ความสอบกลับได้ของผลการวัดทางเคมี
Metrological traceability of measurement results in chemistry

กิตติยา เชียร์แมน

สถาบันมาตรวิทยาแห่งชาติ (มว.)

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เอกสารอ้างอิงสำหรับ Metrological traceability

- **IUPAC Technical Report (2011) (Metrological traceability of measurement results in chemistry: Concepts and implementation)**

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Metrological traceability of measurement results in chemistry: Concepts and implementation (IUPAC Technical Report)*

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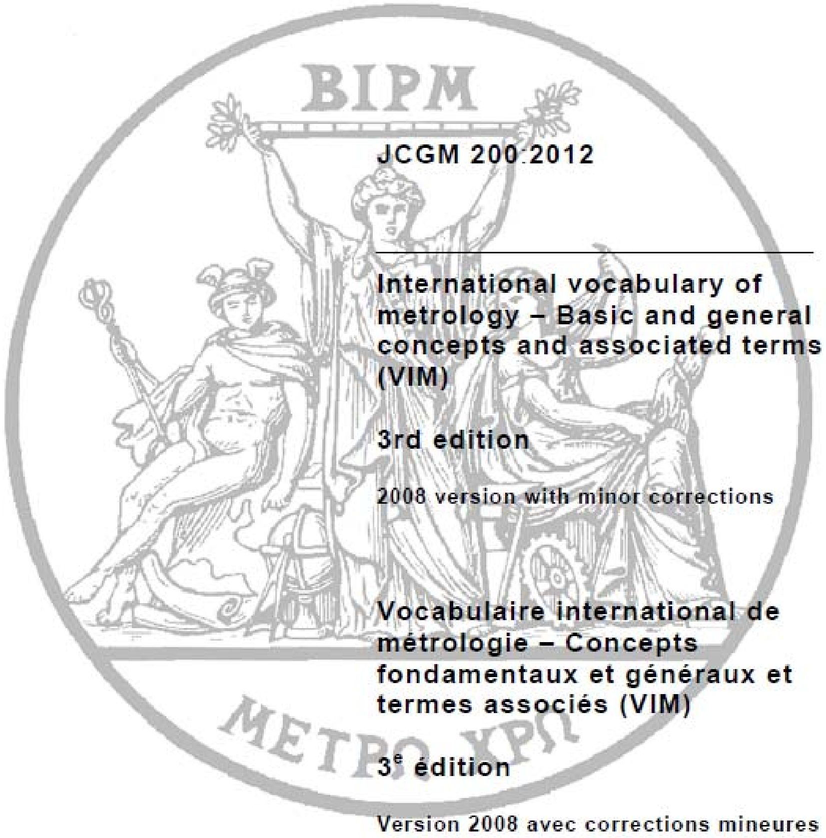
Abstract: This IUPAC study aims at formulating recommendations concerning the metrological traceability of a measurement result in chemistry. It is intended to provide the chemical measurement community with a consistent view of the creation, meaning, and role of metrological traceability and its underpinning concepts. No distinction is made between measurement results obtained in "high metrology" and in the "field". A description is given of the calibration hierarchies needed in different circumstances to arrive at metrological traceability along a metrological traceability chain. Flow charts of generic calibration hierarchies are presented as well as a variety of examples. The establishment, assessment, and reporting of metrological traceability are discussed, including the provision of metrological references by a metrological institutional framework and the role of interlaboratory comparisons.

Keywords: calibration hierarchies; IUPAC Analytical Chemistry Division; measurand; measurement; metrological comparability; metrological reference; metrological traceability.

เอกสารอ้างอิงสำหรับ Metrological traceability

- **International vocabulary of metrology- (2012)-Basic and general concepts and associated terms (VIM)**



เอกสารอ้างอิงสำหรับ Metrological traceability

UNSW
THE UNIVERSITY OF NEW SOUTH WALES

Are our measurement results metrologically traceable? – and does it matter?

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National Institute of Metrology (Thailand)

Content

- Introduction:
- Definition: Metrological traceability
- Metrological reference:
- RMs/CRMs
- Examples of metrological traceability chain of measurement results

Introduction:

Measurement

… is the comparison between a known value and an unknown value

$$\frac{Q_1}{Q_2} = \frac{\text{quantity}_1}{\text{quantity}_2} = \text{numerical value}$$

If quantity_2 is a realisation of the unit then

$$\text{quantity}_1 = \text{numerical value} \,.\, \text{unit}$$

Measurement results

Measurement results

Measurement results

Measurement results

From different locations, times

reliability

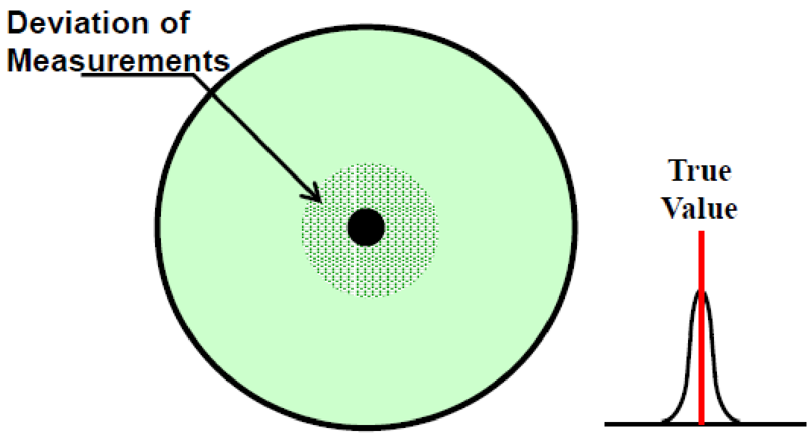
Metrological comparability of measurement results

Concepts “calibration hierarchies”

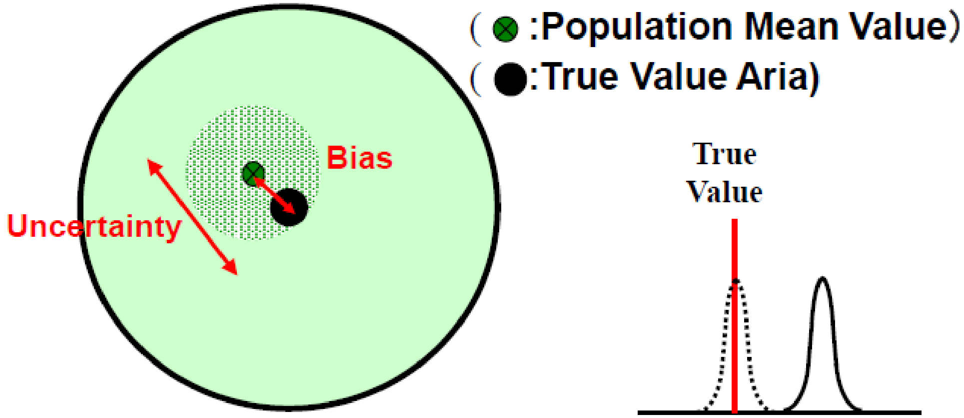
create “metrological traceability chain”

establish “metrological traceability”

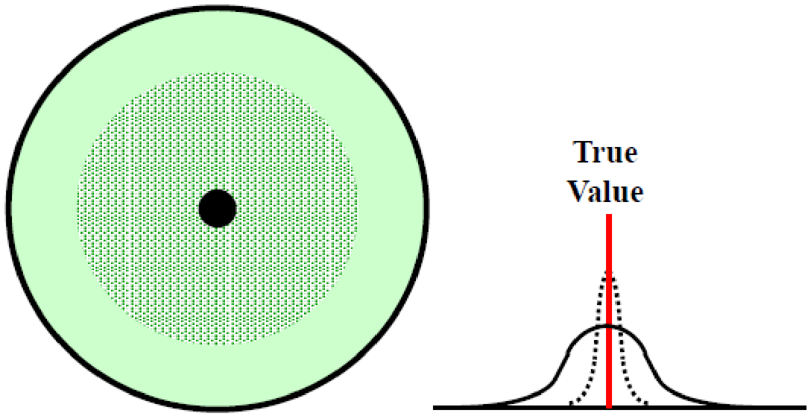
Trueness



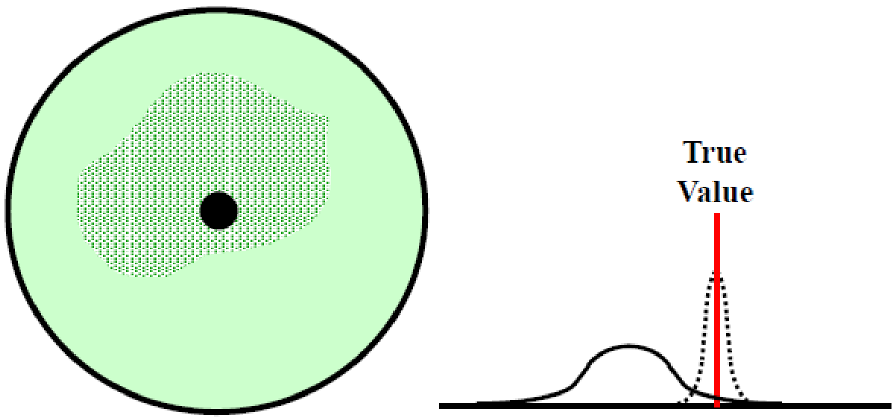
Trueness; excellent
Precision; excellent



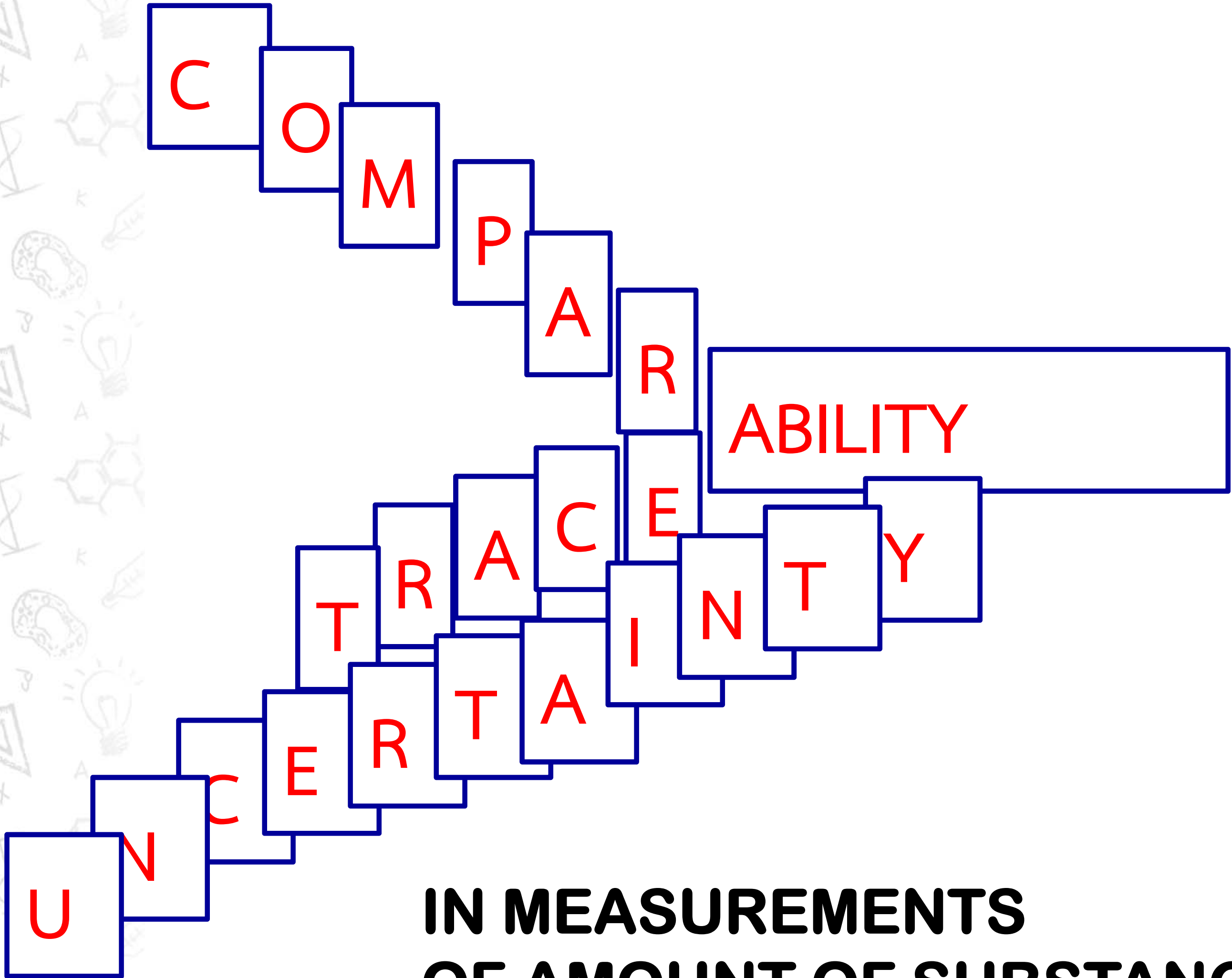
Trueness; poor
Precision; excellent



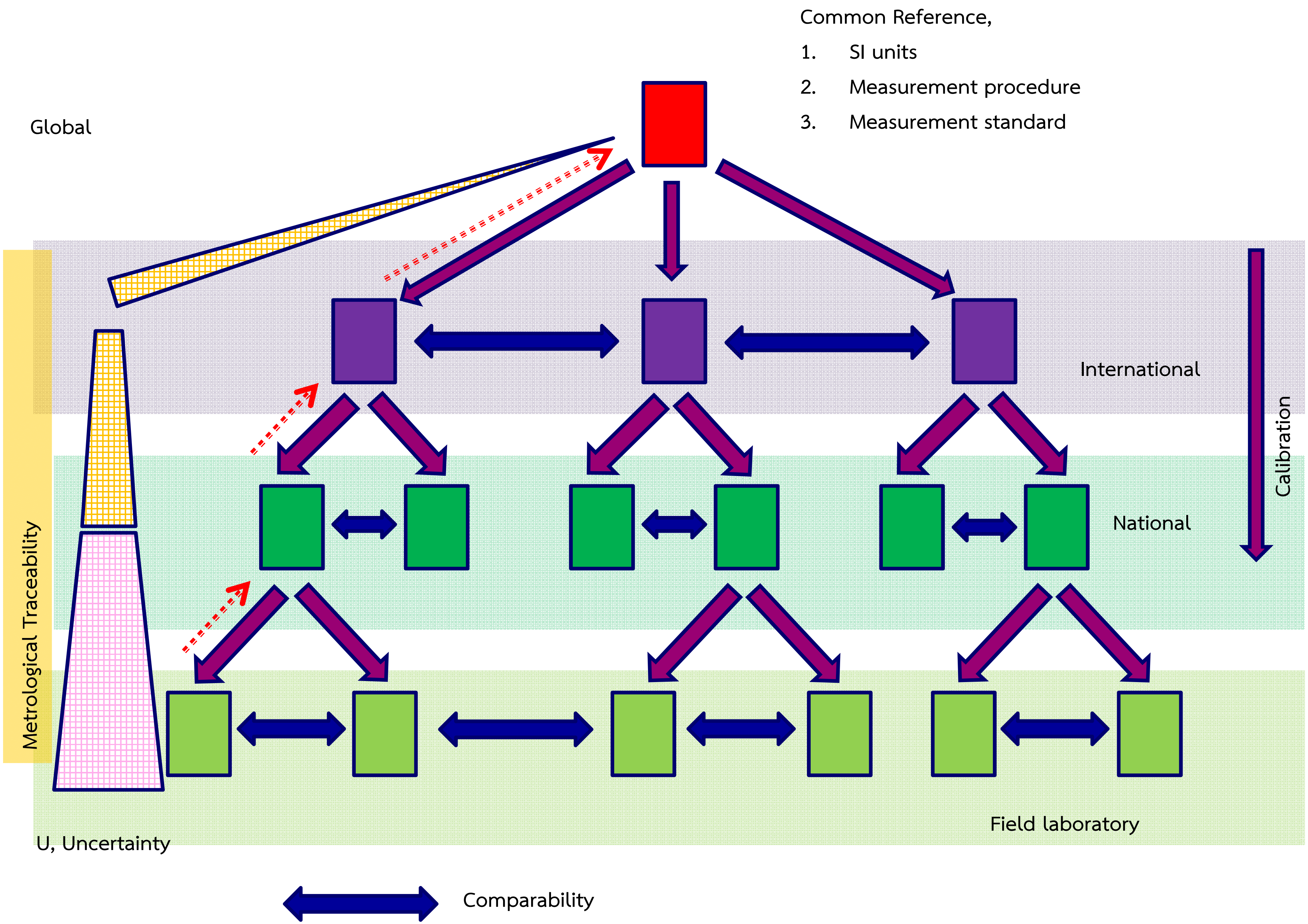
Trueness; fair
Precision; poor



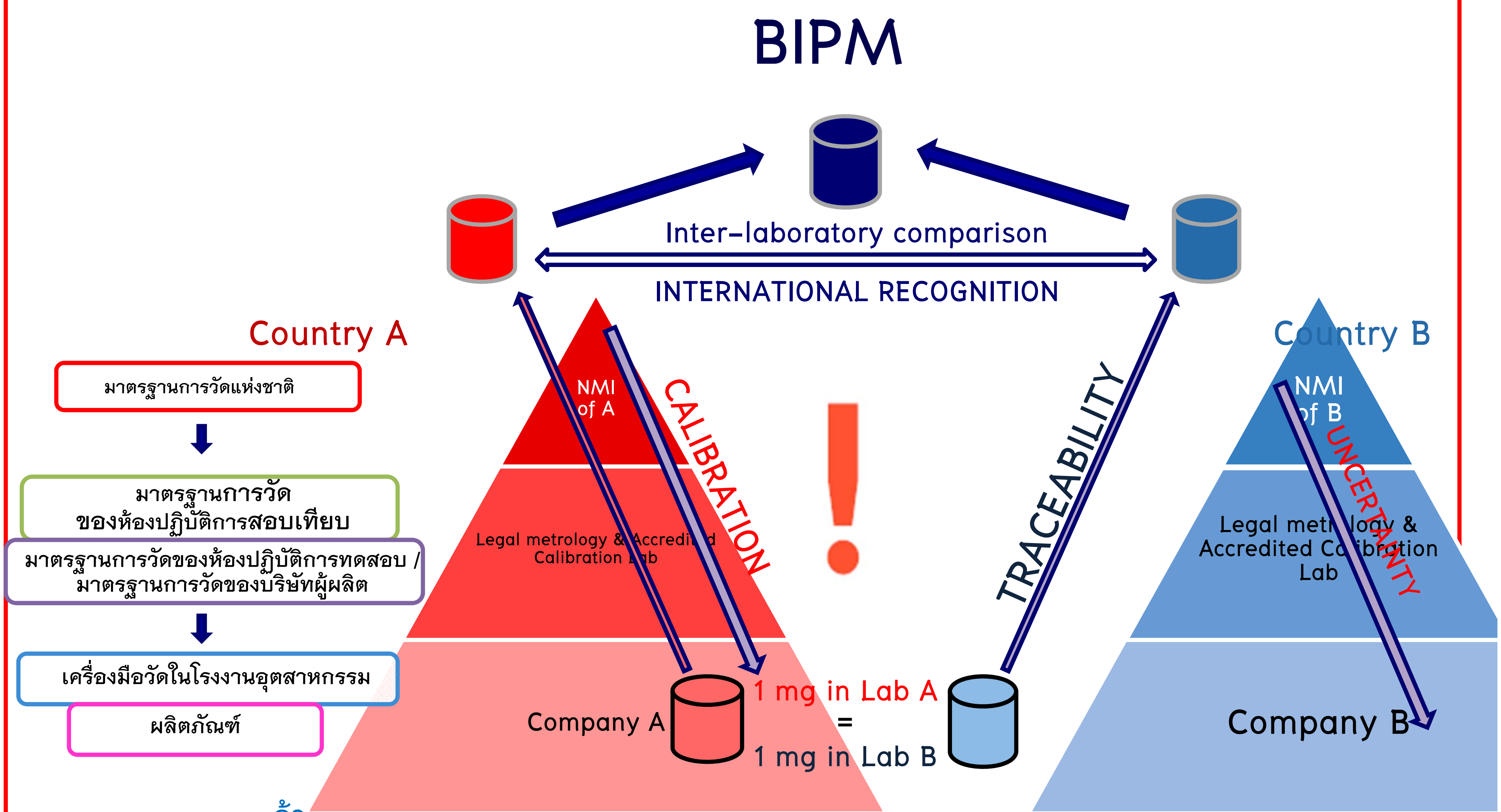
Trueness; poor
Precision; poor



IN MEASUREMENTS
OF AMOUNT OF SUBSTANCE

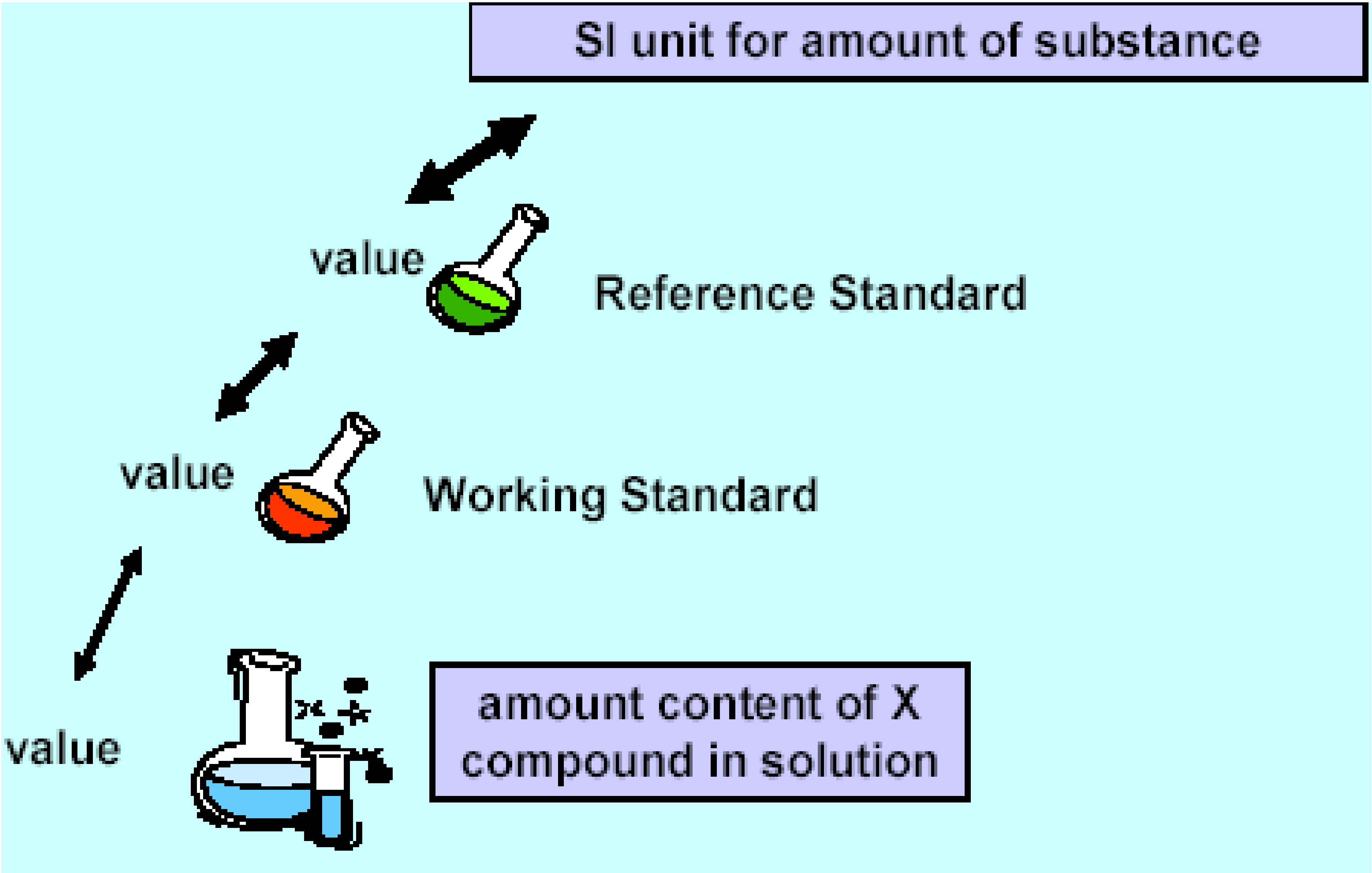

Common Reference,
1. SI units
2. Measurement procedure
3. Measurement standard
Global
International
Calibration
National
Metrological Traceability
Field laboratory
U, Uncertainty
Comparability

กระบวนการทางมาตรวิทยาเพื่อประกันความเชื่อมั่นในผลการวัด



อ้างอิง: Clemens Sanetra, Rocio M. Marban, 2007

Metrological Traceability



ISO 17025: 2005

5.6 Measurement traceability

5.6.1 General

All equipment used for tests and/or calibrations, including equipment for subsidiary measurements (e.g. for environment conditions) having a significant effect on the accuracy or validity of the result of the test, calibration or sampling shall be calibrated before being put into service. The laboratory shall have an established programme and procedure for the calibration of its equipment.

ISO 17025: 2005

5.6.2 Specific requirements

5.6.2.1 Calibration

5.6.2.1.1 For calibration laboratories, the programme for calibration of equipment shall be designed and operated so as to ensure that calibrations and measurements made by the laboratory are traceable to the SI unit.

A calibration laboratory establishes traceability of its own measurement standards and measuring instruments to the SI by means of an unbroken chain of calibrations or comparisons linking them to relevant primary standards of the SI units of measurement. The link to Si units may be achieved by reference to national measurement standards.

ISO 17025: 2005

National measurement standards may be primary standards, which are primary realizations of the SI units or agreed representations of SI units based on fundamental physical constants, or they may be secondary standards which are standards calibrated by another national metrology institute. When using external calibration services, traceability of measurement shall be use of calibration services from laboratories that can demonstrate competence, measurement capability and traceability. The calibration certificates issued by these laboratories shall contain the measurement results, including the measurement uncertainty and/or a statement of compliance with an identified metrological specification.

ISO 17025: 2005

5.6.2.2 Testing

5.6.2.2.1 For testing laboratories, the requirements given in 5.6.2.1 apply for measuring and test equipment with measuring functions used, unless it has been established that the associated contribution from the calibration contributed little to the total uncertainty of the test result. When this situation arises, the laboratory shall ensure that the equipment used can provide the uncertainty of measurement needed.

Note The extent to which the requirements in 5.6.2.1 should be followed depends on the relative contribution of the calibration uncertainty to the total uncertainty. If calibration is the dominant factor, the requirements should be strictly followed.

ISO 17025: 2005

5.6.2.2.2 Where traceability of measurements to SI units is not possible and/or not relevant, the same requirements for traceability to, for example, certified reference materials, agreed methods and/or consensus standards, are required as for calibration laboratories (see 5.6.2.1.2)

Challenges on the concept and the use of the term " traceability"

- In spite of the definition having traceability as a property of a measurement result , it is common also to refer to the traceability of
  - a document such as a measurement procedure (which is a physical object), or
  - sample (which is a physical object), or
  - measurement (which is a process).

"document traceability" "sample traceability"

- It is often claimed that a measurement result can be traceable to an institution (e.g., a specified National Metrology Institute)

- Despite a growing awareness of the need for metrological traceability of measurement results, some field and routine laboratories still assert that metrological traceability is not "applicable" to their measurement results.

Challenges on the concept and the use of the term “ traceability”

- It is not generally accepted that traceability to a common stated metrological reference is a precondition for metrological comparability of measurement results.

- The colloquial meaning of the term “comparability” often refers to quantity values of the same magnitude (size).

- There is the perception that a measurement unit from the International System of Units (SI) is the only possible metrological reference in the metrological traceability of chemical measurement results.

- There is a belief that the use of a reference material (RM) or a certified reference material (CRM) for quality control purposes automatically establishes metrological traceability.

Challenges on the concept and the use of the term " traceability"

- X Claims are made that satisfactory participation in an interlaboratory comparison, proficiency testing scheme, or external quality assessment scheme automatically provides metrological traceability of the participants' measurement results.

- VIM does not define the concept "metrological reference".

metrological traceability is only considered to be a property of a measurement result (and thereby also of a measured quantity value)

Definition:metrological traceability

Metrological Traceability

- **property of a <u>measurement result</u> whereby the result can be related to <u>a reference</u> through a documented unbroken chain of <u>calibrations</u>, each contributing to the <u>measurement uncertainty</u>**

VIM3 [2.41]

ความสอบกลับได้ทางมาตรวิทยาของผลการวัด

สมบัติของผลการวัด โดยที่<u>ผลการวัด</u>นั้น สัมพันธ์กับ<u>สิ่งอ้างอิง</u>อย่างไม่ขาดช่วง<u>การสอบเทียบ</u>ที่ได้จัดทำเป็นเอกสารไว้ โดยการสอบเทียบแต่ละครั้งมีส่วนต่อ<u>ความไม่แน่นอนการวัด</u>

VIM, 3rd Edition
ฉบับภาษาไทยแปลโดย ราชบัณฑิตยสถาน

Notes to VIM definition (1)

- NOTE 1 For this definition, a 'reference' can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.
- NOTE 2 Metrological traceability requires an established calibration hierarchy.
- NOTE 3 Specification of the reference must include the time at which this reference was used in establishing the calibration hierarchy, along with any other relevant metrological information about the reference, such as when the first calibration in the calibration hierarchy was performed.

Notes to VIM definition (2)

- NOTE 4 For measurements with more than one input quantity in the measurement model, each of the input quantity values should itself be metrologically traceable and the calibration hierarchy involved may form a branched structure or a network. The effort involved in establishing metrological traceability for each input quantity value should be commensurate with its relative contribution to the measurement result.
- NOTE 5 Metrological traceability of a measurement result does not ensure that the measurement uncertainty is adequate for a given purpose or that there is an absence of mistakes.
- NOTE 6 A comparison between two measurement standards may be viewed as a calibration if the comparison is used to check and, if necessary, correct the quantity value and measurement uncertainty attributed to one of the measurement standards.

Notes to VIM definition (3)

- **NOTE 7 The ILAC considers the elements for confirming metrological traceability to be an unbroken metrological traceability chain to an international measurement standard or a national measurement standard, a documented measurement uncertainty, a documented measurement procedure, accredited technical competence, metrological traceability to the SI, and calibration intervals (see ILAC P-10:2002).**

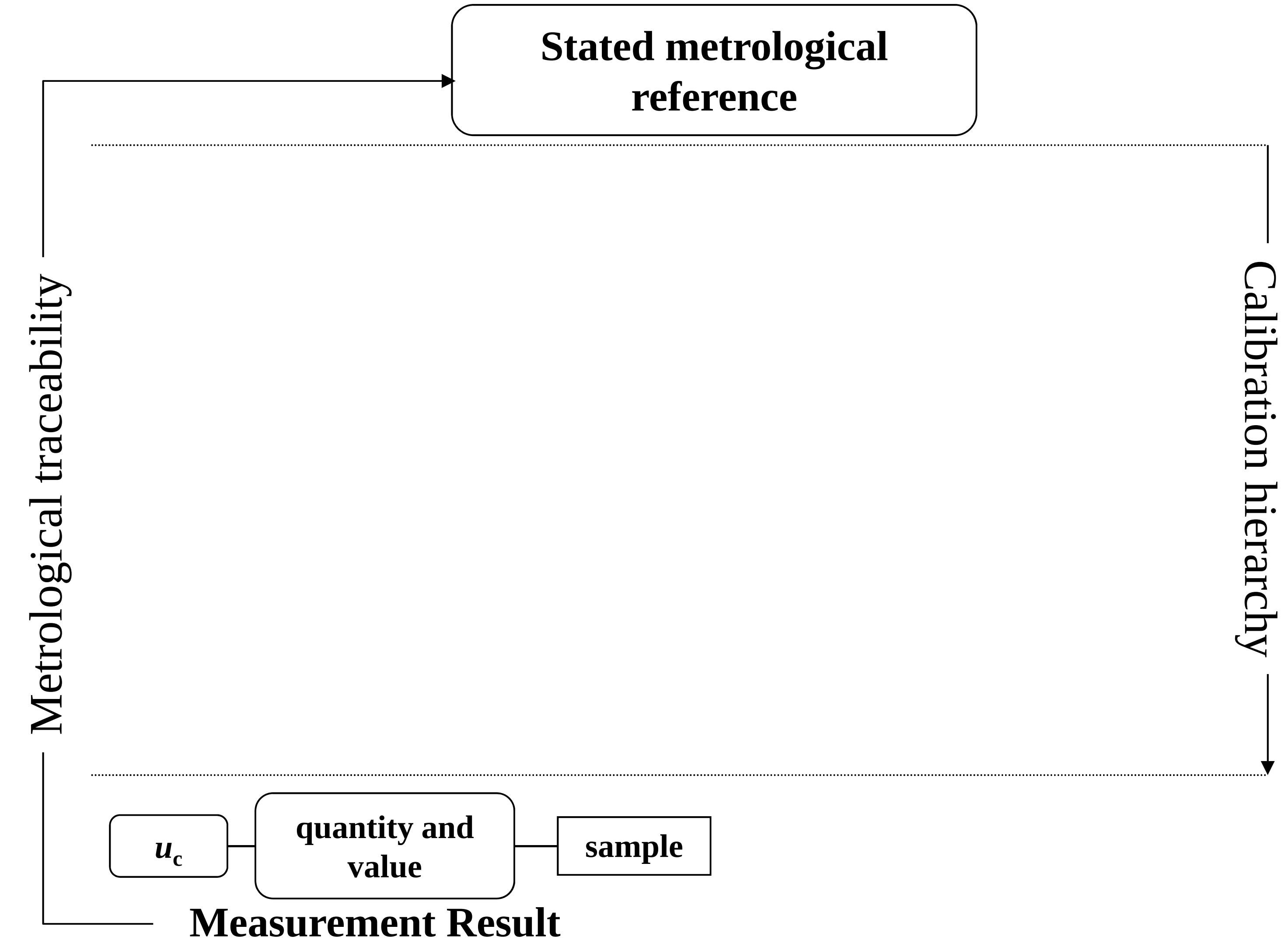
- **NOTE 8 The abbreviated term "traceability" is sometimes used to mean 'metrological traceability' as well as other concepts, such as 'sample traceability' or 'document traceability' or 'instrument traceability' or 'material traceability', where the history ("trace") of an item is meant. Therefore, the full term of "metrological traceability" is preferred if there is any risk of confusion.**

Traceability v trackability

- We will use the term metrological traceability to refer to chain of comparisons used to establish a measurement

- Traceability is also used for audit trails – tracing a document for example. Sometimes this is known as 'trackability'

Metrological Traceability



Ref: B. Hibbert

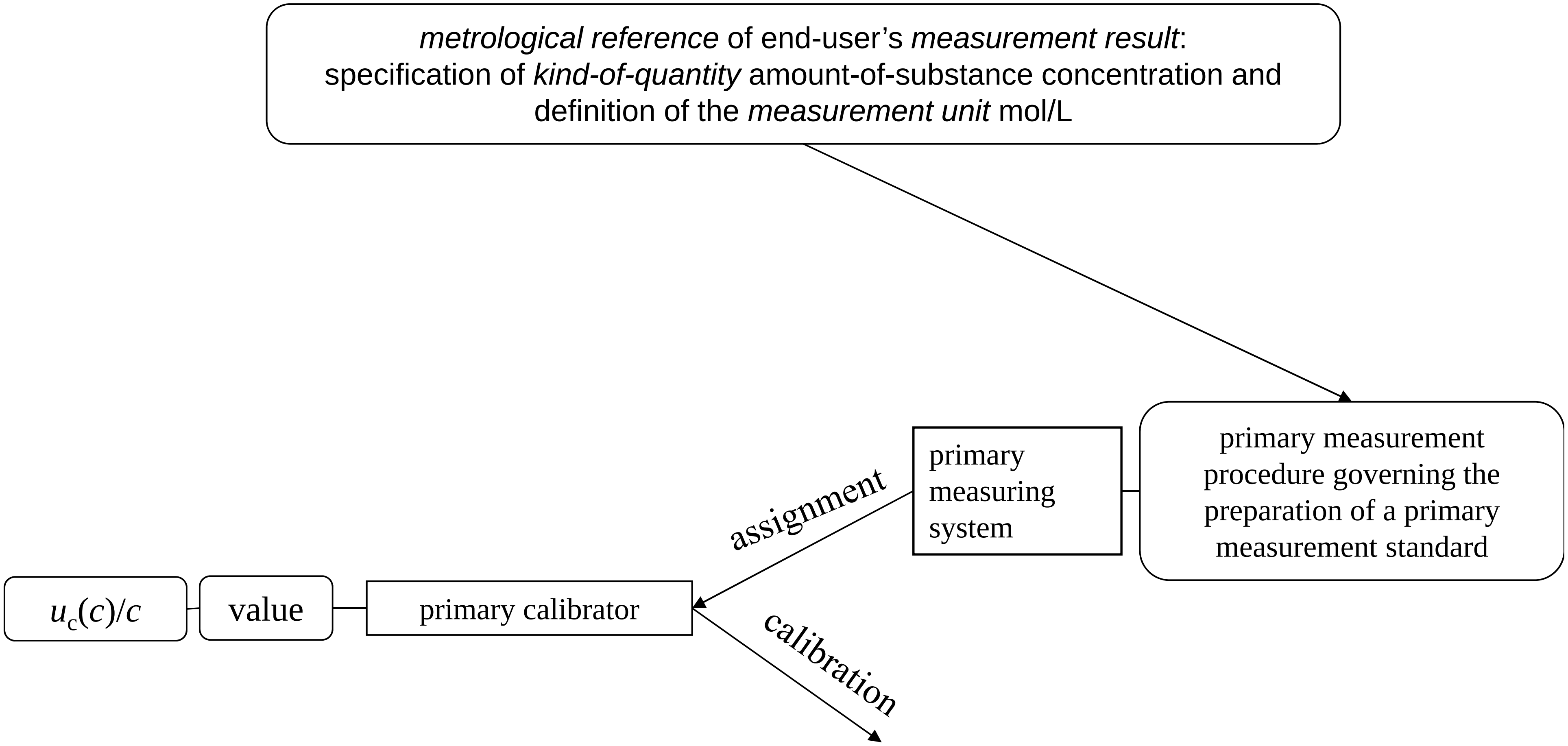
What is a 'metrological reference' ?

- NOTE 1 For this definition, a 'reference' can be a definition of a measurement unit through its practical realization, or a measurement procedure including the measurement unit for a non-ordinal quantity, or a measurement standard.

- We also include the definition of an ordinal quantity value scale

e.g. Rockwell hardness, Beaufort wind

Metrological traceability



Ref: B. Hibbert

Primary methods

CCQM Definition:

Ref: B. Hibbert

"A primary method of measurement is a method having the highest metrological qualities, whose operation is completely described and understood, for which a complete uncertainty statement can be written down in terms of SI units, and whose results are, therefore accepted without reference to a standard of the quantity being measured."

Features :

- **Highest metrological quality**
- **Operation of the method is fully understood**
- **A complete knowledge of uncertainty of its results in SI units**
- **No reference to a standard of the quantity being measured required**

Primary methods

- **Gravimetry:** analyte separated from the sample in a weighable form such as precipitation and its mass or amount of substance is calculated from the mass of the weighed compound whose stoichiometric composition is exactly known.
- **Coulometry :** amount of substance is directly determined by electrical current and time measurements in an electrochemical reaction.
- **Titrimetry:** the analyte is determined by measuring the equivalent volume of titrant solution of known concentration required for a reaction to proceed completely stoichiometrically.

Ref: B. Hibbert

Primary methods-continued

- **IDMS**: a known amount of an internal standard is added gravimetrically to the sample. The internal standard is the pure analyte with a different isotopic composition. The amount of substance of the analyte in the sample is determined by a measurement equation which contains only isotopic abundance ratios. These ratios can be measured using a mass spectrometer.

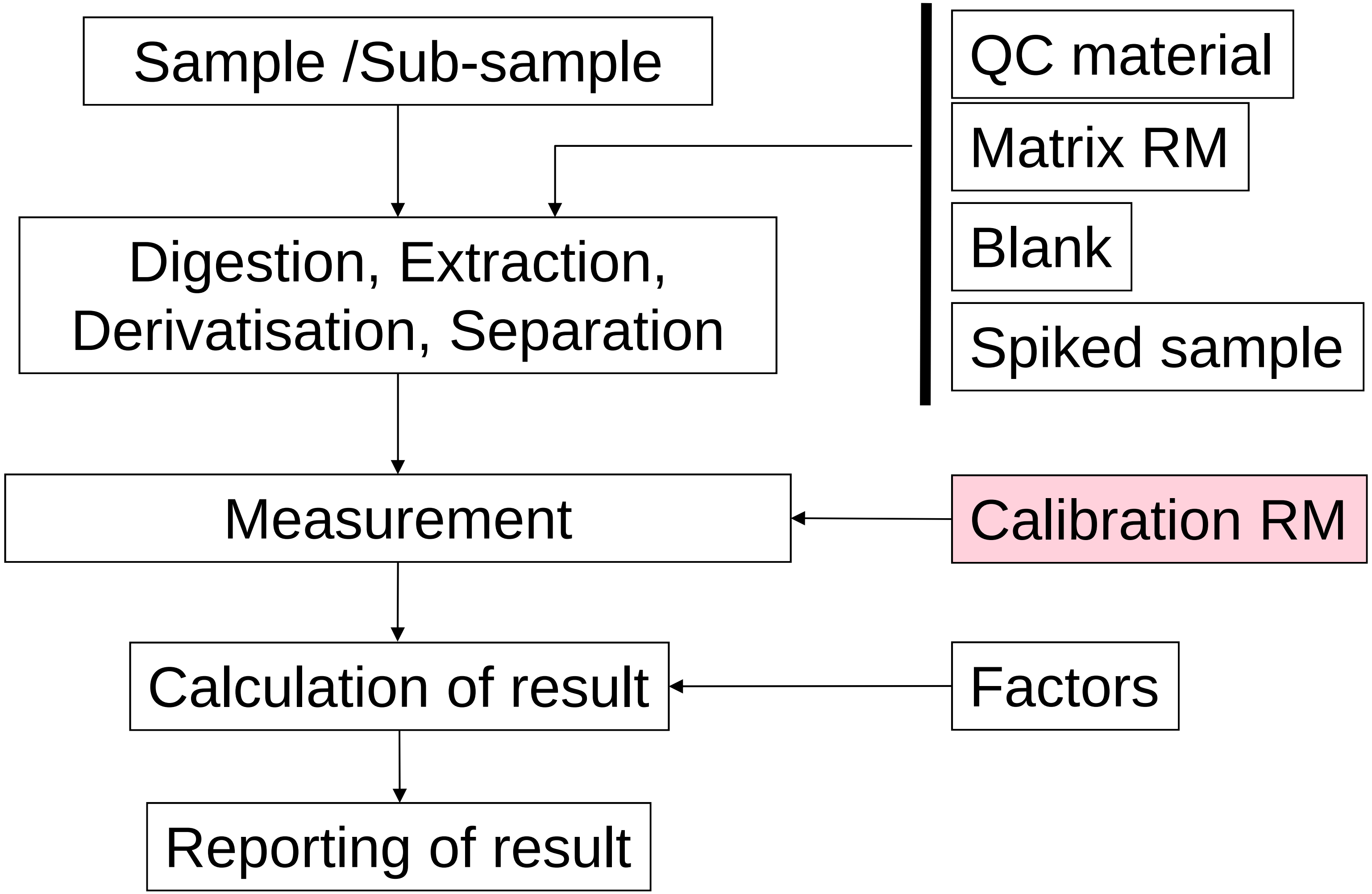
Ref: B. Hibbert

Summary of primary methods in chemistry

Method	Uncertainty contributions	Typical relative uncertainty	Metrological reference
Gravimetry	•Incomplete chemical separations •Volatilization of substance •Stoichiometric proceeding of the reaction •Uncertainties involved with corrections for buoyancy	10^{-4}	Mass standard for the calibration of the balance
Coulometry	•Incomplete chemical reaction •Stoichiometric proceeding of the reaction •Additional current contributing processes •Unwanted side reactions	10^{-5}	Standards of electrical current, mass and time
Titrimetry	•Incomplete chemical reaction •Determination of the equivalence point	10^{-3}	Standard of mass
IDMS	•Non-ideal behavior of real mass spectrometers •Uncertainty associated with sample pretreatment	Lower than gravimetry and titrimetry	Standard of mass

Ref: B. Hibbert

The Analytical Process

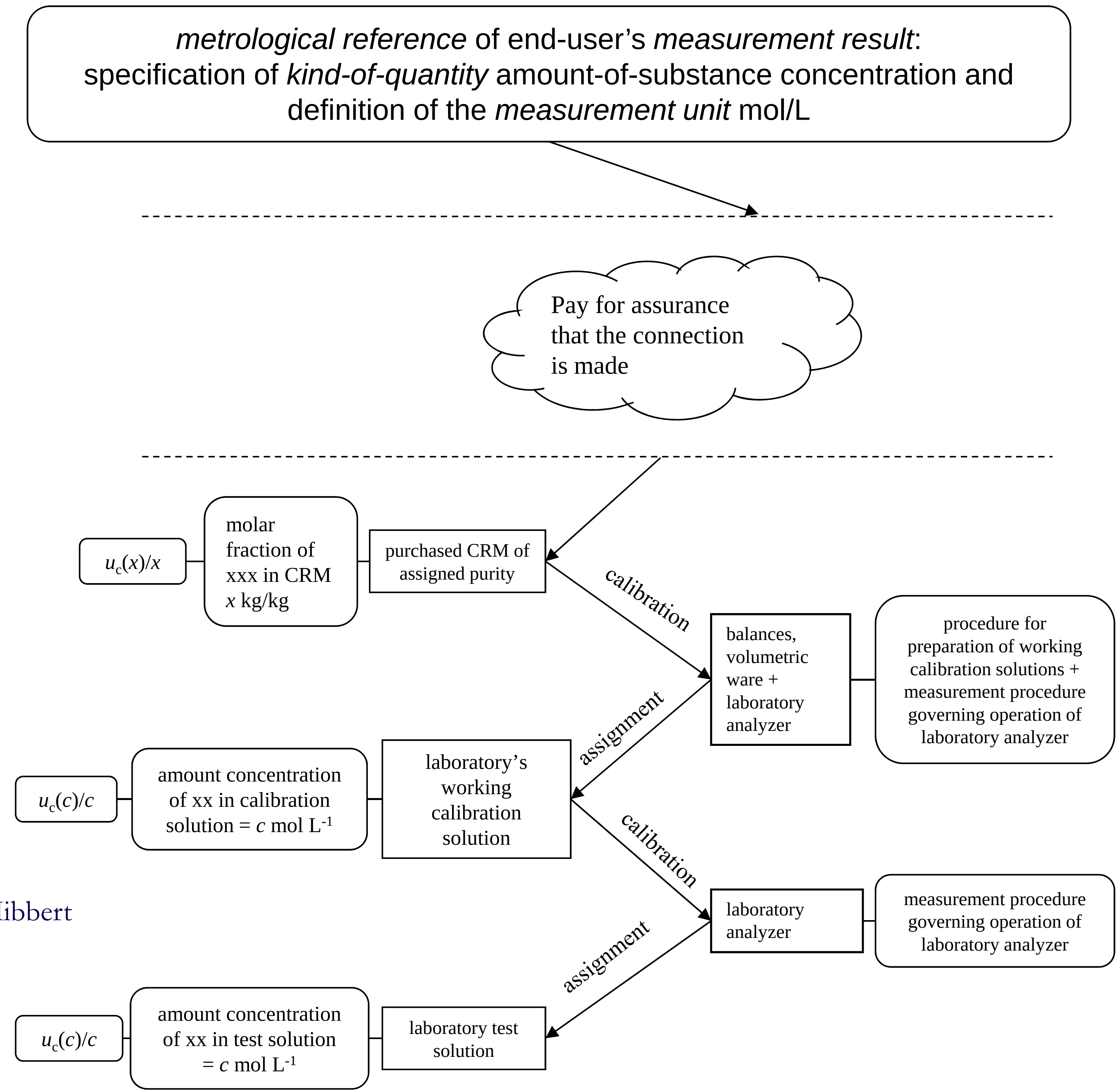


Ref: B. Hibbert

Traceability and CRMs

- **Note that only a traceable reference material used <u>as a calibrator is part of the traceability chain</u>**
- **RMs (or CRM) used to establish bias or check the calibration do not establish traceability**

Ref: B. Hibbert



Ref: B. Hibbert

RMs/CRMs

วัสดุอ้างอิง/วัสดุอ้างอิงรับรอง

สรุปความหมายของ RM และ CRM

- Reference Material, RM → Specified properties value
  → Homogeneous
  → Stable

Certified Reference Material (CRM) หรือ วัสดุอ้างอิงรับรอง คือ วัสดุหรือสารมาตรฐานที่มีความเป็นเนื้อเดียวมีความเสถียร และมีใบรับรองค่าของคุณสมบัติที่เราสนใจ พร้อมแสดงค่าความไม่แน่นอนของผลการวัด และระบุการสอบย้อนกลับได้ของผลการวัด เพื่อใช้สำหรับ

1) สอบเทียบเครื่องมือวัด
2) ตรวจสอบความใช้ได้/ยืนยันความถูกต้องของวิธีทดสอบ
3) ควบคุมคุณภาพของวิธีการทดสอบ

- **Certified Reference Material, CRM**
  - → **Specified property value**
  - → **Homogeneous**
  - → **Stable**

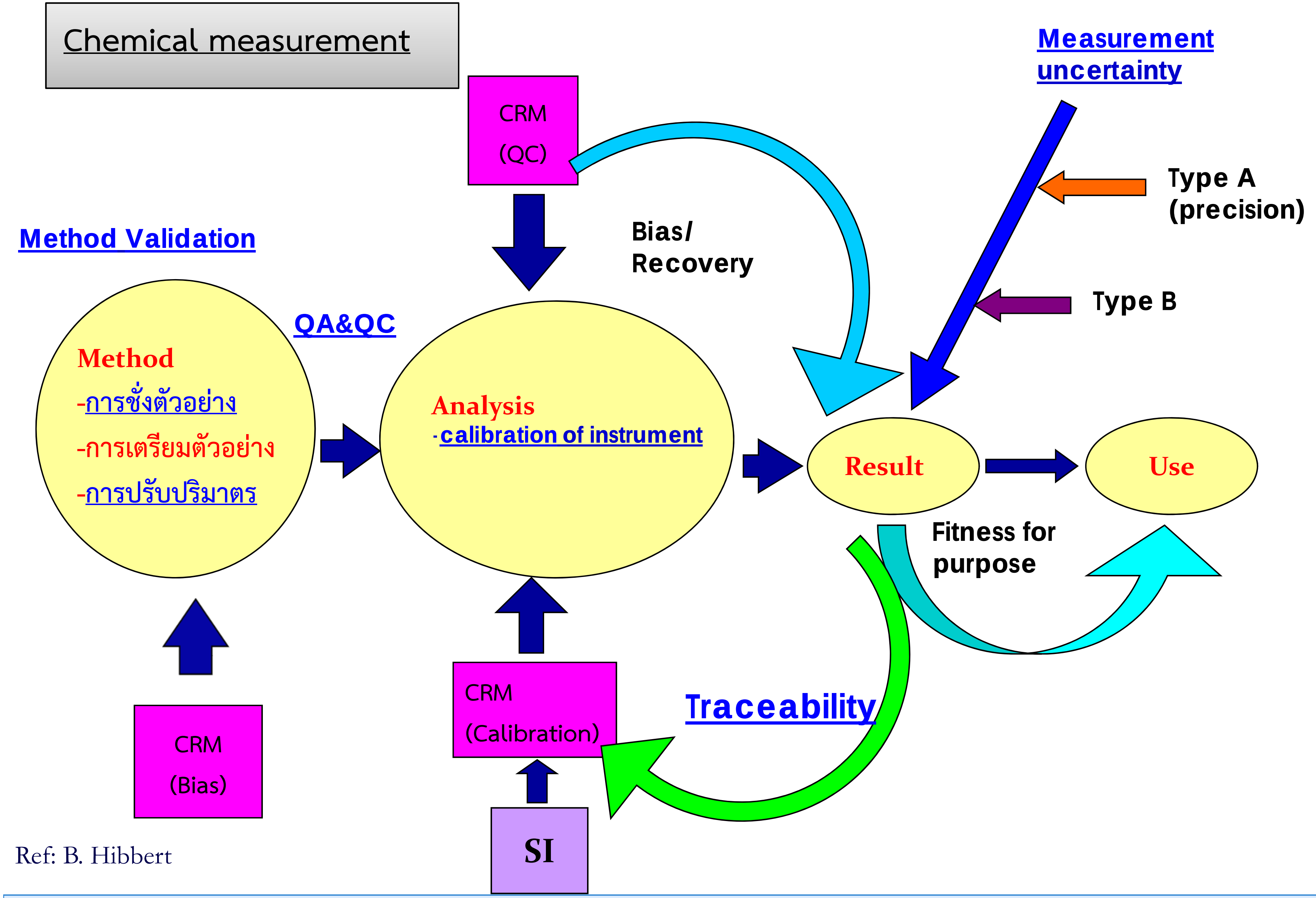
  RM

  - → **Certificate (acc. ISO Guide 31) , issued by an authoritative body + Uncertainty + Traceability**
  - → **Metrologically valid procedure**

- **Reference material** → **Pure substance reference material**
  → **Matrix reference material**

ปัจจัยที่ควรพิจารณาในการเลือกใช้ RMs ที่เหมาะสม

- **Analyte ที่ต้องการวัด**
- **ระดับความเข้มข้น (อยู่ในช่วงที่ต้องการ)**
- **Matrix ใกล้เคียงกับตัวอย่าง**
- **ปริมาณต้องเพียงพอต่อการใช้งานได้ตลอด**
- **เสถียรภาพและความเป็นเนื้อเดียวกัน (Homogeneity and stability)**
- **ค่าความไม่แน่นอน (Measurement uncertainty)**



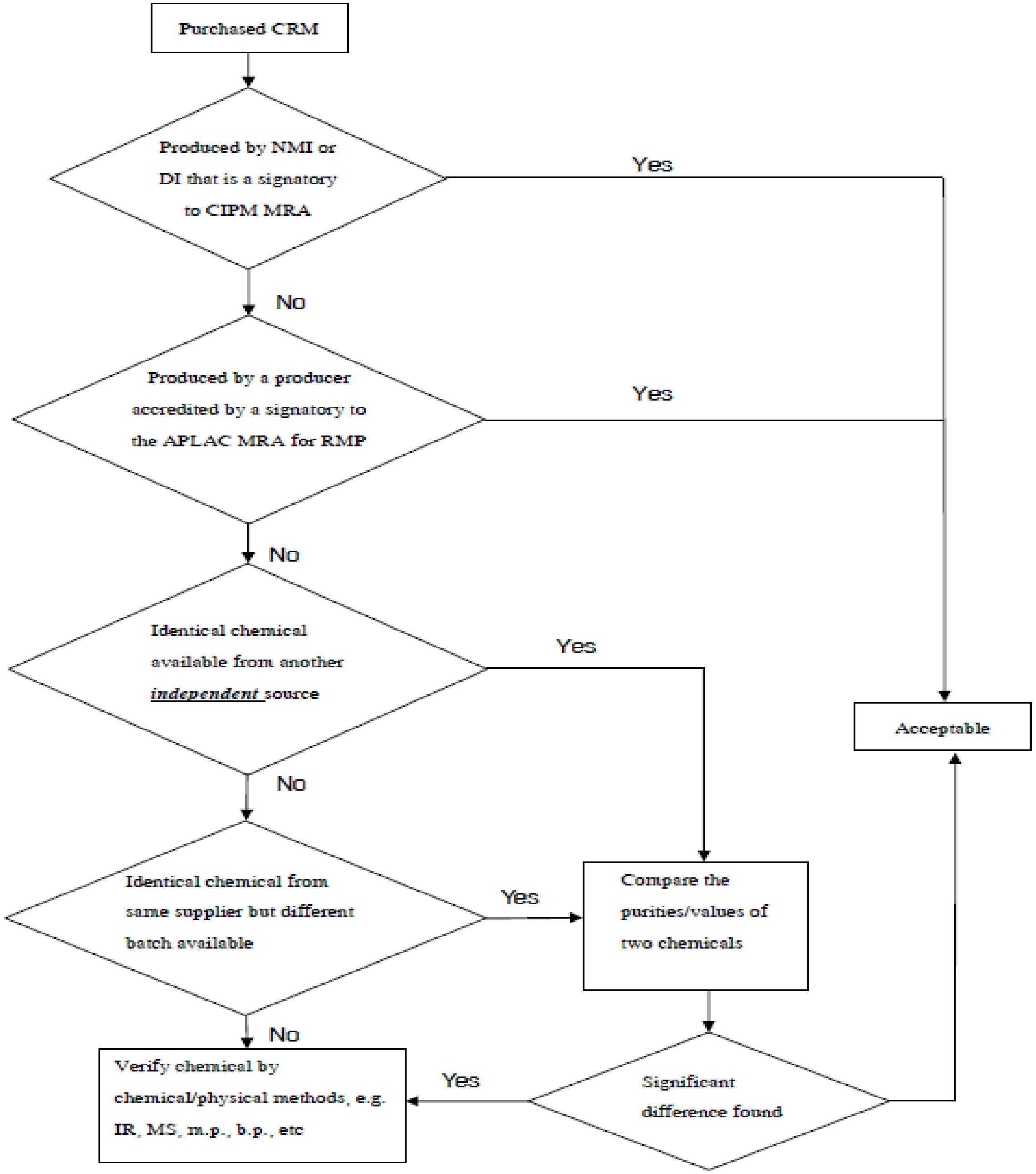
Ref: B. Hibbert

Instrument Calibration/Source of traceability

Analyte	Supplier	Stated Concentration	Traceable to SI?
Cd	NIST SRM	10,000 mg/kg	Yes
Cd	Sigma-Aldrich	1,000 mg/kg	Yes

Annex I

Flowchart for Acceptability of RM



การเลือกใช้ CRM สำหรับการสอบเทียบเครื่องมือวัดทางเคมี

Use of Reference materials in chemical measurement

- Method development and validation
  - evaluation of trueness
  - uncertainty estimation
- Calibration of instrument
- Proof of method performance
  - statistical quality control
  - establishing traceable results
  - equipment qualification
- Proficiency testing
  - training and verification of competence

Use of Reference materials in chemical measurement

- Method development and validation
  - evaluation of trueness
  - uncertainty estimation

Uncertainty from recovery

ERM Aplication Note 1

1. Determine difference (Δ_m) measured vs. certified value

$$\Delta_m = |C_m - C_{CRM}|$$

2. Determine uncertainty of the cert. value (u_{CRM})
3. Determine uncertainty of the measurements (u_m)
4. Combine u_{CRM} and u_m to u_Δ

$$u_\Delta = \sqrt{u_m^2 + u_{CRM}^2}$$

5. Compare Δ_m with $2 * u_\Delta$

   if $\Delta_m < 2 * u_\Delta \Rightarrow$ no difference

T-test

Within the uncertainty of the certified value

Use of Reference materials in chemical measurement

- Calibration of instrument
  - Traceability of measurement result
  - CRM is needed
  - Uncertainty estimation

Use of Reference materials in chemical measurement

- Proof of method performance
  - statistical quality control
  - establishing traceable results
  - equipment qualification

QCRM

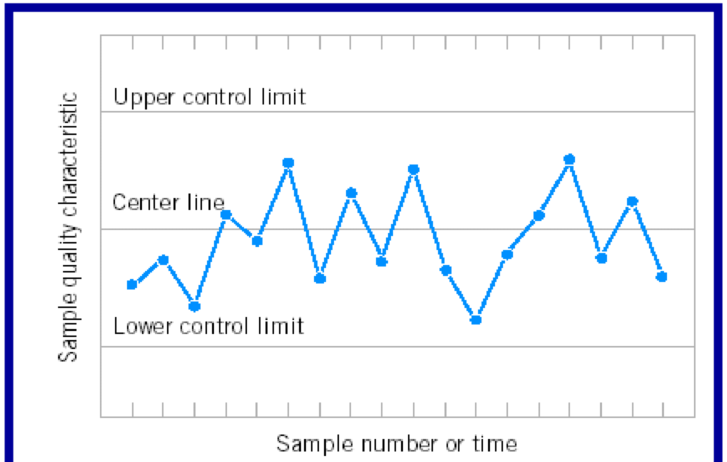


Figure 8-1 A typical control chart.

- Proficiency testing
  - training and verification of competence

RM & CRM

วิธีดำเนินการวัด: Measurement procedure

วิธีดำเนินการวัด มี 2 ประเภท คือ แบบ Empirical และ แบบ Rational

- **แบบ Empirical** หมายถึง วิธีดำเนินการวัดที่ผลการวัดขึ้นอยู่กับ measurement procedure ที่กำหนดรายละเอียดขั้นตอนต่าง ๆ ไว้ในวิธีดำเนินการวัดนั้นๆ และผลการวัดจะสอบกลับได้ (traceable to) ไปยัง measurement procedure นั้นๆ ที่ใช้ แต่ไม่สามารถสอบกลับถึงระบบหน่วยระหว่างประเทศ เอสไอ (International System of Units ; SI units) เช่น การหาความชื้นในตัวอย่าง โดยทำการอบตัวอย่างที่อุณหภูมิ 115 องศาเซลเซียส เป็นเวลา 2 ชั่วโมง แต่ถ้าอบที่อุณหภูมิ หรือเวลาต่างกัน ผลการวัดที่ได้จะต่างกันด้วย ดังนั้นการวัดด้วยลักษณะวิธีแบบนี้จะต้องทำตามวิธีดำเนินการอย่างเคร่งครัด ห้ามทำการดัดแปลงใด ๆ

วิธีดำเนินการวัด: Measurement procedure

วิธีดำเนินการวัด มี 2 ประเภท คือ แบบ Empirical และ แบบ Rational

- **แบบ Rational** หมายถึงวิธีดำเนินการวัดที่ผลการวัด ไม่ขึ้นกับวิธีดำเนินการวัดที่ระบุไว้ การใช้วิธีดำเนินการวัดที่แตกต่างกัน จะให้ผลการวัดที่ไม่แตกต่างกัน และความสามารถสอบกลับได้ทางมาตรวิทยาของผลการวัดจะไปยังระบบหน่วยระหว่างประเทศ (SI units) โดยตรง หรือ โดยผ่านวัสดุอ้างอิงรับรอง (certified reference material, CRM) เช่น การหาปริมาณสารเมลามีน โดยวิธีใช้เครื่องมือ LC-MS/MS กับวิธีใช้เครื่องมือ GC-MS จะให้ผลการวัดที่ไม่ต่างกัน

Note: สำหรับการวัดทางเคมีส่วนใหญ่วิธีดำเนินการวัดจะเป็นแบบ Rational ยกเว้นกรณี การวัด pH ซึ่งจะเป็นการวัด pH = -lg aH+ การสอบกลับได้ของผลการวัด pH จะไปยัง primary measurement procedure ที่เรียกว่า Harned cell measurement system

การวัดโดยใช้วิธีที่ยอมรับ

(Measurement using an accepted procedure)

- การแสดงความสอบกลับได้โดยวิธีนี้ ห้องปฏิบัติการสามารถทำได้โดยการใช้วิธีที่เป็นที่ยอมรับและปฏิบัติตามขั้นตอนที่ระบุในวิธีนั้น ๆ อย่างเคร่งครัดซึ่งอาจขึ้นอยู่กับตัวแปรต่าง ๆ เช่น เวลาที่ใช้ในการสกัด ขนาดของสารตัวอย่าง เป็นต้น และตัวแปรที่มีผลกระทบต่อกระบวนการทดสอบดังกล่าวต้องพิสูจน์ได้ว่าสามารถสอบกลับไปยังค่าอ้างอิงได้ ค่าความไม่แน่นอนของผลการวัดได้จากค่าความไม่แน่นอนที่เกิดจากตัวแปร และความไม่ชัดเจนของเกณฑ์กำหนดของตัวแปร
- กรณีที่มีการปรับเปลี่ยนวิธีการที่ยอมรับ ห้องปฏิบัติการต้องทำการเปรียบเทียบผลการวัดกับวิธีที่เป็นที่ยอมรับ เพื่อแสดงความสอบกลับได้ของผลการวัด

การแปลผลใบรับรอง
วัสดุอ้างอิง/วัสดุอ้างอิงรับรอง

NIMT

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Australian Government

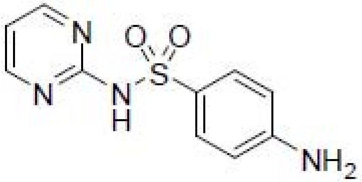
National Measurement Institute

NATA ACCREDITED FOR TECHNICAL COMPETENCE

CERTIFIED REFERENCE MATERIAL CERTIFICATE OF ANALYSIS

Report ID: M317.2016.01

Compound Name: **Sulfadiazine**
Collection Number: M317
Chemical Formula: $C_{10}H_{10}N_4O_2S$
CAS Number: 68-35-9
Structure:



Description: Off white solid
Batch Number: 16-AV-01
Molecular Weight: 250.3
Release Date: 28th June 2016

Other names: *N*[1]-2(1*H*)-Pyrimidinylidene-sulfanilamide, *N*[1]-2-pyrimidinyl-sulfanilamide, 4-Amino-*N*-2-pyrimidinylbenzenesulfonamide, 2-(4-Aminobenzenesulfonamido)pyrimidine, 2-(4-Aminobenzene-sulfonylamino)pyrimidine, 2-(p-Aminobenzenesulfonamido)pyrimidin, 2-Sulfanilamidopyrimidine, 2-Sulfanilylaminopyrimidine, 4-Amino-*N*-(2-pyrimidyl)benzenesulfonamide, 4-[[(Pyrimidin-2-yl)amino]sulfonyl]aniline, A 306, Adiazin, Adiazine, Coco-Diazine, Cremodiazine, Debenal, Deltazina, Di-Azo-Mul, Diazin, Diazolone, Diazovit, Diazyl, Eskadiazine, Honey diazine, Lipo-Diazine, Lipo-Levazine, Liquadiazine, Microsulfon, *N*[1]-2-Pyrimidinylsulfanilamide, *N*[1]-2-Pyrimidylsulfanilamide, NSC 35600, Neazine, Piridisir, Pirimal, Pyrimal, RP 2616, SDA, SN 112, Sanodiazine, Spofadrizine, Sterazine, Sulfadiazin, Sulfadiazina Reig Jofre, Sulfapirimidin, Sulfapyrimidine, Sulfazin, Sulfazin, Sulfazine, Sulfolex, Sulphadiazine, Sulphadiazine E, Theradiazine

Purity (mass fraction): 99.7 ± 0.4% (95% coverage interval)

Homogeneity assessment
The homogeneity of the material was assessed using purity assay by HPLC with UV detection on nine randomly selected 1-2 mg sub samples of the material. The material was judged to be sufficiently homogeneous at this level of sampling as the variation in analysis results between samples was not significantly different at a 95% confidence level from that observed on repeat analysis of the same sample.

Metrological Traceability
The certified purity value is traceable to the SI unit for mass (kg) through Australian national standards via balance calibration. The purity was derived by subtraction of the mass of impurities from the mass of the reference material. Organic purity is traceable to the SI-derived coherent unit one through chromatographic separation and response factor determination of individual components. Volatile and non-volatile residue content is directly traceable to mass through use of Karl Fischer and thermogravimetric analysis. Quantitative NMR provides an independent direct measure of the mass fraction of the analyte of interest, calibrated with an internal standard certified for purity (mass fraction).

Recommended storage
When not in use, this material should be stored at or below 25 °C in a closed container in a dry, dark area.

Intended Use
For *in vitro* laboratory analysis only.

Caution
Treat as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact, ingestion or inhalation of dust.

Legal notice
Neither NMI nor any person acting on NMI's behalf assumes any liability with respect to the use of, or for damages resulting from the use of, this reference material or the information contained in this certificate.

Authorised by:

S. R. Davies

Dr Stephen R. Davies,
Team Leader,
Chemical Reference Materials, NMI.
Dated: 28 June, 2016.

National Institute of Metrology (Thailand)
Ministry of Science and Technology

NIMT

TRM Thailand Reference Material

Certificate of Analysis

Elements in Acrylonitrile Butadiene Styrene (ABS) Plastic (High Levels)

TRM CODE : TRM-M-4002 **Lot Number : 010914**

This Thailand Reference Material (TRM) was produced based on NIMT's quality system in compliance with ISO GUIDE 34, for use in assessment for method performance, i.e. for checking accuracy of analytical results. As any reference material, it can also be used for control charts or validation studies. The certified values for 4 elements are given in the following table. They are expressed in mass fractions. Their expanded uncertainties were estimated using coverage factor k=2, corresponding to an estimated confidence interval of approximately 95 %.

Elements in Acrylonitrile Butadiene Styrene (ABS) Plastic (High levels)		
Parameter	Mass fraction (mg/kg)	Expanded uncertainty (mg/kg)
Cadmium (Cd)	102	4
Chromium (Cr)	172	7
Lead (Pb)	837	36
Mercury (Hg)	1025	61

Determination of Certified Values

The certified values for Cd, Cr, Pb and Hg are based on the results of an exact-matching double Isotope Dilution Mass Spectrometric (IDMS) technique.

Metrological Traceability

The certified values were metrologically traceable to the established to the SI units with NIST SRM standard solutions. This was carried out through the use of gravimetric preparation with calibrated balances and that of a primary ratio measurement procedure, an exact-matching double IDMS for Cd, Cr, Pb and Hg.

Example of metrological traceability chains of measurement results

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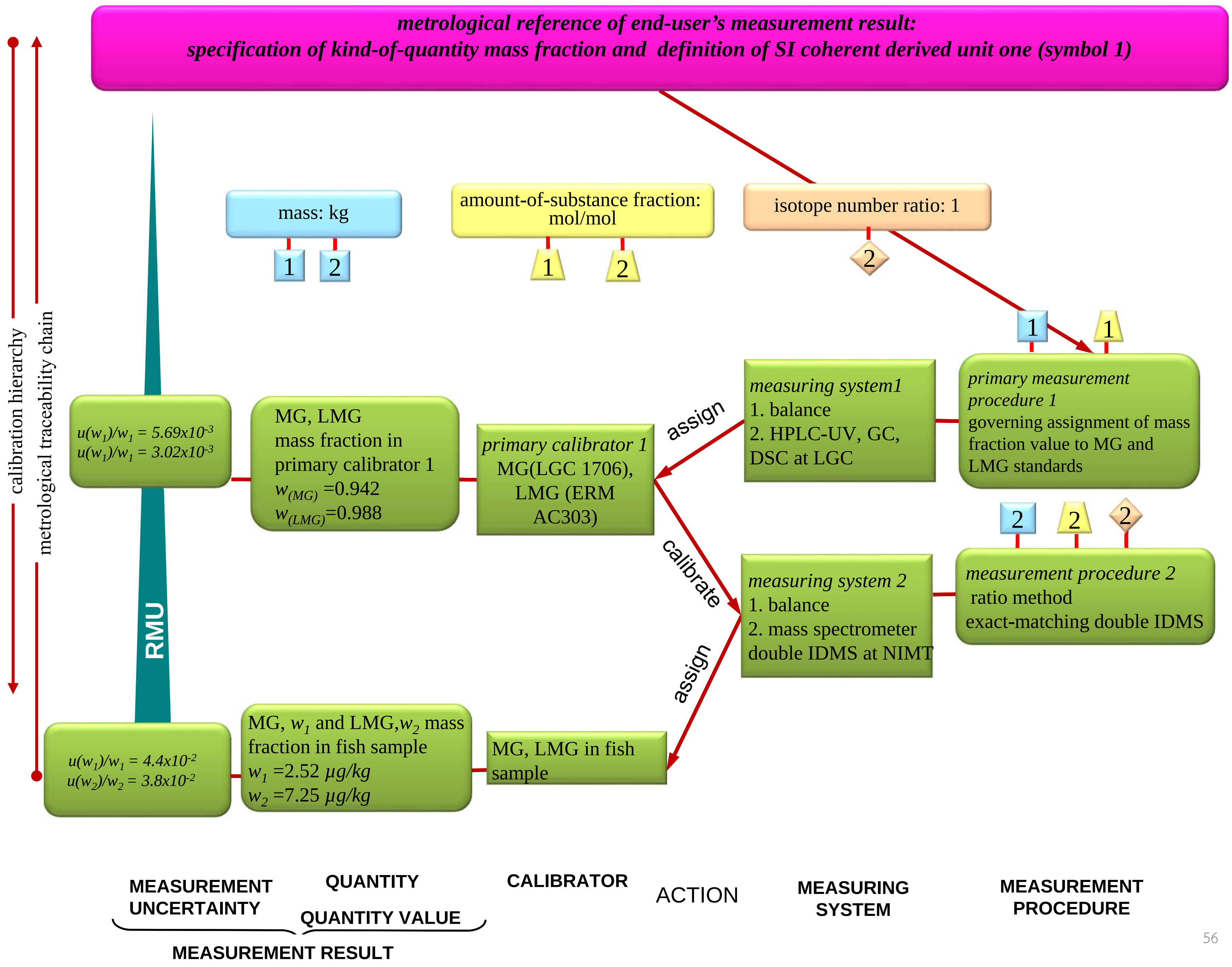
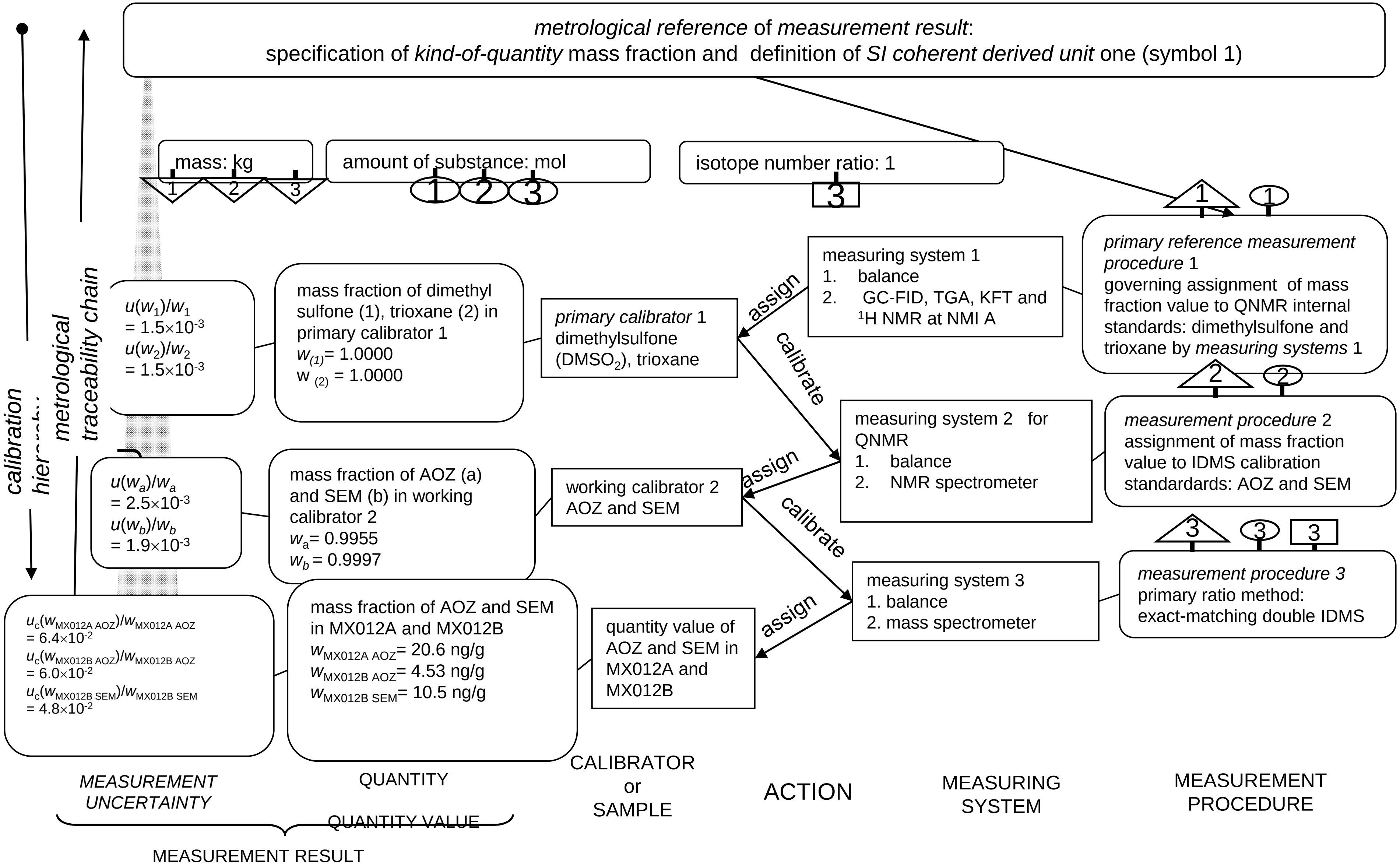

metrological reference of end-user's measurement result:
specification of kind-of-quantity mass fraction and definition of SI coherent derived unit one (symbol 1)
mass: kg
amount-of-substance fraction: mol/mol
isotope number ratio: 1
calibration hierarchy
metrological traceability chain
RMU
u(w1)/w1 = 5.69x10-3
u(w1)/w1 = 3.02x10-3
MG, LMG
mass fraction in
primary calibrator 1
w(MG) =0.942
w(LMG)=0.988
primary calibrator 1
MG(LGC 1706),
LMG (ERM
AC303)
assign
calibrate
assign
measuring system1
1. balance
2. HPLC-UV, GC,
DSC at LGC
primary measurement procedure 1
governing assignment of mass fraction value to MG and LMG standards
measuring system 2
1. balance
2. mass spectrometer
double IDMS at NIMT
measurement procedure 2
ratio method
exact-matching double IDMS
u(w1)/w1 = 4.4x10-2
u(w2)/w2 = 3.8x10-2
MG, w1 and LMG,w2 mass fraction in fish sample
w1 =2.52 µg/kg
w2 =7.25 µg/kg
MG, LMG in fish sample
MEASUREMENT UNCERTAINTY
QUANTITY
QUANTITY VALUE
MEASUREMENT RESULT
CALIBRATOR
ACTION
MEASURING SYSTEM
MEASUREMENT PROCEDURE


metrological reference of measurement result:
specification of kind-of-quantity mass fraction and definition of SI coherent derived unit one (symbol 1)
mass: kg
amount of substance: mol
isotope number ratio: 1
calibration hierarchy
metrological traceability chain
u(w1)/w1 = 1.5×10-3
u(w2)/w2 = 1.5×10-3
mass fraction of dimethyl sulfone (1), trioxane (2) in primary calibrator 1
w(1)= 1.0000
w (2) = 1.0000
primary calibrator 1
dimethylsulfone (DMSO2), trioxane
assign
calibrate
measuring system 1
1. balance
2. GC-FID, TGA, KFT and 1H NMR at NMI A
primary reference measurement procedure 1
governing assignment of mass fraction value to QNMR internal standards: dimethylsulfone and trioxane by measuring systems 1
u(wa)/wa = 2.5×10-3
u(wb)/wb = 1.9×10-3
mass fraction of AOZ (a) and SEM (b) in working calibrator 2
wa= 0.9955
wb = 0.9997
working calibrator 2
AOZ and SEM
measuring system 2 for QNMR
1. balance
2. NMR spectrometer
measurement procedure 2
assignment of mass fraction value to IDMS calibration standardards: AOZ and SEM
uc(wMX012A AOZ)/wMX012A AOZ = 6.4×10-2
uc(wMX012B AOZ)/wMX012B AOZ = 6.0×10-2
uc(wMX012B SEM)/wMX012B SEM = 4.8×10-2
mass fraction of AOZ and SEM in MX012A and MX012B
wMX012A AOZ= 20.6 ng/g
wMX012B AOZ= 4.53 ng/g
wMX012B SEM= 10.5 ng/g
quantity value of AOZ and SEM in MX012A and MX012B
measuring system 3
1. balance
2. mass spectrometer
measurement procedure 3
primary ratio method: exact-matching double IDMS
MEASUREMENT UNCERTAINTY
QUANTITY
QUANTITY VALUE
MEASUREMENT RESULT
CALIBRATOR or SAMPLE
ACTION
MEASURING SYSTEM
MEASUREMENT PROCEDURE

Conclusions

- All results are traceable – but you might not know to what
- Use of properly certified reference materials for calibration is at the heart of metrological traceability
- Look out for changes in the SI (kg, A, K, mol)

UNSW
THE UNIVERSITY OF NEW SOUTH WALES

Ref: B. Hibbert


Thank you for your attention