

Nuclear and related analytical techniques in archaeological and ecological studies



Supervisors: Dr. A. Dmitreiv; Dr. Wael Badawy

Group of neutron activation analysis

Frank Laboratory of Neutron Physics

Group members



Fouad Ismail El Agwany

Assistant Lecturer , Physics department, Faculty of
Science – Menoufia University

fouadesmail@yahoo.com

Romisaa Gamal Mahmoud

Assistant Lecturer, Faculty of Engineering and
technology, Future University.

romisaa.abdelrhman@fue.edu.eg



2023/06/02



Outlines

- History and Discovery of the neutron
- Neutron Activation Analysis (NAA).
- Types of NAA
- Pross and Cons of NAA
- Applications
- NAA Procedures
- Genie 2000
- Case Study
- Outcomes and Capacity building

Neutron Discovery



James Chadwick
(1891-1974)

1932 “Possible Existence of a Neutron”– February

“The Existence of a Neutron”

George Charles de Hevesy was a Hungarian radiochemist and Nobel Prize in Chemistry laureate 1943. The discovery of Neutron Activation Analysis in 1936.

The discovery of Neutron Activation Analysis in 1936.

Hilde Levi was the assistant of George Charles de Hevesy, applying Hevesy's radioactive indicator technique in biology, an application which had recently been made possible by the discovery of artificial radioactivity in 1934.



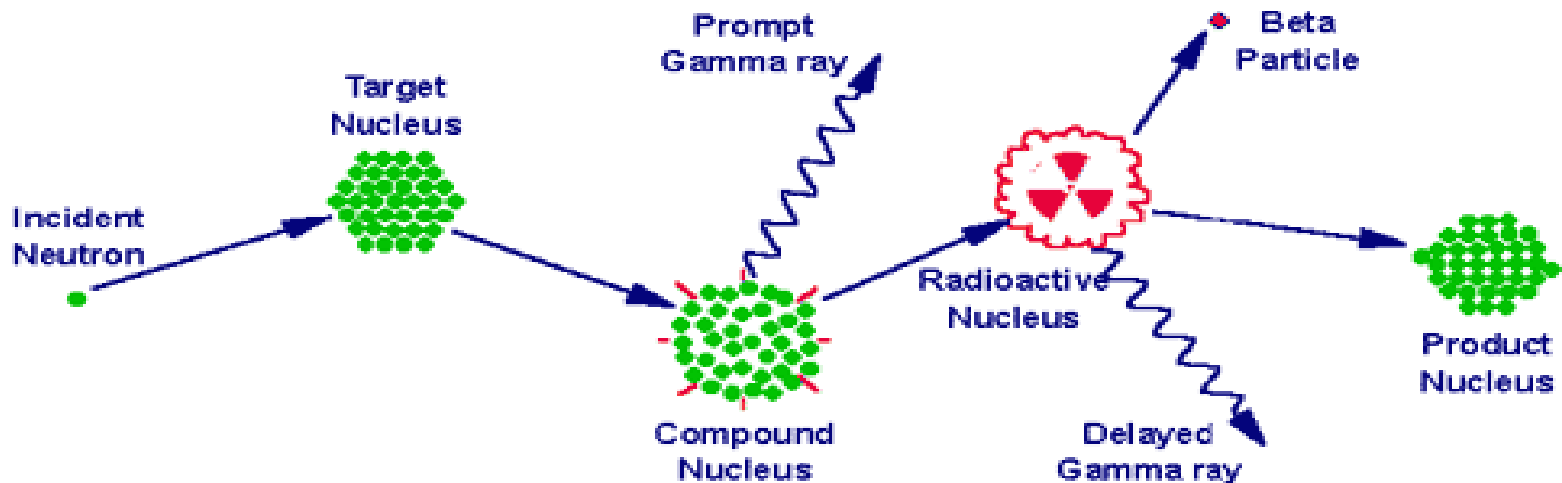
George Charles de Hevesy



Hilde Levi

Neutron activation analysis

- It is a sensitive multi-element analytical technique used for both qualitative and quantitative analysis of major, minor, trace and rare elements in samples from almost every field of scientific or technical interest.



Non-destructive

The resulting radioactive sample is kept intact

2023/06/02

Types
of
NAA

Destructive

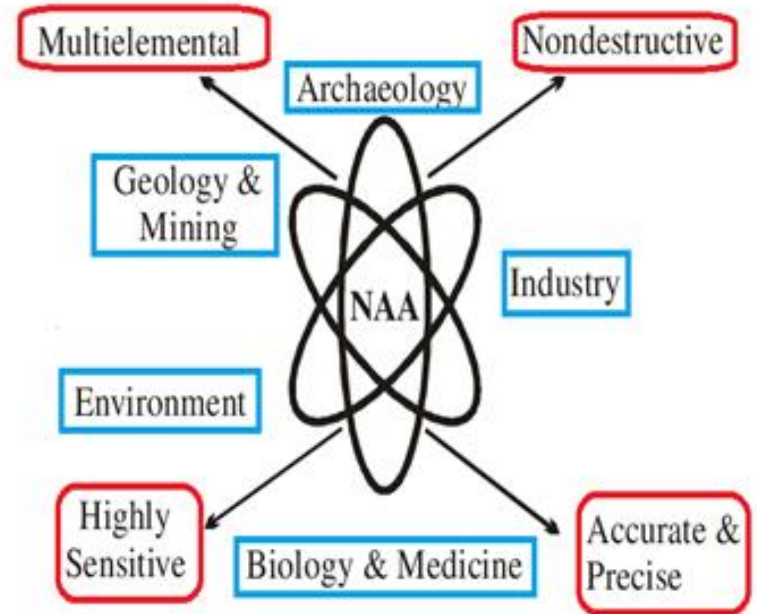
The resulting radioactive sample is decomposed



Why NAA?????

Pros

- ❖ **Industry** (synthesis of diamonds).
- ❖ **Biology** (Natural dispersion of elemental content in normal and tumor tissues).
- ❖ **Medicine** (Neutron activation analysis used to study of some indicators of water-salt metabolism).
- ❖ **Environment** (Investigation of element content of natural water by the neutron activation analysis method using the adsorption complexes).
- ❖ **Archeology** (Analysis of archaeological and museum objects from Russia and other countries)

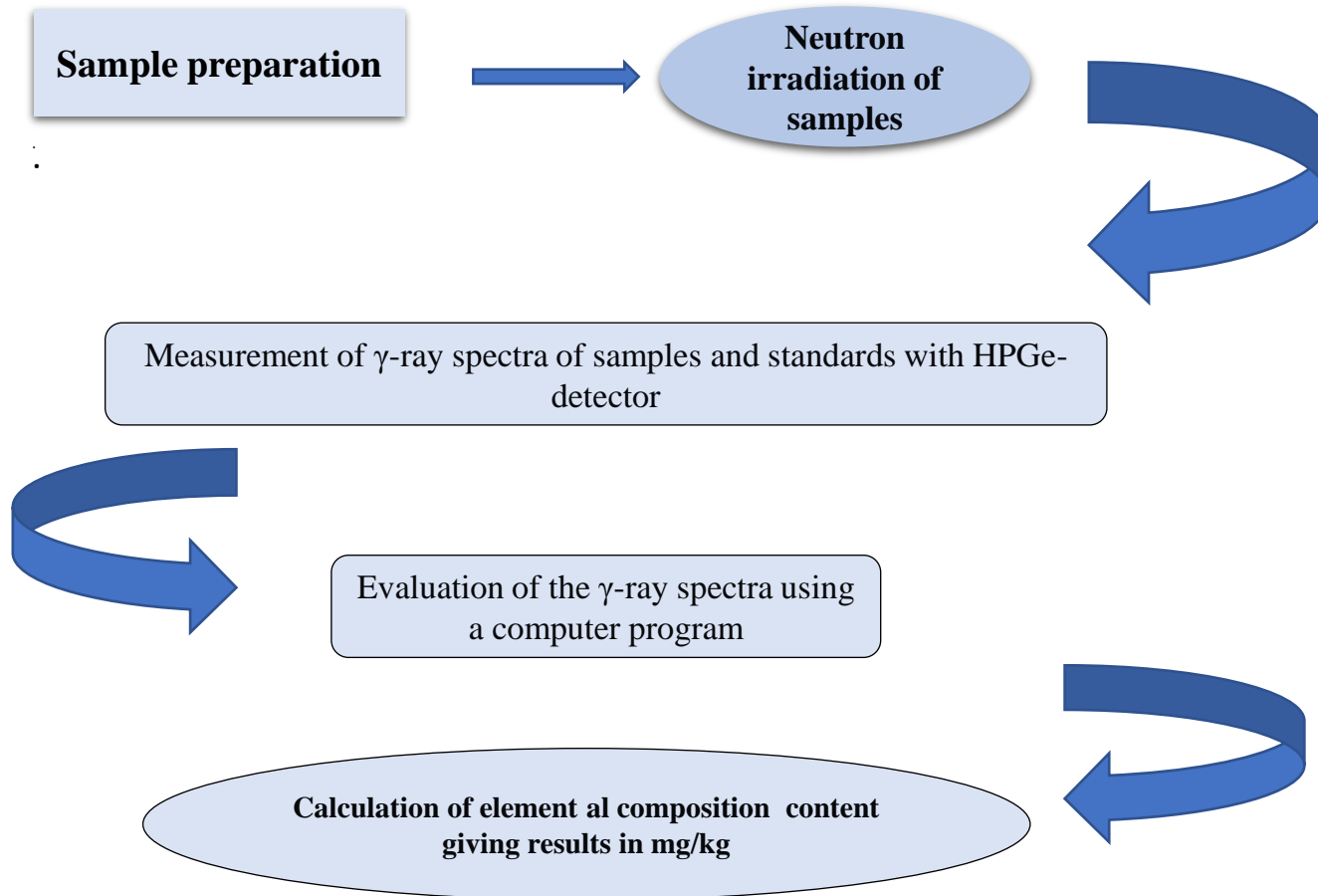


Cons

- **Need for neutron source**
- **Work with radioactive materials**
- **Time of analysis.**

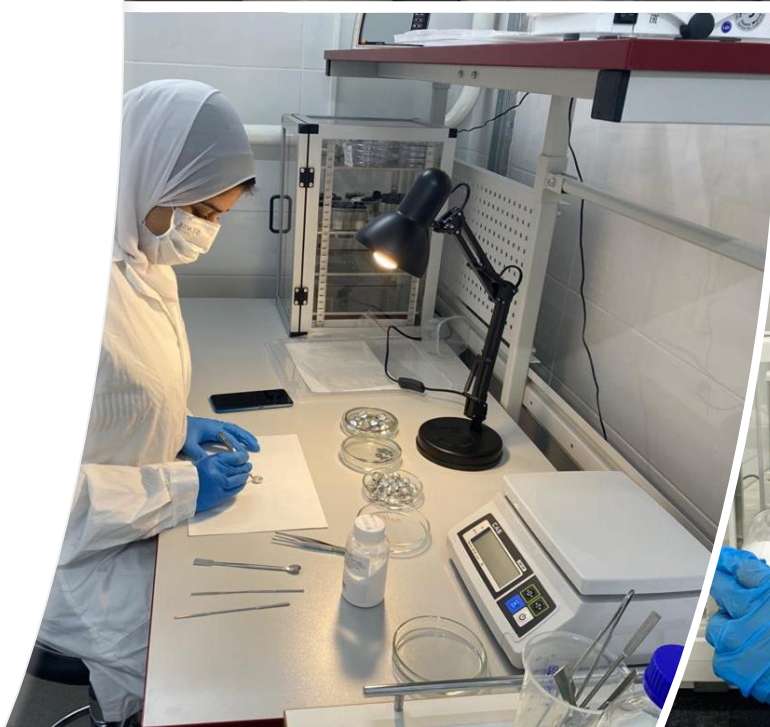
| | | | | | | | | | | | | | | | | | | | |
|----|----|------|----|----|----|----|----|----|----|----|----|----|----|----|----|----|--|--|----|
| H | | | | | | | | | | | | | | | | | | | He |
| Li | Be | | | | | | | | | | B | C | N | O | F | | | | Ne |
| Na | Mg | | | | | | | | | | Al | Si | P | S | Cl | | | | Ar |
| K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br | | | Kr |
| Rb | Sr | Y | Zr | Nb | Mo | Tc | Ru | Rh | Pd | Ag | Cd | In | Sn | Sb | Te | I | | | Xe |
| Cs | Ba | La* | Hf | Ta | W | Re | Os | Ir | Pt | Au | Hg | Tl | Pb | Bi | Po | At | | | Rn |
| Fr | Ra | Ac** | | | | | | | | | | | Rf | Db | Sg | Bh | | | Hs |
| | | | | | | | | | | | | | | | | | | | |
| | | * | Ce | Pr | Nd | Pm | Sm | Eu | Gd | Tb | Dy | Ho | Er | Tm | Yb | Lu | | | |
| | | ** | Th | Pa | U | Np | Pu | Am | Cm | Bk | Cf | Es | Fm | Md | No | Lw | | | |

NAA Procedure



Sample Preparation

- Samples are washed and air-dried
- Samples are homogenized by an agate ball mill machine
- samples are weighed and capsulated



Sample Packing for irradiation



Samples wrapped in polyethylene bags and aluminium container for short- and long-lived irradiations, respectively.



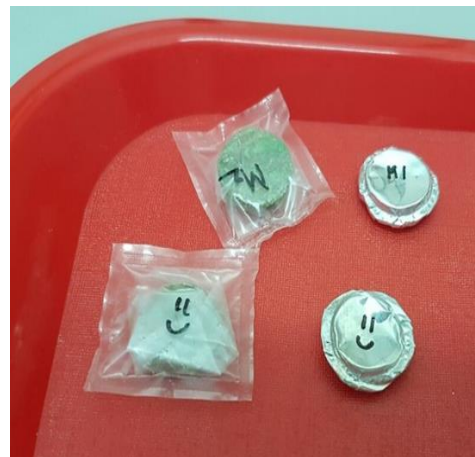
Samples placed in transport capsules



Short-lived isotopes



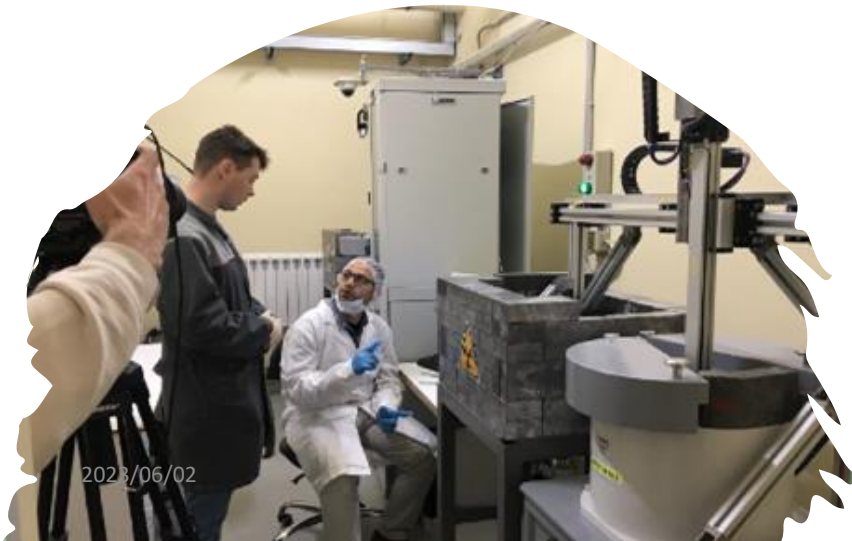
Long-lived isotopes





Pneumatic transport system at the IREN Facility is designed for sample irradiation

- Pneumatic transport system at the IREN Facility is designed for sample irradiation,
- the samples are delivered through the pneumatic system
- the channel is chosen, the time of irradiation is set,
- after that the samples transported for irradiation,
- after that the sample are delivered back after the irradiation after that we can detect gamma rays by using High Purity Germanium Detector.

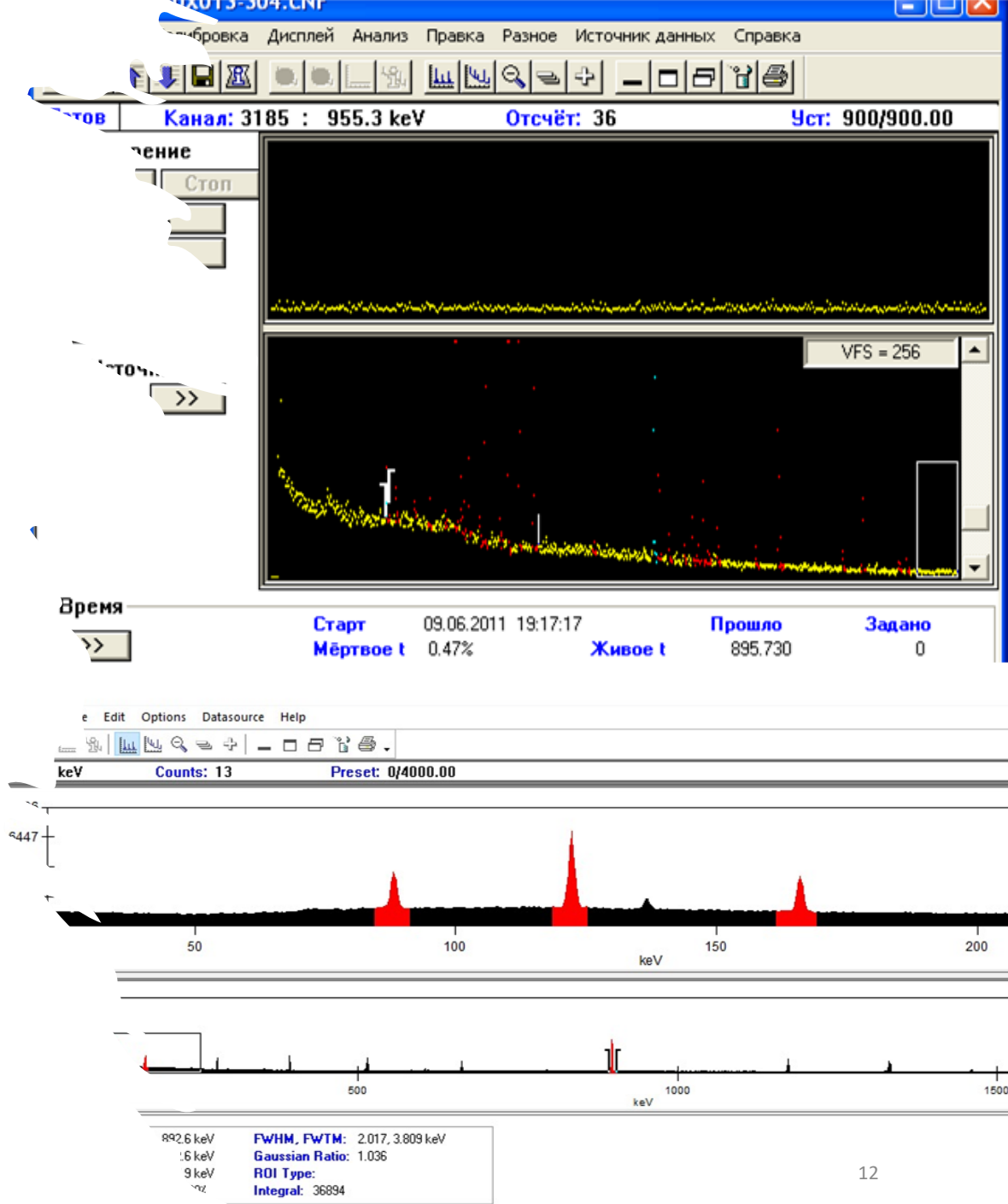


2023/06/02

Spectra processing using Genie2000

The program allows minimizing human involvement in routine long-term

➤ Software systems: **Genie 2000™ (Canberra)**



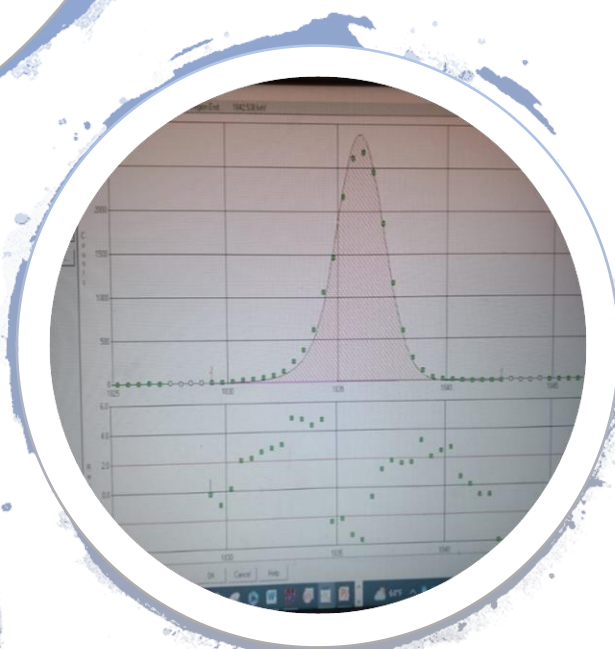
***** NUCLIDE IDENTIFICATION REPORT *****

Sample Title: CERNOEYL
 Nuclide Library Used: C:\GENIE2K\CAMELIES\STDLIB.NIB

IDENTIFIED NUCLIDES

| Nuclide Name | Id Confidence | Energy (keV) | Yield (%) | Activity (uCi/) | Activity Uncertainty |
|--------------|---------------|--------------|-----------|------------------|----------------------|
| BE-7 | 0.948 | 477.59* | 10.42 | 8.23319E-003 | 2.71737E-004 |
| NA-22 | 0.996 | 1274.54* | 99.94 | 3.77786E-004 | 1.39391E-005 |
| SC-46 | 0.983 | 889.25* | 99.98 | 6.67236E-005 | 1.14934E-005 |
| | | 1120.51* | 99.99 | 1.10917E-003 | 2.34273E-005 |
| MN-54 | 0.879 | 834.83* | 99.97 | 2.17337E-004 | 1.18859E-005 |
| CO-58 | 0.914 | 810.76* | 99.40 | 4.67983E-003 | 1.11019E-004 |
| Y-93 | 0.372 | 266.90* | 6.90 | 6.80759E+000 | 9.72548E-001 |
| | | 947.10 | 1.95 | | |
| | | 1917.80 | 1.40 | | |
| NB-95 | 0.997 | 765.79* | 99.81 | 7.43515E-003 | 1.70976E-004 |
| ZR-95 | 0.970 | 724.18* | 43.70 | 1.13056E-002 | 3.28527E-004 |
| | | 756.72* | 55.30 | 6.78248E-003 | 2.07216E-004 |
| TC-96 | 0.467 | 778.22* | 99.76 | 2.39376E-003 | 6.85586E-005 |
| | | 812.54* | 82.00 | 2.86873E-002 | 1.58081E-003 |
| | | 849.86 | 98.00 | | |
| | | 1126.85* | 15.20 | 8.71123E-003 | 7.39228E-004 |
| ZR-97 | 0.615 | 254.15* | 1.25 | 7.36708E+000 | 2.63815E-001 |
| | | 355.39* | 2.27 | 5.77123E-001 | 1.08670E-001 |
| | | 507.63 | 5.30 | | |
| | | 602.52 | 1.39 | | |
| | | 743.36* | 92.80 | 3.70526E-002 | 2.72029E-003 |
| | | 1021.30 | 1.21 | | |
| | | 1147.00 | 2.60 | | |
| | | 1362.66 | 1.35 | | |
| | | 1750.46 | 1.35 | | |
| ~99 | 0.955 | 140.51* | 88.70 | 8.03649E-002 | 3.10891E-003 |
| | | 181.06* | 6.20 | 8.01185E-002 | 7.02843E-003 |
| | | 366.43 | 1.37 | | |
| | | 739.58* | 12.80 | 8.33675E-002 | 5.58813E-003 |
| | | 778.00* | 4.50 | 8.67447E-002 | 2.30437E-003 |
| 0.994 | | 497.08* | 89.00 | 3.53492E-002 | 2.10182E-003 |
| | | 610.33* | 5.60 | 7.12951E-002 | 1.47775E-003 |
| | | 306.10 | 5.13 | | |
| | | 318.90* | 19.20 | 1.55839E-002 | 8.52958E-004 |
| | | ~1.84* | 9.80 | 4.46245E-002 | 2.48110E-003 |
| | | 17 | 1.73 | | |

Spectra processing and reporting the results



.\GENIE2K\CAMFILES\NAA\certified RM\5100332.cnf

Generated On : 5/27/2023 11:53:23 AM

Sample Title : 1944
 Sample Description : Dmitriev A.Yu.
 Sample Identification : s-1944-01-10
 Sample Type : LLI-1
 Sample Geometry : 2,5

Peak Locate Threshold : 3.00
 Peak Locate Range (in channels) : 150 - 8000
 Peak Area Range (in channels) : 150 - 8000
 Identification Energy Tolerance : 2.000 keV

Sample Size : 1.0176E-01 gram

Sample Taken On : 10/16/2021 8:08:17 AM
 Acquisition Started : 10/25/2021 8:12:16 PM

Live Time : 1800.0 seconds
 Real Time : 2023/06/02 1924.0 seconds

Lead Time : 6.44 %

Concentration Program

Mass fractions - 7.4 (ed. TMO).

calculation of SRMs activity Group standard Mass fractions Table of isotopes Absolute method Clear form Help

rk directory: not selected

rk directory: not selected

Select work directory Clear

calculation of SRMs activity

File of SRM flux monitor activity: not selected

File of SRM flux monitor activity: not selected

e(s) of SRM activity: not selected

Recalculate and save SRMs activity

Group standard

es of SRM activity: not selected

Create a summary table of SRMs activity

ata for the table of SRMs check

Calculated uncertainty Z-scores Reference value

e(s) of SRM activity: not selected

File of group standard: not selected

Calculate SRM(s) on the group standard and save the table of SRMs check

es of analyzed sample activity: not selected

File of group standard: not selected

File of SRM flux monitor activity: not selected

File of sample flux monitor activity: not selected

eselect flux monitors file Coefficient of neutrons flux change

File of SLI data SLI-1 and SLI-2 Systematic error, %:

Calculate and save mass fractions

es of elements mass fractions of analyzed samples: not selected

Create the intermediate table of elements mass fractions

Create the final table of elements mass fractions

ss fractions calculation by absolute method

Thermal and resonance neutrons fluxes calculation

5100332; 5100333; 5100342; 5100343; 5100344.

Nuclide ID confidence Wt mean activity Reference concentration

| Nuclide name | Nuclide ID confidence | Wt mean activity, uCi/gram | Calculated uncertainty of activity, % | Reference concentration, ugram/gram | Reference uncertainty of concentration, % |
|--------------|-----------------------|----------------------------|---------------------------------------|-------------------------------------|---|
| AG-110M | 0.795 | 1.41E+00 | 3.42 | 6.40E+00 | 26.56 |
| AS-76 | 0.677 | 1.73E+01 | 3.39 | 1.89E+01 | 14.81 |
| AS-76 | 0.634 | 1.98E+02 | 2.76 | 1.86E+02 | 1.61 |
| AS-76 | 0.652 | 7.61E-03 | 15.92 | 8.60E-03 | 2.23 |
| BA-131 | 0.992 | 2.14E+00 | 9.49 | 1.13E+03 | 2.93 |
| BA-131 | 0.996 | 7.42E-03 | 9.54 | 2.58E+02 | 2.54 |
| BA-131 | 0.991 | 1.43E+00 | 9.48 | 1.14E+03 | 30.00 |
| BR-82 | 0.774 | 3.65E-01 | 6.09 | 1.00E+00 | 30.00 |
| CA-47 | 0.975 | 2.24E-02 | 13.86 | 1.09E+04 | 1.25 |
| CA-47 | 0.960 | 1.23E-02 | 22.32 | 7.03E+02 | 0.26 |
| CD-115 | 0.913 | 3.27E-01 | 6.58 | 8.80E+00 | 15.91 |
| CE-141 | 0.998 | 3.29E+00 | 4.63 | 1.80E+02 | 30.00 |
| CE-141 | 0.999 | 1.06E-03 | 6.27 | 5.45E+00 | 1.71 |
| CE-141 | 0.994 | 2.63E-03 | 4.96 | 6.23E+03 | 30.00 |
| CO-58 | 0.998 | 4.83E-01 | 3.57 | 1.32E+02 | 7.56 |
| CO-58 | 1.000 | 2.97E-01 | 3.22 | 7.61E+01 | 7.24 |
| CO-58 | 1.000 | 1.37E+02 | 16.64 | 5.37E+00 | 6.55 |
| CO-58 | 1.000 | 2.18E-02 | 27.02 | 2.60E+00 | 6.23 |
| CO-60 | 0.999 | 1.33E+02 | 2.07 | 4.29E+01 | 8.16 |

Restore deleted line Invert selection Create group standard automatically Save the group standard

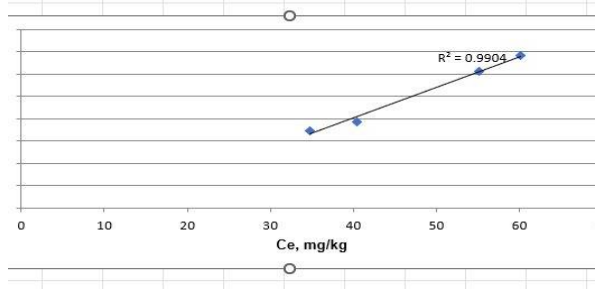
table_check_stand.xlsx - Excel (Product Activation Failed)

View Tell me what you want to do...

File Home Insert Draw Layout References Send Review View Help

| | E | F | G | H | I | J | K | L | M | N |
|--------------------|------------------|----------------|--------------------|--------------------|------------------|----------------|--------------------|--------------------|------------------|---|
| NA 24 | | | | CA 47 | | | | SC 46 | | |
| pass. conc., ug/gr | calc. uncert., % | stand. dev., % | calc. conc., ug/gr | pass. conc., ug/gr | calc. uncert., % | stand. dev., % | calc. conc., ug/gr | pass. conc., ug/gr | calc. uncert., % | |
| 19000 | 3.4 | 3 | | | | | 10.09326 | 10.2 | 3.2 | |
| 35906 | 3.3 | 0 | 5331.689 | 7025 | 26.2 | -24.1 | 4.105684 | 5.1 | 3.2 | |
| | | | 10903.65 | 10870 | 19.4 | 0.3 | 1.270364 | 1.24 | 3.3 | |

| | | Sc | Cr | Fe |
|------|-----|------------|------------|------------|
| /kg | % | MDC, mg/kg | MDC, mg/kg | MDC, mg/kg |
| | | 9.55 | 0.0348 | 1.91 |
| 5300 | 3.4 | 627 | 0.0589 | 2.54 |
| 8000 | 3.4 | 614 | 0.0377 | 1.87 |
| 5100 | 3.5 | 982 | 0.0349 | 2.43 |
| 2800 | 3.5 | 1020 | 0.0311 | 1.78 |



Most Common Programmes Used For Data Interpretation

❖ ArcGIS

❖ Statistica

❖ Origin-Lab

❖ CorelDraw

MEDIEVAL WALL PAINTING

SAMPLES FOR INVESTIGATION:

pigments and plasters of wall painting, mortars from Old Russian buildings

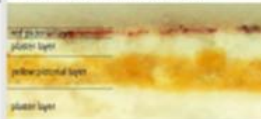
RECENT STUDIES:

comprehensive study of wall painting cycles of the Cathedrals from Veliky Novgorod, Pskov and Moscow monasteries



OBJECTIVE: investigation of pigment composition
METHODS: in situ XRF, FTIR, chemical microanalysis, stratigraphy of polished cross-sections
RESULTS: paint composition (pigments and binders) of antique pre-Mongolian paintings was studied; data will be used for the restoration of the Transfiguration Cathedral of the Mirozhsky Monastery (Pskov, 12th c.)

OBJECTIVE: study of wall painting technique
METHODS: stratigraphy of polished cross-sections
RESULTS: on the basis of the number and order of the painting layers, it was concluded that mixed (cross-section) techniques were used in the unique pre-Mongolian painting of St. George Cathedral of the Yuzev Monastery (Oldly Novgorod, 12th c.)



OBJECTIVE: digital reconstruction of presamable original coloration of mural fragment
METHODS: XRF, digital photo processing
RESULTS: based on the pigment composition and change in coloration, a presamable original view of the mural fragment of the Smolensk Cathedral of the Novodevichy Convent (Moscow, 16th c.) was created

OBJECTIVE: comparative analysis of mortars
METHODS: IFA, IBS, chemical microanalysis
RESULTS: according to the component content and ratio of some elements, an assumption was made about the later time of creation of some fragments of medieval buildings in Ukraine, Veliky Novgorod, Pskov-Publy



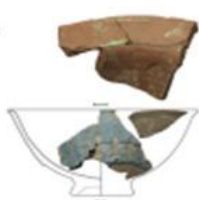
ARCHAEOLOGICAL SAMPLES

SAMPLES FOR INVESTIGATION:

ceramic, glass, and metal artifacts

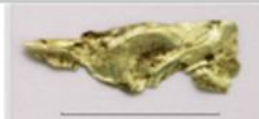
RECENT STUDIES:

analysis of red clay and kashin ceramics, fragments of glass bracelets and gold jewelry



OBJECTIVE: identification of raw material provenance for archaeological ceramic artifacts
METHODS: IFA, IBS
RESULTS: based on the element contents, a conclusion was made about the origin of raw materials (Azerbaijan Peninsula) for the manufacture of antique terracotta found during the construction of the Clinian Bridge

OBJECTIVE: formation of reference groups for determination of red clay ceramic manufacture locations
METHODS: IFA, statistical analysis
RESULTS: criteria were found that allow to classify unknown ceramic samples according to their provenance



OBJECTIVE: determination of the origin of metal artifacts: natural or artificial
METHODS: IFA, IFAA, XRF
RESULTS: on the basis of mass fractions of certain trace elements, a conclusion was made about the natural origin of the alloy under investigation - electrum

OBJECTIVE: identification of manufacturing centers for fragments of glass bracelets
METHODS: IFA
RESULTS: comparison of the obtained data with the recipes of the major glass manufacturing makes it possible to conclude about the Old Russian origin of artifacts



HUMAN REMAINS

SAMPLES FOR INVESTIGATION:

bones, hair, teeth, brain fragments, organics from skulls

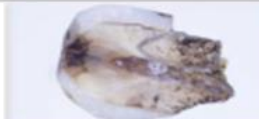
RECENT STUDIES:

study of the remains of medieval Russian nobility; bones of ancient blacksmiths



OBJECTIVE: determination of the mercury and arsenic content - basic of medieval poisons
METHODS: IFA
RESULTS: high mercury content was found in the rib bone of Ivan Ivanovich - the son of Ivan IV the Terrible which confirms the treatment with mercury ointments

OBJECTIVE: distribution of mercury and arsenic along the length of the hair
METHODS: IFA
RESULTS: based on the hair growth rate (1 cm per month), relations between the element content and time to death of the first Russian Tsarina Anastasia Romanovna were plotted



OBJECTIVE: creation of a database of the elemental composition of the remains of the medieval nobility
METHODS: IFA
RESULTS: elemental analysis of various remains of ten medieval high-ranking nobles was carried out, work has been initiated on the creation of a database of the remains of the medieval nobility

OBJECTIVE: determination of the content of copper and main trace constituents of copper ores
METHODS: IFA, IBS
RESULTS: copper was found in the remains, which means that the person was a blacksmith; the detection of arsenic suggests a specific source of raw material



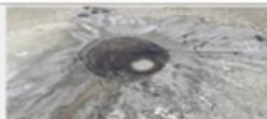
GEOLOGICAL, ALIEN, AND ECOLOGICAL SAMPLES

SAMPLES FOR INVESTIGATION:

solid emissions from mud volcanoes, soils, sediments, rocks, vegetation, air filters, meteorites

RECENT STUDIES:

environmental research of samples from Egypt and the Czech Republic; analysis of characteristics of Azerbaijan mud volcanoes, meteorite Chelyabinsk



OBJECTIVE: determination of elemental composition, natural gamma activities and microclimate
METHODS: IFA, IBS, gamma-ray spectroscopy, microclimate analysis
RESULTS: correlation investigation of solid emission elemental compositions was performed for mud volcanoes from Shemakhi-Gobustan region of Azerbaijan

OBJECTIVE: determination of trace element composition in rocks to detect elements with increased mass fractions
METHODS: IFA, statistical analysis
RESULTS: the fact of enrichment with uranium and thorium of the rocks from Sukani and Harashid gold mines (Red Sea governorate, Egypt) was revealed



OBJECTIVE: determination of environmental hazardous trace element content in samples of marine sediments
METHODS: IFA, statistical analysis
RESULTS: the sources of pollution and the degree of contamination of the marine sediments of the Egyptian Mediterranean coast were identified

OBJECTIVE: determination of air metal pollution using air filters
METHODS: IFA, statistical analysis
RESULTS: the source of air pollution in the Czech Republic (Moravian-Silesian Region) was identified to be the transboundary transfer of combustion products of coal used for local heating in Poland



Joint projects – Egypt

1st Phase

- Assessment of the environmental situation in the basin of the River Nile using nuclear and related analytical techniques (2011-2014).

2nd Phase

- Environmental studies in Egypt using neutron activation analysis and other analytical techniques (2015-2018).

3rd Phase

- Assessment of the environmental situation in the marine ecosystems in Egypt using neutron activation analysis and other analytical techniques (2018-2019).

4th, 5th
Phase

- Assessment of ecological situation of different ecosystems by using neutron activation and related analytical techniques: an approach of baseline concentrations, pollution indicators, and health hazard (2020-till now)
- Application of Nuclear and related analytical techniques to investigate the Egyptian archaeological materials.



The international student practice at JINR offered us several outcomes

- **Enhanced Knowledge and Skills**
- **Research Collaboration**
- **Cultural Exchange**
- **Professional Development**
- **Future Opportunities**

Acknowledgment

- We are deeply grateful to ASRT and JINR for the invaluable opportunity to participate in the international student practice. Our time at JINR has been enriching, with special thanks to the University Centre team and the Group of Neutron Activation Analysis – Frank Laboratory of Neutron Physics for their warm welcome, collaboration, and expertise sharing. We appreciate the stimulating scientific discussions and access to cutting-edge research equipment.
- Dr. Andrey Dmitriev and Dr. Wael Badawy, our supervisors at JINR, for their guidance, mentorship, and expertise. Their knowledge and encouragement have been instrumental in shaping our understanding of neutron activation analysis.
- Thank you all for making our international student practice at JINR a remarkable and unforgettable journey.

References

- P. Bode, J. J. M. de Goeij, Activation Analysis, Encyclopedia of Environmental Analysis and Remediation
- P. Bode, Instrumental and organizational aspects of a neutron activation analysis laboratory.

Born to be explorers!



Thank you for
your attention!

