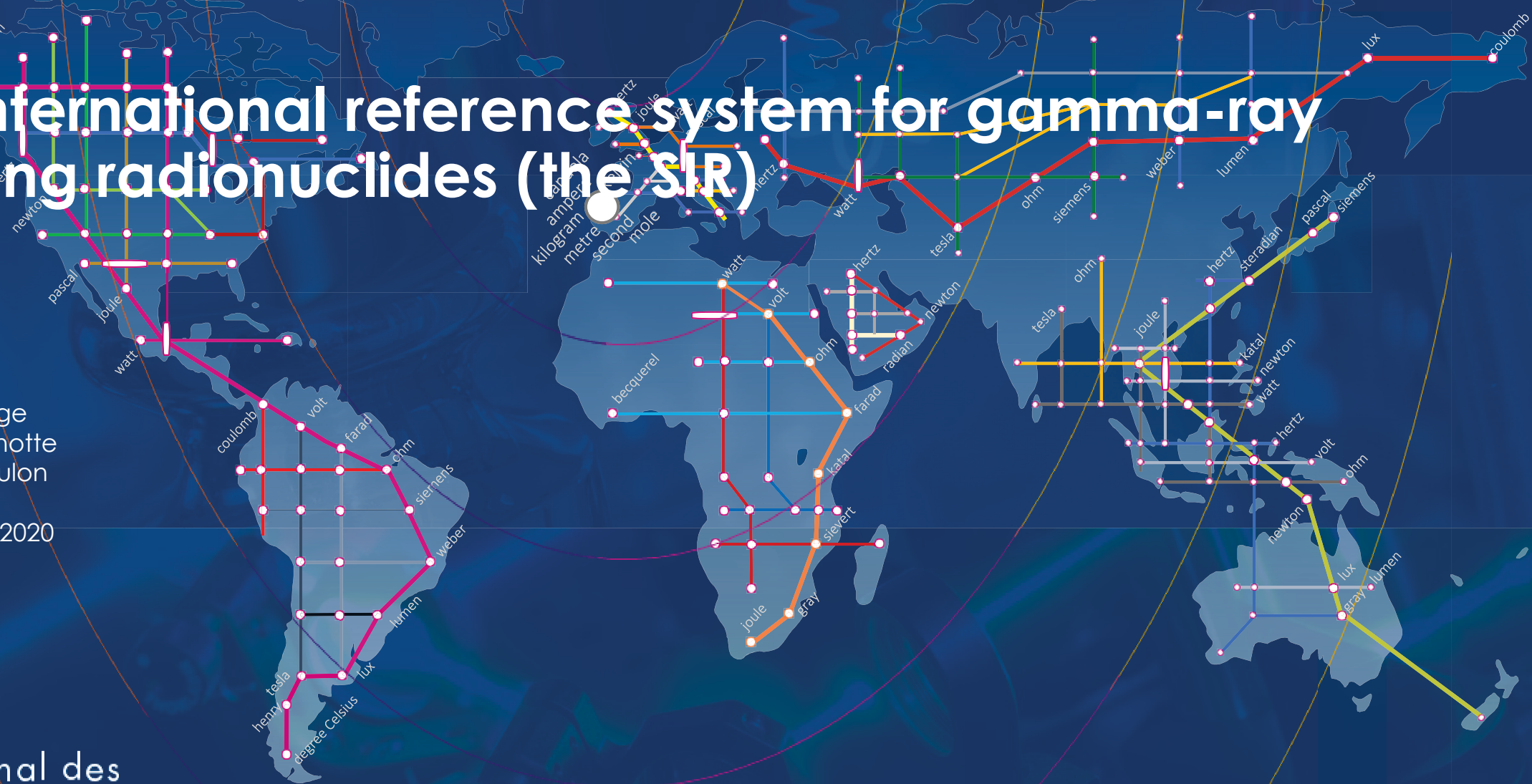


# The international reference system for gamma-ray emitting radionuclides (the SIR)

Steven Judge  
Carine Michotte  
Romain Coulon

December 2020

**B**ureau  
International des  
**P**oids et  
**M**esures



# Applications of the SIR

# Comparisons in radionuclide metrology

How do you support a CMC claim?

CIPM-MRA-D-05

...the range and uncertainty of the CMCs submitted must be consistent with information from some or all of the following sources:

1. Results of key and supplementary comparisons
2. Documented results of past CC, RMO or other comparisons (including bilateral)
3. Knowledge of technical activities by other NMIs, including publications
4. On-site peer-assessment reports
5. Active participation in RMO projects
6. Other available knowledge and experience

<https://www.bipm.org/utils/common/documents/CIPM-MRA/CIPM-MRA-D-04.pdf>

How do you support a CMC claim?

How do you show that the CMC is still valid?

CIPM-MRA-G-02

The comprehensive periodic review of the quality system includes examination of evidence for the **continued validity and vitality** of published CMCs.

In addition to the 5-year review of the supporting QMS, CMCs published in the KCDB undergo **continual monitoring to ensure their validity.**

<https://www.bipm.org/utis/common/documents/CIPM-MRA/CIPM-MRA-G-02.pdf>

How do you support a CMC claim?

How do you show that the CMC is still valid?

How can you comply with ISO17025?

## ISO17025:2017

**6.2.3** The laboratory shall ensure that the personnel have the competence to perform laboratory activities for which they are responsible and to evaluate the significance of deviations.

**7.2.1.5** The laboratory shall verify that it can properly perform methods before introducing them by ensuring that it can achieve the required performance. Records of the verification shall be retained.

**7.2.2.1** The laboratory shall validate non-standard methods, laboratory-developed methods and standard methods used outside their intended scope or otherwise modified.

**7.2.2.2** When changes are made to a validated method, the influence of such changes shall be determined and where they are found to affect the original validation, a new method validation shall be performed.

**7.7.2** The laboratory shall monitor its performance by comparison with results of other laboratories, where available and appropriate. This monitoring shall be planned and reviewed...

How do you support a CMC claim?

How do you show that the CMC is still valid?

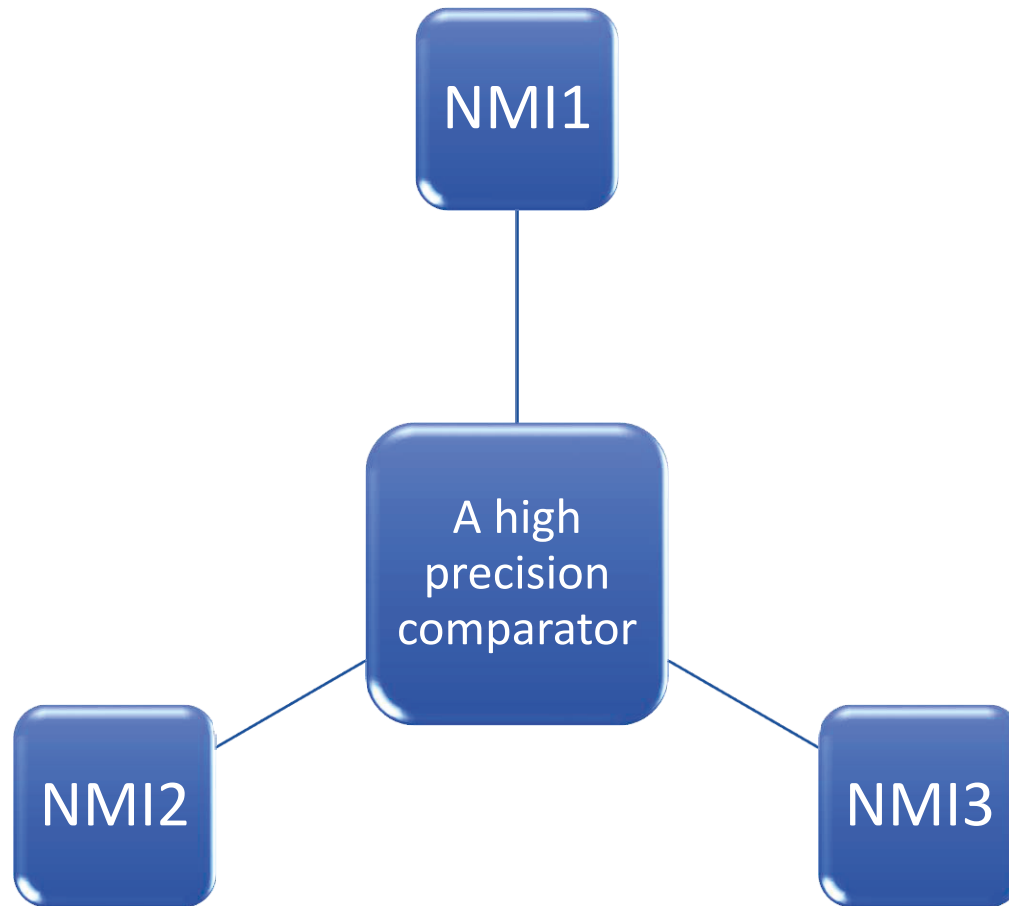
How can you comply with ISO17025?

How can you check a new primary standardization technique?

The science!

- To test new techniques

# The international reference system for gamma-ray emitters (SIR)



## Advantages

- You can compare your standards to those at other institutes and also to your results from the past
- Available when you need it
- Free of charge to NMIs and DIs
- Easy to use
- Results can be used to support CMCs for other radionuclides (for discussion at another webinar)

How the SIR works



The SIR

The International Reference System for gamma-emitting radionuclides

S. Judge, C. Michotte,

R. Coulon

CCRI webinar

Dec. 2020



**B**ureau  
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Poids et  
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# The SIR in a nutshell

BIPM.RI(II)-K1

- Highly stable comparator of activity measurements **since 1976**
- Total of ~ 800 SIR results for **72 different radionuclides**
- Measurements carried out relatively to  $^{226}\text{Ra}$  source
- SIR Result = **Equivalent activity** / kBq

$$A_{e,i} = \frac{A_i(t_m) C_{\text{imp}}}{I_i(t_m) / I_{\text{Ra N}^\circ 5}(t_0)}$$

~ inverse of a calibration factor pA/MBq

IG 11 – N<sub>2</sub> – 2 MPa



# The SIR impurity correction: how is it estimated?

---

$$C_{imp} = 1 + \sum_k \frac{A_k(t_m)}{A(t_m)} \frac{A_e}{A_{e,k}}$$

from SIR measurement

$\gamma$ -spectrometry at NMI

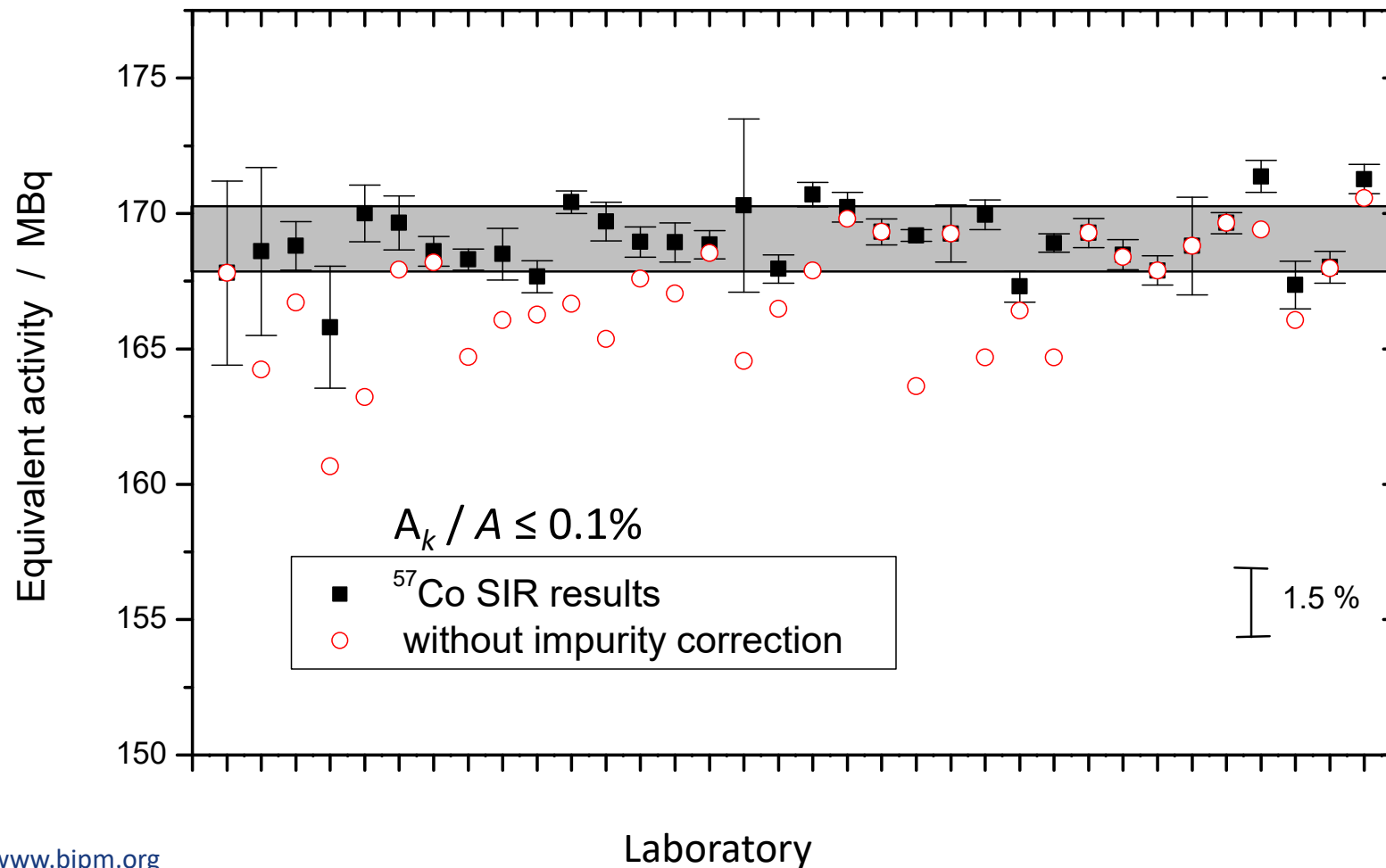
$A_{e,k}$ : Equivalent activity for impurity  $k$

obtained

- from SIR measurement
- or calculated (SIR efficiency curve + nuclear data)

# The SIR impurity correction: how is it estimated?

For low-energy  $\gamma$ -emitter:  $C_{\text{imp}}$  reaches several (tens of) percents  
precise  $\gamma$ -spectrometry is needed



# The SIR range

- Bg current: 30 fA Factor 50  $u(\text{Bg})$  is supposed negligible
- SIR measurement range: from 1.5 pA to 500 pA, which corresponds to a different activity range for each RN

$$A_{e,i} = \frac{A_i(t_m) C_{\text{imp}}}{I_i(t_m) / I_{\text{Ra N}^\circ 5}(t_0)}$$

- Five  $^{226}\text{Ra}$  sealed sources to cover the whole range
- Current ratios  $F_j$  between the 5 sources is well-known

$$F_j = I_{\text{Ra N}^\circ 5} / I_{\text{Ra N}^\circ j}$$

See SIR-T-20 pro

New version on the BIPM website

Radionuclide	Minimum /kBq	/MBq
Cr-51	2 600	50
Mn-54	100	29
Co-57	900	150
Co-60	40	11
Ge-68	90	20
Y-88	40	10
Cd-109	43 000	500
Cs-137	150	41
Ce-139	700	200
Eu-152	80	22
Am-241	11 000	50

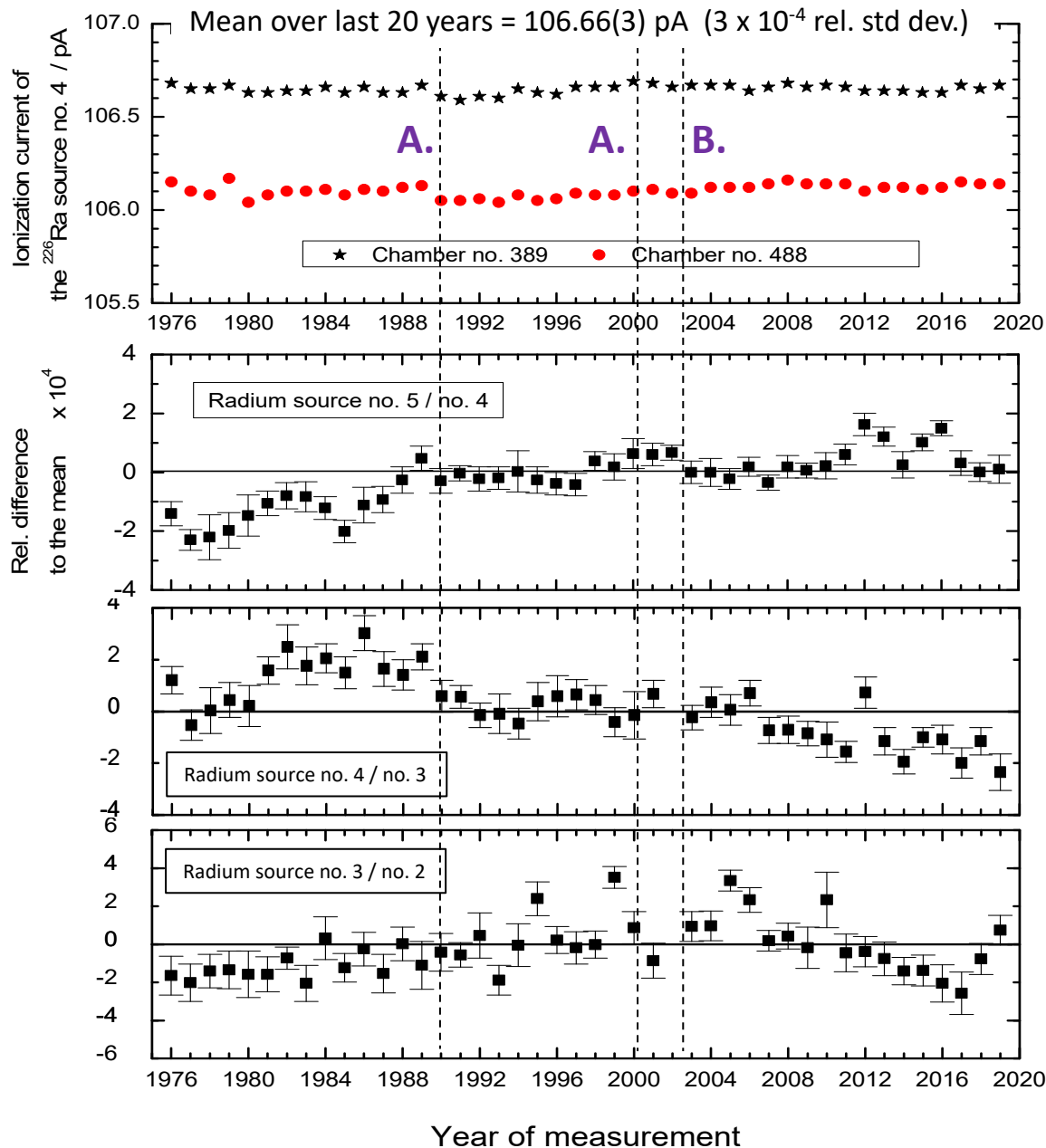
# The SIR

## long-term stability

- $^{226}\text{Ra}$  current is monitored : global stability check of SIR IC, electronics and  $^{226}\text{Ra}$  sources
- Current ratios between the  $^{226}\text{Ra}$  sources are monitored and used in calculation of  $A_e$

- A. Move of SIR to another room
- B. Complete renew of electrometer, electronics, DAQ
- C. 2020 update of DAQ

Fluctuations observed within  $\pm 3 \times 10^{-4}$



# The SIR uncertainty budget

**Combined relative uncertainty**  
**Typ. 0.03 – 0.15 %**

Uncertainty component	Relative uncertainty / $10^{-4}$	Type A/B evaluation method	Comment
Current measurement	2 - 10	A	Standard deviation of the mean
Ratio of $^{226}\text{Ra}$ sources $F_j$	0 - 7	B	Depends on $^{226}\text{Ra}$ source used for the measurement
Decay (RN)	Typ. < 5	B	Can reach several $10^{-3}$
Decay ( $^{226}\text{Ra}$ )	0.8	B	Today
Decay (impurity)	Typ. < 1	B	
Impurity correction	Typ. < 5	A/B	Propagation of uncertainty provided by NMI Can reach several $10^{-3}$
Ampoule wall thickness (liquids)	2 - 10	B	Interpolation/extrapolation of Rytz's study using $^{241}\text{Am}$ and $^{60}\text{Co}$
Background current	-		Negligible (within the activity range of the SIR)
Solution volume	-		Height of ampoule holder adjusted to compensate

# The SIR:

## case of complex decay-scheme not at equilibrium

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- ◆  $A_e$  is always calculated using the activity of the parent nuclide
- ◆ Equilibrium with daughter nuclides is assumed
- ◆ In some case, equilibrium is never reached  
( $A_{\text{daughter}}/A_{\text{parent}}$  is not constant), so  $A_e$  depends on time
- ◆ Specific procedure is being developed to enable comparison  
(J. Keightley, NPL)
- ◆ Examples: Th-227, Pa-231, Zr-95



## The SIR is a reliable tool for

- Long-term comparison of primary/secondary activity standards
- To test/validate a new method, new nuclide...  
« Pilot comparison »: no degrees of equivalence calculated

+ extension to short-lived radionuclides (SIRTI)  
+ extension to beta emitters (ESIR)

# The SIR and the MRA

# The SIR and the MRA

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- ◆ Degrees of Equivalence for lab  $i$   $D_i = x_i - x_{\text{ref}}$ ;  $U_i$  ( $k = 2$ )
- ◆ The CCRI(II) decided that  $D_i$  remains valid for 20 years
- ◆ The CCRI(II) decided that  $x_{\text{ref}} = \text{KCRV} = \text{power moderated weighted mean}$   
(S. Pommé *et al*, *Metrologia* 2015)

$$x_{\text{ref}} = \sum_{i=1}^N w_i x_i \frac{1}{u^2(x_{\text{ref}})} = \sum_{i=1}^N \left[ \left( \sqrt{u_i^2 + s^2} \right)^\alpha S^{2-\alpha} \right]^{-1},$$

$$w_i = u^2(x_{\text{ref}}) \left[ \left( \sqrt{u_i^2 + s^2} \right)^\alpha S^{2-\alpha} \right]^{-1} \quad S = \text{'characteristic uncertainty per datum'}$$

- $s = \text{'dark uncertainty'}$  such that  $\chi^2 = 1$
- Power  $\alpha$ : between 0 (arithmetic mean) and 2 (weighted mean)
- CCRI(II) decided that  $\alpha = 2 - 3/N$

# Which data for the KCRV?

---

## SIR data only

- No data from linked comparisons (except if ampoules also measured in the SIR)

## No limit in time

- old good results remain valid  
(because the SIR demonstrates long-term stability to a few  $10^{-4}$ )  
=> more entries in KCRV => more robust


## Most recent primary result for each participant

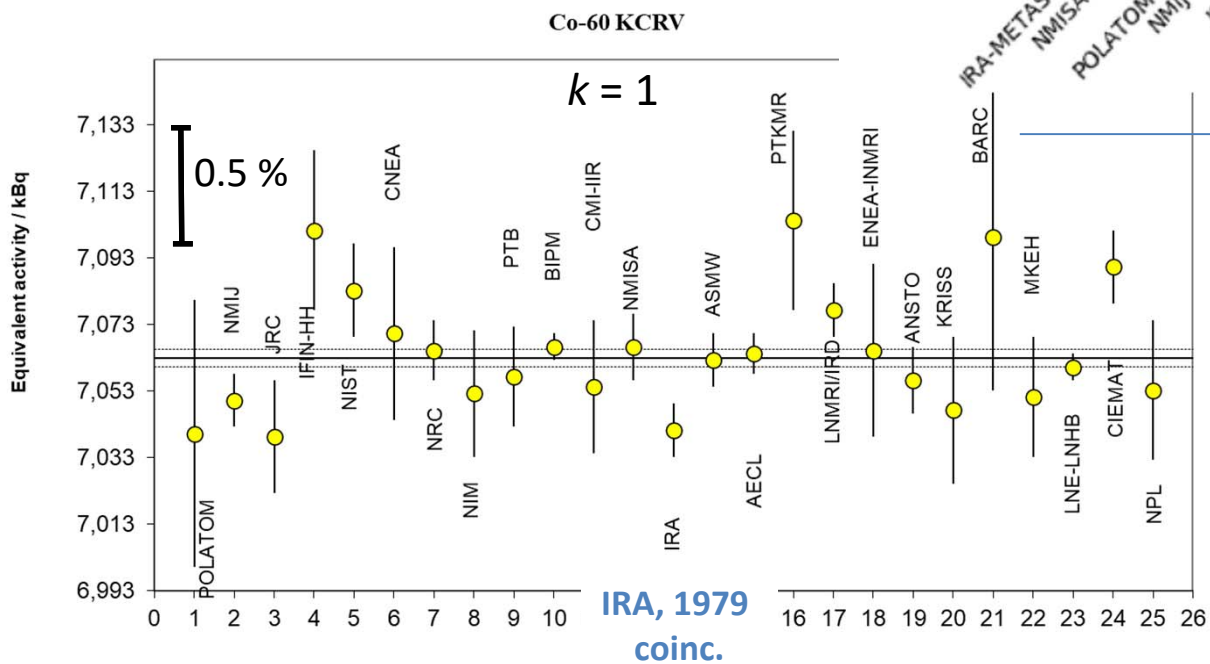
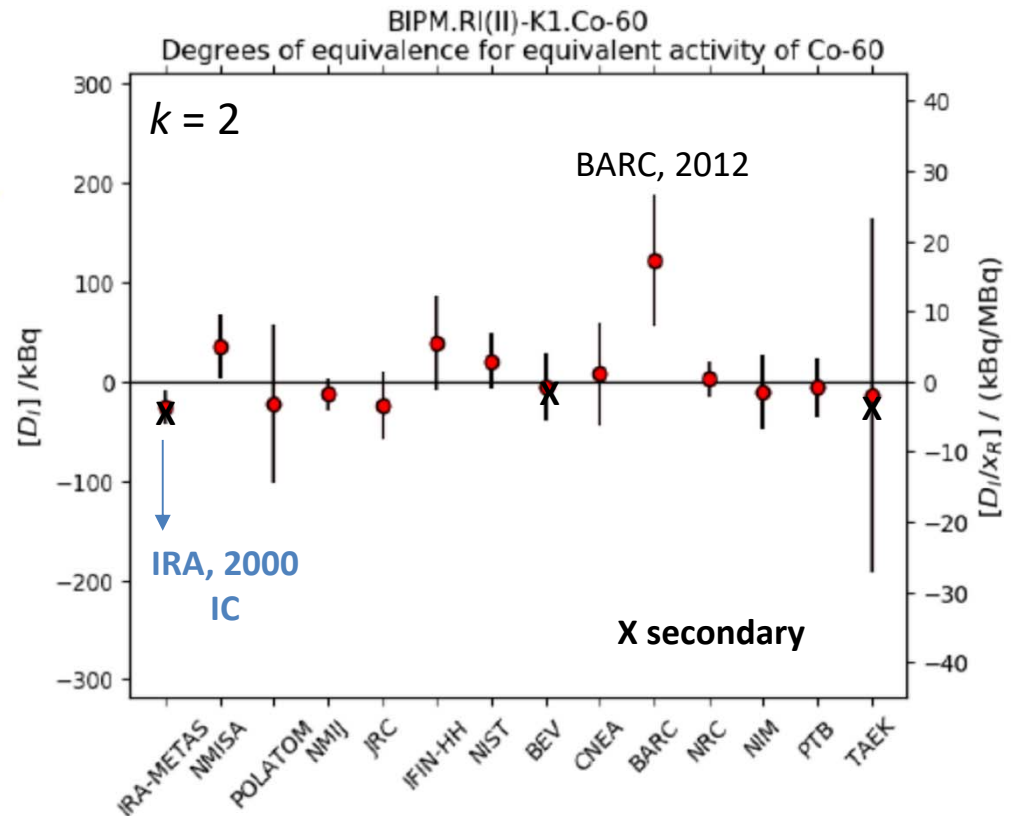
- Exception of old IC results for historical reason

## Exclusion

- KCWG(II) to recommend excluding some results on technical ground
- Outlier rejection if  $D_i / u(D_i) > +/- 2.5$  to be agreed by CCRI(II)

# Example: Co-60

- ◆ 72 ampoules measured since 1976
- ◆ 14 in the last 20 years 
- ◆ KCRV: one data for each NMI (if primary, not outlier,...): 25 entries



PMM	
mean	7062.7
std dev	2.7
chi	1.17
dark unc	6.4
power $\alpha$	1.88

# The SIR: link of RMO KC

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Ampoule(s) (mass  $M_L$ ) from the RMO KC measured in the SIR

For Lab  $j$  from RMO KC:  
 $A_{ej} = (A/m)_j \times \text{Link to SIR}$

Linking laboratory L

$$\begin{aligned} \text{Link} &= A_{eL} / (A/m)_L \\ &= \cancel{(A/m)_L} M_L / (I_L / I_{\text{Ra-5}}) / \cancel{(A/m)_L} \\ &\text{independent on linking lab K2 result} \end{aligned}$$

Example :  
recent Ge-68  
SIR Final report

$$A_{ej} = (A/m)_j M_L / (I_L / I_{\text{Ra-5}})$$

## The SIR: improved service

# The SIR : improved service

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## Quicker

- New NMI reporting form (Excel) + LabView
- Automation to calculate the comparison result
- start of use in January

## Quicker

- Automation to produce reports
- see R. Coulon's presentation

## Safer

- $^{226}\text{Ra}$ : almost 50 years old;  $\alpha$ -emitter
- To be replaced by  $^{166\text{m}}\text{Ho}$  next year

## More robust

- Transfer of KCRVs to 2<sup>nd</sup> SIR IC
- Project starting in January with NPL

## More robust

- Buy new IC (2020-2023 work program)



# The SIR : improved service



<http://www.magnicon.com>

## ULCA = Ultra-stable Low-noise Current Amplifier

### More robust

- ULCA to replace the old Keithley electrometer
- Absolute current traceable to SI
- Calibration each 20 years?
- Highly linear

- ◆ Workshop at NIST to review options
- ◆ Support of the new CCEM-CCRI Task Group on low current measurement
- ◆ First tests at BIPM on-going

I thank you for your attention,  
and invite my colleague  
Romain Coulon to continue.



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Mesures

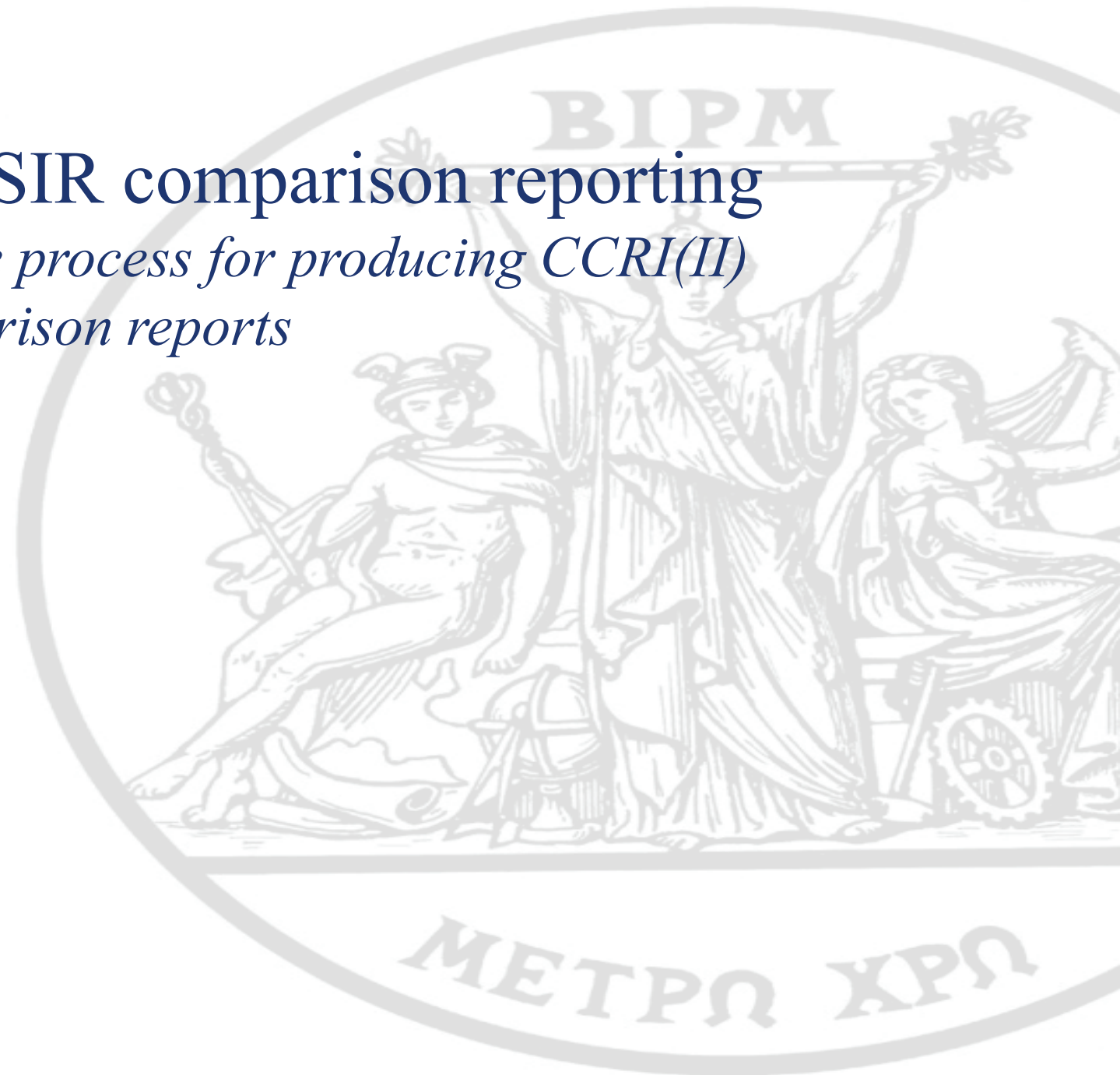
[www.bipm.org](http://www.bipm.org)

# Digitalization of the SIR comparison reporting

*\_ A faster and more secure process for producing CCRI(II) comparison reports*

BIPM Ionizing Radiation Department  
CCRI webinar 15<sup>th</sup> December 2020

**B**ureau  
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† **P**oids et  
† **M**esures



# How to build an efficient reporting of SIR comparison data?

## Objectives

**Optimize the workforce and the velocity of SIR comparisons**

**Limit risk factors and their frequency of occurrence**

**Give opportunities toward more digital data exchange between BIPM and NMIs**

## Strategies

### Maximize the automatization

- Software must support the maximum of work

### Build a scheme as simple as possible

- One database
- One software
- One repository

### Secure the data and the software

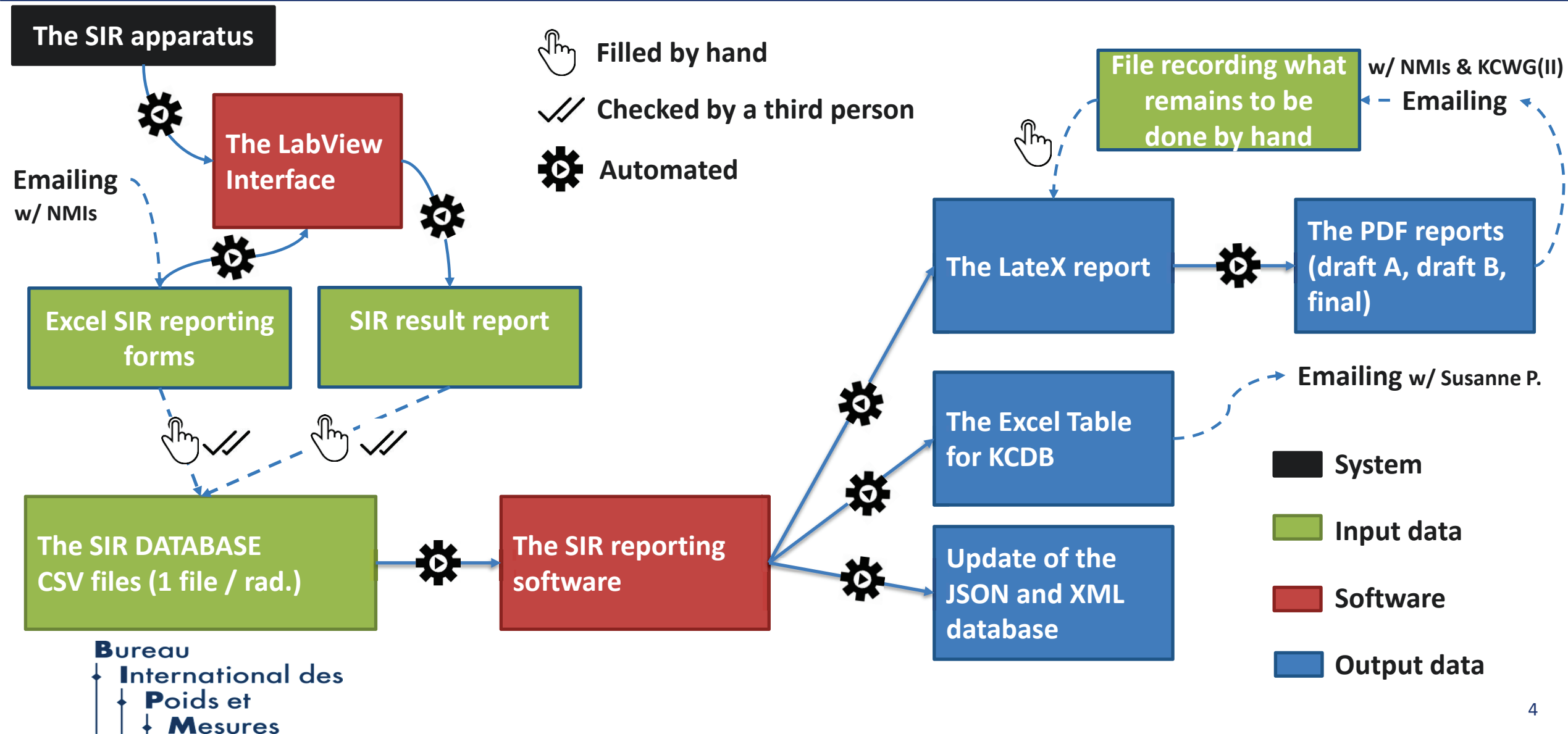
- A robust Life Cycle process
- A robust version control system

### Comply with FAIR principles

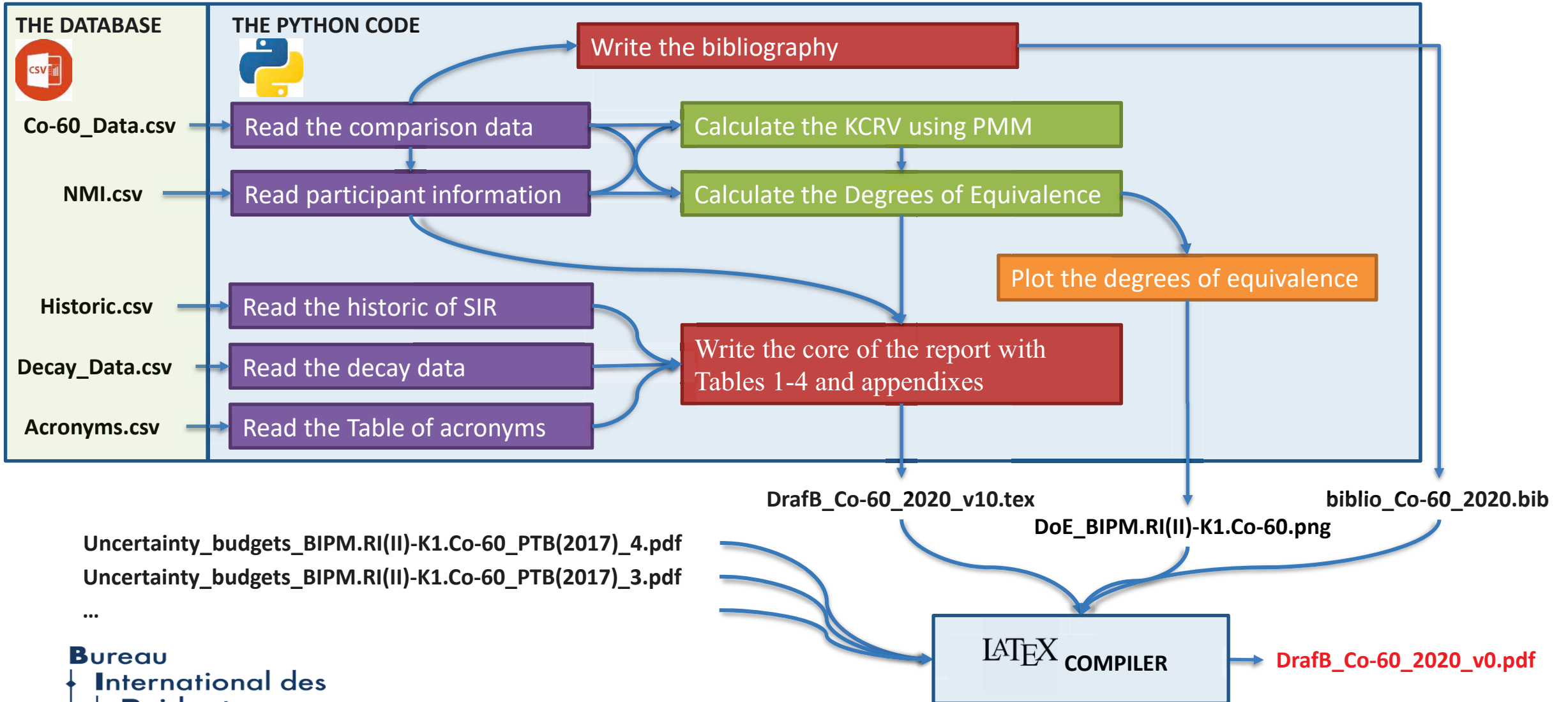
- Machine readable format of the database
- Availability on the KCDB API



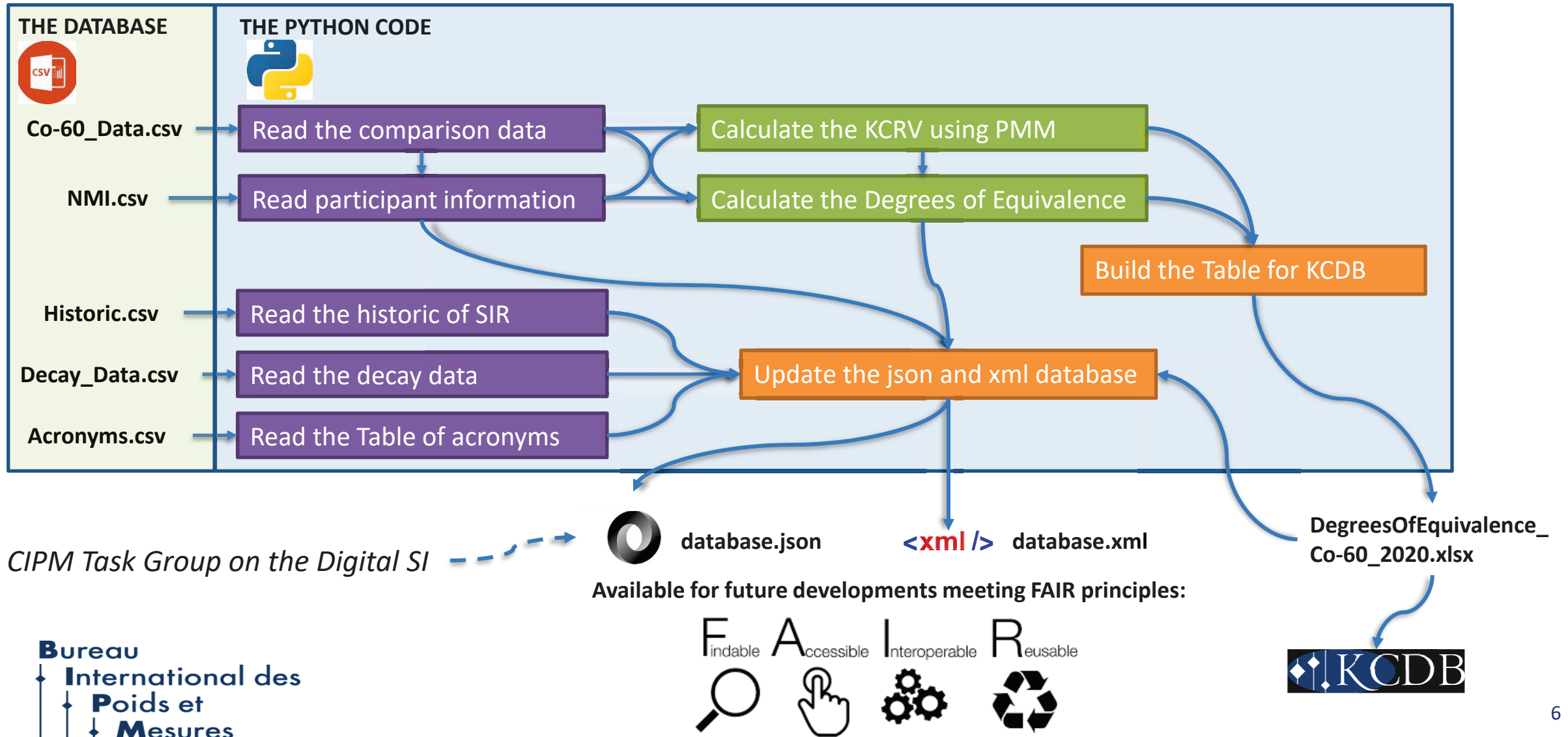
# The new SIR reporting flowchart



# The SIR reporting software - Production of comparison reports

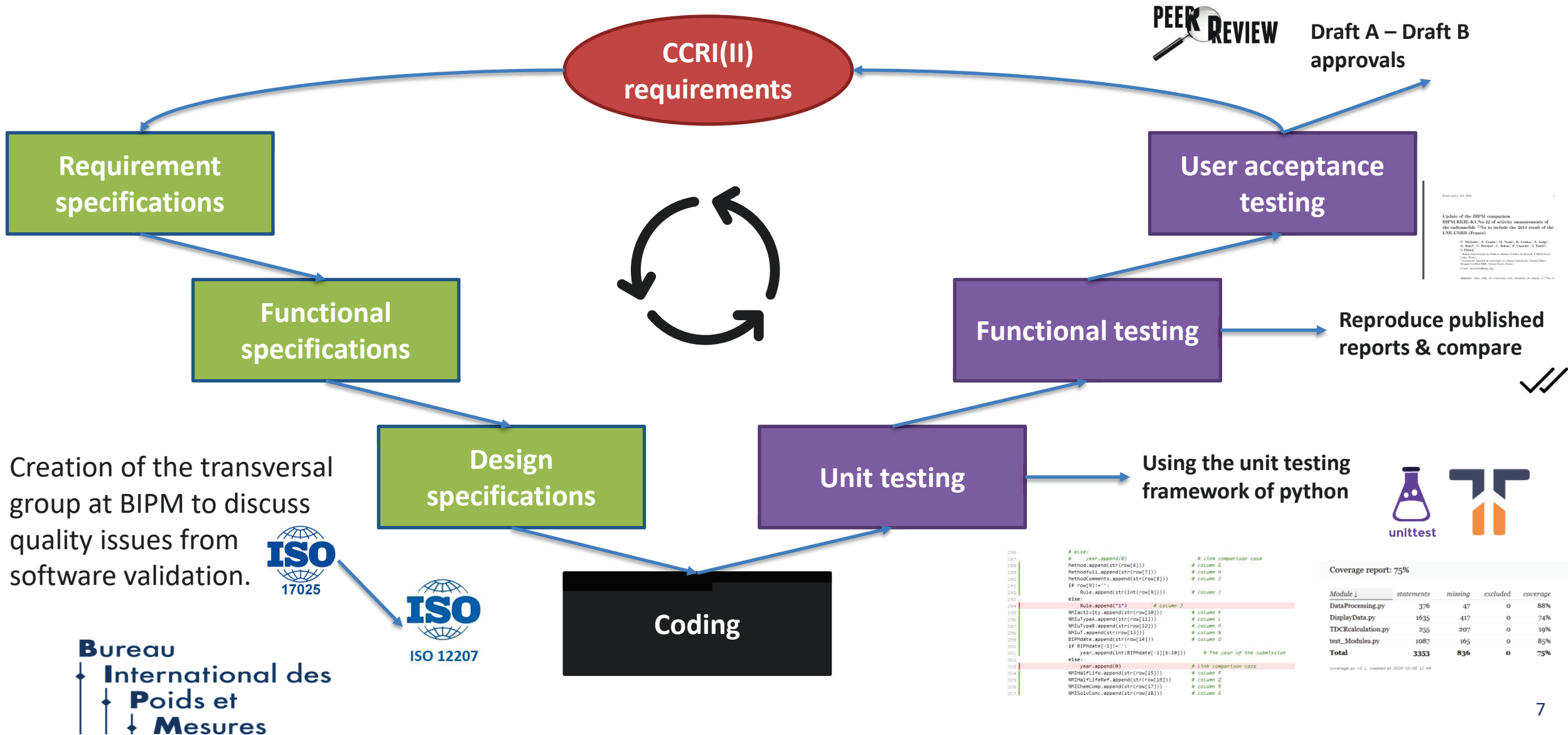


# The SIR reporting software – Production of KCDB Table and machine-readable databases





# The SIR reporting software – The Life Cycle





# Documentation of the digitalized SIR reporting

## 5 BIPM procedures



Python Docstrings - embedded into the script

ENTERING DATA IN THE SIR MASTER FILE				Bureau International des Poids et Mesures Qualité Management System
Author	Date	Authorized	BIPM/RI-SIR-100	
R Coulon	08/10/2020 Version: 1.0	S M Judge		

**ENTERING DATA IN THE SIR MASTER FILE**

**1. Purpose and Objectives**

1.1. This procedure describes the actions necessary to enter historical data into the SIR Master File.

ENTERING NEW DATA IN THE SIR DATABASE				Bureau International des Poids et Mesures Qualité Management System
Author	Date	Authorized	BIPM/RI-SIR-101	
R Coulon	08/10/2020 Version: 1.0	S M Judge		

**ENTERING NEW DATA IN THE SIR DATABASE**

**1. Purpose and Objectives**

1.1. This procedure describes the actions necessary to enter new data into the SIR database.

GENERATING A COMPARISON REPORT FROM THE DATABASE				Bureau International des Poids et Mesures Qualité Management System
Author	Date	Authorized	BIPM/RI-SIR-102	
R Coulon	08/10/2020 Version: 1.0	S M Judge		

**GENERATING A COMPARISON REPORT FROM THE DATABASE**

**1. Purpose and Objectives**

1.1. This procedure describes the actions necessary to produce comparison reports (draft A, draft B and final), associated tables (Table for KCDB) and plots (DoEs and PomPlots) from the SIR database.

Updating the xml and json databases				Bureau International des Poids et Mesures Qualité Management System
Author	Date	Authorized	BIPM/RI-SIR-103	
R Coulon	08/10/2020 Version: 1.0	S M Judge		

**UPDATING THE XML AND JSON DATABASES**

**1. Purpose and Objectives**

1.1. This procedure describes the actions necessary to produce convert the secured CSV database to API readable formats xml and json.

DOCUMENTATION AND VALIDATION OF THE SOFTWARE FOR AUTOMATED REPORTING OF SIR COMPARISON RESULTS				Bureau International des Poids et Mesures Qualité Management System
Author	Date	Authorized	BIPM/RI-SIR-104	
R Coulon	08/10/2020 Version: 1.0	S M Judge		

**DOCUMENTATION AND VALIDATION OF THE SOFTWARE FOR AUTOMATED REPORTING OF SIR COMPARISON RESULTS**

**1. Purpose and Objectives**

1.1. This procedure describes the actions necessary to automatically update the documentation of the digitalized SIR database and to produce validation records.



```

_build/html/DocStrings.html#structure-of-the-code-sirreport-py
    
```

### From module DataProcessing.py

This module contains a set of functions used for the processing of measurement data and time series.

Created on Wed May 20 15:31:18 2020

Updated on Wed May 20 15:36:29 2020

@author: [romain.coulon@bipm.org](mailto:romain.coulon@bipm.org)

`DataProcessing.PMM(x, u, *, autoRej=False, k=2.5, conv=0.0001)`

This function calculates the Power-Modered Mean (PMM) on a data sample.

References:

- [Accred Qual Assur (2008)13:83-89, Metrologia 52(2015)S200]
- <https://link.springer.com/article/10.1007/s00769-007-0330-1>
- <https://iopscience.iop.org/article/10.1088/0026-1394/52/3/S200/pdf>

This method is applied to calculate de Key Comparison Reference Value (KCRV) related to international inter-laboratory comparisons of the CIPM/CCRI(II). It was validated from PMM v1.xlsm provided by S. Pommé [email of the 14 Jan 2020]

- Parameters:**
- **x** (array of floats) – Sample of values
  - **u** (array of floats) – Sample of standard uncertainties related to the values
  - **r** – (Optional) Set if the auto-rejection capability is activated or not (False by default)
  - **k** (float) – (Optional) Threshold (see as quantiles of a Normal density) of the frequentist test (default value = 2.5)
  - **conv** (float) – (Optional) Convergence paramater (set ny default equal to 10<sup>-4</sup>)

# Conclusion

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## ◆ Summary

- A clear specification from the needs
- A process as simple as possible (1 database – 1 software – 1 repository)
- Defining a dedicated life cycle process
- Using only text encoded files (.csv, .json, .xml, .tex, .bib, .py)
- Using only licence-free and largely supported software
  - ◆ Python, Git, GitHub, Sphinx...

## ◆ Achievement

- Draft reports produced for 10 radionuclides
- Draft B in circulation for approval: Ga-67, Sr-85, Tl-201, Ra-223, Cd-109, Cs-137, Ba-133
- Final reports: Na-22, Co-60, Ag-110m

How to use the SIR

# Step 1: Check that the radionuclide is covered

- Download <https://www.bipm.org/utis/en/pdf/RI-SIR-T-20.pdf>
- Check that the radionuclide is listed in the table in the document

Procedure to submit a sample for measurement on the SIR				Bureau International des Poids et Mesures Quality Management System
Author : C Michotte	Date: 6/10/2020 Version : 1.1	Authorized: S M Judge	BIPM/RI-SIR-T-20	

## Procedure to submit a sample for measurement on the SIR

### 1. Purpose and objectives

- 1.1. This procedure is for use by national metrology institutes or designated institutes wishing to send a sample for measurement on the SIR.

### 2. Comments

- 2.1. Please follow this procedure carefully to avoid possible delays in shipments and measurements.
- 2.2. Samples for measurement on the SIR must be supplied in ampoules that the BIPM will provide.
- 2.3. Please contact the BIPM on [sir@bipm.org](mailto:sir@bipm.org) for help in following this procedure if required.

# Step 2: Prepare the ampoule

- Contact [SIR@bipm.org](mailto:SIR@bipm.org) to arrange a date for the measurement
- You will receive an empty ampoule by post
- You **must** use this ampoule for the sample
- Dispense 3.6 g of standardized solution into the ampoule
- Don't forget to determine any radioactive impurities, estimate the measurement uncertainties and note the chemical form
- Seal the ampoule and check it is not leaking



# Step 3: Dispatch your ampoule

- Complete the BIPM shipping form
- Package the ampoule following the IATA regulations
- Complete all the IATA documents and dispatch the ampoule to the BIPM

Administration procedure/Shipping instructions for comparisons			Bureau International des Poids et Mesures Admin
<u>Authors:</u> L. Dell'Oro E. Van	<u>Date:</u> 2019/11/28 <u>Version:</u> 4.0	<u>Authorized:</u> M. Milton	ADM-DOU-F-02

(to be filled by the NMI and emailed to : [customsbipm@bipm.org](mailto:customsbipm@bipm.org) and technical contact for comparison)

## 1. SHIPPING INSTRUCTION FOR COMPARISONS :

• Name of the NMI: \_\_\_\_\_  
• Contact: \_\_\_\_\_  
• Address: \_\_\_\_\_  
• Tel : \_\_\_\_\_ • e-mail: \_\_\_\_\_

## 2. TYPE OF SHIPMENT: CARGO TRANSPORT

ATA carnet:  Temporary Importation  
Diplomatic bag:

## 3. SHIPPING INFORMATION: TO F

• Description of the equipment: (including UN \_\_\_\_\_)



<https://www.bipm.org/en/bipm/ionizing/radionuclides/sir/participation.html>



# Step 4: Send the details of the source

- Download and complete the Excel form
- The form asks for information on the radionuclide, uncertainties, measurement technique used, chemical form and impurities
- Email the form to SIR@bipm.org

	A	B	C	D	Formu
1	<b>SIR/SIRTI reporting form - radioactive solution</b>				page 1
2	BIPM.RI(II)-K1 or BIPM.RI(II)-K4				
3		Date			
4	<b>Participant laboratory</b>				
5		Acronym			
6		Full name			
7		Country			
8		Address			
9		Contact person			
10		E-mail address			
11	Persons who participated in the measurement				
12					
13	<b>Radionuclide (e.g. Ho-166m)</b>				
14		Half-life used by the participant			
15		Standard uncertainty (k=1)			
16		Unit			
17		Reference			
18					
19	<b>Participation mode</b>				
20		To generate a degree of equivalence			
21		Or as pilot study (no publication in the KCDB)			

<https://www.bipm.org/en/bipm/ionizing/radionuclides/sir/participation.html>

# Step 5: Results

- BIPM staff carry out the measurements and generate a Draft A report.
- The Draft A report is sent to you for checking.
- Any typographical errors can be corrected.
- For key comparisons, BIPM staff circulate the Draft B report to CCRI(II) for review and then arrange publication on the KCDB.
- The comparison is then finished - you will have an entry in the database and a publication in the Metrologia Technical Supplement

New software



# Summary

