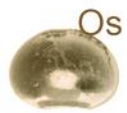
Precious Metals and Rhenium Consortium PGM Experts meeting

Conference Call

16th November 2011

1. Introduction and welcome

- Competition Law reminder
- Approval of the Agenda
- Approval of minutes last meeting
- Objectives



2. PGM Project Scope

Scope – Recent Changes

Substance	Feedback	Conclusion
RhCl3 and RuCl3	• Anhydrous form not produced, • Not available for testing	• No testing of anh. forms • Prepare dossiers for hydrated forms only
Diamminedichloropalladium	• Mixture of several isomers • Equilibirum mixture primarily trans isomer	• Test representative commercial product
PtCl2	• Not supported any longer	• Omit from scope
IrCl4	• Need feedback on no dispersive use => Annex III exemption	• Added back to scope (1-10 tpa)
PdO2	• Substance not available for testing	• Tests waived b/c non-availability of test material

Metal "blacks" vs. powders

- Most metals available in 2 grades: standard powders and very fine "blacks"
  - Different particle sizes = different properties possible (e.g., inhalation potential)
  - Dustiness: testing both grades
  - No difference in classification, though (blacks were not self heating)
- Need to decide which grade(s) to test

Metal "blacks" vs. powders

- Caveat
  - New EU (Commission Recommendation) definition for nanomaterials published
    - D50 (50% of particles) < 0.1 µm, OR
    - Surface area > 60 m^2/cm^3
  - Need to confirm whether or not blacks fall under nano definition
- Next steps
  - Confirm with members who is producing blacks
  - Determine PSD of blacks
  - Evaluate results dustiness tests

2013 Registrations

	Highest tonnage	DL	Dossier
Diammonium hexachloropalladate *(Intermediate)*	• SCC: 100-1000 tpa • Non-SCC: 10-100 tpa	• 2013 • 2018	• Available information • Full dossier
Diammonium hexachloroplatinate *(Intermediate)*	• SCC: 100-1000 tpa • Non-SCC: 10-100 tpa	• 2013 • 2018	• Available information • Full dossier
Diamine dichloropalladium *(Substance)*	• 100-1000 tpa	• **2013**	• **Full dossier**

Required by 31 May 2013

- 1 full dossier
- 2 intermediate dossiers (reporting available information)
- Caveat: need robust documentation of SCC!
  - Responsibility of the concerned company(ies)
  - WCA and PMC have to assume SCC compliance

Options Diamine dichloropalladium

Substance 100-1000 → 2013 registration

- **OPTION 1: Prepare full dossier by 2013**
  - Very limited time available = expedited testing approach needed
  - Discussion detailed timing: agenda point 5
- OPTION 2: Prepare as much as technically feasible and update afterwards
  - Test concurrently with other PGMs
  - Consider risks; e.g., tentative DNEL = higher assessment factor = lower margin of safety
- Recommend Option 1 = “Fast tack” approach

Options Diamine dichloropalladium

- "Fast tack" approach
  - All testing to be completed in 2012
  - Initial discussion with PEG: drafted fast-track testing plan (email 15 Nov 2011)
  - Note: also fast-track approach to requesting quotes required
  - Tests that can be started now: already started (irritation, dustiness) or in process of being started (ecotox tests)
  - Other tests to start asap
  - Time line: agenda point 5.2

Options Diammonium hexachloroPt and Pd

Intermediates 100-1000 t/a SCC registrations by 2013, followed by 10-100 t/a non-SCC registrations before 2018

- **OPTION 1: Prepare 'all available information' dossiers by 2013 and update afterwards**
  - Legal compliance ensured
  - Double registration preparation/submission work (unless updates are foreseen anyway)
- OPTION 2: Prepare full 10-100 t/a dossiers by 2013 and no need to update afterwards
  - Legal compliance ensured
  - No double registration preparation/submission (unless updates are foreseen anyway)
- Recommend Option 1

Scope – Karstedt Concentrate

- Background
  - Significant data gaps
  - Important product for silicones industry; will also register
- Meeting Reconsile consortium 23 Sept. 2011, Bxl
  - Constructive discussion, both parties interested in sharing efforts/data/costs
  - Need for pre-studies: evaluate environmental fate/ biologically active form
  - Probable scenario: hydrolysis to siloxane ligand and Pt(0)
    - Reconsile hold data on siloxane ligand
    - PMC currently developing data on Pt (0)

Scope – Karstedt Concentrate

- Conclusions/ Next Steps
  - Agree on substance identity
  - Work out details of collaboration
    - Letter of intent (done)
    - Non-disclosure agreement (done)
    - Need to decide on lead registrant
  - WCA and PFA (Peter Fisk Associates; siloxane consultants) to meet and recommend way forward
- Other
  - Siloxane ligand might be persistent and bioaccumulative; properties currently under evaluation

ITS Reports - update on progress

Background *(slide from last PGM call)*

Overall ITS strategy

1. Identify exemptions: Annex III, intermediates - ***done***
2. Clarify read across options - ***done***
3. Enabling tests (pH, water solubility, bioaccessibility, particle size, etc) - ***ongoing***
4. Develop integrated testing strategy (ITS) – ***next slides***

ITS

- Scope
  - Ambitious testing scope: 66 Substances x 35 Reach endpoints x n-Studies = many studies, complex discussions
- ITS reports and Enabling Tests
  - Main area of activities in past months
  - Detailed technical discussions
  - Close involvement of PEG
  - External peer reviewers where necessary (TK, genotoxicity, etc)

Recent developments - Timing

- ITS reports
  - 2 Phases
    - Initial ITS/ enabling tests recommendation + initial test recommendations
    - Updated ITS with enabling tests/ full test recommendations
  - Timing
    - Initial ITS reports slightly delayed: reports received end August/September instead of June
    - September: PEG commenting round
    - Early November: draft final reports
    - Revised ITS reports when results enabling tests available = Q1 2012
    - Bottleneck: shipping of samples
  - Fast track testing plan for 2013 substance

ITS

Test	No of substances Estimates
Phys.-chem. tests	
Boiling point	1
Rel. density	31
Vapour pressure	12
Water solubility	19
Flammability	6
Self ignition	5
Oxidising prop.	6
Explosivity	1
Enabling tests	
Dustiness	19
Bioaccessibility test	12
TD test	2
Mammalian tox	
In vitro eye and skin irritation	6
Sensitisation	6
Genetic toxicology	10
In vivo acute studies	6
Repeated dose/reprotox	ca 11
Environment	
Algeae/fish/daphnia	6
Activated sludge	5

2012 Outlook

- ITS update
  - Update ITS with data from enabling tests
  - Agree on final testing scope
- Testing Programme
  - Start majority of testing programmes
- Exposure Scenarios & CSR
  - Collect use/ exposure data
- Update CLP

Enabling Tests

Oxidising Properties – tests at BAM

Substance	CAS	State	Status
Iridium dioxide	12030-49-8	solid	done
Platinum dioxide	1314-15-4	solid	done
Platinum dinitrate	18496-40-7	solution	done
Dipotassium tetraoxoruthenate	31111-21-4	Solution in aqueous KOH	Not available in pure form test K2OsO4 instead, testing ongoing
Ruthenium IV oxide	12036-10-1	solid	Ongoing, substance provided late
Rhodium trinitrate	10139-58-9	Solid (hydrate)	done

- Tests ongoing at BAM, Berlin
- Results in November

Water Solubility -Standard tests

- OECD 105 (shake flask test)
- Testing at JMTC
- Results by end of year

~~10025-65-7 Platinum dichloride~~
Dicarbonyl(pentane-2,4-dionato-O,O')rhodium 14874-82-9
Triammonium hexachlororhodate 15336-18-2
Diammonium sodium hexakis(nitrito-N)rhodate 64164-17-6
Diammonium hexachlororuthenate 18746-63-9
Tetraammonium decachloro-mu-oxodiruthenate(4-) 85392-65-0
Rhodium trichloride (anhydrous) 10049-07-7
Ruthenium trichloride 10049-08-8 (anhydrous)
Iridium dioxide 12030-49-8

Water Solubility – Non standard tests

Substance	Comment
Rhodium trihydroxide 21656-02-0	cannot be isolated as dry solid
Rhodium tris(2-ethylhexanoate) 20845-92-5	solution in 2-EH
Ruthenium trihydroxide 12135-42-1	damp unisolated solid
16970-55-1 Dihydrogen tetrachloropalladate	only available as solution
68478-92-2 Platinum, 1,3-diethenyl-1,1,3,3-tetramethyldisiloxane complexes / Karstedt concentrate (10-100 tonne substance)	decomposes in water
14221-01-3 Tetrakis(triphenylphosphine)palladium	decomposes in water
10102-05-3 Palladium dinitrate (and solution)	likely to precipitate in water
18496-40-7 Platinum dinitrate	likely to precipitate in water
12036-04-3 Palladium dioxide (and hydrate)	substance not isolated
16919-73-6 Dipotassium hexachloropalladate	substance not isolated
19168-23-1 Diammonium hexachloropalladate	substance not isolated

- Testing at JMTC
- Results by end of year

Water Solubility – TD - Other

Substance	Status
Palladium monoxide (hydrate) 1314-08-5	TD test CANMET
Palladium dihydroxide 12135-22-7	TD test CANMET
14024-61-4 Palladium di(4-oxopent-2-en-2-oate)	Existing data sufficient, no TD test required
Dirhodium trisulphate 10489-46-0	Check if limit value available - done
Potassium tetraoxoruthenate 31111-21-4	Check if limit value available - done
61495-96-3 Tetramminepalladium(2+) diacetate (1-10 tonne substance)	Check if limit value available - done

- Testing at CANMET
- Results by end of year

Bio-elution tests

- Testing at CIMM, Chile
- Currently working on sample preparation
- Need to decide if testing of blacks required
- Scope:

Substance	CASRN	Comment
Platinum	7440-06-4	
Platinum black		further discussion needed
Palladium	7440-05-3	
Palladium black		further discussion needed
Palladium monoxide	1314-08-5	
Palladium dihydroxide	12135-22-7	
Palladium (II) di(4-oxopent-2-en-2-oate)	14024-61-4	
Rhodium	7440-16-6	
Rhodium black		further discussion needed
Diammonium sodium hexakis(nitrito-N)rhodate	64164-17-6	
Ruthenium	7440-18-8	
Ruthenium black		further discussion needed

Dustiness tests

- To estimate potential for inhalation exposure
- Scope
  - Testing of first batch completed at DMT (9 substances) completed, reports available
  - No testing of chloroplatinates (3 substances)
  - **Testing ongoing: Pt, Pd, Rh, Ru, PdO, PtCl2**
  - **“Blacks”: also testing Pt-, Pd-, Rh-, Ru-black**
- Next steps
  - Lung deposition modelling (MPPD) based on dustiness data
- Timing
  - 9 substances: MPPD modelling by end November
  - 10 new substances
    - dustiness tests by end November (if samples arrive on time)
    - MPPD modelling by end December

Dustiness tests - Draft results diammine-dichloropalladium (fast track substance)

Fraction	Amount (mg/g)
Total dustiness	220.18
Inhalable fraction	138.77
Thoracic fraction	41.40
Respirable fraction	8.50

Dustiness data

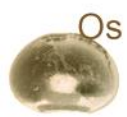
Model	Mode	Head (%)	TB (%)	Pulmonary (%)	Total (%)
Symmetric	oral	87.5	7.4	2.2	97.2
	Oronasal augmenter	52.2	0.31	0.44	52.9
5-Lobe	oral	87.5	7.2	2.2	97.0
	Oronasal augmenter	52.2	**0.29**	**0.44**	52.9

MPPD modeling

- Compared several models/modes => similar outcomes
- Results highlighted in bold use parameters used in Ag dossiers => consistence
- <1% of exposure via lung
- Recommendation: **no inhalation tox testing needed** (DNEL can be derived via route-to-route extrapolation) = will save significant cost and time

In-vitro irritation/corrosion testing

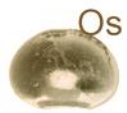
- Testing strategy
  - In-vitro skin corrosion(EPISKIN) – if positive: stop
  - In-vitro eye irritation (ICE or BCOP) – if positive: stop
  - In-vivo eye irritation
- Skin corrosion data needed to decide on other tests
  - Eye irritation
  - Sensitisation
  - Acute toxicity
  - (Genotoxicity)



In-vitro irritation testing

- Testing ongoing
  - CiToxLAB (EPISKIN/ ICE)
  - Harlan (EPISKIN/ BCOP)

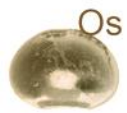
Substance	In vitro skin (EPISKIN) test	In vitro eye (e.g. BCOP or ICE) test	In vivo eye irritation test	
Diammonium hexachlororuthenate (CAS 18746-63-9)	X	X	X	Low water solubility / liable to sediment rapidly from suspension – ICE recommended
Carbonyl(pentane-2,4-dionato-O,O')(triphenylphosphine)rhodium (CAS 25470-96-6)		-	-	Due to reduction in tonnage, substance now qualifies for Annex III exemption. No testing required any longer.
Diammonium sodium hexakis(nitrito-N)rhodate (CAS 64164-17-6)	X	X	X	Low water solubility / liable to sediment rapidly from suspension – ICE recommended
Diamminedichloropalladium (CAS 13782-33-7)	X	X	X	Low water solubility / liable to sediment rapidly from suspension – ICE recommended
Palladium (II) di(4-oxopent-2-en-2-oate) (CAS 14024-61-4)	X	-	X	N/A
Tetraammineplatinum dinitrate (CAS 20634-12-2)	X	X	X	Supplied as solution, max. conc. 6.6%. Makeup of 20% solution from solid is not possible since solid is not isolated (explosivity); suggest to test 6.6% solution in BCOP test.
Platinum, 1,3-diethenyl-1,1,3,3-tetramethyldisiloxane complexes / Karstedt concentrate (CAS 68478-92-2)	(X)	(X)	(X)	Data sharing under discussion with Silicones sector. Suggest to put testing on hold. (Will decompose in aqueous solution. Consider waiver possibility. Predicted to be a marked irritant.)
Dihydrogen hexahydroxyplatinate (CAS 51850-20-5)	X			



Ecotox testing

Overall Testing Plan

- Palladium
  - Full testing for 2 substances: diamminedichloropalladium and dipotassium hexachloropalladate
  - Confirmatory testing for 2 substance: palladium di(4-oxopent-2-en-2-oate) and diammonium hexachloropalladate
- Platinum
  - Full testing for 2 substances: platinum dichloride and dihydrogen hexahydroxyplatinate
- Rhodium
  - Full testing for 1 substance: diammonium sodium hexakis(nitro-N)rhodate
- Ruthenium
  - Full testing for 1 substance: diammonium hexachlororuthenate
- Iridium
  - No testing required



Mammalian toxicity part

Genotoxicity

- Initial testing recommendations ready
- Expert assessment required on 2 issues
  - Impact of extreme pH on genotox study results
  - (Apparently) conflicting study results on Rh (III) substances
  - Currently consulting with independent genotoxicity expert (David Kirkland)

Sensitisation

- Initial testing recommendations ready
- Final scope will depend on outcome irritation tests

Irritation

- In-vitro skin irritation tests ongoing
- Scope of other tests will depend on outcome EPISKIN tests

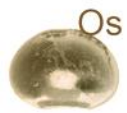
- Testing strategy
  - In-vitro skin irritation (EPISKIN) – if positive: stop
  - In-vitro eye irritation (ICE or BCOP) – if positive: stop
  - In-vivo eye irritation

Acute and repeated dose toxicity requirements

- Acute oral
  - Testing recommendations ready
- Acute inhalation/dermal
  - Depending on data from
    - Dustiness (is inhalation relevant at all?)
    - Bio-elution (is dermal/inhal. uptake possible at all?)
    - Irritation (no testing for irritants)
    - Practicalities (can stable testing atmosphere be created?)
- Repeated dose
  - Route of exposure/ dose selection, based on data from acute tests

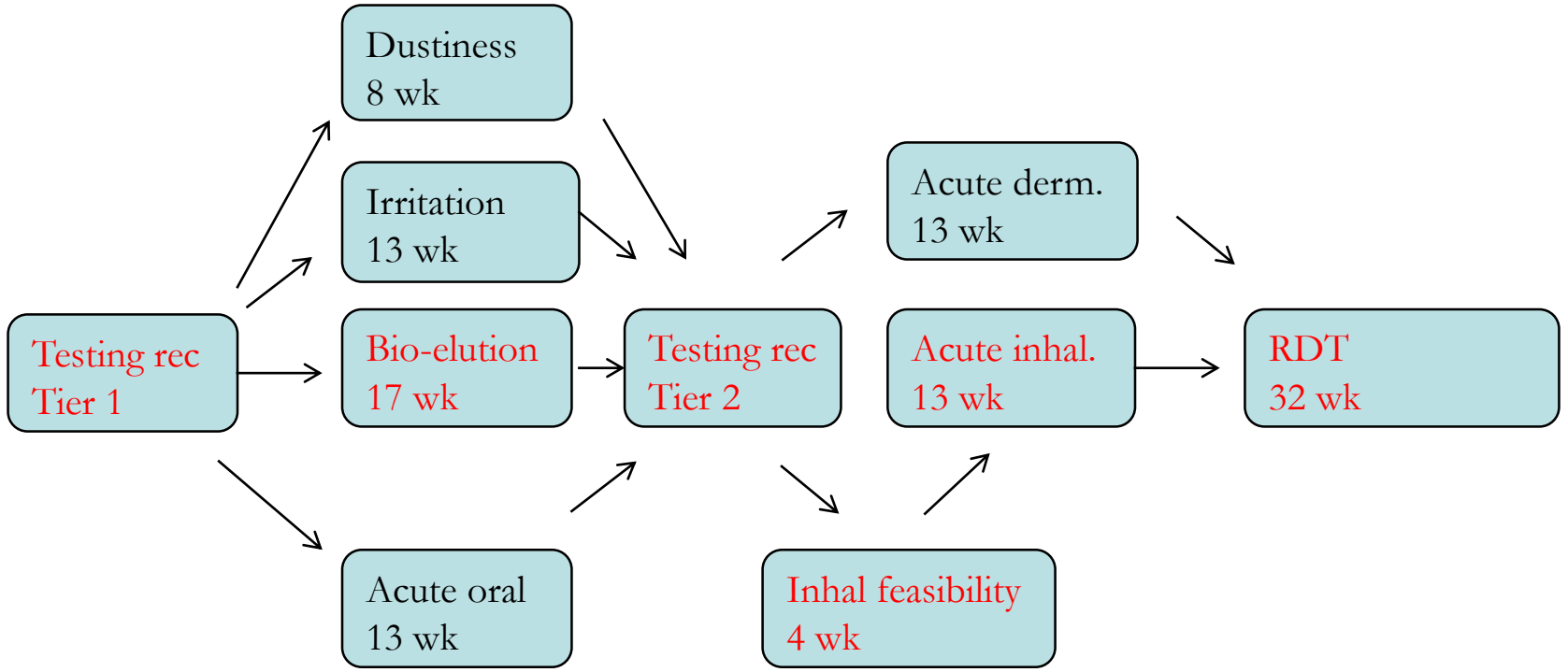
CLP

- Changes to PGM classification
  - Initial changes discussed at 6th April 2011 PGM WG call
  - Further changes possible as new test data are generated
  - 2nd ATP to CLP
    - To be implemented by Dec. 2012
    - New/ changed endpoints
      - Sensitization (skin/ resp.)
      - Environment classification:
        - » chronic effects
        - » new M factors
      - Pending: “removal from water column” (UWM)
      - (Effects on ozone layer)
- PMC preparing CLP update for Q1 2012



Next steps - Timeline

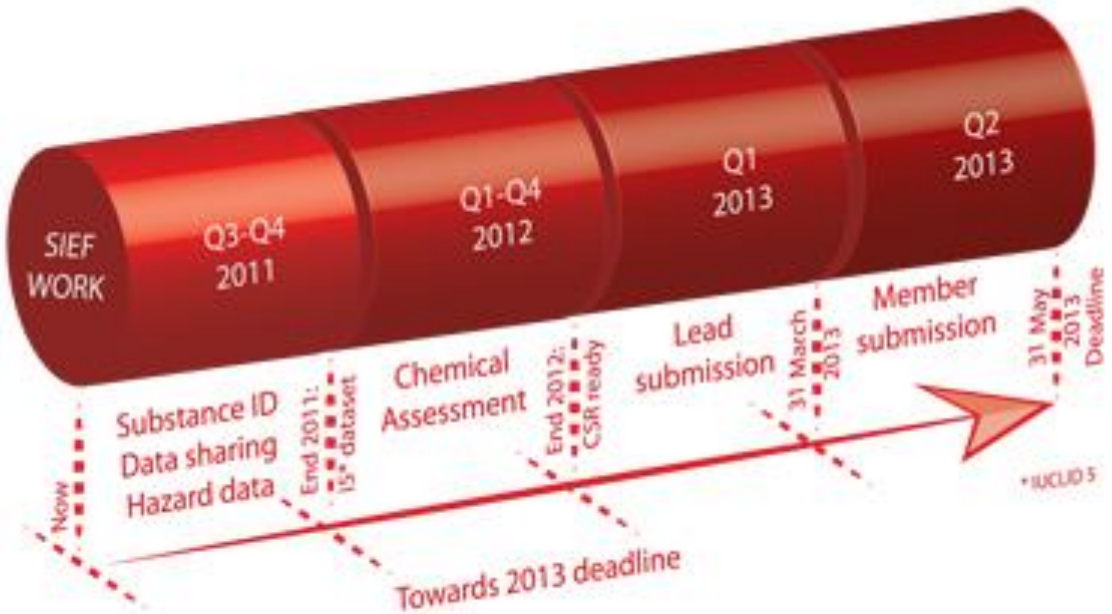
Timeline "normal track" testing - Likely too long for 2013 deadline



66 weeks needed, back-to-back, w/o slack

Nov 2011 → CSR

Timeline for successful submission
(from ECHA website)



Timeline fast-track - for 2013 deadline