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Preparation of Working Standards for Safeguards . Planning -part 1

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To cite this version:

C. Maillard, D. Roudil, P. Moisy. Preparation of Working Standards for Safeguards . Planning -part 1. TM on the preparation of working standards for safeguards, Jun 2016, Vienne, Austria. hal-02441968

HAL Id: hal-02441968

https://cea.hal.science/hal-02441968v1

Submitted on 16 Jan 2020

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Preparation of Working Standards for Safeguards

Planning – part I

IAEA / JRC-IRMM / NBL / CETAMA

IAEA
International Atomic Energy Agency

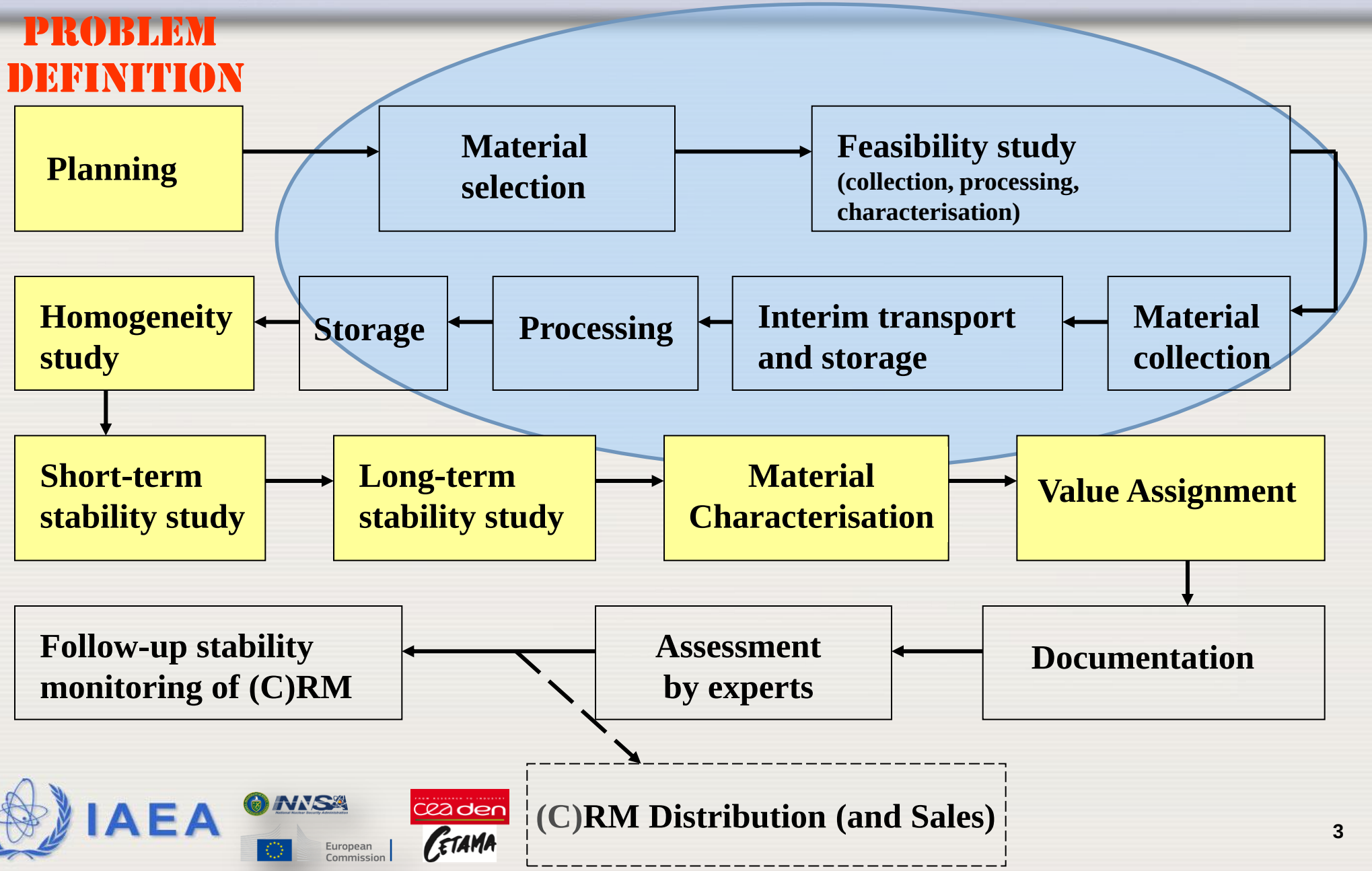
In cooperation with:

cea den
CETAMA
NNSA National Nuclear Security Administration
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

(C)RM Production – part I



(C)RM planning

What (C)RM to be produced

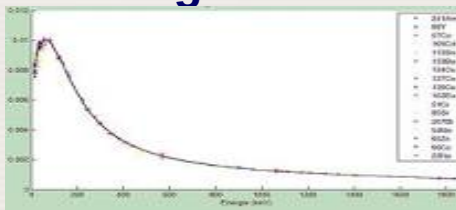
High chemical purity
Matrix
Impurity

- **Material for instrument calibration**
  - High chemical purity RM
  - Standard solutions
  - Sources

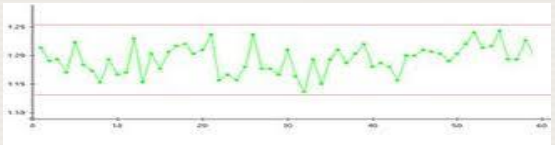
Sources étalons CERCA-LEA électrodéposées

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

Intial characterisation of analytical method

QC material (global measurement process)

- Isotopic spikes

traceability

collaborators

(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

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

<u>Therefore:</u> PPF is the basis of good (C)RM production

I NEED A BUDGET ESTIMATE FOR MY PROJECT, BUT I DON'T HAVE A SCOPE OR A DESIGN FOR IT YET.
Dilbert.com DilbertCartoonist@gmail.com
OKAY, MY ESTIMATE IS $3,583,729.
12-1-09 ©2009 Scott Adams, Inc./Dist. by UFS, Inc.
YOU DON'T KNOW ANYTHING ABOUT MY PROJECT.
THAT MAKES TWO OF US.

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

PPF – Resources draft

- Human resources
- Financial resources
- Technical ressources

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 radiolysi
2. Determination of radiological stability : expiration date (fixed by $[H_2] \leq 4$ %)

$$dn_{H2} = \frac{G_{H2} * C * P_{therm} * A_{mass} * t}{2680}$$

➢ Conclusions

The pressure is an important parameter to consider

Important gas production, H_2, O_2, N_2 but also NOx …

Assessment of the overpressure limit value in vials

- H_2 and O_2 composition evolution in the gas volume

H_2/O_2 increase but limited energy

planning — traceability — collaborators

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

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

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

CERTIFICAT DE MATERIAU DE REFERENCE

" MORILLE "

U_3O_8 : « 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

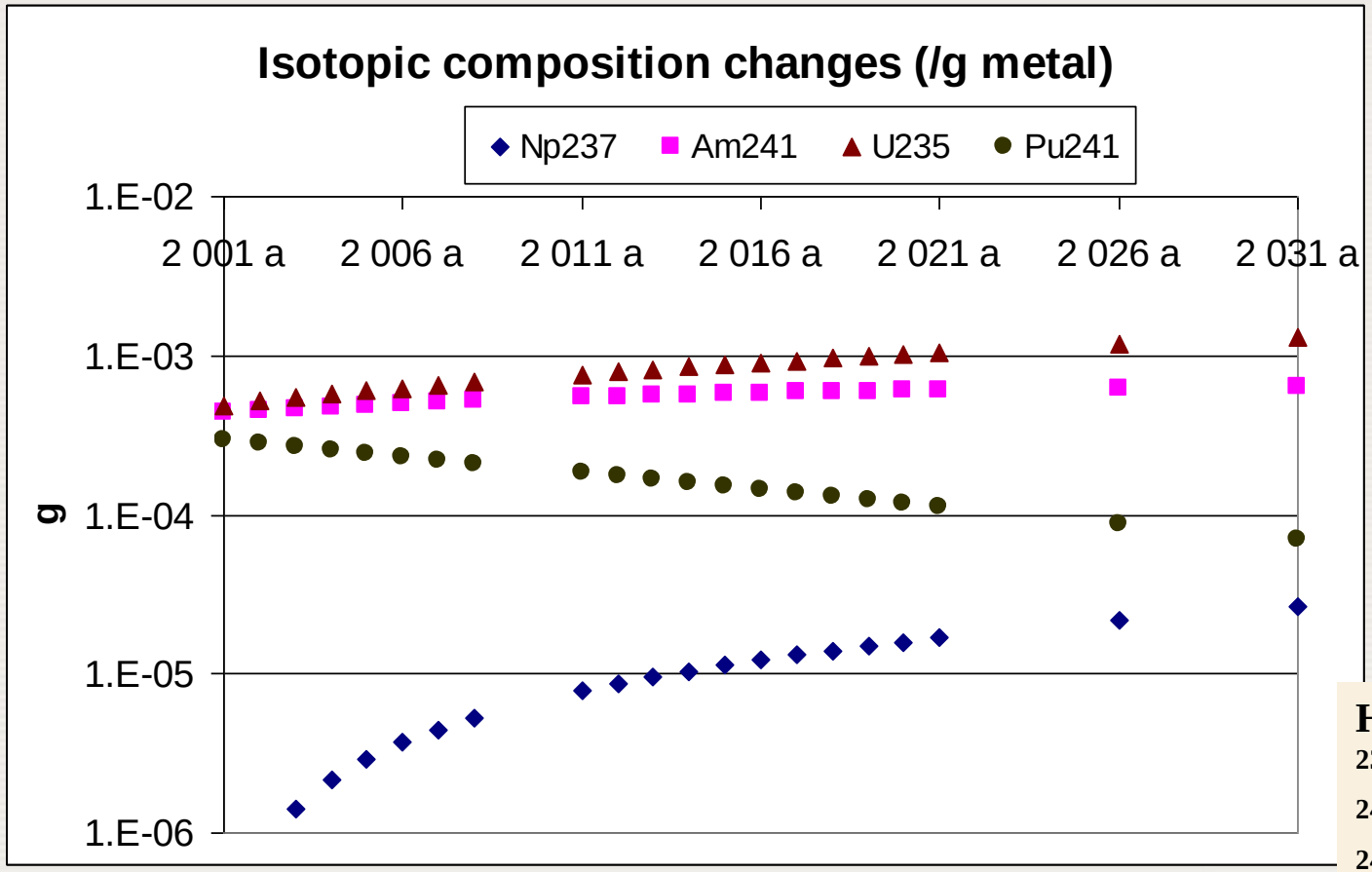
- Material selection (availability of the material)
  - Raw material :
    - Sample directly collected on process flow
    - Synthetic material
    - Old or other CRM

    Physical change?
    Dissolution, evaporation…

  - Initial purity or composition to composition of candidate (C) RM
    - i.e. radioactive decay
  - Sampling of raw material
  - Homogenization

planning — traceability — collaborators

PPF – Material selection



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

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

planning — traceability — collaborators

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

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}

IAEA NNSA European Commission cea den CETAMA

Example: ADR for U

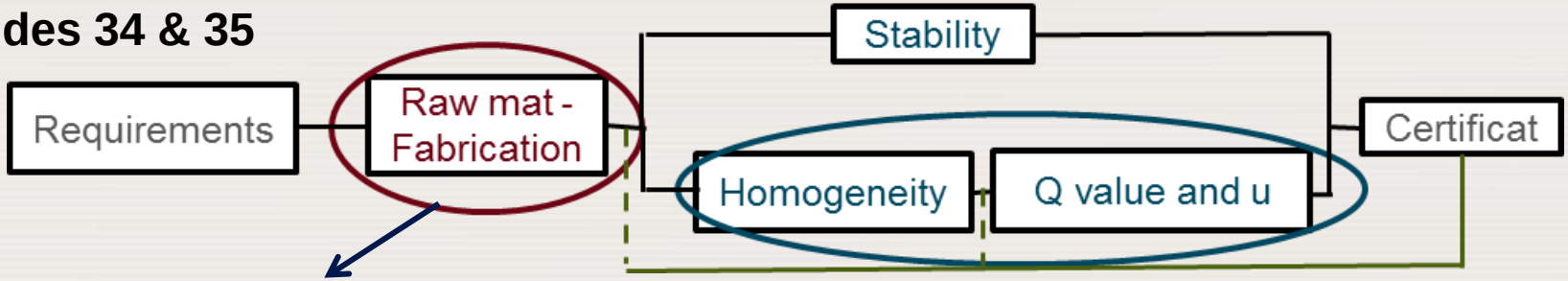
Radionucléide (numéro atomique)	A_1	A_2	Limite d'activité massique pour les matières exemptées	Limite d'activité pour un envoi exempté
	(TBq)	(TBq)	(Bq/g)	(Bq)
Uranium (92)				
U-230 (absorption pulmonaire rapide) (a) (d)	4×10^1	1×10^{-1}	1×10^1 (b)	1×10^5 (b)
U-230 (absorption pulmonaire moyenne) (a) (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

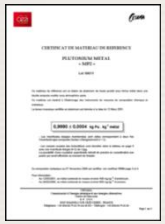
PPF – Material processing - draft

- 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**

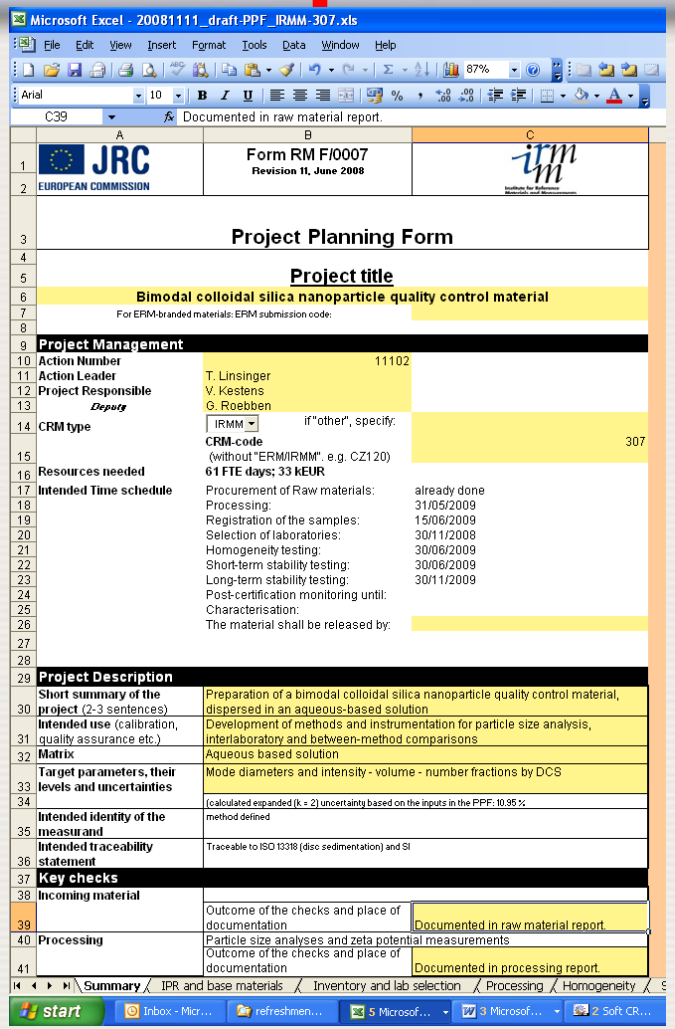
PPF – Feasibility Study

- 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

PPF – Project Planning Form – update with nuclear ppf example



Microsoft Excel - 20081111_draft-PPF_IRMM-307.xls

C39: Documented in raw material report.

	A	B	C
1–2	JRC EUROPEAN COMMISSION	Form RM F/0007 Revision 11, June 2008	irmm
3	**Project Planning Form**		
4–5	**Project title**		
6	**Bimodal colloidal silica nanoparticle quality control material**		
7–8	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** (without "ERM/IRMM". e.g. CZ120)	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:	
27–28			
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**		
39		Outcome of the checks and place of documentation	Documented in raw material report.
40	**Processing**	Particle size analyses and zeta potential measurements	
41		Outcome of the checks and place of documentation	Documented in processing report.

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