



DEVELOPMENTS AT THE NUCLEAR ANALYTICAL FACILITIES AT MLZ, GARCHING

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MLZ is a cooperation between:



Helmholtz-Zentrum Geesthacht Zentrum für Material- und Küstenforschung







Shut down since March 2020, until ~Sep 2024, then only thermal operation

Cold neutron beam (PGAA)

- Prompt Gamma Activation Analysis (PGAA)
 - In-beam activation analysis
 - In-beam liquid scintillation
 - Neutron radiography-driven PGAA + PGAI
- Neutron Depth Profiling (NDP)

Thermal neutrons – irr. channel in reactor, counting lab at RCM

• Neutron Activation Analysis (NAA)

Fast neutron beam (MedApp and Nectar)

- Fast Neutron induced Gamma Spectrometry (FaNGaS)
 - Eric Mauerhofer, Niklas Ophoven (Jülich Center for Neutron Research)
- GRAINS at Nectar (with Adrian Losko)





PGAA and in-beam NAA







PGAA facility at the neutron guide hall





The PGAA facility







Shielding arrangement at PGAA









- HPGe detectors in BGO
 - 60% PopTop +Lithoflex





• PGAA facility and the low-background counting station

- ⁶Li-containing plastic (2.5 mm)
- Sn sheets (4 mm) instead of Cd
- 10 cm of lead

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- 5mm boron rubber (40% B_4C)
- 5cm boron plastic (20% H_3BO_3)
- BGO annulus (Anti-Compton)

Status 2023

electronics

PGAA

low-bkg counting station

64k spectrometers, cooling without LN2 filling

New Focusing Guide Extension

More homogeneous High flux at every setup at sample:4×10¹⁰ cm⁻² s⁻¹

15

20

25

30

distance [cm]

10

-30

20

64k spectrometers

- Better resolution
- Better peak shapes for narrow peaks
- Simultaneous acquisition of Comptonsuppressed and – unsuppressed spectra
- List mode
 - Complete time dynamics

Cyclic activation for short-lived nuclides (offline)

Prompt and decay y spectra of Ag

In decay spectra baseline is much lower, ~1 OoM better DL ~3--4 OoM is possible in a separate counting position

MORIS: upgrade of

Prompt Gamma Activation Imaging (PGAI)

Feasibility shown (ANCIENT CHARM project, FRM2-Budapest collaboration)

- Collimate neutron beam and gamma detection.
- Acquire prompt gamma spectra of single voxels (in isocenter), non-destructive analysis.
- Test: 3D elemental map, too long
- Only selected spots

2009: inside an object at many spots

Fibula from Frankish Kingdom (8th century) found in Hungary, fragile, unknown production, unknown composition

(a) Layers front (c) Positions in middle (d) Positions (b) Positions in in back layer laver layer

3D elemental map: 3hours/voxel, 77 (30+27+20) voxels, 2×2×2 mm³, 10 days in FRM2

Applications of PGAI-NR

Test samples (U, Fe, Cu, Al) in closed lead container Analysis of a few interesting spots

- Radiography to find spots of interest inside sensitive complex objects nondestructively.
- Then gamma counting

16

2018-19: PGAI experiment at MLZ

Guided copper reliquiary pendant (phylacterion), rare piece from Mainz, 12th century Inside rich in Ca, probably bone **PGAI-NT**, attenuation is also used for identifying phases 16 voxel, 3 hours/voxel, voxel ~ 2 × 2 × 3mm³ (too large)

Heinzel, M., Schillinger, B., Kemper, D., Stieghorst, C., & Kluge, E. (2022). Discovery of a 12th-Century Enamelled Reliquary Pendant: Elemental Analysis and Content Visualisation Using Prompt Gamma Neutron Activation Analysis and Neutron Tomography. In INTERIM MEETING OF THE ICOM-CC METALS WORKING GROUP Roman amulet 16 pixel, 3 hours/pixel voxel ~ $2 \times 2 \times 3$ mm³

Detector upgrade for PGAI

Analysis of several voxels at the same time Detector cluster with a multichannel spectrometer

Detector cluster (7 - 15 detectors)7—15 spots in parallel

Like Advanced Gamma Tracking Array (AGATA)

Improved collimators

- One detector one voxel
- Multi-channel collimators to increase detection efficiency (goal: 10-20x faster)

Speeding up the measurement with 2 OoM

NAA

Neutron Activation Analysis collaboration with Radiochemie München

NAA instrument

- The unique possibilities of FRM2 reactor have not been exploited until now.
 - Highly thermalized
 - High flux
- Recent developments
- NAA is now an instrument
- 3 HPGe detectors with digital spectrometers (Lynx, Canberra)
- Evaluation: Hyperlab + k0-SolCoi
- Too much manual work
- During working hours only
- High demand \rightarrow 40 \leftrightarrow 168 hours/week

increase sample throughput.

• Automation of counting

Forschungs-Neutronenquelle Heinz Maier-Leibnitz

- enabling measurements 24/7 with automatic
- SMART-controlled sample changers (automatically measurement optimizing conditions)
- Shorten transfer time between reactor and lab
 - Rabbit system to RCM
- Improve the evaluation procedure (ErUM-Data proposal).
- Following the time dynamics with list mode acquisition
- Automation of the spectrum evaluation and the analysis

MORIS upgrade plans

- In radioactive decay, a β particle is emitted, often followed by γ radiation.
 - Famous pure β emitters: ³²P, ³⁵S, ²⁰⁹Pb, ...
 - Isomers (pure γ emitters): e.g. Hg, ²⁰⁷Pb
 - (Neutrino remains undetectable)
 - β spectrum is not characteristic
- One can use β-γ coincidence/anticoincide
 - The pure emitters
 - Low-activity decay products
 - β detection ~100%, γ should be high too
- Several geometries depending on the emphasis
 - 4πβ-γ
 - 4πβ + ~4πγ
 - γ-γ concidence
- List-mode acquisition
- Dynamic on-the-fly evaluation

23

The PGAA database project

Budapest

1997–1998 establishment of PGAA data library in thermal beam

2001—2004 measurements in cold beam

2006 – partial revision of data (NIST, Budapest, Garching)

2012 – 2020, new measurements for the library

2021, 2022 library updates

2023 new standardization measurements in Budapest

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5	3 Li	6.941	1051.817	0.048	7	0.00426	2.00	2.00	10.95	15.90		3		updated 2022.03	2.120591		AgCuCe	Ag -0.45	0.002083	Cu -0.40	0.000147	Ce 0.71	7.57E-05				
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4	4 Be	9.012	5956.602	0.092	9	0.00011	5.00	5.50	2.31	1.60		0		updated 2022.03	4.809895		Ir A-C-	IF 1.49	0.009299	0-455	0.000000						
5	4 Be	9.012	6809.579	0.099	9	0.00474	2.50	3.50	100.00	59.00		4		updated 2022.03	5.135796		Asse	AS 0.53	0.002137	Se 1.55	0.000369						-
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/	6 C	12.011	1261./08	0.057	12	0.00117	1.00	1.00	45.10	100.00	1	1		updated 2022.03	2.300976		ZnBaSm	Zn -0.54	0.000662	Ba -0.17	7.61E-05	Sm 0.35	0.186094				
8	6 C	12.011	3684.016	0.069	12	0.00116	0.80	0.80	44.70	38.00		3		updated 2022.03	3.807789												
9	6 C	12.011	4945.302	0.066	12	0.002594	0.60	0.60	100.00	60.00		2		updated 2022.03	4.392283												
0	7 N	14.007	1678.244	0.029	14	0.006254	1.50	1.50	26.34	47.15	3	3			2.622466												
1	7 N	14.007	1681.174	0.043	14	0.001296	2.70	2.70	5.46	9.76	14	4			2.624588		CeGd	Ce 1.05	5.1E-05	Gd 1.10	0.766319						
2	7 N	14.007	1853.944	0.052	14	0.000474	4.50	4.50	2.00	3.31	19	9		unc updated 2022.03	2.746814		Ва	Ba -0.73	5.39E-05								
3	7 N	14.007	1884.853	0.031	14	0.0145	0.50	0.50	61.07	100.00	1	1			2.768112		SnSc	Sn 0.39	2.23E-05	Sc 0.93	0.001991						
4	7 N	14.007	1988.532	0.077	14	0.000294	5.80	5.80	1.24	1.94	22	2			2.838384		Zr	Zr -0.39	5.56E-05								
5	7 N	14.007	1999.693	0.032	14	0.003208	1.70	1.70	13.51	21.12	11	1			2.845845		Cr	Cr 1.32	0.000377								
6	7 N	14.007	2520.446	0.039	14	0.004246	1.80	1.80	17.88	22.99	10	0			3.174539												
7	7 N	14.007	2830.8	0.048	14	0.001331	2.60	2.60	5.61	6.47	17	7			3.355151												
8	7 N	14.007	3013.631	0.061	14	0.000642	3.80	3.80	2.70	2.94	20	0			3.457137		F	F 0.98	2.14E-05								
9	7 N	14.007	3531.98	0.051	14	0.006857	1.80	1.80	28.88	26.56	9	9			3.731156		Pr	Pr 0.74	0.000214								
0	7 N	14.007	3677.802	0.05	14	0.011359	1.30	1.30	47.84	42.06	2	4			3.804688		CoPrLa	Co -0.85	0.001646	Pr 0.25	0.000283	La 1.44	0.000985				
1	7 N	14.007	3855.552	0.08	14	0.000606	3.80	3.80	2.55	2.13	21	1			3.892441												
2	7 N	14.007	3884.353	0.106	14	0.000424	4.30	4.30	1.79	1.47	23	3			3.906474		Dy	Dy 0.82	0.025652								
3	7 N	14.007	4508.687	0.063	14	0.012862	1.60	1.60	54.17	37.24	(6			4.199168												
4	7 N	14.007	5268.984	0.072	14	0.023743	1.50	1.50	100.00	55.69		2			4.530137		CrSnCoSiOs	Cr -0.88	0.000877	Sn 0.05	1.68E-05	Co 0.93	0.006098	Si 1.38	6.41E-05 (Os 1.85	0.0
5	7 N	14.007	5297.662	0.153	14	0.016728	1.60	1.60	70.46	38,94		5			4,542149		Snl	Sn 0.30	1.88E-05	10.89	0.000256						
6	7 N	14.007	5533.251	0.076	14	0.015677	1.60	1.60	66.03	34.28		7			4.63965		Tlir	TI 0.10	0.00026	Ir 1.48	0.007234						
7	7 N	14.007	5561.948	0.078	14	0.008627	1.80	1.80	36.33	18.72	13	2			4 651387		LSnBaSh	1-1.81	0.000492	Sn -0.35	2.33E-05	Ba -0.30	2.76E-05	Sh 1 43	0.000164		
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3	80	15.999	8/0.082	0.034	16	1.55E-04	2.00	2.00	100.00	100.00	1	1		updated 2022.03	1.951561		GurdiNBBa	600.17	2.702187	10 0.35	0.000415	NB 0.41	6.18E-05	58 0.85	9.01E-05		
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PGAA Evaluation

ProSpeRo 5

fits masses and energies to E and σ values of the library

O from stoichiometry detection limits corrects for backgrounds N using pressure corrects for interference with B interference correction unc-s may increase self-attenuation iteratively from Hubbel (NIST) γattenuation database

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7	11	Na	22.99	2.10E	-3	2.6	6 05E 6	0.0	2.10E-3	1	2.83E-3	2.6	1.2		2.8	0.91	2.9	0.51	2.7	0.69	2.9		
9	14	Si	28.09	0.0	75	2.6	0.002-0	0.0	0.08	4	0.05	2.6	36		1.8	33	2.0	18	2.2	39	1.8		
10	17	CI CI	35.45	2.22E	-5	5.		0.0	2.22E-5	-1	2.22E-5	5.	80	ppm 2	5.	100 ppm	5.	54 ppm	5.	54 ppm	5.		
12	20	Ca	40.08	1.91E	-2	2.3		0.0	1.91E-2	2	2.68E-2	2.3	6.4		2.5	8.3	2.4	4.7	2.2	6.5	2.4		
13	21	Sc	44.96	3.51E	-5	5.		0.0	3.51E-5 6.21E-3	3	5.38E-5	5. 22	110	ppm	2.5	150 ppm	5. 25	90 ppm	5.	130 ppm	5.		
15	23	v	50.94	8.65E	-5	6.		0.0	8.65E-5	5	1.54E-4	6.	230	ppm	6.	370 ppm	6.	210 ppm	6.	380 ppm	6.		
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MORIS: Gamma-Ray Analysis from Inelastic Neutron Scattering (GRAINS)

- New, unique instrument
- Offers simultaneous elemental mapping and fast neutron radiography
 - n and γ penetrate through matter easily
 - Sample can be several mm thick
 - Any complex sample
 - Cultural heritage, energy, electronic waste (heavy elements)
- Principle: fast neutron in, fast neutron and gamma-ray out in coincidence
 - Delayed coincidence because of neutron ToF
- Fast neutrons detected by a scintillator screen
- Gamma rays detected perpendicularly
 - Neutron energy, detection angle, niveau lifetime cause peak broadening
 - Worse energy resolution
 - Affect coincidence timing

Planned at NECTAR

- Flux: 5×10⁷ cm⁻² s⁻¹
- Low gamma background
 - (<MedApp, <Fangas)
- Fast neutron imaging is practically available
 - Neutron direction is assumed to remain the same
 - Time resolution ~10ns
- Gamma detection perpendicular to beam at sample
 - Energy shift due to angle change
 - Time resolution ~10ns

Utilizing prompt gamma in combination with neutron Time-of-Arrival

28

- Many new developments in lacksquareimaging
- Timepix detector

FRM II

• To reduce beam background:

Scraping/Collimating the incoming beam

Using as many modules as SR10 can fit

"portable" design on wheels to move in/out

29

Applications

User statistics: PGAA and in-beam AA

- Archeology
- D/H rate in deuterated samples
- B in different matrices
- H in difefernt matrices
- Air filters, other environmental
- Nuclear data measurements
- Nuclear physics
- High-flux irradiation

Overbooking factor around 2

Archaeometry

Chlorine in iron objects

- Slow down the corrosion of artifacts
- Understand the corrosion of iron
- Britta Schmutzler, Friedrich Wagner

Carbonate layers in aqueducts

- Trace elements
- Cornelius Passchier

Applications

Lichen checking air pollution

z	EI	м	m	unc	m	unc	m	ox.	m	unc	mol%	unc	w%	unc
			meas	%	Bkg	%	net	st.	ΟΧ	%		%	el/el	%
1	Н	1.008	1.18E-2	0.4	2.44E-7	1.7	1.18E-2	1	0.11	0.4	48.6	2.2	6.17	4.
5	В	10.81	1.14E-6	0.4	3.87E-9	0.9	1.13E-6	3	3.65E-6	0.4	4.35 ppm	2.2	5.92 ppm	4.
6	С	12.01	0.107	3.1	3.50E-2	3.0	0.07	4	0.26	5.	25	4.	38	5.
7	Ν	14.01	2.32E-3	2.1	_	0.0	2.32E-3	5	8.95E-3	2.1	0.69	3.0	1.21	5.
13	ΑΙ	26.98	7.32E-4	2.1	1.38E-4	2.0	5.93E-4	3	1.12E-3	2.6	0.091	3.4	0.31	5 .
14	Si	28.09	1.85E-3	3.1		0.0	1.85E-3	4	3.95E-3	3.1	0.27	3.7	0.97	5 .
16	S	32.07	2.47E-4	2.6		0.0	2.47E-4	6	6.17E-4	2.6	320 ppm	3.3	0.129	5.
17	CI	35.45	9.91E-5	1.7	9.27E-8	2.4	9.90E-5	-1	9.90E-5	1.7	116 ppm	2.7	0.052	4.
19	Κ	39.1	9.92E-4	1.7		0.0	9.92E-4	1	1.20E-3	1.7	0.105	2.7	0.52	4.
22	Ti	47.87	3.50E-5	3.0		0.0	3.50E-5	4	5.84E-5	3.0	30 ppm	3.7	180 ppm	5.
25	Mn	54.94	5.05E-6	7.		0.0	5.05E-6	3	7.25E-6	7.	3.8 ppm	8.	26 ppm	9.
48	Cd	112.4	3.06E-8	4.0		0.0	3.06E-8	2	3.49E-8	4.0	0.0113ppm	5.	0.16 ppm	6.
62	Sm	150.4	3.73E-8	2.8		0.0	3.73E-8	3	4.33E-8	2.8	0.0103ppm	3.5	0.19 ppm	5.
64	Gd	157.3	4.45E-8	6.		0.0	4.45E-8	3	5.13E-8	6.	0.012ppm	6.	0.23 ppm	7
		0	_											
8	0	16	0.096	7.		0.0	0.10	-2		7.	25	6.	50	4.
		0					-							
11	Na	22.99	5.22E-5	0.7		0.0	5.22E-5	1	7.03E-5	0.7	94 ppm	2.3	273 ppm	4.
17	CI	35.45	1.02E-4	3.0		2.4	1.02E-4	-1	1.02E-4	3.0	120 ppm	3.7	0.053	5.
20	Ca	40.08	5.54E-3	13.		0.0	5.54E-3	2	7.75E-3	13.	0.6	13.	2.9	13.
21	Sc	44.96	3.36E-8	4.		0.0	3.36E-8	3	5.15E-8	4.	0.031ppm	5.	0.18 ppm	6.
23	V	50.94	6.64E-7	23.		0.0	6.64E-7	5	1.19E-6	23.	0.5 ppm	23.	3 ppm	23.
25	Mn	54.94	5.06E-6	2.2		0.0	5.06E-6	3	7.28E-6	2.2	3.8 ppm	3.1	26 ppm	5.
26	Fe	55.85	1.00E-4	15.		8.	1.00E-4	3	1.43E-4	15.	70 ppm	15.	0.05	15.
27	Co	58.93	1.25E-7	12.		0.0	1.25E-7	2	1.59E-7	12.	0.09 ppm	13.	0.7 ppm	13.
35	Br	79.9	2.00E-6	8.		0.0	2.00E-6	-1	2.00E-6	8.	1.0 ppm	9.	10 ppm	9.
38	Sr	87.62	1.09E-5	8.		0.0	1.09E-5	2	1.29E-5	8.	5.2 ppm	8.	57 ppm	9.
63	Eu	152	7.21E-9	8.		0.0	7.21E-9	3	8.35E-9	8.	0.0020ppm	9.	0.038ppm	9.
		0												

−PGAA

stoichiometry

- ibNAA

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Applications

Typical sample: many major and minor components

Micro-meteorites

- Mass ~0.3—2mg
- In 0.5µm Mylar (PET) foil
- No Teflon string

1 mg

z	EI	М	m meas	unc %	m Bka	unc %	m net	ox. st.	m ox	unc %
1	Н	1.008	5.47E-6	0.8	1.36E-6	1.5	4.11E-6	1	3.67E-5	1.2
5	В	10.81	6.74E-7	0.3	1.15E-8	1.0	6.63E-7	3	2.13E-6	0.3
13	AI	26.98	4.51E-5	2.2	1.96E-5	2.5	2.55E-5	3	4.82E-5	4.
14	Si	28.09	9.22E-5	2.8	1.96E-5	3.7	7.26E-5	4	1.55E-4	3.7
16	S	32.07	4.67E-6	14.		0.0	4.67E-6	6	1.17E-5	14.
17	CI	35.45	1.09E-6	18.	1.15E-7	22.	9.71E-7	-1	9.71E-7	20.
20	Ca	40.08	2.10E-5	19.		0.0	2.10E-5	2	2.94E-5	19.
22	Ti	47.87	2.45E-4	2.6	1.40E-7	22.	2.45E-4	4	4.09E-4	2.6
24	Cr	52	4.03E-6	14.		0.0	4.03E-6	ო	5.89E-6	14.
25	Mn	54.94	8.75E-6	2.0		0.0	8.75E-6	ო	1.26E-5	2.0
26	Fe	55.85	2.31E-4	2.4	1.16E-6	10.	2.30E-4	ო	3.28E-4	2.4
27	Со	58.93	3.06E-6	3.1		0.0	3.06E-6	2	3.89E-6	3.1
60	Nd	144.2	8.89E-7	13.		0.0	8.89E-7	3	1.04E-6	13.
62	Sm	150.4	5.32E-8	6.		0.0	5.32E-8	3	6.16E-8	6.
64	Gd	157.3	9.89E-8	7.		0.0	9.89E-8	3	1.14E-7	7.

R.B. Firestone

Thank you!

1.08.2023

IN

US