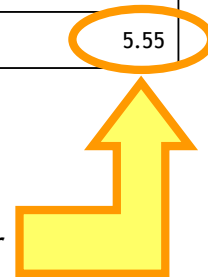
Rhenium concentrations and molar extinction coefficients calculated using Beer's law

Table 1 Perrhenate concentrations and uv absorbance

	Formula	Molar mass	g	ml	molL ⁻¹	path/cm	Absorbance	Molar extinction /1000mol ⁻¹ cm ²
Na rhenate	NaReO ₄	273.19	0.507	5.00	0.37	1.00	3.00	8.08
Perrhenic acid	HReO ₄	251.21	1.012	10.00	0.40	1.00	3.00	7.45
Ammonium perrhenate	NH ₄ ReO ₄	268.24	0.999	10.00	0.37	1.00	3.00	8.06
rhenium	Re	186.21	1.007	10.00	0.54	1.00	3.00	5.55

Dominant species in solutions is the perrhenate ion → supports read-across strategy

Re metal: Perrhenate is formed by dissolution of a an oxide layer from rhenium metal in contact with water



Discussion of results - UV vis

- Difference in spectrum suggested to be due to:
 - Re filtered before measurement
 - Na perrhenate, Ammonium perrhenate not filtered before measurement
 - Elementary mistake
- **For discussion:**
 - Sufficient for read-across strategy or re-measure spectra with fresh, filtered solutions?
 - Need to perform a UV test on K perrhenate?



Phase III: Testing programme - Tier 1



- T/D:



- T/D test conducted on Re



- Aim of T/D is to fill water solubility data gap, confirm read-across strategy and refine/avoid ecotox classification



- Start with 24h screening test (100 mg/l for 24h in a pH range from 6 to 8,5)



- K. Rothenbacher (EPMF) and A. Peters (WCA) visited testing house and discussed preliminary T/D results on 9 March 2010



Phase III: Testing programme - Tier 1



- T/D (Cont.):



- AQura reported on preliminary results on 12 March 2010:
 - pH Control done by addition of $\text{KH}_2\text{PO}_4/\text{Na}_2\text{HPO}_4$ instead of CO_2 control
 - No filtration: analysis after sedimentation of Re
 - Solubility of rhenium in the screening test is approximately 1,7 mg/l
- Discussion of results (PMC with WCA, WCA with AQura):
 - Sample not filtered
 - Influence of phosphate in Re's solubility





Phase III: Testing programme - Tier 1



• T/D (Cont.):



- AQura repeating T/D screening test with filtered sample and CO₂ controlled pH



- Meanwhile, full test:

- Short term transformation: 100 mg/l for 7 days in a pH range from 6 to 8.5
- Long term transformation: 1 mg/l for 28 days in a pH range from 5.5 to 8.5



on hold...



- Q: OK to continue with AQura?



Phase III: Testing programme - Tier 1



Melting/freezing point:



- Test conducted on Perrhenic acid



- Regular test method incompatible with Perrhenic acid
→ agreement by group to follow specific method (DSC analysis with corrosion resistant gold-plated steel crucibles)



- Confirmation from PMC given on 7 March 2010 to WCA



- When can the report from AQura be expected?



Phase III: Testing programme - Tier 1



• *Algae, Daphnia, fish:*



- No information received on progress of these tests on APR



- Awaiting T/D results?



- WCA?



Phase III: Testing programme - Tier 1



• *Respiration inhibition:*



- Oxygen consumption/liter/hour measured and built onto a respiration curve



- % inhibition calculated at each test concentration is plotted against concentration to derive an EC50



- For APR, 3h EC50 > 1000 mg/l
 - Guide to the likely toxicity of APR to activated sludge sewage treatment or to waste water micro-organisms
 - APR not likely to be toxic to activated sludge or waste water micro-organisms (even in presence of high quantities of phosphates)



- Test valid
 - Q = how does this feed into the derivation of a PNEC_{stp} for APR?
 - Any other comment?





Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

Tier 2



Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

Phase III: Testing programme - Tier 2

- Testing recommendations pending outcomes of tier 1
- Agreed to test in tiers:
 - Tier 2 scope: CLP and tox endpoints
 - Tier 2A: CLP
 - Tier 2B: Tox



Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

Tier 2A



Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

Phase III: Testing programme - Tier 2

- **CLP** - for all materials in scope!

- Flammability
- Explosive properties
- Self-ignition
- Oxidising properties
- Corrosivity to metals

- **Re WG to judge:**

- Likelihood for classification
- Need for test to discard/confirm classification



Likely classification, derogations or waivers, or testing needs

- Ag 
- Au 
- Ir 
- Os 
- Pd 
- Pt 
- Re 
- Rh 
- Ru 

CLP endpoint	Flamm. (pyrophoricity)	Self-ignition	Explosive properties	Oxidising properties	Corrosivity to metals
Rhenium	> 300 °C powder Re oxidises - T	No test	No test	No test	No test
Perrhenic acid	No test	No test	No test	Test	Yes (no test)
APR	No test	Required	No test	No test (info available)	Transported in plastics - check
Na rhenate	No test	No test	No test	Test	Dr Mitchell to check
K perrhenate	No test	Check MP	No test	Test	Dr Mitchell to check
Dirhenium heptasulphide	Test	Check MP	No test	No test	Check



Tier 2B

- Ag 
- Au 
- Ir 
- Os 
- Pd 
- Pt 
- Re 
- Rh 
- Ru 



Phase III: Testing programme - Tier 2

- **Tox** - All on APR:

- Skin sensitisation
- Eye irritation (also Re)
- Cytogenicity (Micronucleus)
- Mutagenicity (Ames replaced by Mouse Lymphone Assay)
- Acute toxicity
- 28 days repeated dose toxicity

See WCA's comments

Discussion around route of administration - see next slide



Route of administration for Acute and repeated dose toxicity studies

- Oral EC50 already exists for APR
 - No need to conduct Acute toxicity study (oral) for APR
- Next most relevant route of exposure may be inhalation
 - Dependant on potential for inhalation of typical APR powder
 - Can only be confirmed by the assessment of a combined PSD + particle form + aerodynamic diameter evaluation
 - APR's LR and WCA have been in discussion to resolve the above (see next slide)



Is APR inhalable/respirable?

Refinement of assessment

- LR's APR is representative of what is placed on the market
- LR's APR powder sample close to spherical as per REM but also proportion of larger (100 μm borderline) non-spherical particles
- Way forward (for consideration by group):

- 1 **Worst case approach** → Accept REM and go ahead with inhalation test (without certainty of reaching a test atmosphere + costly)
- 2 **Stepwise approach** → Obtain Mass Median Aerodynamic Diameter by cascade impactor or dustiness test + respiratory tract deposition modelling

2 preferred



Tier 2 Timeline



Phase III: Testing programme - Tier 2



• Timeline:



- Tier 2A → CLP asap to complete CLP notification by 3 Jan 2010



- WCA to confirm testing house - mid May?
- Members to prepare samples - mid May?
- CB to prepare formal sample request - mid May
- Members to send samples (including certificate of analysis and D10, D50, D90 values) - end May ?



- Tier 2B → Tox - to be decided by Re WG



- WCA to confirm testing house for cascade impactor, dustiness and deposition modelling work
- WCA to confirm testing house for tox tests
- CB to circulate sample preparation/submission request in due course



Phase IV





Uses, exposure and emissions questionnaire



- Draft questionnaire received from WCA
- Internally reviewed - comments sent to make it more user-friendly
 - Adjusted version available when?
- Ag experience shows:
 - Gathering information is not straightforward
 - Needs to done at manufacturing and DU level
 - Received information not always high quality
 - Requires refinement loops
 - Exposure calculation depends on PNEC and DNEL which can only be derived once full effects data (testing data set) is available



Uses, exposure and emissions questionnaire



- Options proposed to Re WG:
 - Circulate questionnaire asap?
 - Circulate questionnaire in 2011, after effects data is (almost entirely) gathered
- How long will it take to complete questionnaire?

Preference of PGM WG,
Ideally Re and PGM in parallel



Phase V



IUCLID 5 Hosting System

- System set-up
- Launched for PMC in tiered approach, starting with Ag
- Each LR will be given a username and password to follow the completion of their IUCLID 5 file “live”



Project timeline towards registration



Status of Re project

Phase	Status
I: Literature search	✓
II: Draft test derogations & Integrated Testing Strategy report	✓
II: Final test derogations & Integrated Testing Strategy report	Awaiting today's conclusions
III: Testing programme, tier 1	Started in Jan 2010 Finalisation expected in March 2010 but need to repeat tests
III: Testing programme, tier 2	Awaiting results of tier 1 and today's conclusions (CLP and Tox)
IV: CSA/CSR (including ES)	Draft Questionnaire received & commented
V: IUCLID 5 preparation	Ongoing

✓ Completed phase

Earliest registration deadline
Jan 2012



Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

3. AOB, next meeting and closing remarks



Ag



Au



Ir



Os



Pd



Pt



Re



Rh



Ru

Next meetings

- 15 June 3 pm: Re WG Telcon
- 18 June: Hamburg, PMC Mtg
- 23 Sep: Brussels, Re WG Mtg
- 2 Dec: Brussels, PMC Mtg
- 3 Dec: Brussels, Re WG Mtg



To be discussed at Assembly meeting



- Re project progress
- Consortium Agreement review:
 - Integration of so far standalone parts
 - Clarification/tidying up
 - Cost-sharing for intermediates
 - Lead Registrant's Declaration of Commitment
- Budget predictions (till 2020)
- Letter of Access
- And many other topics! → We need you to be present or represented



IUCLID 5 training session



- Guidance document prepared in two batches:
 - Preparation for registration
 - 2 pages check-list
 - Print screens providing idea of amount of detail that will be needed from each registrant
 - Registration recommendations
 - Inspired from Ni consortia guidance
 - Addressing registration recommendations for:
 - LR vs non-LR
 - Substance, simple intermediates, complex intermediates (Refinables)
 - ~ 25 pages of recommendations
 - Print screens with examples



Preparation for registration



• Pre-registration

- Those companies having not pre-registered need to find solution with ECHA



TODAY

• Start preparing now

- Company-specific IUCLID 5.2 sections



• Not before autumn 2010

- Preparation for registration following LR's submission



Start preparing now



Substance identification

- Solids: ICP-MS or XRF, XRD and IR spectroscopy
- Liquids: ICP-MS or XRF and Raman spectroscopy



Applicable to Re materials too?



Granulometry

Sieving with 100 μm sieve

- If all particles > 100 μm \rightarrow stop testing
- If some/all particles < 100 μm :
 - Determine relative density and water solubility
 - If water
 - soluble: microscopy, sedimentation, laser dropller
 - insoluble: microscopy, sedimentation, laser dropller, electrical sensing
 - If inhalation route of exposure relevant (and inhalation test possibly needed): laser diffraction, cascade impactor, rotating drum/dustiness, continuous drop method to obtain Mass Median Aerodynamic Diameter





Start preparing now



• IUCLID 5.2



- PMC will pre-populate files in due course



- Meanwhile, Members shall install IUCLID 5.2 (≠ from pre-registration version) and start completing company-specific information



• Live IUCLID 5.2 show



To be circulated by mid-May 2010, before Assembly meeting



• Consortium Agreement



• Budget predictions



• Letter of Access



• IUCLID 5.2 guidance document



THANK YOU!!!!