New Practice on Methods for Determination of U Th and Ra in Uranium Geology and NORM Samples

GUO Dongfa's Team

Analytical Laboratory
Beijing Research Institute of Uranium Geology, China

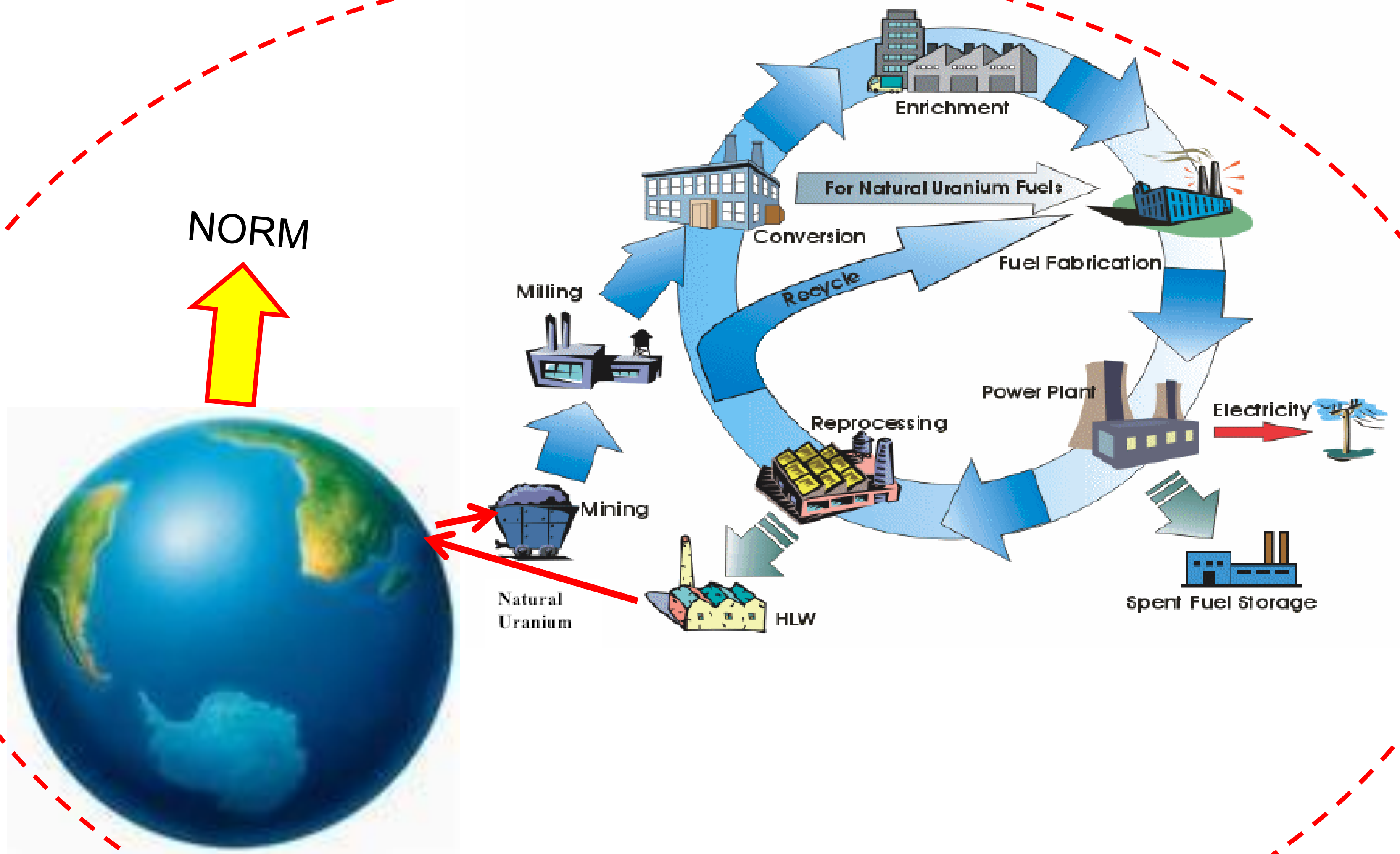
2017-09-23

Outline

1. Background for uranium geology (UG) and NORM sample analysis
2. New Practice on Methods for Determination of U Th Ra
3. Summary

NORM: naturally occurring radioactive materials

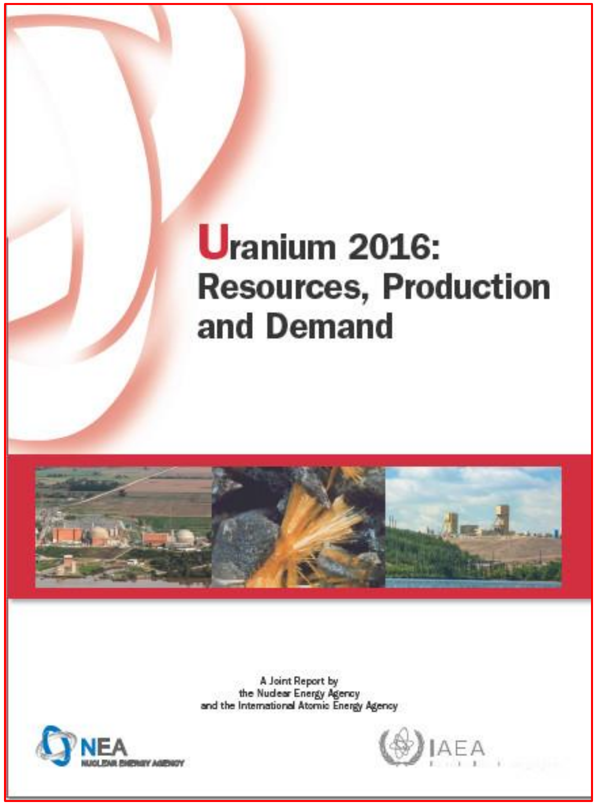
1. Background for UG & NORM S.A.

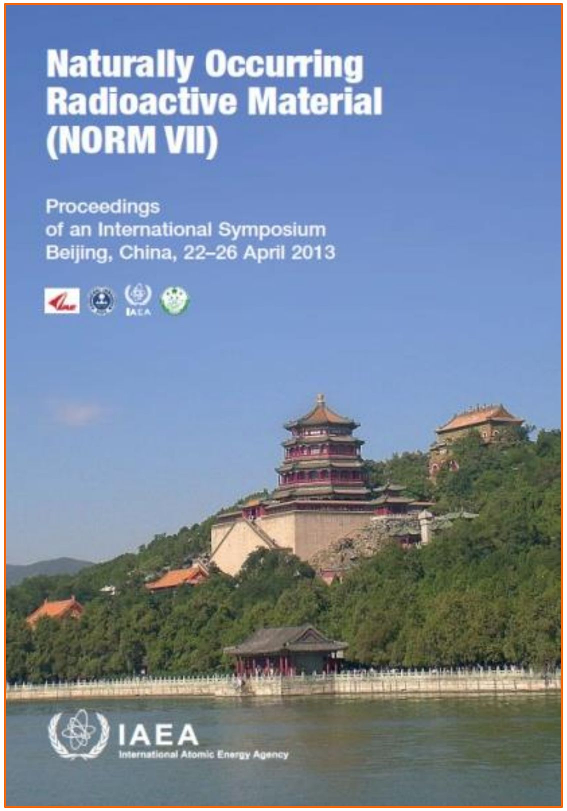


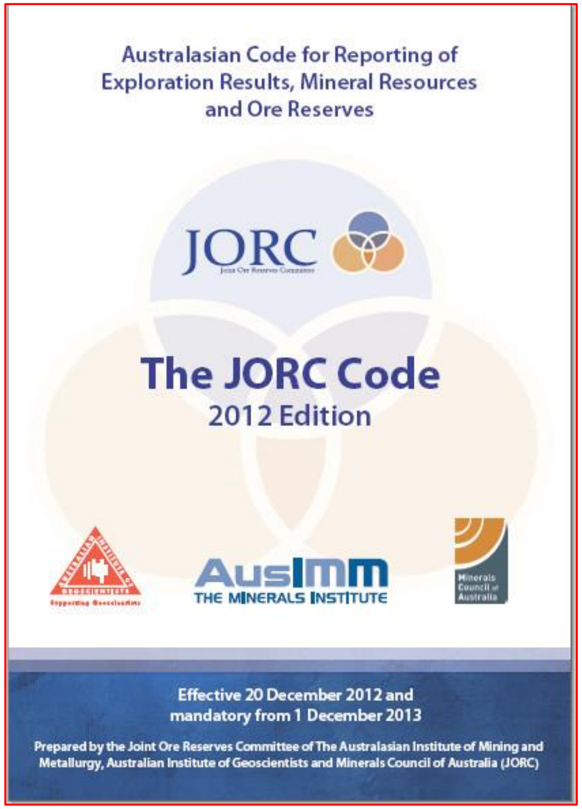
UG & NORM Samples come from……

1. Background for UG & NORM S.A.

Very Important References





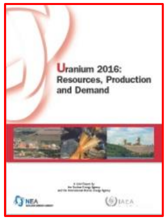


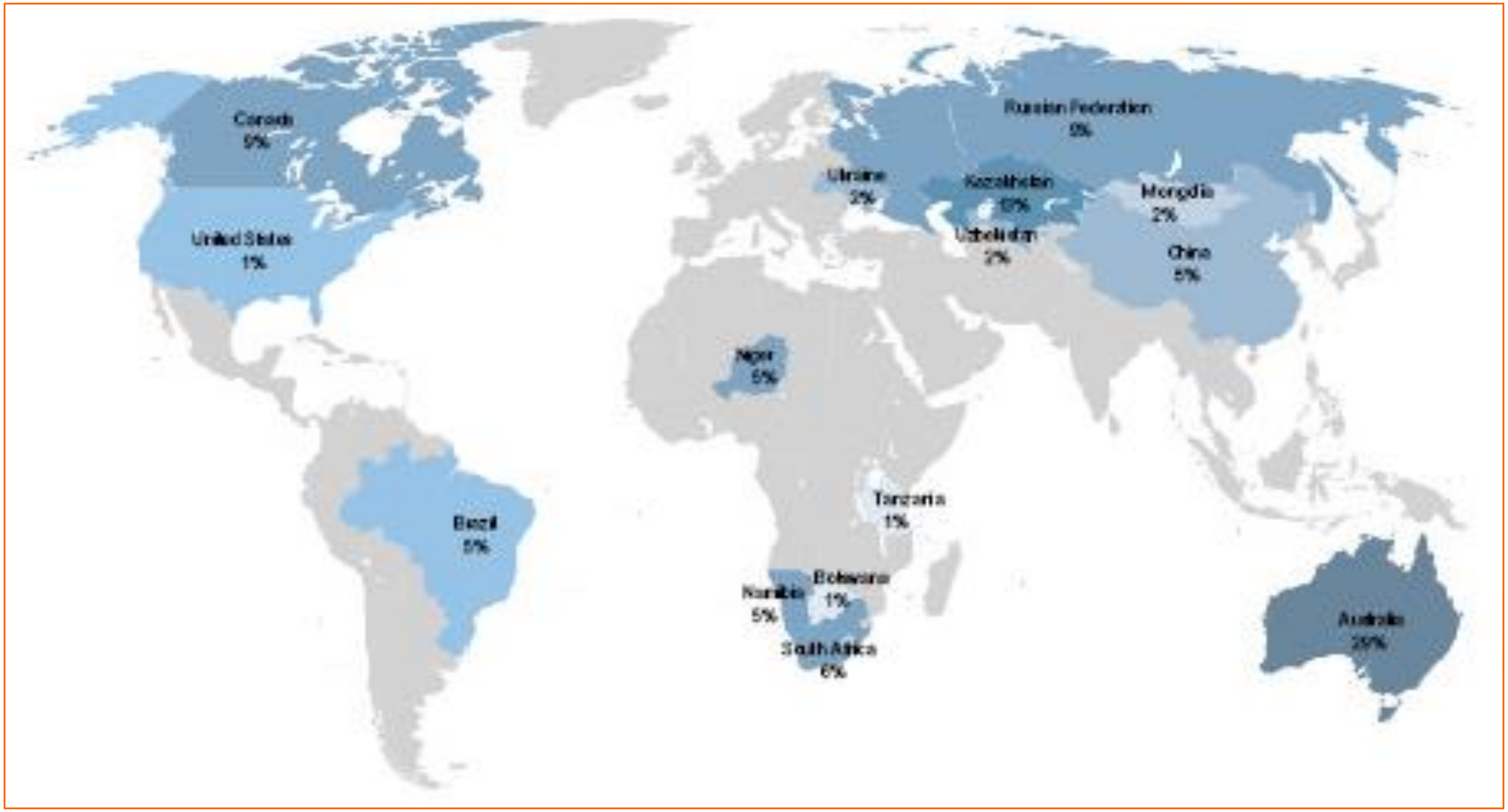
中核集团核工业北京地质研究院
CNNC Beijing Research Institute of Uranium Geology

1. Background for UG & NORM S.A.

Very Important References

Global distribution of identified resources (<USD 130/kg U as of January 2015)





1. Background for UG & NORM S.A.

Very Important References

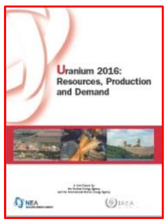
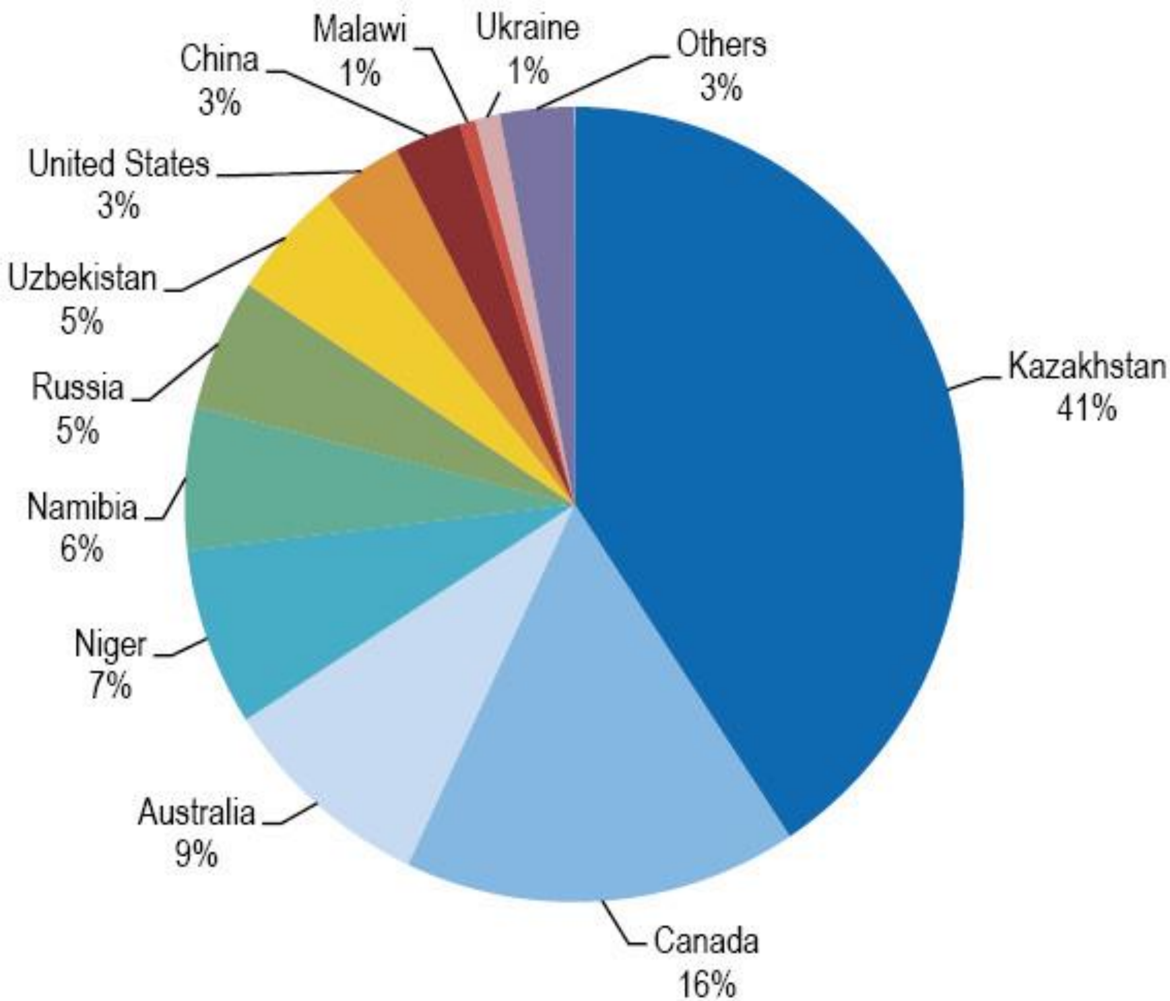
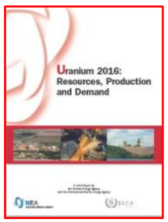


Figure 1.5. Uranium production in 2014: 55 975 tU



1. Background for UG & NORM S.A.

Very Important References



Uranium production centre technical details

(as of 1 January 2015)

	Centre #1	Centre #2	Centre #3	Centre #4	Centre #5		Centre #6
Name of production centre	Fuzhou	Chongyi	Yining	Lantian	Benxi		Shaoguan
Production centre classification	Existing	Existing	Existing	Existing	Existing	Existing	Existing
Date of first production	1966	1979	1993	1993	1996	2007	N/A
Source of ore:							
Deposit name(s)			Kujieertai	Lantian	Benxi	Qinglong	
Deposit type(s)	Volcanic	Granite	Sandstone	Granite	Granite	Volcanic	Granite
Resources (tU)	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Grade (% U)	N/A	N/A	N/A	N/A	N/A	N/A	N/A
Mining operation:							
Type (OP/UG/ISL)	UG	UG	ISL	UG	UG	UG	UG
Size (tonnes ore/day)	700	500	N/A	200	100	200	500
Average mining recovery (%)	92	90	N/A	80	85	85	90
Processing plant:							
Acid/alkaline	Acid	Acid	Acid	Acid	Acid	Acid	Acid
Type (IX/SX)	IX	IX	IX	IX	SX	IX	SX
Size (tonnes ore/day); for ISL (l/day or l/h)	700	500	N/A	N/A	N/A	N/A	N/A
Average process recovery (%)	90	84	N/A	90	90	96	90
Nominal production capacity (tU/year)	350	200	480	100	120	100	200
Plans for expansion	Up to 500	Up to 300	Up to 800	N/A	N/A	Up to 200	Up to 300
Other remarks	N/A	N/A	N/A	N/A	N/A	N/A	N/A

China's Uranium Production

Very Important References

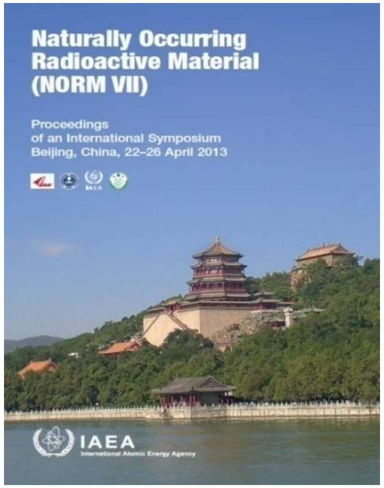
(a) If, in every process material, the activity concentrations of all radionuclides in the ^{238}U and ^{232}Th decay series are 1 Bq/g or less and the activity concentration of ^{40}K is 10 Bq/g or less, the material is not regarded as radioactive material (NORM), the industrial activity is not regarded as a practice and the requirements for **existing exposure situations** apply.

(b) If, in any process material, the activity concentration of any radionuclide in the ^{238}U or ^{232}Th decay series exceeds 1 Bq/g, or if the activity concentration of ^{40}K exceeds 10 Bq/g, that material is regarded as radioactive material (NORM), the industrial activity is regarded as a practice and the requirements for **planned exposure situations** apply.

KEYNOTE ADDRESS

MANAGING EXPOSURE TO NATURAL SOURCES: INTERNATIONAL STANDARDS AND NEW CHALLENGES

P.P. Haridasan
International Atomic Energy Agency,
Vienna



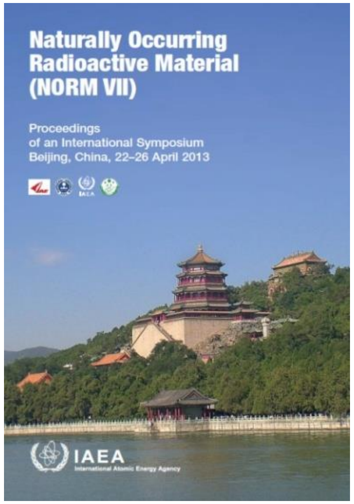
1. Background for UG & NORM S.A.

Very Important References

TABLE 18. SUMMARY OF OCCUPATIONAL EXPOSURES TO NATURAL SOURCES, 1996–2000

	No. of monitored workers	Annual av. individual dose (mSv)	Annual av. collective dose (man Sv)
Coal mines	6 500 000	2.40	14 600
Metal mines	1 000 000	5.53	5 530
Other mines	3 000 000	0.688	2 060
Underground workplaces other than mines	50 000	1.56	78.0
Aircrews	38 400	2[a] 6[b]	77.7
Overall	10 588 400	2.1	22 300

[a] Domestic routes.
[b] International routes.



Invited Paper

NORM EXPOSURE IN CHINA

SENLIN LIU*, ZIQIANG PAN**

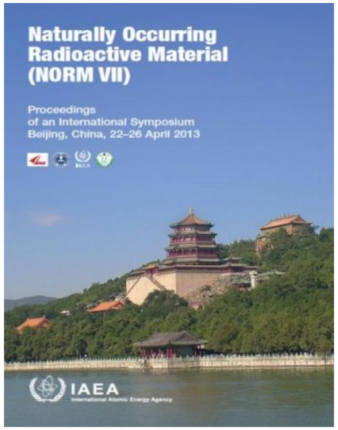
* China Institute of Atomic Energy,
Email: slliu65_325@sohu.com

** China National Nuclear Corporation

Beijing, China

1. Background for UG & NORM S.A.

Very Important References



(1) Extraction of rare earth elements;
(2) Production and use of thorium and its compounds;
(3) Production of niobium and ferroniobium;
(4) Mining of ores other than uranium ore;
(5) Production of oil and gas;
(6) Manufacture of titanium dioxide (TiO_2) pigments;
(7) The phosphate industry;
(8) Zircon and zirconia industries;
(9) Production of tin, copper, aluminium, zinc, lead, and iron and steel;
(10) Combustion of coal;
(11) Water treatment.

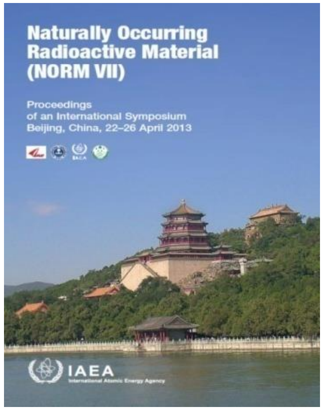
IAEA List of NORM

INTERNATIONAL ATOMIC ENERGY AGENCY, Assessing the Need for Radiation Protection Measures in Work Involving Minerals and Raw Materials, Safety Reports Series No. 49, IAEA, Vienna (2006).

1. Background for UG & NORM S.A.

Very Important References

(1) Extraction of rare earths from monazite;
(2) Production of thorium compounds and manufacture of thorium containing products;
(3) Processing of niobium–tantalum ore;
(4) Oil and gas production;
(5) Geothermal energy production;
(6) TiO_2 pigment production;
(7) Thermal phosphorus production;
(8) Zircon and zirconium industry;
(9) Production of phosphate fertilizers;
(10) Cement production, maintenance of clinker ovens;
(11) Coal fired power plants, maintenance of boilers;
(12) Phosphoric acid production;
(13) Primary iron production;
(14) Tin/lead/copper smelting;
(15) Groundwater filtration facilities.



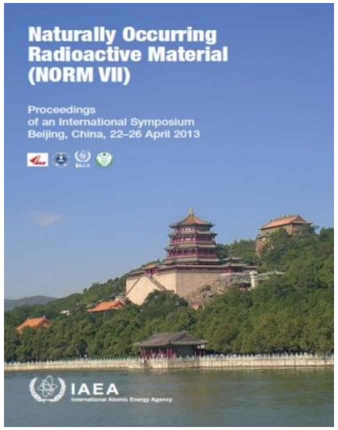
EU List of NORM

EUROPEAN COMMISSION, Draft Euratom Basic Safety Standards Directive (2010).

Very Important References

(1) Uranium overburden;
(2) Phosphate waste, including phosphogypsum and slag;
(3) Phosphate fertilizers;
(4) Coal combustion residues, comprising fly ash, bottom ash and slag;
(5) Oil and gas scale and sludge;
(6) Water treatment sludges and resins containing radium;
(7) Metal mining and processing waste associated with rare earths, zircon, hafnium, titanium, tin and large volume industries such as copper and iron and steel;
(8) Geothermal energy production wastes.

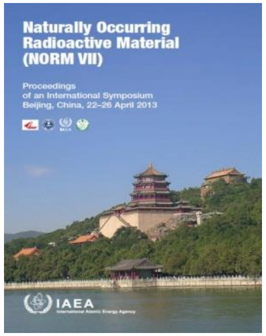
USA List of NORM



Very Important References

TABLE 1. THE REGULATORY DIRECTORY ON RADIATION ENVIRONMENTAL SAFETY IN THE UTILIZATION OF MINERAL RESOURCES (FIRST BATCH)

Mineral	Industrial activities
1. Rare earths	Mining, beneficiation and processing of various rare earth ores, including monazite, bastnäsite, xenotime and ion adsorption clays
2. Niobium–tantalum	Mining, beneficiation and processing of minerals containing niobium and/or tantalum
3. Zircon and zirconia	Mining, beneficiation and processing of zircon sand and baddeleyite
4. Vanadium	Mining and processing of vanadium ore
5. Anthracite	Mining and utilization of anthracite



China List of NORM

Typical NORM (1st Catalog, Published by MOEP, P.R.China)

UG & NORM Samples are very complicated!

- Hard Rock Samples
- Sandstone Samples
- NORM Samples

1. Background for UG & NORM S.A.

Very Important References

JORC TABLE 1

Section 1 Sampling Techniques and Data

(Criteria in this section apply to all succeeding sections.)

Criteria	Explanation
Sampling techniques	• *Nature and quality of sampling (eg cut channels, random chips, or specific specialised industry standard measurement tools appropriate to the minerals under investigation, such as down hole gamma sondes, or handheld XRF instruments, etc). These examples should not be taken as limiting the broad meaning of sampling.* • *Include reference to measures taken to ensure sample representivity and the appropriate calibration of any measurement tools or systems used.* • *Aspects of the determination of mineralisation that are Material to the Public Report. In cases where 'industry standard' work has been done this would be relatively simple (eg 'reverse circulation drilling was used to obtain 1 m samples from which 3 kg was pulverised to produce a 30 g charge for fire assay'). In other cases more explanation may be required, such as where there is coarse gold that has inherent sampling problems. Unusual commodities or mineralisation types (eg submarine nodules) may warrant disclosure of detailed information.*

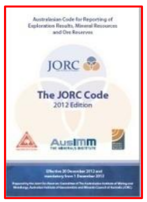
JORC- requirement

=【DZ/T 130-2006】？

Sampling should be based on the criteria!

1. Background for UG & NORM S.A.

Very Important References



Quality of assay data and laboratory tests	• *The nature, quality and appropriateness of the assaying and laboratory procedures used and whether the technique is considered partial or total.* • *For geophysical tools, spectrometers, handheld XRF instruments, etc, the parameters used in determining the analysis including instrument make and model, reading times, calibrations factors applied and their derivation, etc.* • *Nature of quality control procedures adopted (eg standards, blanks, duplicates, external laboratory checks) and whether acceptable levels of accuracy (ie lack of bias) and precision have been established.*

JORC- requirement

【=DZ/T 130-2006】？

Laboratory test should be based on the criteria!

1. Background for UG & NORM S.A.

Determination of U ,Th and Ra in UG & NORM sample analysis are getting more and more significant!

- ✓Uranium Exploration
- ✓NORM Regulatory control
- ✓Improvement of processing
- ✓Protection of radiation
- ✓Resource utilization

Outline

1. Background for uranium geology (UG) and NORM sample analysis
2. New Practice on Methods for Determination of U Th Ra
3. Summary

NORM: naturally occurring radioactive materials

➢Typical methods used for U Th Ra

Method	Category	References
Neutron activation analysis	NDA	[1]
Fission track method	NDA	[1]
X ray fluorescence spectrometry	NDA	[1]
Laser ablation spectroscopy	NDA	[1]
Direct measurement γ spectrometry	NDA	[1]
gravimetric method	DA	[2]
Colorimetry	DA	[2]
Volumetric method	DA	[2]
Laser fluorescence method	DA	[2]
Polarography	DA	[2]
Emanation method	DA	[2]
Atomic Emmision Spectrometry	DA	[2]
Alpha spectrometry	DA	[2,3]
Mass spectrometry	DA	[2]

[1], Liu Likun, Guo Dongfa, Huang Qiuhong. Advances in the analysis methods of uranium and thorium in rock and mineral materials -- non destructive analysis [J]. Chinese inorganic analytical chemistry, 2012,2 (04): 10-14.[2017-08-27].
[2] [2] Liu Likun, Guo Dongfa, Huang Qiuhong. Advances in the analysis methods of uranium and thorium in rocks and minerals -- wet chemical method [J]. Chinese inorganic analytical chemistry, 2012,2 (02): 6-9.[2017-08-27].
[3] IAEA Analytical Quality in Nuclear Applications Series No. 34. A Procedure for the Sequential Determination of Radionuclides in Phosphogypsum[OL]. http://www-pub.iaea.org/books/IAEABooks/10711/A-Procedure-for-the-Sequential-Determination-of-Radionuclides-in-Phosphogypsum-Liquid-Sci. 2017-08-27

➢Typical methods used for U Th Ra analysis at BRIUG

Wet Chemical Analysis

Methods	Analysed for	Type of samples	References
Auto titration(Davie s and Grad Titration)	U	UOC, UF_4, other uranium compounds	EJ/T 266-1993
Emanation	^{226}Ra	Rocks, ores, water	GB/T 13073-2010, [4]
UV Spectrophotoscopy	Th	Rocks, ores, water	EJ/T 349 3-1988
Laser-induced fluorescence	Trace U	Rocks, ores, water	EJ/T 550-2000, [5,6]

[4] Huang Qiuhong, Dong Chen, Guo Dongfa, Fan Zengwei, Tian Fei, Hu Xiaohua, Pan Jingrong, Wang Tiejian. Experimental study on the determination of radium using a new generation of radium radon pc-2100 analyzer [J]. uranium geology, 2014,30 (01): 51-56.
[5] Li Qingzhen, Zhang Yanan. Direct determination of uranium in soil, rock, mineral, biology and other samples by laser fluorescence method. China Nuclear Science and technology report, 1993, (00): 222-235
[6] Liu Likun, Guo Dongfa, Li Binkai, Zhang Yanhui, Huang Qiuhong. Determination of mass concentration of uranium in Saline Lake water by ultraviolet pulse fluorescence [J]. uranium geology, 2011,27 (03): 180-184

➢Typical methods used for U Th Ra analysis at BRIUG

Mass Spectrometry

Methods	Analysed for	Type of samples	References
ICP-MS	U, Th, Ra, other elements	Rocks, ores, water	GB/T 14506.30-2010 , [7]
ID-MS	U, Th, other elements	Rocks, ores, water	[8]
TIMS	U, Th, other elements	Rocks, ores, water	[9]
LI-TOF-MS	U Clusters	U metal, UO_2,UF_4, other uranium compounds for identification	Doctoral Dissertation by TAN Jing
LG-SIMS +FIB-SEM LA-ICP-MS	U Particles	Swipe samples for U isotopic identification, imaging	SCMS 2017, Chengdu

[7] Guo D F, Zhang Y H, Zhao-Hui W U, et al. Determination of ~(234)U/~(238)U,~(230)Th/~(232)Th,~(228)Ra/~(226)Ra Ratios in Uranium Ores by High-resolution Inductively Coupled Plasma-Mass Spectrometry[J]. Rock & Mineral Analysis, 2009, 28(2):101-107.
[8] Guo Dongfa, Wu Zhaohui, Cui Jianyong, et al. Arbitration analysis method of uranium content in sandstone uranium geological samples -- isotope dilution inductively coupled plasma mass spectrometry [J]. atomic energy science and technology, 2008, 42 (3): 277-283.
[9] Zhu Mingyan, Guo Dongfa, Cui Jianyong, Liu Yuang, Tan Jing, Zhang Yanhui, Determination of (234) U/ (238) U, (230) Th/ (232) Th, (228) Ra/ (226) Ra in uranium ore samples by TIMS [J]. uranium geology, 2009,25 (03): 184-192.

➢Typical methods used for U Th Ra analysis at BRIUG

XRF & Alpha & Gamma Spectrometry

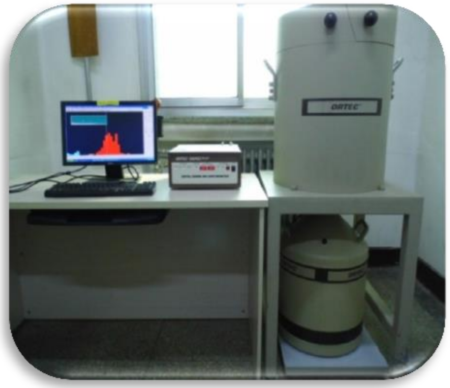
Methods	Analysed for	Type of samples	References
Wavelength dispersive X-ray fluorescence spectrometry	U, Th, other elements	Rocks ,ores	**GB/T 14506.29-2010, [10]**
Gamma Spectrometry	^{238}U, ^{232}Th, ^{226}Ra	Rocks and ores	GB/T 11713-2015, [11]
Alpha spectrometry	$^{234}U/^{238}U$; $^{230}Th/^{232}Th$ Ra isotopes	Rocks, ores, water	GB/T 13071-2010, [3, 12]

[10] Xiao Deming, Gan Xuanji, Zhang Hongwen. Determination of of uranium and in geological samples by X ray fluorescence spectrometry[J Chinese Journal of Analytical Chemistry.1983(10): 750-753
[11] IAEA TECHNICAL REPORTS SERIES No 341 . Analytical techniques in uranium exploration and ore processing[R]. http://www-pub.iaea.org/mtcd/publications/pdf/trs341_web.pdf. 2017-08-27
[12] IAEA Analytical Quality in Nuclear Applications Series No. 19. Analytical Methodology for the Determination of Radium Isotopes in Environmental Samples [R] https://nucleus.iaea.org/rpst/referenceproducts/analytical_methods/aq-19.pdf, 2017-08-24

Typical Methods for UG & NORM

Typical Methods for UG & NORM Sample Analysis

XRF



HPGe γ-Spec.

CRMs

ICP-MS

MUA (U)

PC2100(Ra)

Method Validation for Determination of Major and Minor Elements by Fusion-WDXRF

②Flux temperatur is 1050℃, 22min

①Sample:Flux=1:10
Flux is
67%$Li_2B_4O_7$+33%$LiBO_2$

③Fused Bead

XRF sample preparation (Fused Bead)

Yuan Jian†, XIA Cheng-Guang……., Determination of Determination of Major and Minor Elements in UGSA samples by Fusion-WDXRF[R]. BRIUG Report, 2017

Method Validation for Determination of Major and Minor Elements by Fusion-WDXRF

The method is :

GB/T 14506.28- Methods for chemical analysis of silicate rocks-Part 28: Determination of 16 major and minor elements content.

SiO_2,Al_2O_3,Fe_2O_3,MgO,CaO,NaO_2,K_2O,MnO,TiO_2,P_2O_5,Sr,Cu,Ni,Zr,Cr,Ba.

GB/T 14506.14- Methods for chemical analysis of silicate rocks-Part 14: Determination of ferrous oxide content.

FeO

Yuan Jian†, XIA Cheng-Guang. Determination of Determination of Major and Minor Elements in UGSA samples by Fusion-WDXRF[R]. BRIUG Report, 2017

Method Validation for Determination of Major and Minor Elements by Fusion-WDXRF

More than 30 CRMs were used for calibration curve

Uranium-bearing sandstone	GBW 04130
Rock	GBW07103, GBW07104, GBW07105 GBW07106, GBW07107, GBW07108, GBW07109, GBW07110, GBW07111, GBW07112, GBW07113, GBW07114
Sediment	GBW 07301a, GBW 07302a, GBW 07303a, GBW 07304a, GBW 07305a, GBW 07307a, GBW 07308a,GBW07309, GBW07310, GBW07311, GBW07312, GBW07315, GBW07316
Soil	GBW07401, GBW07402, GBW07403, GBW07404, GBW07405, GBW07406, GBW07407, GBW07408, GBW07423, GBW07424, GBW07425, GBW07426, GBW07427, GBW07428, GBW07429, GBW07430

Yuan Jian†, XIA Cheng-Guang. Determination of Determination of Major and Minor Elements in UGSA samples by Fusion-WDXRF[R]. BRIUG Report, 2017

Method Validation for Determination of Major and Minor Elements by Fusion-WDXRF

WD-XRF Interference Control

Instrument monitor would be used to calibrate instrument drift

Matrix interference ,we use theory α coefficient, fundamental parameters

Some elements need calibrate the overlap interference

Select CRMs as much as possible to calibrate curve

Yuan Jian†, XIA Cheng-Guang. Determination of Determination of Major and Minor Elements in UGSA samples by Fusion-WDXRF[R]. BRIUG Report, 2017

Detection Limits

Component	D.L（%）	Component	D.L（%）
SiO_2	0.010	Sr	0.005
Al_2O_3	0.010	Cu	0.004
Fe_2O_3	0.010	Ni	0.004
MgO	0.010	Zr	0.010
CaO	0.010	Cr	0.006
NaO_2	0.010	Ba	0.010
K_2O	0.010	FeO	0.10
MnO	0.004	L.O.I	0.10
TiO_2	0.006		
P_2O_5	0.006		

Precision

Component	Sample 42851 Content(%)	RSD%	Y_c%	Evaluation
SiO_2	75.40	0.29	0.67	Accepted
Al_2O_3	10.15	0.15	3.06	Accepted
Fe_2O_3	4.63	0.08	4.18	Accepted
MgO	0.083	4.29	12	Accepted
CaO	0.199	1.42	10	Accepted
NaO_2	3.63	0.60	4.55	Accepted
K_2O	3.36	0.53	4.67	Accepted
MnO	0.026	2.77	15	Accepted
TiO_2	0.078	0.91	12	Accepted
P_2O_5	0.014	5.24	17	Accepted
FeO	0.79	1.31	11	Accepted
L.O.I	0.48	7.44	12	Accepted

2. New Practice on Methods for D. U Th Ra

Trueness

Component	GBW04132		RE%	Y_B%	Evaluation
	Certified Value, %	Measured Value, %			
SiO_2	75.55	75.64	0.12	0.47	Accepted
Al_2O_3	10.29	10.31	0.18	2.15	Accepted
Fe_2O_3	2.91	2.88	0.95	3.46	Accepted
MgO	0.42	0.429	2.19	5.92	Accepted
CaO	1.63	1.60	1.58	4.14	Accepted
NaO_2	2.08	2.10	0.97	3.85	Accepted
K_2O	3.60	3.60	0.03	3.23	Accepted
MnO	0.14	0.137	1.96	7.61	Accepted
TiO_2	0.40	0.402	0.42	5.99	Accepted
P_2O_5	0.53	0.527	0.60	5.59	Accepted
FeO	0.86	0.90	4.65	7.41	Accepted
L.O.I	2.26	2.27	0.31	5.63	Accepted

Detection Limits by PPP-WDXRF

Element	晶体	D.L.(mg/kg）
U (Lα1, 0.0911nm)	LiF 200	1.04
Th((Lα1, 0.0956nm)	LiF 220	0.93

[12] Zhang Hongwen, Gan Xuanji, Xiao Deming. Determination of low content uranium and thorium by X - ray fluorescence spectrometry[J]. Analysis Bulletin, 1986 (05) : 19-23

Trueness by PPP-WDXRF

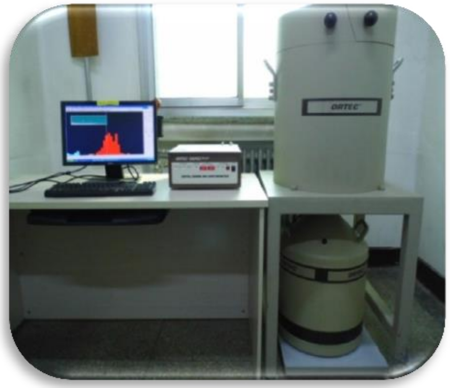
Element	GSD-2		RE%	Y_B%	Evaluation
	Certified Value, mg/kg	Measured Value, mg/kg			
U	17.0	16.9	-0.6%	11.65	Accepted
Th	70.0	71.2	1.7%	9.15	Accepted

$$Y_B = \frac{1}{\sqrt{2}} Y_C = \frac{1}{\sqrt{2}} C \times (14.37 X_0^{-0.1263} - 7.659)$$

[12] Zhang Hongwen, Gan Xuanji, Xiao Deming. Determination of low content uranium and thorium by X - ray fluorescence spectrometry[J]. Analysis Bulletin, 1986 (05) : 19-23

Typical Methods for UG & NORM Sample Analysis

XRF



HPGe γ-Spec.

CRMs

ICP-MS

MUA (U)

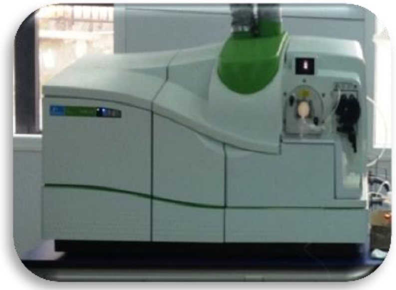
PC2100(Ra)

2. New Practice on Methods for D. U Th Ra

Instrument Configuration

Element Analysis

Q-ICP-MS



QQ-ICP-MS

Speciation Analysis

Micro Zone Analysis

LA-ICP-MS

HR-ICP-MS

Nuclide Analysis

Isotope Analysis

MC-ICP-MS

Q-ICP-MS

Specific Analysis

2. New Practice on Methods for D. U Th Ra

Different Detection Limits;
Interference concerned!

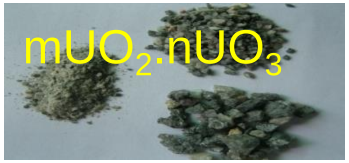
	1A	2A	3B	4B	5B	6B	7B	8			1B	2B	3A	4A	5A	6A	7A	0
1	1 H																	2 He
2	3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
3	11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
4	19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
5	37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 ^{103}Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
6	55 Cs	56 Ba	*	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg	81 Tl	82 Pb	83 Bi	84 Po	85 At	86 Rn
7	87 Fr	88 Ra	#															

*	57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
#	89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

Elements can be determined by ICP-MS

2. New Practice on Methods for D. U Th Ra

Expl.

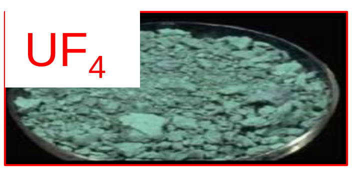


Mining

Con.

Enrich.

Re-Con.

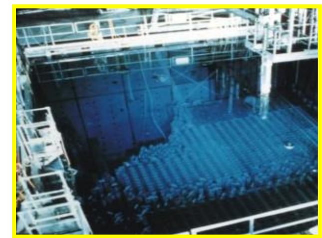


Fab.

Zr-4 Alloy

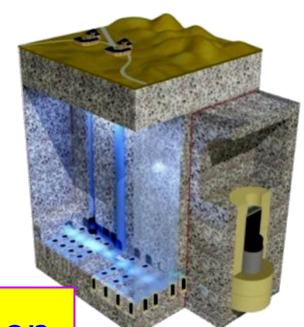
ICP-MS Application

Reactor



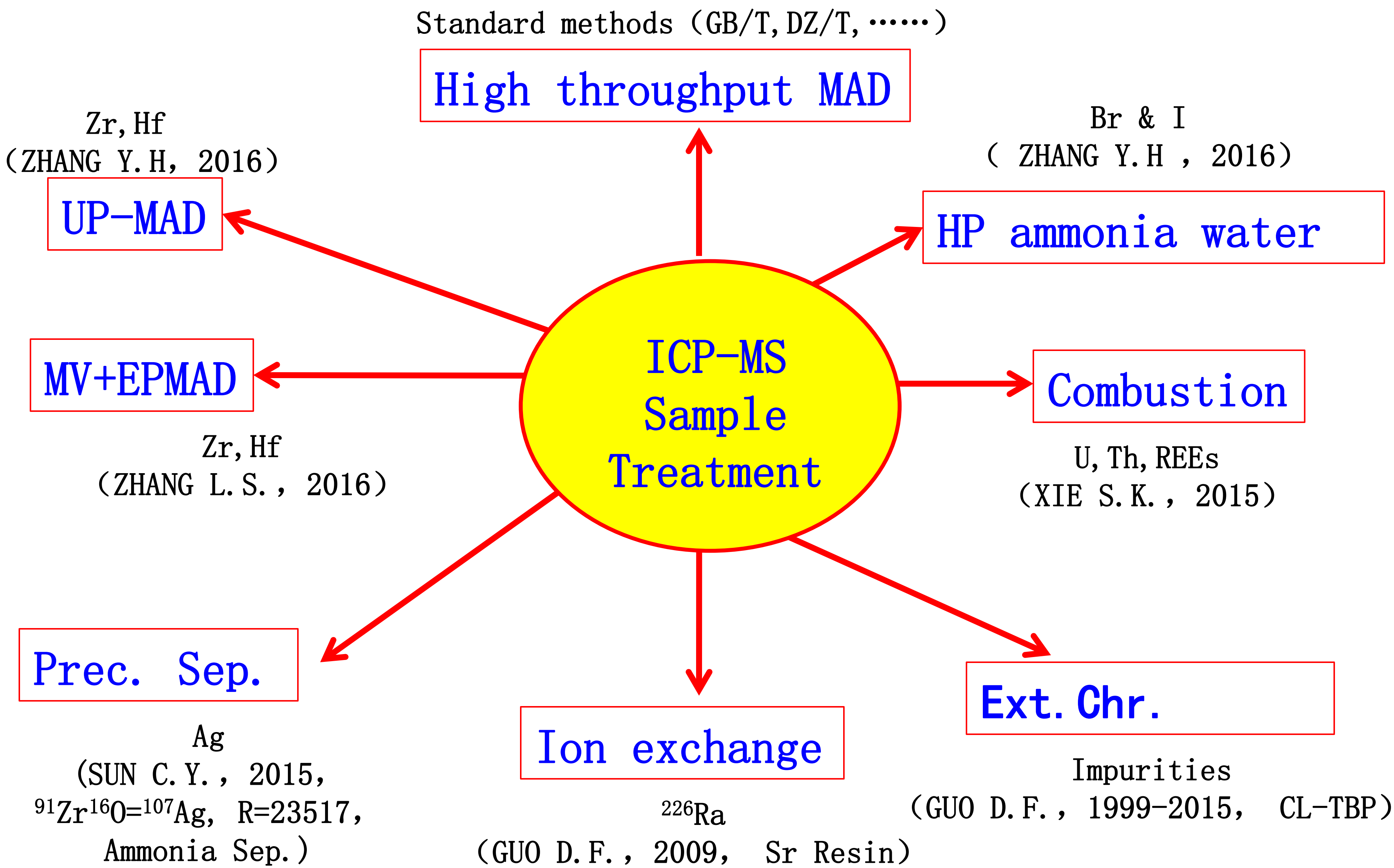
Spent Fuel

Reprocessing



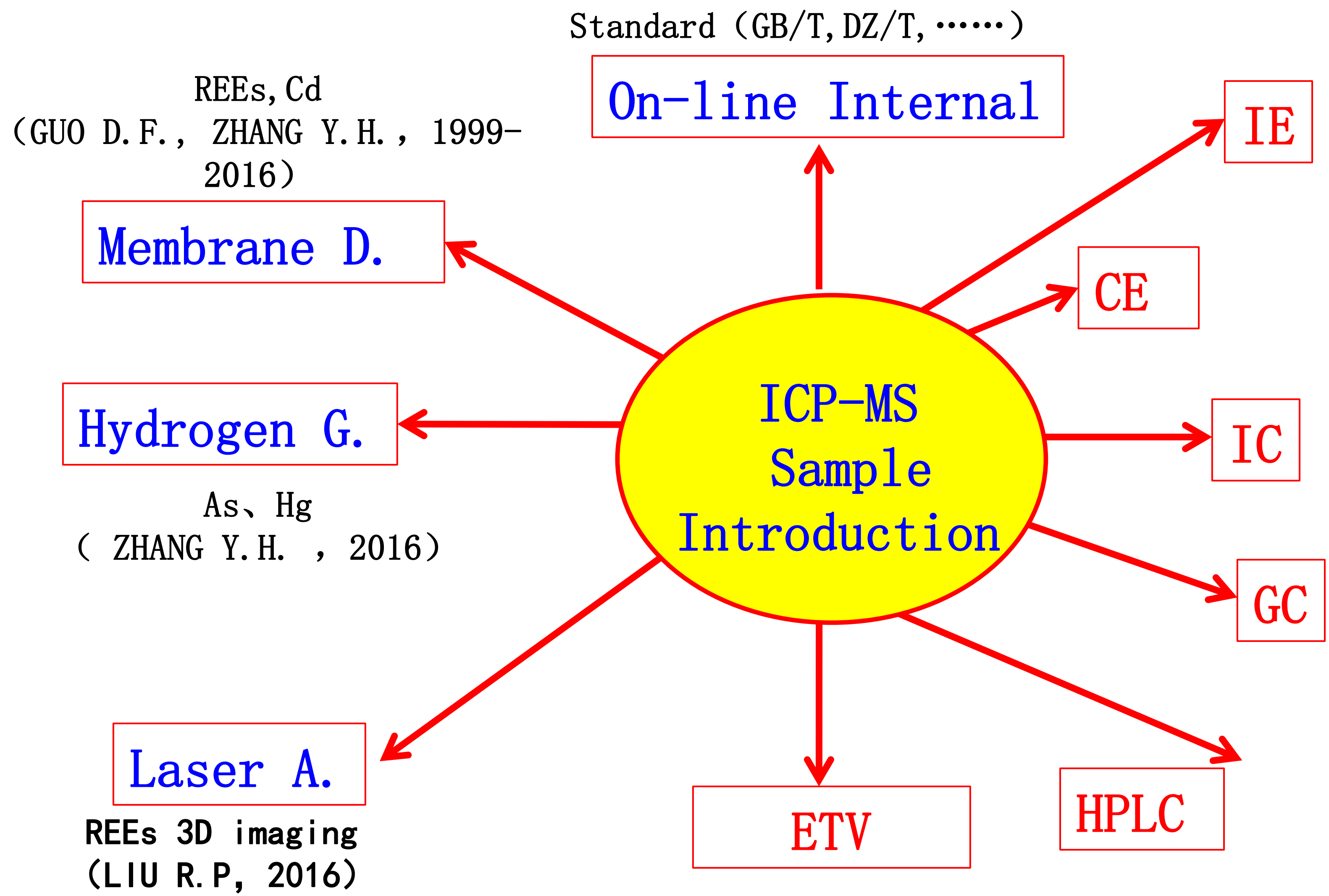
Waste Disposal

2. New Practice on Methods for D. U Th Ra

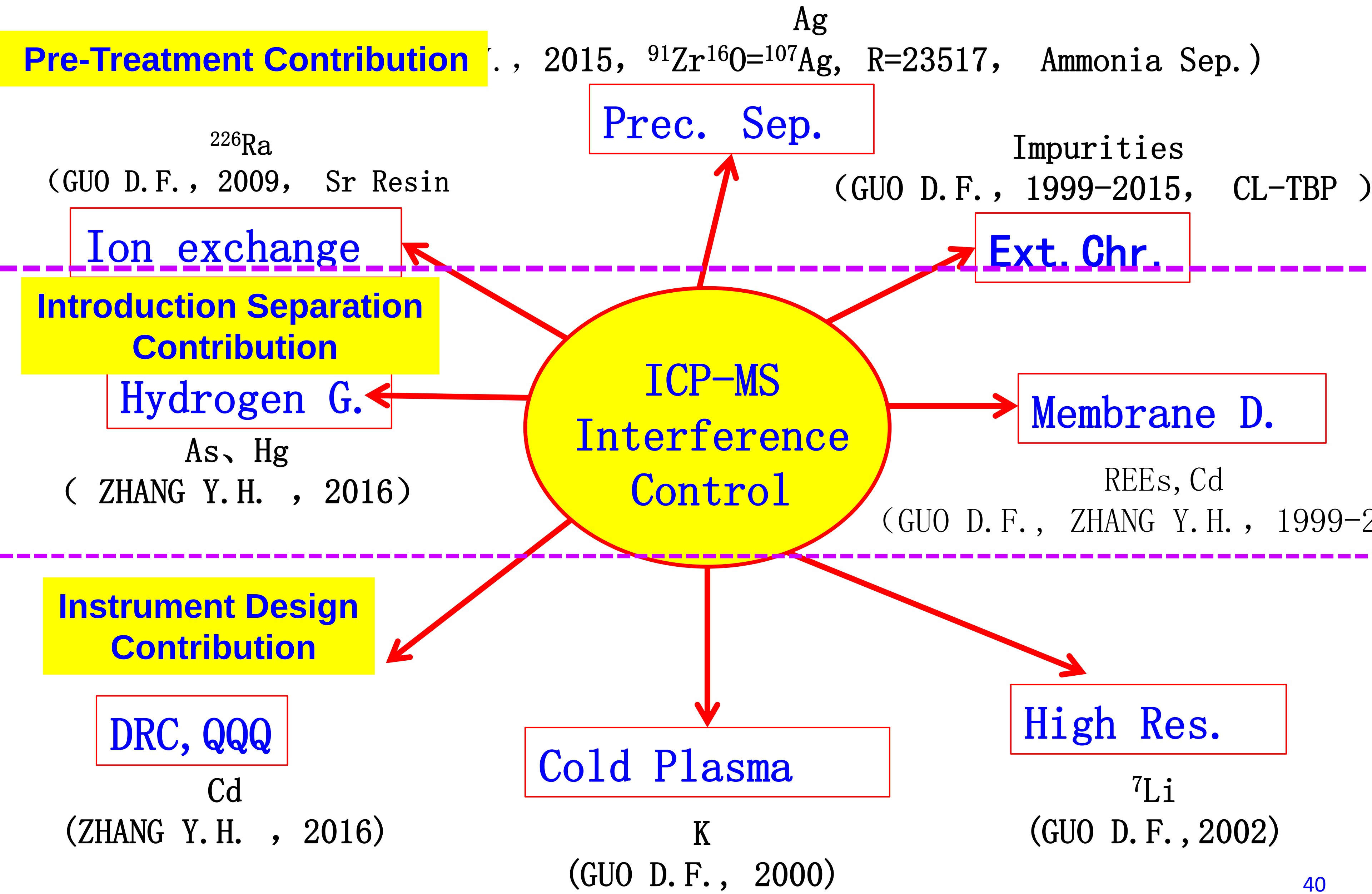


2. New Practice on Methods for D. U Th Ra

从铀矿勘查到高防废物地质处置样品分析的若干新技术进展（二）



2. New Practice on Methods for D. U Th Ra



Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Most Powerful Tool
for Trace Inorganic Elements！

U,Th……

XIE Shengkai[†], GUO Dong-fa*, HUANG Qiu-hong, LI Li, WANG Tie-jian, TIAN Fei， ZHOU Lianghui, QU Ying. Determination of Nb,Ta,Zr,Hf,U,Th and REEs in Alkaline Rock Samples by Lithium Borate Fusion- Mixed Acid Digestion-ICP-MS[R]. BRIUG Report, 2017

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

↓

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

1. Weighing（0.1000g）in Graphite Crucible
2. Add 0.4g High Pure $LiBO_2$
3. Cover with $LiBO_2$ (about 0.2g)
4. Put in muffle furnace (1030℃) melting for 30min
5. Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry
6. Add 10mL7mol/LHNO_3
7. Add water to 52.40g
8. Transfer 5mL, and to 50.00g
9. Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Selection of Crucible

Type of Crucible	Number of Sample	Results Measured	
		U(μg/g)	Th(μg/g)
Pt-Au	GBW07157	14.0	14.3
	GBW07157	13.3	14.5
Graphite	GBW07157	13.7	16.1
	GBW07157	14.1	15.5
Graphite powder	GBW07157	12.9	15.5
	GBW07157	13.1	15.4

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

↓

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Selection of Flux

Flux weight(g)	Number of Sample	Result Measured	
		U(μg/g)	Th(μg/g)
0.2	GBW07157	13.1	15.8
0.3	GBW07157	11.2	14.7
0.4	GBW07157	13.1	15.3
0.5	GBW07157	14.3	15.4
0.6	GBW07157	14.1	15.9
0.7	GBW07157	13.8	15.9
0.8	GBW07157	12.8	15.5
0.9	GBW07157	13.4	15.5

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

↓

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Selection of Temperature

Temperature(℃)	Number of Sample	Results Measured	
		U(μg/g)	Th(μg/g)
1000	GBW07157	13.7	16
	GBW07157	14.1	15.5
1030	GBW07157	13.1	15.8
	GBW07157	13.3	15.7

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

- Weighing（0.1000g）in Graphite Crucible
- Add 0.4g High Pure $LiBO_2$
- Cover with $LiBO_2$ (about 0.2g)
- Put in muffle furnace (1030℃) melting for 30min
- Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry
- Add 10mL7mol/LHNO_3
- Add water to 52.40g
- Transfer 5mL, and to 50.00g
- Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

The purpose of acid dissolution

- Extraction;
- Remove Boron;
- Reduce Silicon;
- Remove Fluorine.

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

↓

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

Add 0.4g High Pure $LiBO_2$

Cover with $LiBO_2$ (about 0.2g)

Put in muffle furnace (1030℃) melting for 30min

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

Add 10mL7mol/LHNO_3

Add water to 52.40g

Transfer 5mL, and to 50.00g

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

↓

Measured by ICP-MS

$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Weighing（0.1000g）in Graphite Crucible

↓

Add 0.4g High Pure $LiBO_2$

↓

Cover with $LiBO_2$ (about 0.2g)

↓

Put in muffle furnace (1030℃) melting for 30min

↓

Add 4mLHNO_3, 4mLHF & 0.5mL$HClO_4$, heat at (180℃) to dry

↓

Add 10mL7mol/LHNO_3

↓

Add water to 52.40g

↓

Transfer 5mL, and to 50.00g

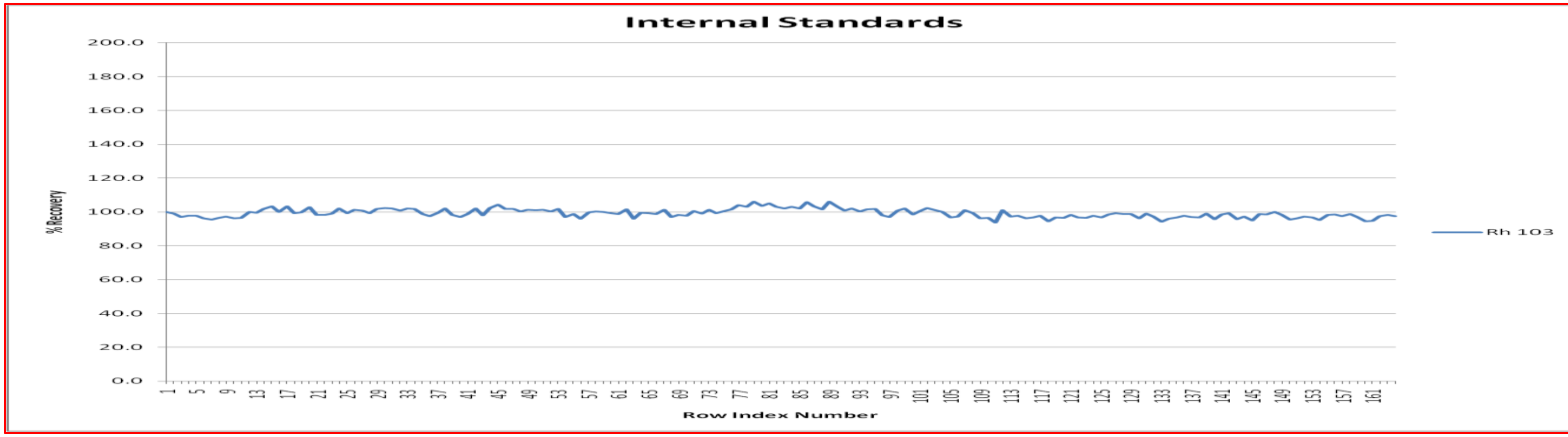
↓

Measured by ICP-MS

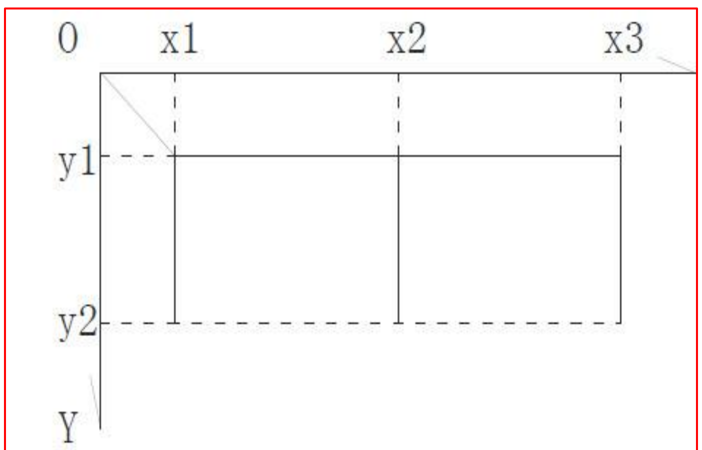
$LiBO_2$ Alkali Fusion- Mixed Acid Digestion Procedure

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Internal Standard



Monitoring of ^{103}Rh during measurement



On-line internal standard and sample introduction system

2. New Practice on Methods for D. U Th Ra

Interference control

Nr.	Isotope	Interference	Control Measures	Working Curve (0,5,10,50,100 ng/mL) Group
15	Eu 151	135Ba16O	Coef. Correction	A
19	Ho 165	149Sm16O	Coef. Correction	A
20	Er 166	150Sm16O, 150Nd16O	Coef. Correction	A
21	Tm 169	153Eu16O	Coef. Correction	A
22	Yb 172	156Gd16O	Coef. Correction	A
23	Lu 175	141Pr16O	Coef. Correction	A
24	W 182	142Nd40Ar, 166Er16O	Coef. Correction	B
25	Tl 205	165Ho40Ar	Coef. Correction	A
26	Th 232	192Pt40Ar	Coef. Correction	A
27	U 238	198Pt40Ar	Coef. Correction	A
28	Nb 93	53Cr40Ar, 77Se16O	Coef. Correction	B
29	Ta 181	141Pr40Ar, 165Ho16O	Coef. Correction	B
30	Zr 90	50Ti40Ar, 50Cr40Ar, 180Hf++	Coef. Correction	B
31	Hf 177	161Dy16O, 137Ba40Ar	Coef. Correction	B

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS

Performance of LBF-MAD ICP-MS

XIE Shengkai†, GUO Dong-fa*, HUANG Qiu-hong, LI Li, WANG Tie-jian, TIAN Fei， ZHOU Lianghui, QU Ying. Determination of Nb,Ta,Zr,Hf,U,Th and REEs in Alkaline Rock Samples by Lithium Borate Fusion- Mixed Acid Digestion-ICP-MS[R]. BRIUG Report, 2017

Detection Limits

Nr.	Isotop	D.L（μg/g）
15	Eu 151	0.08
17	Tb 159	0.12
18	Dy 163	0.46
19	Ho 165	0.14
20	Er 166	0.35
21	Tm 169	0.04
23	Lu 175	0.04
24	W 182	0.24
25	Tl 205	0.06
26	Th 232	0.59
27	U 238	0.09

Precision

Nr	Element	Sample 47679 Content(mg/kg)	RSD%	Yc%	Evaluation
20	Er	744	8.1	12	Accepted
21	Tm	125	8.5	17	Accepted
22	Yb	509	8.2	13	Accepted
23	Lu	62	7.4	20	Accepted
24	W	3.8	13	31	Accepted
25	Tl	1.0	15	38	Accepted
26	Th	983	10	12	Accepted
27	U	620	4.5	13	Accepted
28	Nb	12036	3.7	6.4	Accepted
29	Ta	744	3.8	12	Accepted
30	Zr	5137	5.2	8.0	Accepted
31	Hf	89	5.1	18	Accepted

Trueness

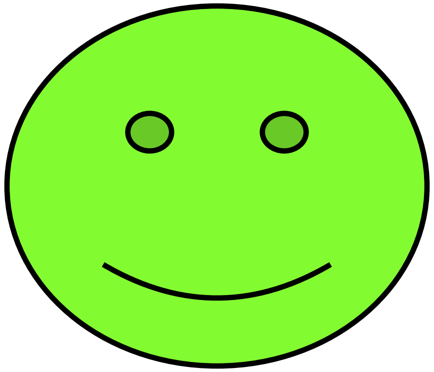
Nr.	Element	GBW07161(REEs ）		RE%	Y_B	Evaluation
		Certified Value,, μg/g	Measured Value, μg/g)			
11	Ce	187	173	-7.6%	11.4%	Accepted
12	Pr	433	443	2.3%	9.7%	Accepted
13	Nd	1595	1535	-3.7%	7.4%	Accepted
14	Sm	285	272	-4.4%	10.5%	Accepted
15	Eu	65	64	-1.5%	13.8%	Accepted
16	Gd	226	202	-10.5%	11.0%	Accepted
17	Tb	35	36	4.0%	15.4%	Accepted
18	Dy	192	181	-5.5%	11.3%	Accepted
19	Ho	36	35	-1.7%	15.3%	Accepted
20	Er	96	104	7.9%	12.9%	Accepted
21	Tm	13	13	1.3%	18.1%	Accepted
22	Yb	88	86	-1.9%	13.1%	Accepted
23	Lu	12	12	4.4%	18.4%	Accepted
26	Th	24	24	1.0%	16.4%	Accepted

Trueness

No	ELEMENT	GBW07301a		RE%	Y_B	Evaluation
		Certified Value,, µg/g	Measured Value, µg/g)			
1	Be	3.10	2.79	-9.9%	22.8%	Accepted
…	…	…	…	…	….	Accepted
22	Yb	2.30	2.38	3.6%	23.9%	Accepted
23	Lu	0.390	0.385	-1.3%	31.2%	Accepted
24	W	1.00	1.25	24.9%	27.1%	Accepted
25	Tl	0.670	0.595	-11.1%	28.8%	Accepted
26	Th	27	26	-2.3%	16.0%	Accepted
27	U	4.60	4.35	-5.5%	21.4%	Accepted
28	Nb	32	31	-0.6%	15.6%	Accepted
29	Ta	3.00	3.29	9.6%	22.9%	Accepted
30	Zr	316	298	-5.7%	10.3%	Accepted
31	Hf	9.30	8.72	-6.2%	19.1%	Accepted

2. New Practice on Methods for D. U Th Ra

Determination of U, Th and Other Elements in UGSA Samples by ICP-MS



U & Th

LBF-MAD

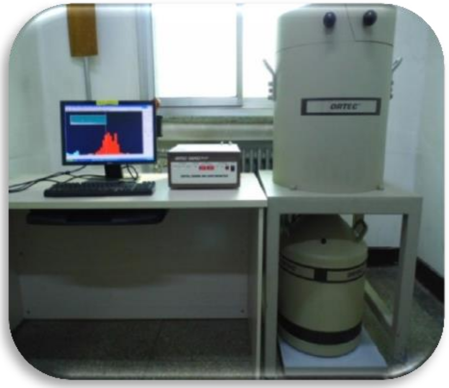
ICP-MS

Ra

XIE Shengkai†, GUO Dong-fa*, HUANG Qiu-hong, LI Li, WANG Tie-jian, TIAN Fei， ZHOU Lianghui, QU Ying. Determination of Nb,Ta,Zr,Hf,U,Th and REEs in Alkaline Rock Samples by Lithium Borate Fusion- Mixed Acid Digestion-ICP-MS[R]. BRIUG Report, 2017

Typical Methods for UG & NORM Sample Analysis

XRF



HPGe γ-Spec.

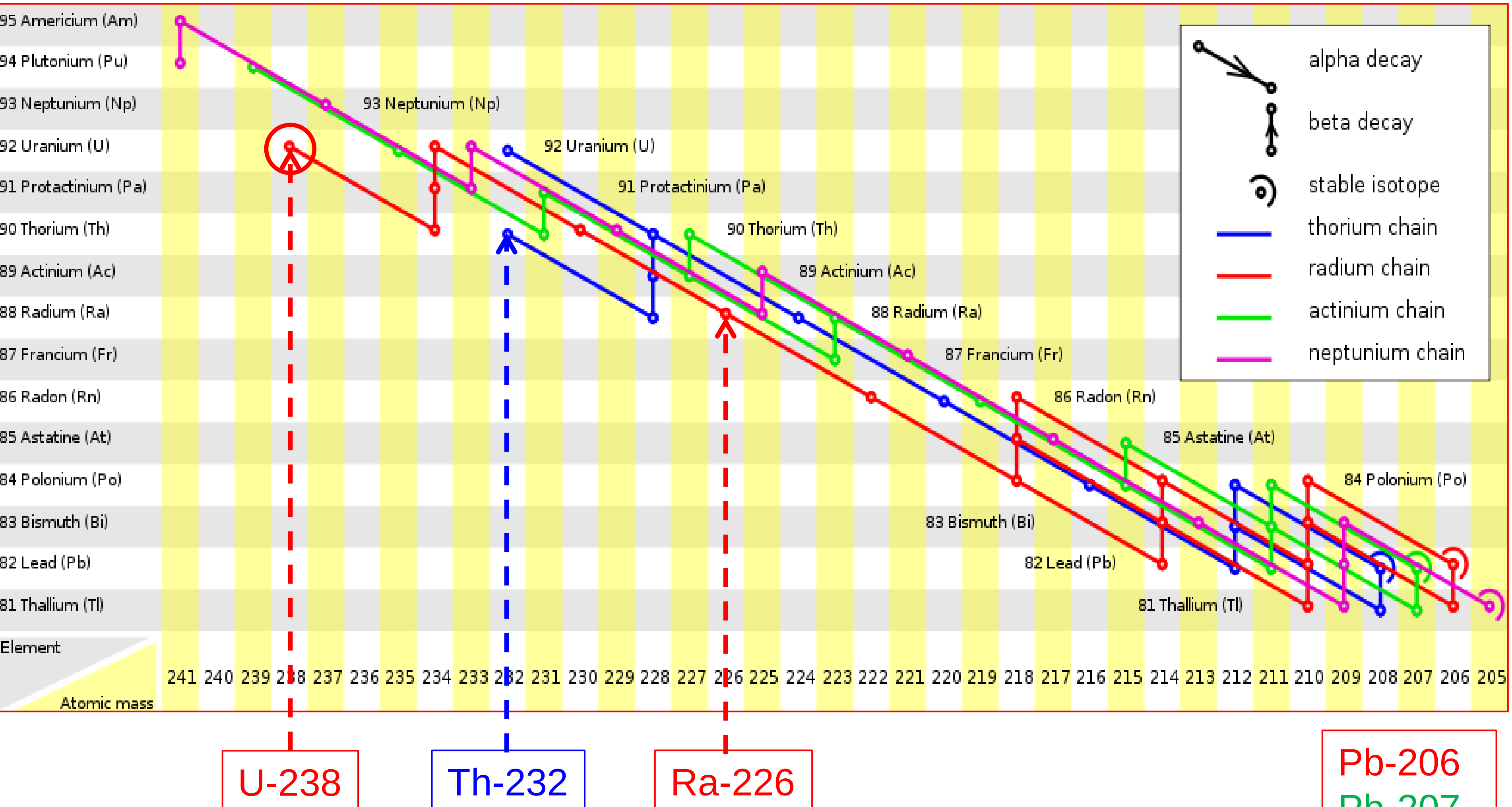
CRMs

ICP-MS

MUA (U)

PC2100(Ra)

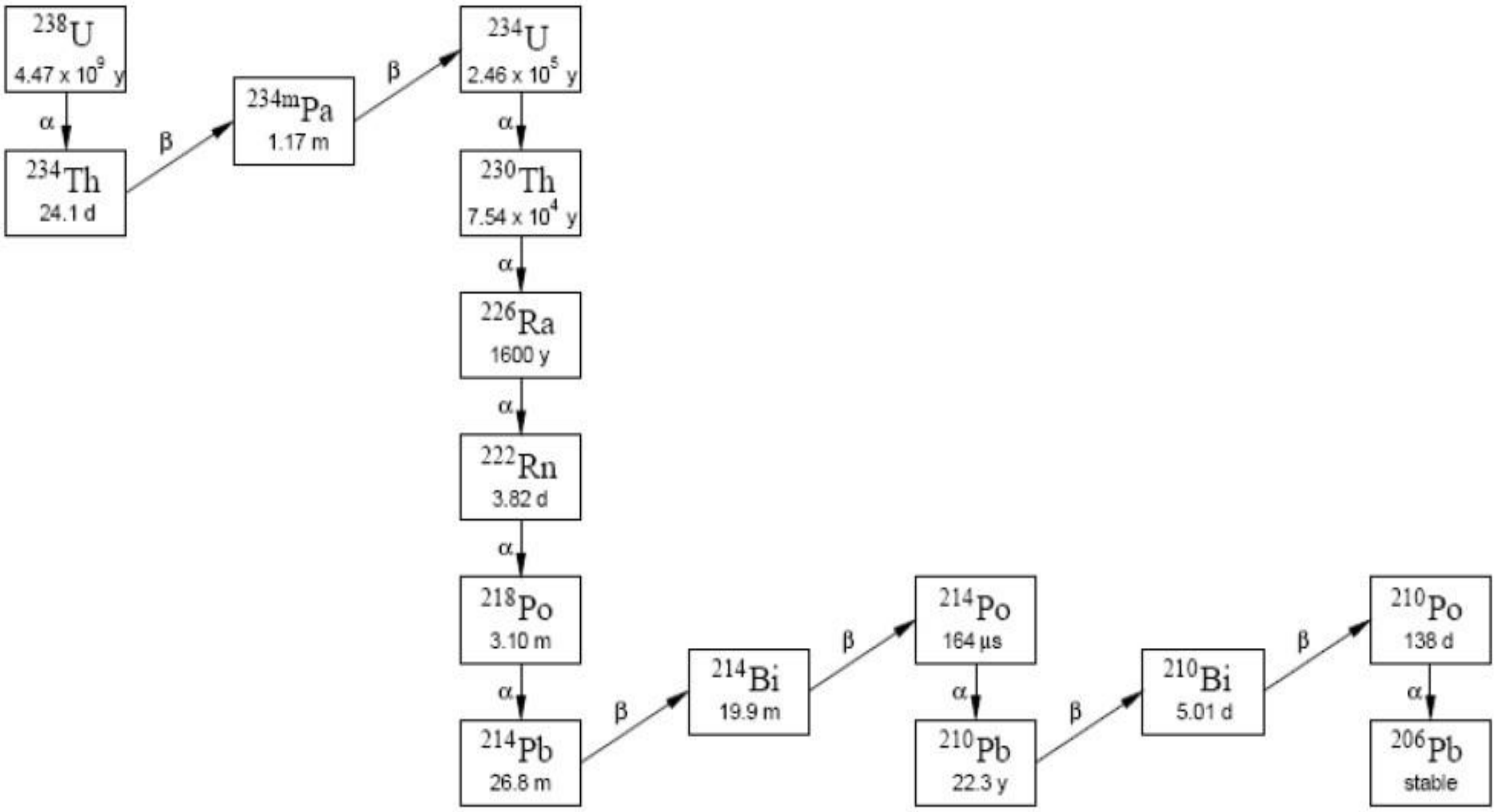
Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



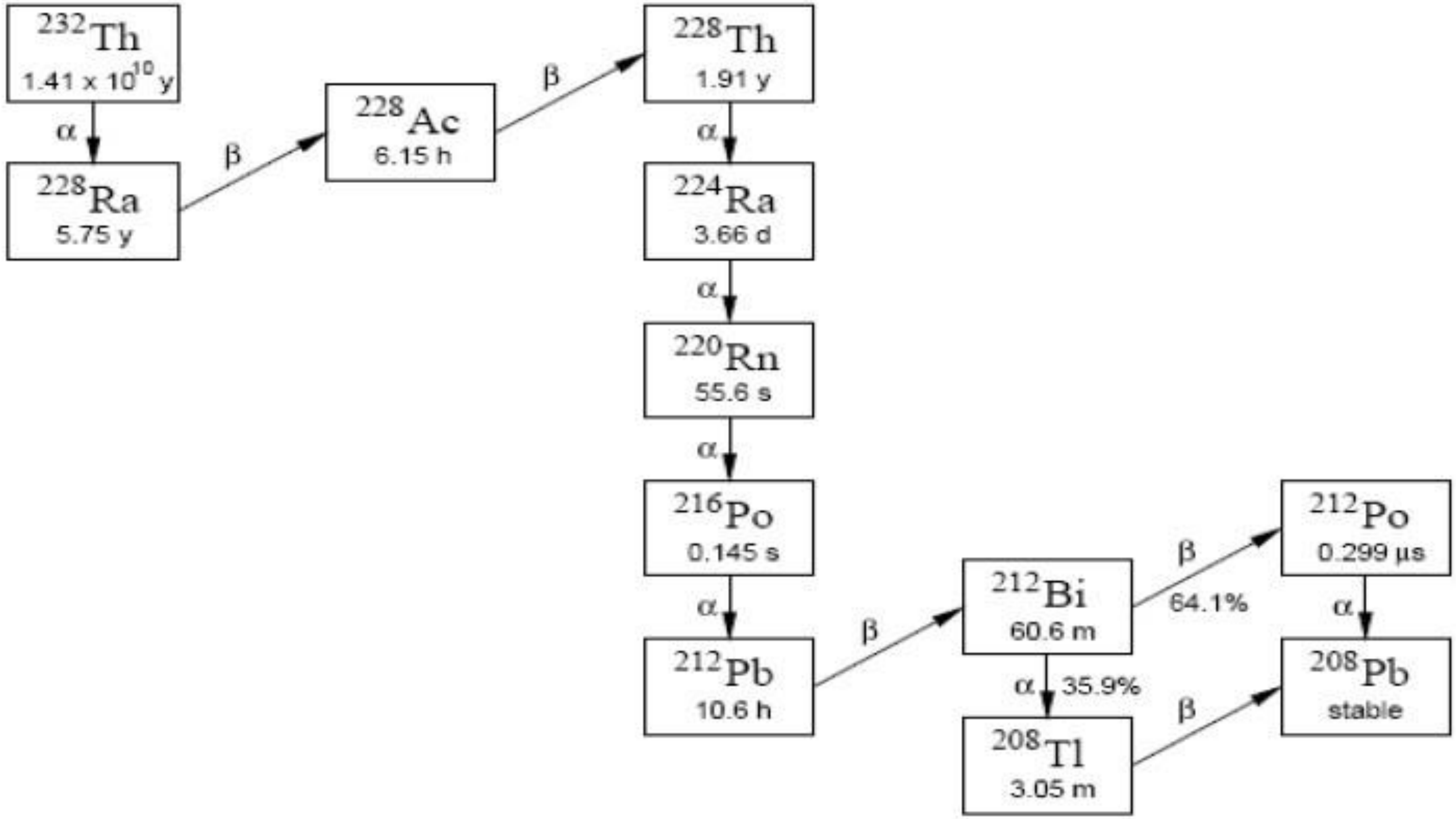
Pb-206
Pb-207
Pb-208

https://commons.wikimedia.org/wiki/File:Radioactive_decay_chains_diagram.svg

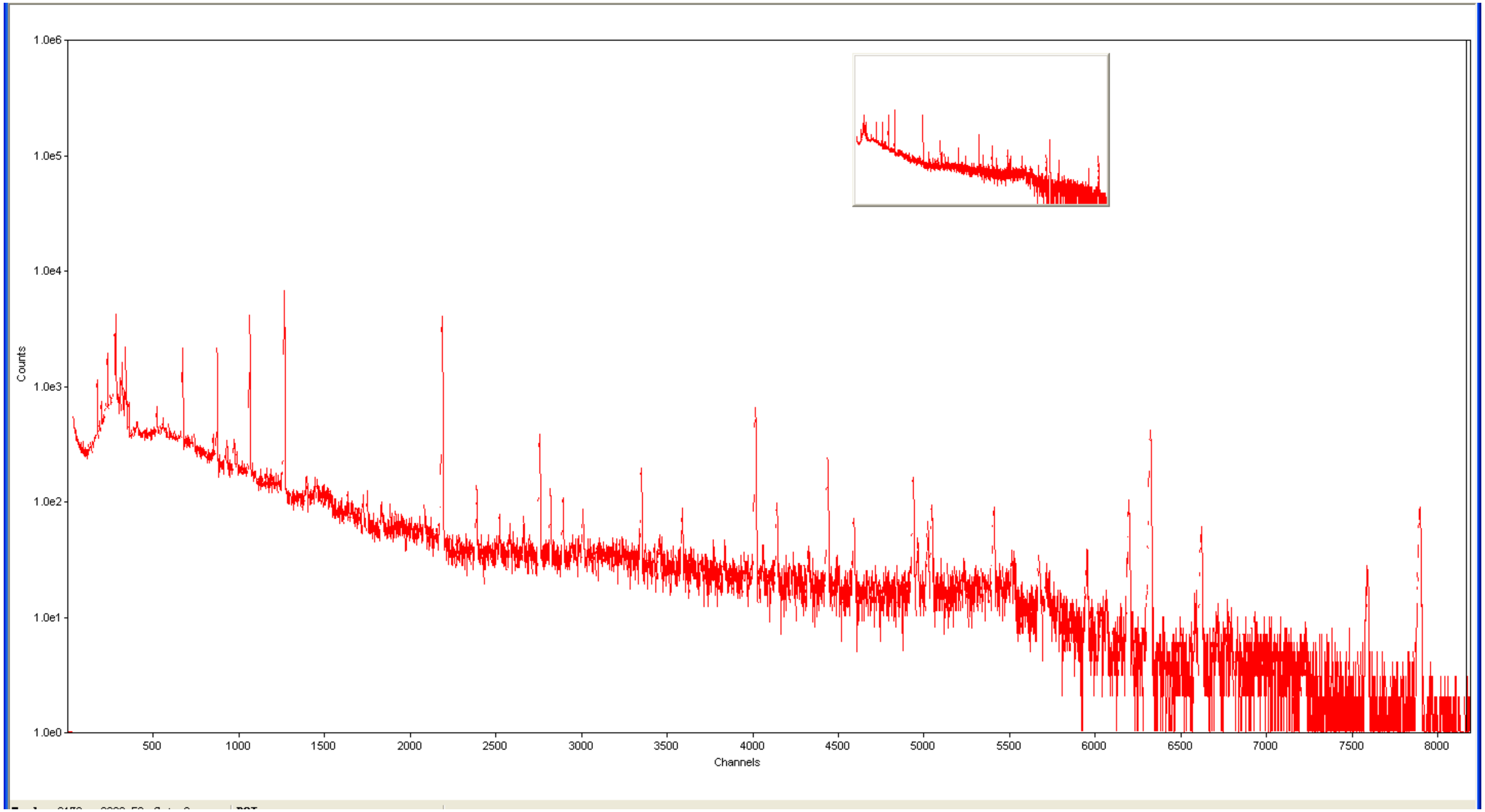
Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

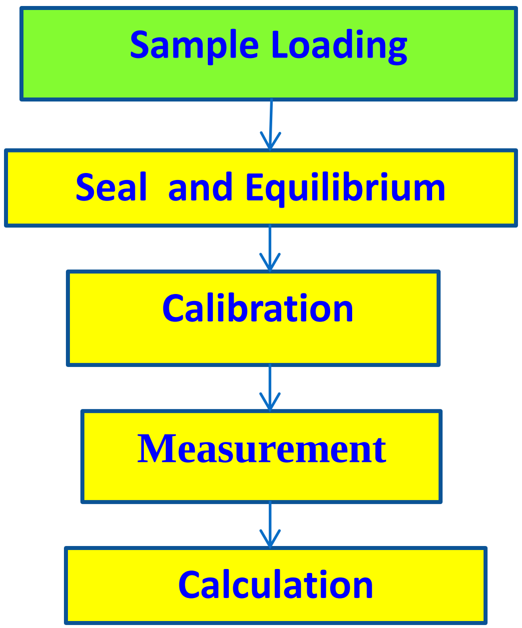


Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



HUANG Qiu-hong†, GUO Dong-fa* , WANG Tie-jian, TIAN Fei， ZHOULianghui, QUYing, LI Li. Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGeγSpectrometry [R]. BRIUG Report, 2017

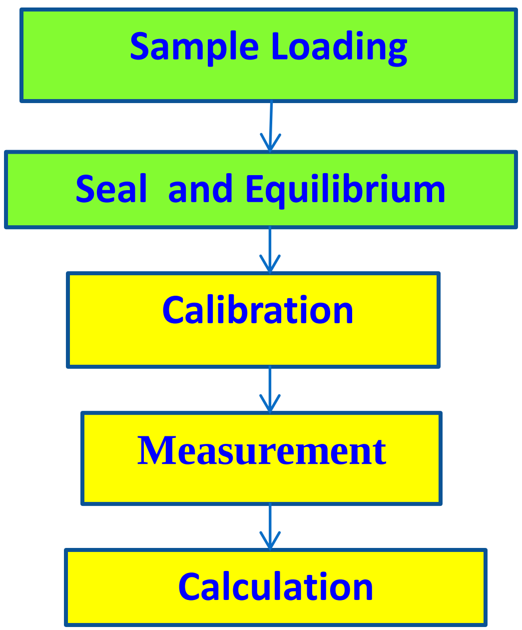
Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



10～300g

HPGe γ-Spectrometry Procedure

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



Plastic Cylinder

HPGe γ-Spectrometry Procedure

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

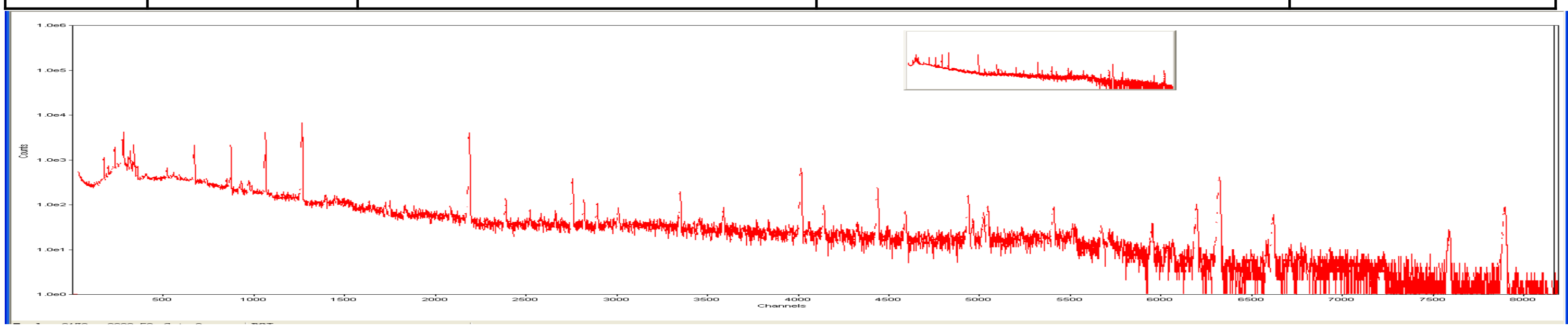
Sample Loading
↓
Seal and Equilibrium
↓
Calibration — Energy & Efficiency
↓
Measurement
↓
Calculation

HPGe γ-Spectrometry Procedure

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

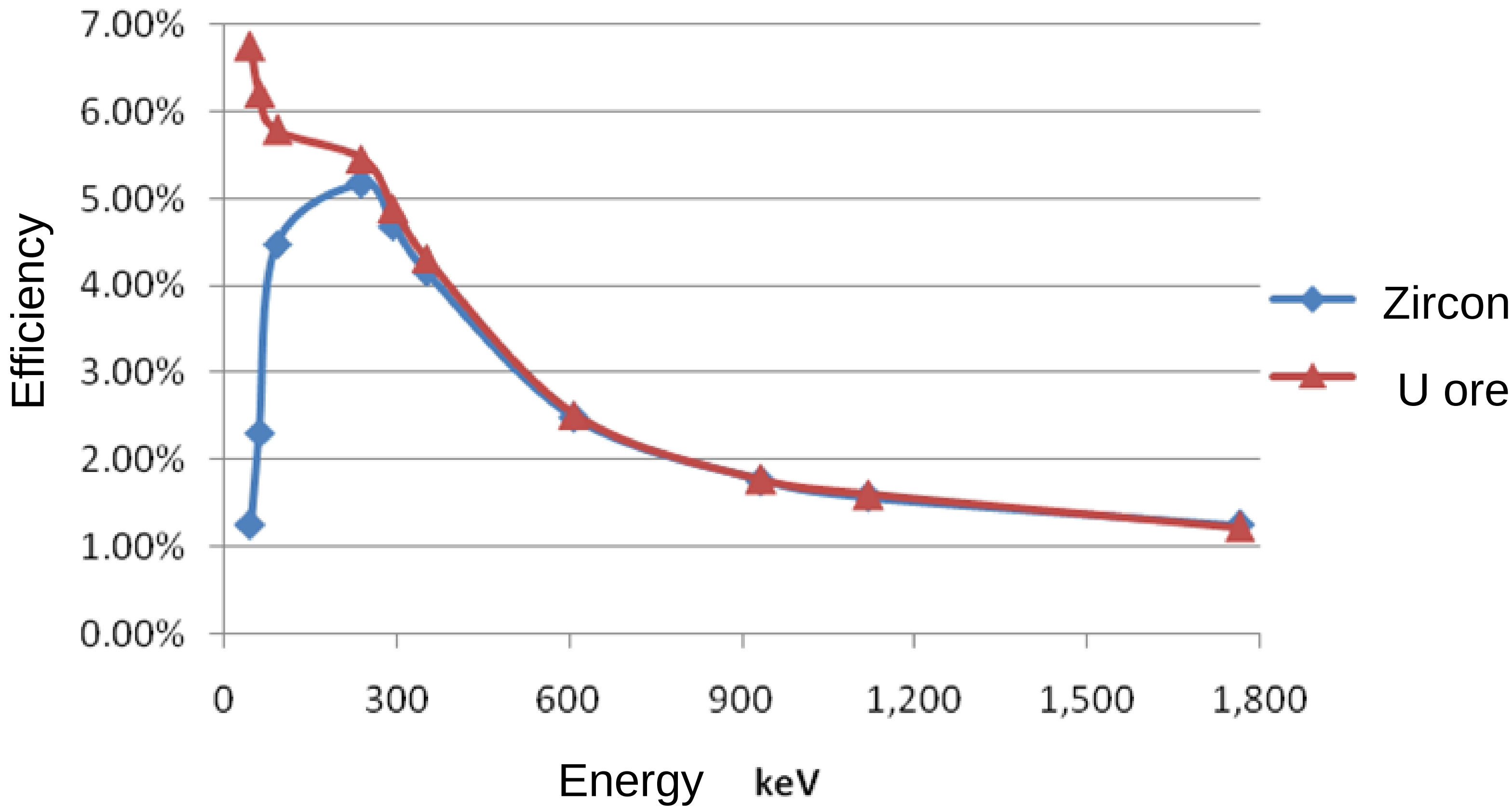
Energy Calibration and Peak Selection

Nr.	Parent nucleus	Equilibrium Daughter	Peak Energy （keV）	Selected Peak (keV）
1	^{238}U	^{234}Th、^{234m}Pa	63.3、92.6、1001.0	1001.0
2	^{232}Th	^{228}Ac、^{212}Pb、^{208}Tl	238.6、583.1、 911.1、 2614.7	911.1
3	^{226}Ra	^{214}Pb、^{214}Bi	295.2、351.93、609.31、 1120.4、1764.7	609.31



Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Detection Efficiency



Efficiency for Different Samples

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Energy Peak Selection

^{238}U(Bq/g)							
Nr.	Sample	Code	^{238}U Certified	^{238}U (63.3 keV) Measured	RE （%）	^{238}U(1001.3keV) Measured	RE （%）
1	Uranium Ore Uranium Thorium Radium CRMs	EJ180-80	23690.8	24867	4.73	23858	0.48
2	Uranium Ore Uranium Thorium Radium CRMs	EJ181-80	86048.5	85774	-0.32	83308	-3.18
3	Thorium CRMs	GBW04325		27.2	/	54.8	/
4	Uranium Ore Uranium Thorium Radium CRMs	GBW04119	1046.7	1148	8.82	958	-9.19
5	Zircon Sand CRMs	BCS-388	1961.0	1177	-66.61	3195	-9.36

Energy Peak Selection

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γSpectrometry

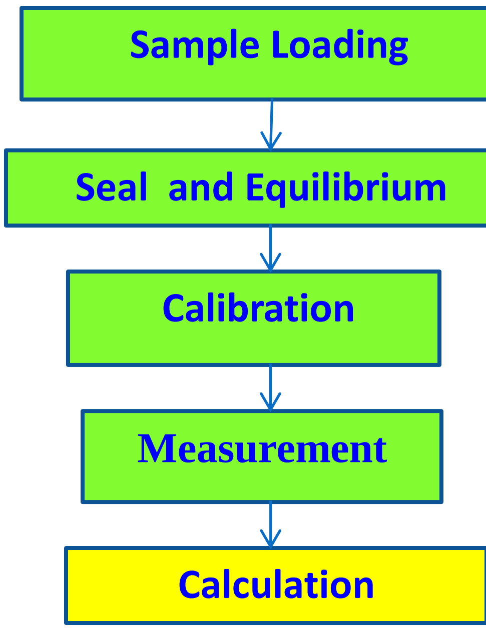
^{232}Th(Bq/g)							
Nr.	Sample	Code	^{232}Th Certified	^{232}Th(238.6 keV) Measured	RE（%）	^{232}Th(911.1 keV) Measured	RE（%）
1	Uranium Ore Uranium Thorium Radium CRMs	EJ180-80	174.1	162.4	-7.2	167.7	-4.17
2	Uranium Ore Uranium Thorium Radium CRMs	EJ181-80	9403.1	9108	-3.24	9561	1.57
3	Thorium CRMs	GBW04325	180.9	180.4	-0.28	184.6	2.05
4	Uranium Ore Uranium Thorium Radium CRMs	GBW04119	48.2	44.6	-8.04	50.1	3.3
5	Zircon Sand CRMs	BCS-388	671.3	603	-11.32	698	2.65

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Energy Peak Selection

^{226}Ra((Bq/g)							
Nr.	Sample	Code	^{226}Ra Certified	^{226}Ra (352 keV) Measured	RE （%）	^{226}Ra (609.3keV) Measured	RE （%）
1	Uranium Ore Uranium Thorium Radium CRMs	EJ180-80	23422.5	23031	-1.7	23019	-1.75
2	Uranium Ore Uranium Thorium Radium CRMs	EJ181-80	86180.9	88938	3.1	88728	2.86
3	Thorium CRMs	GBW04325		19.3	/	24.9	/
4	Uranium Ore Uranium Thorium Radium CRMs	GBW04119		1035	/	1040	/
5	Zircon Sand CRMs	BCS-388	3503.3	3248	-7.86	3374	-4.28

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



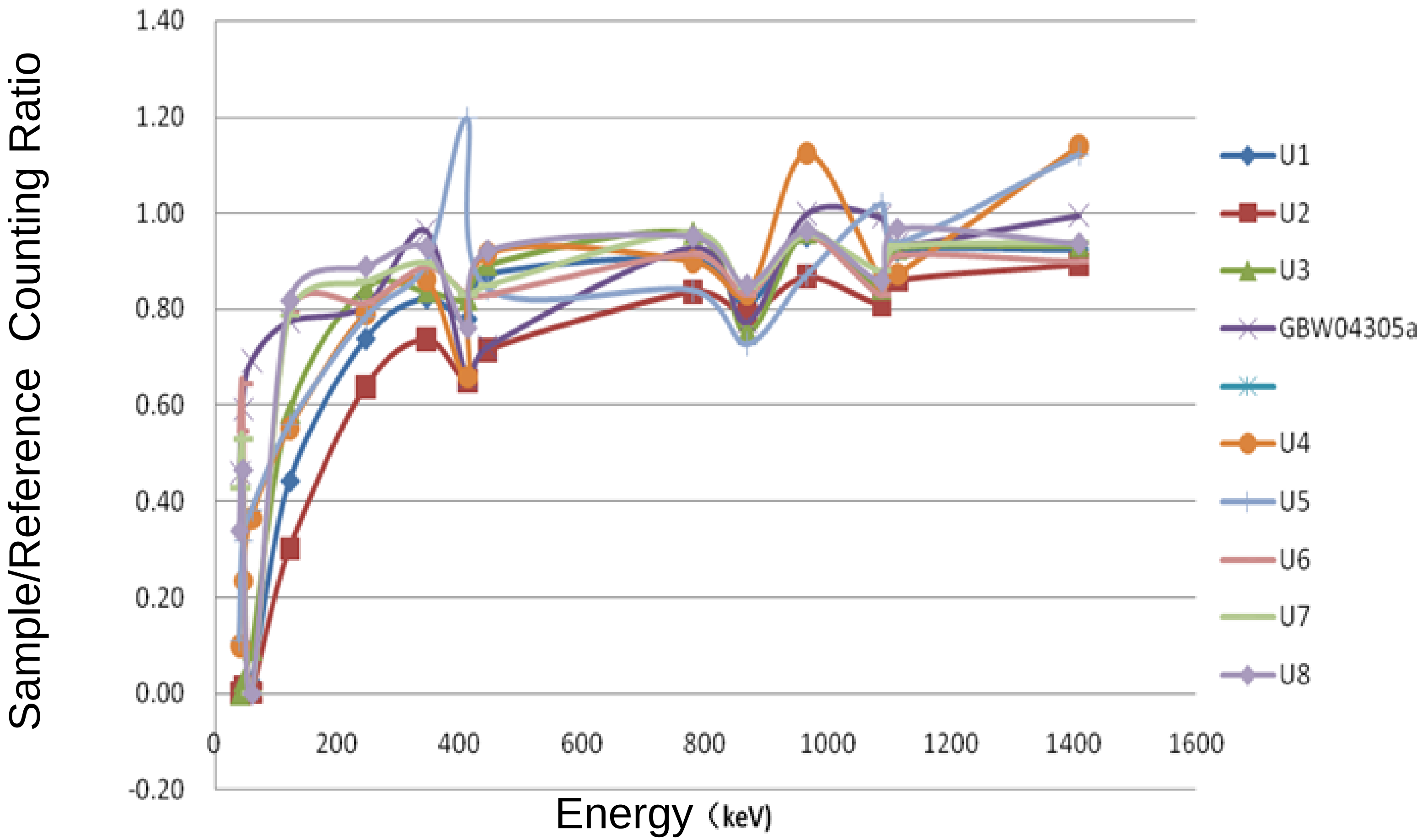
Self-absorption & Time

HPGe γ-Spectrometry Procedure

Samples Tested

Nr	Sample Code	Sample Type	Comment
1	U1	Zircon Sand	ZrO_2 52%
2	U2	Zircon Sand	ZrO_2 62%
3	U3	Monazite	
4	GBW04305a	Uranium Ore	U 0.32%
5	U4	Uranium Ore	
6	U5	Uranium Ore	
7	U6	Tantalum Ore	
8	U7	REEs	Nb_2O_5, Ta_2O_5
9	U8	REEs	ZrO_2 HfO_2

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry



Self-Absorption Curve for Different Samples

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Sample Loading

↓

Seal and Equilibrium

↓

Calibration

↓

Measurement

↓

Calculation — **Peak area method**

HPGe γ-Spectrometry Procedure

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Performance of HPGe γ-Spectrometry

- ✓Precision
- ✓Trueness
- ✓Detection Limits

HUANG Qiu-hong†, GUO Dong-fa* , WANG Tie-jian, TIAN Fei，ZHOULianghui, QUYing, LI Li. Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGeγSpectrometry [R]. BRIUG Report, 2017

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

^{238}U(Bq/g)

Precision

Nr.	Level-1	Level-2	Level-3	Level-4	Level-5
1	528	2679	3182	6673	9466
2	544	2669	3319	6745	9831
3	520	2730	3170	6730	9347
4	526	2639	3030	6537	10128
5	521	2623	3243	6773	9268
6	537	2703	3143	6790	9347
7	534	2693	3180	6848	9335
8	510	2737	3293	7081	9332
9	570	2543	3269	6745	9397
Average	532	2668	3203	6769	9495
SD	17.4	60.3	88.8	145.7	289.7
RSD%	3.3%	2.3%	2.8%	2.2%	3.1%

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Precision

^{232}Th(Bq/g)

Nr.	Level-1	Level-2	Level-3	Level-4	Level-5
1	70. 9	585	823	1529	5180
2	73. 5	553	815	1481	4857
3	70	587	838	1523	5083
4	65. 3	542	736	1414	4718
5	66. 9	541	783	1412	4830
6	68. 1	568	801	1508	5059
7	66. 7	530	758	1537	4773
8	68. 8	587	843	1514	5107
9	69. 7	571	801	1441	4780
Average	68. 9	563	800	1484	4932
SD	2. 5	21. 9	35. 7	49. 7	173. 7
RSD%	3. 6%	3. 9%	4. 5%	3. 3%	3. 5%

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

^{226}Ra((Bq/g)

Precision

Nr.	Level-1	Level-2	Level-3	Level-4	Level-5
1	416	2724	3094	8235	6530
2	473	2635	3076	8352	6715
3	412	2733	3160	8353	6620
4	397	2700	2963	8034	6419
5	444	2758	3160	8250	6491
6	407	2633	2990	8140	6460
7	450	2854	3374	8257	6490
8	414	2797	3103	8295	6632
9	403	2669	3083	8132	6508
Average	424	2723	3111	8228	6541
SD	25.6	73.5	118.8	106.9	95.2
RSD%	6.0%	2.7%	3.8%	1.3%	1.5%

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

^{238}U(Bq/g)

Trueness

Sample Type	REEs	Zircon	Vanadium	Anthracite	Niobium-Tantalum
Certified（Bq/g）	5133	9310	552	240	46905
Measures (Bq/g）	5457	9221	552	270	45973
RE（%）	6.32%	-0.96%	0.00%	12.45%	-2.00%
Acceptable RE(%)	13.80%	12.20%	20.80%	23.90%	8.60%
Evaluation	OK	OK	OK	OK	OK

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

^{232}Th(Bq/g)

Trueness

Sample Type	REEs	Zircon	Vanadium	Anthracite	Niobium-Tantalum
Certified（Bq/g）	3264	2960	21	42	4310
Measures (Bq/g）	3297	3110	21	41	4477
RE（%）	1.01%	5.07%	0.00%	-2.38%	3.87%
Acceptable RE(%)	12.10%	12.40%	29.70%	26.60%	11.60%
Evaluation	OK	OK	OK	OK	OK

Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

^{226}Ra((Bq/g)

Trueness

Sample Type	REEs	Zircon	Vanadium	Anthracite	Niobium-Tantalum
Certified（Bq/g）	60	9450	580	150	42324
Measures (Bq/g）	63	9078	583	153	43134
RE（%）	5.00%	-3.94%	0.52%	2.00%	1.91%
Acceptable RE(%)	25.10%	9.60%	16.90%	21.50%	6.60%
Evaluation	OK	OK	OK	OK	OK

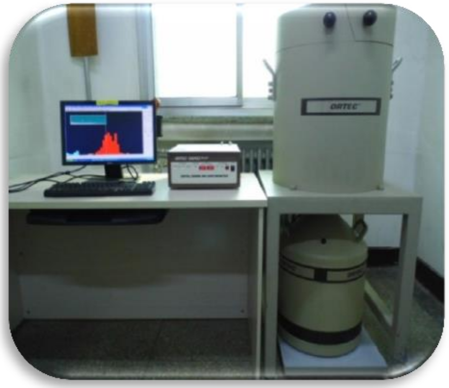
Method Validation for the Determination of Uranium,Thorium and Radium in UGSA samples by HPGe γ Spectrometry

Detection Limits

Nuclide	^{238}U			^{232}Th			^{226}Ra	
Energy（keV)	63.39	92.59	1001	238	583	911	351.7	609
Blank count rate 1（cps）	0.032	0.0378	0.00017	0.0266	0.00616	0.0081	0.0288	0.0174
Blank count rate 2（cps）	0.0316	0.0385	0.00016	0.0246	0.00554	0.0072	0.0276	0.0162
Blank count rate 3（cps）	0.034	0.0392	0.00015	0.0259	0.00616	0.008	0.0297	0.0185
Average Blank count rate（cps）	0.0325	0.0382	0.00016	0.0253	0.00595	0.0076	0.0283	0.0168
Calibration Factor（Bq/cps）	655	445	7741	73.6	190	252	91.1	121
Sample Weight（g）	300	300	300	300	300	300	300	300
Detection Limit (Bq/g)	0.00965	0.0071	0.008	0.00096	0.0012	0.00179	0.00125	0.00128

Typical Methods for UG & NORM Sample Analysis

XRF



HPGe γ-Spec.

CRMs

ICP-MS

MUA (U)

PC2100(Ra)

Typical Methods for UG & NORM Sample Analysis

Determination of U in UG & NORM Samples by ultraviolet pulse light –induced fluorescence micro uranium analyzer (MUA)

MUA

- ✓High sensitivity
- ✓Low detection (0.02ng/mL)
- ✓Suitable for most samples

(500，522，546 nm) (337nm)

$$F = 2.3 \times \phi \times I_0 \times \varepsilon \times C \times L$$

2. New Practice on Methods for D. U Th Ra

Sensitivity and Detection Limit

MUA

Measured Trial	1	2	3	4	5	6	7	8	9	10	11	12
Readout	76	76	74	74	78	77	78	75	74	75	77	74
Standard Deviation S_0	1.557											
$3S_0$	4.67											
Sensitivity	546											
D.L (ng/mL)	0.009											

Weight 0.1g, Volume 50mL, Sub-volume 0.5mL, Measuring Volume 5.0 mL, Method Detection Limit 0.15μg/g, Low determination limit 0.45μg/g U。

Precision Unit: μg/g

Element	Sample Type	Range /m	Repeatability	Reproducibility
U	Zircon	38.4～734	S_r=0.203+0.031m	S_R=2.22+0.05m

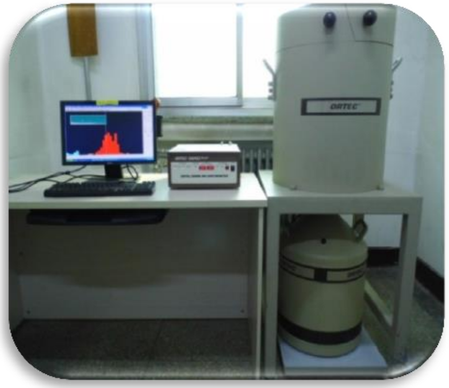
Trueness Unit: μg/g

Trail	Sample Nr.	Measured	Certified	RE（%）
1	BCS-CRM-388	278.4	288	-3.33
2	BCS-CRM-388	282.9	288	-1.77
3	BCS-CRM-388	279.3	288	-3.02
4	BCS-CRM-388	262.8	288	-8.75

MUA

Typical Methods for UG & NORM Sample Analysis

XRF



HPGe γ-Spec.

CRMs

ICP-MS

MUA (U)

PC2100(Ra)

Typical Methods for UG & NORM Sample Analysis

Determination of Ra-226 in UG & NORM Samples by Emanation Analyzer (PC2100)

PC2100

$$LLD \approx \frac{4.66 \times \sqrt{N_b} \times k}{(1-e^{-\lambda t}) \times m \times t_b}$$

LLD-Bq/g）； N_b-Background count； t_b-Time for background count（s）；
t-Radon accumulated time（s）； m-Sample weighted（g）

Lo w Limit of Determination of Ra-226

Trail	1	2	3	4	5	6	7	8	9	10	11	12
Counting time（s）	240	240	240	240	240	240	240	240	240	240	240	240
Count	16	20	12	15	23	20	17	13	22	18	21	12
Average Count	17.42											
LLD（Bq/g）	0.02											

PC2100

Precision Unit: Bq/g

Nuclide	Sample Type	Range /m	Repeatability	Reproducibility
Ra-226	Zircon	0.464~6.73	S_r=0.016+0.022m	S_R=0.019+0.038m

Trueness

Trail	Sample	^{226}Ra（Bq）	Added（Bq）	Recovery（%）
1	BCS-388	3.30	/	
2	BCS-388	67.3	66	97
3	BCS-388	70.9	66	102

PC2100

Outline

1. Background for uranium geology (UG) and NORM sample analysis
2. New Practice on Methods for Determination of U Th Ra
3. Summary

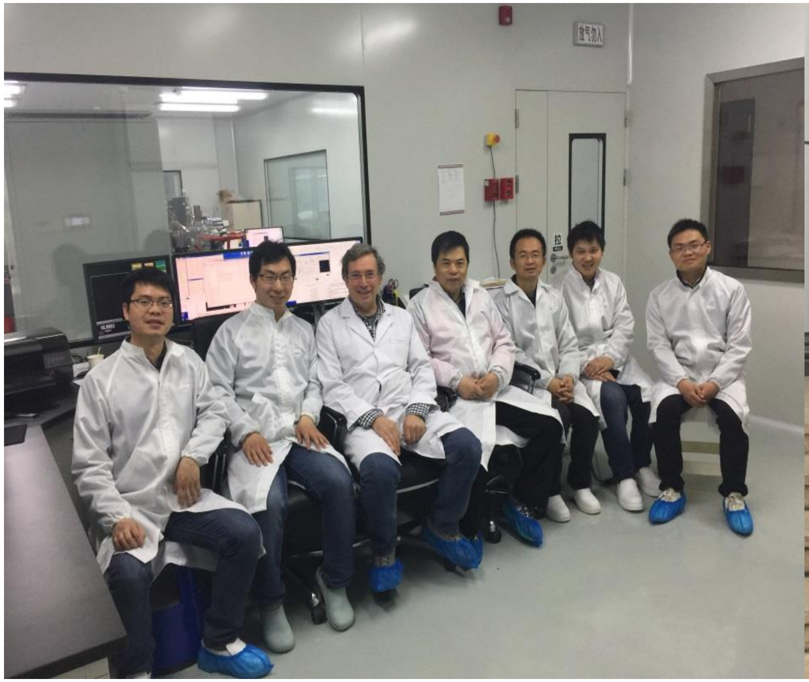
NORM: naturally occurring radioactive materials

UG & NORM Sample Analysis Strategy

XRF+GAMMA+ICP-MS+MUA+PC2100+X

- ✓**XRF: Multi-element analysis in solid samples, but not for Ra-226;**
- ✓**ICP-MS: U, Th and Others, Low Detection Limits, but pre-treatment;**
- ✓**HPGe γ Spectrometry: ^{238}U,^{232}Th, ^{226}Ra, NDA, but nuclide equilibrium needed;**
- ✓**MUA: high sensitivity and different sample type, but U only;**
- ✓**PC2100: different sample type, but Ra-226 only.**
- ✓**X: which one is a high sensitivity method for Th only?**

Thank you for your attention!



LG-SIMS Team

ICP-MS Team

http://www.albriug.com
guodongfa@263.net
13511062560