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Preparation of Working Standards for Safeguards . Planning -part 1

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Preparation of Working Standards for Safeguards

Planning – part I

IAEA / JRC-IRMM / NBL / CETAMA



IAEA
International Atomic Energy Agency

In cooperation with:



European
Commission

Planning part I - Goal

- Inform on main steps in project planning
 - Resources, timing (health and safety issues)
 - Collaboration/resources
 - Material selection (availability of the material)
 - Material handling (interim transport and storage)
 - Material processing – sub division into respective units, packaging and storage
 - If relevant, transport/shipment

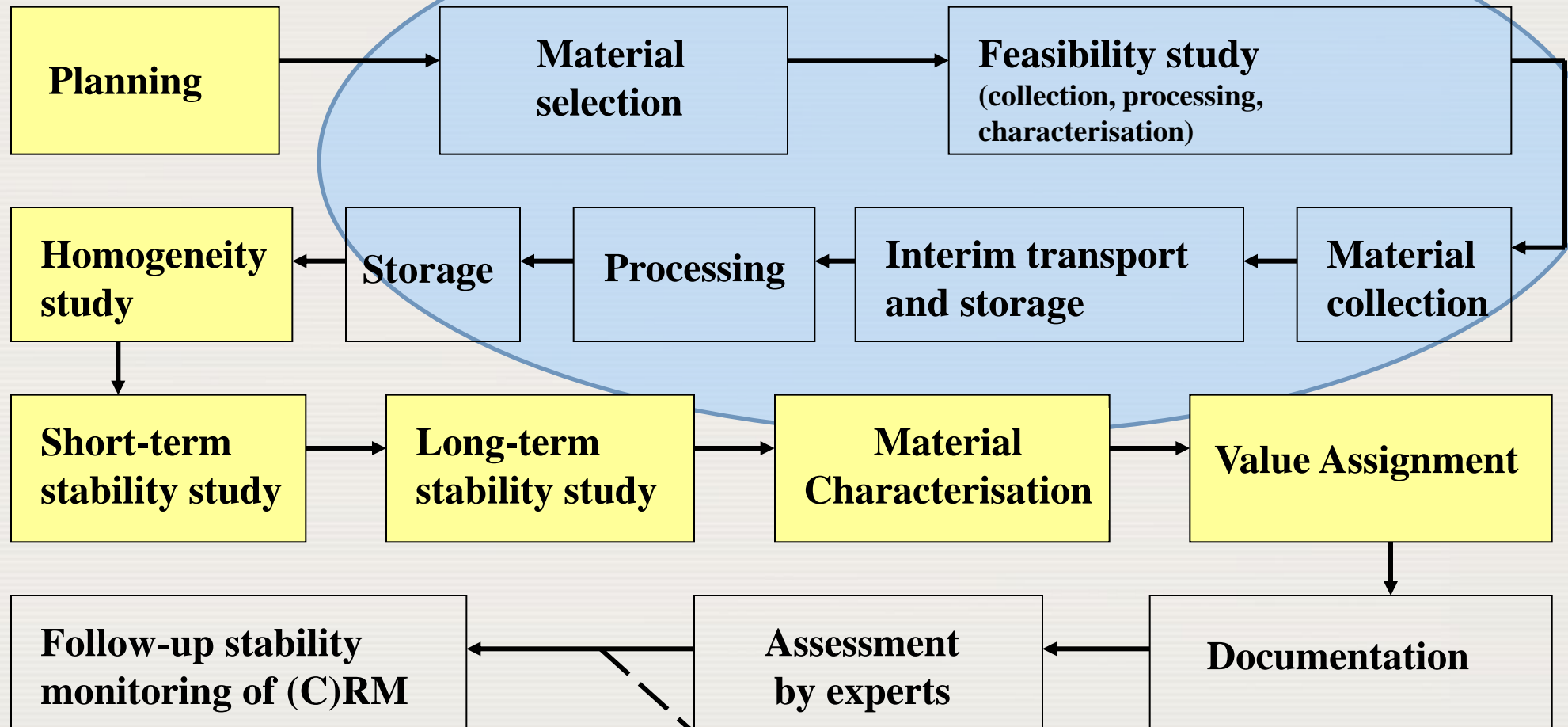


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(C)RM Production – part I

PROBLEM DEFINITION



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(C)RM Distribution (and Sales)

(C)RM planning

What (C)RM to be produced

High chemical purity

Matrix

Impurity

■ Material for instrument calibration

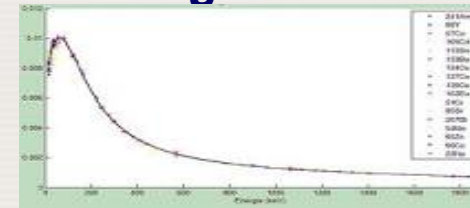
- High chemical purity RM
- Standard solutions
- Sources



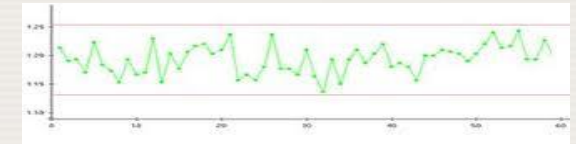
Sources étalons CERCA-LEA électrodéposées

2 main uses

Calibration curve setting on



Calibration QC



■ Material for method trueness and precision

- assessment
RM « matrix »



Matériau de référence de cartouche de charbon actif Nuclitec



Matériau de référence de végétaux CERCA-LEA

2 main uses

Initial characterisation of analytical method

QC material (global measurement process)

Isotopic spikes

traceability

collaborators



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European Commission



(C)RM project planning topics

- **(C)RM properties: target values (measurands?), uncertainty, traceability**
- Base material
 - procurement, restriction, transport, storage
- Processing (conversion into final material)
 - drying, cutting, mixing, filling, labelling etc.
- Availability of analytical resources
 - methods, labs etc
- Health & Safety issues
 - risk assessment, safety advisor



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Planning and PPF

proper and careful planning allows

- getting the desired result at the first attempt
- setting go/no go criteria for a (C)RM
- execution without budgetary problems
- responsibility of project responsible

Therefore: PPF is the basis of good (C)RM production



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Dilbert.com DilbertCartoonist@gmail.com



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CETAMA

PPF

Project summary:

Preparation of a Material for the determination of analyte y in matrix y with an uncertainty z . This material will be used by for Based on (*use of similar materials, educated guess, market study, etc.*) the demand/use is expected to be per year. Traceability to is required, because ...

Realistic estimates of time and costs



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PPF – Resources **draft**

- Human resources
- Financial resources
- Technical resources



PPF – Resources (health/safety)

- Impact on homogeneity/stability studies
- Other type of (C)RM available?
- Health
 - Chemical risk assessment
 - Radiological risk assessment
- Safety
 - Radiolysis risk assessment
 - Criticality

- Radioprotection
- Material handling
- Material conditioning



planning

traceability

collaborators



PPF – Resources (health/safety)

- Radiolysis risk assessment

- Context

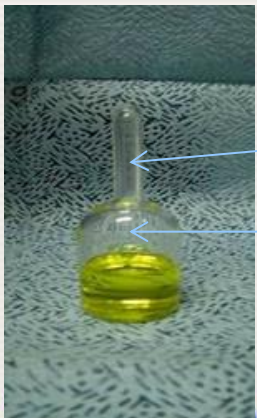
- Alpha radiolysis induced by radioactive decay of actinides in solution
- What about radiolysis risk management?

- Methodology

1. Calculation of H₂ and O₂ production due to water radiolysis
2. Determination of radiological stability : expiration date (fixed by [H₂] ≤ 4 %)

- $$dn_{H_2} = \frac{G_{H_2} * C * P_{therm} * A_{mass} * t}{2680}$$

- Conclusions



The pressure is an important parameter to consider

Important gas production, H₂, O₂, N₂ but also NO_x ...

Assessment of the overpressure limit value in vials

- H₂ and O₂ composition evolution in the gas volume

H₂/O₂ increase but limited energy



PPF – Timing

- Intended time schedule
- Amount of raw material needed
 - Depending on : Process, homogeneity/stability tests, volume or mass per samples
 - Needed/waited performances : composition and uncertainties
- Number of samples needed
 - Stability : shelf life of CRM
 - Possible use : life time of RM



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Examples of associated uncertainties



REFERENCE MATERIAL CERTIFICATE

PLUTONIUM METAL
« MP2 »

0.9990 ± 0.0004 kg Pu . kg⁻¹ metal

- The uncertainties assigned are expanded uncertainties with a coverage factor k=2.

isotope amount ratio(s)	
$n(^{238}\text{Pu})/n(^{239}\text{Pu})$	0.000 030 83(29)
$n(^{240}\text{Pu})/n(^{239}\text{Pu})$	0.022 432 4(51)
$n(^{241}\text{Pu})/n(^{239}\text{Pu})$	0.000 237 8(31)
$n(^{242}\text{Pu})/n(^{239}\text{Pu})$	0.000 075 70(78)

amount fraction (·100)		mass fraction (·100)	
$n(^{238}\text{Pu})/n(\text{Pu})$	0.003 015(29)	$m(^{238}\text{Pu})/m(\text{Pu})$	0.003 002(28)
$n(^{239}\text{Pu})/n(\text{Pu})$	97.773 05(58)	$m(^{239}\text{Pu})/m(\text{Pu})$	97.763 80(59)
$n(^{240}\text{Pu})/n(\text{Pu})$	2.193 28(49)	$m(^{240}\text{Pu})/m(\text{Pu})$	2.202 27(49)
$n(^{241}\text{Pu})/n(\text{Pu})$	0.023 25(30)	$m(^{241}\text{Pu})/m(\text{Pu})$	0.023 44(31)
$n(^{242}\text{Pu})/n(\text{Pu})$	0.007 402(76)	$m(^{242}\text{Pu})/m(\text{Pu})$	0.007 494(77)



CERTIFICAT DE MATERIAU DE REFERENCE

" MORILLE "

U₃O₈ : « MORILLE »
Teneurs exprimées en mg.kg⁻¹ d'U

ELEMENT	TENEUR CERTIFIEE	ELEMENT	TENEUR CERTIFIEE
Ag	10,4 ± 1,6	Li	5,0 ± 0,2
Al	99 ± 6	Mg	19,3 ± 1,5
B	3,8 ± 1,6	Mn	24,5 ± 0,5
Ba	9,6 ± 0,4	Mo	147 ± 5
Be	5,4 ± 0,6	Ni	147 ± 3
Bi	24,4 ± 1,9	Pb	101 ± 3
Ca	93 ± 8	Si	100 ± 8
Cd	4,9 ± 0,7	Sm	0,50 ± 0,12
Co	9,8 ± 2,0	Sn	18,5 ± 5,6
Cr	99 ± 2	Th *	6,2 ± 0,8
Cu	50,2 ± 1,0	Ti	49,2 ± 2,6
Dy	0,50 ± 0,06	V	48,7 ± 2,8
Eu	0,52 ± 0,03	W	100 ± 9
Fe	211,6 ± 6,5	Zn	98,6 ± 5,5
Gd	0,56 ± 0,06	Zr *	59,9 ± 4,1
In	9,4 ± 1,0		



European Commission



PPF – Collaboration - draft

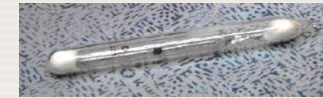
- Collaboration/resources



PPF – Material selection

- Material selection (availability of the material)
 - Raw material :
 - Sample directly collected on process flow
 - Synthetic material
 - Old or other CRM
 - Initial purity or composition to composition of candidate (C) RM
 - i.e. radioactive decay
 - Sampling of raw material
 - Homogenization

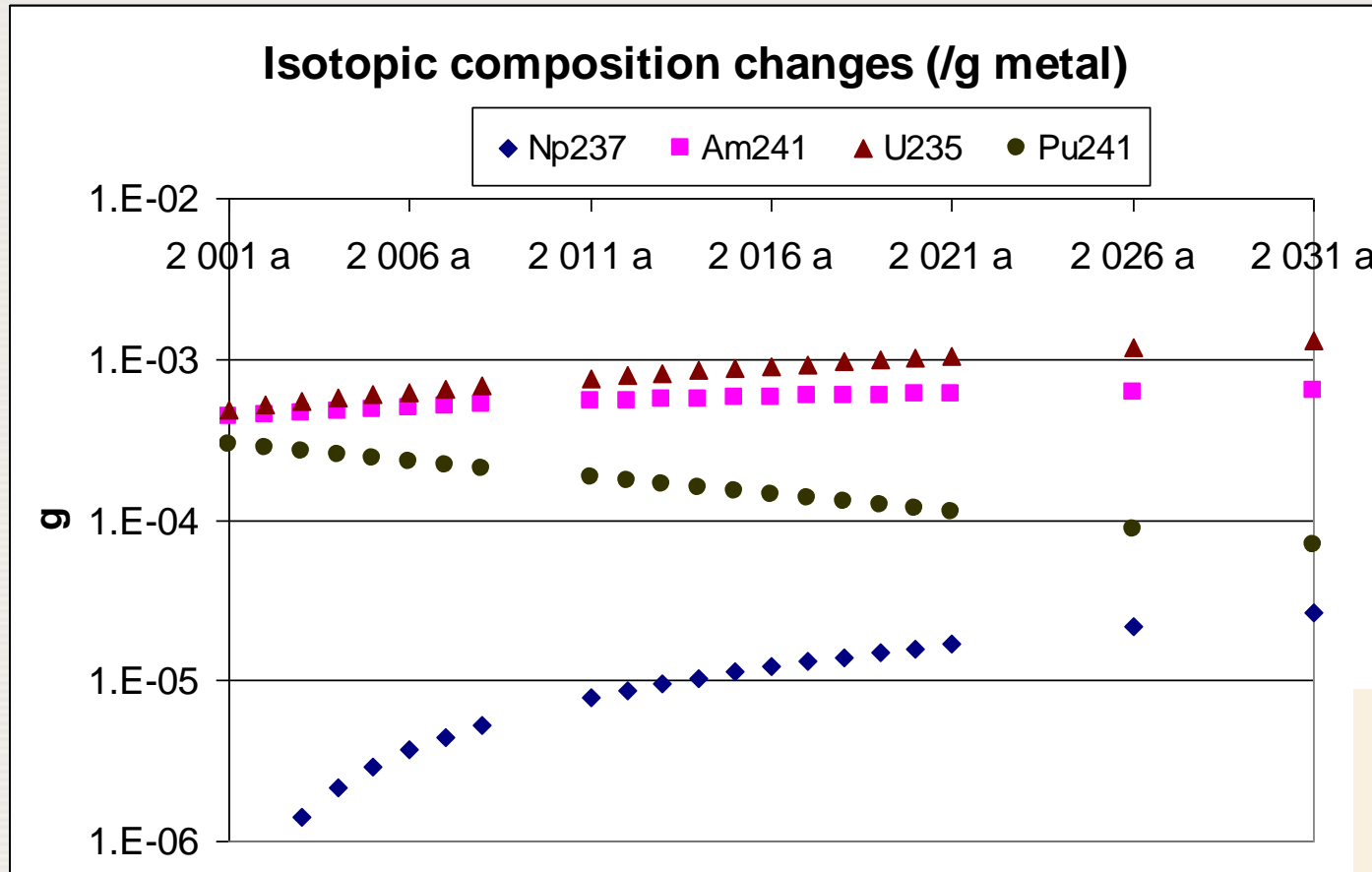
Physical change?
Dissolution, evaporation...



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PPF – Material selection

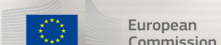


mass fraction (·100)	
$m(^{238}\text{Pu})/m(\text{Pu})$	0.003 227(40)
$m(^{239}\text{Pu})/m(\text{Pu})$	97.757 76(98)
$m(^{240}\text{Pu})/m(\text{Pu})$	2.202 62(95)
$m(^{241}\text{Pu})/m(\text{Pu})$	0.029 38(17)
$m(^{242}\text{Pu})/m(\text{Pu})$	0.007 015(70)

Half lives used
 ^{239}Pu : $2.411 \cdot 10^4$ y
 ^{240}Pu : $6.563 \cdot 10^3$ y
 ^{241}Pu : 14.29 y



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PPF – Material handling

- Supervised or nuclear controlled area
 - Glove box, fume hood,.....
- Transport of raw material and potential transport of RM
 - ONU material code
 - European agreement concerning international transport of dangerous material : ADR and conditioning type A or B
 - Global activity of the sample (Bq) and power
 - Comparison to A1 (isotope or mixed) and A2 (special form) values

- Interim storage



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Example: ADR for Pu

Radionucléide (numéro atomique)	A ₁	A ₂	Limite d'activité massique pour les matières exemptées	Limite d'activité pour un envoi exempté
	(TBq)	(TBq)	(Bq/g)	(Bq)
Plutonium (94)				
Pu-236	3×10^1	3×10^{-3}	1×10^1	1×10^4
Pu-237	2×10^1	2×10^1	1×10^3	1×10^7
Pu-238	1×10^1	1×10^{-3}	1×10^0	1×10^4
Pu-239	1×10^1	1×10^{-3}	1×10^0	1×10^4
Pu-240	1×10^1	1×10^{-3}	1×10^0	1×10^3
Pu-241 (a)	4×10^1	6×10^{-2}	1×10^2	1×10^5
Pu-242	1×10^1	1×10^{-3}	1×10^0	1×10^4
Pu-244 (a)	4×10^{-1}	1×10^{-3}	1×10^0	1×10^4

Example: ADR for U

Radionucléide (numéro atomique)	A ₁	A ₂	Limite d'activité massique pour les matières exemptées	Limite d'activité pour un envoi exempté
	(TBq)	(TBq)	(Bq/g)	(Bq)
Uranium (92)				
U-230 (absorption pulmonaire rapide) (a) (d)	4×10^1	1×10^{-1}	1×10^1 (b)	1×10^5 (b)
U-230 (absorption pulmonaire moyenne) (a) (e)	4×10^1	4×10^{-3}	1×10^1	1×10^4
U-230 (absorption pulmonaire lente) (a) (f)	3×10^1	3×10^{-3}	1×10^1	1×10^4
U-232 (absorption pulmonaire rapide) (d)	4×10^1	1×10^{-2}	1×10^0 (b)	1×10^3 (b)
U-232 (absorption pulmonaire moyenne) (e)	4×10^1	7×10^{-3}	1×10^1	1×10^4
U-232 (absorption pulmonaire lente) (f)	1×10^1	1×10^{-3}	1×10^1	1×10^4
U-233 (absorption pulmonaire rapide) (d)	4×10^1	9×10^{-2}	1×10^1	1×10^4
U-233 (absorption pulmonaire moyenne) (e)	4×10^1	2×10^{-2}	1×10^2	1×10^5
U-233 (absorption pulmonaire lente) (f)	4×10^1	6×10^{-3}	1×10^1	1×10^5
U-234 (absorption pulmonaire rapide) (d)	4×10^1	9×10^{-2}	1×10^1	1×10^4
U-234 (absorption pulmonaire moyenne) (e)	4×10^1	2×10^{-2}	1×10^2	1×10^5
U-234 (absorption pulmonaire lente) (f)	4×10^1	6×10^{-3}	1×10^1	1×10^5
U-235 (tous types d'absorption pulmonaire) (a), (d), (e), (f)	Illimitée	Illimitée	1×10^1 (b)	1×10^4 (b)
U-236 (absorption pulmonaire rapide) (d)	Illimitée	Illimitée	1×10^1	1×10^4
U-236 (absorption pulmonaire moyenne) (e)	4×10^1	2×10^{-2}	1×10^2	1×10^5
U-236 (absorption pulmonaire lente) (f)	4×10^1	6×10^{-3}	1×10^1	1×10^4
U-238 (tous types d'absorption pulmonaire) (d), (e), (f)	Illimitée	Illimitée	1×10^1 (b)	1×10^4 (b)
U (naturel)	Illimitée	Illimitée	1×10^0 (b)	1×10^3 (b)
U (enrichi à 20% ou moins) (g)	Illimitée	Illimitée	1×10^0	1×10^3
U (appauvri)	Illimitée	Illimitée	1×10^0	1×10^3

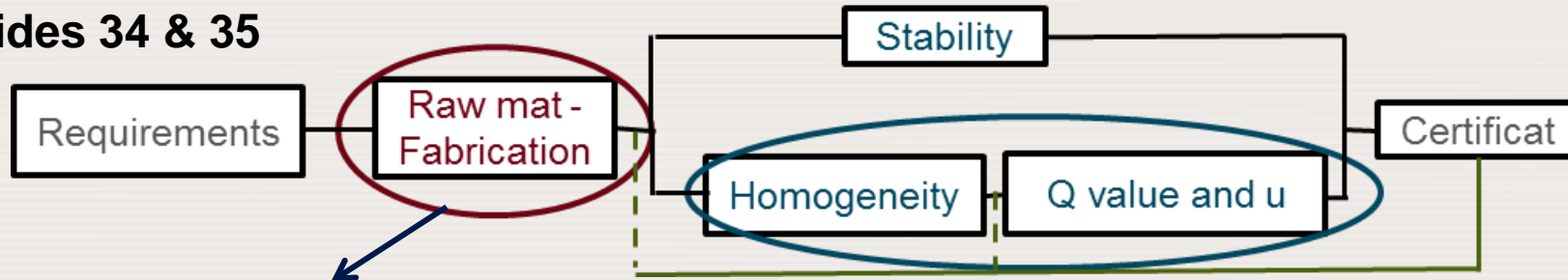
PPF – Material processing - **draft**

- Material processing – sub division into respective units, packaging and storage (transport)
- Containment (ampoules, vials etc...)



PPF – Material processing

ISO guides 34 & 35



Support laboratory

Raw material



Standard in solution:

- Chemical purification
- High yield separation process
- Conditioning : sealed vials
- Radiolysis risk management

Solid standard:

- Homogeneity (powder < pellet...)
- Grinding & homogenization

planning

traceability

collaborators



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PPF – Feasibility Study

- Facultative step: prior knowledge available ?
 - Optimal / validated/ adapted method o preparation
 - Short and/or long term stability knowledge
 - Adaptation to analytical needs
- Or preselection of expert laboratory for final certification
 - A batch of a small number of samples needed: to be defined
- Duration of feasibility study step



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PPF – Project Planning Form – update

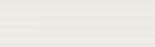

with nuclear ppf example

Microsoft Excel - 20081111_draft_PPF_IRMM-307.xls

File Edit View Insert Format Tools Data Window Help

Arial 10

C39 Documented in raw material report.

1	 JRC EUROPEAN COMMISSION	Form RM F0007 Revision 11, June 2008	
3	Project Planning Form		
5	Project title		
6	Bimodal colloidal silica nanoparticle quality control material		
7	For ERM-branded materials: ERM submission code:		
9	Project Management		
10	Action Number	11102	
11	Action Leader	T. Linsinger	
12	Project Responsible	V. Kestens	
13	Deputy	G. Roebben	
14	CRM type	IRMM if "other", specify:	
15	CRM code	307	
16	Resources needed	61 FTE days; 33 KEUR	
17	Intended Time schedule	Procurement of Raw materials: already done	
18		Processing: 31/05/2009	
19		Registration of the samples: 15/06/2009	
20		Selection of laboratories: 30/11/2008	
21		Homogeneity testing: 30/06/2009	
22		Short-term stability testing: 30/06/2009	
23		Long-term stability testing: 30/11/2009	
24		Post-certification monitoring until:	
25		Characterisation:	
26		The material shall be released by:	
29	Project Description		
30	Short summary of the project (2-3 sentences)	Preparation of a bimodal colloidal silica nanoparticle quality control material, dispersed in an aqueous-based solution	
31	Intended use (calibration, quality assurance etc.)	Development of methods and instrumentation for particle size analysis, interlaboratory and between-method comparisons	
32	Matrix	Aqueous based solution	
33	Target parameters, their levels and uncertainties	Mode diameters and intensity - volume - number fractions by DCS	
34		(calculated expanded (k = 2) uncertainty based on the inputs in the PPF: 10,95 %	
35	Intended identity of the measurand	method defined	
36	Intended traceability statement	Traceable to ISO 13318 (disc sedimentation) and SI	
37	Key checks		
38	Incoming material	Outcome of the checks and place of documentation	Documented in raw material report
39	Processing	Particle size analyses and zeta potential measurements	
40		Outcome of the checks and place of documentation	Documented in processing report
41			

Summary | IPR and base materials | Inventory and lab selection | Processing | Homogeneity

planning

traceability

collaborators

Outline – planning part I

- Exercise
 - Prepare a project planning form
 - use provided PPF (excel) template