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S C I E N T I F I C

ИНСТРУМЕНТАЛНИ
ПРИЛОЖЕНИЯ ПРИ КОНТРОЛ НА
ХРАНИТЕ И ОПАЗВАНЕ НА
ОКОЛНАТА СРЕДА



АСМ2

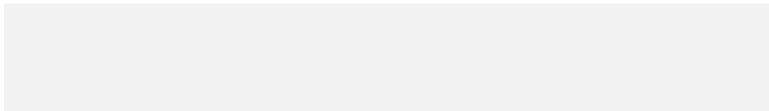
СЕМИНАР

Пловдив, 5-6 Юни 2012



Определяне на следи от тежки метали в храни и обекти на околната среда

FBA – *Food and Bio-product Analysis Work Group*
/Работна група по анализ на храни и био-продукти/





Faculty of Chemistry – working groups in BioSupport

title

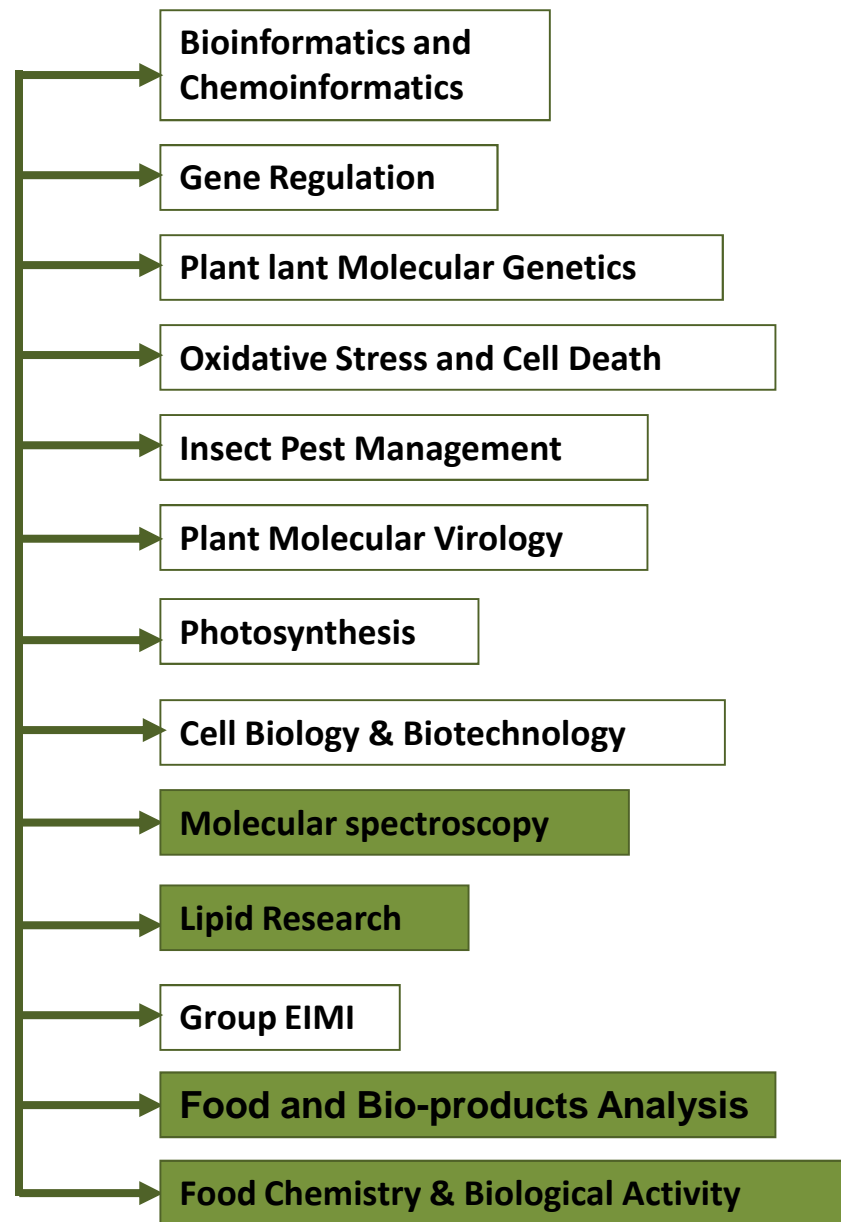
Strengthening the University of Plovdiv Research Potential in Plant Systems Biology and Food Biotechnology
(BioSupport)

funding scheme:

Coordination and support actions (Support)
THEME
CAPACITIES – RESEARCH POTENTIAL

Work programme topics addressed

Area: 4.1.
Unlocking and developing the Research Potential of research entities established in the EU's Convergence Regions and Outermost regions



Prevention

Atom Economy

Less Hazardous Chemical

Designing Safer Chemicals

Safer Solvents

Energy Efficiency

Renewable Feed stocks

Reduce Derivatives

Catalysis

Design for Degradation

Real-time analysis

Accident Prevention

Green Analytical Methods Academic Centre
<http://gama.argon.uni-plovdiv.bg/>

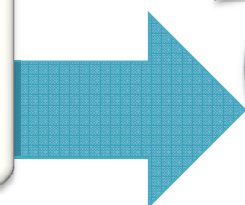


ФОНД
НАУЧНИ
ИЗСЛЕДВАНИЯ

Министерство на образованието, младежта и науката

Национален Фонд „Научни изследвания”
ИНТЕГРИРАНИ НАУЧНИ ЦЕНТРОВЕ В
УНИВЕРСИТЕТИТЕ

УНИВЕРСИТЕТСКИ ЦЕНТЪР ЗА РАЗВИТИЕ
НА ЕКОЛОГИЧНО ЦЕЛЕСЪОБРАЗНИ
МЕТОДИ
ОБЕКТ



MW

SPE

CPE

ASDI

MNPs

FBA

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СЕМИНАР 5-6 Юни 2012 г.

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АКРЕДИТИРАН
АНАЛИТИЧЕН ЦЕНТЪР
ЗА ЛАБОРАТОРНИ ИЗПИТВАНИЯ
НА ХРАНИ, НАПИТКИ
И КОЗМЕТИКА

FDA

U.S. Food and Drug Administration

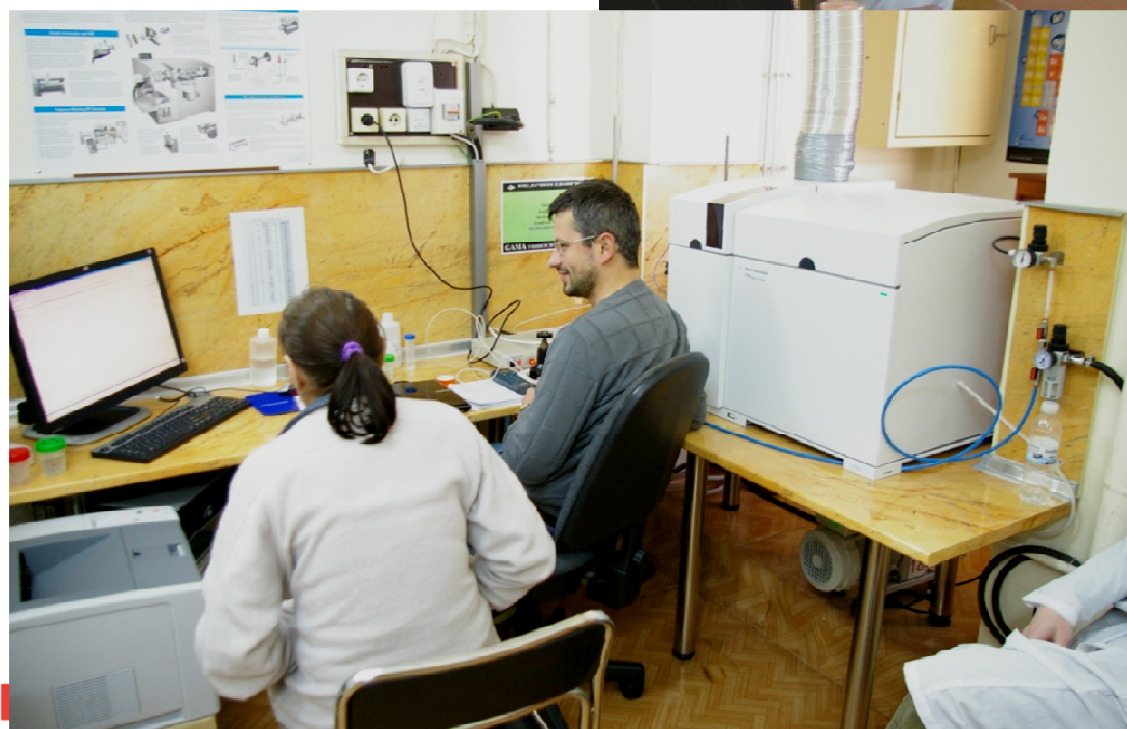
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СЕМИНАР 5-6 Юни 2012 г.

ФВА инструментална екипировка

**ETAAS with
Zeeman
Perkin
Elmer 5100**

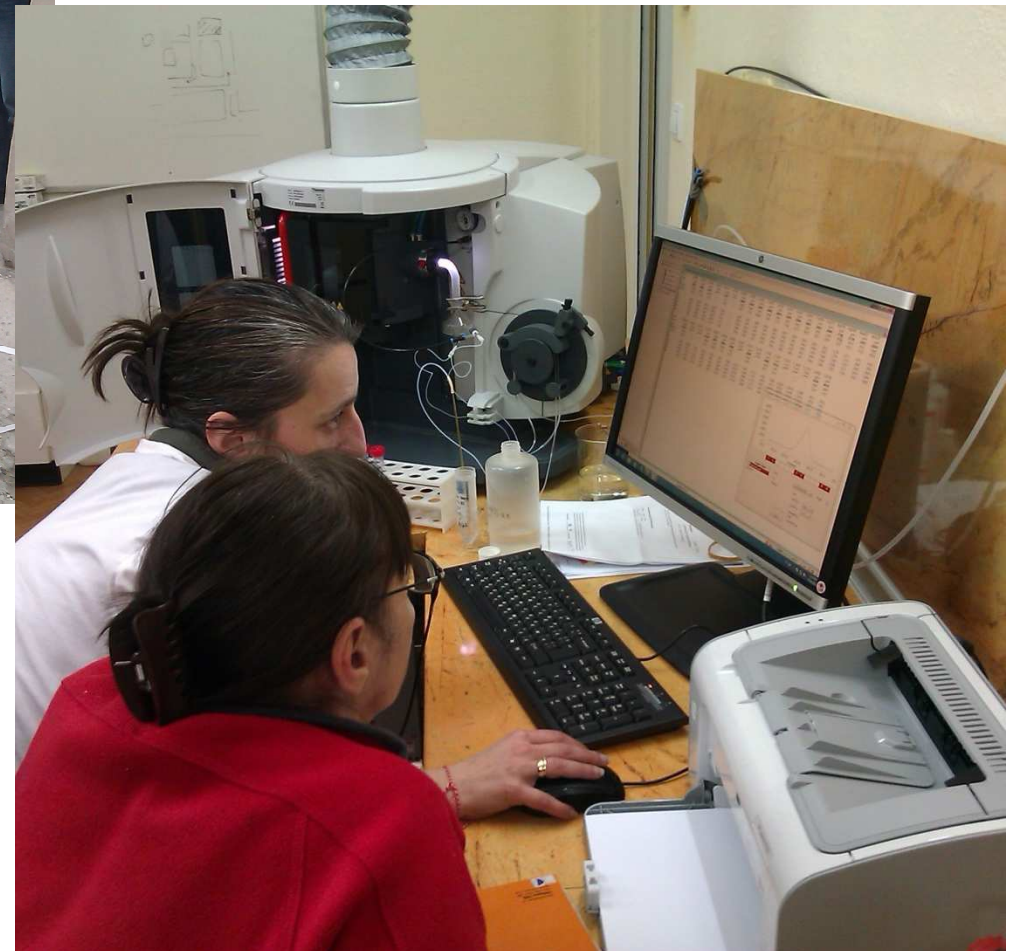


**ICP-MS with
helium
collision cell
Agilent
7700**

FBA апаратура закупена от BioSupport



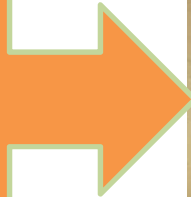
ACM2



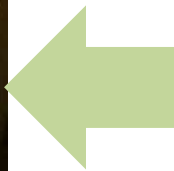
ICP-OES
Duel view
plasma
iCAP 6300
Thermo

FBA апаратура закупена от BioSupport

Microviwe
High pressure
30 At
oven with TV
Milestone
Ehtos One



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MDS-81D,
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Journal of Analytical Atomic Spectrometry

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Kiril Simitchiev^{a,*}
Alfredo Sanchez^b

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^b Department of Analytical Chem

Unmodified manganese ferrite nanoparticles as a new sorbent for solid phase extraction of trace metals - APDC complexes followed by inductively coupled plasma mass spectrometry analysis

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Received (in XXX, XXX) Xth XXXXXXXXXX 20XX, Accepted Xth XXXXXXXXXX 20XX
DOI: 10.1039/b000000x

Abstract

Ultrasound-assisted (ICP-OES) analysis. A multivariate approach was completely optimized in 30 min time-at-preparation which lowers the preparation time, 90 °C temperature methods were validated yielding microwave Cu, Pb and Zn. We not able to be determined limitations that microwave is considerably cheaper © 2004 Elsevier B

Keywords: Ultrasound

Abstract

An automated, fast Mn in moss. The method determination of all the diverse analytical solutions as well. An absorption detection tolerates the introduction application of data containing high Ca worsening the analytical MS one. The ASDI MS determination. reference materials © 2002 Elsevier Sci

Keywords: FAAS; Dis

ARTICLE INFO

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Keywords:
ICP-MS
Platinum group metals
Road dust
Correction of spectral interference
Uncertainty budget analysis
Microwave-assisted cloud point

The applicability of MnFe₂O₄ nanoparticles as a new sorbent for group pre-concentration of V, Co, Ni, Cu, Zn, As, Se, Cd and Pb was investigated and compared with magnetite nanoparticles. A solid phase extraction (SPE) of target analytes based on sorption of their hydrophobic complexes with ammonium pyrrolidine dithiocarbamate (APDC) on the surface of unmodified nanoparticles (NPs) was optimized. Magnetic NPs with retained metal complexes were easily separated from the bulk solution by permanent magnet for 5 min. Analytes restoration in final solution was accomplished by heating with 0.5 mL of 7 mol L⁻¹ nitric acid. The obtained solutions were suitable for continuous nebulization in ICP-MS. Spectral and non-spectral matrix effects for urine analysis (direct and after SPE) were studied and adequate calibration strategies were suggested. Under optimized conditions the magnetic assisted SPE procedure enables enrichment of target analytes by factors between 7.4 – 10, with linear dynamic ranges 1–100 µg L⁻¹ for V, Co, Ni, Cd, Pb and 10–1000 µg L⁻¹ for Zn, As, Se. The method detection limits of proposed SPE were improved by factor between 3 and 20 compared to the direct analysis of diluted urine. The accuracy of magnetic NPs-SPE-ICP-MS method was evaluated analysing urine certified reference material Seronorm™ Trace Elements Urine 201205. For correct determination of As and Se in urine, a preliminary microwave sample treatment with a mixture HNO₃+H₂O₂ was needed, but it led to worsening of MLOD. The developed method was successfully applied for analysis of human urine



Investigation the ecological state of the *Maritza-iztok* region
 in Bulgaria using different methods for analysis

INVESTIGATION THE ECOLOGICAL STATE OF THE *MARITZA-IZTOK* REGION
 IN BULGARIA USING DIFFERENT METHODS FOR ANALYSIS

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²Joint Institute of Nuclear Research, Dubna, Russia

³Regional Inspection of the Ministry of Ecology, Stara Zagora, Bulgaria

⁴Agrarian University of Plovdiv, Plovdiv, Bulgaria

⁴Regional Inspection of the Epidemiological Institute, Plovdiv, Bulgaria

Abstract. Soil, water and vegetable samples from the region of *Maritza-Iztok* Coal-Fired Power Plants (CFPP) in Bulgaria have been studied using a large variety of physical methods of analysis: γ - and neutron activation analyses, γ -spectrometry, radiometry, massspectrometry, physicochemical and radiochemical analyses, soil sciences studies.

An information about the concentration of ecologically important elements in the chain of the products (bottom of environmental

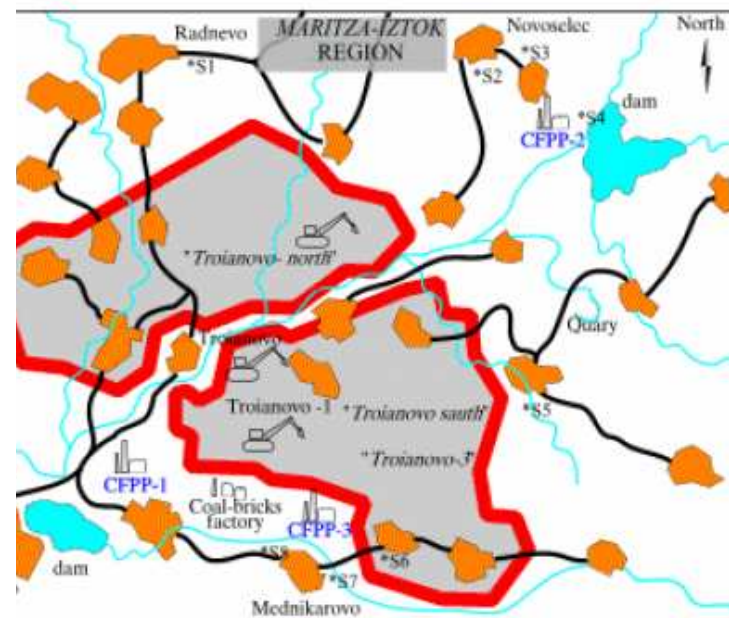
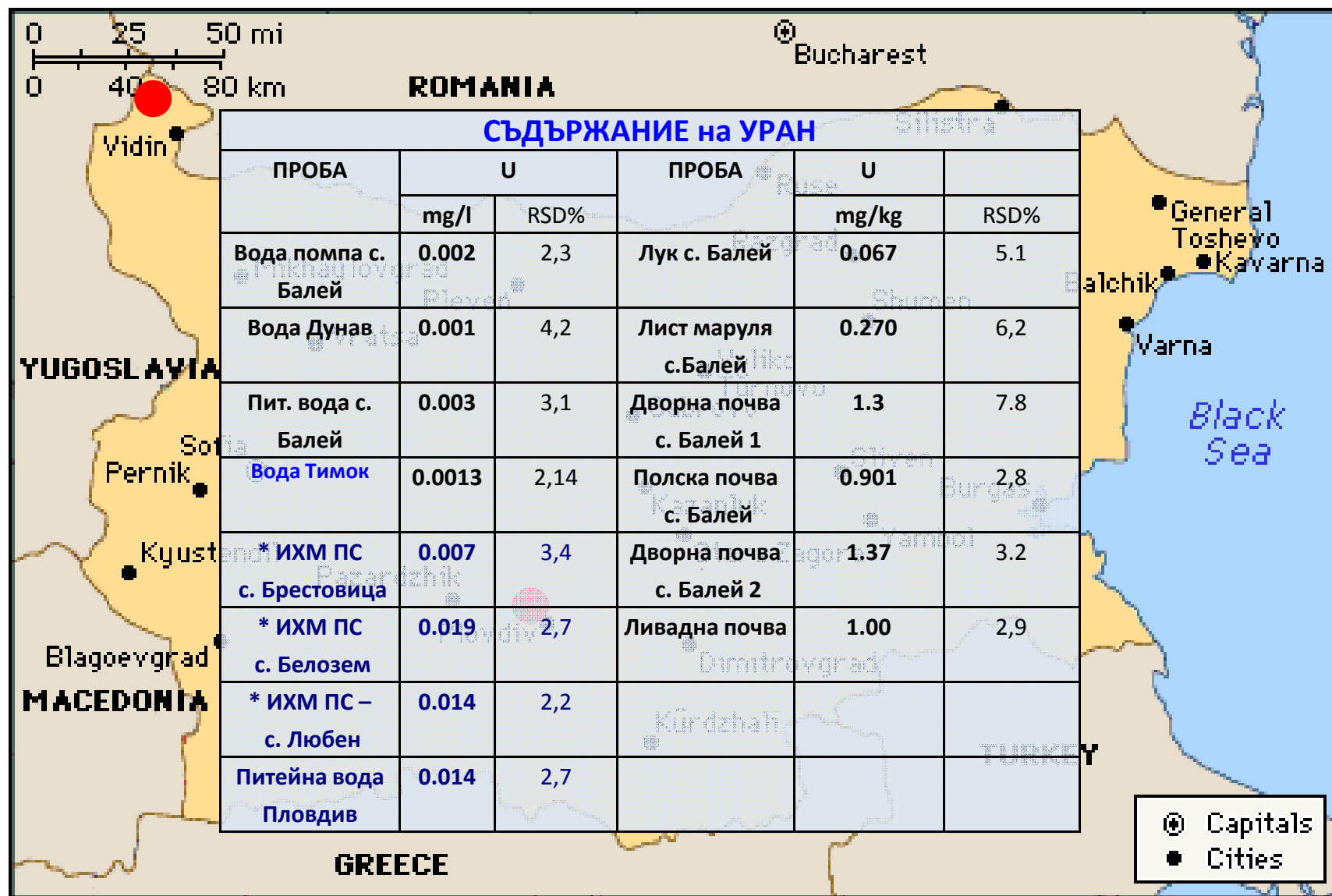


Fig.1. Map of the *Maritza-Iztok* region

Table 1. Element's concentrations in soil samples obtained by IGAA and MSA methods

Element	C _x (IGAA), [ppm]		C _x (MSA), [ppm] + (SD%)		MAC (for pH>7.0)
	Sample S1	Sample S9	Drift Soil	Depository CFPP3	
Mn	600±46	391±44	319(2.0)	425(8.7)	
Cu	-	-	18.2(3.3)	58.8(4.2)	260
Ni	42±11	33±5	15.5(3.0)	63.9(5.2)	70
Cr	40±12	64±16	13.9(3.9)	47.7(6.4)	200
Zn	-	-	16.9(7.5)	20.6(7.5)	340
As	8±1.5	8±3	2.9(6.3)	5.9(8.4)	25
Cd	-	-	<0,1(-)	0.11(10.7)	3,0
Ba	478±42	345±33	117(2.8)	<0,1(-)	
Pb	46±15	70±25	12.2(2.0)	8.7(2.7)	80
U	2±0.2	3.5±0.6	1.3(3.4)	6.04(1.8)	
Hg	-	-	<0.1(-)	<0.1(-)	1
Th*	7.5±0.6	8.3±1.3	-	-	

Проби от Село Бaley и помпени станции в района на Пловдив Института по Хидрология и Метеорология – БАН филиал Пловдив



Данни от анализа на As в минерални води



Данни от анализа на As в минерални води

Вода закупена на	Бутилка от	ICP-MS As (ppb)	RSD%	GFAAS As (ppb)	RSD %
M 13.04.05	1500 ml	100	4.4	94	9
M 13.04.05	500 ml	102	4.3	98	8
M 14.04.05	1500 ml	103	4.2	99	8
M 14.04.05	500 ml	109	5.6	93	9
M 18.05.05	500 ml	106	4.3	99	8
GB 18.05.05	500 ml	< 5	–	< 10	–
K 18.05.05	300 ml	< 5	–	< 10	–
D 18.05.05	500 ml	< 5	–	< 10	–
DG 18.05.05	1500 ml	< 5	–	< 10	–

Съдържание на **As** в бутилирана минерална вода **M**
(\pm) съответната разширена неопределеност **U** при **K=2**

4 mg/L

2 mg/L

DETERMINATION OF TRACE ELEMENTS IN COMMERCIAL BOTTELED BULGARIAN MINERAL WATERS BY MEANS OF ICP-MS AND TXRF


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² Plovdiv University "Paisii Hilendarski", Tsar Assen Str. 24, 4000 Plovdiv, Bulgaria

Рамкова директива за водите и планиране на водните ресурси

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ENVIRONMENT

European Commission


European Commission > Environment > Water > Water Framework Directive

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- Flood Risk Management ▶
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- Bathing Water ▶
- Water Pollution ▶
- Adaptation to Global Change ▶
- Blueprint ▶
- Candidate European Innovation Partnership on Water ▶
- EU Water Initiative
- Water Eurobarometer

The EU Water Framework Directive - integrated river basin management for Europe

Following extensive consultations on Water Framework Directive River Basin Management Plans should since 22.12.2009 be available in all River Basin Districts across the EU. Follow this link or click on the map to see the current status of adoption of river basin management plans in the different Member States.




On 23 October 2000, the "[Directive 2000/60/EC of the European Parliament and of the Council establishing a framework for the Community action in the field of water policy](#)" or, in short, the **EU Water Framework Directive** (or even shorter the WFD) was finally adopted.

The [Directive](#) was published in the Official Journal (OJ L 327) on 22 December 2000 and entered into force the same day. Some amendments have been introduced into the Directive since 2000, and you can download the consolidated version in all EU languages [here](#).

However, even after reading the Directive, you may have still questions, such as: What is this Directive about? What do I need to know about the Directive? What is currently happening in relation to the Directive?

Twelve "Water notes" which intend to give an introduction and overview of key aspects of the implementation of the Water Framework Directive are available to [download in all EU languages](#) !



Your gateway to European water information.

Контролни пунктове – 121 пробни точки от четирите Басейнови дирекции



Анализ на повърхностни води

No	КОД	Al ppb	Al (RSD) %	Cr ppb	Cr (RSD) %	Mn ppb	Mn (RSD) %	Fe ppb	Fe (RSD) %	Ni ppb	Ni (RSD) %	Cu ppb	Cu (RSD) %	As ppb	As (RSD) %	Cd ppb	Cd (RSD) %	Hg ppb	Hg (RSD) %	Pb ppb	Pb (RSD) %	Zn ppb	Zn (RSD) %
1	ССМ	48.6	5	0.22	10	4.2	5	91	7	0.44	8	17.3	7	0.42	8	0.007	25	< 0.01	—	0.1	11	0.99	12
2	Бл 1 А	2	7	0.004	20	4.1	5	5.8	6	0.02	18	12	5	< 0.05	—	0.004	25	< 0.003	—	0.91	5	55	4
3	Бл 1 Б	0.65	10	0.004	20	2.2	6	2.2	6	0.014	20	11.6	5	< 0.05	—	0.003	25	< 0.003	—	0.56	5	54	4
4	ДР1	7.6	5	0.085	9	0.2	6	2.3	6	0.093	9	0.48	4	0.082	9	0.005	11	< 0.003	—	0.053	6	0.86	12
5	ДР3	2.8	6	0.13	10	9.7	5	13.2	6	0.19	9	0.81	4	0.68	6	0.003	14	1.7	10	0.068	6	0.65	13
6	ДР5	1.8	7	0.052	9	12.5	5	42.3	6	0.33	9	0.81	4	0.47	7	< 0.002	—	0.3	9	0.04	6	0.88	12
7	ДР7	1.65	7	0.017	10	105	4	3.8	6	0.32	9	0.79	4	0.32	7	0.016	8	0.1	13	0.064	6	1.1	12
8	ДР9	0.81	7	0.076	9	2.1	5	7.7	9	0.21	9	0.44	4	0.44	7	0.002	19	1	12	0.033	6	0.44	16
9	ДР13	0.62	10	0.37	8	8.1	5	6.3	6	0.27	9	0.35	4	0.73	7	0.004	13	0.9	10	0.034	6	0.64	15
10	ДР16	4.6	6	0.05	10	0.99	5	3.0	6	<0.02	—	0.85	4	0.24	7	0.002	19	0.3	11	0.034	6	0.92	13
11	ДР18	23	6	0.26	8	2.7	5	19.5	6	0.16	9	1.0	4	0.55	7	0.002	19	1.1	8	0.079	6	0.83	12
12	ДР22	1.45	7	0.081	9	4.4	6	13.1	6	0.31	9	0.44	4	0.48	7	0.004	14	1.9	10	0.265	5	0.91	12
13	ДР26	0.9	9	0.42	8	0.39	6	1.8	6	0.48	9	0.46	4	0.98	7	0.002	17	0.5	9	0.028	6	0.66	15
14	ЗБР1	2.7	6	0.059	10	1.52	4	2.6	6	0.062	10	0.65	4	0.22	7	0.012	8	0.02	10	0.05	6	0.02	12
15	ЗБР2	7.9	5	0.043	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
16	ЗБР3	9.4	6	0.018	11	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
17	ЗБР4	3.7	6	0.048	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
18	ЗБР5	5.2	6	0.078	9	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
19	ЗБР6	3.9	6	0.014	12	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
20	ЗБР7	19.5	6	0.075	9	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
21	ИБР1	22	5	0.025	11	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
22	ИБР2	7	6	0.01	12	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
23	ИБР3	4	6	0.05	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
24	ИБР4	9	6	0.09	7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
25	ИБР5	1.2	6	0.06	7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
26	ИБР6	1.7	6	0.25	5	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
27	ИБР7	1.6	6	0.054	7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
28	ИБР8	13.7	5	0.032	7	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
29	ИБР9	1.6	6	0.021	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
30	ИБР10	14.4	6	0.052	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
31	ИБР11	6.8	6	0.065	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
32	ЧР24	3.1	6	0.075	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
33	ЧР25	2.4	6	0.18	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
34	ЧР27	2	7	0.037	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
35	ЧР28	6.5	8	0.074	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
36	ЧР29	1.4	7	0.035	9	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
37	ЧР30	3.2	6	0.19	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
38	ЧР31	4	25	0.27	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
39	ЧР32	1.7	6	0.039	8	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
40	ЧР33	1.8	8	0.066	10	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—

ПРИЛОЖЕНИЕ НА ХЕМОМЕТРИЧНИ ПОДХОДИ ЗА ИНТЕРПРЕТАЦИЯ НА ДАННИ ОТ ICP-MS АНАЛИЗ НА ПОВЪРХНОСТНИ ВОДИ

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София 1164

Резюме

Обект на изследването са повърхностни води от територията на България, от гледна точка на елементния им състав. Изотопите в диапазона 7- 238 аму бяха измерени с ICP-MS модел Agilent 7700x в 120 пробни води от пунктове за мониторинг, регламентирани в законодателството. За обработка на информацията и решаване на класификационни задачи, върху данните от анализа бяха приложени хемометрични методи – СА, РСА с FA, DA. Бяха потърсени елементни маркери за обособените региони с цел идентификация на произход на неизвестни водни проби.

Анализ на повърхностни води

GAMA - lab performance check

River water Reference Material
for Trace Metals SLRS-5

Element	Measured GAMA lab Agilent ICP-MS 7700 $\bar{x} \pm u_m$		Certified SLRS-5 $X_a \pm U (k=2)$		Z	Z'	zeta- score
	x	u_x	X_a	U_a			
Al ppb	48.6	± 2.4	49.5	± 5	-0.4	-0.4	-0.3
Cr ppb	0.22	± 0.022	0.208	± 0.023	1.0	0.5	0.5
Mn ppb	4.2	± 0.21	4.33	± 0.18	-1.4	-0.6	-0.6
Fe ppb	91	± 6.4	91.2	± 5.8	-0.1	0.0	0.0
Ni ppb	0.44	± 0.035	0.476	± 0.064	-1.1	-1.0	-0.8
Cu ppb	17.3	± 1.2	17.4	± 1.3	-0.2	-0.1	-0.1
As ppb	0.42	± 0.034	0.413	± 0.039	0.4	0.2	0.2
Cd ppb	0.007	± 0.0018	0.006	± 0.001	1.4	0.6	0.5
Hg ppb	< 0.01						
Pb ppb	0.1	± 0.011	0.081	± 0.006	6.3	1.7	1.7
Zn ppb	0.99	± 0.12	0.845	± 0.095	3.1	1.2	1.1

Приоритетни вещества:
Hg, Cd, Pb, Ni + Zn

директива 2009/90/ ЕО

критерии за анализ :

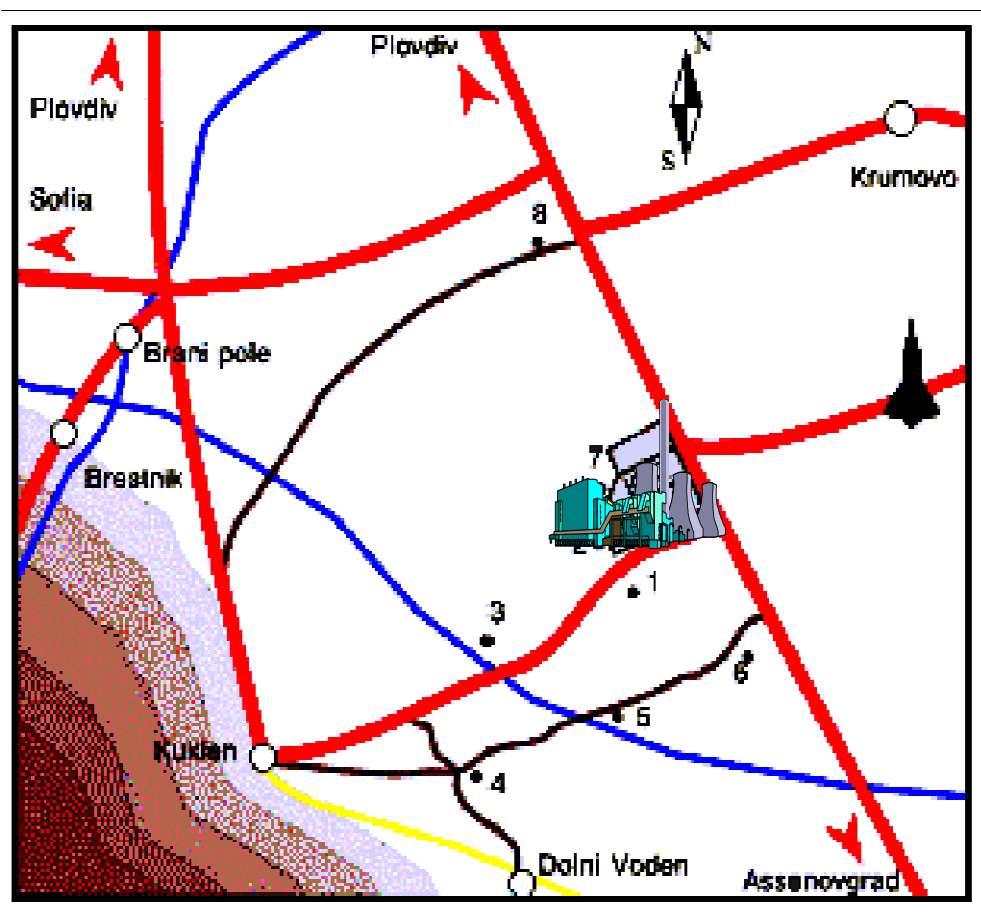
максималните стойности за
неопределеността и
границата на количествено
определяне както следва:

$$U_{\max} = 50\% (k=2)$$

$QL_{\max} = 30\%$ от определените допустими
стойности.

INCO-Copernicus Project CT 98-0334

Оценка на здравния риск в хранителната диета на деца в индустриални райони на Източна Европа



Обследване на източниците на замърсяване с Pb и Cd в ежедневноната храна на деца от Куклен – 4 km до КЦМ АД Пловдив

Резултати за Pb и Cd определени в храни чрез ICP-MS и ETAAS

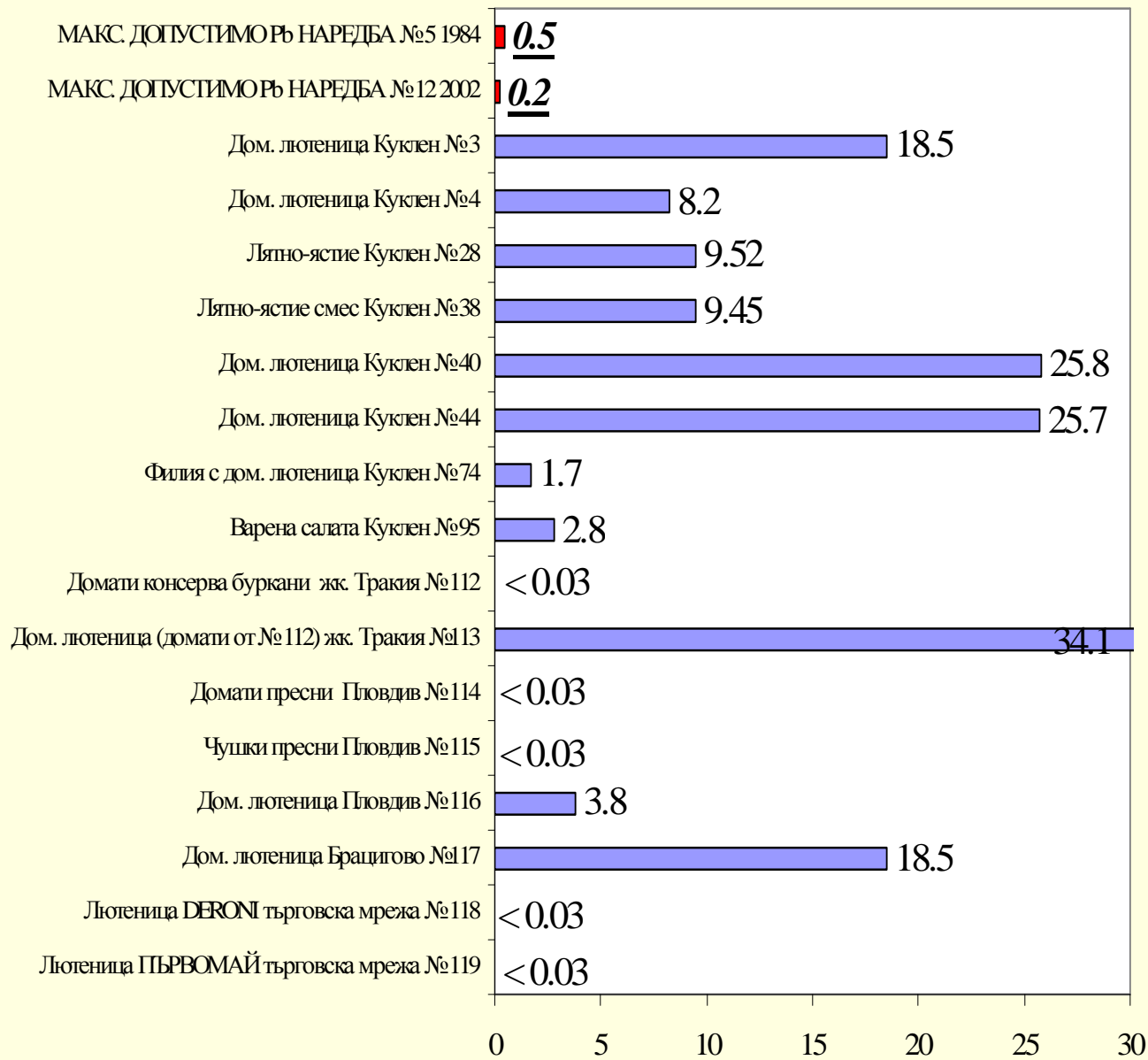
ПРОБА	ICP-MS		ETAAS		ICP-MS		ETAAS	
	Cd µg/kg	SD µg/kg	Cd µg/kg	SD µg/kg	Pb µg/kg	SD µg/kg	Pb µg/kg	SD µg/kg
Зеленчуков а салата -1	164	13	161	11	138	24	110	19
Зеленчуков а салата -2	23	4.8	30	5.6	11	1.5	13	1.6
Свински дроб	53	6.1	51	3	71	2.6	67	3.9
Мляко Куклен	9	2.1	8	0.82	11	1.5	9	1
Мляко Руен	12	2.3	13	1	8	2.4	8	1.2

Анализ на храни – деца от Куклен

Вид на пробата	Cd mg/kg	SD mg/kg	Pb mg/kg	SD mg/kg	Вид на пробата	Cd mg/kg	SD mg/kg	Pb mg/kg	SD mg/kg	Вид на пробата	Cd mg/kg	SD mg/kg	Pb mg/kg	SD mg/kg
Меню – детска градина	< 20	-	< 40	-	Хляб с яйца	< 15	-	< 20	-	Яйца	32	4	< 50	-
Компот сливи	< 5	-	< 10	-	Боза	< 5	-	< 10	-	Ориз с месо и зеле	< 30	-	< 40	-
Лютеница	35	4	18500	450	Сирене	< 30	-	< 50	-	Ориз със зеле и месо	< 30	-	< 50	-
Лютеница	70	8	8200	130	Грозде	< 10	-	330	8	Баница	17	2	21	4
Целодневно меню	< 30	-	< 50	-	Хляб с наденица	< 30	-	< 40	-	Хляб с шоколад	< 15	-	< 20	-
Домати	< 10	-	< 10	-	Домати и сирене	14	1	12	1	Ястие с пилешко	< 30	-	< 50	-
Хляб	< 30	-	< 40	-	Домати	24	3	24	2	Месо + зеленчуци	< 30	-	< 50	-
Сирене	< 30	-	< 50	-	Месо с картофи	< 30	-	< 50	-	Компот кайсии	< 5	-	38	3
Дом. сок	< 30	-	< 40	-	Чушка със сирене	23	2	11	2	Консерва	46	6	9450	160
Наденица	< 30	-	< 50	-	Дом.сок	24	2	12	1	Компот сливи	< 5	-	< 10	-
Зеле	< 20	-	< 40	-	Дом. сок	11	2	< 10	-	Лютеница	65	4	25800	550
Кис. мляко	< 5	-	< 5	-	Конфитюр сливи	< 30	-	< 50	-	Праз	< 15	-	< 40	-
Мляко	< 5	-	12	1	Компот кайсии	< 5	-	< 10	-	Чушки	29	3	< 40	-
Макарони + сирене	< 15	-	< 30	-	Компот праскови	< 5	-	< 10	-	Грозде	< 5	-	23	2
Смляно месо	< 30	-	< 50	-	Пресни яйца	< 15	-	< 40	-	Лютеница	60	7	25700	700
Бисквити	< 15	-	< 20	-	Пърж. яйца	< 20	-	42	4	Пърж. картофи сирене	51	4	< 40	-
Ябълка	< 5	-	< 10	-	Салата зеле моркови	< 30	-	< 50	-	Баница	< 15	-	< 20	-
Мусака	< 30	-	< 50	-	Принцеса с кайма	< 30	-	< 50	-	Домати	15	2	< 10	-
Кис. мляко	12	2	8	2	Черен дроб	53	6	71	3	Сок череша	< 5	-	15	2
Пр. мляко	< 5	-	< 5	-	Хляб с яйца	< 15	-	< 20	-	Грозде	< 5	-	75	3
Сирене	< 30	-	< 50	-	Мляко	< 5	-	< 10	-	Айран	< 5	-	< 5	-
Кис. мляко	< 5	-	< 5	-	Хляб с лютеница	< 20	-	1700	80	Салата домати	11	1	11	1
Палачинка + мармалад	< 30	-	220	9	Мляко	< 10	-	< 10	-	Месо	< 30	-	< 50	-
Салата домати	18	2	16	2	Ориз	< 30	-	< 50	-	Круша	< 5	-	25	1
Ориз с месо	< 30	-	145	10	Грозде	< 5	-	50	3	Ябълка	6.2	2	81	4
Баклава	< 30	-	< 50	-	Диня	< 5	-	< 10	-	Салата домати чушки	14	1	13	1
Компот	< 5	-	15	2	Компот сливи	6	2	10	1	Картофи	< 30	-	< 50	-
Ястие с домати	129,40	513	9520	316	Месо с картофи	< 35	-	< 50	-	Макарони с сирене	< 15	-	41	3
Ябълка	< 5	-	76	3	Яйца	< 20	-	< 40	-	Дом. сок	< 15	-	< 10	-
Царев. брашно	< 30	-	390	20	Кюфте	< 30	-	< 50	-	Ориз + домати	< 20	-	< 50	-
Изсушени яйца	< 20	-	160	15	Картофено кюфте	38	4	< 50	-	Айран	< 5	-	< 10	-
Изсушен праз	164	13	138	12	Мляко	9	2	11	2	Яйца със салата	20	3	< 50	-
Айран	< 5	-	< 5	-	Брашно	< 30	-	< 50	-	Кашкавал	< 20	-	140	10
Салата зеле и моркови	48	5	2800	112	Баница	< 30	-	66	5	Компот праскови	< 5	-	< 10	-
Компот череша	< 5	-	< 10	-	Брашно	< 30	-	< 50	-	Ориз	< 30	-	92	8



Съдържание на Pb (mg/kg) в хранителни продукти



КОЙ Е ВИНОВНИКЪТ ЗА ВИСОКОТО СЪДЪРЖАНИЕ НА Рb В ДОМАШНАТА ЛЮТЕНИЦА!!!



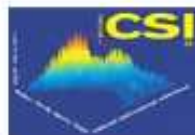
ФВА ЛАБОРАТОРИЯ BioSupport





Аграрен Университет
анализ на съдържание на
тежки метали в почви и
подвижни форми на фосфор

Проф. Красимир Иванов



Colloquium Spectroscopicum
Internationale XXXVII

TU32

Comparison of ICP-MS and colorimetric determination of total and extractable phosphorous in soils

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Sample №	blank	El c.v.	El HClO4	PC1 MWc.v.	PC1 c.v.	PC1 HClO4	PC2 MWc.v.	PC2 c.v.	PC2 HClO4	PC3 MWc.v.	PC3 c.v.	PC3 HClO4	303 MWc.v.	303 c.v.	303 HClO4	386 MWc.v.	386 c.v.	386 HClO4	113 CRM	
Be	mg/l	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	
	SD	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
B	mg/l	<1	<1	15.57	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	
	SD	-	-	0.3	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Na	mg/l	0.19	21.4	2.8	5.5	20.5	6.6	3.7	36.8	5.0	4.2	34.9	7.1	34.9	54.0	31.7	3.7	31.7	7.0	6.0
	SD	0.003	0.64	0.07	0.19	0.82	0.36	0.25	1.18	0.23	0.14	0.72	0.28	1.61	0.89	0.96	0.13	0.81	0.18	0.18
Mg	mg/l	0.04	0.18	1.66	65.3	21.5	70.3	24.9	29.7	31.0	42.3	43.8	47.5	75.5	84.5	86.7	6.3	11.2	12.0	30.6
	SD	0.004	0.01	0.09	3.05	0.66	1.83	0.45	0.35	0.30	0.52	0.51	0.32	2.56	3.35	3.49	0.25	0.23	0.34	0.41
Al	mg/l	0.01	0.16	0.50	76	39	130	61	98	117	87	105	141	128	159	172	199	363	451	139
	SD	0.001	0.01	0.04	1.98	1.20	2.08	1.10	2.22	3.57	2.01	3.52	2.81	5.91	3.33	4.05	4.72	9.19	22.15	4.05
P	mg/l	0.10	0.04	0.23	2.96	1.45	2.77	2.23	2.09	2.26	3.01	2.86	2.81	5.12	5.06	4.88	0.96	1.15	1.29	4.82
	SD	0.03	0.03	0.15	0.36	0.24	0.36	0.32	0.28	0.26	0.27	0.25	0.37	0.48	0.34	0.42	0.21	0.09	0.28	0.24
S	mg/l	<0,5	0.80	7.62	0.95	8.74	<0,5	0.62	17.95	<0,5	15.85	<0,5	5.63	20.10	<0,5	1.00	15	<0,5	23.3	
	SD	0.000	0.22	1.71	0.11	0.74	0.00	0.37	1.11	0.19	0.09	0.63	0.00	0.78	1.62	0.77	0.58	0.62	0.34	1.40
Ca	mg/l	0.20	0.98	12.94	370	118	379	42	54	57	174	179	183	123	125	129	3.35	15	9	90
	SD	0.028	0.071	0.797	32.3	8.9	37.0	3.2	4.9	4.0	11.0	12.4	12.8	8.7	10.1	9.5	0.8	1.5	0.9	7.2
Cr	mg/l	0.01	0.00	0.02	0.39	0.14	0.36	0.14	0.18	0.15	0.26	0.27	0.20	0.14	0.20	0.14	0.35	0.41	0.22	1.27
	SD	0.001	0.0002	0.004	0.05	0.02	0.05	0.02	0.02	0.02	0.03	0.03	0.02	0.02	0.02	0.01	0.03	0.04	0.02	0.13
Fe	mg/l	0.04	0.48	1.25	92	48	100	86	97	99	109	108	117	244	244	238	333	354	364	141
	SD	0.004	0.07	0.12	8.1	4.8	5.6	7.7	7.9	9.9	7.0	10.7	9.5	25.8	20.6	23.8	27.0	27.3	31.7	14.7
Mn	mg/l	0.00	0.01	0.08	2.8	1.4	2.9	2.3	2.5	2.6	2.5	2.5	2.8	37	37	37	4.9	5.3	5.5	1.4
	SD	0.0001	0.001	0.006	0.24	0.12	0.18	0.27	0.24	0.18	0.14	0.15	0.16	2.6	1.9	1.6	0.37	0.42	0.41	0.13
Co	mg/l	<0,001	<0,001	0.00	0.05	0.02	0.05	0.04	0.04	0.04	0.05	0.05	0.16	0.05	0.05	0.05	0.04	0.05	0.05	0.08
	SD	-	-	0.0003	0.004	0.002	0.005	0.003	0.002	0.005	0.004	0.004	0.016	0.004	0.004	0.004	0.003	0.004	0.004	0.006
Ni	mg/l	0.00	0.00	0.07	0.26	0.11	0.29	0.14	0.14	0.16	0.22	0.21	0.24	0.09	0.09	0.10	0.15	0.16	0.18	1.11
	SD	0.0008	0.0003	0.008	0.02	0.01	0.02	0.01	0.01	0.01	0.02	0.02	0.02	0.01	0.01	0.01	0.01	0.02	0.02	0.06
Cu	mg/l	<0,002	0.00	0.06	0.21	0.08	0.21	0.31	0.28	0.30	0.24	0.20	0.23	0.27	0.26	0.28	0.54	0.60	0.64	0.41
	SD	-	0.000	0.005	0.015	0.007	0.015	0.024	0.018	0.027	0.021	0.015	0.015	0.023	0.023	0.027	0.034	0.046	0.054	0.026
Zn	mg/l	<0,004	0.04	17.76	1.21	0.42	1.12	0.29	0.31	0.32	1.04	0.52	0.60	14.79	14.11	15.55	2.34	2.06	2.07	1.32
	SD	-	0.008	0.93	0.13	0.03	0.08	0.03	0.04	0.03	0.07	0.05	0.05	0.72	0.66	0.80	0.20	0.18	0.18	0.13
As	mg/l	<0,004	<0,004	0.00	0.04	0.02	0.04	0.03	0.02	0.03	0.03	0.02	0.03	0.79	0.76	0.80	1.62	1.60	1.69	0.08
	SD	-	-	0.001	0.006	0.003	0.006	0.004	0.003	0.004	0.004	0.004	0.004	0.094	0.087	0.086	0.12	0.13	0.15	0.006
Se	mg/l	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	<0,004	0.01	0.01	0.01	<0,004
	SD	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	0.002	0.002	0.002	-
Mo	mg/l	<0,001	<0,001	0.05	0.01	0.01	0.01	0.01	0.01	0.01	0.00	0.01	0.01	0.01	0.01	0.01	0.02	0.02	0.02	0.02
	SD	-	-	0.004	0.002	0.002	0.002	0.002	0.002	0.002	0.000	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002	0.002
Cd	mg/l	<0,001	<0,001	0.00	0.02	0.01	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.10	0.11	0.00	0.00	0.00	0.00
	SD	-	-	0.0005	0.0012	0.0004	0.0007	0.0003	0.0003	0.0003	0.0006	0.0004	0.0004	0.0041	0.0046	0.0039	0.0002	0.0004	0.0005	0.0003
Pb	mg/l	<0,001	0.01	0.07	0.51	0.18	0.55	0.18	0.23	0.22	0.34	0.35	0.37	10.87	10.75	11.49	2.22	2.33	2.43	1.56
	SD	-	0.00	0.02	0.50	0.07	0.06	0.04	0.05	0.03	0.08	0.07	0.07	0.51	0.31	0.37	0.04	0.08	0.09	0.07
Bi	mg/l	<0,001	<0,001	0.01	0.01	<0,001	<0,001	0.01	<0,001	<0,001	0.01	0.01	<0,001	0.01	0.01	<0,001	0.16	0.15	0.17	<0,001
	SD	-	-	0.004	0.005			0.004			0.003	0.004		0.004	0.002		0.016	0.018	0.019	
U	mg/l	<0,0005	<0,0005	0.00	0.00	0.00	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.03	0.03	0.03	0.00	
	SD	-	-	0.0001	0.0001	0.0001	0.0002	0.0001	0.0003	0.0001	0.0002	0.0002	0.0003	0.0003	0.0003	0.0002	0.0003	0.0007	0.0006	0.0001



Аграрен Университет анализ на съдържание на тежки метали в почви

доц. Аланджийски



1 g – въздушно суха проба
5 ml HNO₃ + 5 ml H₂O
MW E=800W T 600 (R_t=10min H_t=20 min)
Отваряне за едно денонощие
+ 3 ml HNO₃ + 2 ml H₂O₂
MW E=800W T 600 (R_t=10min H_t=20 min)



Аграрен Университет

анализ на съдържание на тежки метали в почви

доц. Аланджийски



	Soil 1.1 DF=100	Soil 1.2 DF=100	Soil 2.1 DF=100	Soil 3.1 DF=100	Soil 3.2 DF=100	Soil 1 DF=200	Soil 2.1 + 1 ppm	Solil 2.1 + 5 ppm
						0.5	1.00	5.00
Cd2144	1.00	1.01	1.00	1.00	1.02	0.55	0.78	3.90
Cd2265	0.97	0.99	0.92	0.66	0.67	0.55	0.75	3.85
Cd2288	0.96	0.98	0.96	1.02	1.03	0.54	0.81	4.14
Cu2178	1.00	1.07	1.00	1.00	1.00	0.53	0.77	3.96
Cu2247	0.94	1.01	0.92	0.90	0.90	0.55	0.72	3.74
Cu3247	0.90	0.97	0.91	0.92	0.92	0.52	0.87	4.42
Cu3273	0.90	0.97	0.90	0.90	0.91	0.53	0.86	4.45
Pb1822	1.00	1.01	1.00	1.00	0.95	0.58	0.70	3.65
Pb2169	0.92	0.94	0.91	0.78	0.76	0.54	0.74	3.84
Pb2203	1.03	1.05	1.04	1.04	1.00	0.57	0.68	3.56
Pb2833	0.88	0.90	0.87	0.78	0.76	0.54	0.81	4.05
Zn2062	1.00	1.02	1.00	1.00	1.01	0.57	0.67	3.48
Zn2138	1.04	1.07	0.98	0.93	0.94	0.59	0.61	2.98
Zn3345	1.00	1.03	1.04	1.11	1.13	0.54	0.72	3.77
Zn4810	0.99	1.03	1.02	1.02	1.03	0.53	0.73	3.63
					Aver	0.55	0.75	3.83
					Faktor	0.910	0.75	0.77

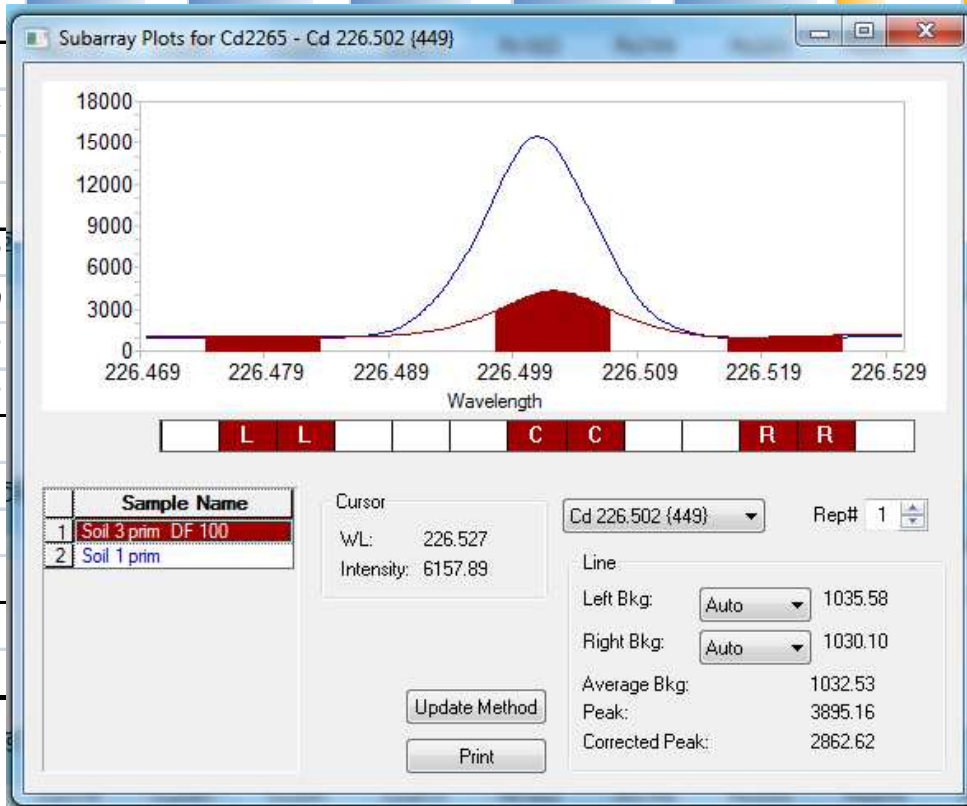


Аграрен Университет анализ на съдържание на тежки метали в почви

доц. Аланджийски



	Soil 1.1 DF=100	Soil 1.2 DF=100	Soil 2.1 DF=100	Soil 3.1 DF=100	Soil 3.2 DF=100	Soil 1 DF=200	Soil 2.1 + 1 ppm	Solil 2.1 + 5 ppm
						0.5	1.00	5.00
Cd2144	1.00	1.01	1.00	1.00	1.02	0.55	0.78	3.90
Cd2265	0.97	0.99	0.92	0.66	0.67	0.55	0.75	3.85
Cd2288							0.81	4.14
Cu2178							0.77	3.96
Cu2247							0.72	3.74
Cu3247							0.87	4.42
Cu3273							0.86	4.45
Pb1822							0.70	3.65
Pb2169							0.74	3.84
Pb2203							0.68	3.56
Pb2833							0.81	4.05
Zn2062							0.67	3.48
Zn2138							0.61	2.98
Zn3345							0.72	3.77
Zn4810							0.73	3.63
							0.75	3.83
							0.75	0.77



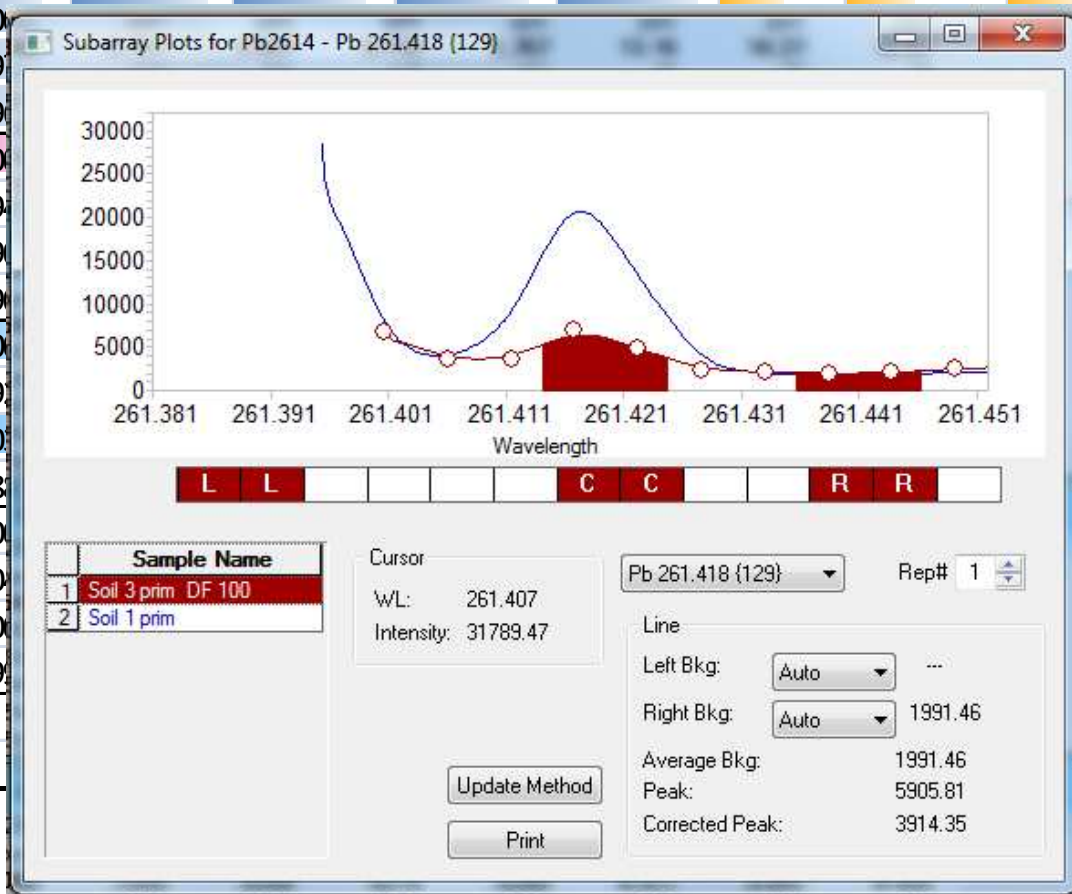


Аграрен Университет анализ на съдържание на тежки метали в почви

доц. Аланджийски



	Soil 1.1 DF=100	Soil 1.2 DF=100	Soil 2.1 DF=100	Soil 3.1 DF=100	Soil 3.2 DF=100	Soil 1 DF=200	Soil 2.1 + 1 ppm	Solil 2.1 + 5 ppm
						0.5	1.00	5.00
Cd2144	1.0							
Cd2265	0.9							
Cd2288	0.9							
Cu2178	1.0							
Cu2247	0.9							
Cu3247	0.9							
Cu3273	0.9							
Pb1822	1.0							
Pb2169	0.9							
Pb2203	1.0							
Pb2833	0.8							
Zn2062	1.0							
Zn2138	1.0							
Zn3345	1.0							
Zn4810	0.9							





Line Library

Search Results

Element	Wavelength	Rel. Intensity	State	BEC	Det. Limit
Pb	247.638	9000	I	9999.00	0.00
Pb	252.669	10	II	9999.00	0.00
Pb	257.66	10	II	9999.00	0.00
Pb	257.727	7000	I	9999.00	0.00
Pb	260.838	2	II	9999.00	0.00
Pb	261.365	3000	I	9999.00	0.00
Pb	261.418	70000	I	9999.00	0.00
Pb	262.826	192	I	9999.00	0.00
Pb	263.426	5	II	9999.00	0.00
Pb	265.709	12	I	9999.00	0.00
Pb	266.316	15000	I	9999.00	0.00

Search Criteria:

Elements: Pb

Wavelength: (130 nm to 999.999 nm)

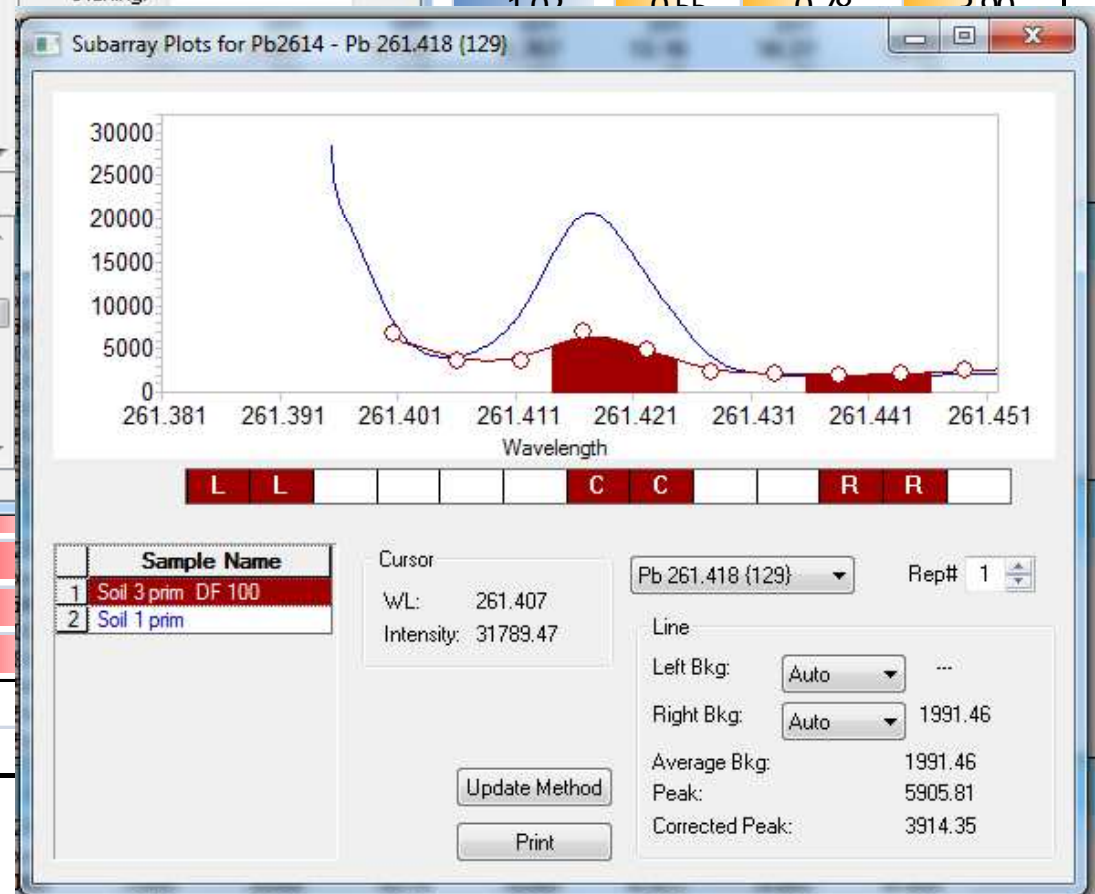
Starting: 130.000

Interfering Lines For Element: Pb Wavelength: 261.418

Element	Wavelength	Rel. Intensity	State	BEC	Det. Limit
Fe	261.382	120000	II	9999.00	0.00
S	261.383	22	II	9999.00	0.00
Nb	261.385	3500	II	9999.00	0.00
U	261.395	4000	II	9999.00	0.00
Ho	261.399	10000	II	9999.00	0.00
Pb	261.418	70000	I	9999.00	0.00

Soil 3.2 DF=100	Soil 1 DF=200	Soil 2.1 + 1 ppm	Solil 2.1 + 5 ppm
	0.5	1.00	5.00
1.03	0.55	0.78	2.00

Zn2062	1.00	
Zn2138	1.04	
Zn3345	1.00	
Zn4810	0.99	





Аграрен Университет
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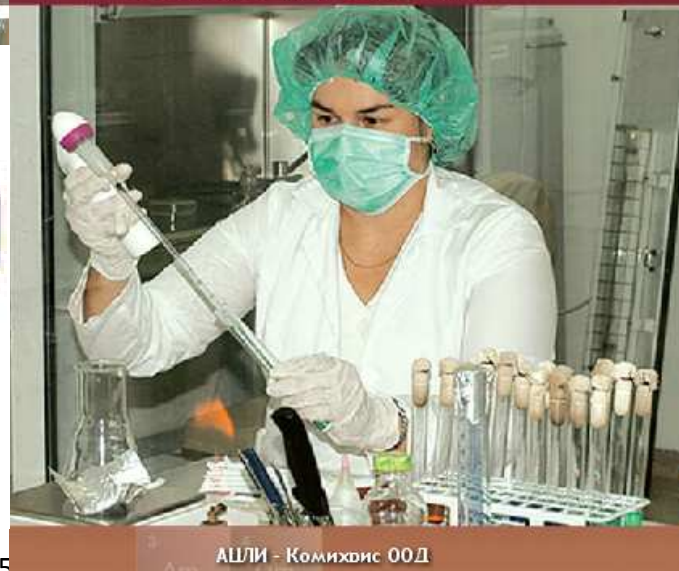


Проба	Pb ppm	<i>Pb</i> RSD%	Cd ppm	<i>Cd</i> RSD%	Cu ppm	<i>Cu</i> RSD%	Zn ppm	<i>Zn</i> RSD%
I Първоначално сито 0.315	1750	6	40	3	160	4	1800	7
II Първоначално	1100	6	27	4	140	5	1150	7
I след сито 0.315	310	7	6	8	105	5	420	7

- Месо
- Мляко
- Риба
- Яйца
- Мед
- Подправки
- Захарни изд.
- Консерви, Не
- Зърнени
- Растителни п
- Вода
- Козметика
- Хигиенен ко



акредитиран аналитичен цент
за лабораторни изпитвания



АШЛ - Колхицис ООД



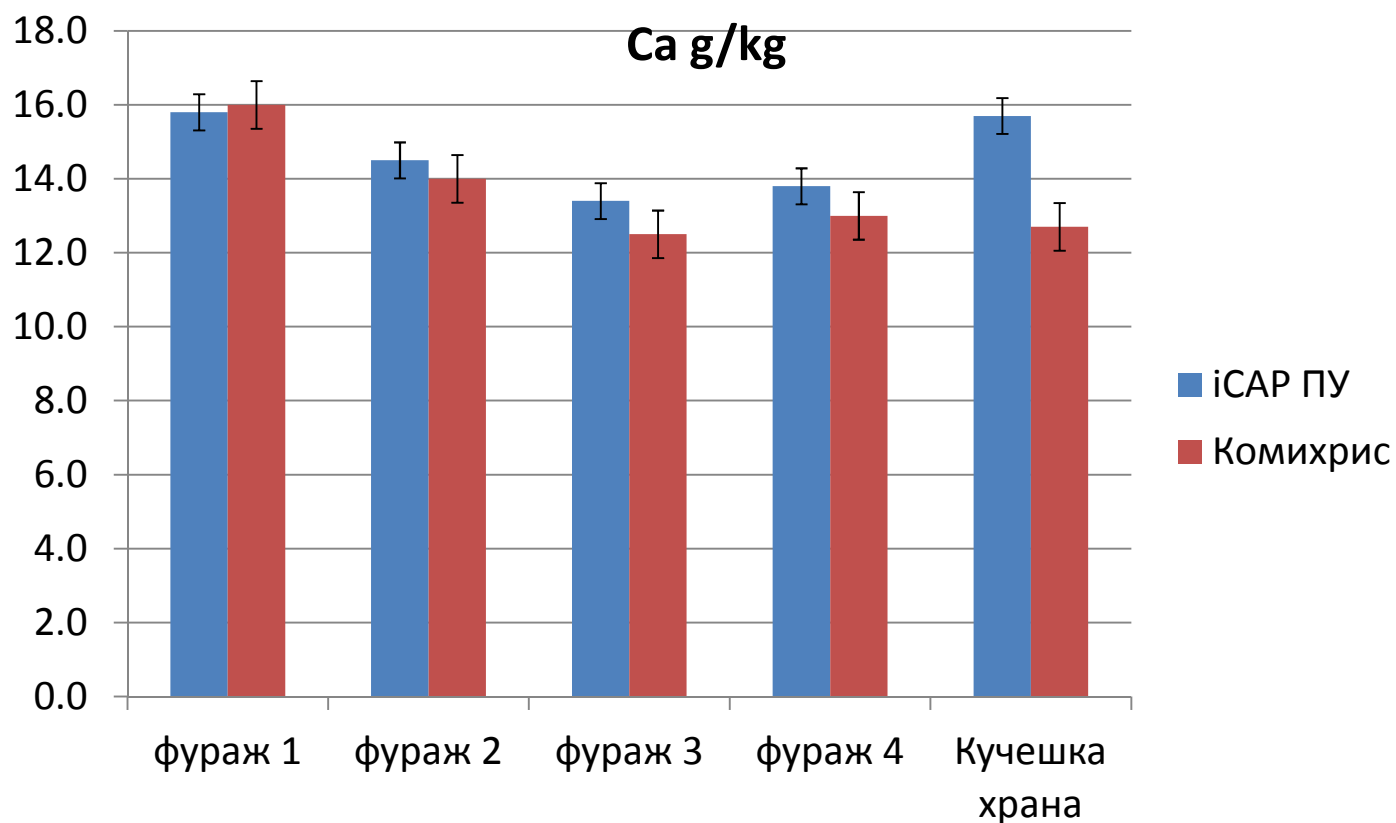


FVA анализ на храни и обекти на околната среда



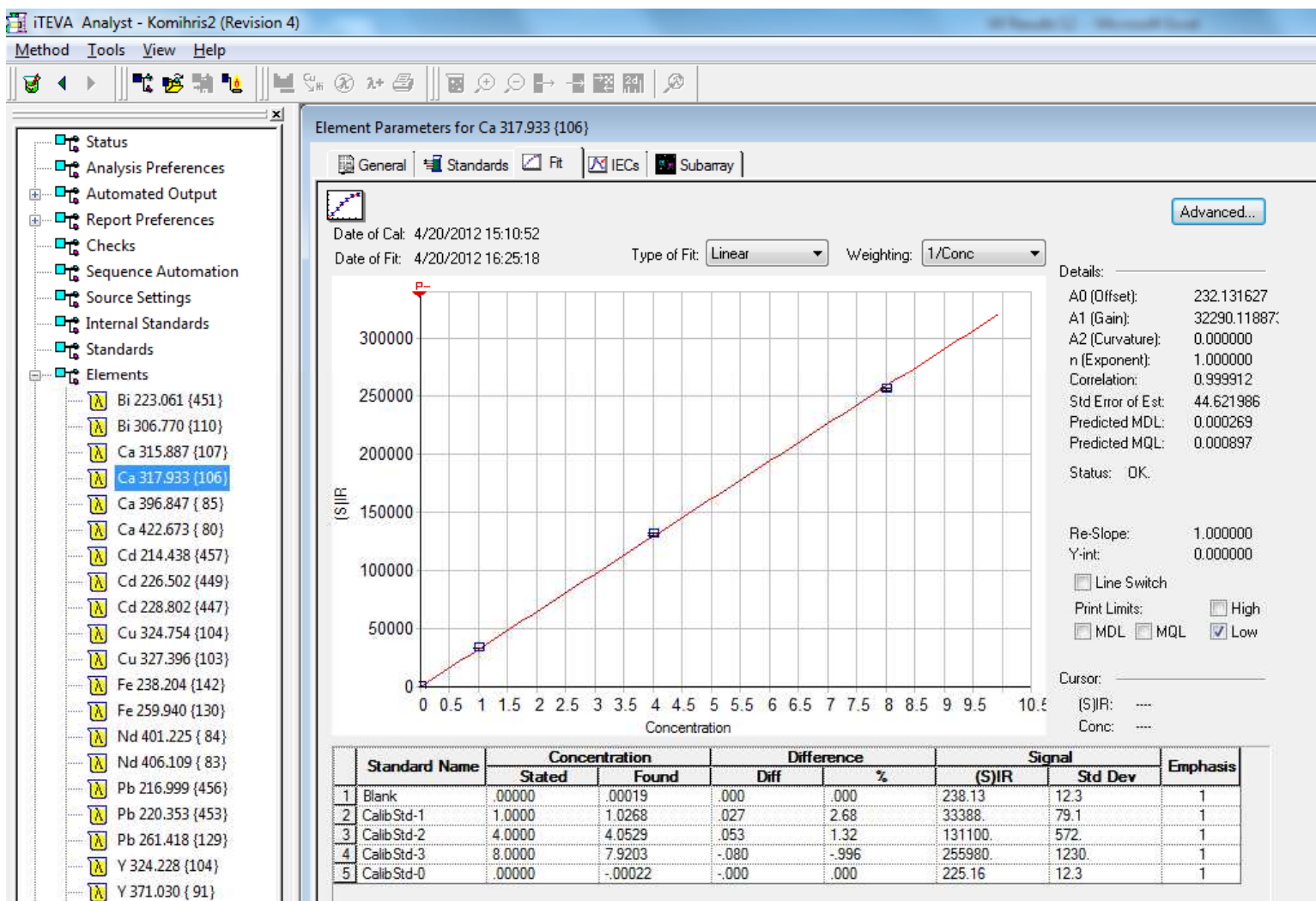
Междулабораторни сравнения при анализ на храни ПУ и Комихрис

Ca – 317.9 nm





Междулабораторни сравнения при анализ на храни ПУ и Комихрис





Междулабораторни сравнения при анализ на храни ПУ и Комихрис

ITEVA Analyst - Komihris2 (Revision 4)

Method Tools View Help

Element Parameters for Ca 317.933 (106)

Date of Cal: 4/20/2012 15:10:52
Date of Fit: 4/20/2012 16:25:18

Subarray Plots for Ca3179 - Ca 317.933 (106)

Wavelength

Standard Name	Concentration Stated	Concentration Found
1 Blank	.00000	0.00000
2 CalibStd-1	1.0000	1.0000
3 CalibStd-2	4.0000	4.0000
4 CalibStd-3	8.0000	7.9999
5 CalibStd-0	.00000	-.00000

Sample Name	WL	Intensity
1 KOMIHRIS SS (10)DF10	---	---
2 KOMIH DogF (1) DF100	---	---
3 BLANK KOMIHRIS DF10	---	---

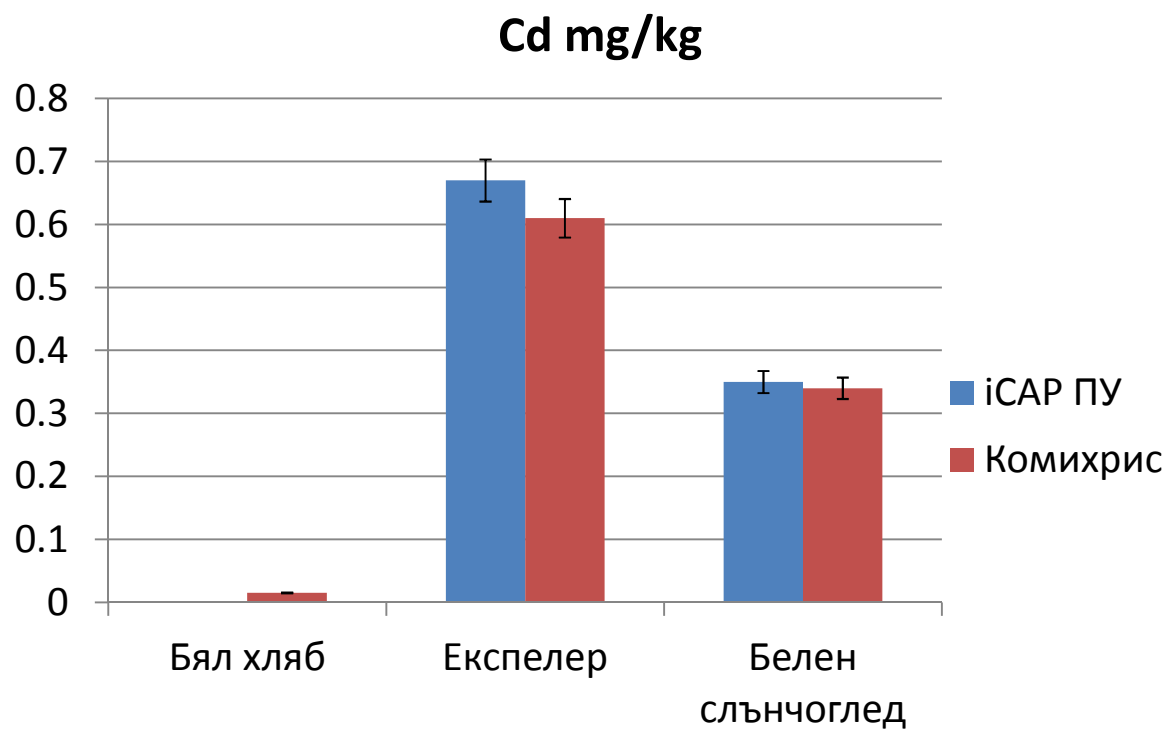
Ca 317.933 (106) Rep# 1

Left Bkg: Auto 1456.29
Right Bkg: Auto 1526.27
Average Bkg: 1494.46
Peak: 50135.11
Corrected Peak: 48640.65

Update Method
Print

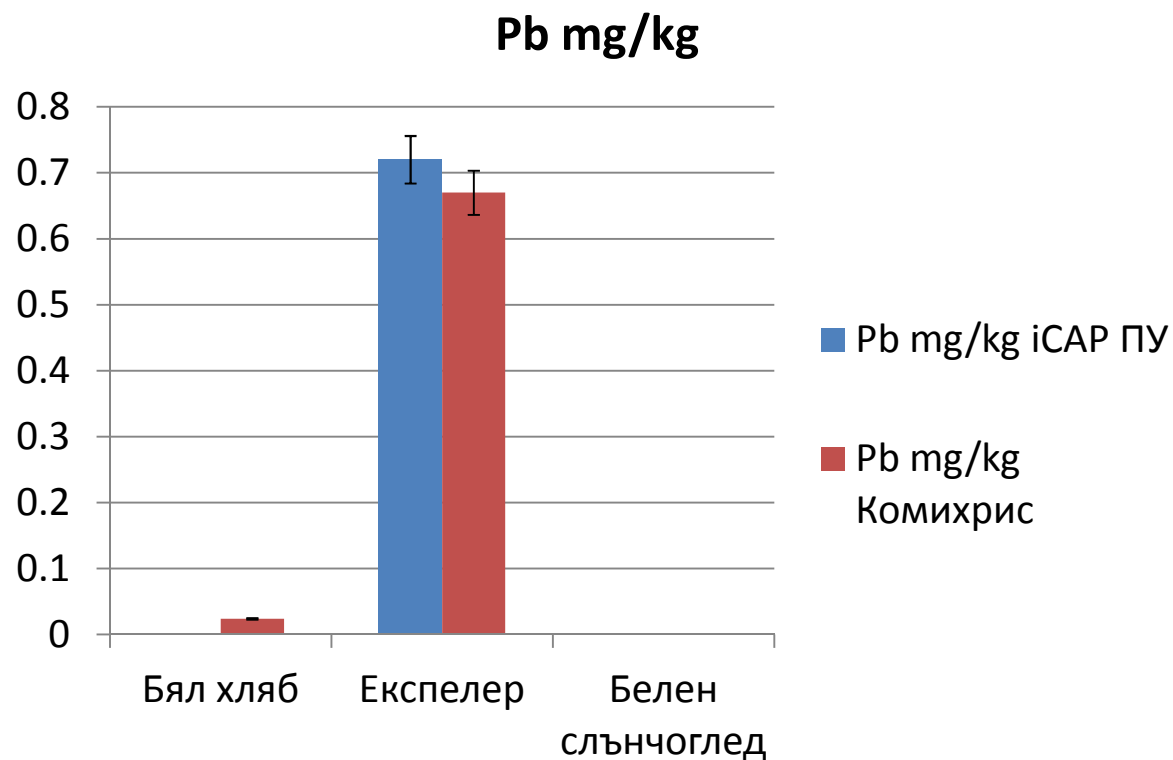


Междулабораторни сравнения при анализ на храни ПУ и Комихрис



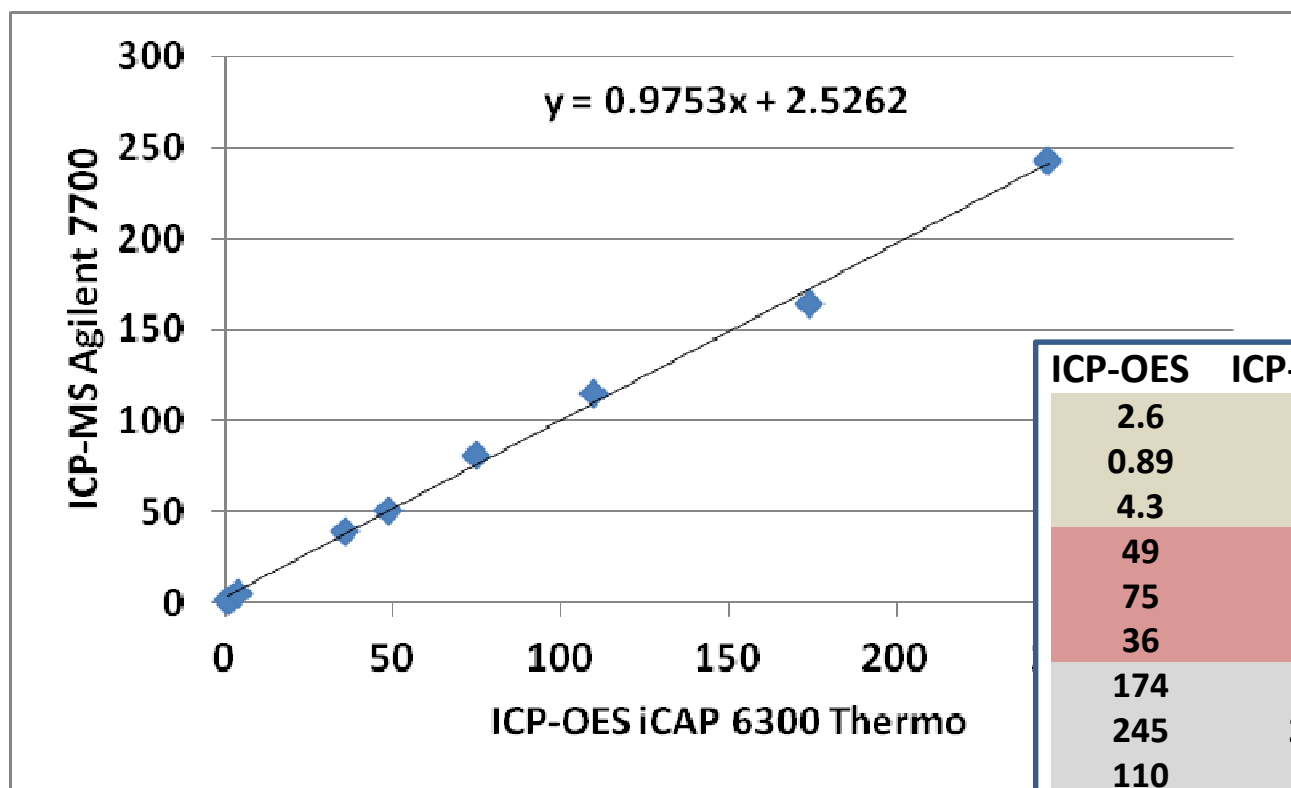


Междулабораторни сравнения при анализ на храни ПУ и Комихрис





Междулабораторни сравнения при анализ на храни ПУ и Комихрис



ICP-OES	ICP-MS	ppb
2.6	2.8	Cd Бел. Слънчогед 1
0.89	1.0	Cd Бел. Слънчогед 13
4.3	5	Cd Бял хляб
49	51	Cu Бел. Слънчогед 1
75	81.0	Cu Бел. Слънчогед 13
36	39.5	Cu Бял хляб
174	164	Zn Бел. Слънчогед 1
245	243.0	Zn Бел. Слънчогед 13
110	115	Zn Бял хляб

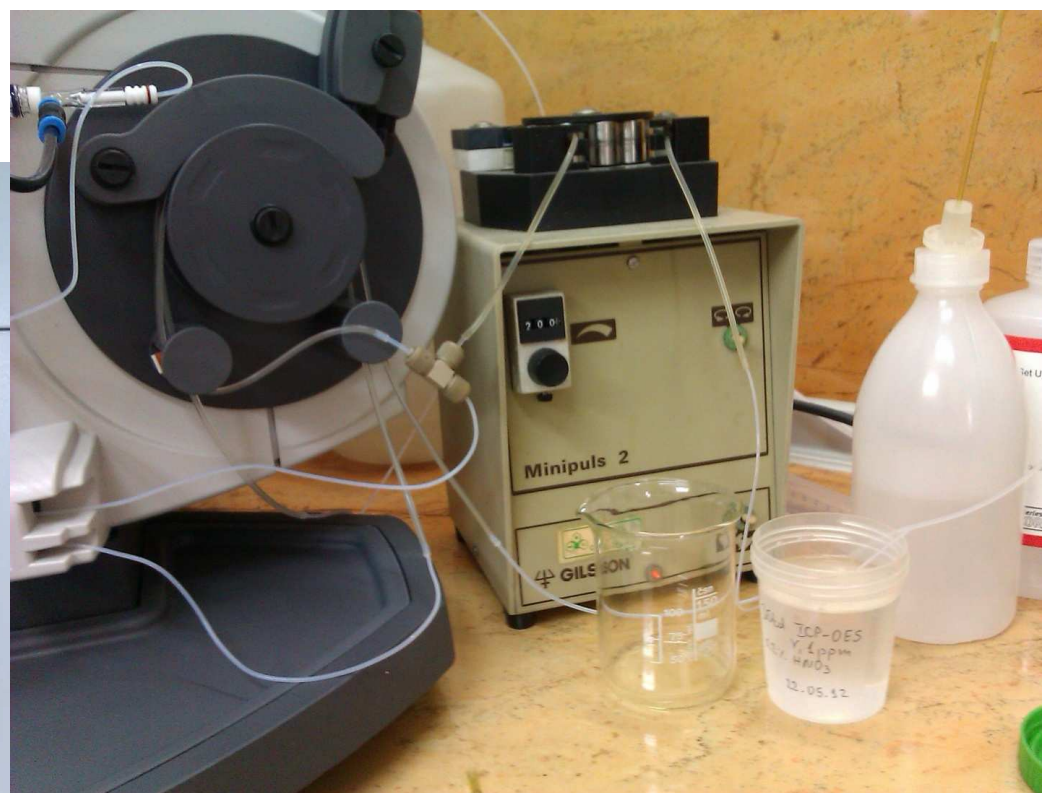
Y Rh IS дрейф
< 5 % < 5 %

Анализ на алуминиево фолио за опаковъчни капачки за храни



Елемент	ICP OES		ICP MS		FAAS	
	Конс., ppm	RSD, %	Конс., ppm	RSD, %	Конс., ppm	RSD %
Cu	43	0.9	45	1.6	NA	
Fe	1.19E+04	1.4	1.21E+04	3.4	1.12E+04	3.6
Mg	70	0.5	78	4.1	67	4.4
Zn	104	0.3	102	2.9	NA	

On-line въвеждане на вътрешен стандарт чрез смесени потоци



ФВА ЛАБОРАТОРИЯ BioSupport



Благодаря за
вниманието
и успех на
семинара !!