Simposio Metrologia 2008
CENAM, Santiago de Queretaro
22-24/Oct./2008

Growing Demands for Metrology in Quality of Life

- Development of Standards in Chemistry -

National Metrology Institute of Japan

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National Metrology Institute of Japan

Today's Topics

1. National Standard System in Japan
2. Growing Demands for Metrology in Quality of Life
3. New Strategy of Metrology in Chemistry
4. Conclusions

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1. **National Standard System in Japan**
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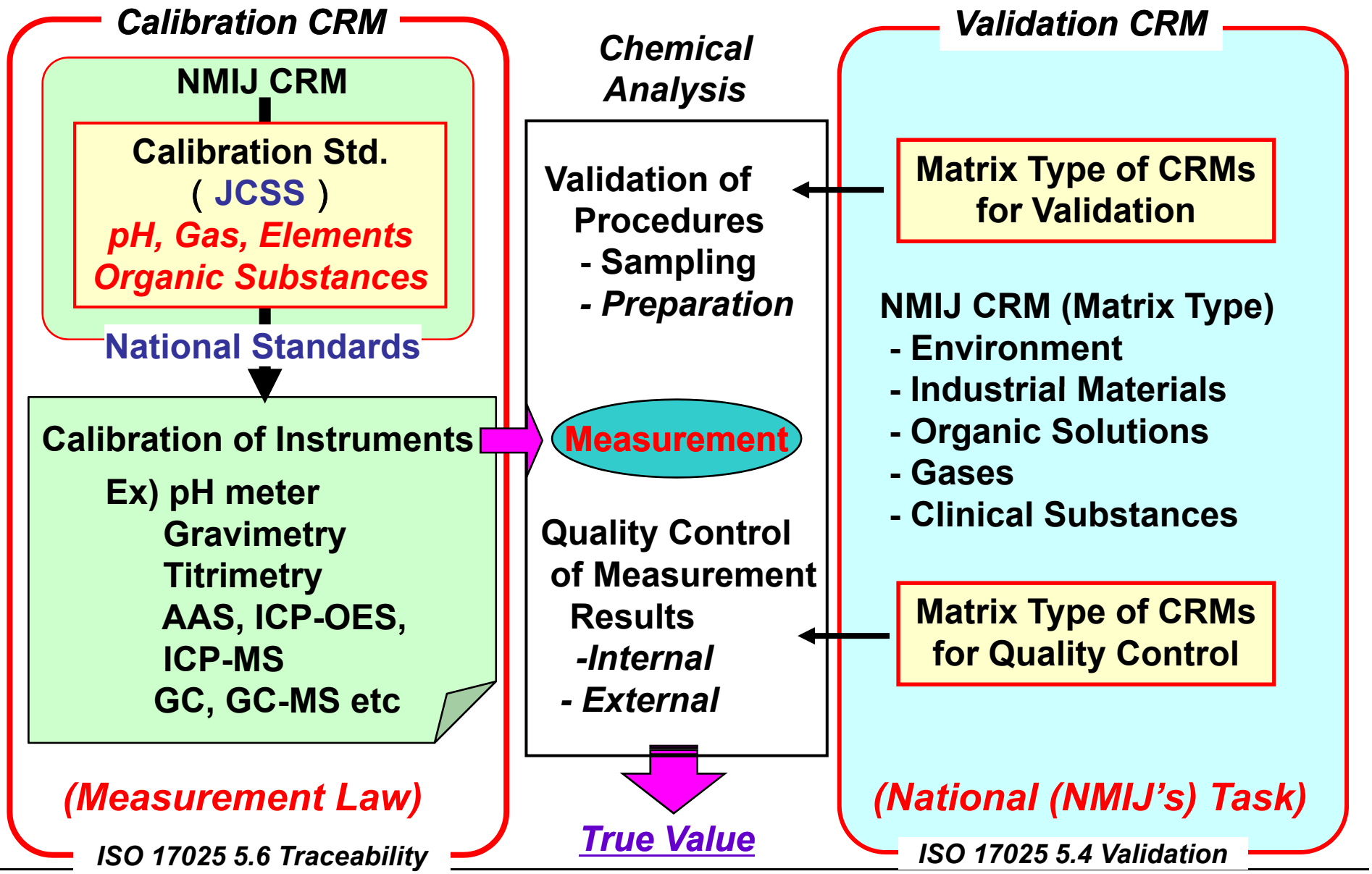
Traceability in Chemistry



Japan Measurement Law

- The Measurement Law of Japan was first constituted in 1951.
- It was originally designed for providing legal metrology.

- In 1992, the Japan Calibration Service System (JCSS) was established to provide the calibration standards under the Measurement Law.

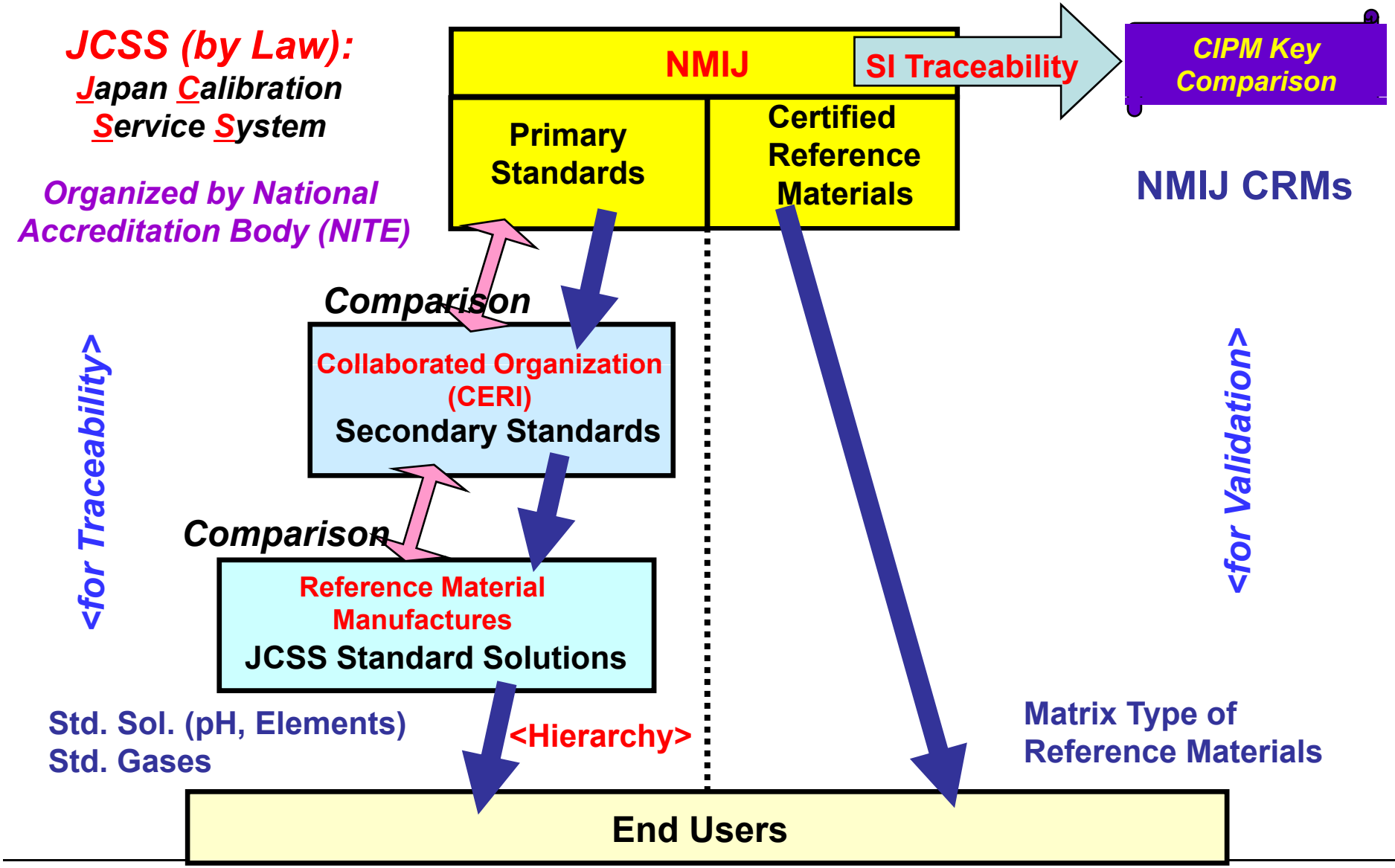
  The purposes are as follows;

  - to harmonized measurement systems internationally
  - to protect the consumers
  - to maintain the infrastructure for the technological innovation

JCSS is characterized by its relative rigidity of hierarchical structure.

JCSS has played a vital role in the establishment of traceability system in Japan, because it has extremely high name recognition and reliability.

Supply Scheme of Chemical Standards in Japan



Inorganic Calibration Standards through JCSS

June 2008

	I A	II A	III A	IV A	V A	VI A	VII A	VIII		I B	II B	III B	IV B	V B	VI B	VII B	0
1	H																He
2	Li	Be										B	C	N	O	F	Ne
3	Na	Mg										Al	Si	P	S	Cl	Ar
4	K	Ca	Sc	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	Zn	Ga	Ge	As	Se	Br	Kr
5	Rb	Sr	Y	Zr	Nb	Mo	Tc	Ru	Rh	Pd	Ag	Cd	In	Sn	Sb	Te	I	Xe
6	Cs	Ba	*La*	Hf	Ta	W	Re	Os	Ir	Pt	Au	Hg	Tl	Pb	Bi	Po	At	Rn
7	Fr	Ra	*Ac*															

pH Std. Sol.; 6 Levels

Lanthanoid	La	Ce	Pr	Nd	Pm	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
Actinoid	Ac	Th	Pa	U	Np	Pu	Am	Cm	Bk	Cf	Es	Fm	Md	No	Lr

JCSS Available (6 + 29 +10)

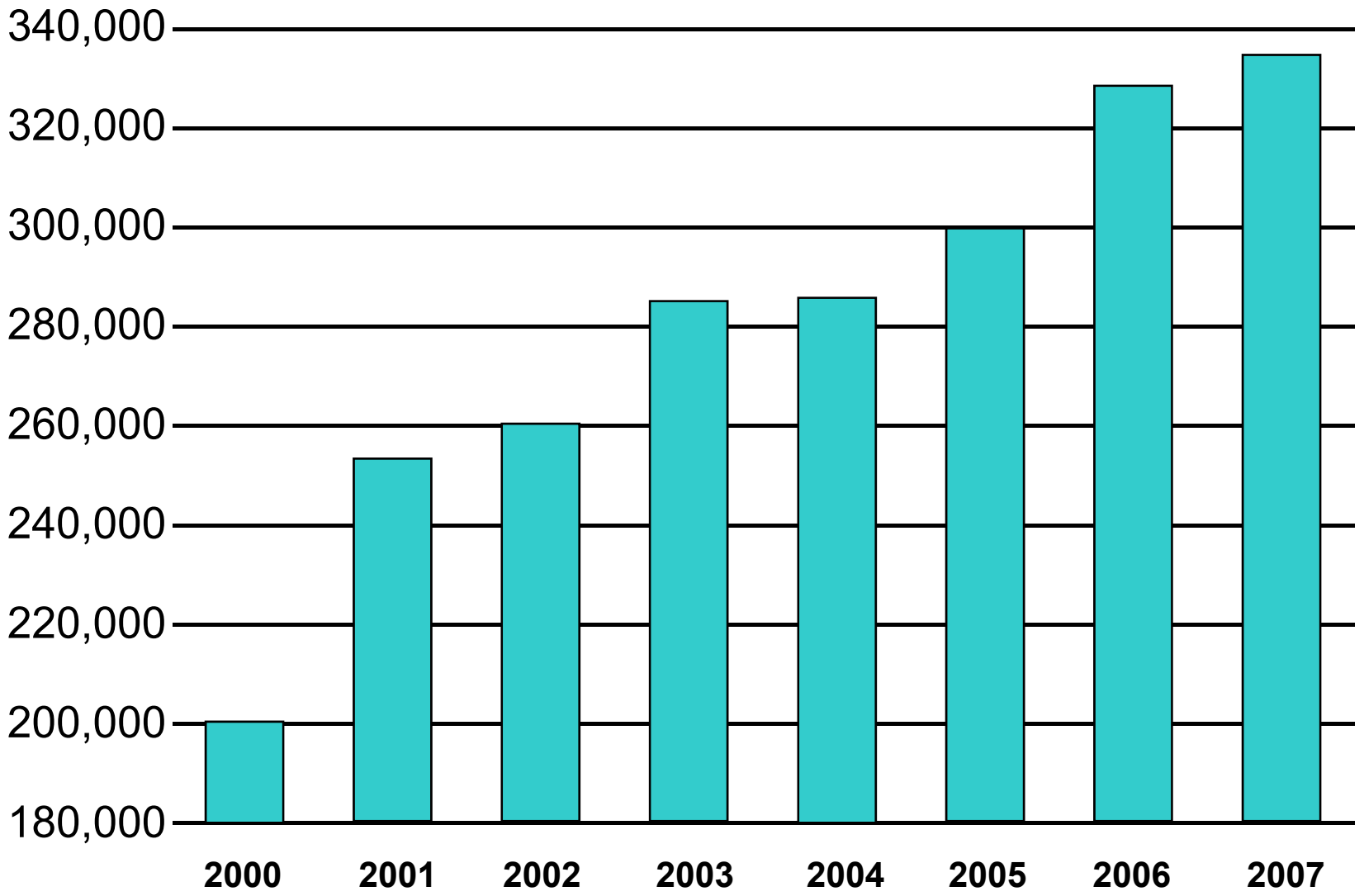
Coming Soon (2)

under Development (15)

Other Ions ⇒ PO_4^{3-} SO_4^{2-} NO_3^- NO_2^- NH_4^+ CN^-

Dissemination of JCSS Calibration Solutions

(NITE Web Site)



Development of Matrix Type of CRMs

Environment
NMIJ CRM7300 Series :
Marin Sediment
for Trace Elements
for Butyltins
for PCB Congeners
for Organochloride Pesticides

RoHS Regulation;
NMIJ CRM 8103-a Series
for Cd, Cr, Hg, Pb,

NMIJ Reference Materials (2008. 4.)

1. JCSS Reference Materials (121)

Gas Standards	37	(Inorganic 16、Organic 21)
Inorganic Solution Standards	39	(Metallic 29、Non-metallic 10)
pH Solution Standards	6	
Organic Solution Standards	40	(VOC 23、EDC 10, etc)

2．NMIJ Certified Reference Materials (132)

Gas Standards	15	
Organic Substances	39	
Inorganic Substances	7	
Environmental Materials	18	
RoHS Materials	11	
Polymer Standards	16	
Advanced Materials	23	(for EPMA, Multilayer, Pore)
Bio/Medical Material	3	

Total Number 229

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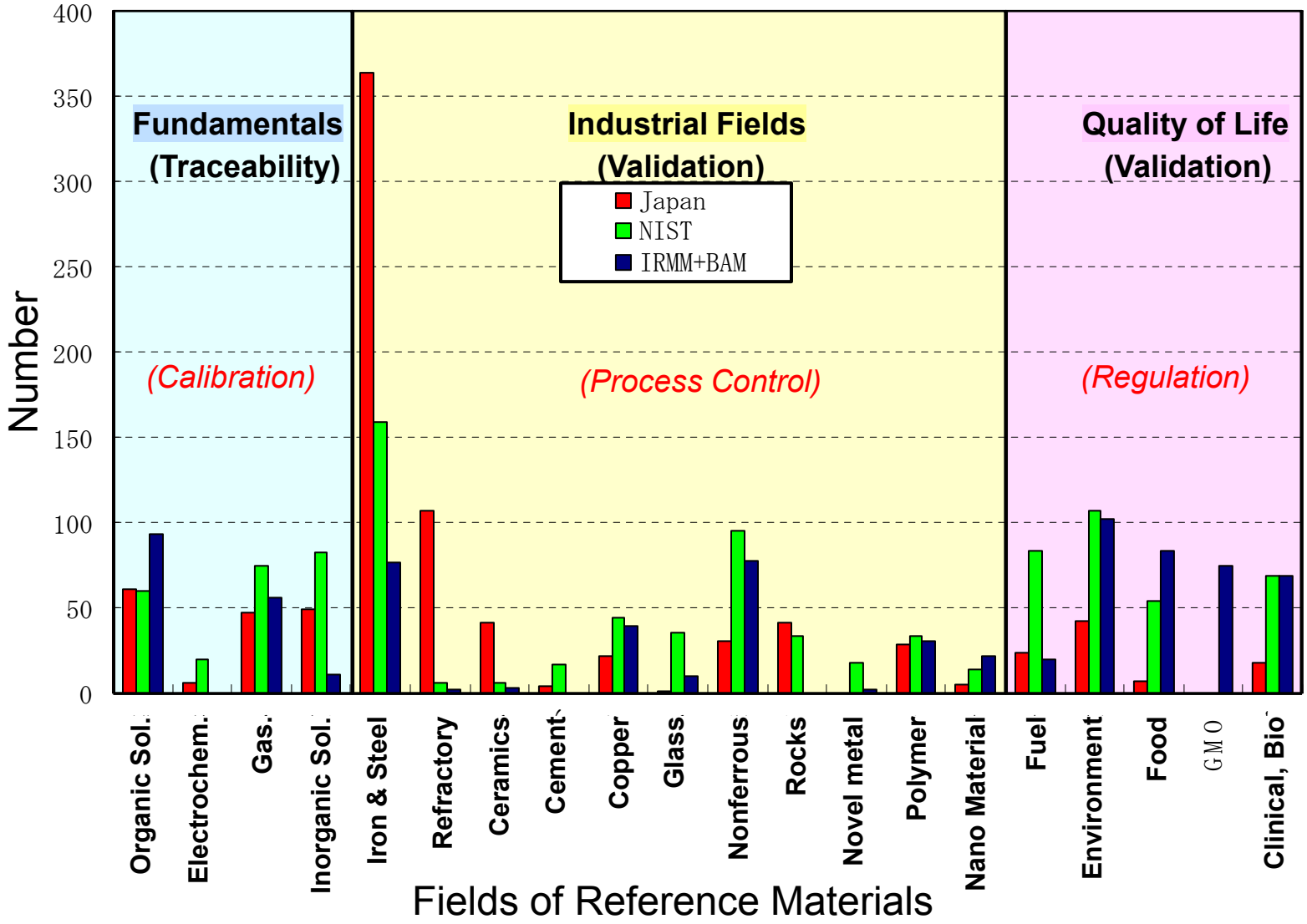
Reference Materials Developed in Japan, NIST, and EU

08/2008



CCQM toward the Quality of Life

Consulta ttee for Amount o Substanc Metrology in Chemistry

- Increasing numbe of relations with inter-governmental and international organizations, like WMO, WHO, IFCC, JCTLM, IAEA, ISO REMCO, IUPAC, CIPAC, Codex Alimentarius Commission, Inter Agency Meeting, ILAC, WADA (World Anti Doping Agency), IAFS (International Association of Forensic Sciences), ENFSI (European Network of Forensic Science Institutes), Pharmacopeia, USP, etc.

Global Warming

Clinical

Food

Safety

Drugs

Medicine

BIPM

Growing Demands for Metrology

Ministry	Regulation for Quality of Life in Japan
Ministry of Economy, Trade and Industry : METI	Explosives Control Law
	Industrial Water Law
	Law on Prohibition of Chemical Weapons and Regulation, etc., of Special Chemicals
Ministry of the Environment	Waste Management and Public Cleansing Law
ME	Water Pollution Control Law
	Law concerning Special Measures for Total Emission Reduction of Nitrogen Oxides from Automobiles in Specified Areas
	Law concerning Special Measures for Conservation of Lake Water Quality
	Air Pollution Control Law
	Offensive Odor Control Law
	Law Concerning Special Measures against Dioxins
Ministry of Health, Labour and Welfare	Poisonous and Deleterious Substances Control Law
MHLW	Waterworks Law
	Pharmaceutical Affairs Law
	Law for the Control of Household Products containing Harmful Substances
	鉛中毒予防規則
	Industrial Safety and Health Law
Ministry of Land Infrastructure	Sewerage Law
and Transport MLIT	Law Relating to the Prevention of Marine Pollution and Maritime Disaster
	危険物船舶運送及び貯蔵規則
	Civil Aeronautics Law
	Purification Tank Law
	消防法
Ministry of Agriculture, Forestry,	Agricultural Chemicals Regulation Law
and Fisheries MAFF	Agricultural Land Soil Pollution Prevention Law
METI & ME	Law concerning Reporting, etc. of Releases to the Environment of Specific Chemical Substances and Promoting Improvements in Their Management [Ministry of Environment]
METI & ME	Law Concerning the Protection of the Ozone Layer through the Control of Specified Substances and Other Measures
METI & ME & MHLW	Law concerning the Evaluation of Chemical Substances and Regulation of Their Manufacture

Growing Demands for Reference Materials in Quality of Life

Waterworks Law **(revised 2003)**

- **51 items to be regulated**
- **26 items to be monitored including 101 pesticides**

Regulation of residual pesticides **(revised 2006)**

- **shifted to a positive-list system from a negative-list system**
- **about 800 pesticides with uniform regulation values (0.01 ppm)**

Reference Materials in Current Use for Water Quality Testing

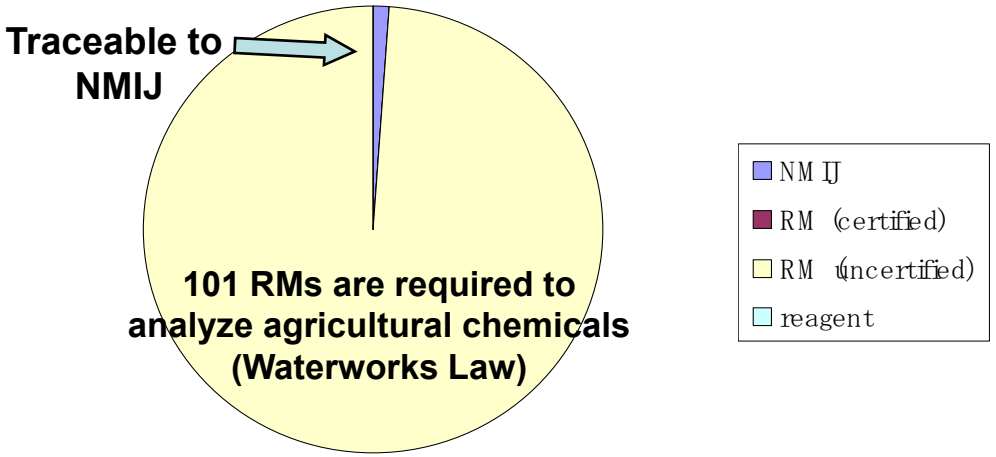
	Regulation item	Regulation value	CRM	RM
1	bacillus			
2	colon bacillus			
3	cadmium and its	0.01	JCSS	
4	mercury	0.0005	JCSS	
5	selenium	0.01	JCSS	
6	lead	0.01	JCSS	
7	arsenic	0.01	NIST	
8	chromium	0.05	JCSS	
9	cyanogen	0.01		self prep.
10	nitric oxide and nitrous oxide	10	JCSS	
11	fluorine	0.8	JCSS	
12	boron	1.0	NIST	
13	tetrachloromethane	0.002		commercial
14	1,4-dioxane	0.05		commercial
15	1,1-dichloroethylene	0.02		commercial
16	cis-1,2-dichloroethylene	0.04		commercial
17	dichloromethane	0.02		commercial
18	tetrachloroehylene	0.01		commercial
19	trichloroethylene	0.03		commercial
20	benzane	0.01		commercial
21	chloroacetic acid	0.02		commercial
22	chloroform	0.06		commercial
23	dichloroacetic acid	0.04		commercial
24	dibromochloromethane	0.1		commercial
25	bromic acid	0.01		commercial

	Regulation item	Regulation value	CRM	RM
26	trihalomethane	0.1		
27	trichloroacetic acid	0.2		commercial
28	bromodichloromethane	0.03		commercial
29	bromoform	0.09		commercial
30	formaldehyde	0.08		commercial
31	zinc	1.0	JCSS	
32	aluminium	0.2	JCSS	
33	lead	0.3	JCSS	
34	copper	1.0	JCSS	
35	sodium	200	JCSS	
36	manganese	0.05	JCSS	
37	chloride ion	200	JCSS	
38	calcium, magnecium (hardness)	300	JCSS	
39	ignition residue	500	-	-
40	ionic surfactant	0.2		commercial
41	musty odor substances	0.00001		commercial
42	2-MIB	0.00001		commercial
43	nonionic surfactant	0.02		self prep.
44	phenol	0.005		commercial
45	TOC	5		self prep.
46	pH	5.8-8.6	JCSS	
47	taste			
48	smell			
49	color	5		self prep.
50	turbidity	2		commercial

Inorganic Element

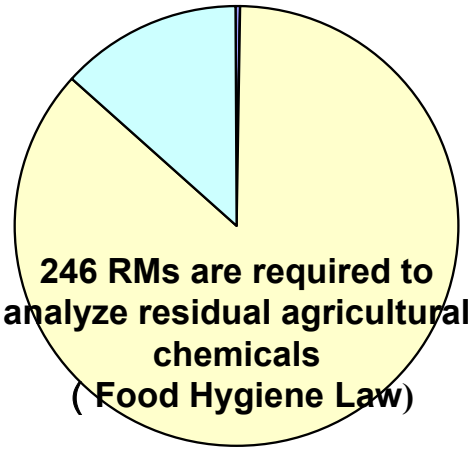
Reference Materials Used in Japan

Environmental analysis

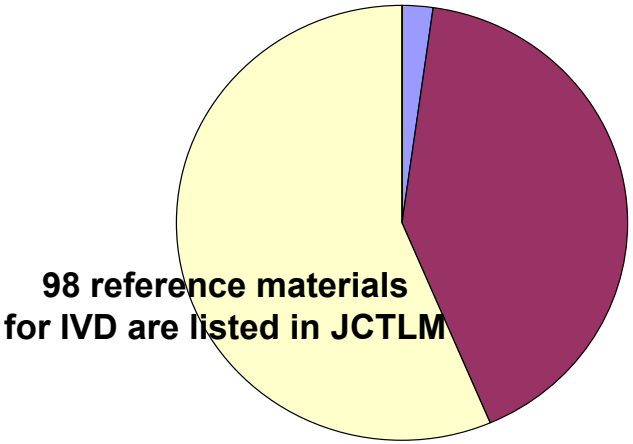


Ensuring the reliability of measurement values in fields such as environmental analysis, food analysis and clinical laboratory testing requires a vast number of reference materials.

Food analysis



Clinical laboratory testing



Difficulty in Supply of Sufficient Number of Measurement Standards by NMIJ

<Now>

Testing laboratories are performing calibrations using in-house standards supplied by private reagent manufacturers.

The reliable measurement standards,
which guarantee the traceability and realize the comparability among values measured.

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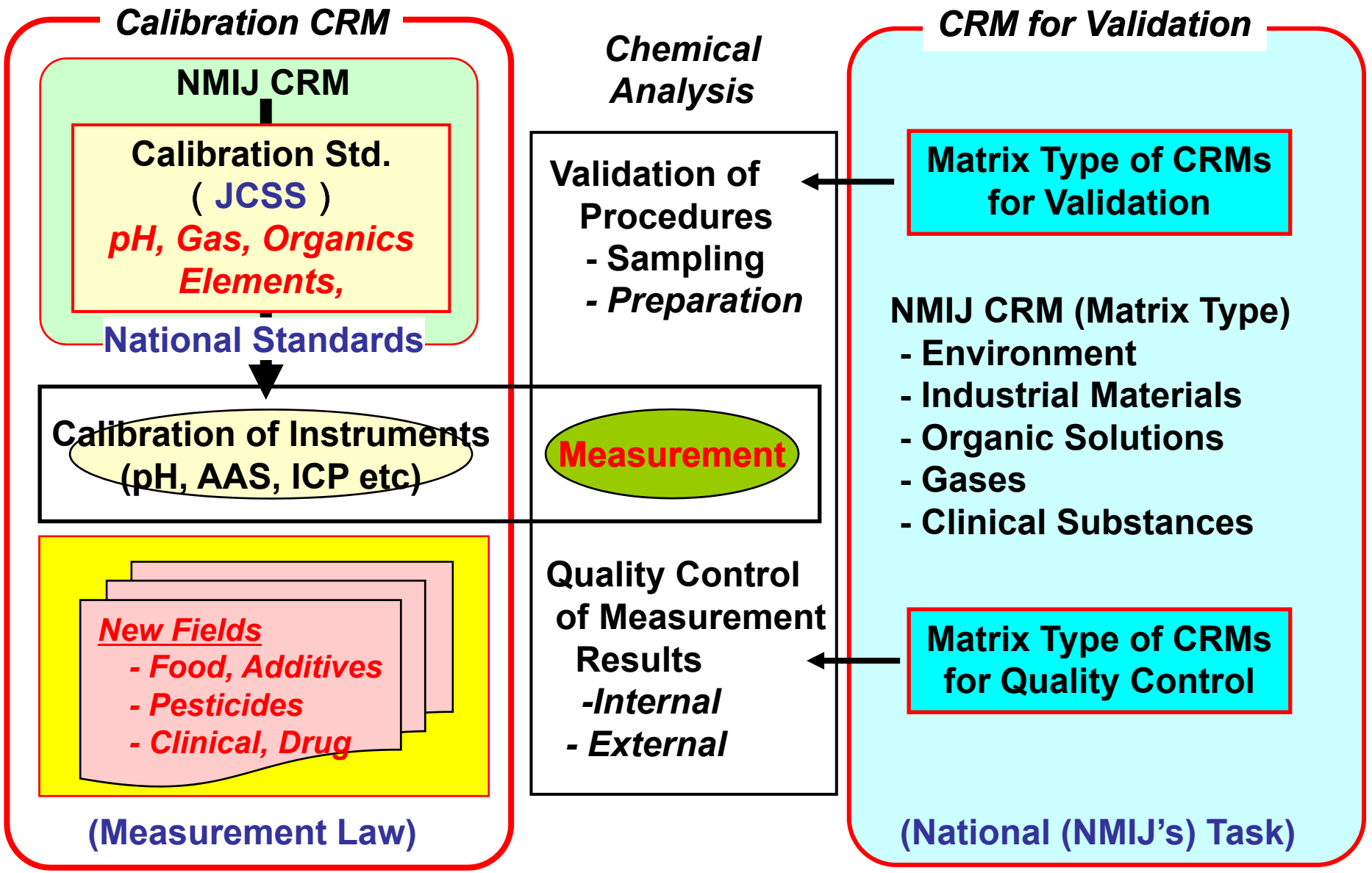
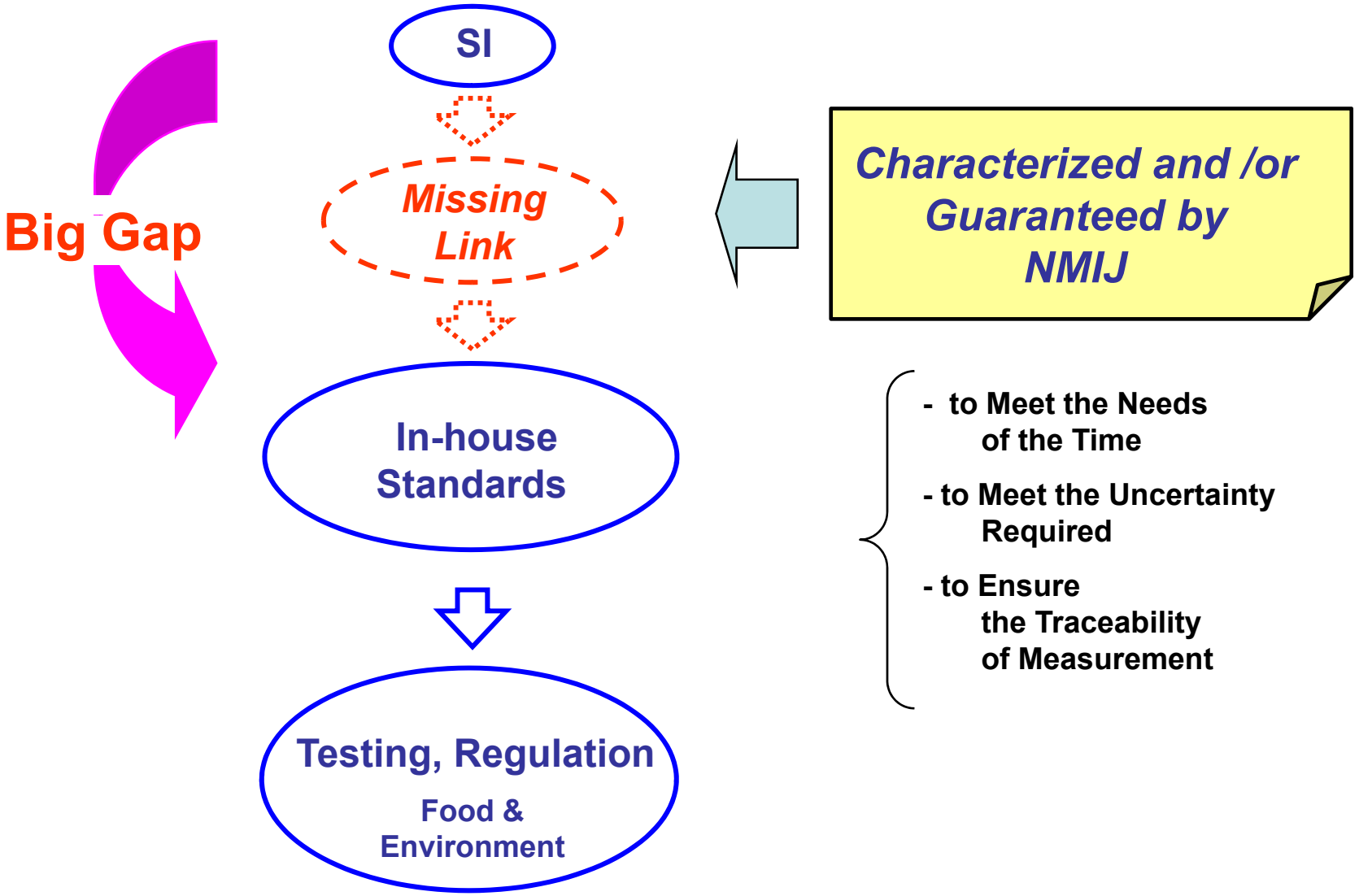
Traceability in Chemical Analysis



New Dissemination System for Metrological Standards of Organic Substances

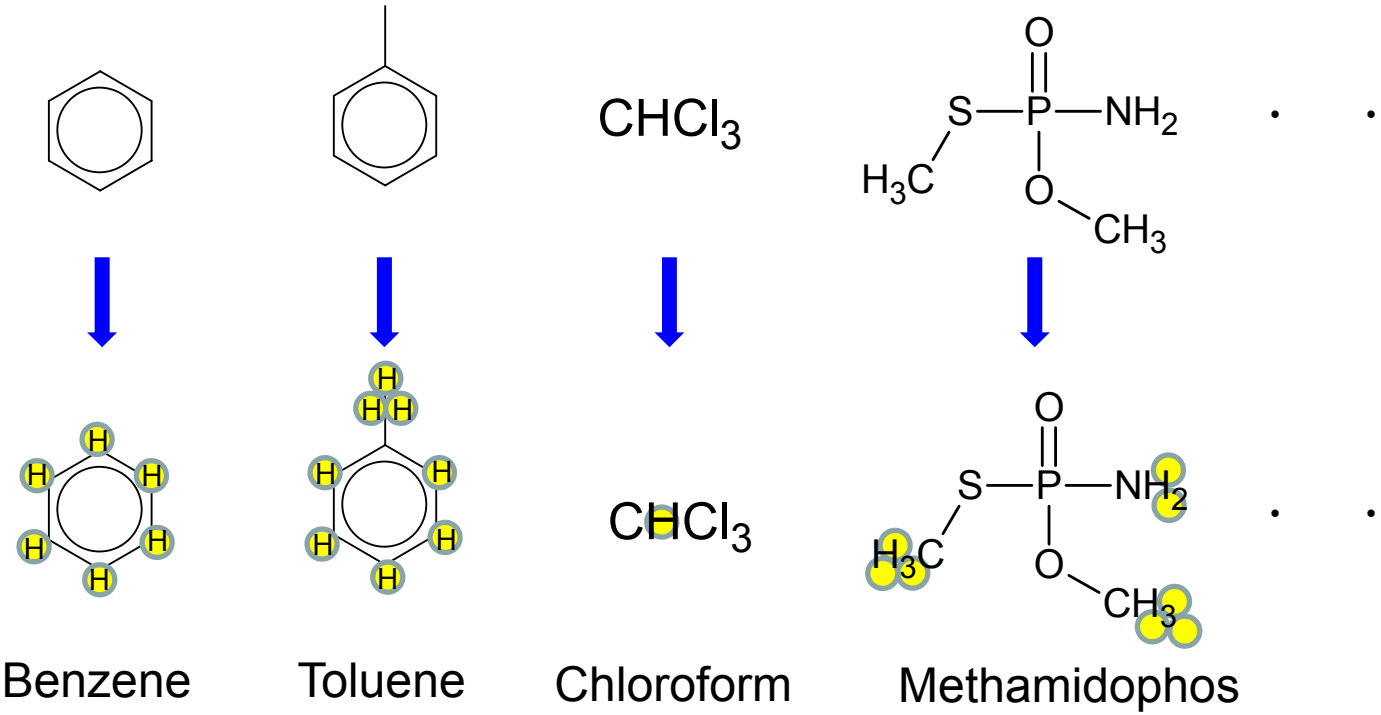


Universal Calibration by Quantitative NMR

National Std. (Conventional)

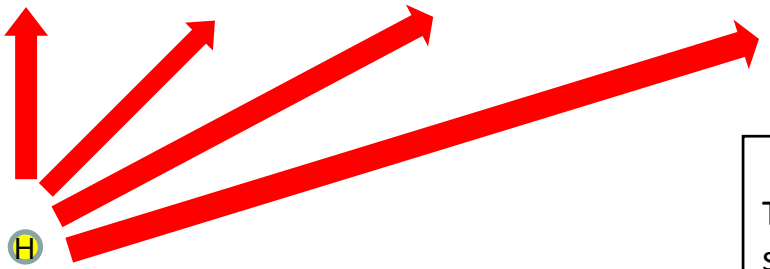
"Substance to Substance" Calibration

Working Std.



National Std. (Proton Basis)

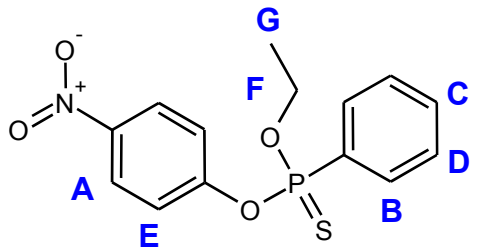
Hydrogen basis of Universal Calibration



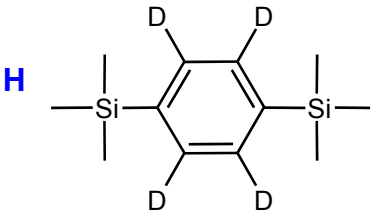
【qNMR】
To calibrate the amount of a substance by measuring the amount of hydrogen containing in the substance .

NMR spectrum of EPN and TMDS

EPN

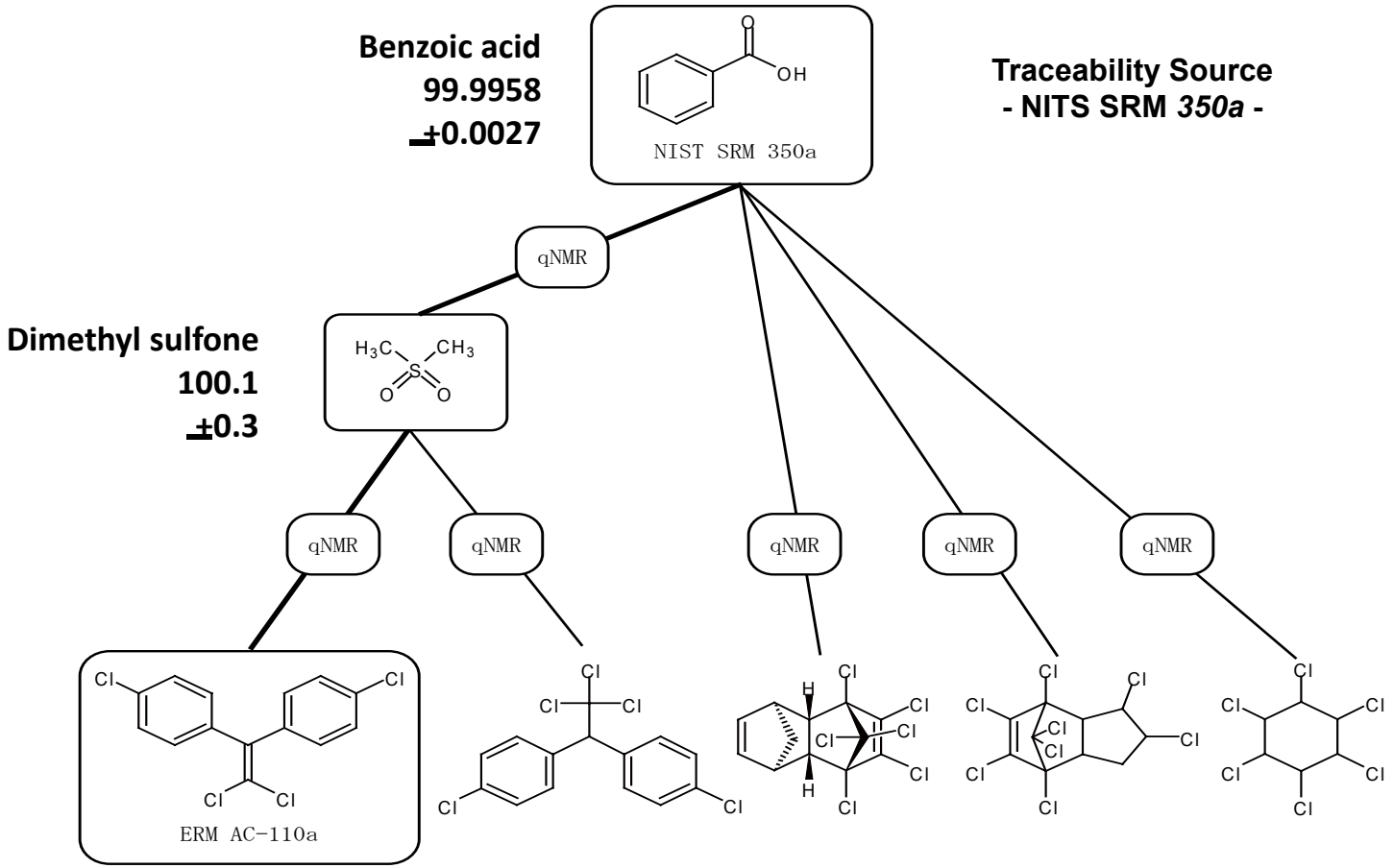


TMDS (Std.)



Acquisition Time (sec)	4.0000	***Comment***	EPN-14BTMSB-d6, run1		
Date	Jun 20 2008	***Date Stamp***	Jun 20 2008		
File Name	\\150.29.164.185\vnmr2\saito\auto_20.06.08\EPN-14BTMSBd4-32-4-60d-11.fid\fid				
Frequency (MHz)	599.90	***Nucleus***	1H	***Number of Transients***	32
Original Points Count	239880	***Points Count***	262144	***Pulse Sequence***	s2pul
Receiver Gain	20.00	***Solvent***	DICHLOROMETHANE-d2		
Spectrum Offset (Hz)	3446.3828	***Sweep Width (Hz)***	59970.02	***Temperature (degree C)***	25.000

Traceability of qNMR Calibration

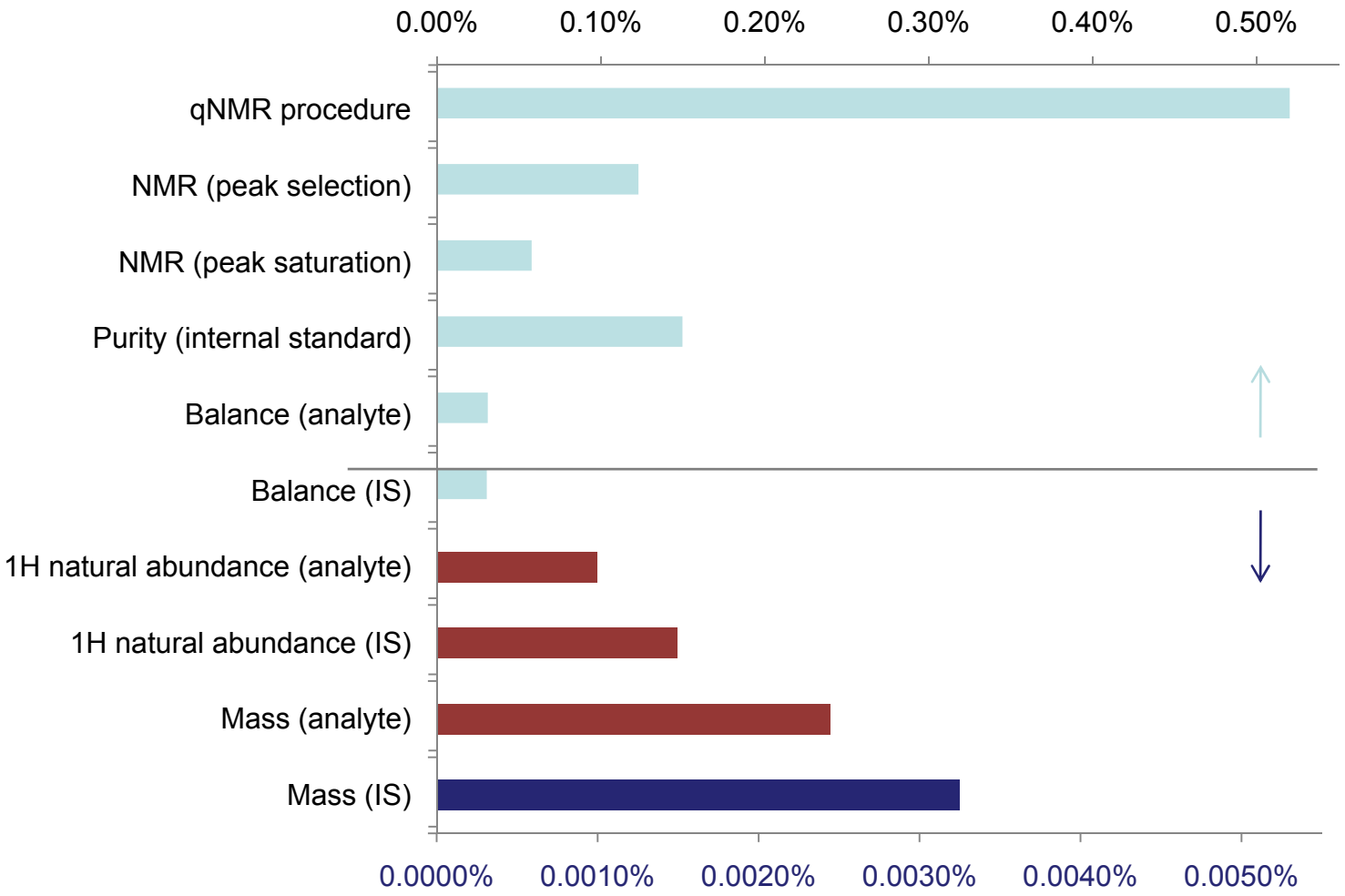


Name	*4,4'*-DDE	*4,4'*-DDT	Aldrin	*cis*-Chlordane	*α*-HCH
Purity (mass %)	99.6	99.9	98.7	99.1	99.2
Uncertainty (mass %)	0.4	1.2	0.5	0.5	0.6

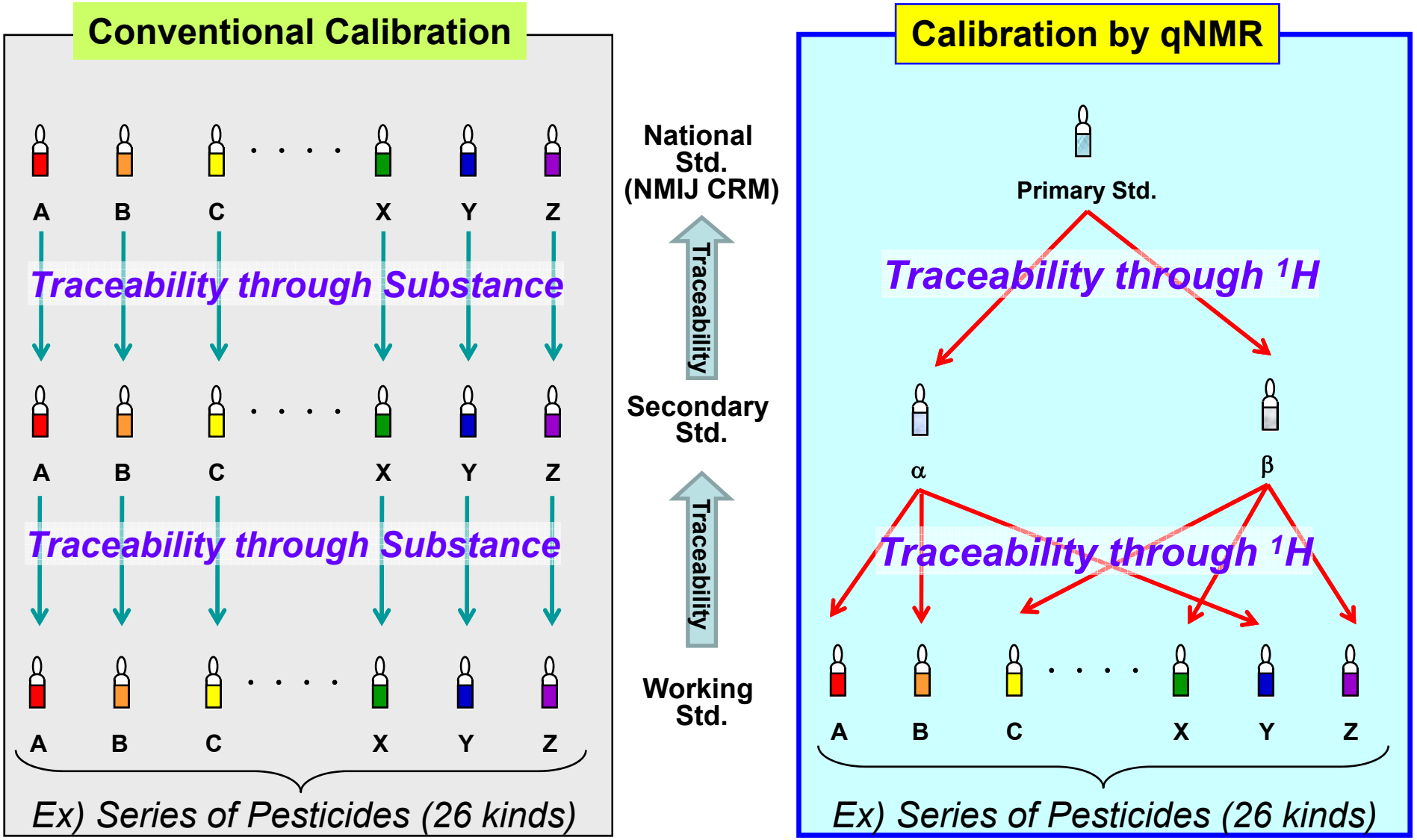
Analytical Performance of q-NMR

Compound	NMR		DSC		GC
	Purity (%)	Uncertainty (%, *k*=2)	Purity (%)	Uncertainty (%, *k*=2)	Purity (%)
p,p'-DDT	99.9	1.1	99.6	0.3	99.5
p,p'-DDE	99.8	0.6	99.7	0.3	99.6
p,p'-DDD	99.9	0.5	99.8	0.2	99.6
Aldrin	98.7	0.4	99.8	0.1	98.8
Dieldrin	97.8	0.7	99.8	0.2	99.0
Endrin	99.2	0.9	99.7	0.2	97.7
trans-Chlordane	99.5	0.6	99.8	0.2	99.6
trans-Nonachlor	99.5	0.6	99.7	0.2	99.0
cis-Nonachlor	99.9	0.5	99.8	0.2	99.8
Oxychlordane	99.3	0.5	99.9	0.1	99.6
Heptachlor	99.3	0.3	99.7	0.3	99.3
α-HCH	99.2	0.6	99.6	0.3	99.2
δ-HCH	99.0	0.6	99.9	0.1	99.2

Uncertainty Budget for *4,4'*-DDT

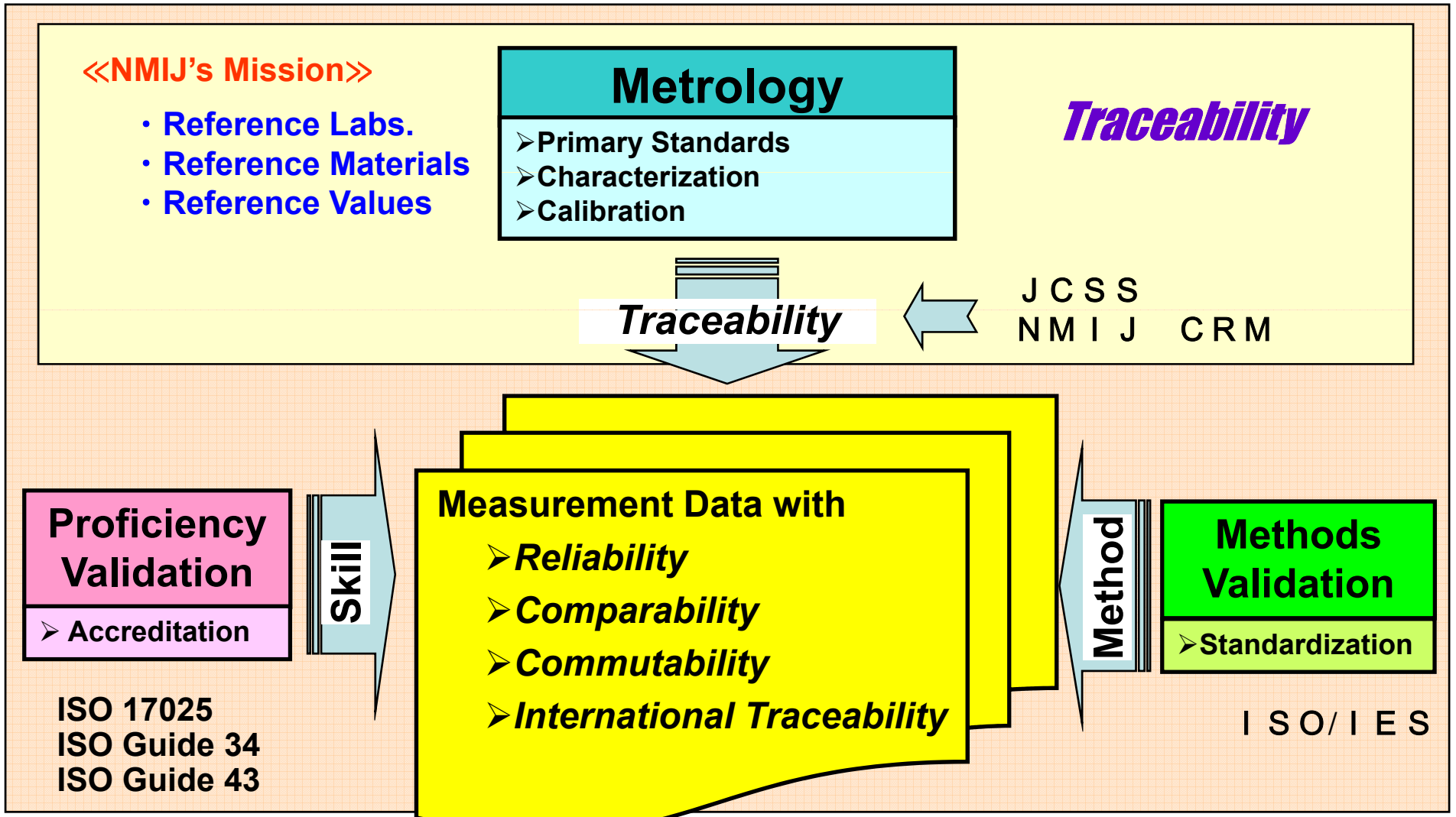


Innovation in Dissemination of Chemical Standards



Development of Standards for Safety and Security

Comparability and Reliability of Measurement Data



New Horizon in Metrology

- Metrology in Quality of Life -

Thank you, Queretaro!