

Creating and Improving a National Quality Infrastructure for Chemical Measurement

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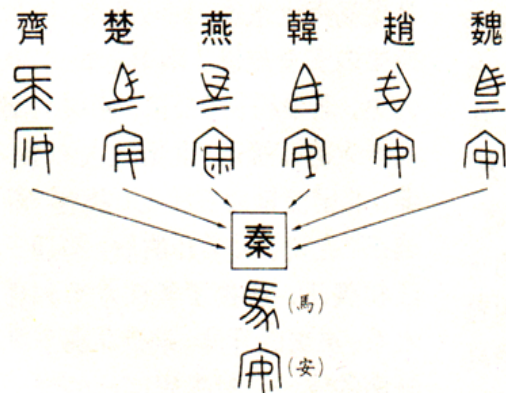


Quality Concept in Ancient CHINA



秦始皇(259-210 B.C.)

Qin Shi Huang First Emperor of Qin



秦统一文字示意图

(采自中国文史出版社《中国通史》)



Unified the weights and measures and characters

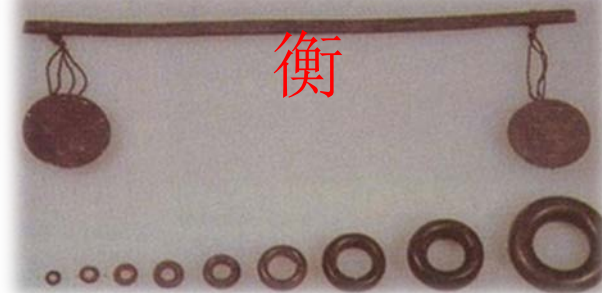
Length
度



Volume
量

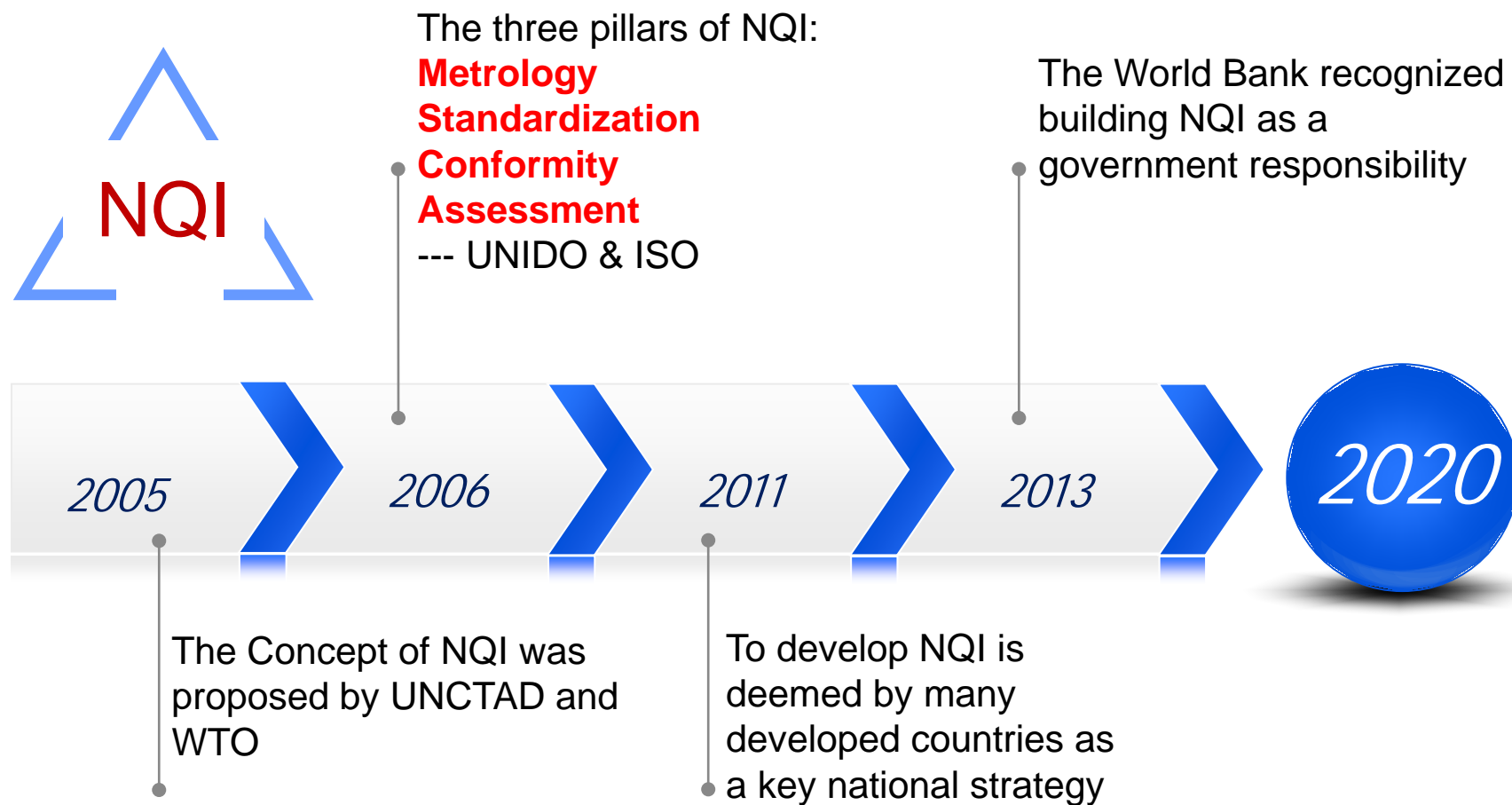


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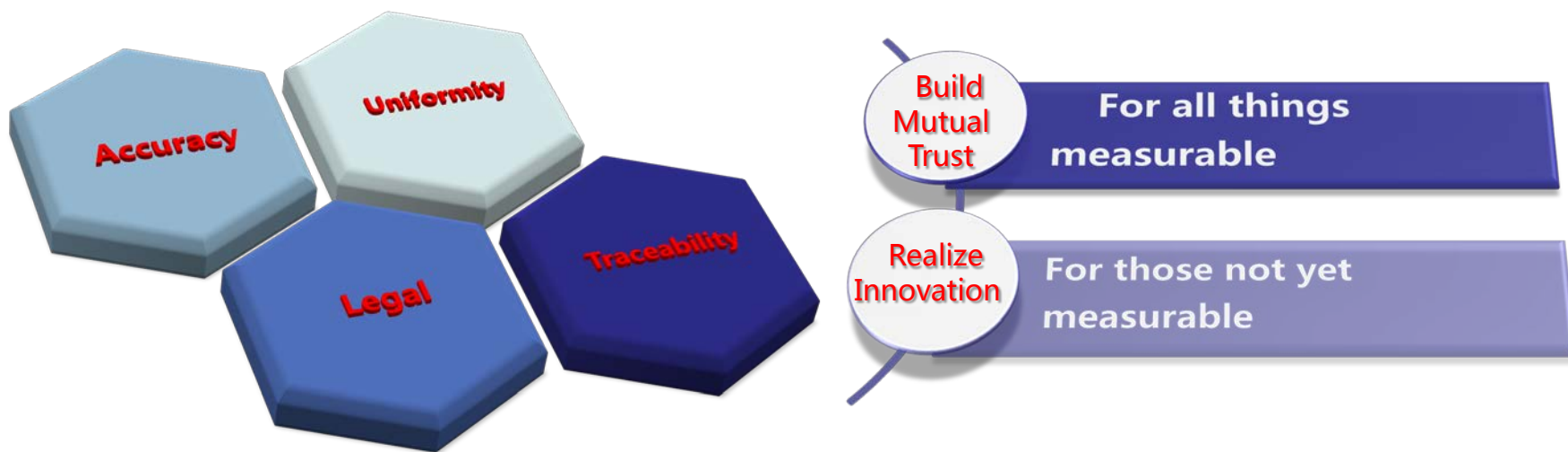
What is National Quality Infrastructure(NQI)?

A general name for all institutional frameworks that are needed in order to establish and implement metrology, standardization, certification, accreditation, inspection and testing. NQI includes regulatory system, management system and technical system, etc.



—— the science of measurement

ISO/IEC GUIDE 99:2007: The science of measurement, embracing both experimental and theoretical determinations at any level of uncertainty in any field of science and technology."

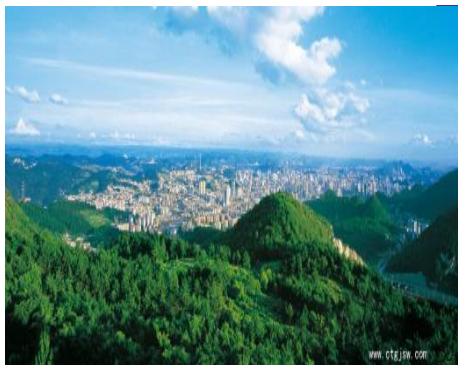


- A core national competitiveness

Chemical Measurements



Green
Production



Environmental
Protection

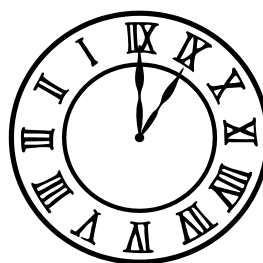


Food
Safety



Medical
& Clinical

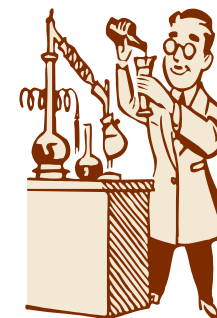
■ Chemical Measurements are more than 50% in total measurements



Different time



Different place



Different labs
/ methods

- **Measurement Methods**

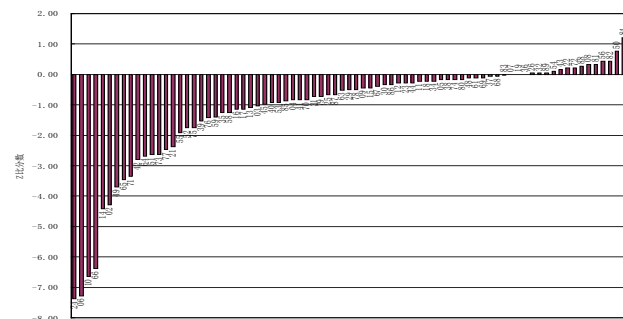
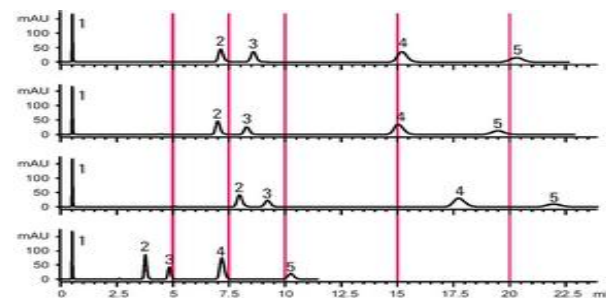
- Primary Methods
- Reference Methods
- Routine Methods
- Rapid Methods
- New Techniques

- **Certified Reference Materials**

- High Purity (for traceability)
- Solutions (for calibration)
- Matrix (for method validation)

- **Chemical Metrology Service**

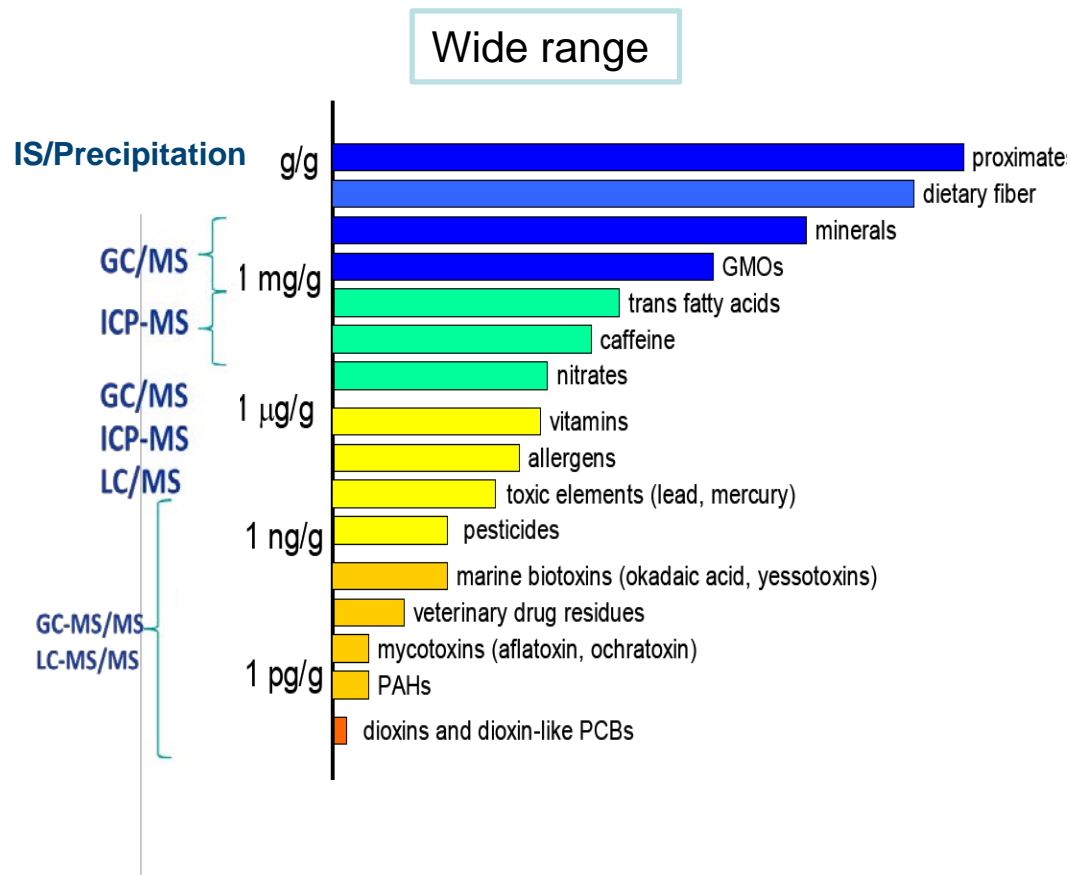
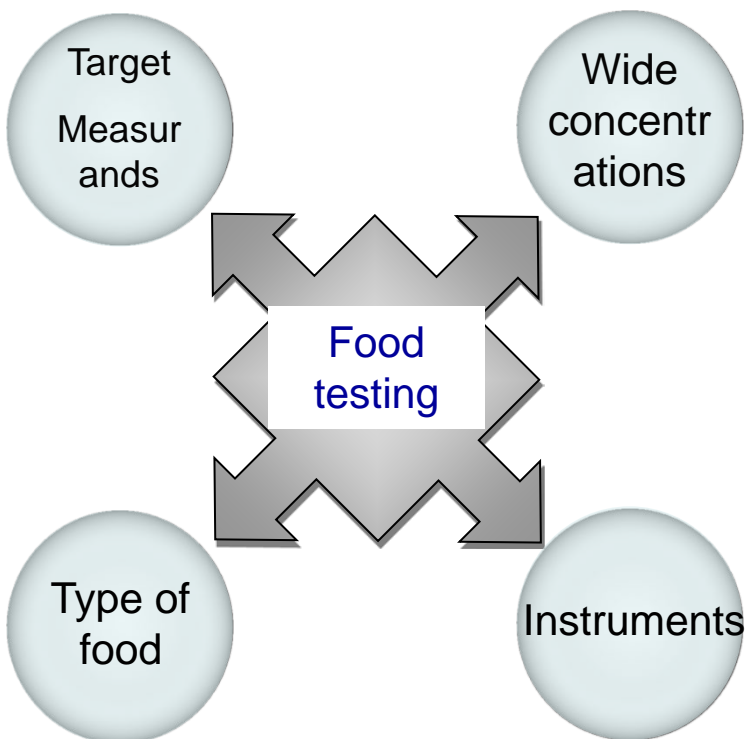
- CRM service
- Proficiency Testing
- Test results assessment





Chemical Metrology Research for Food Safety in NIM, China



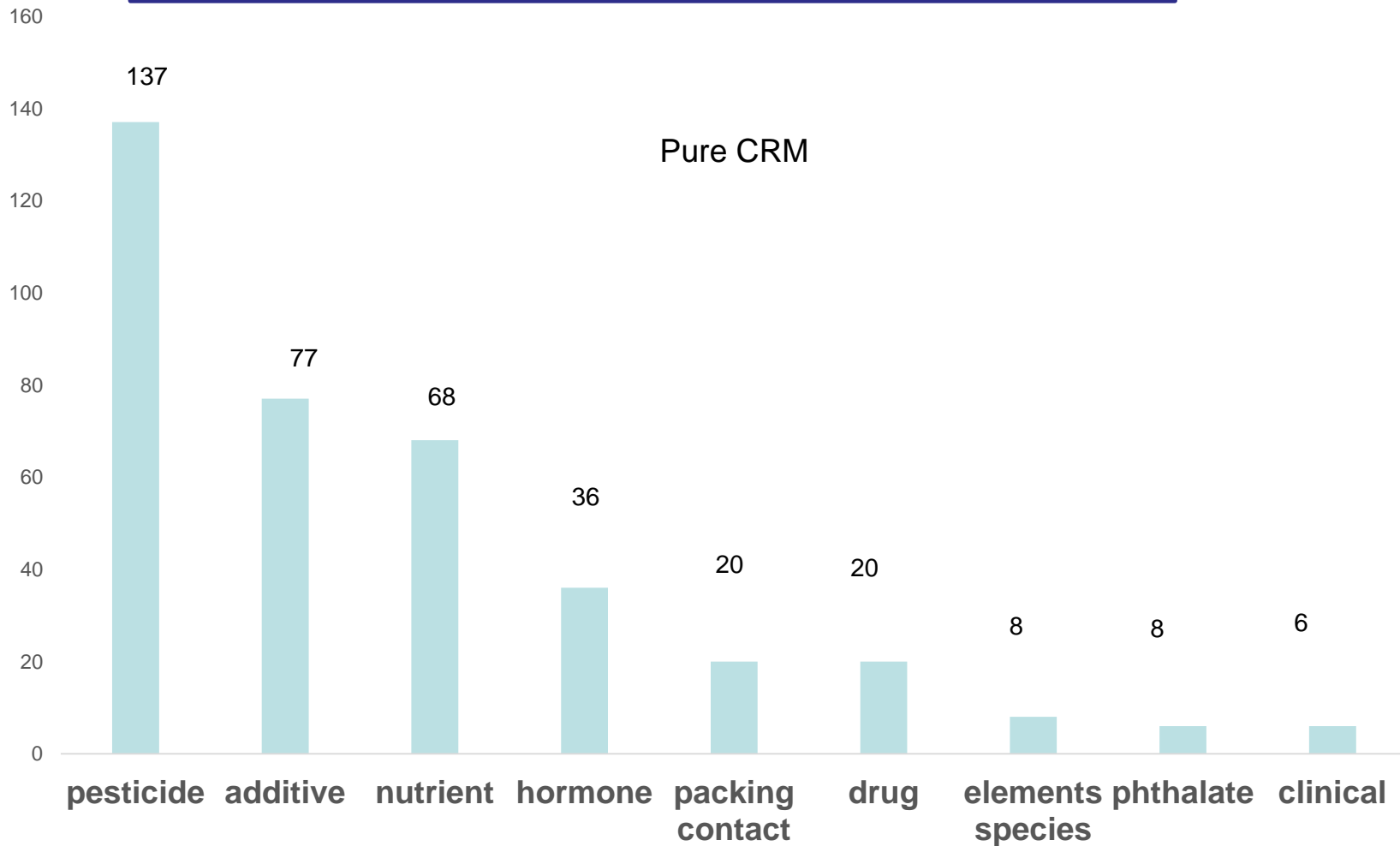


Challenges :

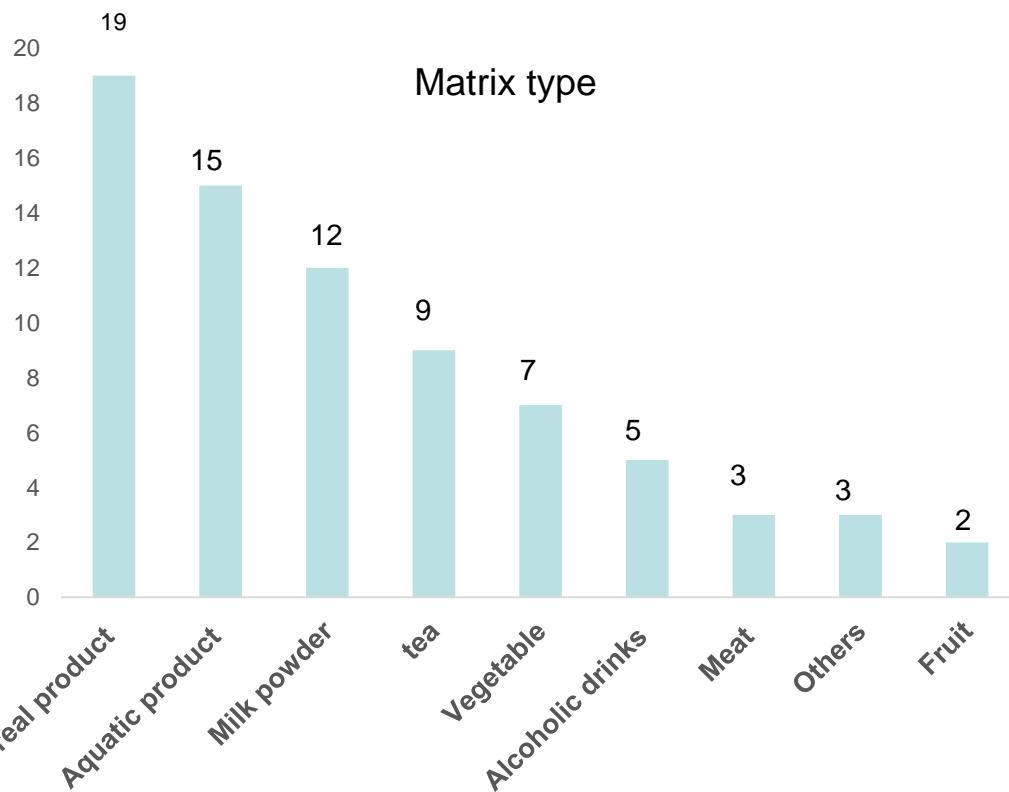
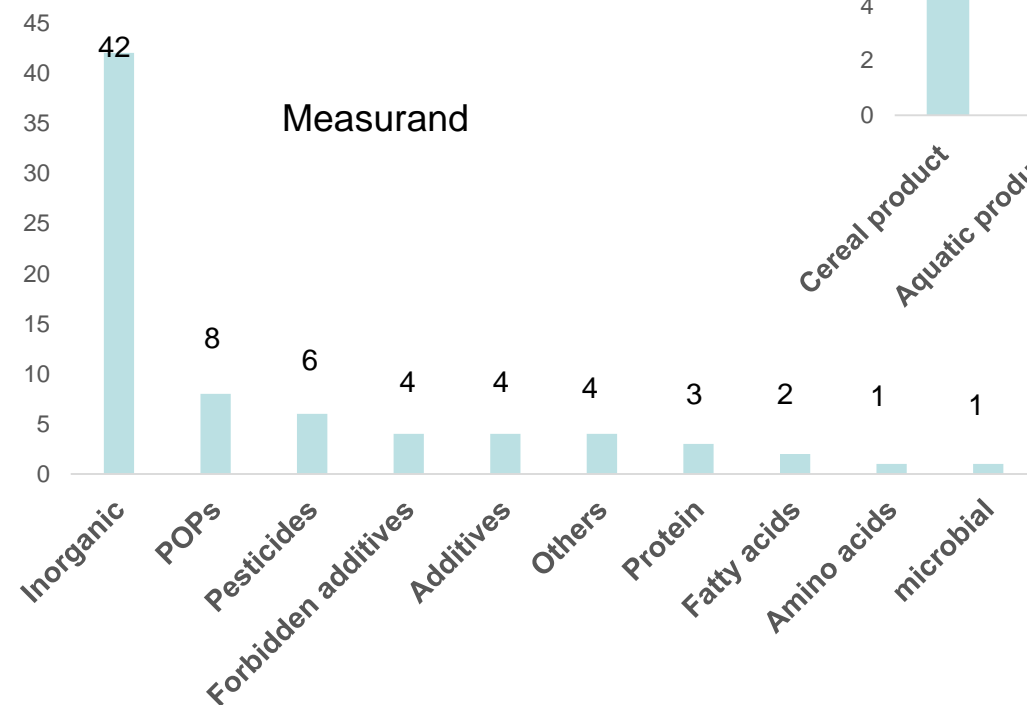
- **Different Matrix**---different pretreatment & different separation
- **Different Concentration**--- different sensitivity, enrich/dilute technique
- **Different Measurand**--- different instrument & resolution

- Food related CRM: 455
- purity & solution: 380

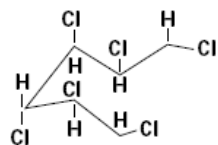
Pure CRM



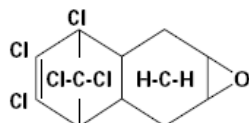
- Food related CRM: 455
- Food matrix: 75



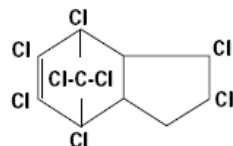
CRMs of Pesticides



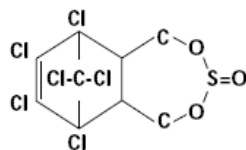
Lindane



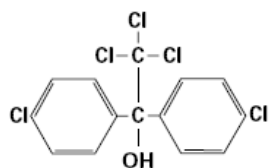
Dieldrin



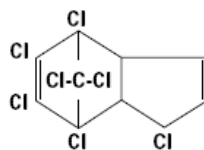
Chlordane



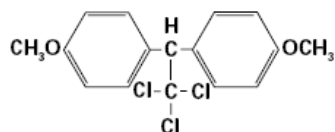
Endosulfan



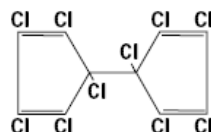
Dicofol



Heptachlor

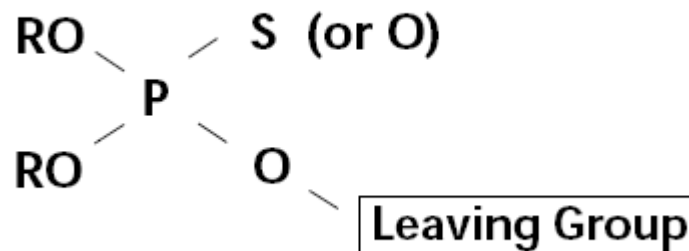


Methoxychlor



Dientochlor

Organochlorine pesticides



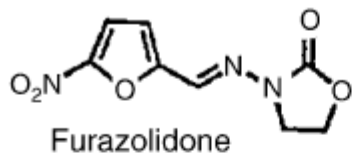
Organophosphate pesticides

More than 140 purity and solution CRMs have been developed including various types of pesticides

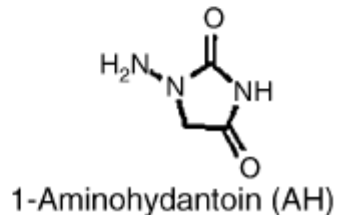
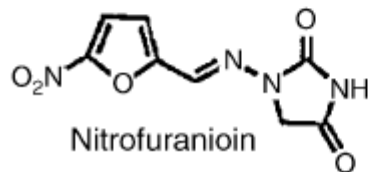
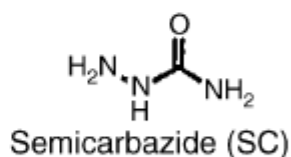
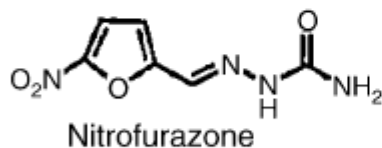
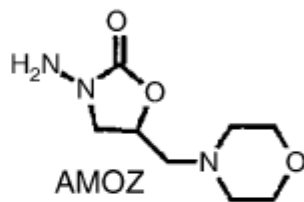
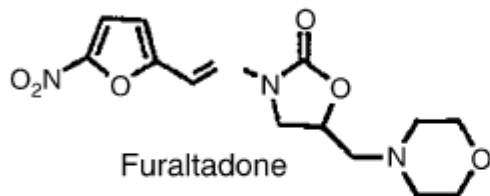
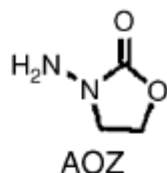
CRMs of Veterinary Drugs

Nitrofurans and metabolites

Parent drug



Metabolite



	Purity %	U% (k=2)
Furazolidone	99.9	0.9
Furaltadone	99.8	0.5
Nitrofurazone	99.8	0.4
Furadantin	99.8	0.6
AOZ	98.8	0.5
AMOZ	99.4	0.9
SEM	98.2	0.9
AHD	99.4	0.6

More than 40 purity and solution CRMs have been developed including nitrofurans, malachite, sulfanilamide et.al

Inorganic targets CRMs in food Matrix

CRM No.	Food Matrix	Certified Methods	Elements and Speices
GBW08509a	Skimmed Milk Powder	IC, Kjeldahl, ICP-MS, ICP-OES, AFS, AAS	Se, Zn, Fe, As, K, Na, Ca, Cu, Mn, Pb, Mg, N, P, Cl
GBW08521	Laver Powder	IDMS, INAA, ICP-MS, ICP-OES, AFS, AAS	As, Pb, Cd
GBW10029	Fish Tissue Powder	IDMS, ICP-MS, CVAFS, CVAAS, ID-GC-ICPMS, ID-LC-ICPMS, HPLC-CVAFS	Hg, MeHg
GBW10035	Wheat Powder	IDMS, ICP-MS, ICP-OES	Pb, Cd, Cr
GBW10036	Corn Powder	IDMS, ICP-MS, ICP-OES	Pb, Cd, Cr
GBW10054	defatted soybean powder	IDMS, ICP-MS, AAS, ICP-OES	K , Mg , Ca , Fe , Zn , Mn , Cu , Na
GBW10055	whole fat soybean powder	IDMS, ICP-MS, AAS, ICP-OES	K , Mg , Ca , Fe , Zn , Mn , Cu , Na
GBW(E)100194	Rice Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100195	Wheat Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100196	Corn Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100197	Bovine Muscle Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100198	Egg Yolk Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100199	Cushaw Powder	AAS, ICP-OES, ICP-MS	K, Mg, Ca, Fe, Mn, Zn, Cu, Na
GBW(E)100227	Infant Formula Milk Powder	ICP-MS, ICP-OES, AAS, XRF, SP	Se, Zn, Fe, K, Na, Ca, Cu, Mn, Mg, P

CRMs of Vitamins

	GBW(E)100227 Infant Formula		GBW(E)100228 Multivitamin Tablets		GBW(E)100273 Multivitamin Powder	
	certificate mg/kg	U (k=2)	certificate mg/kg	U (k=2)	Certificate m/m(10⁻²)	U (k=2)
Nicotinic Acid	39.8	3.0				
Nicotinamide			9.89*	1.8		
VitaminB₁	6.60	7.0	2.76*	4.0	0.101	0.008
VitaminB₂	20.2	6.0	4.28*	4.0	0.124	0.010
Vitamine B₅					0.883	0.057
VitaminB₆	4.71	6.6	0.30*	6.7	0.180	0.012
Ca	4.37*	5.5	92.7*	2.9		
Cu	4.5	4.5	675	1.7		
Fe	78.7	4.0	7.24*	6.1		
K	6.11*	2.0	7.10*	3.1		
Mg	849	1.6	1.10*	4.6		
Mn	1.06	5.7	613	4.6		
Na	1.95*	2.1	1.13*	3.6		
Zn	58.1	4.5	353	5.1		
P	2.98*	2.4	6.5*	2.0		
Se	1.23	9.8				

Participated in 18 international comparisons focus on **organic analysis** for food matrix

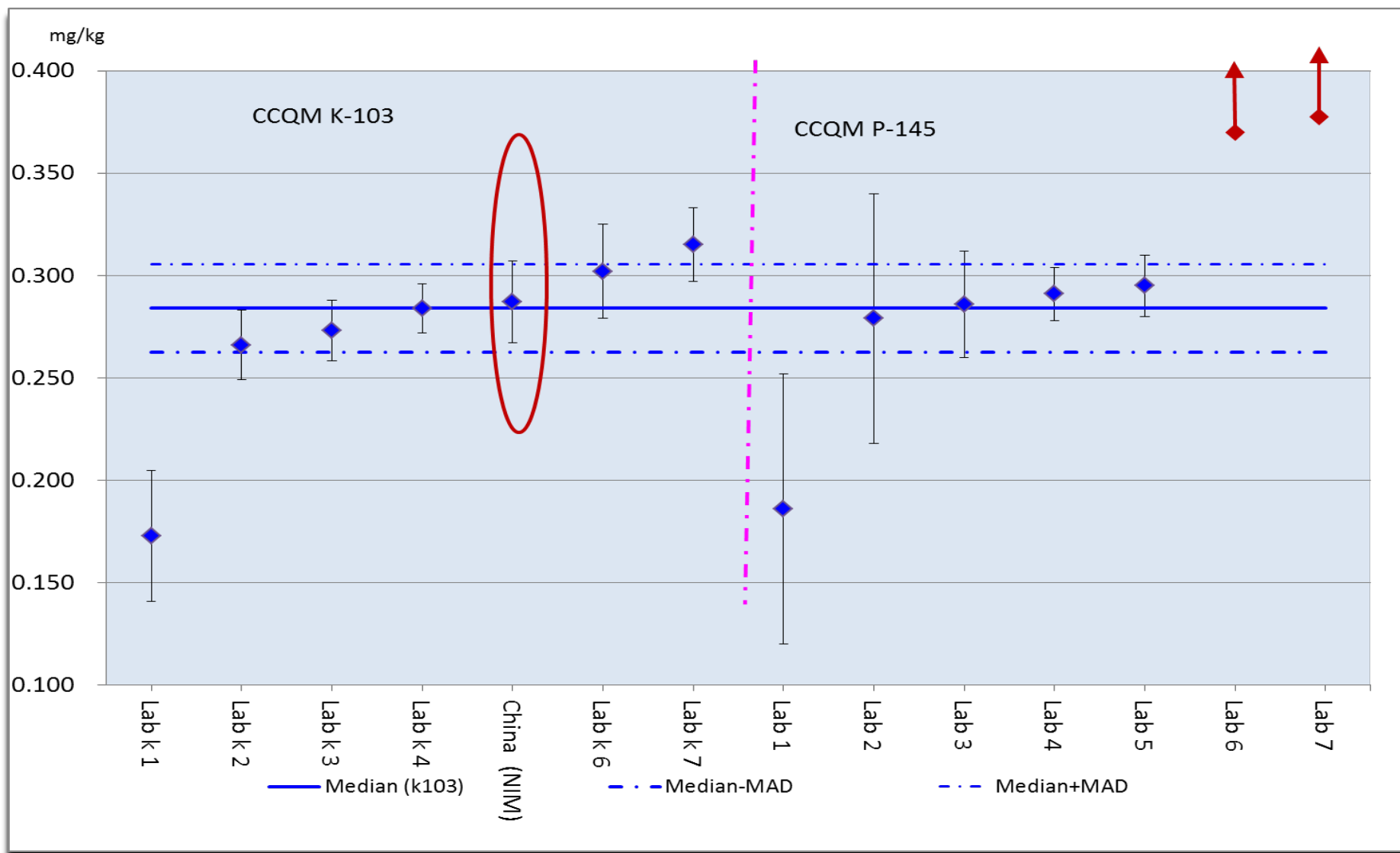
WR	No.	Description	Time
OAWG	CCQM-P10	Gamma-HCH in fish oil	1999
OAWG	CCQM-K5	p,p'-DDE in fish oil	1999
OAWG	CCQM-P4	p,p'-DDE in corn oil	1998
OAWG	CCQM-K21	p,p'-DDT in fish oil	2000
OAWG	CCQM-P91	Pesticides in Foods: Pyrethroids in apple juice	2008
OAWG	CCQM-K95	Organochlorine pesticides in tea	2011
OAWG	CCQM-P57	PCB Congeners in Tissue Extract	2004
OAWG	CCQM-P67	PCBs Congeners in Tissue	2004
OAWG	CCQM-K62	Nutrients in Infant/Adult Formula: Vitamins	2007-2008
OAWG	CCQM-P78	Nutrients in infant/ adult formula	2006
OAWG	CCQM-P109	Determination of Acrylamide in Potato Chips	2008
OAWG	CCQM-K103	Melamine in Milk Powder	2012
APMP	APMP.QM-P19	Melamine in Milk Powder	2010
APMP	APMP.QM-P23	Determination of Mass Fraction of Benzoic Acid in Orange Juice	2012
OAWG	CCQM-P122	Chloramphenicol as residue in pig muscle(only P,K81)	2009
OAWG	CCQM-K85	Malachite green in Salmon	2010
OAWG	CCQM-P88	Malachite green in fish	2006
APMP	APMP.QM-S6	Clenbuterol in meat	2013

International Comparisons – CCQM/IAWG

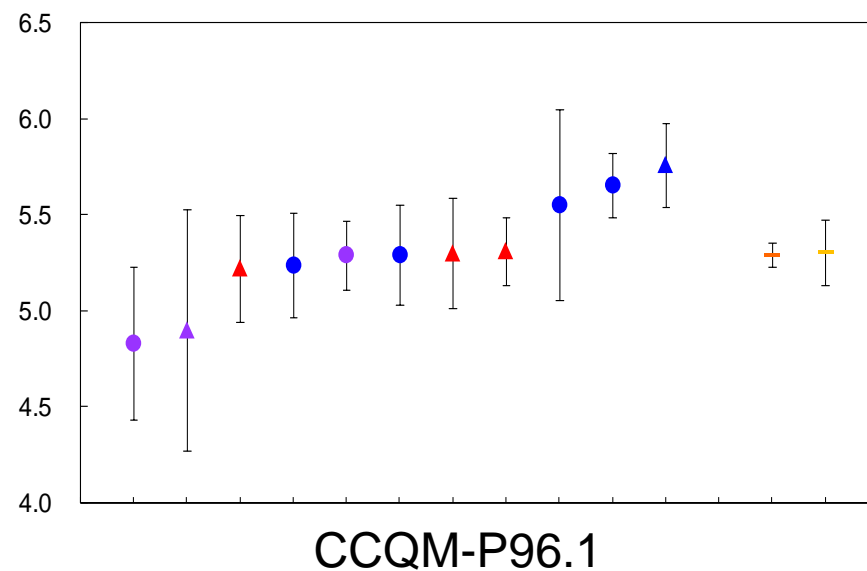
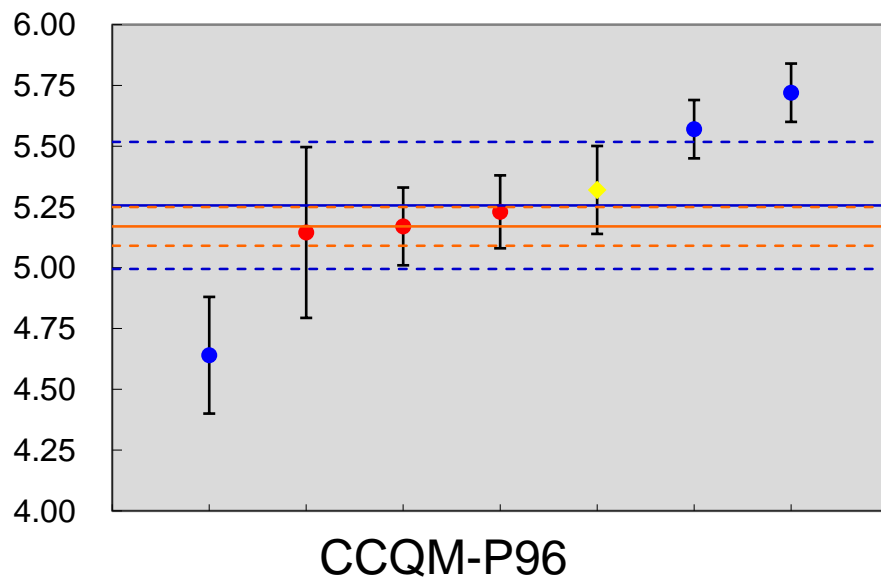
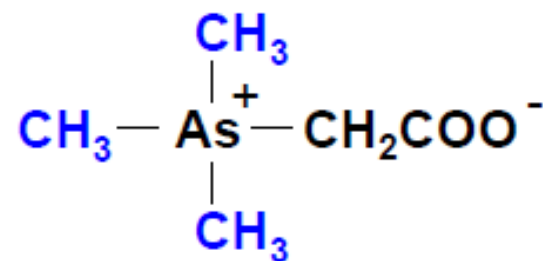
Participated in 18 international comparisons focus on **inorganic analysis** for food matrix

WG	No.	Description	Time
IAWG	CCQM-K2	Cd and Pb in natural water	1998
IAWG	CCQM-K24	Cd in Rice	2001
IAWG	CCQM-K30.1	Pb in wine	2009
IAWG	CCQM-K43.1	As, Hg, Se and methylmercury content in marine fish	2005
IAWG	CCQM-K49	Toxic and essential elements in bovine liver	2006
IAWG	CCQM-K56	Trace elements in Soybean powder	2006
IAWG	CCQM-K60	Total Se and Se speciation analysis of Se-rich wheat flour	2008
IAWG	CCQM-K75	Toxic metals in algae	2009
IAWG	CCQM-K89	Trace and essential elements in Herba Ecliptae	2010
IAWG	CCQM-K97	Arsenobetaine in solution and fish	2011
IAWG	CCQM-K108	Determination of arsenic species, total arsenic and cadmium in brown rice flour	2013
IAWG	CCQM-K124	Trace elements and chromium speciation in drinking water	2014
IAWG	CCQM-K125	Iodine and other elements in infant formula	2014
IAWG	CCQM-P39.1	Methyl-mercury in salmon fish	2005
IAWG	CCQM-P85	Toxic and essential elements in bovine liver	2006
IAWG	CCQM-P86	Analysis of total Se and Se methionine in pharmaceutical supplements	2006
IAWG	CCQM-P96	As and arsenobetaine content in marine fish	2007
IAWG	CCQM-P96.1	As and arsenobetaine in calibration solution and Japanese sea bass sample	2010

Melamine in Milk Powder – CCQM-K103 & P145



CCQM-P96 & P96.1 Arsenobetaine in fish



CCQM-K97/P133 Arsenobetaine in fish

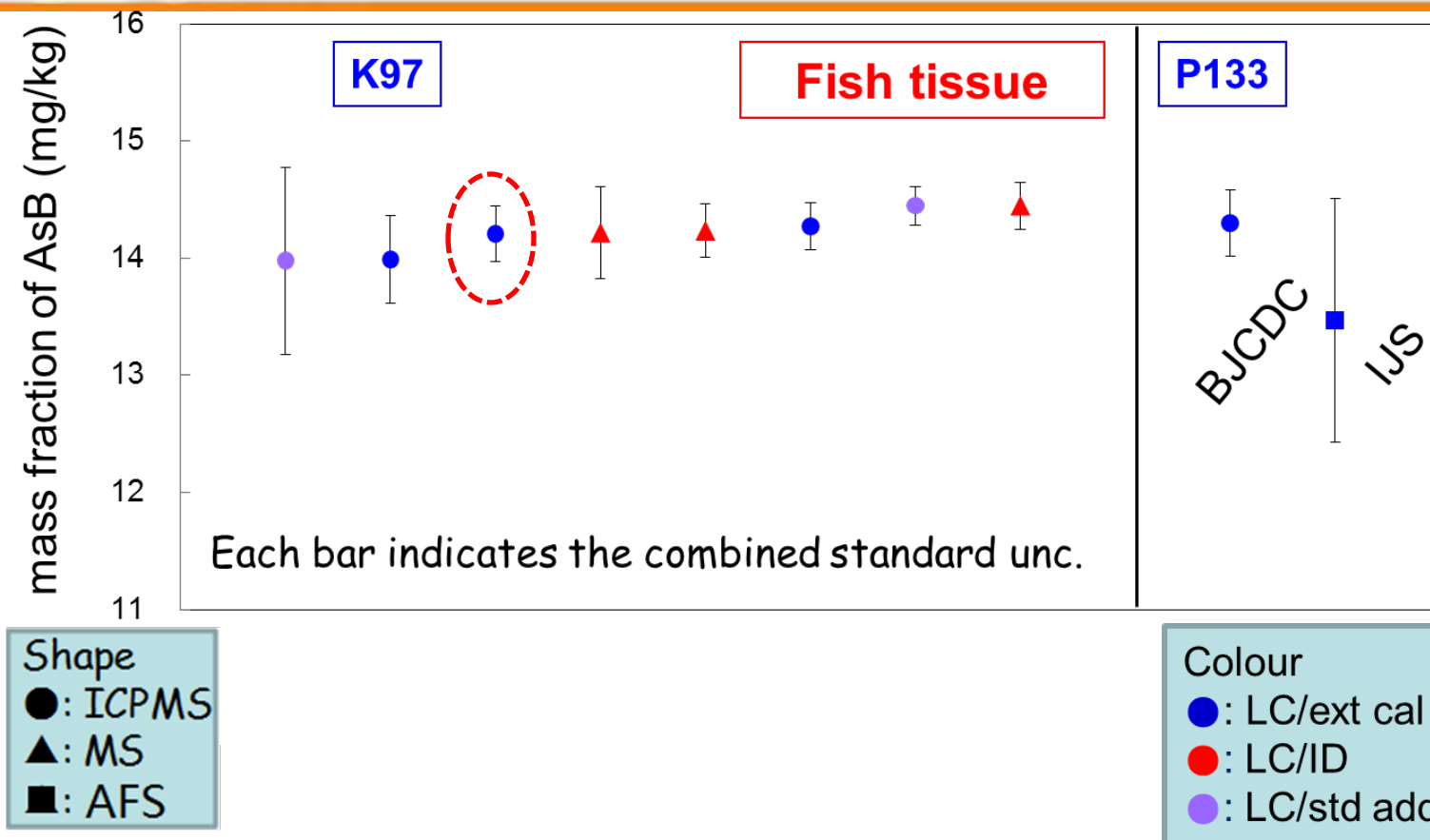


Figure 2 CCQM-K97/P133 All Results of AsB in fish tissue

The results by different methods (organic IDMS and inorganic LC-ICPMS) were comparable. Organic IDMS could be used as a well-validated reference method for speciation of organo-metallic measurement, especially for some monoisotopic elements. The research open a new field across inorganic and organic measurement

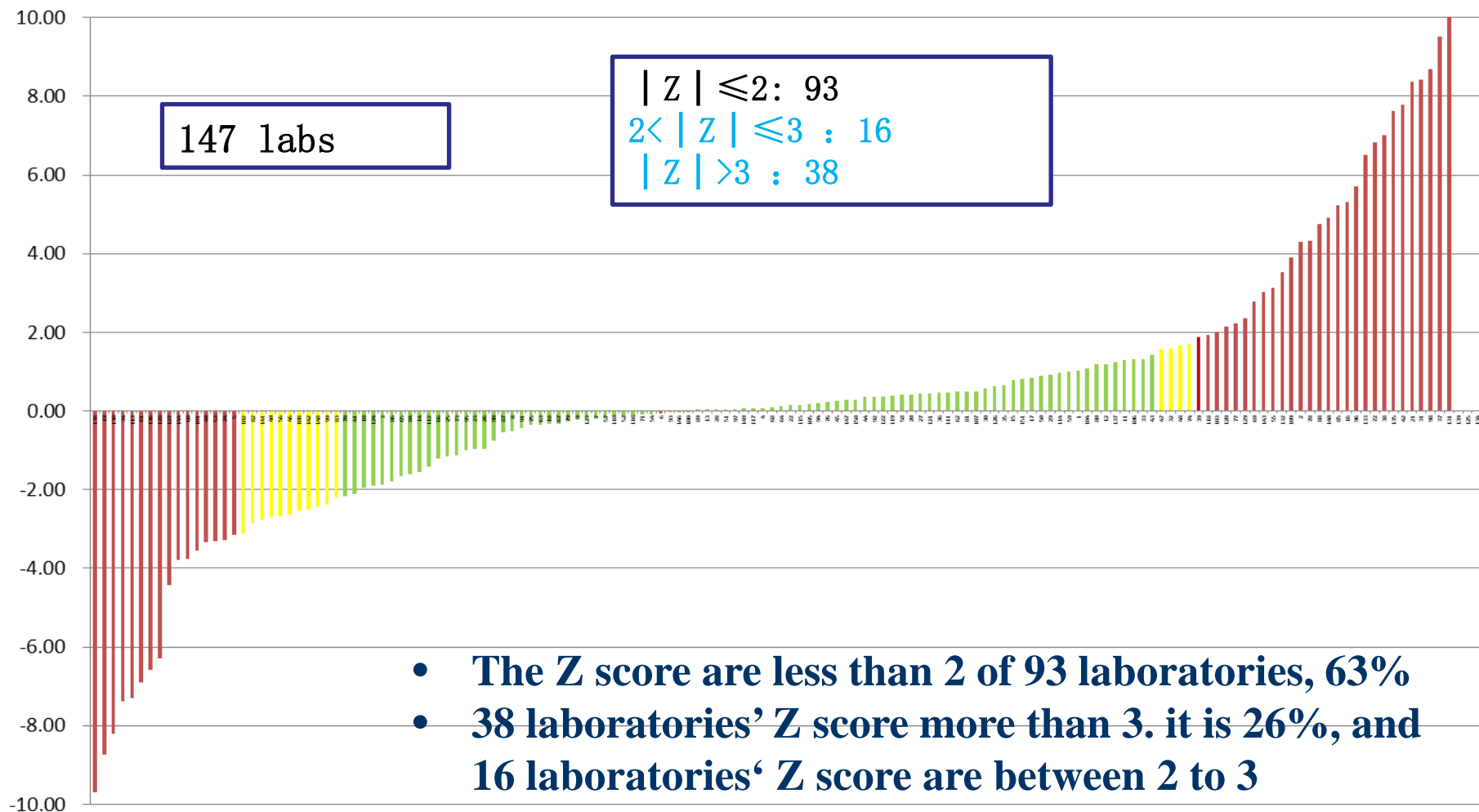
Over 30 National PTs Provided in last 5years

Year	Name	Number of participants
2010	Determination of chloropropanol in soy sauce	23
2011	Determination of benzo[a]pyrene in edible oil	25
2011	Determination of sodium thiocyanate in liquid milk	25
2012	Determination of lead and chromium in solution	31
2012	Determination of lead and chromium in milk powder	31
2012	Determination of lead and chromium in tea	31
2012	Determination of pigments in wine	130
2012	Determination of cadmium in rice	510
2013	Determination of aluminum in vermicelli	40
2013	Determination of 16 kinds of phthalate acid esters in Chinese spirit	15
2013	Determination of aflatoxin M1 in liquid milk	15
2014	Determination of 16 kinds of phthalate acid esters in chinese spirit	37
2014	Total Hg in infant poder	147
2014	Determination of lead in infant powder	37
2015	Cu, Se and protein content in infant formula milk powder	10

- Improving the measurement capabilities of testing labs in food field through provided matrix CRMs and technical training.

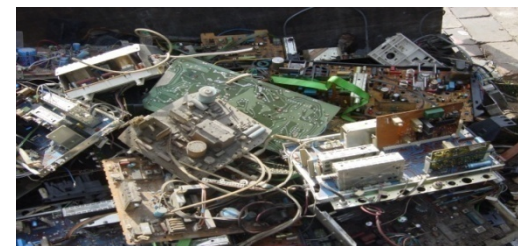
Total Hg in infant milk powder -2014

Z score evaluation



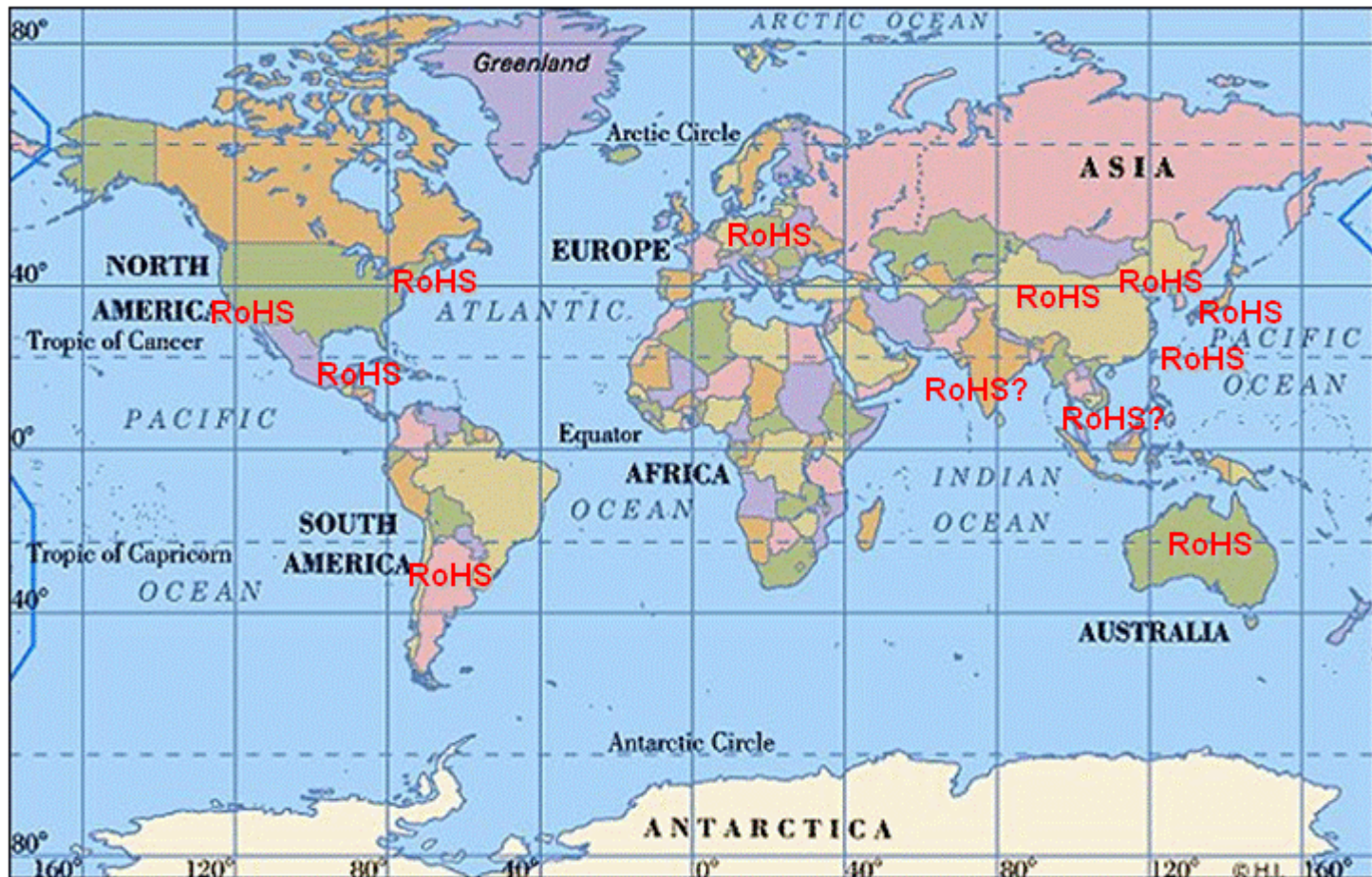


- **Chemical Metrology Research
for RoHS Testing
in NIM, China**



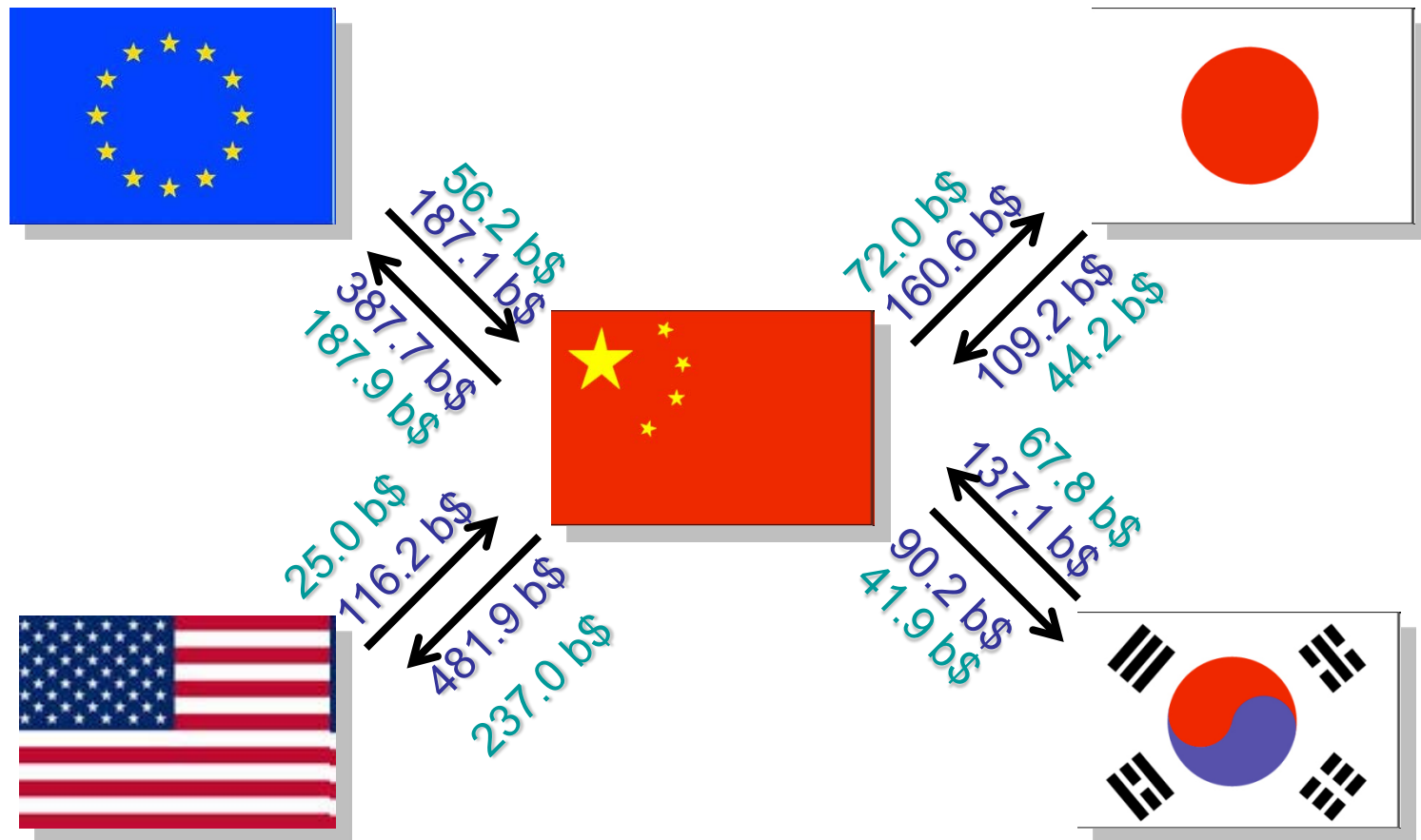
RoHS Directive

(**R**estriction **o**f the use of certain **H**azardous **S**ubstances in Electrical and Electronic Equipment)



Why RoHS is important for China?

Major trading partners



2015 trade data from <http://www.mofcom.gov.cn/>

Blue data are total trade amount,
green data are electrical and electronic products trade amount.

RoHS CRMs in NIM China

➤ Heavy metals in plastic

CRM No	Matrix	Characteristics	levels
GBW08404 ~ 08406	PP(pellet)	Cd/Cr/Hg/Pb	3 levels
GBW(E)081121~ 081123	PP(disks)	Cd/Cr/Hg/Pb	3 levels
GBW08407 ~ 08411	ABS (pellet)	Cd/Cr/Hg/Pb	5 levels
GBW(E)081634 ~ 081638	ABS (disks)	Cd/Cr/Hg/Pb	5 levels
GBW08417 ~ 08421	PVC (pellet)	Cd/Cr/Hg/Pb	5 levels
GBW(E)082144 ~ 082148	PVC (disks)	Cd/Cr/Hg/Pb	5 levels



➤ Heavy metals in glass

Heavy metals (Pb,Cr,(Hg),Cd, As,Ba) in glass powder include 3 levels

	Matrix	Cd (mg/kg)	Cr (mg/kg)	As (mg/kg)	Pb (mg/kg)	Ba (mg/kg)	(Hg) (mg/kg)
GBW08414	Glass	0.031	3.17	0.056	4.3	133	(0.003)
GBW08415	Glass	9.6	95	95	83	228	(0.20)
GBW08416	Glass	94	963	980	784	1078	(1.35)



➤ Pb, Cd in lead free solder

CRM No	Matrix	Cd(mg/kg)	Pb(mg/kg)
GBW08422	Solder	27.2	261
GBW08423	Solder	127	1024



➤ Alloy CRMs for XRF

Matrix		Discs amount	Elements	Range (mg/kg)
Copper Alloy	Pure copper	11	Pb	20-1300
	Aluminum Brass	5	Pb	200-1500
	Brass	7	Pb	20-3100
Steel Alloy	Alloy structural steel	7	Cr, Pb	5000-20000
Aluminum Alloy	Deformation of Aluminum	6	Pb,Cd,Hg	10-600
	Casting Aluminum Alloy	6	Pb,Cr	10-1500
Tin Alloy	Tin Alloy	5	Pb	250-7000
Zinc Alloy	Zinc Alloy	5	Pb,Cd	20-1000



Zinc Alloy



Aluminum Alloy



Tin Alloy



Copper Alloy

➤ PBDE standard solutions

CRM No	Solution	Characteristics
GBW08709	Deca-BDE in iso-octane/toluene	PBDE-209
GBW(E)081124	Industrial Penta-BDE in iso-octane	PBDE-47/100/99/85/154/153
GBW(E)081125	Industrial octa-BDE in iso-octane	PBDE-53/183/197/203/196/207
GBW(E)082213	Toluene	PBB209
GBW(E)082212	isooctane	PBB209
GBW(E)082214	isooctane	HBB



➤ PBDE in Plastic

CRM No	Matrix	Characteristics
GBW08412	Polyethylene	Decabrominated diphenyl ether
GBW08413	Polyethylene	Decabrominated diphenyl ether

International cooperation and comparisons for MRA



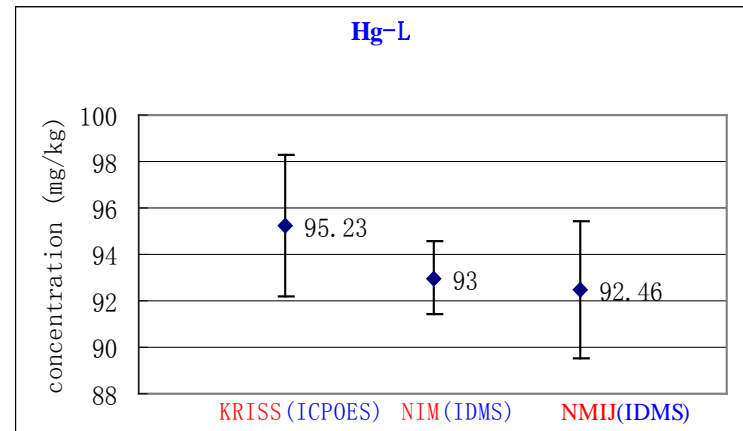
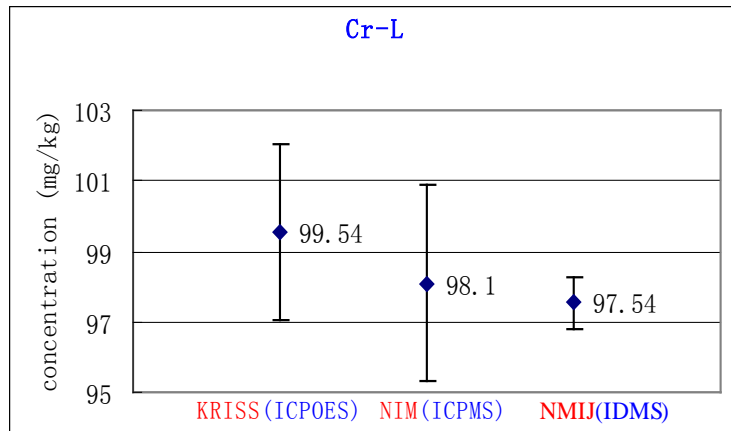
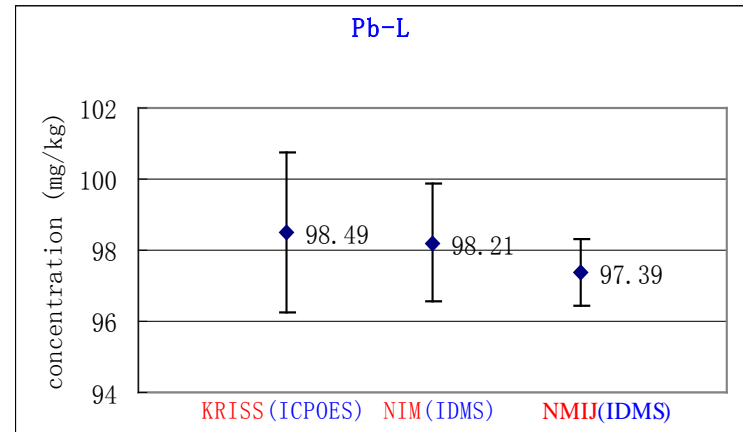
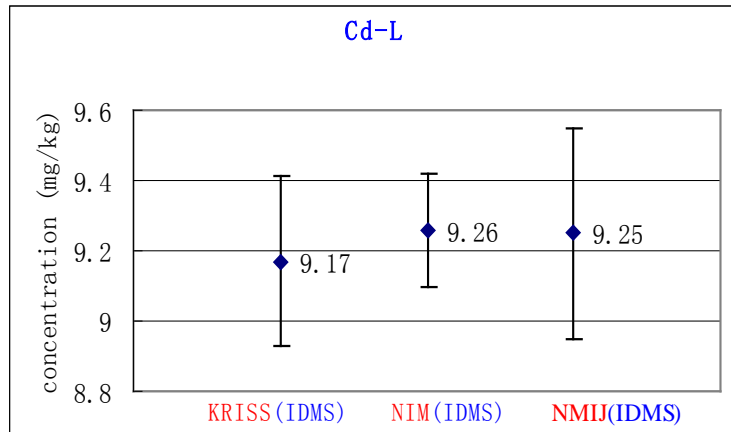
ACRM-WG3
RoHS Group

Asian Collaboration on Certified Reference Materials

*NIM, NMIJ, KRISS established a Regional Network for RoHS
CRMs collaboration*

Action have done	Subject	Institute in charge	Start date
#1	Cd, Cr, Hg and Pb in ABS	NMIJ	2005
#2	Cd, Cr, Hg and Pb in PP	NIM	2006
#3	Cd, Cr, Hg and Pb in PP	KRISS	2006
#4	PBDE solutions (209 and 206)	KRISS	2007
#5	Pb in Pb-free solder	NMIJ	2008
#6	BDE209 in PE	NIM	2009
#7	BDE209 in PS	KRISS	2010
#8	Phthalates in PVC	KRISS	2011
#9	Phthalates in PVC	NMIJ/NIM	2014

Asian Collaboration on CRM - *NIM RoHS CRM for Co-validation*



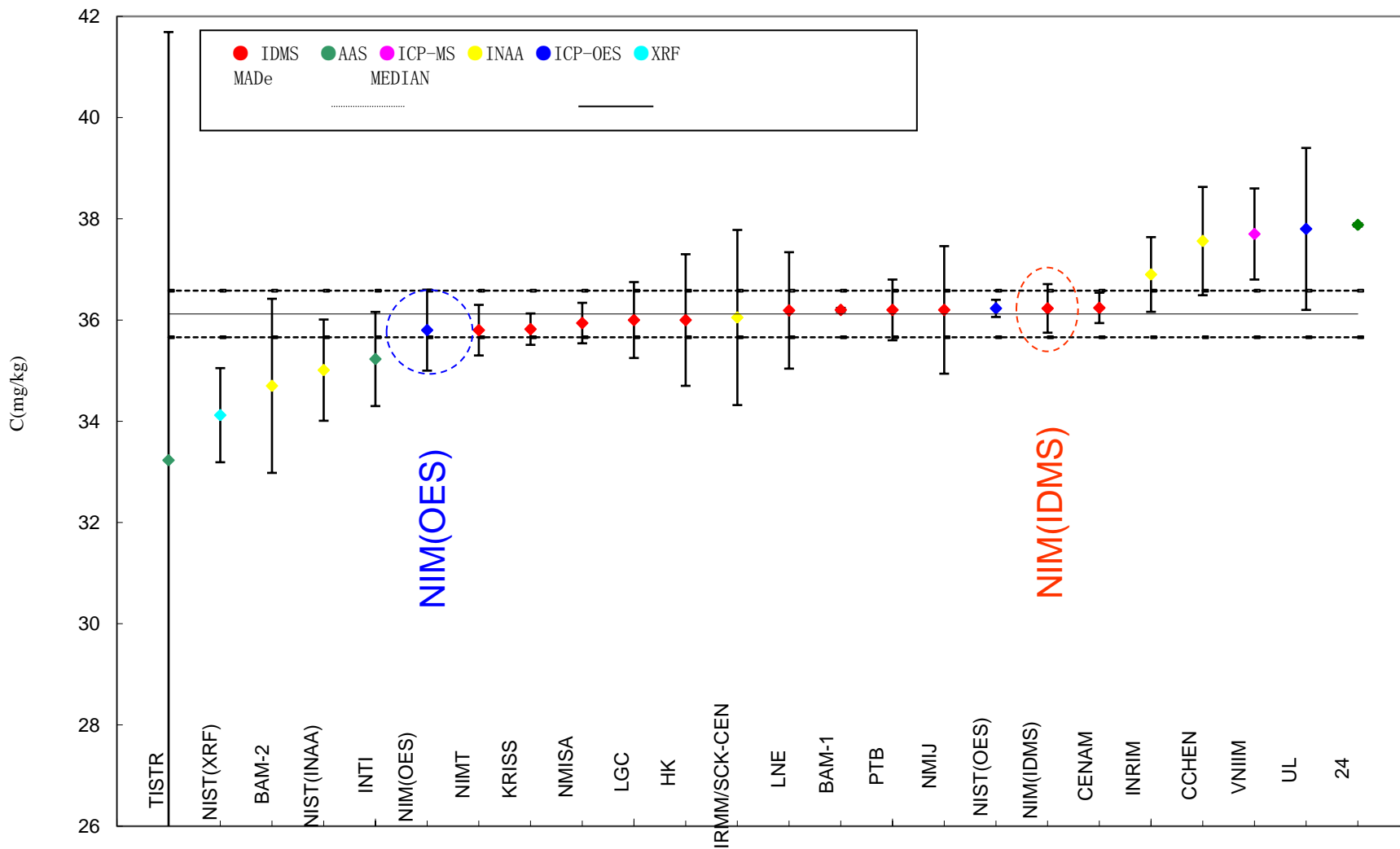
International collaboration and co-validation work can check the established national metrology tractability system, link with other NMIs, make global comparable, and get mutual recognition.

International comparisons

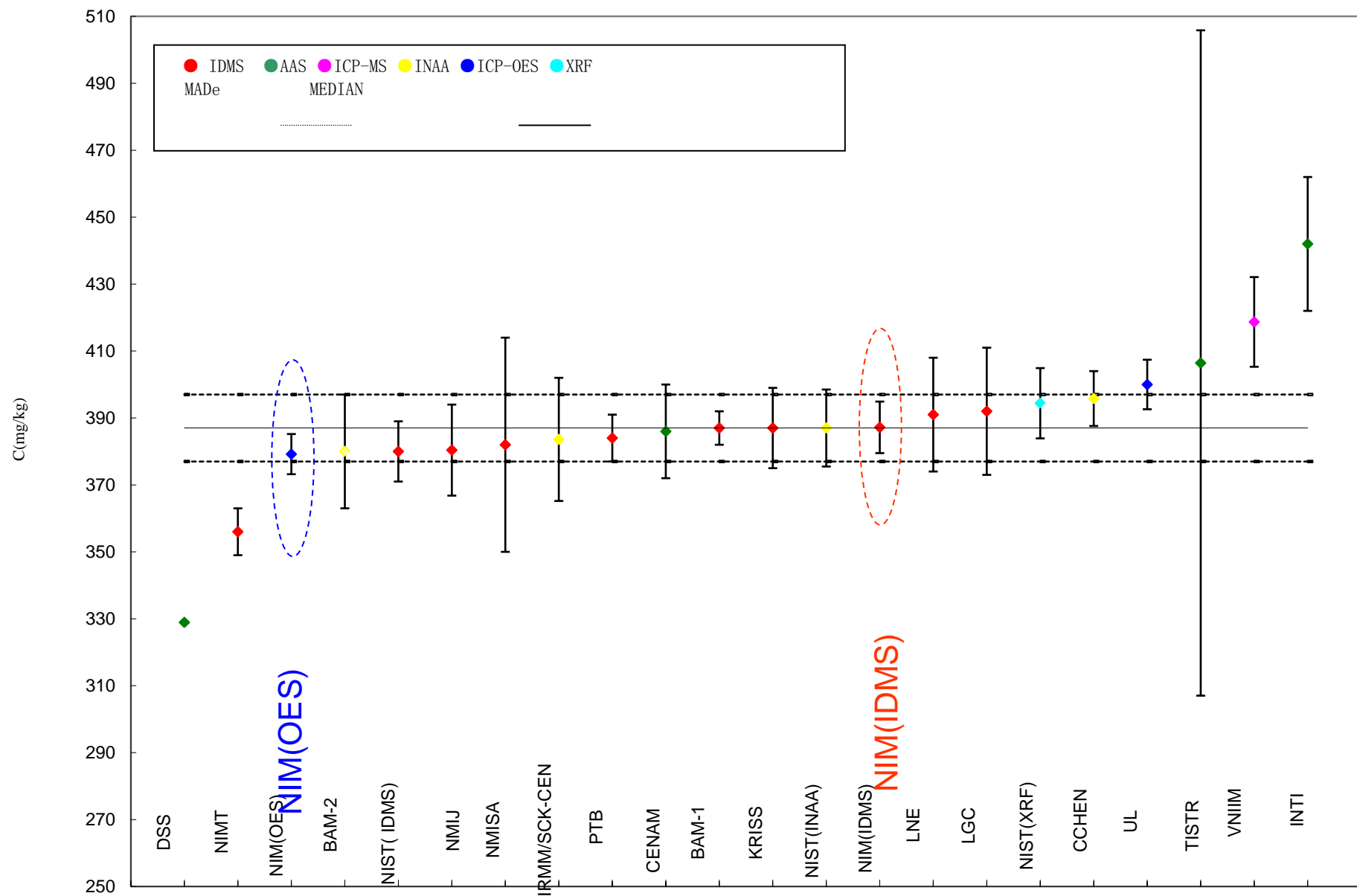
Action	Coordinating Lab	Contents
CIPM/CCQM	NIM, NMIJ, KRISS	CCQM-P106 Cd, Cr, Hg and Pb in Polypropylene
CIPM/CCQM	IRMM	CCQM-P114 PBDE & PBB in Polypropylene
CIPM/CCQM	NMIJ, NIM, KRISS	CCQM-P119 lead in lead-free solder
CIPM/CCQM	NMIJ, NIM, KRISS	CCQM-K88 & P125 lead in lead-free solder

International comparisons

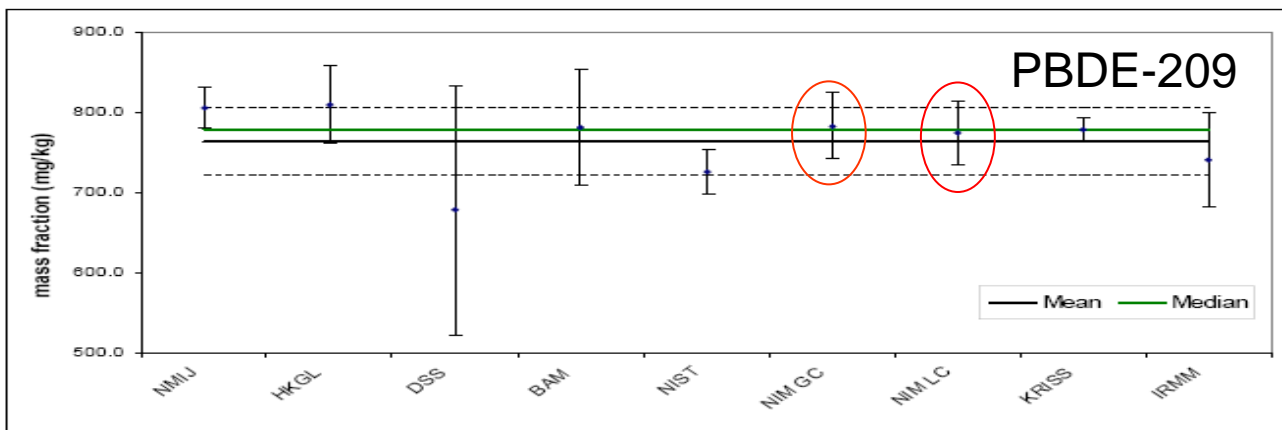
CCQM-P106 Cd in polypropylene



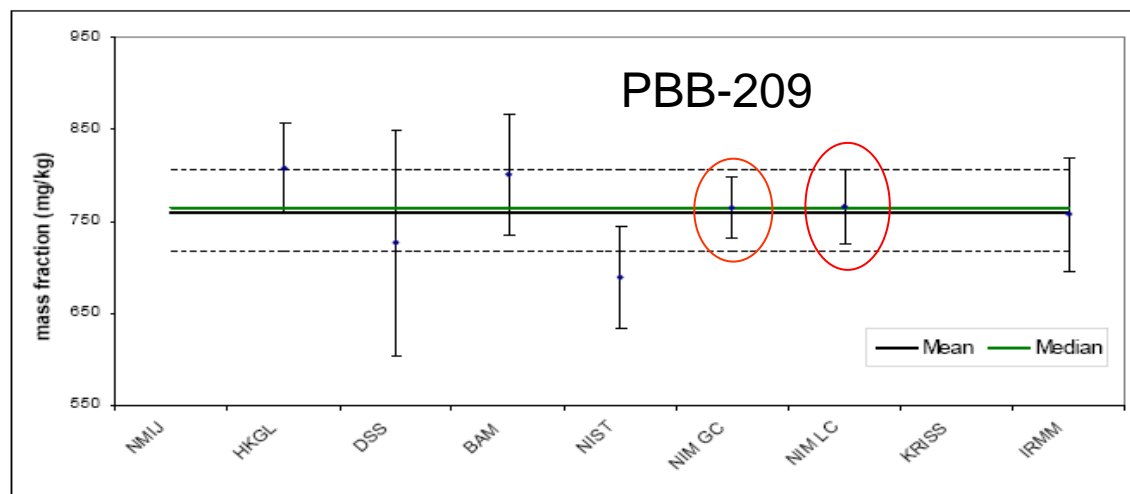
CCQM-P106 Hg in polypropylene



Results of CCQM-P114-Determination of several BFRs in PP



Median 778.80 mg/kg
Mean 764.16 mg/kg
Std.dev. 42.13 mg/kg
RSD% 5.5

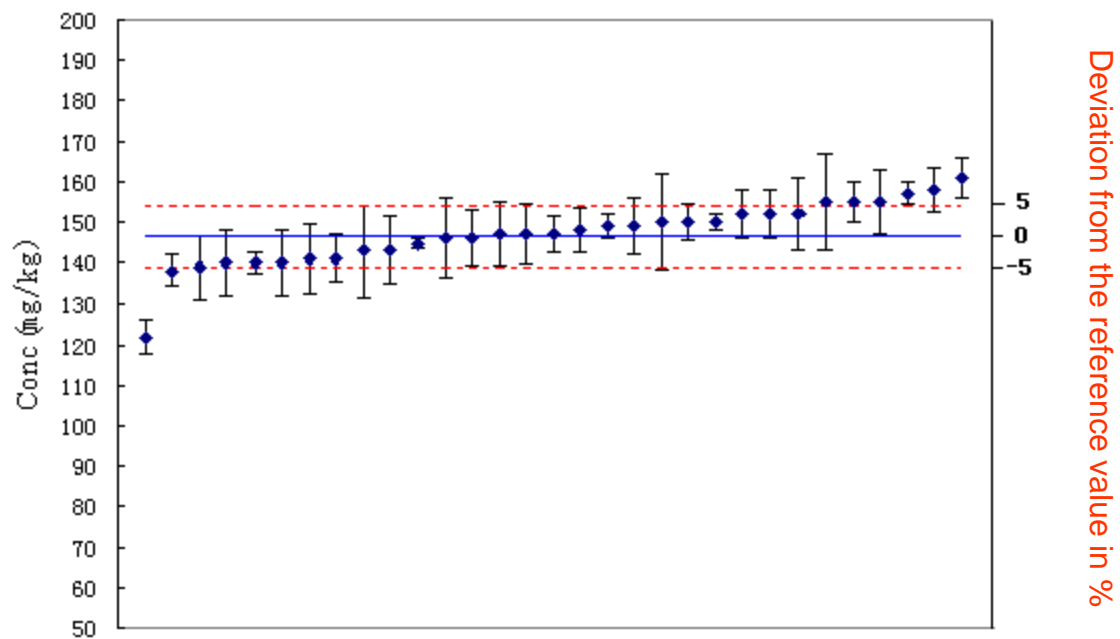
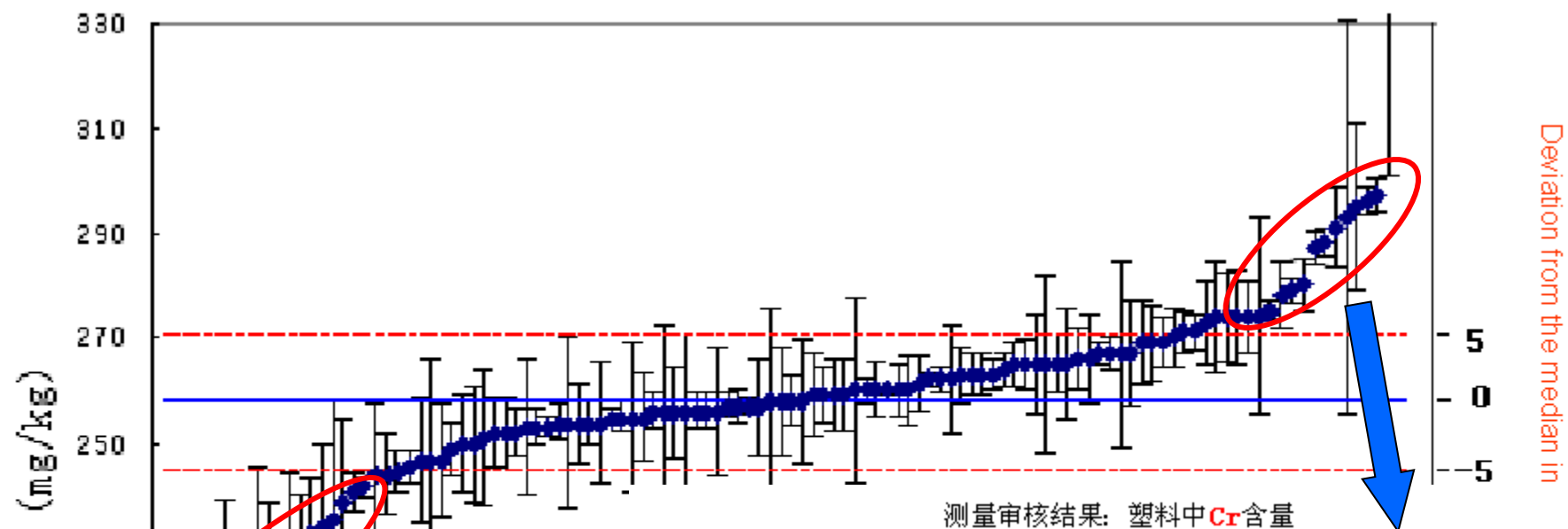


Median 764.10 mg/kg
Mean 758.25 mg/kg
Std.dev. 41.25 mg/kg
RSD% 5.4

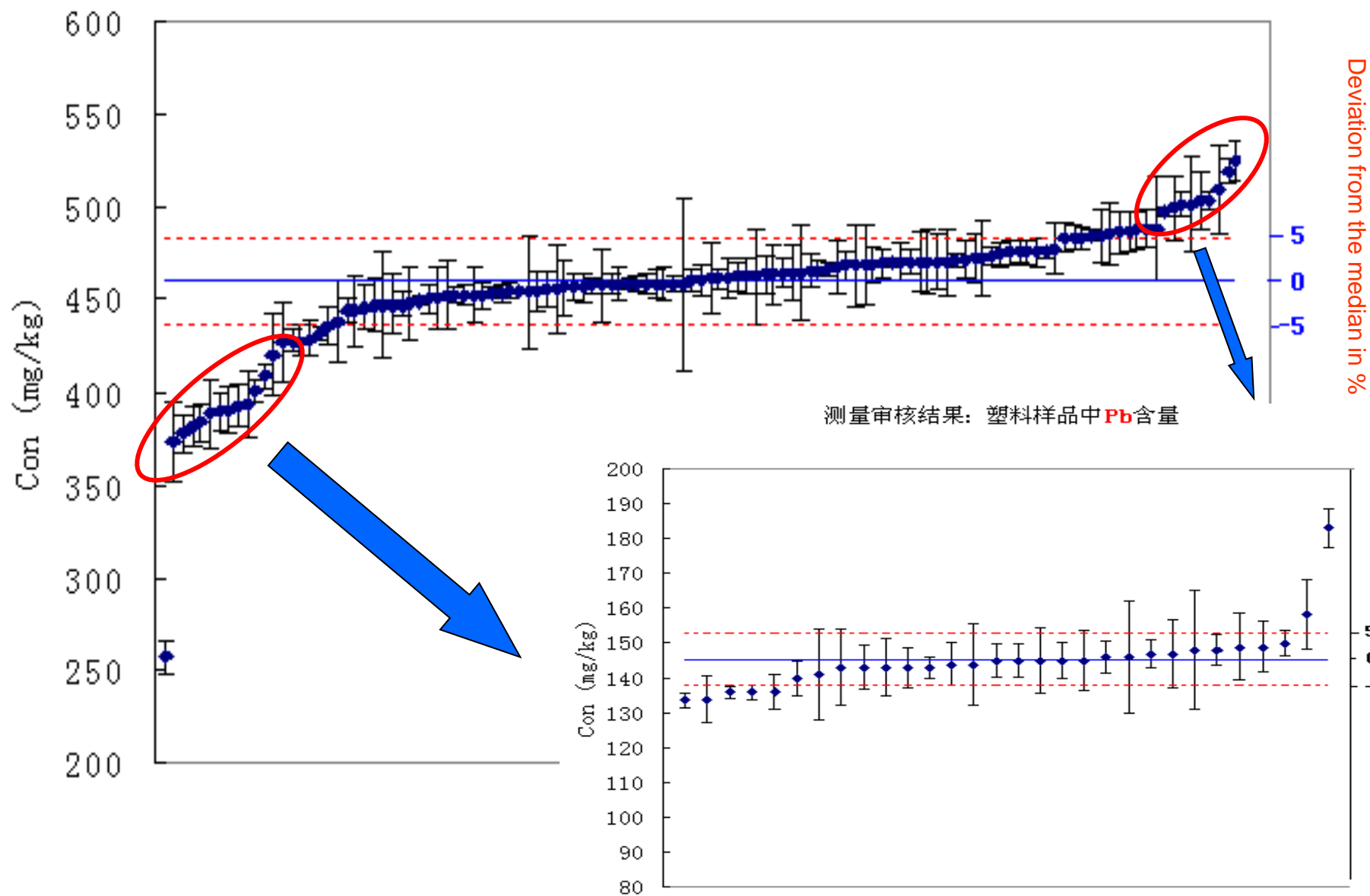
National proficiency tests

- NIM has coordinated 6 PTs in China, about 700 labs/times had participated.
- **CNAS T0329** Determination of heavy metals in PP (123 labs)
- **CNAS T0331** PBDE in Octane Solution (79 labs)
- **CNAS T0399** Determination of heavy metals in ABS (109 labs)
- **CNAS T0398** PBDE in ABS (88 labs)
- **CNAS T0559** PBDES in PS (110 Labs)
- **CNAS T0620** Heavy metals in PVC (170 Labs)

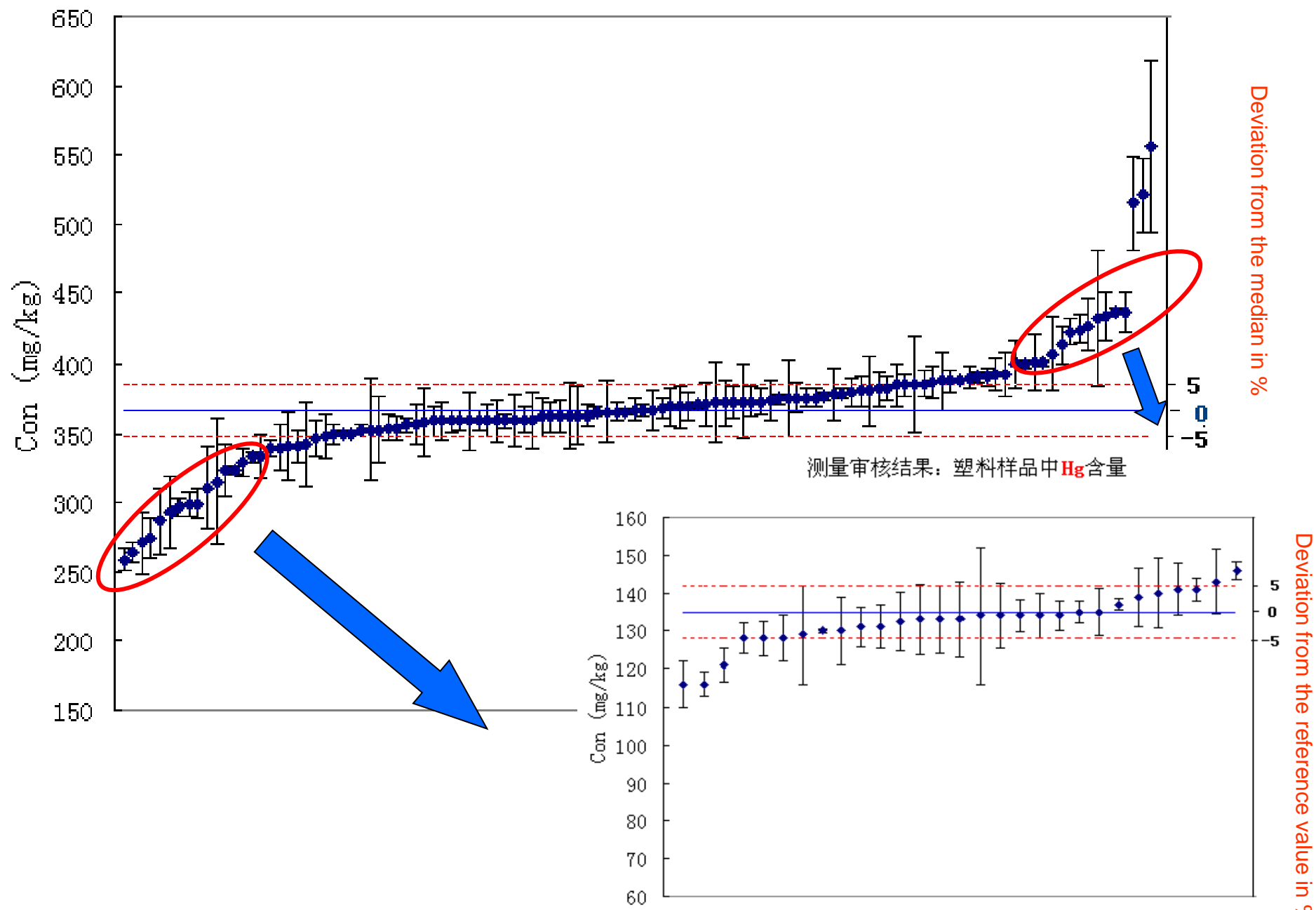
CNAS T0329: Cr in Plastic



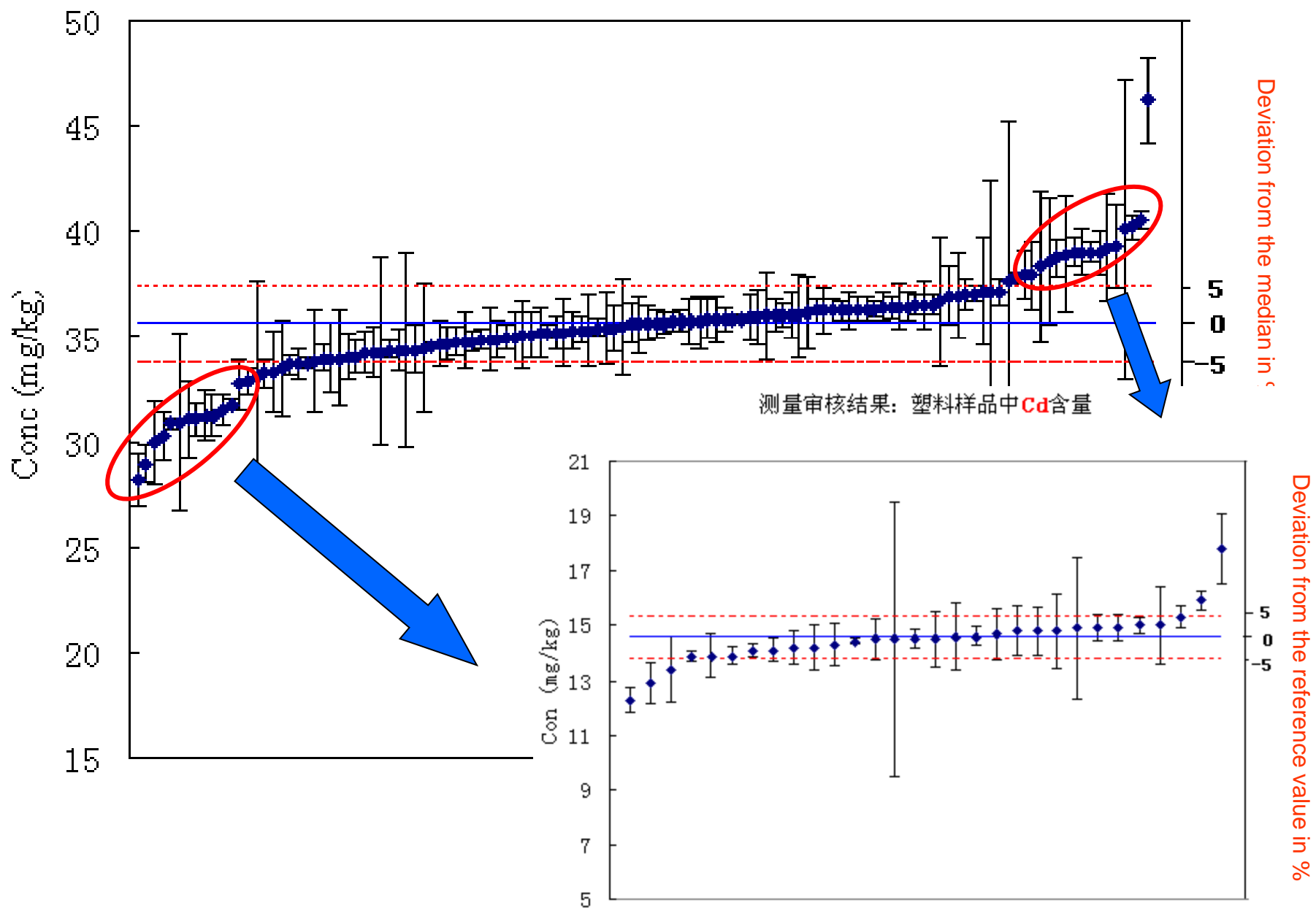
CNAS T0329: Pb in Plastic



CNAS T0329: Hg in Plastic

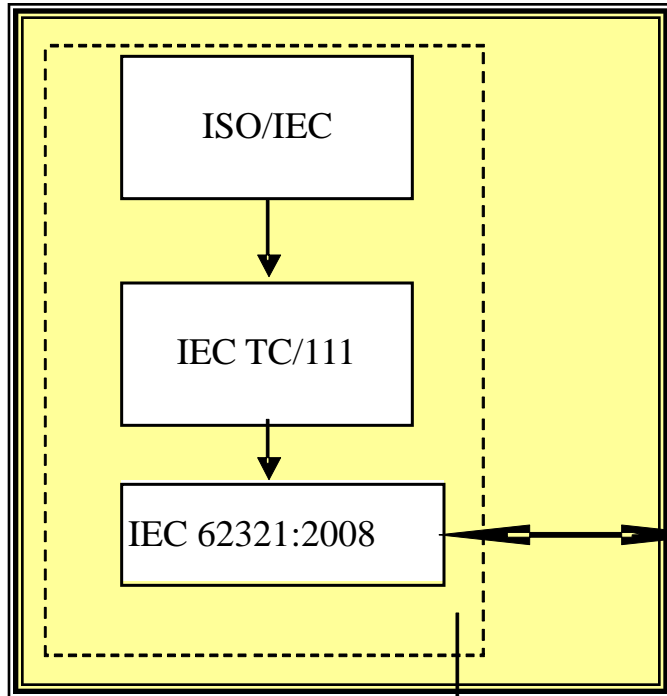


CNAS T0329: Cd in Plastic

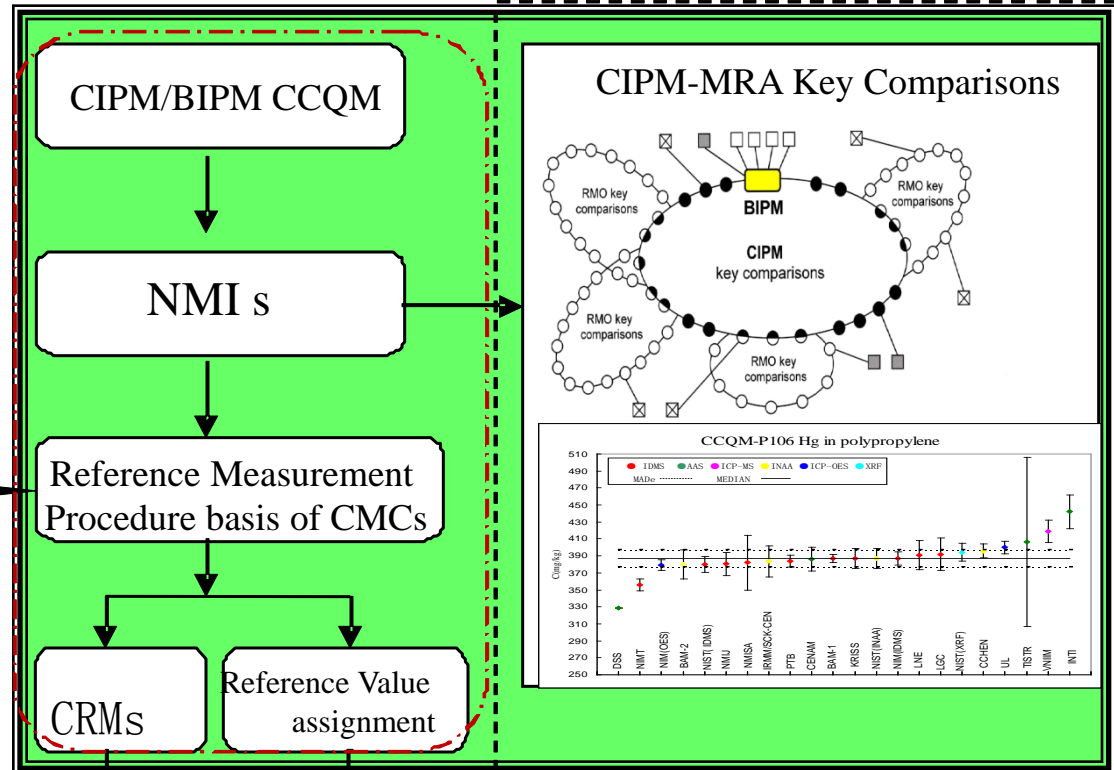


BIPM/CIPM/ISO/IEC/ILAC

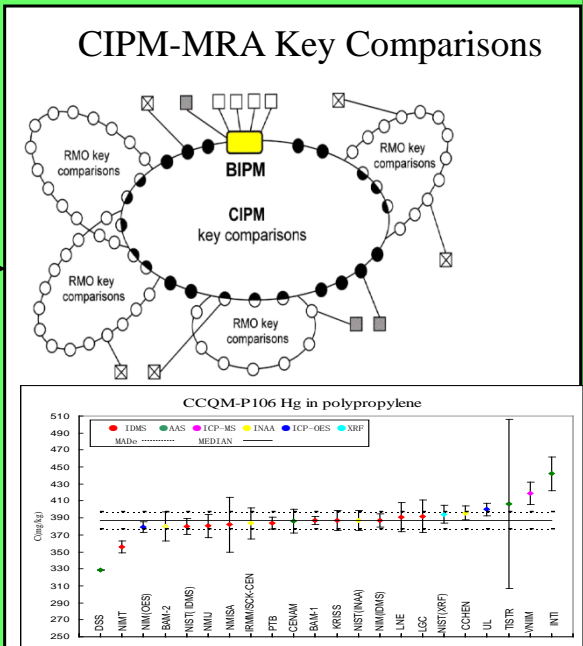
Standardization
ISO/IEC



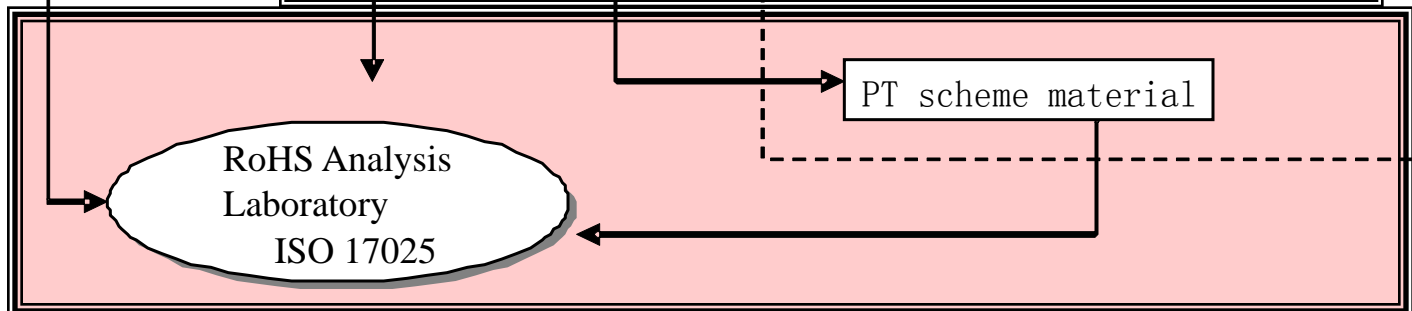
Metrology
CIPM/BIPM



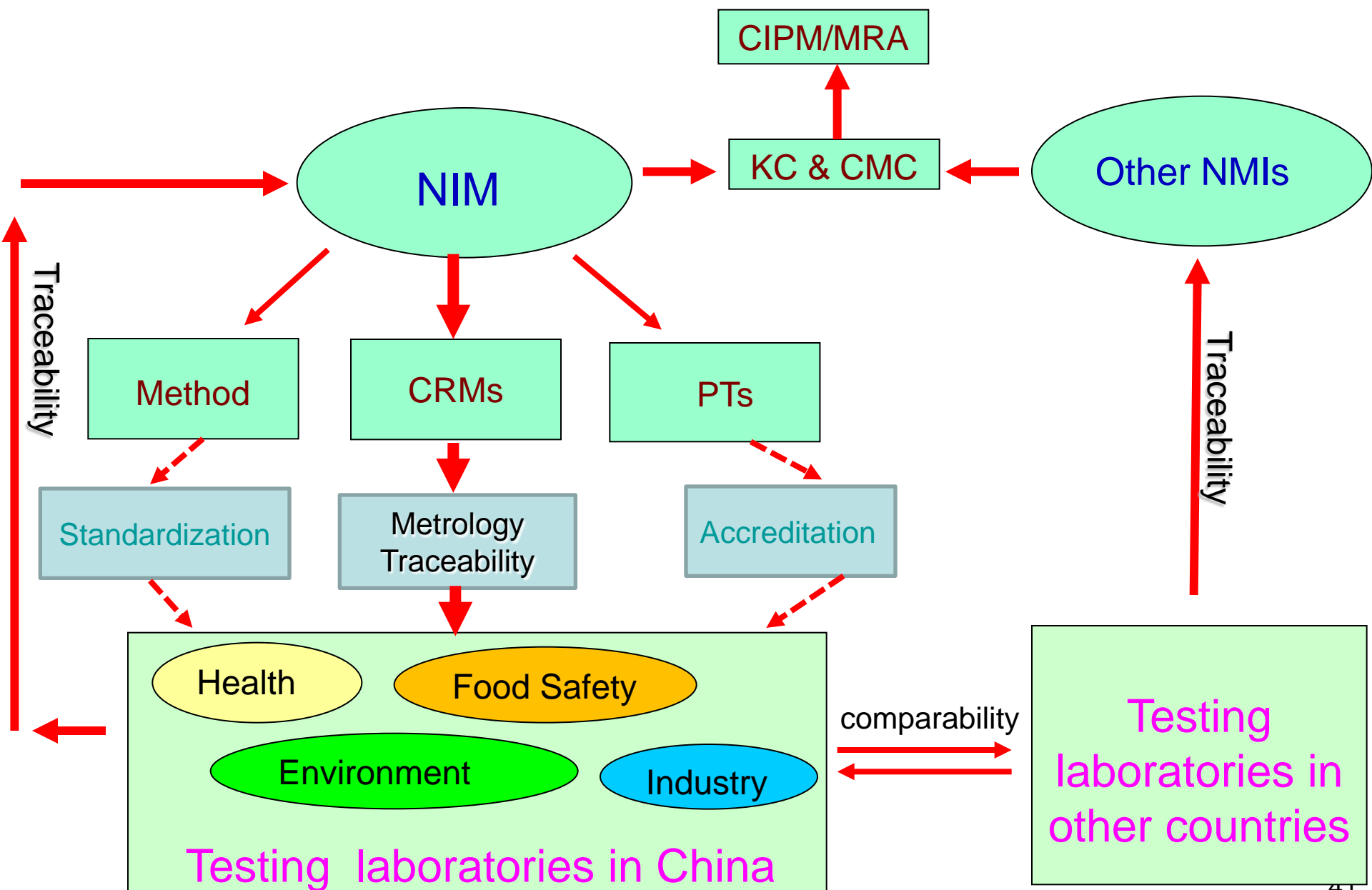
CIPM-MRA



Lab Accreditation
Conformity
Assessment
ILAC

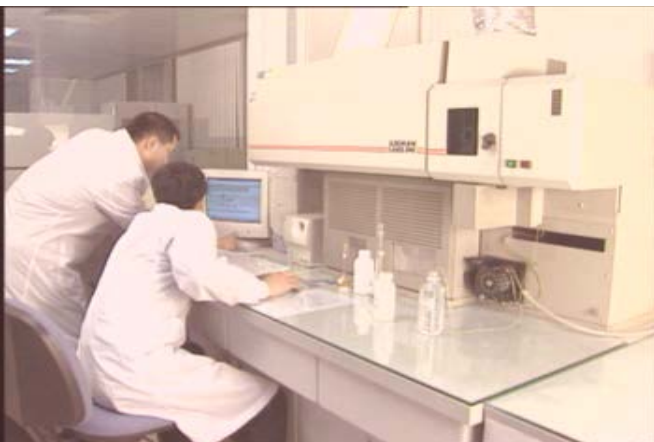


National Measurement Traceability System



Testing Labs in China are more than 30,000

NIM provided CRMs service 450,000 units in 2015



One Measurement, One Management, Accepted all over the World.

Conclusion

- The validity and reliability of results basically come from the measurement traceability.
- This national traceability system will help to enhance the nation's measurement capability, facilitate international trade, promote national economic performance and improve welfare of the people.
- Consolidating the Metrology, Standardization and Conformity Assessment as “national quality driven development strategy” technical foundation , will support high-quality of economic and social development.

Thank You
for Your Attention! ! !



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