Elemental analysis of phosphoric ore with tagged neutron method

Yu.N.Rogov

Diamant Ltd, Dubna

ISINN-27, Dubna,
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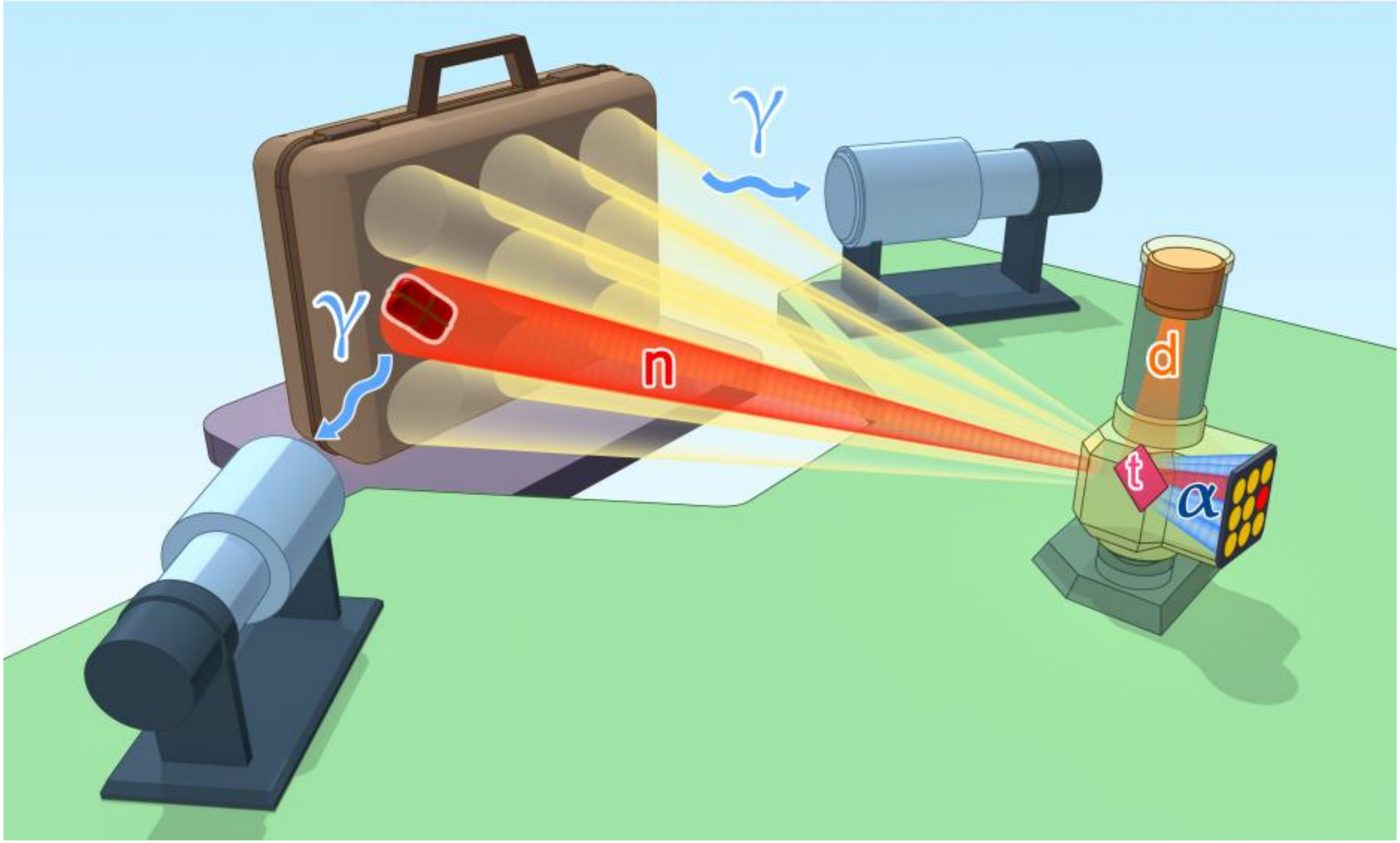
ДИАМАНТ

Report review

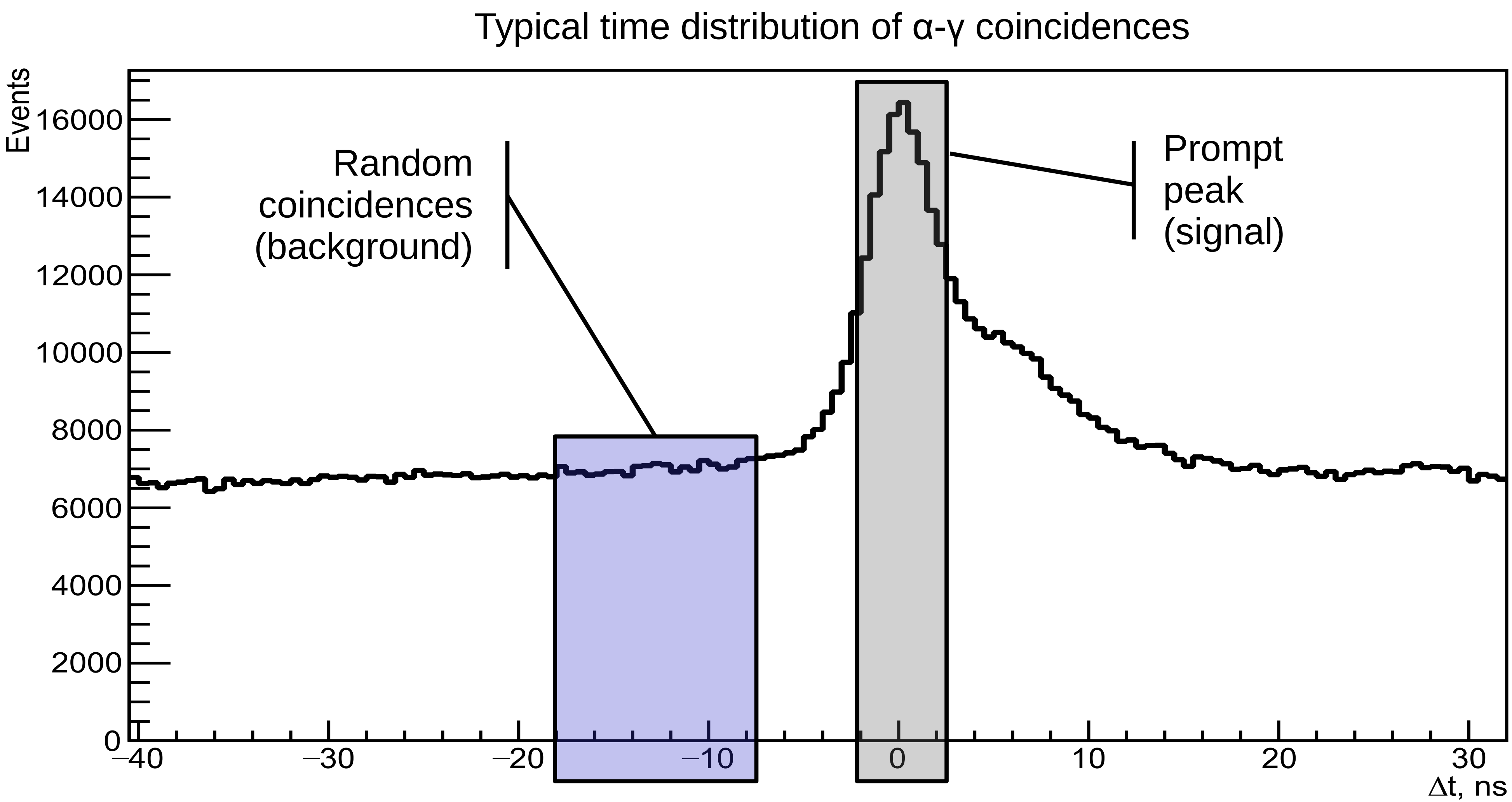
- TNM (tagged neutron method) and its advantages
- Results of operation on apatite ore of Khibini deposit at Apatit JSC, Kirovsk
- Results of tests on ore samples of Kovdor ore refinery
- Conclusions

Object under investigation is irradiated with fast 14 MeV neutrons

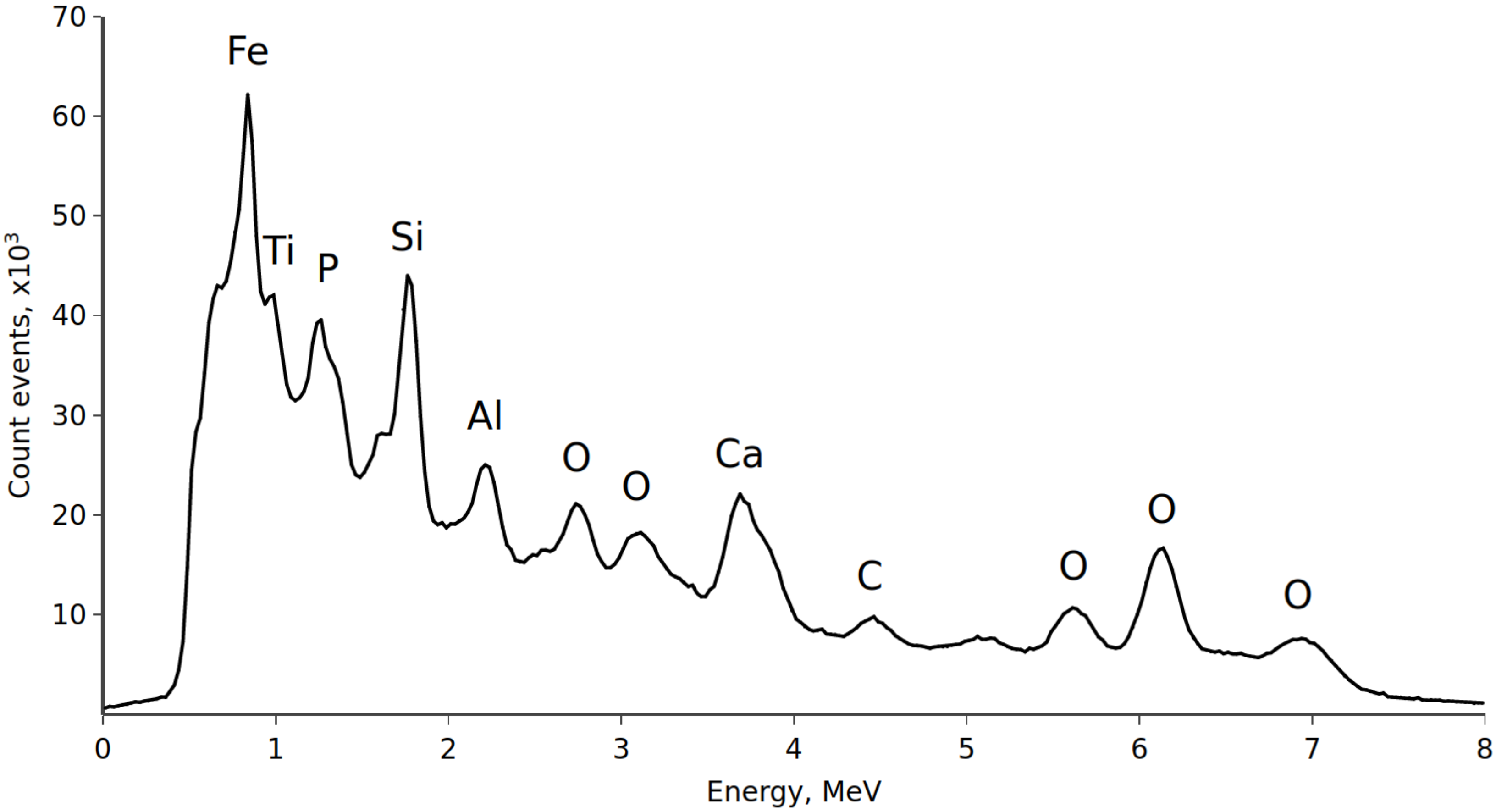


Neutron direction is tagged by α-particle
γ-quanta from object under investigation are detected

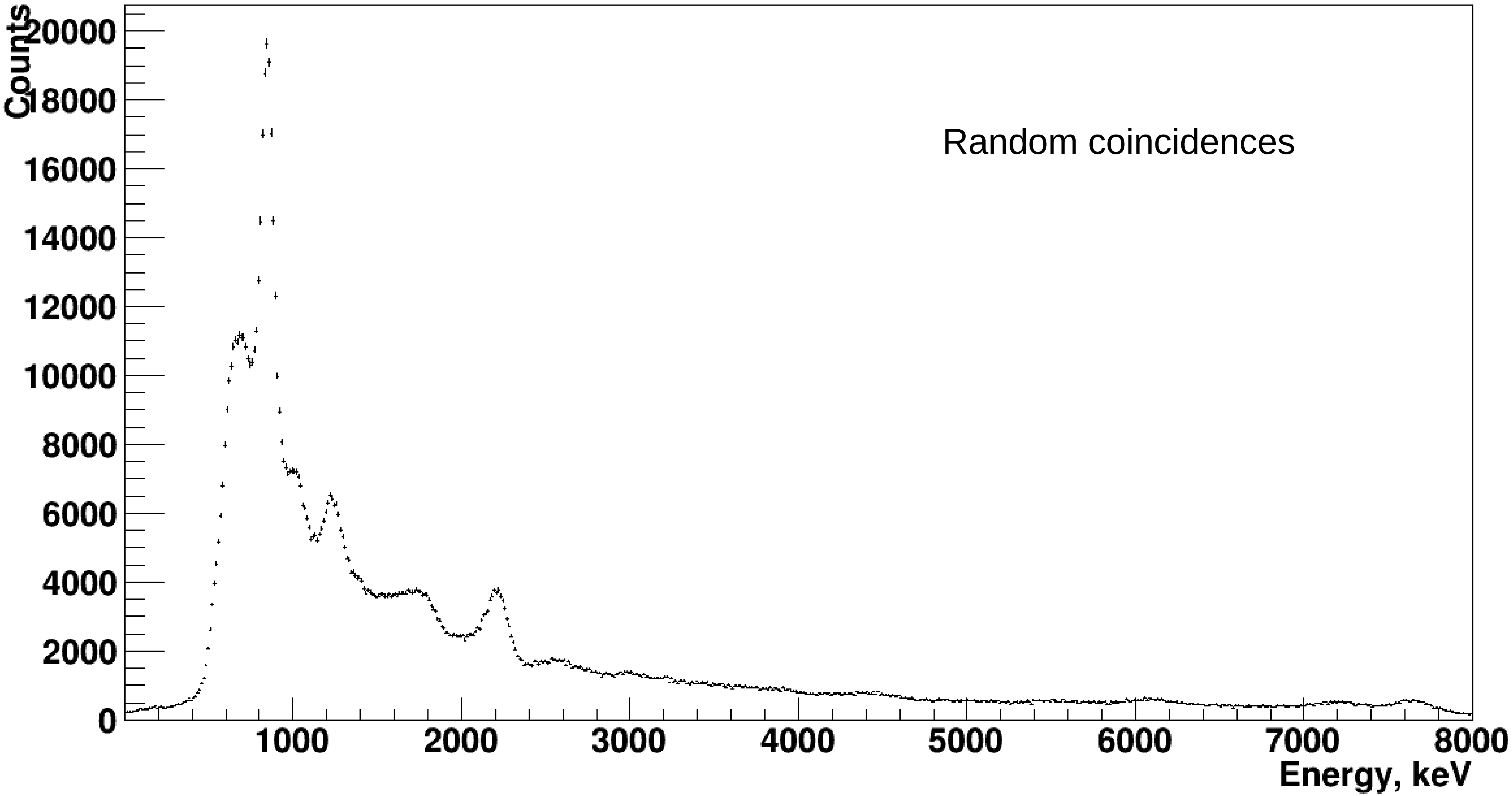
Advantage of TNM – time spectrum



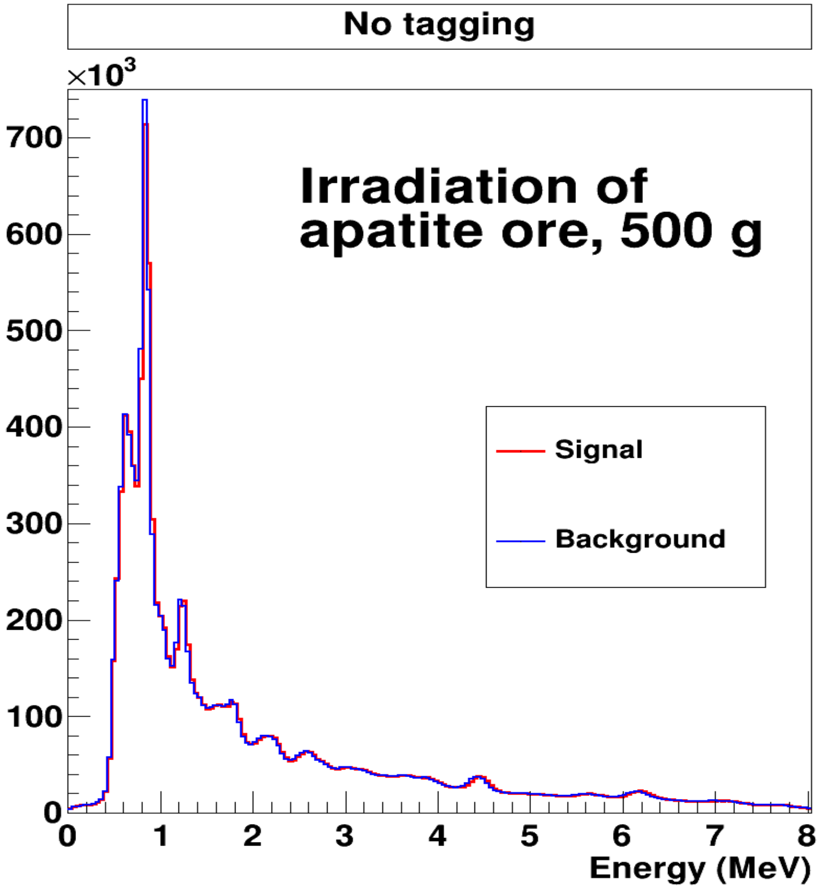
Energy spectrum – signal region

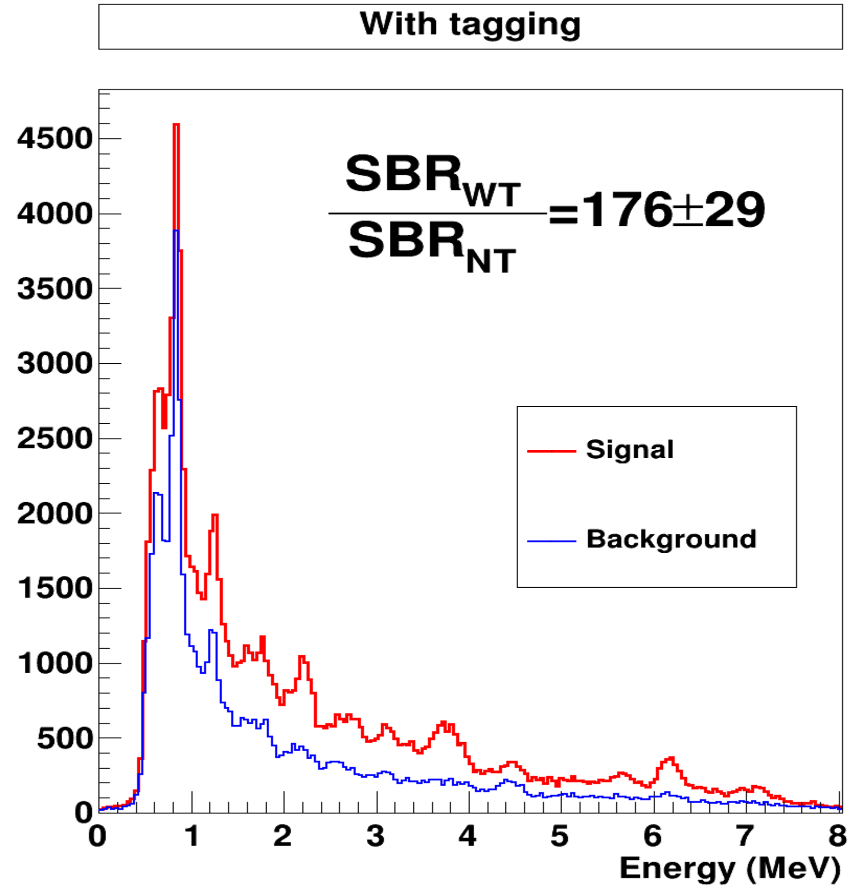


Energy spectrum – random coincidence region



	RFA	NAA	TNM
Dramatic suppression of environment background signal	no	no	yes
What is the benefit?	Signal-to-background ratio is 200 times better		





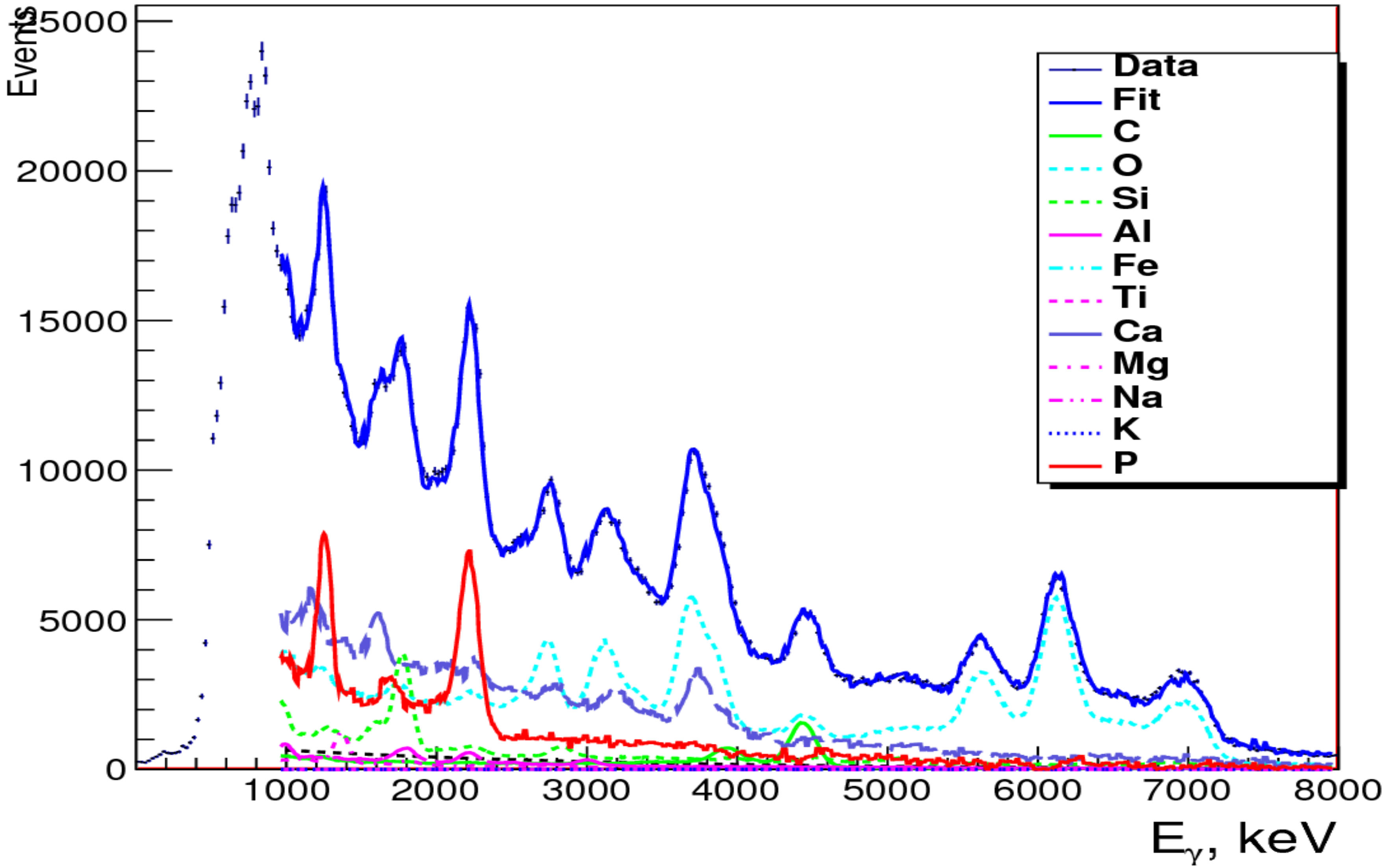
Evaluation of concentrations of 25 elements

	RFA	NAA	TNM
Evaluation of concentration of light elements, C,N,O	no	No	yes
What is the benefit?	• Direct evaluation of carbon concentration • Explosive substances detection		

Detectable elements:

Na, Mg, C,N,O, F, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, Ni, Cu, Zn, Zr, Pb, Sn, Bi, Sb

Spectrum of the sample



Fit function

Fit function for all elements $j = C, O, Si, Mg, Ca, Fe, Al$ is as follows (parameters of the fit are in bold):

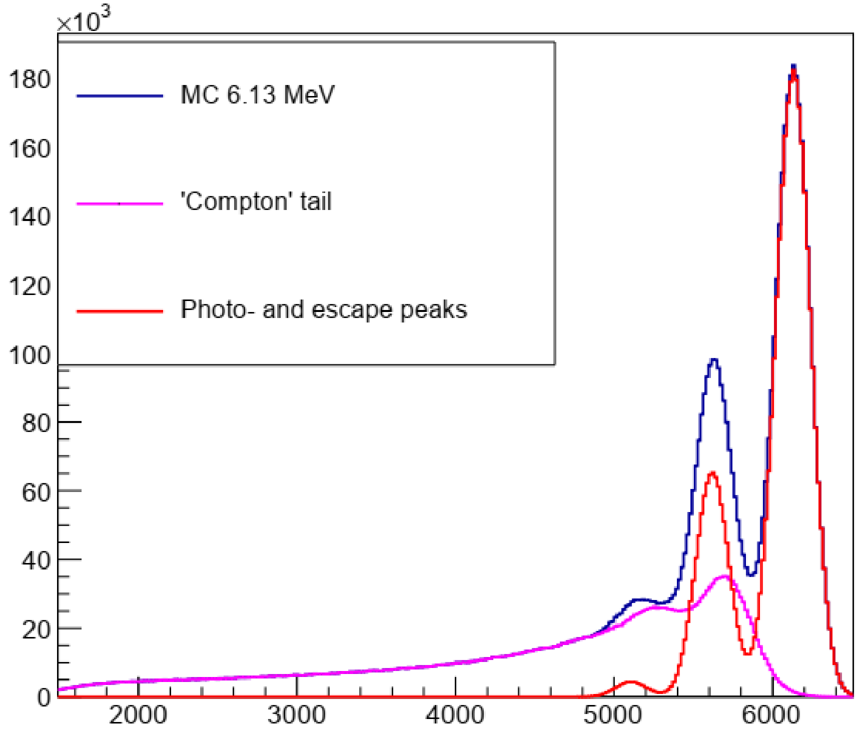
$$S(E) = \sum_{j=C,O,Si...} (\mathbf{N_j} \sum_{i=1}^{i=k_j} \sigma_{ij} P_{ij}(E) \epsilon_{ph}(E_i) + Cont_j(E)) + Bg(E)$$

k_j - number of peaks for the element j, P_{ij} - fit function (response function) for gamma-line i with energy E_i, $\mathbf{N_j}$ - parameter which determines the amount of the element j in the spectrum , σ_{ij} - cross section of the gamma ray with the energy E_{ij}. $Cont_j(E)$ - amplitude of the " Continuum spectrum". Since the production cross section σ_{ij} and total absorption peak efficiency(included in the P_{ij}) are given, the normalization of the element is determined by a single parameter $\mathbf{N_j}$. $Bg(E)$ - function of the "background". $\epsilon_{ph}(E_i)$ - total absorption peak efficiency for the energy E_i (from MC).

Fit function

Fit function (response function) for gamma-line with energy E_i are as follows (parameters of the fit are in bold):

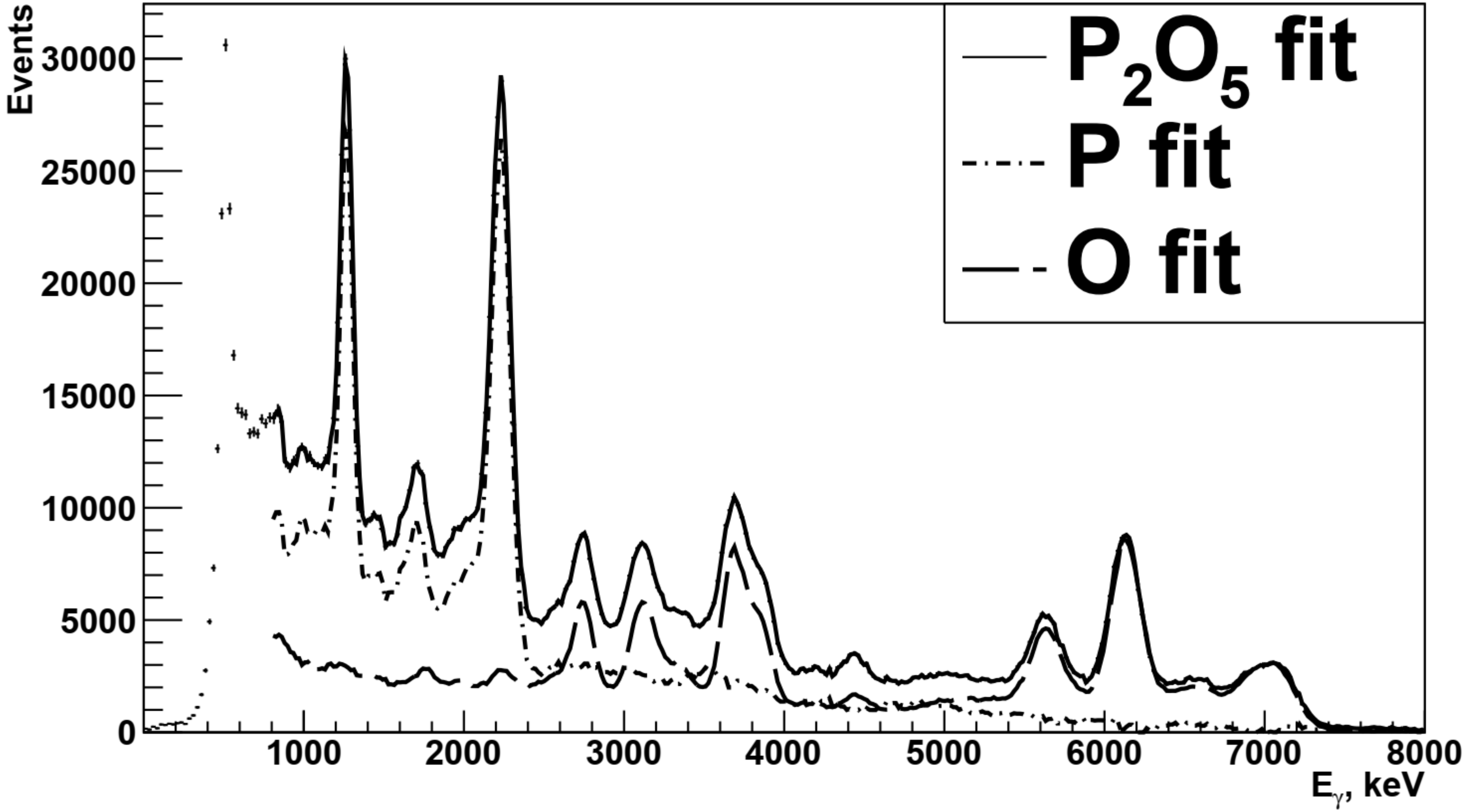
$$P_i(E) = \frac{1}{\sqrt{2\pi}\sigma_{\mathbf{i}}^{\mathbf{ph}}} G(E_i, \sigma_{\mathbf{i}}^{\mathbf{ph}}) + \frac{\mathbf{R_O} r^{es}(E_i)}{\sqrt{2\pi}\sigma_i^{es}} G(E_i - 511, \sigma_i^{es}) + \frac{\mathbf{R_O} r^{es}(E_i) r^{es2}(E_i)}{\sqrt{2\pi}\sigma_i^{es2}} G(E_i - 1022, \sigma_i^{es2}) + N_i^c \mathbf{R_{Compt}} C(E_i, E)$$



- $\sigma_{\mathbf{i}}^{\mathbf{ph}}$ - the energy resolution (depends as $\sqrt{E}$).
- $r^{es}(E)$ and $r^{es2}(E_i)$ - define the amplitudes of escape peaks, normalized relatively to the amplitude of the photopeak. Dependence from the energy is set by MC, but $\mathbf{R_O}$ is used to adjust them.
- $C(E_i, E)$ - Compton "tail" function.
- $\mathbf{R_{Compt}}$ - factor to adjust Compton "tail" amplitude.
- $G(\overline{x}, \sigma)$ -Gauss function.

Why phosphorus?

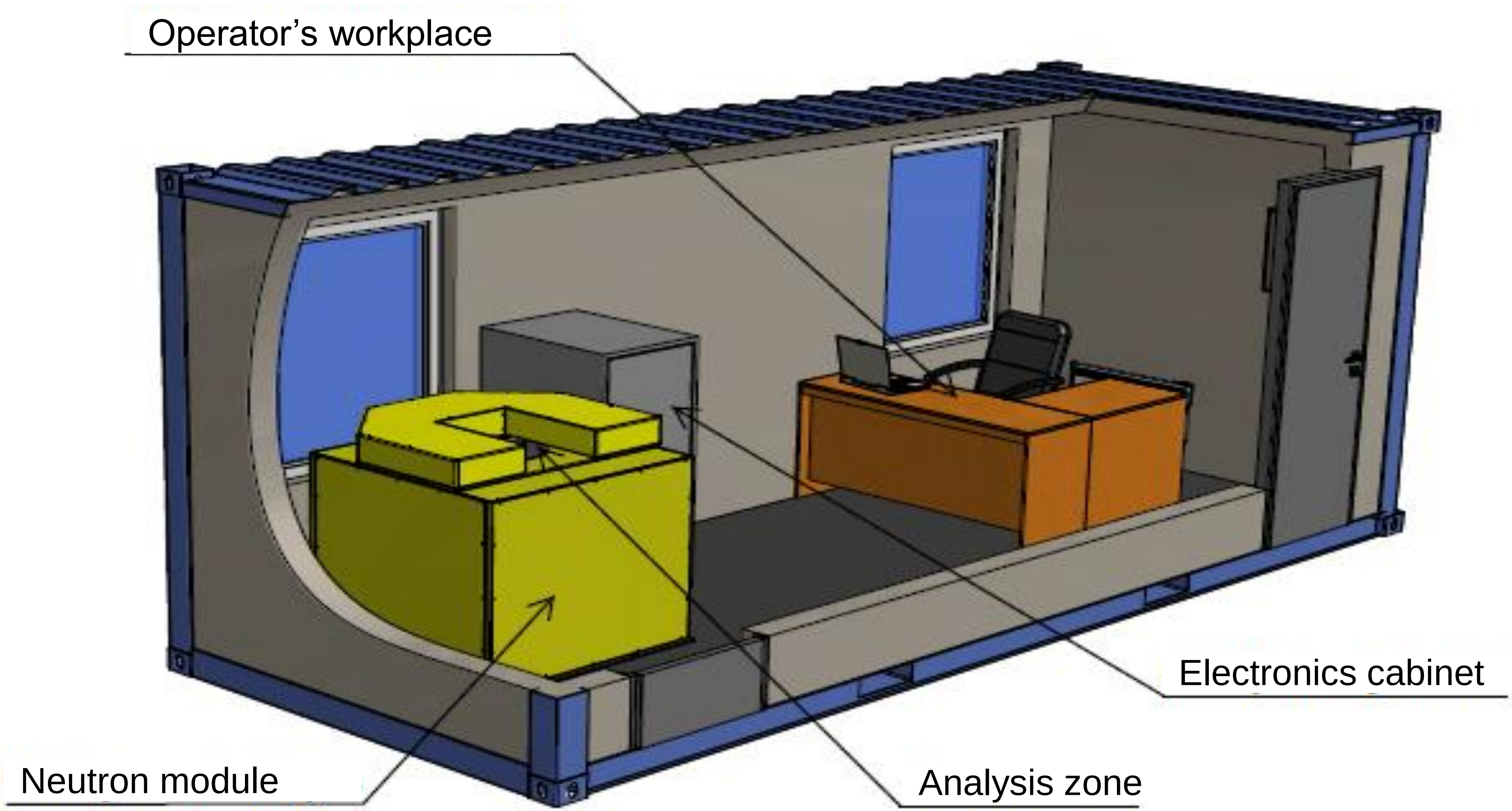
2234 keV	367 mb
1266 keV	180 mb



Test operation at Apatit JSC, Kirovsk

- Problems of geologists
- AGP-F had been operating ore of Khibini deposit for 6 months
- 1614 measurements was carried out
- Remote control

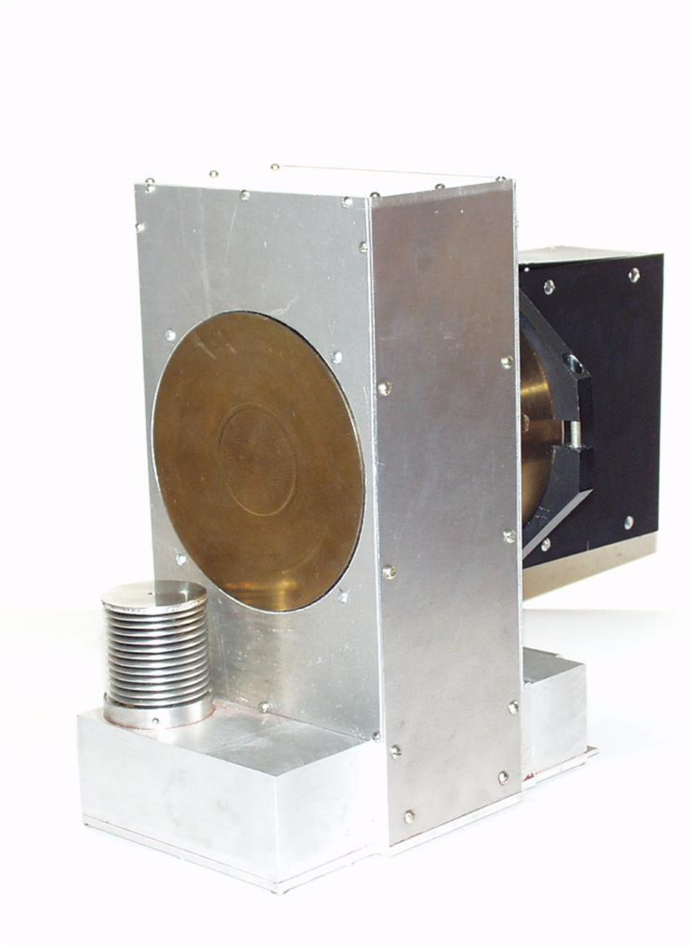
AGP-F system


Operator’s workplace
Electronics cabinet
Analysis zone
Neutron module

Neutron module of AGP

- Dimensions 1200×1176×1149 mm
- ING-27 neutron generator
- 9 tagged beams
- $I = 5 \times 10^{7}\ s^{-1}$
- 12 BGO gamma-detectors

Neutron source



- ING-27 neutron generator produced by Dukhov's VNIIA, Moscow
- $I= 8 \cdot 10^7\ s^{-1}$
- Mass – 8 kg
- Height – 300 mm
- Alpha-detector - 3×3 matrix (10×10mm)
- Actual lifetime:
  617 hours 47 minutes

Gamma-detector

- Scintillator - BGO
- Scintillator size 76×65 mm
- Relative energy resolution at line 4.44 MeV (^{12}C) 3.9±0.1%
- Detectable energy range 0.5-11 MeV
- Environment temperature range from +5 to +50°C
- Mass 3.4 kg
- Dimensions 89×265 mm
- R6233 Hamamatsu PMT

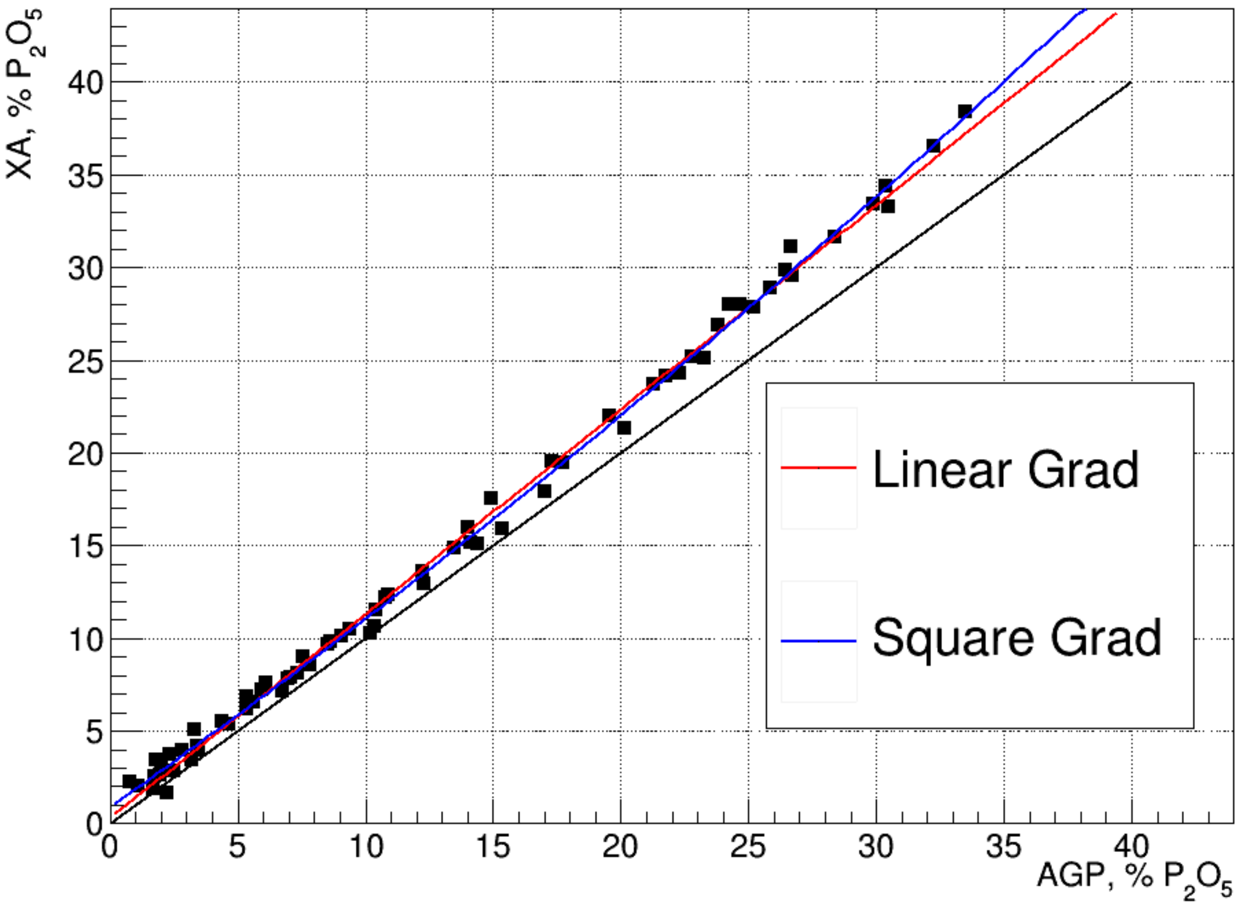
Software

- Place the sample in the bag to inspection tray.
- Turn on neutron generator.
- Start data taking.

Sampling

-100 mm grade samples of 5 kg mass from mining site were measured as is, without any preparation.

Graduation curve



P_2O_5 content range: 1-40%

σ_{rel} = 3,33%

69 samples

Red line – linear dependence.
Blue line – quadratic dependence.
Black line – equal values

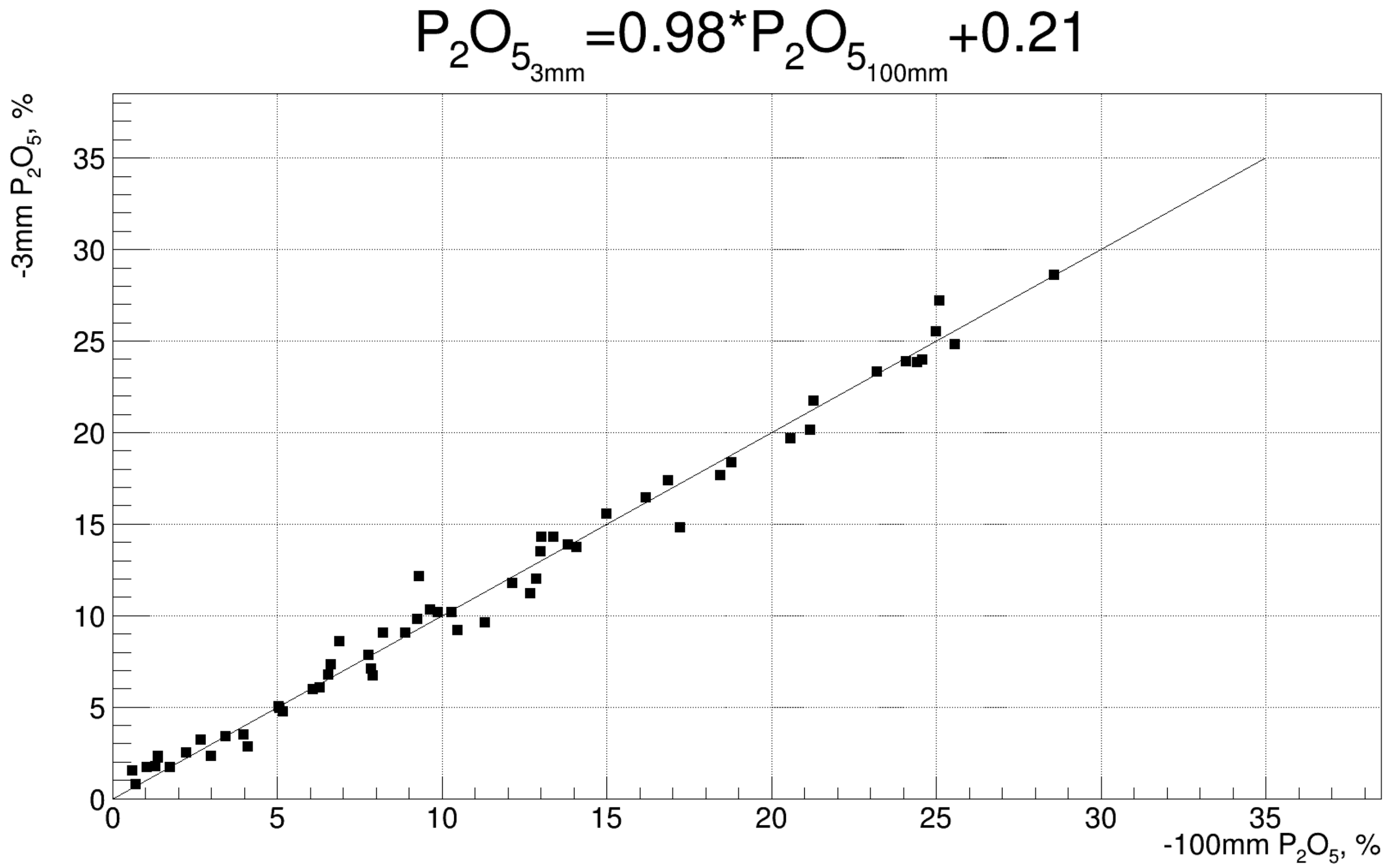
Graduation curve

P_2O_5 content, %	RMS deviation of P_2O_5 content, %	Relative RMS deviation of P_2O_5 content, %
1.00-6.99	0.57	13.50
7.00-14.99	0.43	4.05
15.00-24.99	0.66	3.46
25.00-40.00	0.62	1.95
1.00-40.00	0.55	3.33

$$Y_{ChA} = 0.0041 * (x_{TNM})^2 + 0.98 * x_{TNM} + 0.9$$

Minimal detectable concentration of P_2O_5 is 1.7%

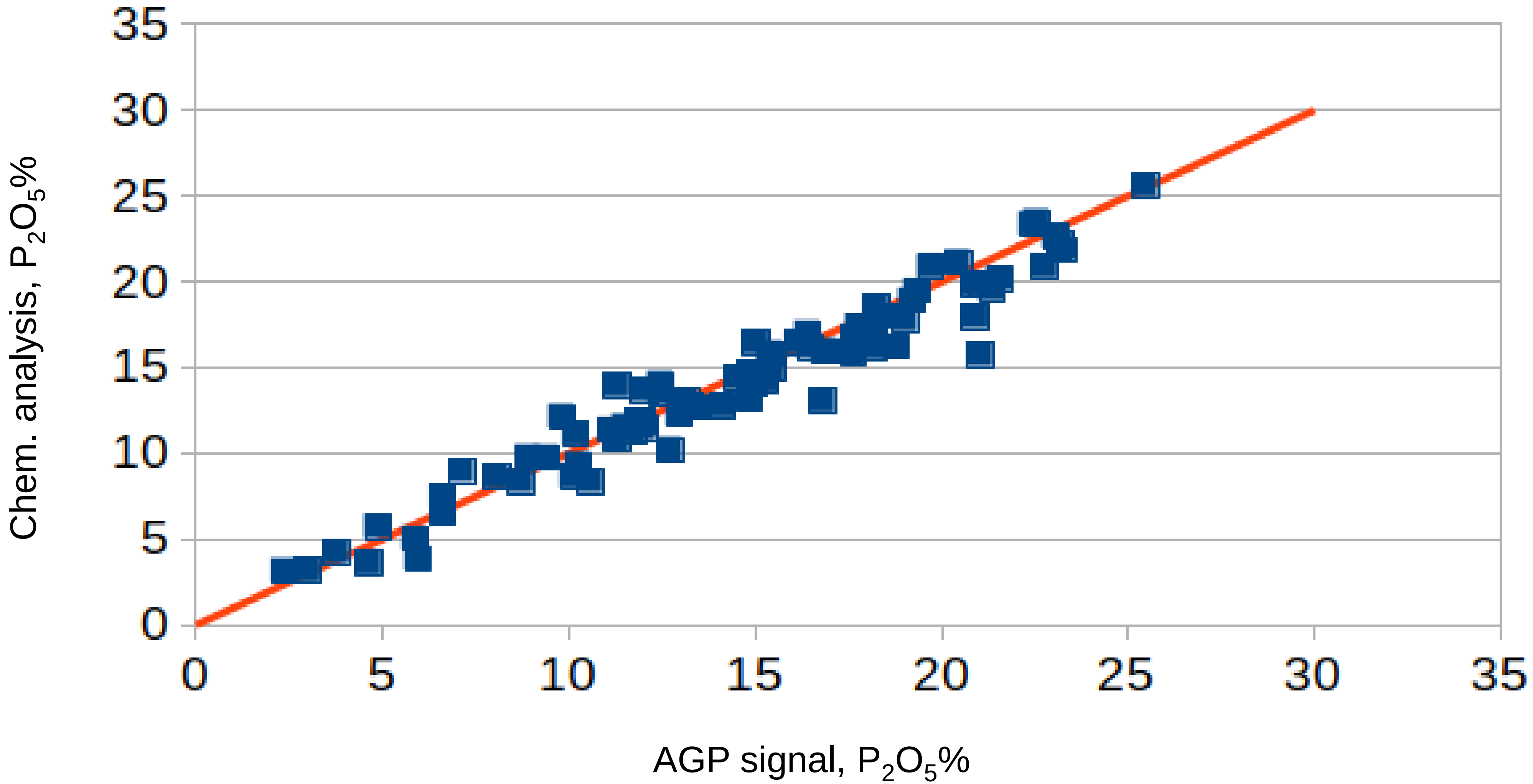
Transition curve



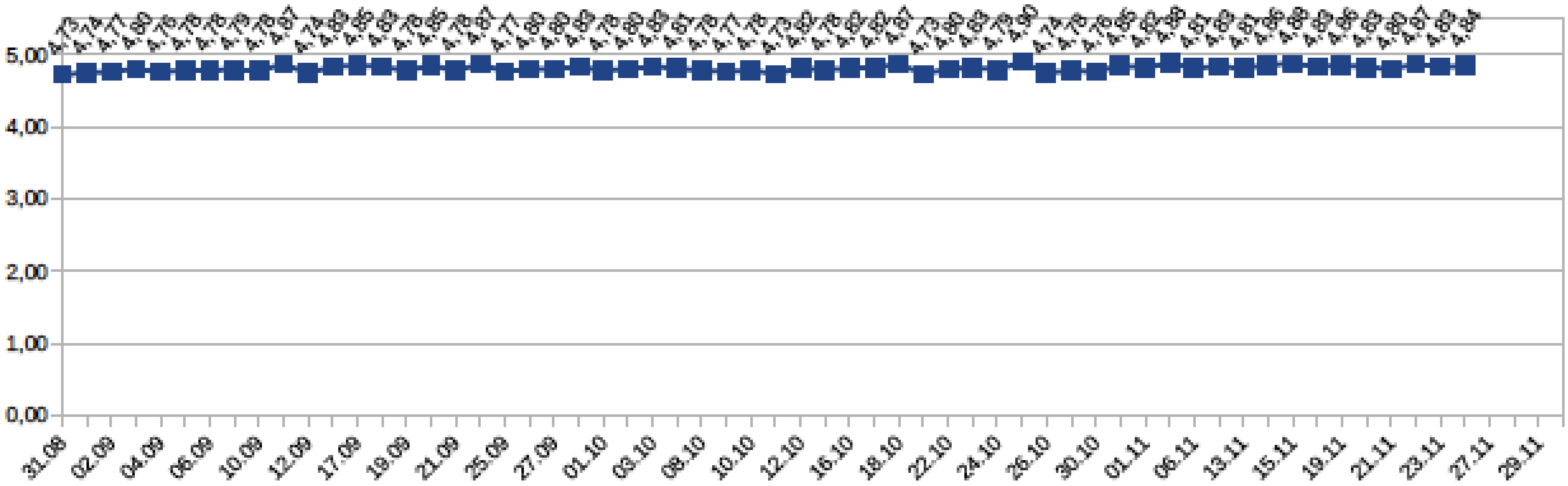
58 measurements

Comparison with chemical analysis for –100 mm samples

P_2O_5 content range: 2÷25%, RMS=1.17, RMS_{rel}=8,3%



Hardware stability



Energy resolution of gamma-detector system.

RMS_{rel}=1,1% over 6 months of operation

Graduation stability

Sample	P_2O_5 content, %	Number of measurements	$s_o(R)$, %
ДК 2-2	7.82	37	5.67
ДК 2-10	12.19	67	6.48
ДК 3-9	21.99	65	6.39
ДК 4-10	33.40	36	2.22

Measurement of control samples over 6 months. RMS of deviations from the first graduational measurement is less than 7%

Repeatability(r) and reproducibility (R)

Sample	AGP parameter, R %	Number of meas.	σ_r, $\%P_2O_5$	σ_r/R, %	σ_R, $\%P_2O_5$	σ_R/R, %
ДК 2-2	7.21	58	0.62	8.61	0.63	8.70
ДК 2-10	11.48	88	0.59	5.14	0.62	5.36
ДК 3-9	19.83	65	0.79	3.99	0.82	4.14
ДК 4-10	29.71	41	0.62	2.09	0.71	2.40

Radiation dose control results

ОБЪЕДИНЕННЫЙ ИНСТИТУТ ЯДЕРНЫХ ИССЛЕДОВАНИЙ

ОТДЕЛ РАДИАЦИОННОЙ БЕЗОПАСНОСТИ

141980 Московская область, г.Дубна
Телефон/факс: (49621) 65794
E-mail: buchnev@jinr.ru

Утверждаю
Начальник ОРБ
__________ В.Н.Бучнев
"25" 12 2018г.

Результаты индивидуального дозиметрического контроля персонала фирмы
ООО «Диамант»,
За 2018 год.

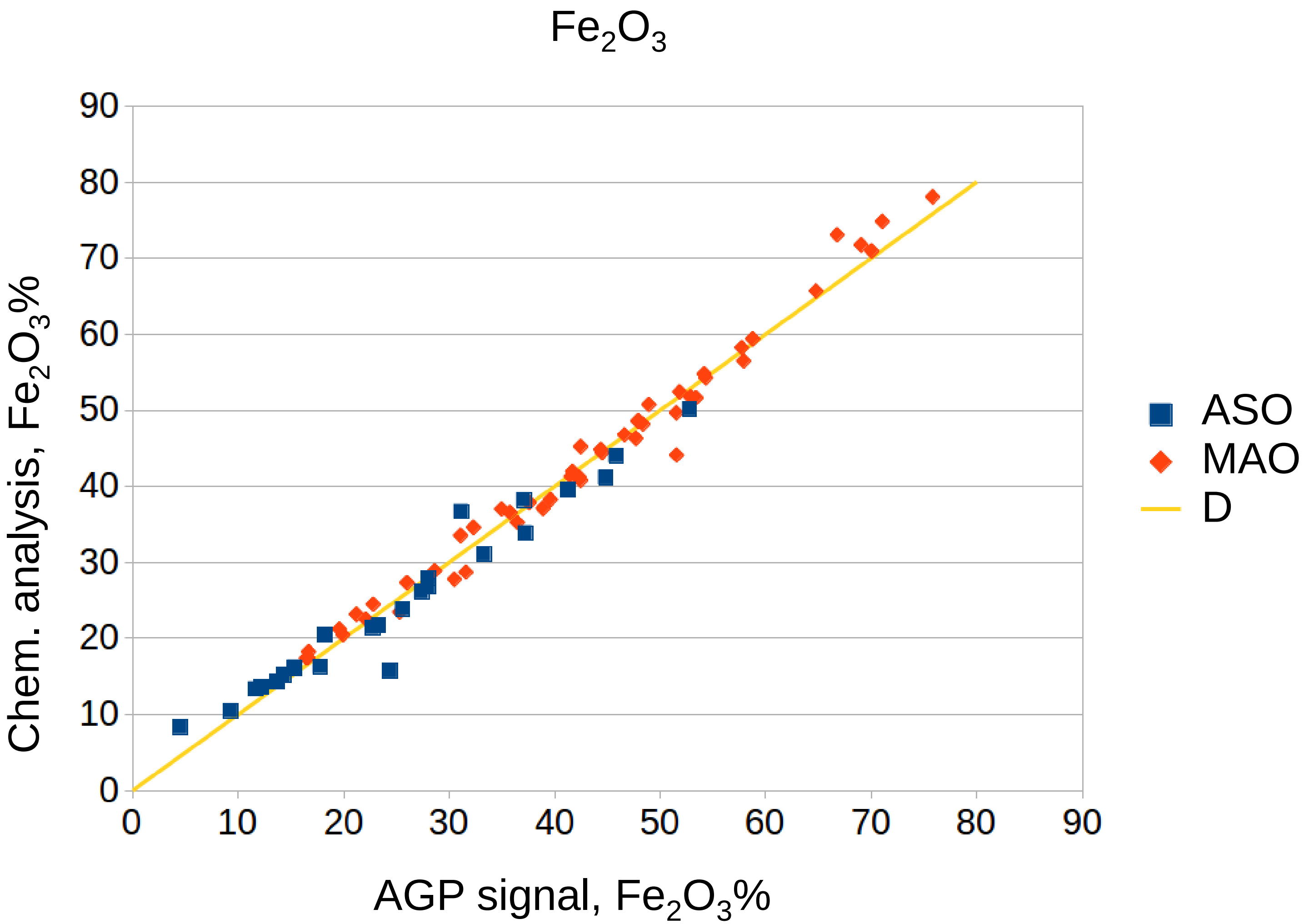
№	Фамилия И. О.	Номера дозиметров		Индивидуальная эффективная доза, мЗв		
				фотоны	нейтроны	*сумма*
1	Ассаулов А.Е.	2971	2972	0.279	0.705	*0.984*
2	Гончаров А.В.	2937	2938	0.037	0.000	*0.037*
3	Зубарев Е.В.	2933	2934	0.107	0.000	*0.107*
4	Кременец В.А.	2939	2940	0.063	0.076	*0.139*
5	Курилкин П.К.	2935	2936	0.033	0.000	*0.033*
6	Разинков Е.А.	2931	2932	0.234	0.498	*0.732*
7	Сапожников М.Г.	2941	2942	0.196	0.109	*0.305*

- Over 6 months of operation AGP-F operator dose was measured to be 2.101 mSv
- Maximum permissible annual dose for population is 1 mSv
- For Group-A personnel annual dose is 20 mSv

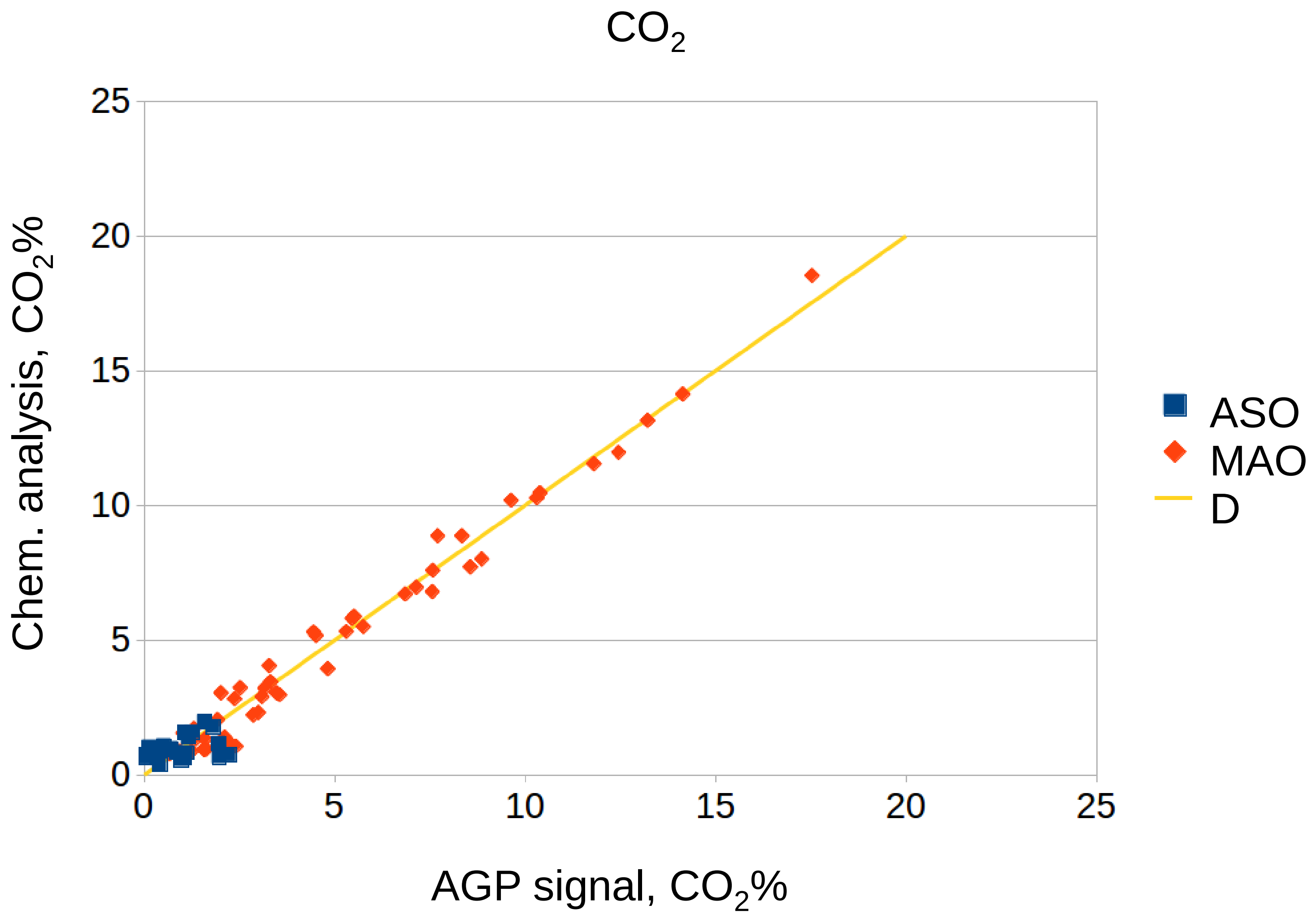
Trial run at Kovdor refinery

- 75 samples with known chemical composition
  - 25 samples of apatite-staffelite ore
  - 50 samples of magnetite-apatite ore
- Sample mass – 3-10 kg.
- Size grade –3 mm.

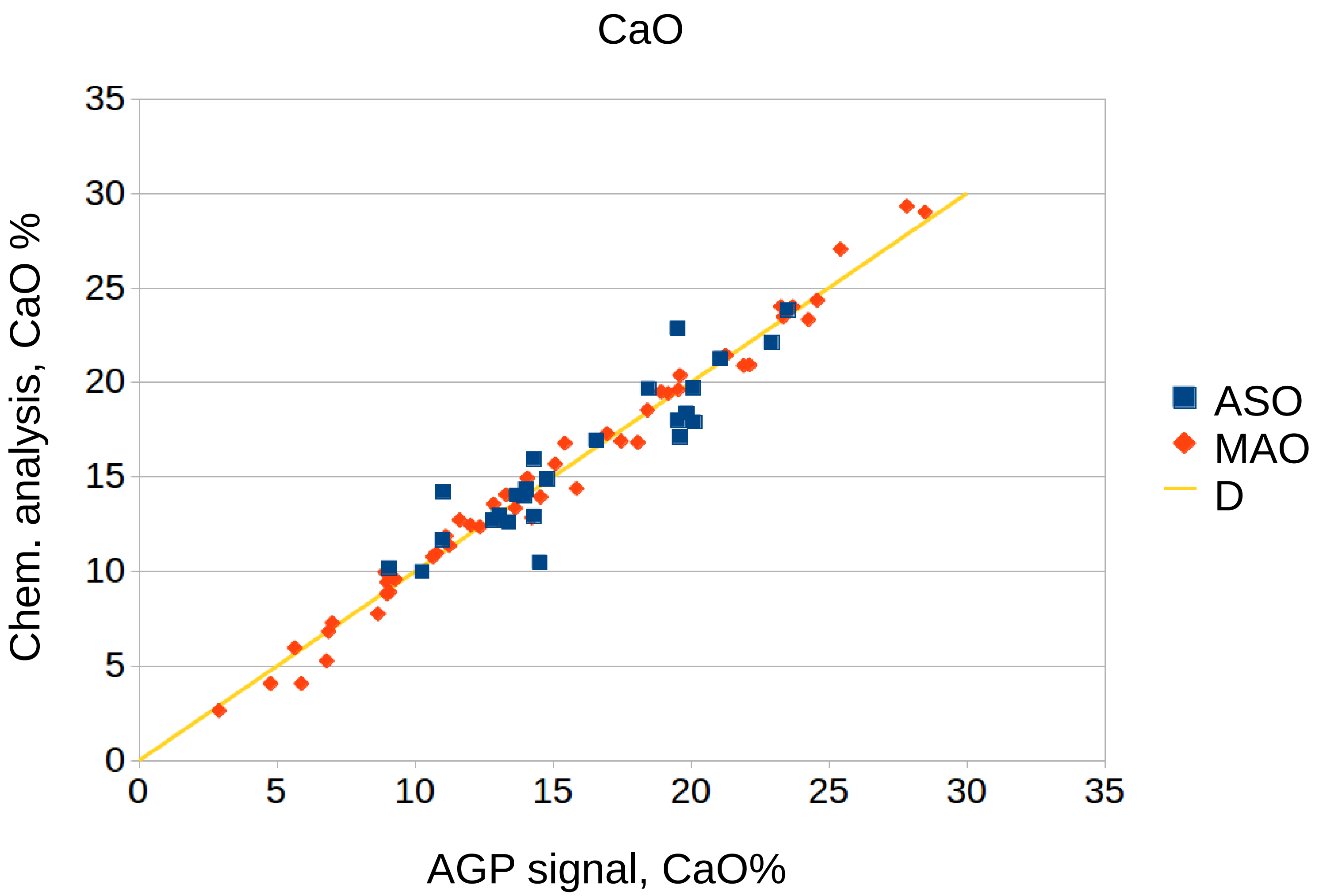
Comparison of AGP-F with chemical analysis



Comparison of AGP-F with chemical analysis



Comparison of AGP-F with chemical analysis

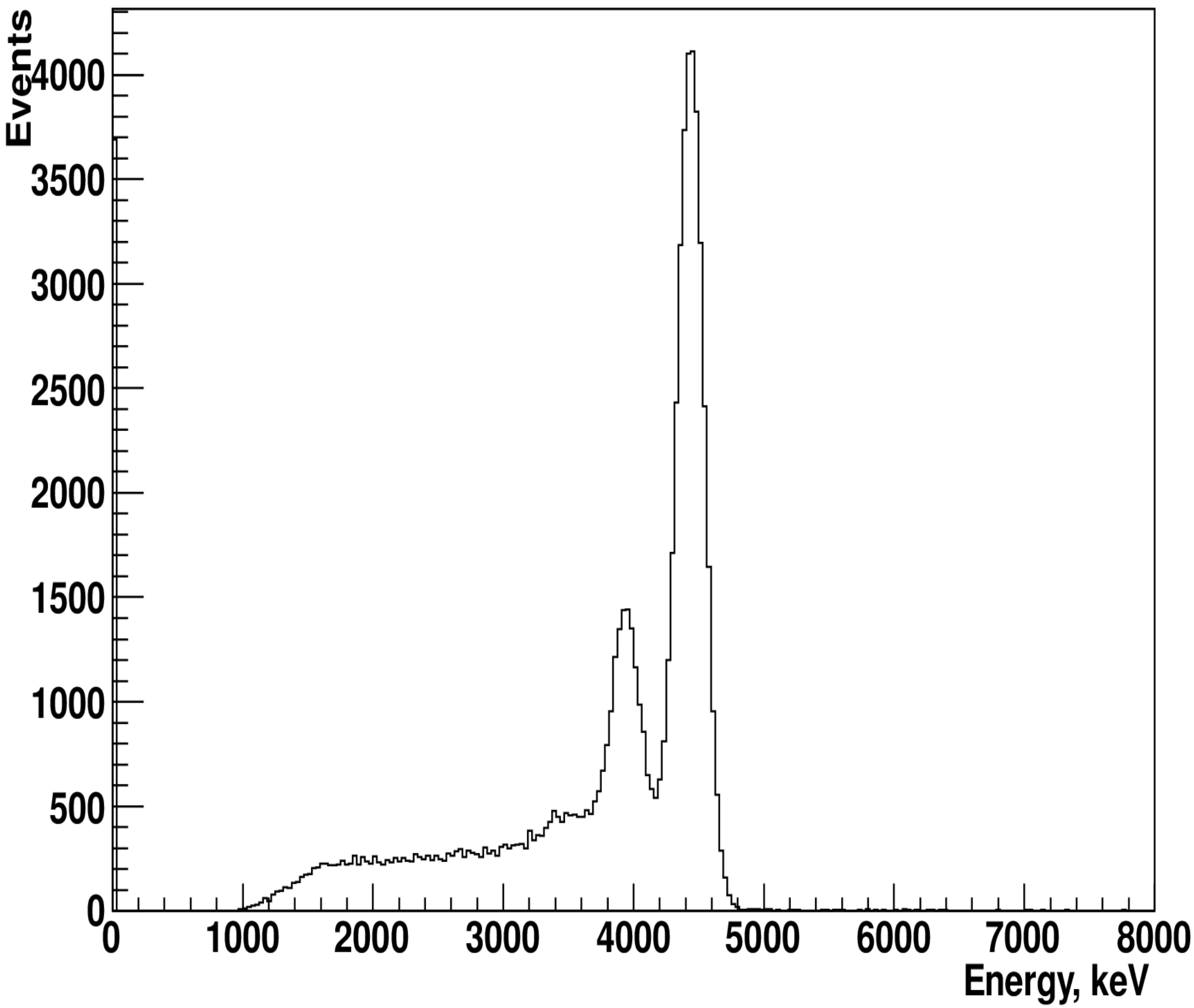


ДИАМАНТ

Conclusions:

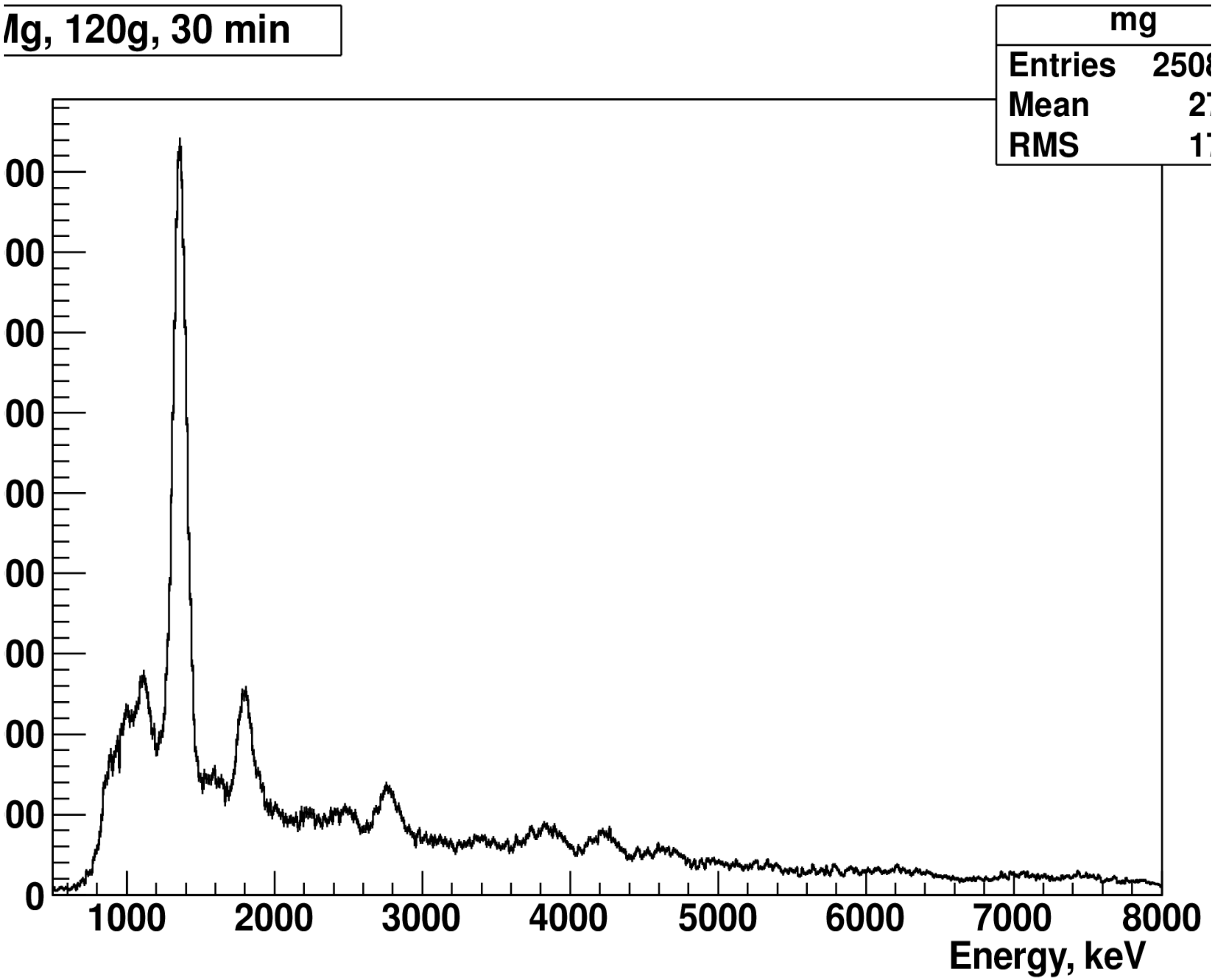
- AGP-F system has been field-operating for 6 months without any technical issues.
- The system allows obtaining results on content of 10 element oxides, namely CO_2 , P_2O_5, Al_2O_3, MgO, SiO_2, CaO, TiO_2, Fe_2O_3, Na_2O, K_2O
- The system allows obtaining results on content of samples of large size grade (-100 mm) without any sample preparation.
- Minimal detectable content – 1.7% of P_2O_5
- Precision of P_2O_5 content evaluation:
  σ_{rel} = 3,33% on graduational samples (-100 mm),
  σ_{rel} = 8,3% on routine samples (-3 mm)
- Graduation curve have been stable for 6 months.
- AGP-F system proved to be stable and reliable.

Carbon



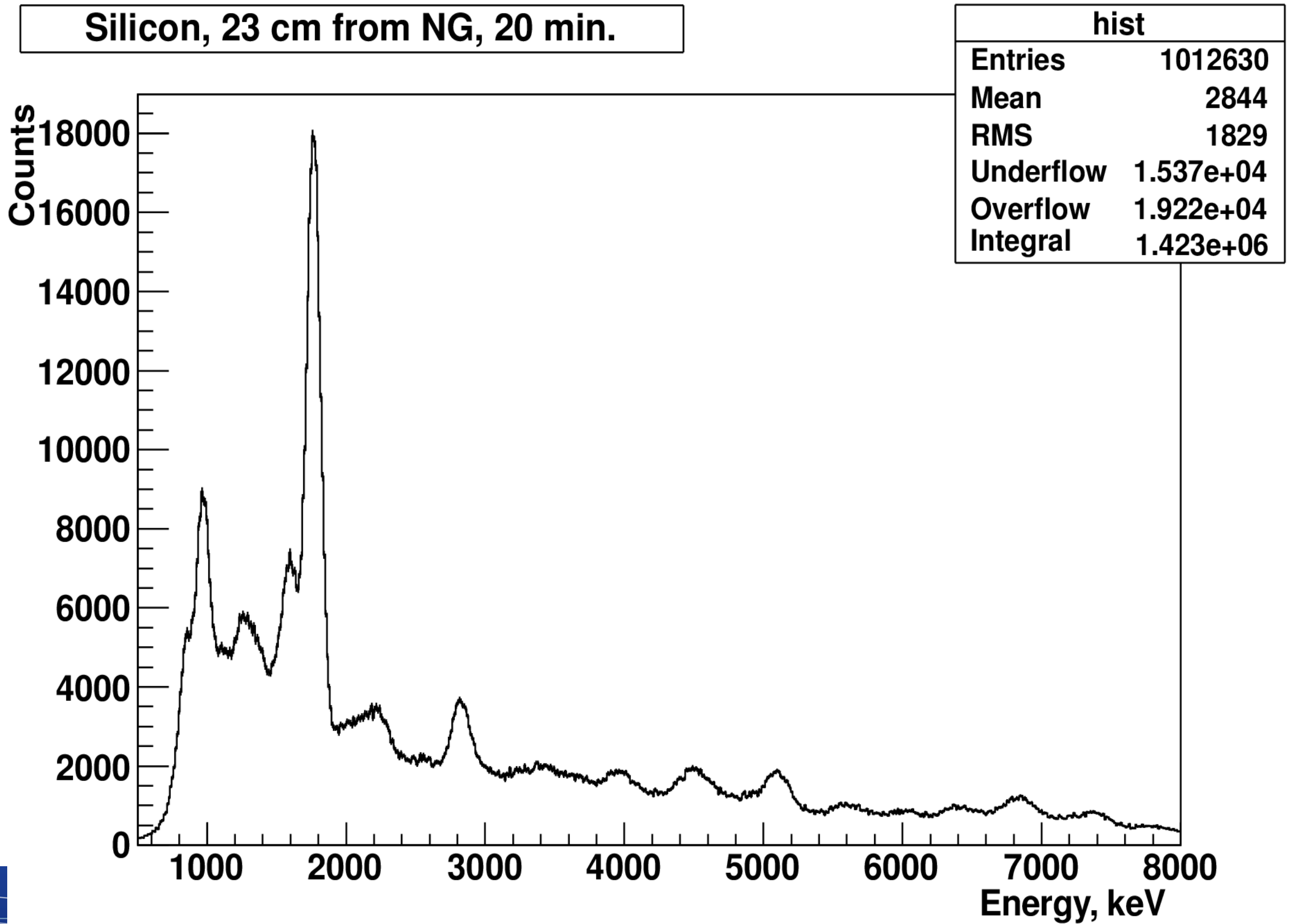
4438 keV	187 mb

Magnesium



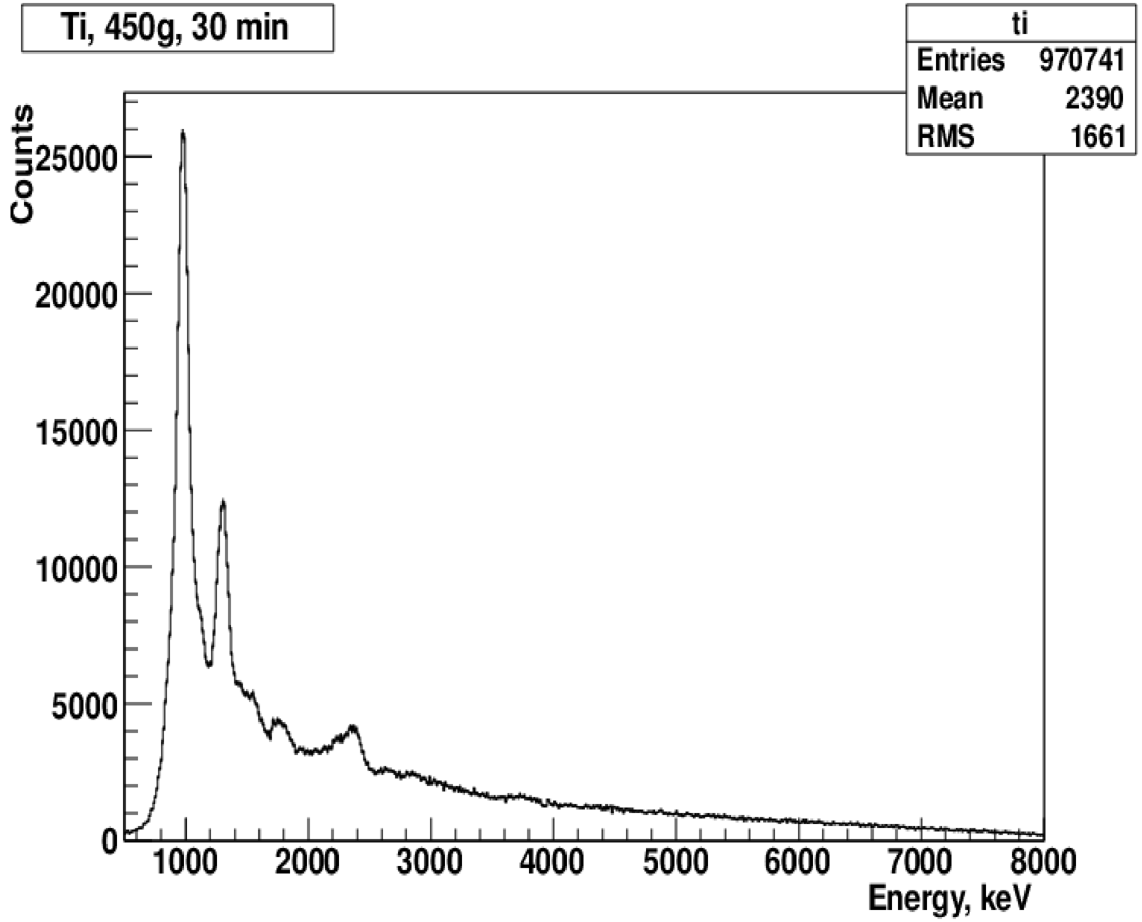
1369 keV	450 mb
1809 keV	81 mb
2770 keV	45 mb
3867 keV	30 mb
4239 keV	30 mb
4640 keV	22 mb

Silicium



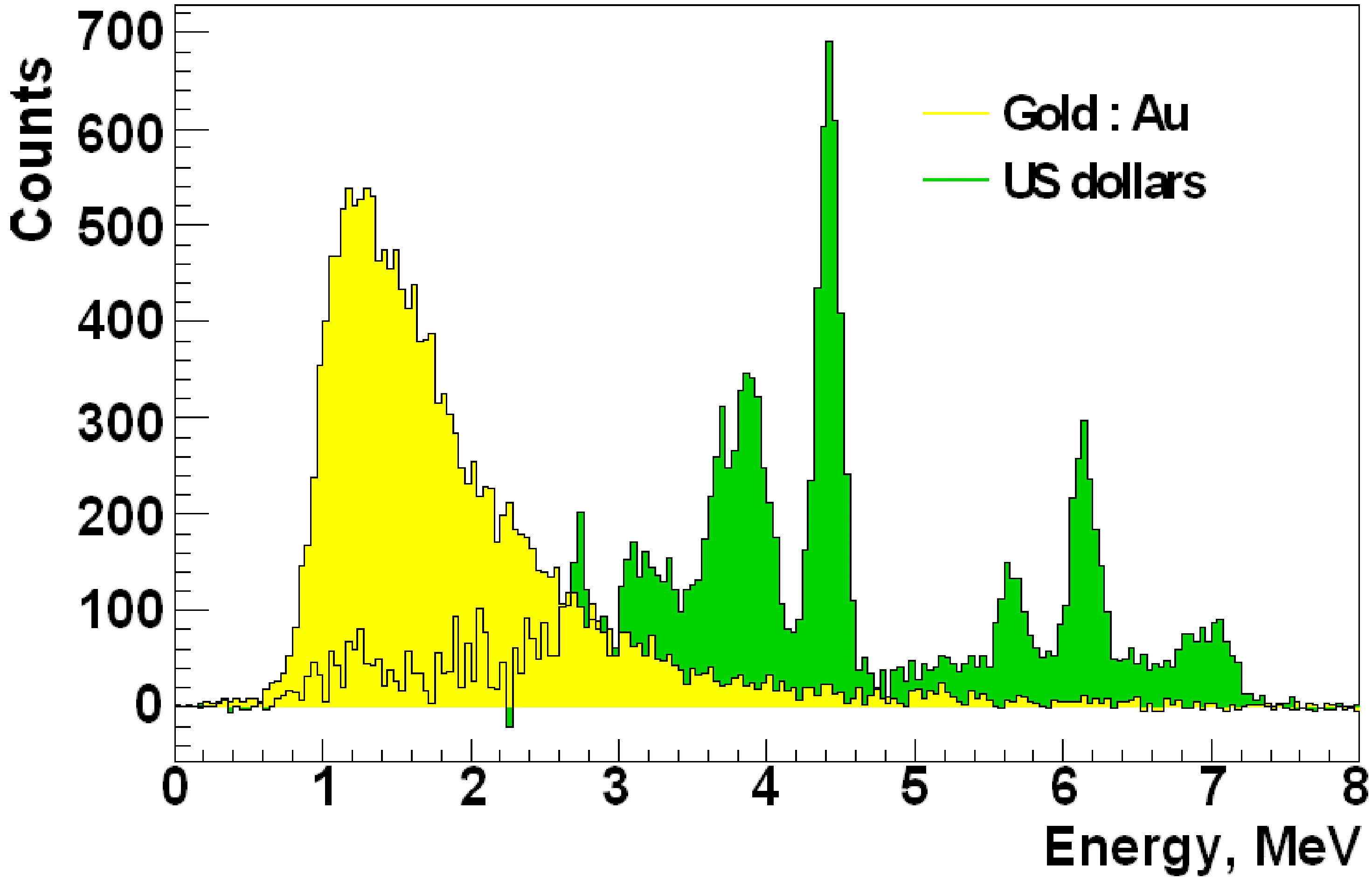
1589 keV	24 mb
1779 keV	403 mb
2839 keV	59 mb
5100 keV	37 mb
6879 keV	36 mb

Titanium



984 keV	666 mb
1312 keV	238 mb
1437 keV	49 mb
1555 keV	32 mb
1762 keV	23 mb
2240 keV	32 mb
2375 keV	54 mb

There are elements without narrow lines



Characterization of Spectrum of Sample

For one element the number of gamma rays entering the detector N_f is

$$N_f = \frac{N_{in} \sigma N_{Av} \rho l W}{A},$$

N_{in} - number of neutrons incident on the target, σ - total cross section of the reaction $(n, n'\gamma)$, N_{Av} - Avogadro's number, ρ - target density, l -target length, A - atomic weight of the element, W - the probability of gamma ray detection.

For the case of a target consisting of various elements j:

$$N_f = N_{in} l \rho W \sum_j n_j \sigma_j$$

σ_j - cross section for the element j, n_j - the number of nuclei of element j in the target.

By measuring the spectrum of gamma rays and knowing the gamma production cross sections σ_j for different elements one can restore the relative proportions of the elements in the target.

ДИАМАНТ