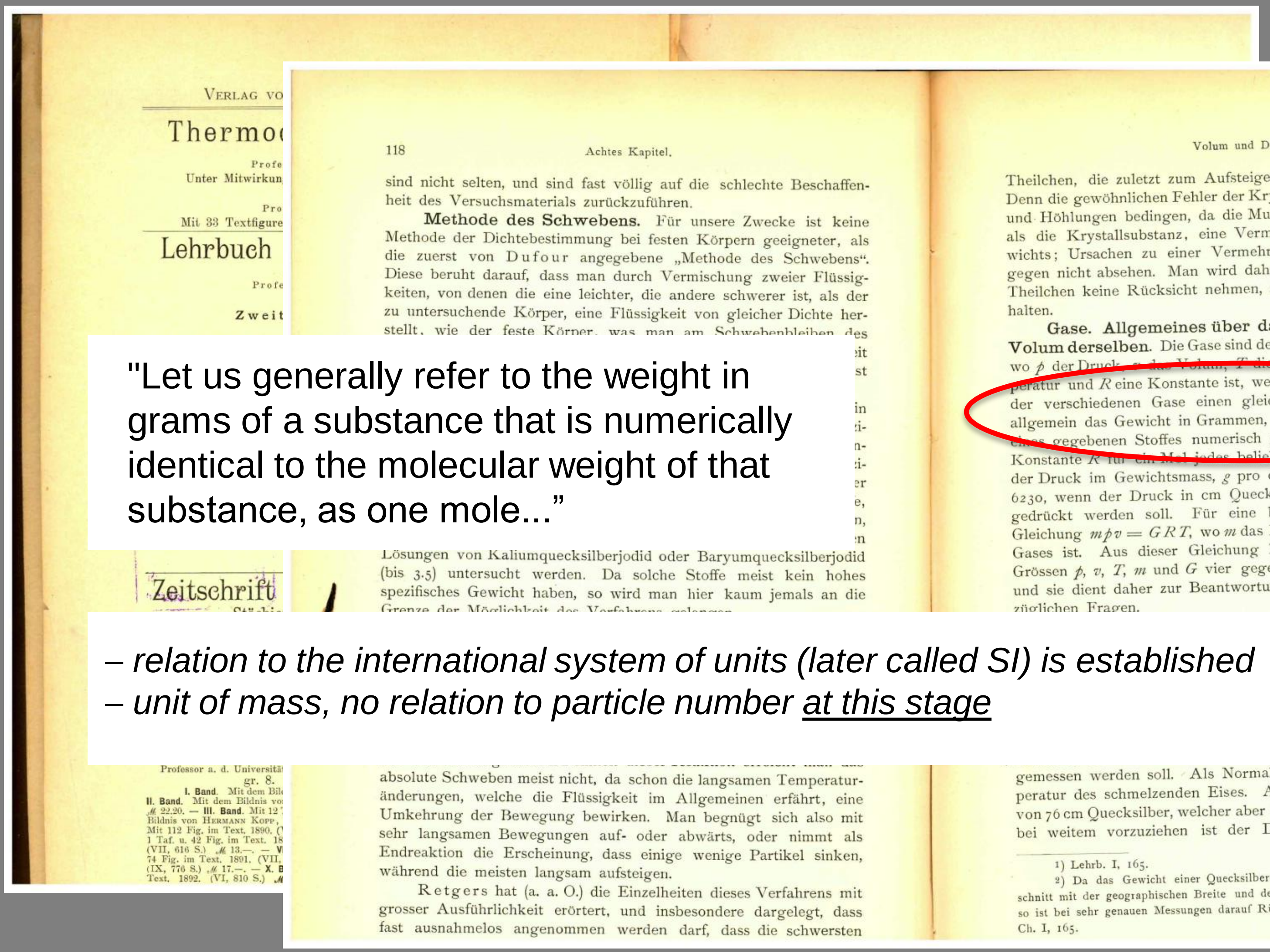
Status of the redefinition of the mole

Dr. Bernd Güttler
Physikalisch-Technische
Bundesanstalt (PTB)
Germany

CCQM
WG on the mole

Symposium at 250^{th} ACS National Meeting, Boston, MA, August 19, 2015

Definition of the mole


Theilchen, die zuletzt zum Aufsteigen ...
Denn die gewöhnlichen Fehler der Kry...
und Höhlungen bedingen, da die Mut...
als die Krystallsubstanz, eine Vermi...
wichts; Ursachen zu einer Vermehru...
gegen nicht absehen. Man wird dahe...
Theilchen keine Rücksicht nehmen, s...
halten.

Gase. Allgemeines über d... Volum derselben. Die Gase sind de... wo p der Druck, ... das Volum, T die ... peratur und R eine Konstante ist, wel... der verschiedenen Gase einen gleichen Werth hat. Nennen wir allgemein das Gewicht in Grammen, welches dem Molekulargewicht eines gegebenen Stoffes numerisch gleich ist, ein Mol, so ist die Konstante R für ein Mol jedes beliebigen Gases gleich 84720, wenn der Druck im Gewichtsmass, g pro cm, gemessen wird [1]), und gleich 6230, wenn der Druck in cm Quecksilberhöhe, auf 0° reduzirt, ausgedrückt werden soll. Für eine beliebige Gasmenge G gilt die Gleichung $mpv = GRT$, wo m das Molekulargewicht des fraglichen Gases ist. Aus dieser Gleichung lässt sich, wenn von den fünf Grössen p, v, T, m und G vier gegeben sind, die fünfte berechnen, und sie dient daher zur Beantwortung aller auf diese Grössen bezüglichen Fragen.

"Let us generally refer to the weight in grams of a substance that is numerically identical to the molecular weight of that substance, as one mole..."

– *relation to the international system of units (later called SI) is established*
– *unit of mass, no relation to particle number* ***at this stage***

Definition of the mole

"Let us generally refer to the weight in grams of a substance that is numerically identical to the molecular weight of that substance, as one mole..."

The mole, mol, is the unit of amount of substance of a specified elementary entity, which may be an atom, molecule, ion, electron, any other particle or a specified group of such particles; its magnitude is set by fixing the numerical value of the Avogadro constant to be equal to exactly 6.022 141X·10^{23} when it is expressed in the unit mol^{-1}.

Ostwald 1893

?

proposed definition

Definition of the mole

"Let us generally refer to the weight in grams of a substance that is numerically identical to the molecular weight of that substance, as one mole..."

Ostwald 1893

$$M(\mathrm{X}) = A_r(\mathrm{X}) \times M_u$$

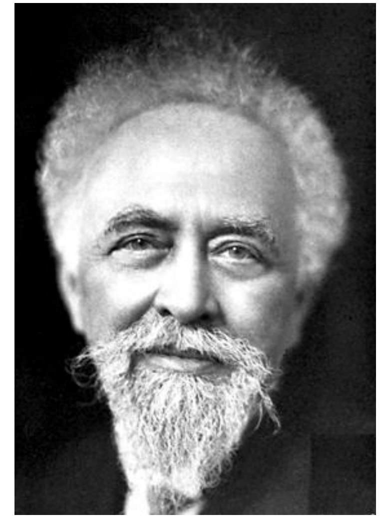
$$n_x = M_\mathrm{x}/M(\mathrm{X})$$

$M(\mathrm{X})$	molar mass of X
$\mathrm{A_r}(\mathrm{X})$	Σ rel. atomic masses (dimension less) of X
M_u	molar mass constant (10^{-3} kg/mol)
M_x	mass of X

Molecules per mole: Avogadro's Constant

$$N_A = (1/\langle x^2 \rangle)(RT/3\pi\eta r)$$

η viscosity
r particle radius
x average displacement



Albert Einstein

Jean Perrin

"Any two gram-molecules always contain the same number of molecules. This invariable number N is a universal constant which may appropriatly be designated Avogadro's constant."

Perrin. J.: Brownian Motion and Molecular Reality
aus: Annales de Chimie et de Physique 18, 1-114 (1909)

dual interpretation and base quantity "Stoffmenge":

8. Mit den Bezeichnungen „Mol“ und „Äquivalent“ verbundene Begriffe und Einheiten

Ulrich Stille

"First of all it (the mole) is understood as a chemical mass unit in accordance with **Ostwald's view of a continuum** and has an individual value for each type of molecule.

The other understanding of the "**mole**" is that of **a number of atoms or molecules** that is comprised in a mole.“

When you intend to find a more precise wording, for example by introducing a base quantity **"Stoffmenge"** (amount of substance) it can be considered as the numerical value of the amount of substance."

Stille U.: Messen und Rechnen in der Physik, Vieweg & Sohn S. 117f. (1955)

Definition of the mole

quantification

"Let us generally refer to the weight in grams of a substance that is numerically identical to the molecular weight of that substance, as one mole..."

1. The mole is the amount of substance of a system which contains as many elementary entities as there are atoms in 0.012 kilogram of carbon 12; its symbol is "mol".

identification

2. When the mole is used, the elementary entities must be specified and may be atoms, molecules, ions, electrons, other particles, or specified groups of such particles.

N_A

Ostwald 1893

current definition (1971)

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current definition (1971)

$$M(\mathrm{X}) = A_r(\mathrm{X}) \times M_u$$

$$M(\mathrm{X}) = m(\mathrm{X}) \times N_A$$

$m(\mathrm{X})$	$A_\mathrm{r}(\mathrm{X})\, m_\mathrm{u}$
m_u	$m(^{12}\mathrm{C})/12$
N_A	Avogadro constant

now: $u(M_\mathrm{u}) = 0$

$u(N_\mathrm{A}) = 2 \times 10^{-8}$

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current definition (1971)

- In the new SI all 7 base units will be defined by constants of nature (SI reference constants),
- the respective SI reference constants will have exact numerical values,
- the new SI is expected to be more stable since there are no artefacts involved.

demands for redefinition

Definition of the mole

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proposed definition

no artefacts!

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proposed definition

$$M(\mathrm{X}) = A_r(\mathrm{X}) \times M_u$$

$$M(\mathrm{X}) = m(\mathrm{X}) \times N_A$$

$$n_x = M_\mathrm{x}/M(\mathrm{X})$$

$$n_\mathrm{x} = N_\mathrm{x}/N_\mathrm{A}$$

$N(\mathrm{X})$ number of particles X

then: $u(M_\mathrm{u}) \leq 7 \times 10^{-10}$

$\boldsymbol{u(N_\mathrm{A}) = 0}$

mise en pratique of the mole

A *mise en pratique* for the definition of a unit is a set of instructions that allows the definition to be realized in practice at the highest level. The *mise en pratique* should describe the primary realizations based on top-level primary methods.

CCU would like to see some homogeneity in their content.

(report of the 97th meeting of the CIPM (2008)).

mise en pratique of the mole

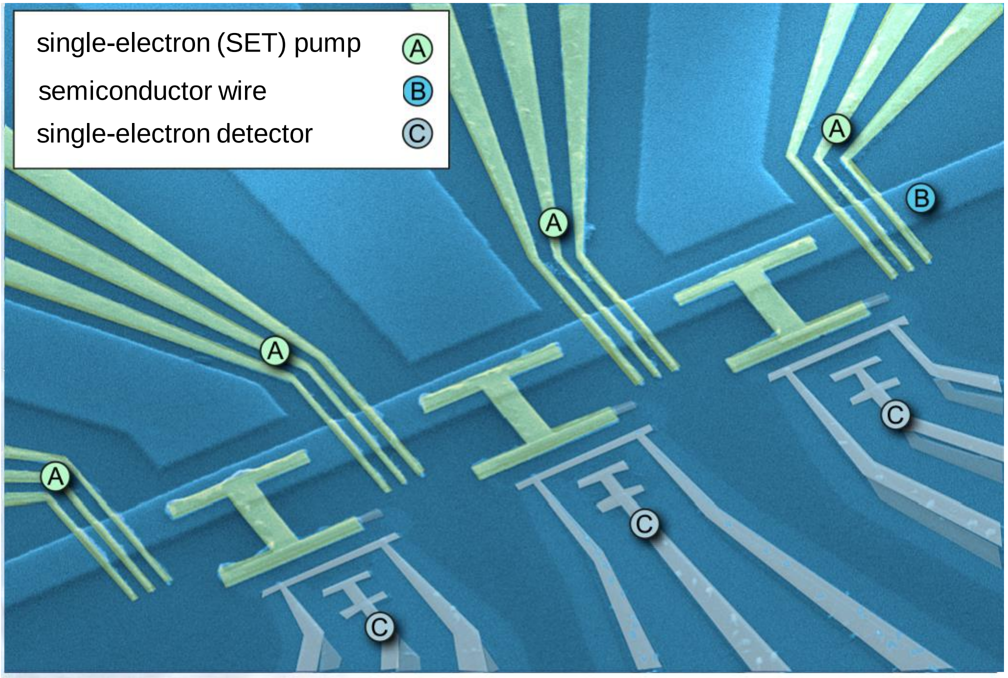
The mole can be realized by counting N_A electrons in a conductor line with a SET device.

$$n_{el} = \langle N_{el} \rangle / N_{\mathrm{A}}$$

Identification:
elementary entities are electrons

Quantification:
by sequential counting

other units involved:
none



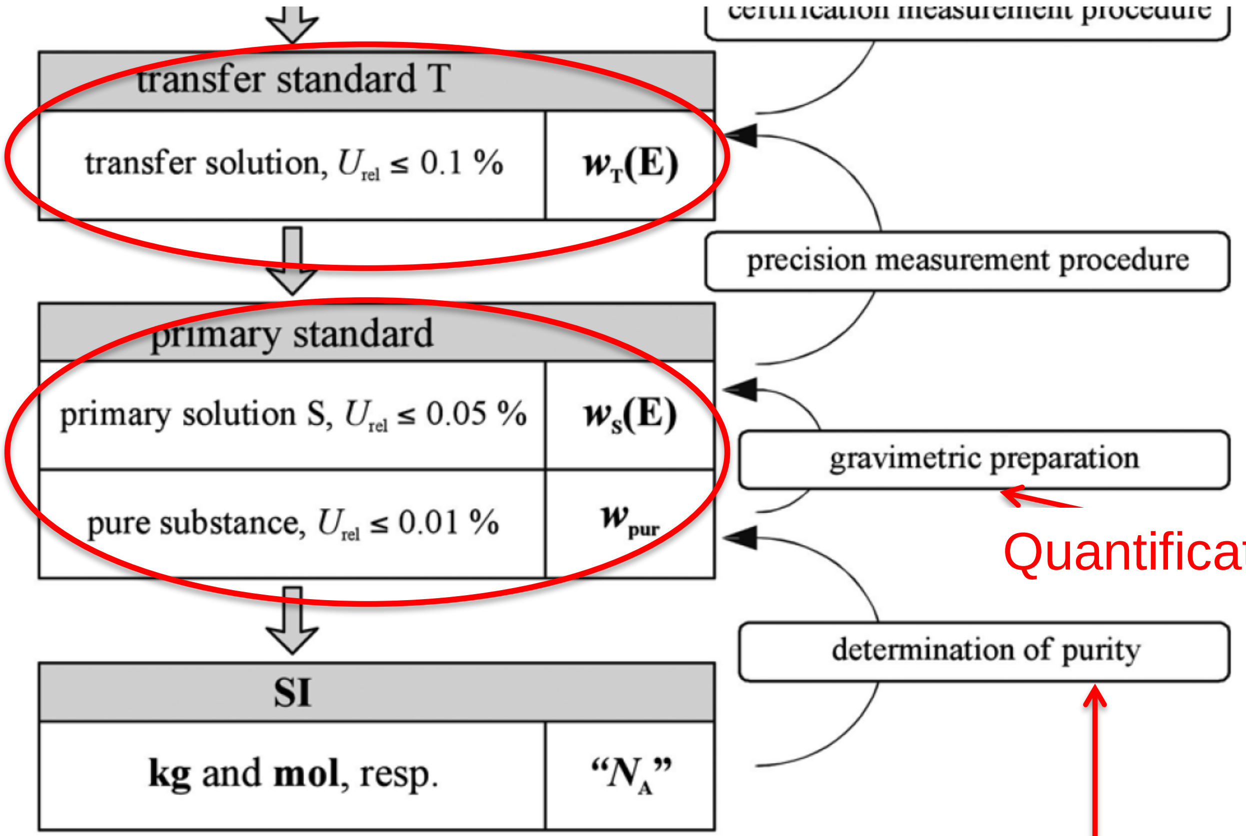
A semiconductor single-electron-tunneling (SET) pump.

$$I = \langle N_{el} \rangle ef$$

S. P. Giblin et al. Nature Communications 3.930
DOI 10.1038 ncomms 1935
from: U. Siegner, PTB

mise en pratique of the mole

Traceability



primary level

transfer standard

$$c_x = \frac{n_x}{V_{sol}}$$

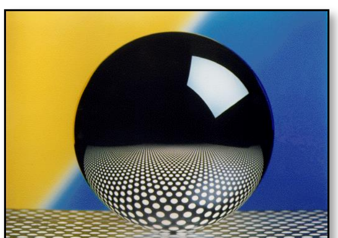
primary standard

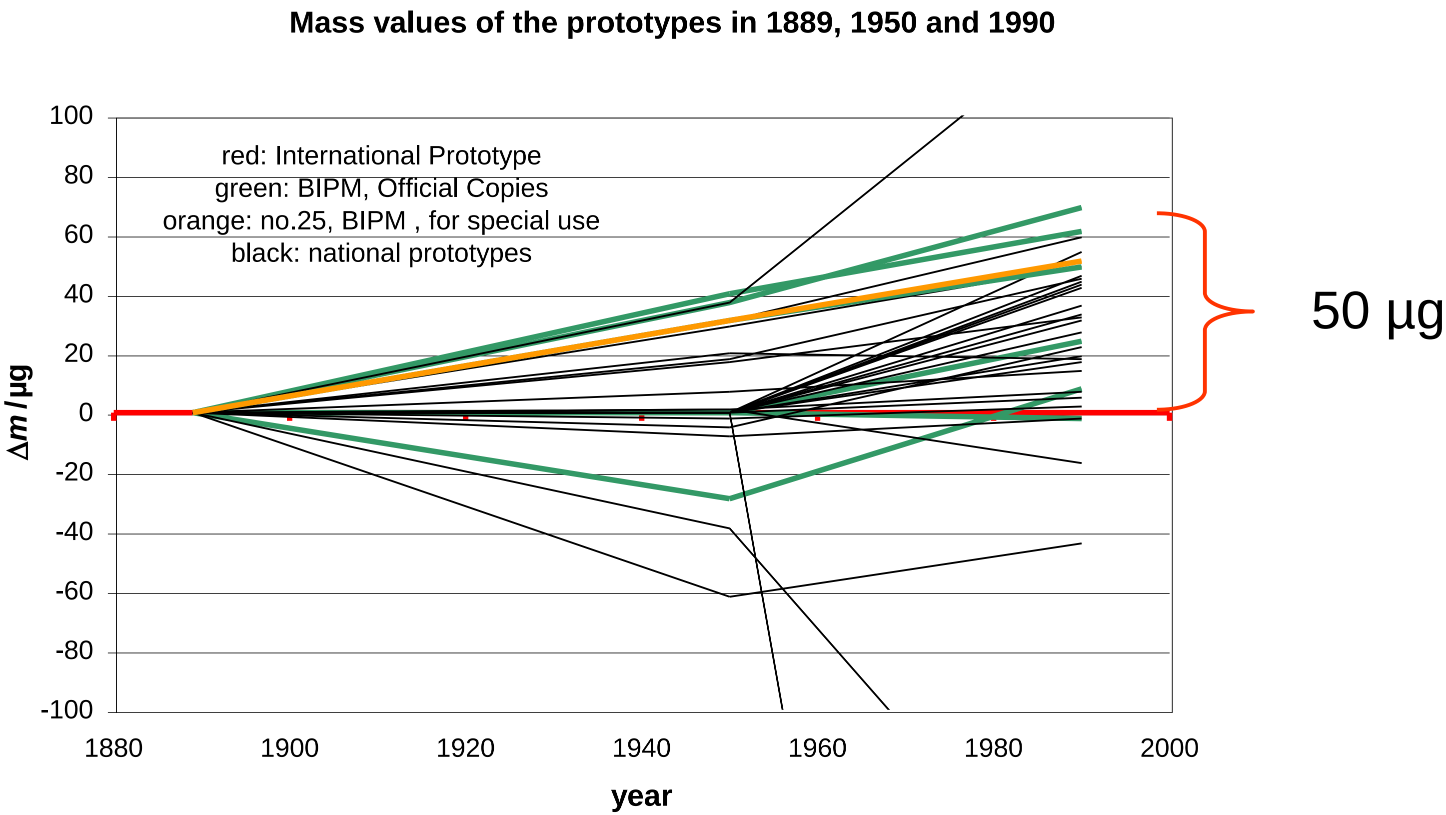
Quantification

$$n_x = \frac{M_X}{M(X)} R(X)$$

Identification

the kilogram artefact problem

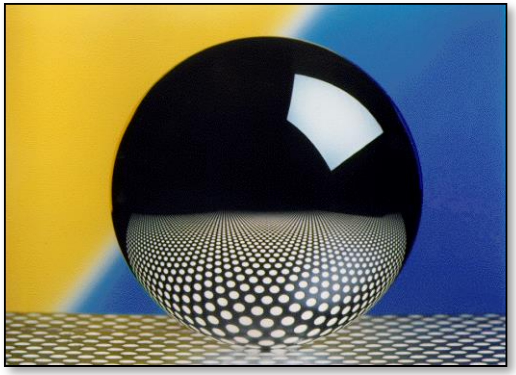


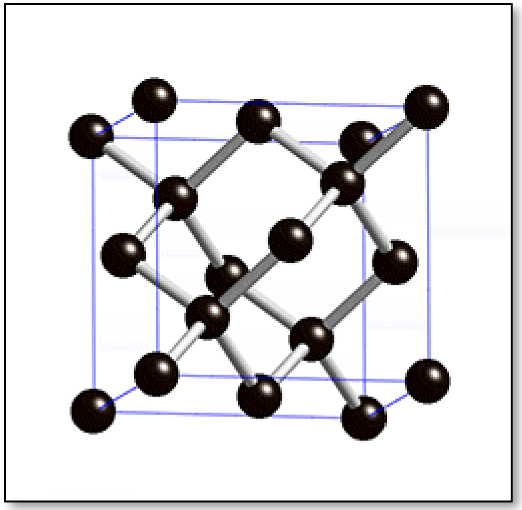


$$N_A = \frac{8 \cdot M(Si) \cdot V_{\text{sphere}}}{M_{Si} a^3}$$

International Avogadro Cooperation (IAC)

Avogadro's constant via Bragg's relationship





$$N_{Si} = V_{\mathrm{Sphere}} / V_{\mathrm{Atom}}$$

$$n_{Si} = N_{Si}/N_{\mathrm{A}} = M_{Si}/M(Si)$$

$$N_{\mathrm{A}} = (M(Si)/M_{Si})\,(V_{\mathrm{Sphere}} / V_{\mathrm{Atom}})$$

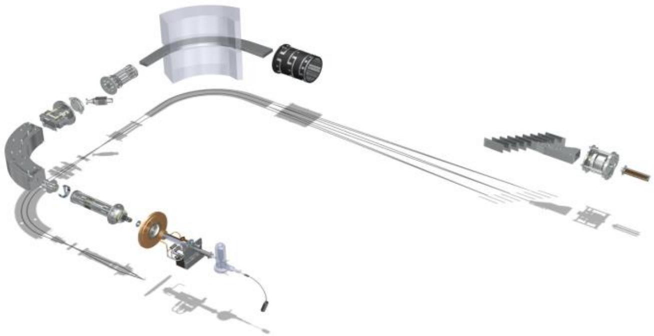
$$N_{\mathrm{A}} = \frac{8 \cdot M(Si) \cdot V_{\mathrm{sphere}}}{M_{Si} \cdot a^3}$$

$$N_A h = \frac{c A_r(e) M_u \alpha^2}{2 R_\infty}$$

http://www.msm.cam.ac.uk/phase-trans/2003/MP1.crystals/MP1.crystals.html

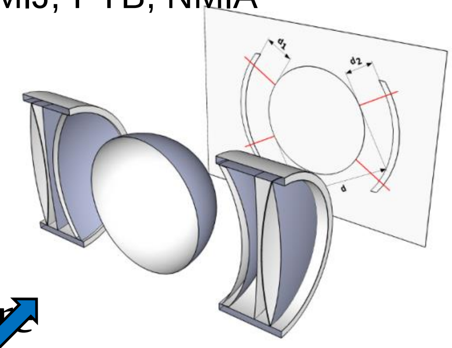
see J. Stenger & E.O. Göbel, Metrologia 49 (2012), L25–L27

International Avogadro Coordination



Multicollector ICPMS
PTB, NMIJ, NIST

Optical sphere interferometer
NMIJ, PTB, NMIA

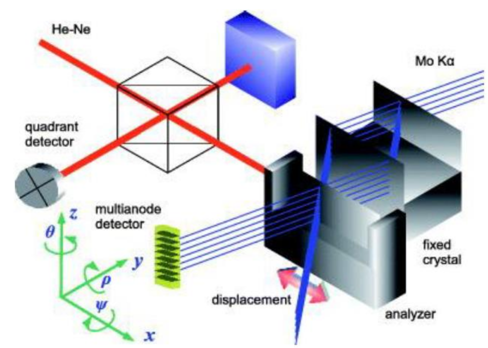


$$N_A = \frac{8 \cdot M(Si) \cdot V_{sphere}}{M_{Si} \cdot a^3}$$

Mass comparator
BIPM, NMIJ, PTB

Surface layer :	XRR, XRF, XPS, opt. ellipsometry
Impurities:	IR, NAA

NMIJ, METAS, PTB



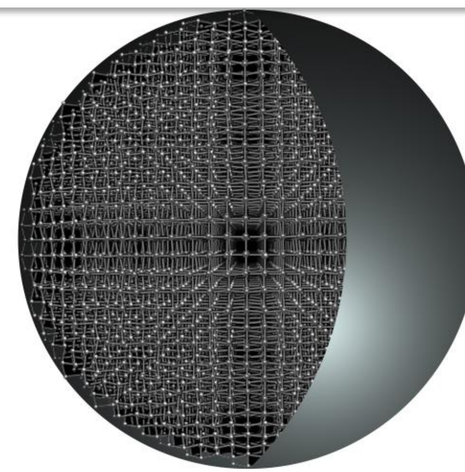
X-Ray Interferometer
INRIM, NIST

interferometric volume determination

Planet Silicon

PTB‘s sphere interferometer enables complete topographies of spheres, $n_{diameter}$ ≈ 600 000.

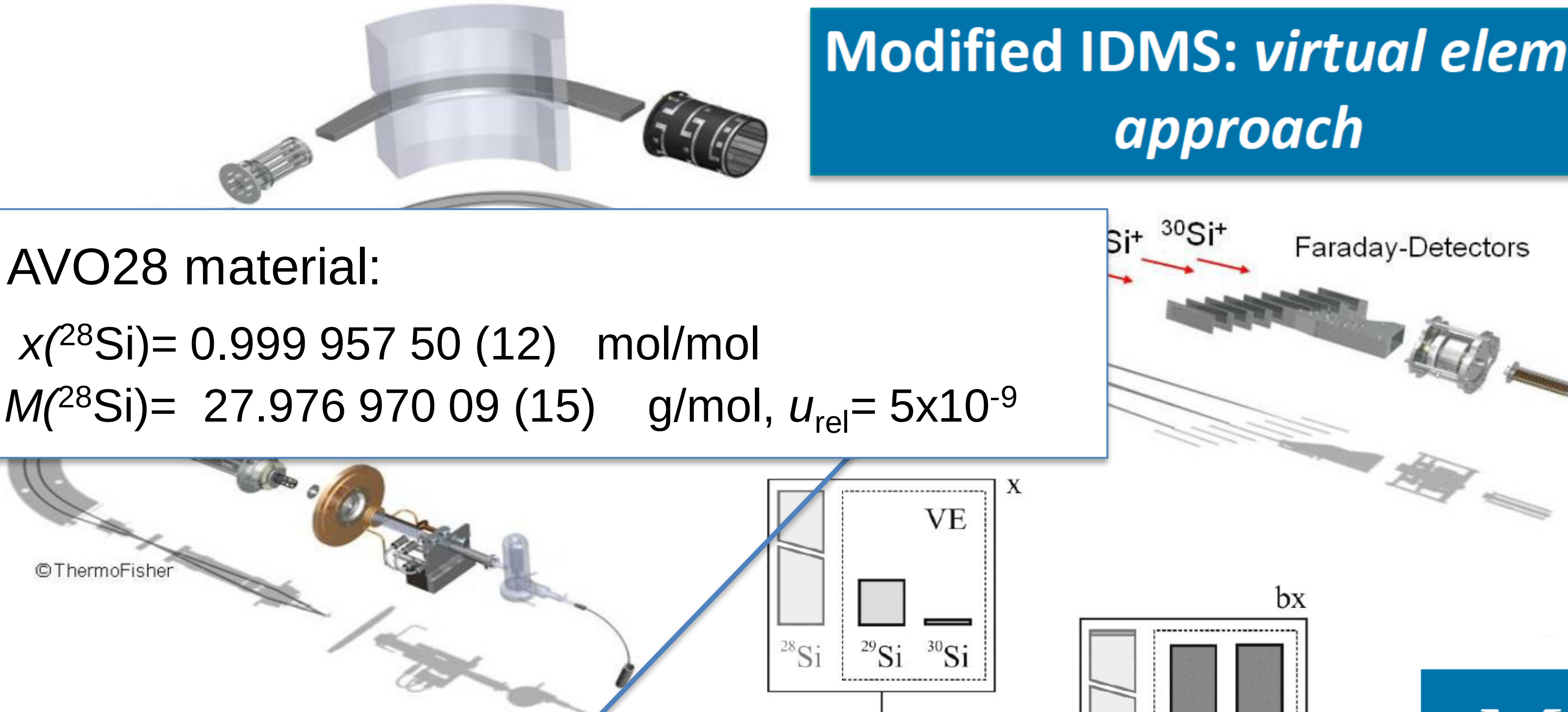
The radius uncertainty is 0.7 nm or 8 $\times 10^{-9}$



Radius topography of ^{28}Si-sphere S8. Peak to valley deviations from roundness amount to < 40 nm.

molar mass determination: the "virtual element"

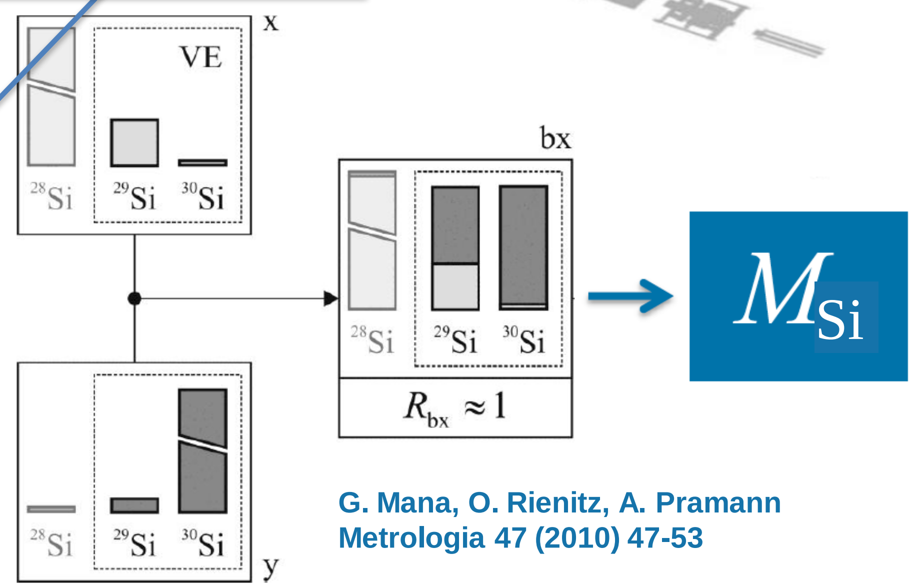
Modified IDMS: *virtual element approach*



AVO28 material:

$x(^{28}\text{Si})$= 0.999 957 50 (12) mol/mol

$M(^{28}\text{Si})$= 27.976 970 09 (15) g/mol, u_{rel}= 5x10^{-9}

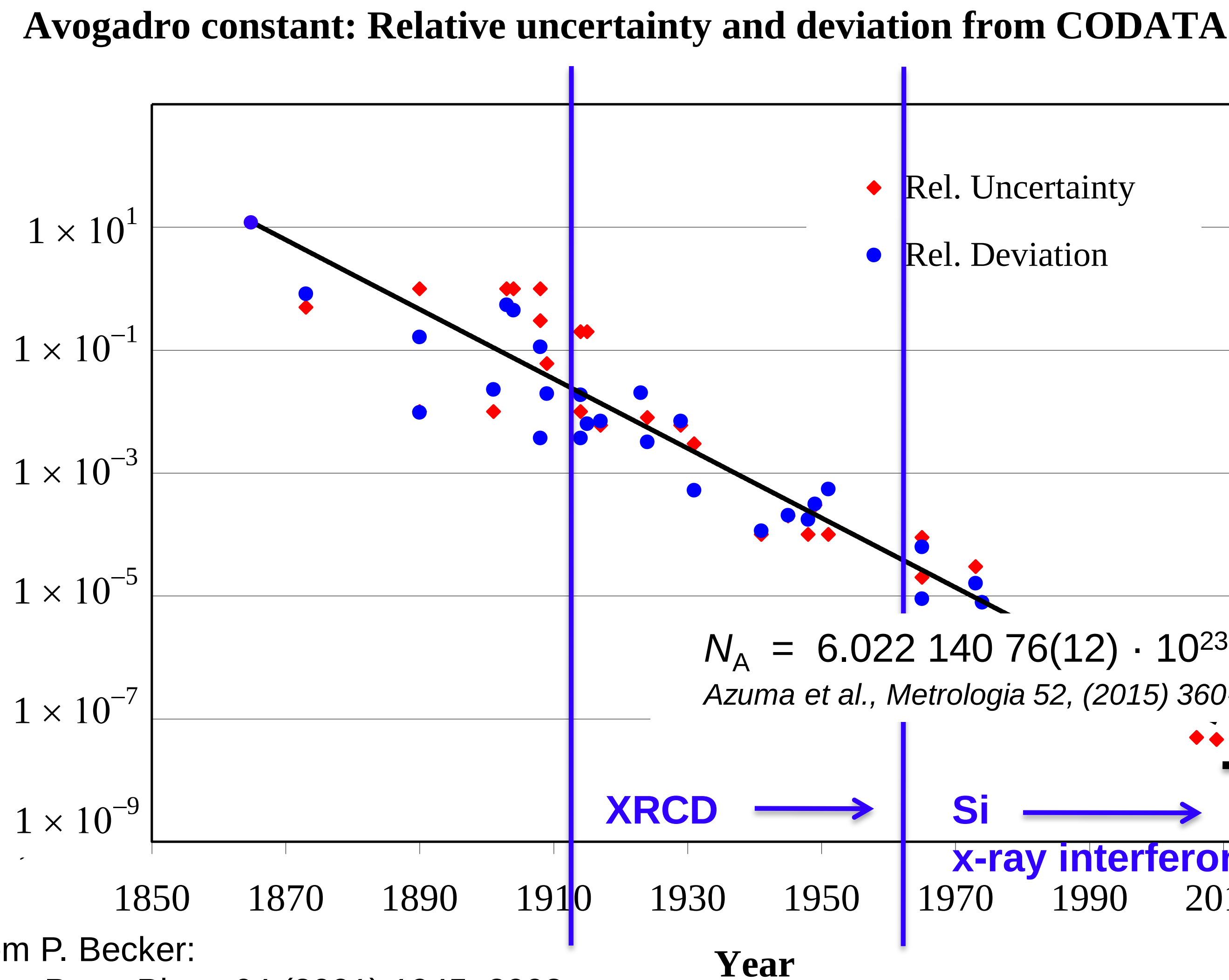


G. Mana, O. Rienitz, A. Pramann
Metrologia 47 (2010) 47-53

Si virtual element appoach

- three samples: ^{28}Si-material („*x*")
  ^{30}Si-enriched („*y*")
  IDMS-blend („*bx*")
- $R(^{30}\text{Si}/^{29}\text{Si})$ measured in *x*, *y*, *bx*

Avogadro constant: accuracy over time



Max von Laue

N_A = 6.022 140 76(12) · 10^{23} mol^{-1} u_{rel}= 2 x 10^{-8}

Azuma et al., Metrologia 52, (2015) 360-375

from P. Becker:
Rep. Prog. Phys. 64 (2001) 1945–2008

2. Realization of the definition of the mole

Currently, the most accurate realization of the definition of the mole is by the determination of the number of ^{28}Si atoms in a single crystal of Si enriched in ^{28}Si using volumetric and X-ray interferometric measurements

$$N_A = \frac{8 \cdot M(Si) \cdot V_{\text{sphere}}}{M_{Si} \cdot a(^{28}Si)^3} \quad (1)$$

$$N_{Si} = 8 V_{sphere} / a(^{28}Si)^3 \quad (2)$$

$$n_{Si} = N_{\text{Si}} / N_{\text{A}} \quad (3)$$

One mol of ^{28}Si atoms is equivalent to the number of ^{28}Si atoms that is contained in a sample of a ^{28}Si single crystal with a volume of 12.05867069 cm^3 at 20 °C and in vacuum. The relative standard uncertainty of this volume would be 2×10^{-8}. (Hypothetical!)

summary

- In the new SI, the definition of the mole will no longer be related to the element carbon and the unit of mass.
- Avogadro constant and number will be exact with no uncertainty.
- The most accurate realisation of the mole is given by the experiment that led to the definition of the Avogadro constant (and also the Planck-constant): the XRCD experiment on ^{28}Si.
- This primary realisation of the mole is independent of the quantity mass but closely linked to the realisation of the unit kilogram so that the interrelation of the units can be made easily transparent.
- The molar mass constant M_u = 1 x10^{-3} Kg mol^{-1} will no longer be exact, but will have a relative standard uncertainty of 7x10^{-10}.
- The continuity with the current definition of the mole is preserved - none of these changes will affect practical measurements in chemistry.

Thank you…

…for your interest!

…and to my colleagues

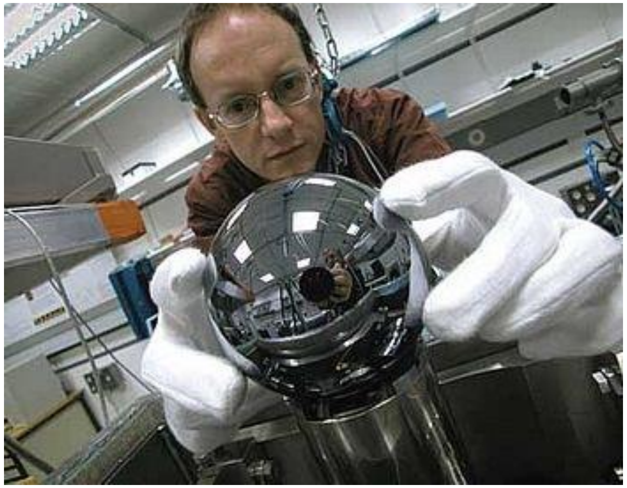
Horst Bettin
Olaf Rienitz
Axel Pramann
Detlef Schiel
Peter Becker
Michael Gläser
Michael Borys
Roman Schwartz
Joachim Ullrich
Richard Davis
Martin Milton
Robert Wielgosz
…

for their work and support.

mise en pratique of the mole

kilogram

mole



Planck-route

Avogadro-route

$$n_x = \frac{N_x}{N_A}$$

primary standard

the path to the "modern" SI-unit mole

1957 Olander and Nier suggest ^{12}C instead of oxygen as reference point for the atomic mass scale
- it replaces the Berzelius-scale (O=16) of the chemists and the $^{16}O=16$ scale of the physicists)

1961 ^{12}C is recommended by IUPAC and IUPAP as reference point

Translation of Stilles term "Stoffmenge" for the base quantity related to the mole into the English term „**amount of substance**“ by Guggenheim (Guggenheim, E.A.: The Mole and Related Quantities, J. Chem. Ed. 38 (2), S. 86 (1961))

1967 Following recommendations by IUPAP, IUPAC and ISO a definition of the mole is fixed and confirmed in 1969

1971 The 14. CGPM accepts the definition of the mole (Res 3 ; CR, S. 78 und Metrologia, **8,** (1972), p. 36)

mise en pratique: example ^{28}Si in AVO28-S5

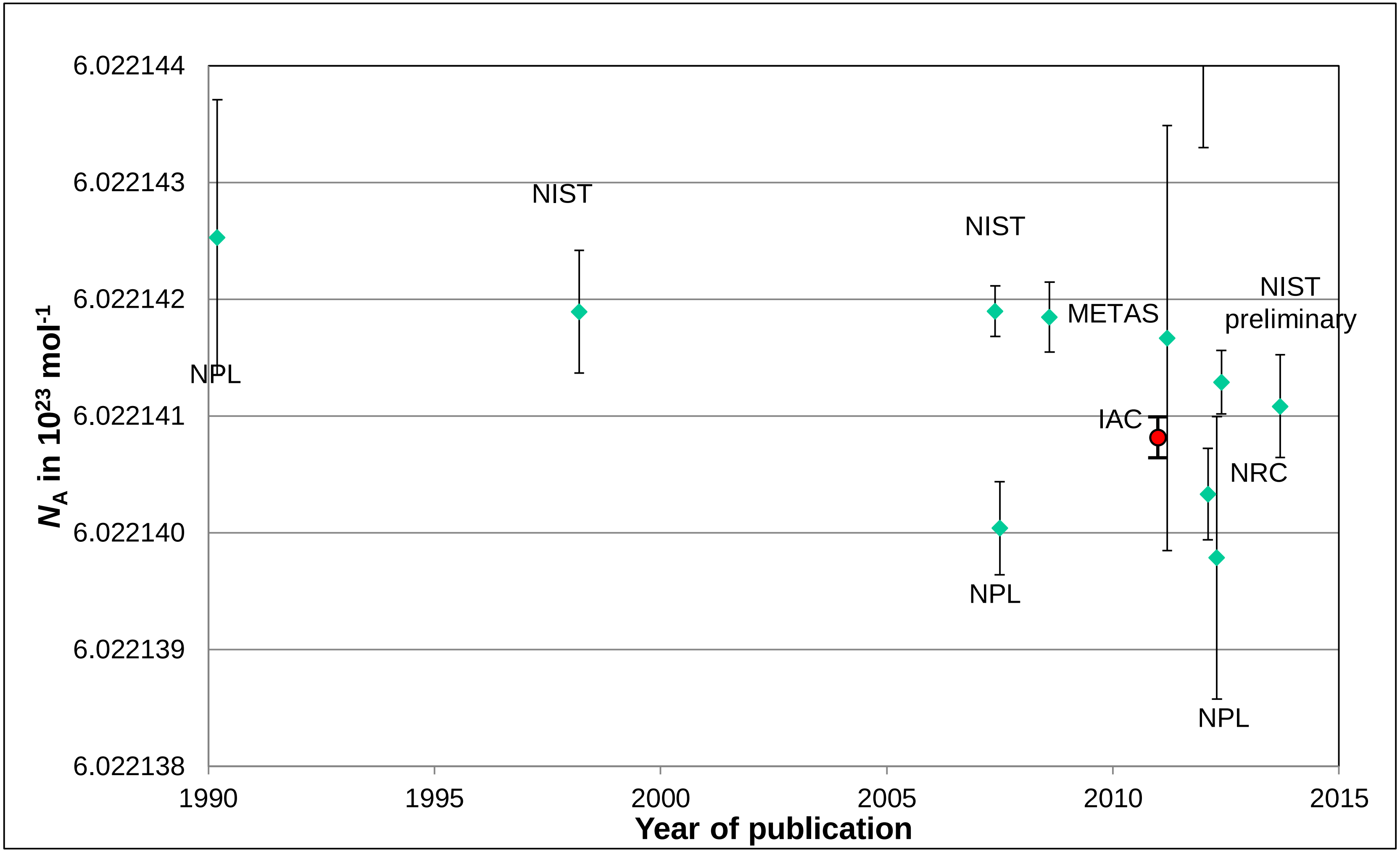
Identification step:

In case that the specified elementary entity is **^{28}Si** and the real crystal is ***not*** purely **^{28}Si** also other elementary entities (elemental impurities, i.e. C, O, B and isotope impurities, i.e. ^{29}Si, ^{30}Si) and the volume of the surface passivation layer must be considered (i.e. excluded). In the ^{28}Si enriched single crystal Si-sphere AVO28-S5 the following statements apply:

One mol of ^{28}Si atoms is equivalent to the number of ^{28}Si atoms that is contained in a sample of the AVO28-S5 single crystal sphere with a volume of 12.05918321 cm^3 at 20 °C and in vacuum.

The AVO28-S5 single crystal sphere contains 35.7452948469 mol of ^{28}Si atoms.

Present situation of N_A results



Experiments for definition N_A and h

- **Counting atoms:**

  Determination of the Avogadro constant with silicon crystal method,

  Current value: $u_{rel}(N_A) = 3 \cdot 10^{-8}$

- **Generating standard forces:**

  Determination of Planck constant with Watt balance experiment,

  Current value: $u_{rel}(N_A) \sim 6 \cdot 10^{-8}$

intended new definition of the mole

The mole, mol, is the unit of amount of substance of a specified elementary entity, which may be an atom, molecule, ion, electron, any other particle or a specified group of such particles;
its magnitude is set by fixing the numerical value of the Avogadro constant to be equal to exactly 6.022 141X·10^{23} when it is expressed in the unit mol^{-1}.

N_A defined
$U(N_A)$ = 0

↓

mol

?

⟷

h defined
$U(h)$ = 0

↓

kg

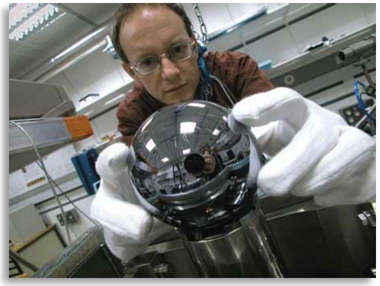


Foto: Okerlandarchiv

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N_A defined

$U(N_A) = 0$

↑

mol

$$N_A h = \frac{c A_r(e) M_u \alpha^2}{2 R_\infty}$$

h defined

$U(h) = 0$

↑

kg

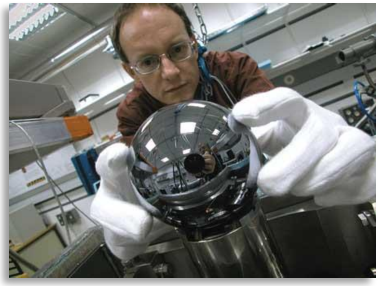


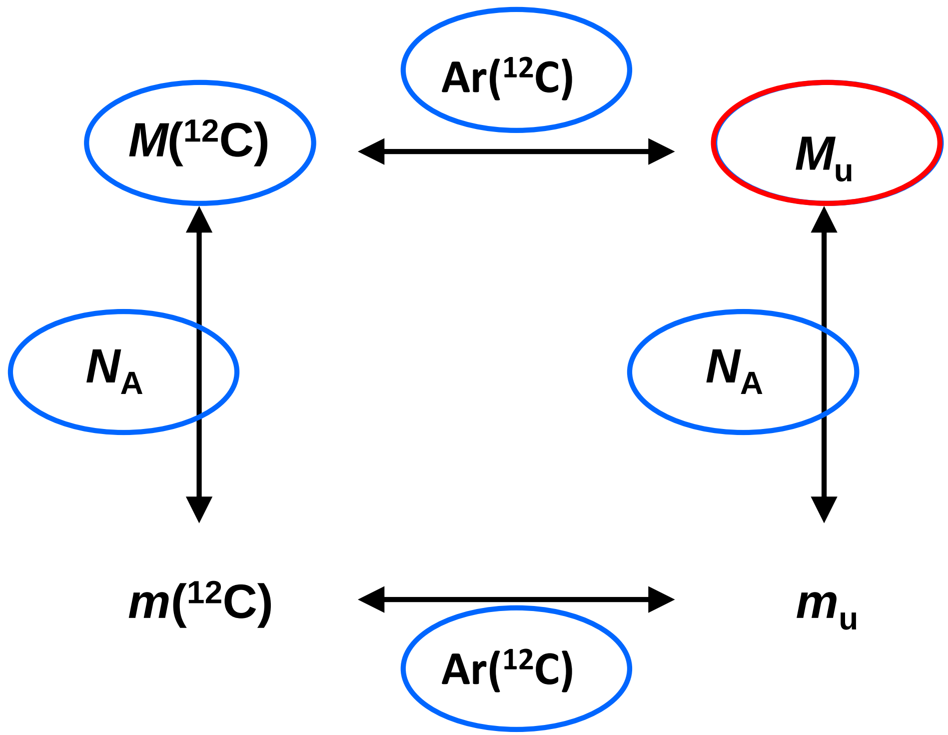
Foto: Okerlandarchiv

c light velocity, $A_r(e)$ relative mass of the electron, $M_u = (10^{-3}$ kg $mol^{-1})$, α fine structure constant, R_∞ Rydberg constant, e elementary charge

After new definition of the mole

$M(\mathrm{A}) = A_r(\mathrm{A})\, M_u$ g/mol

Macroscopic level



$m(\mathrm{A}) = A_r(\mathrm{A})\, m_u$ g/mol

Atomic level

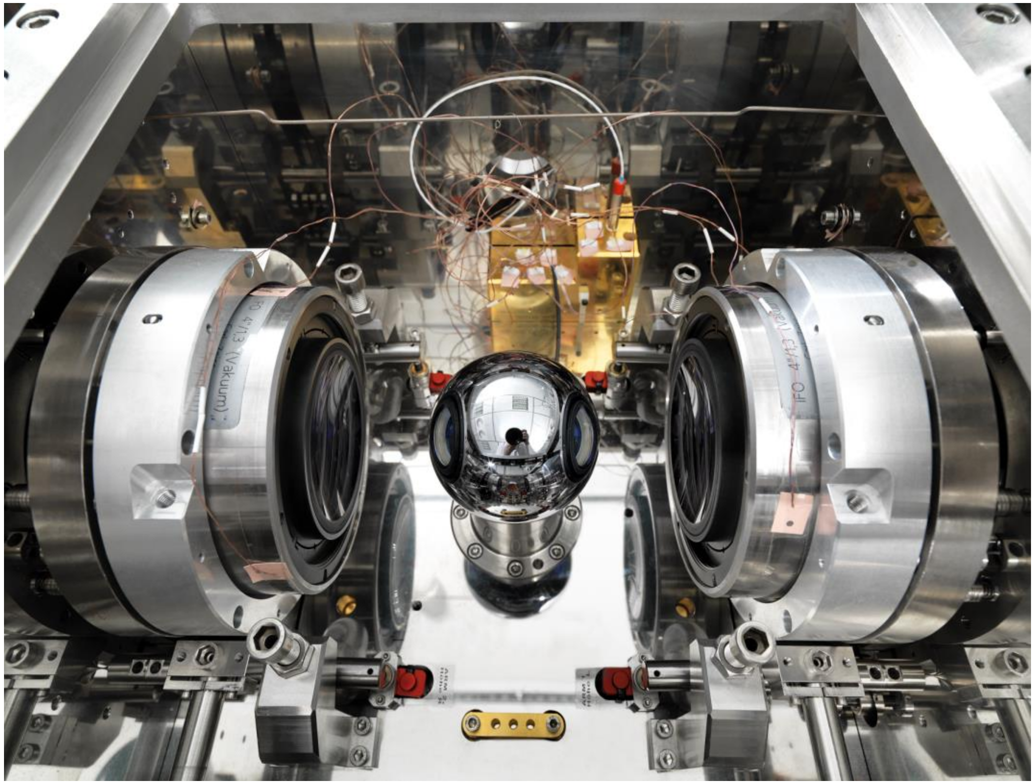
New definition:	N_A	$= 6.0221X \cdot 10^{23}$ 1/mol
	$u(N_A)$	$= 0$
Therefore:	M_u	$= 1$ g/mol
	$u(M_u)$	$\approx 1.4 \times 10^{-9}$

$$M_u = \frac{2hN_A R_\infty}{cA_r(e)\alpha^2}$$

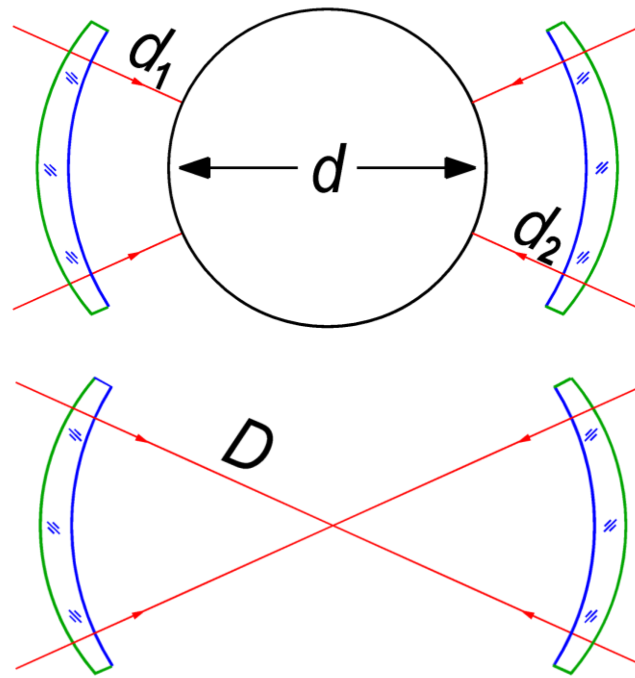
mise en pratique of the mole

- All base units of the SI now have a mise en pratique which is a document giving recommendations for the practical realization of the definition the unit at the highest level of accuracy by means of one or more primary methods.
- The mises en pratique are “living” documents that are updated to take account of new methods and technological improvements.
- The explicit-constant formulation of the definition of the unit does not imply or suggest any particular experiment to realize it. This mise en pratique is based on the state of the art of experimental knowledge ... and will be revised from time to time as new experiments are devised or existing experiments are refined.

The sphere interferometer of PTB



difference measurement



$$d(x,y) = D(x,y) - d_1(x,y) - d_2(x,y)$$

~30 different orientations of the sphere, each 60° result: 1 million diameters

- working group 5.41, Dr. Arnold Nicolaus
- Measurement of ^{28}Si-S5

Letter from Paul De Bievre (CCQM12_16)

Conclusions

1.It would be wise to interrupt the re-definition process of the mole until further in-depth discussions have led to a broad and argued opinion on the existing or arising problems, which is supported by intercontinental understanding in broader (chemical) communities than has been the case so far; it is necessary to create the necessary time for properly pursuing that goal;

2.This recommendation also includes the possibility to address the very fundamental view of acknowledged researchers that the Avogadro constant is not (at all?) a fundamental constant of nature (e.g. see again attachment 2) making the present re-definition all the more worrying; evidently, questions of such a basic nature must be sorted out in a much broader community before any final decision with far reaching consequences is made.

Letter from Gary Price (Australia) (CCQM12-17)

Just two of the many important problems are:

1. Incomprehensibility

Any system of measurement units has a primary purpose to facilitate clear communication – for all users. *The new SI is incomprehensible to the vast majority of its intended users. It is not capable of being taught at less than a specialised post-graduate level.* This must have adverse impacts on understanding and trust at all levels and lead to misunderstandings of all kinds. Educationists of the highest standing regard the new SI as literally unteachable. This alone is serious cause for concern.

2. Vulnerability to systematic error and undetectable drift in the basis of measurements.

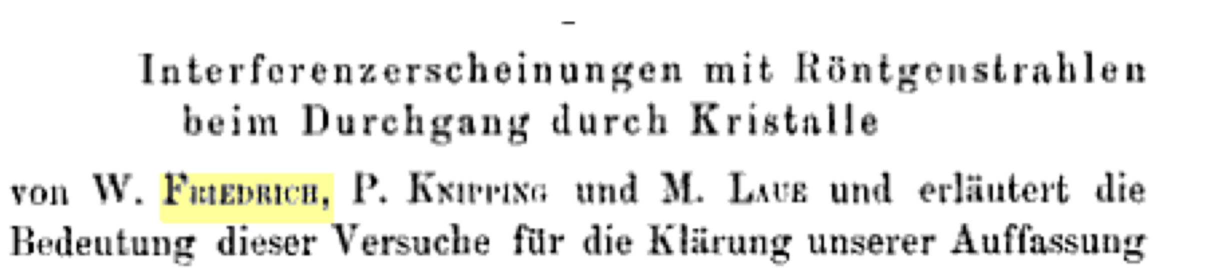
This was shown with great rigour by Franco Pavese [2] in the March AQUAL. He demonstrated the difficulties assailing any measurement system founded on fixing fundamental, inter-related and inter-dependent constants on the basis only of present accuracies and with no base units. He has since elegantly and simply proved that *the rounding errors involved in the new SI procedure must propagate and multiply alarmingly throughout all subsequent calculation and measurement, greatly exceeding best quoted uncertainties* and effectively rendering the new SI unusable in most modern computer data handling systems due to the dangers of non halting computing processes. This is in addition to the already well known problems, discussed by Foster [3], that the current SI is incompatible with computer data systems and informatics generally.

Why do we need to state a "realisation" ?

- The 1971 definition specifies "12g of ^{12}C"
- The proposed definition specifies a fixed number of entities
- No real user can use either of these!
  - We need to give examples of practical ways to realise the mole.
  - *ie* How do we make (valid) measurements with results expressed in terms of the mole?

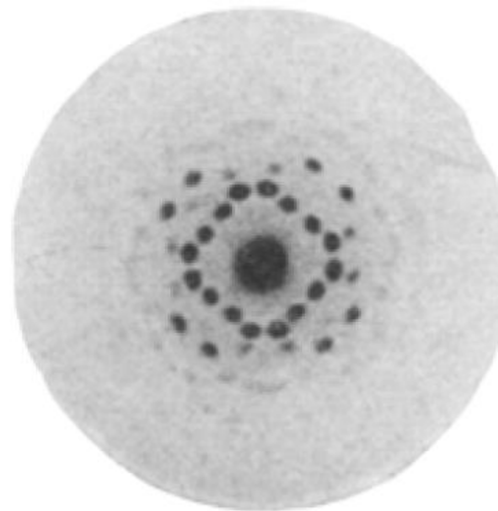
Definition of the mole

Max von Laue



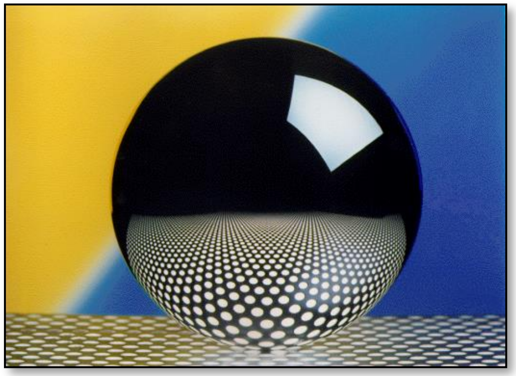
Interferenzerscheinungen mit Röntgenstrahlen
beim Durchgang durch Kristalle

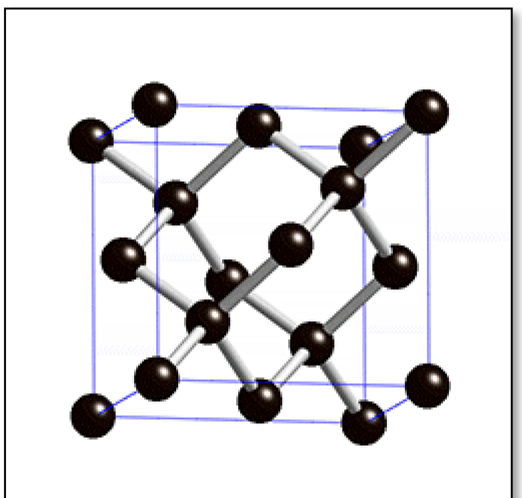
von W. Friedrich, P. Knipping und M. Laue und erläutert die
Bedeutung dieser Versuche für die Klärung unserer Auffassung



Sitzungsberichte der Mathematisch-Naturwissenschaftlichen Klasse der Königl. Bayerischen Akademie der Wissenschaften zu München S. 303 (1912)

Avogadro's constant via Bragg's relationship





http://www.msm.cam.ac.uk/phase-trans/2003/MP1.crystals/MP1.crystals.html

The following quantities must be measured to determine N_A:

(1) The volume occupied by a single Si atom, derived from the knowledge of the structure and the lattice spacing of a highly perfect, highly pure silicon crystal.

(2) The macroscopic density of the crystal.

(1) The content of impurities and self-point defects (incl. surface layer).

(2) The molar mass and, thus, the isotopic composition of the Si crystal (Si has three stable isotopes: ^{28}Si, ^{29}Si, ^{30}Si).

mise en pratique: example ^{28}Si in AVO28-S5

Identification step:

In case that the specified elementary entity is **Si** (Σ^{28}Si, ^{29}Si, ^{30}Si) and the real crystal is ***not*** purely **^{28}Si** also other elementary entities (elemental impurities, i.e. C, O, B and isotope impurities, i.e. ^{29}Si, ^{30}Si) and the volume of the surface passivation layer must be considered (i.e. excluded). In the ^{28}Si enriched single crystal Si-sphere AVO28-S5 the following statements apply:

One mol of Si atoms is equivalent to the number of Si atoms that is contained in a sample of the AVO28-S5 single crystal sphere with a volume of 12.05867069 cm^3 at 20 °C and in vacuum.

The AVO28-S5 single crystal sphere contains 35.74681424 mol of Si atoms.

integration of the Avogadro project and the mise en pratique of the mole

- the most accurate realisation of the mole: the perfect implementation of a mise en pratique of the mole
- possibly the origin of the fixed value of the natural constant that accompanies the unit mole
- the only realisation of the mole that does not require the determination of a mass (i.e. is independent of the mass)
- a description that is closely linked to the mise en pratique of the kilogram so that the interelation of the units can be made easily transparent
- a perfect tool to explain the mole to the world outside of the chemical community

Precondition for new defintion

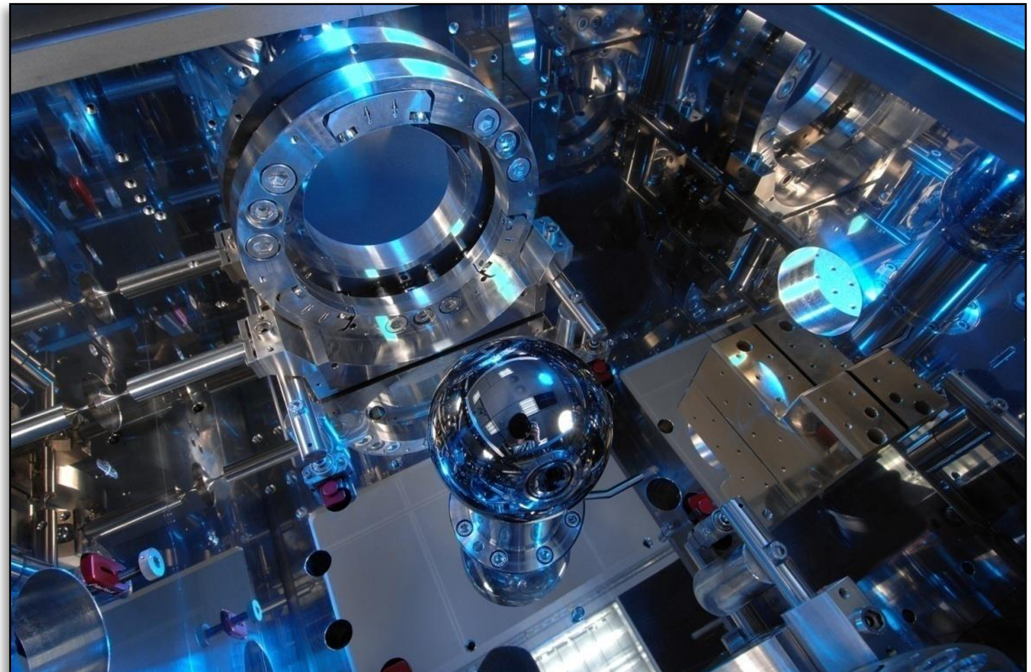
Measurement challenge:

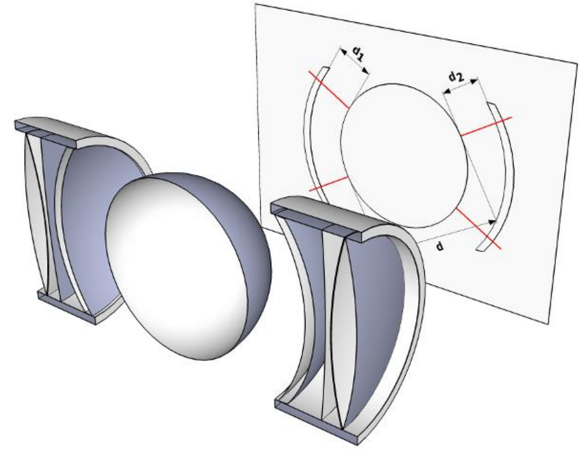
$u(N_A)$ or $u(h)$ ≤ present situation

– three measurement results of N_A or h available
– one of them should have a rel. stand. uncertainty of 2· 10^{-8} and
– two should have a rel. standard uncertainty of 5· 10^{-8}

Recommendation G1 (2013) of the CCM

interferometric volume determination

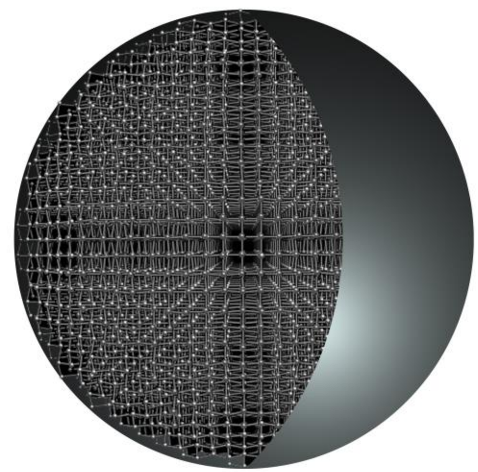




PTB‘s sphere interferometer with spherical symmetry

PTB‘s sphere interferometer enables complete topographies of spheres, $n_{diameter} \approx 600\ 000$.

The radius uncertainty is 0.7 nm or 8×10^{-9}



Radius topography of ^{28}Si-sphere S8. Peak to valley deviations from roundness amount to < 40 nm.

2.2 Realization by the X-ray-crystal-density (XRCD) method

The concept of the XRCD method where the mass of a pure substance can be expressed in terms of the number of elementary entities in the substance...assume that the crystal contains only the isotope ^{28}Si. The number *... is t given by*

$$N_{Si} = 8V_{sphere}/a(^{28}Si)^3$$

Such a number can be measured by the XRCD method in which the lattice constant *a and volume V of a nearly perfect crystal are measured.* To realize the definition of the kilogram, the mass m_s of (a ^{28}Si single crystal) sphere is... expressed in terms of the mass of a single atom

$$m_s = N_{Si}m(^{28}Si)$$

and

$$m_s = hN_{Si}m(^{28}Si)/h$$

The XRCD experiment determines N_{Si}; $m(^{28}Si)/h$ is a constant of nature whose value is... known to high accuracy and h is now exactly defined.

Uncertainty budget of the N_A by AVO28-S5

Quantity	Relative uncertainty/10^{-9}	100 × Contribution
Molar mass	8	5
Lattice parameter	11	9
Surface	15	18
Sphere volume	29	66
Sphere mass	4	1
Point defects	3	1
Total	36	100

The percentage contributions to the total uncertainty are the relevant variance fractions with respect to the total variance. The main contributions are at present due to surface characterization and the volume determination.

from: B Andreas et al., Metrologia, 48 (2011) S1–S13