# РЕЗЮМЕТА НА НАУЧНИТЕ ТРУДОВЕ

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## НАУЧНА ДЕЙНОСТ

## І. РЕЗЮМЕТА НА ПУБЛИКАЦИИ В ПЕРИОДИЧНИ НАУЧНИ ИЗДАНИЯ, КОИТО СА ВКЛЮЧЕНИ В ДОКТОРСКАТА ДИСЕРТАЦИЯ

## Автореферат

**Галя Д. Генчева-Нишева** "Изследване възможностите на атомната спектрометрия за аналитичен контрол при синтеза на оксидни кристални материали". Дисертационен труд за присъждане на образователната и научна степен "доктор", ИОНХ-БАН, 2001 год.

Изследвани са възможностите на атомната спектрометрия - атомноемисионна индуктивно спектрометрия с възбуждане в свързана плазма (ICP-AES) И атомноабсорбционна спектрометрия (AAS) в пламъков и електротермичен вариант за аналитичен контрол при синтеза на монокристали от β-бариев борат, калиевогадолиниев волфрамат, литиевобариеви фосфати, калиев титанилфосфат и негови структурни аналози. Разработени са подходящи процедури за разтваряне на кристалните материали. При ІСР-AES анализа са изучени спектралните пречения около аналитичните линии на определяемите елементи в присъствие на матричните компоненти като е използвана Оконцепцията на Boumans и Vrakking и са избрани най-добрите аналитични линии за всяка матрица. Изследвани са матричните пречения и са оптимизирани условията за AAS определяне на елементи в широк концентрационен интервал в разтвори на пробите. В отделни случаи анализът е проведен посредством спектрофотометрия или титриметрия. Показана е възможността за ускоряване и опростяване на анализа чрез електротермична AAS на суспензии от кристалните материали без предварителното им разтваряне. Въз основа на проведените изследвания са разработени методи за определяне съдържанието на онечистващи, дотиращи и основни елементи в монокристали от β-бариев борат, литиевобариеви фосфати, калиевогадолиниев волфрамат, калиев титанилфосфат и негови структурни аналози. Точността на получените резултати е оценена чрез анализ на пробите по два различни аналитични метода поради липса на сертифицирани стандартни материали за проби от този тип. Възпроизводимостта на резултатите (1-6 % RSD) е напълно достатъчна за аналитичния контрол при синтеза на оксидни кристални материали.

#### А. Публикации в чужди научни списания

**2.** E. Ivanova, N. Daskalova, S. Velichkov, P. Slavova, **G. Gentscheva** (**1996**) "Determination of Dopants and Impurities in Optical Crystals of  $\beta$ -Barium Borate by Inductively Coupled Plasma Atomic Emission Spectrometry and Flame Atomic Absorption Spectrometry" *J. Anal. At. Spectrom.*, **11**, 567-570 **IF=2.545** 

## Abstract

Inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry were applied to the determination of dopants (Ce, Nd, Eu and Er) and impurities (Fe, Na, Mg and Al) in the optical crystals of  $\beta$ -barium borate dissolved in hydrochloric acid. The matrix interferences occurring in analyses carried out by means of ICP-AES and FAAS were studied. The detection limits of the analytes in a 0.8% solution of  $\beta$ -barium borate in 2 mol L<sup>-1</sup> hydrochloric acid were determined. The relative standard deviation of the obtained results lies within 2-6%.

**3.** N. Manuilov, V. Nikolov, **G. Gentscheva**, P. Peshev (**1999**) "Study of some  $K_2W_2O_7 - KGd_{1-x} Ln_x(WO_4)_2$  systems to be used for flux growth of doped KGd(WO\_4)\_2 single crystals", *J. Crystal Growth*, **196**, 181-184 **IF=1.492** 

## Abstract

High-temperature solutions of the systems  $K_2W_2O_7$ -KGd<sub>1-x</sub>Ln<sub>x</sub>(WO<sub>4</sub>)<sub>2</sub> (*x*=0.05, 0.10, 0.20 and 0.50; Ln, Er, Eu and Ho) were investigated. The solubility curves were plotted for each *x* value and each dopant and the concentration and temperature regions of crystallization of the low-temperature monoclinic phase were determined. The distribution coefficients were determined for gadolinium and for the dopants. The data obtained were compared with the distribution coefficients in the case of double doping of KGd(WO<sub>4</sub>)<sub>2</sub> with erbium and neodymium

## Б. Публикации в научни списания в България

**4. G. Gentscheva**, N. Daskalova, E. Ivanova (**1999**) "Analysis of single crystals of copper-doped double lithium-barium phosphates by means of inductively coupled plasma atomic emission spectrometry and atomic absorption spectrometry", *Bulg. Chem. Comm.*, **31**, 200-212

## Abstract

The content of the major constituents lithium, barium and phosphorus, and of the dopant in single crystals of copper-doped lithium-barium phosphates has been determined by inductively coupled plasma atomic emission spectrometry and flame atomic absorption spectrometry (spectrophotometry for phosphorus). The lack of statistical differences between the data obtained by the different methods points to the accuracy of the results. The precision of the results in between 0.5 and 5% (RSD) **5.** G. Gentscheva, E. Ivanova, I. Havezov (2000) "Determination of europium and gadolinium in single crystals of europium-doped potassium gadolinium tungstate by means of electrothermal atomic absorption spectrometry and titrimetry", *Bulg. Chem. Comm.*, **32**, 114-119

### Abstract

A rapid combined method is developed for the analytical control of the content of europium and gadolinium in single crystals of europium-doped potassium gadolinium tungstate. The crystal material is dissolved using hydrofluoric acid, sulphuric acid and hydrogen peroxide. The sum of europium and gadolinium in the sample solution is determined by titration with diethylenetriaminepentaacetic acid using Arsenazo III as the indicator, europium is determined by electrothermal atomic absorption spectrometry (ETAAS) under conditions providing a working concentration of 0.1-1 mg  $1^{-1}$ ; gadolinium is determined by the difference. The relative standard deviation for analyte contents at the mg  $g^{-1}$  level is 1% (titrimetry) and 0.6-1.2% (ETAAS). The accuracy of the results is checked by comparison with data obtained using inductively coupled plasma atomic emission spectrometry.

# В. Публикации (разписани в пълен текст с книгопис и резюме) в рецензирани сборници на научни звена или в сборници от проведени научни форуми

**6. G. Gentscheva**, A. Detcheva, I. Havezov, E. Ivanova (**2000**) "Slurry sampling ETAAS for analysis of iron impurities in optical crystals", Proc.: 4<sup>th</sup> EFS and XV<sup>th</sup> SSC, High Tatras-Podbanske, Slovakia, pp. 225-228

#### Abstract

Slurry Sampling ETAAS is a rapid, simple and reliable technique for trace analysis in hardly soluble materials. This technique was applied to the determination of iron impurities in optical crystals of potassium titanylphosphate (KTP) and potassium gadolinium tungstate (KGW). Calibration curves with aqueous standards could be used for the KTP slurry; the KGW slurry required the standard addition method. The precision of the method is about 3% RSD. The results obtained by this method showed an excellent agreement with those determined by flame AAS after sample digestion

## **II. РЕЗЮМЕТА НА ПУБЛИКАЦИИ В ПЕРИОДИЧНИ НАУЧНИ ИЗДАНИЯ,** КОИТО НЕ СА СВЪРЗАНИ С ДОКТОРСКАТА ДИСЕРТАЦИЯ.

### А. Публикации в чужди научни списания

**7.** E. Ivanova, M. Stoimenova, G. Gentcheva (1994) "Flame AAS determination of As, Cd and Tl in soils and sediments after their simultaneous carbodithioate extraction", *Fresenius J. Anal. Chem.*, 348, 317-319 IF=0.975

## Abstract

The extraction system ammonium tetramethylenecarbodithioate/isobutylmethylketone is applied to the simultaneous preconcentration of the toxic trace elements As, Cd and T1 from soil digests at an acidity of 2- 3 mol/1 sulphuric acid and to their separation from the major soil components. The extraction procedure is easily coupled to AAS. The accuracy of the method is checked by the analysis of a certified reference material. RSD is between 4 and 10%

**8.** E. Ivanova, S. Tsakovski, **G. Gentscheva**, I. Havezov (**1996**) "Anion-exchange enrichment of thallium and cadmium prior to their flame atomic absorption spectrometric determination in soils", *Talanta*, **43**, 1367-1370 **IF=1.228** 

#### Abstract

Модифицирана е процедура за едновременната сорбцията на талий и кадмий върху Dowex 1X8 (200-400 меша) като хлорни компрекси при подходяща киселиност. Количествено елуиране на Tl се постига с 2% аскорбинова киселина, а на Cd с 2 M HNO<sub>3</sub>. Малки обеми от елуатите са достатъчни за количествено елуиране на Tl и Cd. Трябва да бъде подчерта, че емуиравено на Cd трябва да предхожда Tl, в противен случай се регистрират загубите на Cd поради на неговото частично елуиране с талия. Тази техника, в съчетание с пламъковата AAC е приложена за определяне на микропримеси от талий и кадмий в български култивирани почви.

**9.** N. Daskalova, L. Aleksieva, **G. Gentsheva**, S. Velichkov (**2002**) "Analytical control of the preparation of single crystal materials by inductively coupled plasma atomic emission spectrometry", *Spectrochimica Acta* Part B **57**, 4, 755-768 **IF**= **2.695** 

#### Abstract

The present paper shows that inductively coupled plasma atomic emission spectrometry (ICP-AES) and the Q concept, in accordance with Boumans and Vrakking [*Spectrochim. Acta Part B* 43 (1988) 69] can be used in the determination of a large number of dopants with different characteristics (charge and ionic radius) in the single crystals of potassium titanylphosphate [KTiOPO<sub>4</sub>], some of its structural analogues and potassium gadolinium tungstate [KGd(WO<sub>4</sub>)<sub>2</sub>].The basic conclusion from the analytical data obtained in this work is that the incorporation of Me<sup>+</sup>, Me<sup>2+</sup>, Me<sup>3+</sup>, Me<sup>4+</sup> and Me<sup>5+</sup> ions in the crystal lattice depend on its ionic radii. The effect of the ionic charge of the dopant ions is negligible. The light on the regularities of dopant incorporation in the crystal lattice was thrown and hence on the possibilities of modifying the properties of the single crystal materials.

10. A. Detcheva, I. Havezov, G. Gentscheva, E. Ivanova (2002) "Electrothermal atomization of arsenic, antimony and thallium using a graphite atomizer with refractory metal platforms", *Annali di Chimica*, 92, 595-599 IF=0.494

#### Abstract

The electrothermal atomization of the volatile elements arsenic, antimony and thallium from a refractory metal platform consisting of a tungsten coil and/or a refractory metal foil with the dimensions of a conventional graphite platform was studied. Several combinations of refractory metal platforms were investigated, as follows: W platform; Ta platform; W coil; W coil on a W platform and W coil on a Ta platform. The best combination for these elements as regards both thermal stabilization and sensitivity is the W coil on a Ta platform. Thermal stabilization is also achieved with a W coil on a W platform. The presence of Pd-containing chemical modifier favors the thermal stabilization of the analytes. The sufficient amount is 2 micrograms of Pd. The maximal temperatures of pyrolysis are higher (arsenic, antimony) or equal (thallium) to those when using different chemical modifiers, added as solutions. It may be concluded, that the refractory metal platforms act as "built-in modifiers". They are suitable for

the determination of arsenic, antimony and thallium in samples of complex matrix composition where high thermal stability of the analytes during the pyrolysis step is required

11. N. Daskalova, G. Gentsheva, S. Velichkov (2002) "Spectral interference in the determination of Ho, Tm and Ga as dopants in single crystals of potassium titanylphosphate by inductively coupled plasma atomic emission spectrometry", *Spectrochimica Acta* Part B, 57, 8, 1351-1359 IF=2.695

## Abstract

The present article discusses the spectral interferences affecting the determination of Ga, Ho and Tm as dopants in single crystals of potassium titanylphosphate (KTiOPO<sub>4</sub>) by inductively coupled plasma atomic emission spectrometry (ICP-AES). The Q concept, as proposed by Boumans and Vrakking [Spectrochim. Acta Part B 43 (1988) 69] was used for this study, which covers: (a) spectral data for potassium, titanium and phosphorous as interferents (at a concentration of 4 mg/ml) in 400 pm wide spectral windows around the prominent lines of the analytes; (b) a database of Q values for line interferences ( $Q_I$ ) and for wing interferences ( $Q_W$ ); and (c) the interelement interferences in doubly doped crystals of KTP.

**12.** A. Detcheva, **G. Gentscheva**, I. Havezov, E. Ivanova (**2002**) "Slurry sampling ETAAS determination of sodium impurities in optical crystals of potassium titanyl phosphate and potassium gadolinium tungstate", *Talanta*, **58**, 3, 489-495 **IF=2.054** 

## Abstract

Slurry sampling ETAAS was successfully applied to the determination of sodium impurities in single crystals of potassium titanyl phosphate (KTP) and potassium gadolinium tungstate (KGW). Platform atomizers coated with titanium carbide or tungsten carbide, respectively, were used in order to avoid sensitivity drift due to the changes in the composition and the structure of the platform surface. Calibration curves with aqueous standards could be used for the KGW slurry (no matrix effects); analysis of KTP slurry required the standard additions method. The precision of the proposed method was better than 3% R.S.D. The results obtained by the present method showed a good agreement with those obtained by an independent method-flame AAS after sample digestion, which is an evidence for the good accuracy of the proposed method.

**13.** G. Gentscheva, A. Detcheva, I. Havezov, E. Ivanova (2004) "Slurry Sampling Electrothermal Atomic Absorption Spectrometric Determination of Sodium and Iron Impurities in Optical Crystals of Rubidium Titanyl Phosphate", *Microchimica Acta*, **144**, 1-3, 115-118 IF=0.851

## Abstract

A simple and rapid slurry sampling electrothermal atomic absorption spectrometric (ETAAS) method was developed for the determination of traces of sodium and iron in single crystals of rubidium titanyl phosphate (RTP). The finely ground crystal material was dispersed in 5mL of 2% (v/v) HNO<sub>3</sub>, containing 0.005% (v/v) Triton-X-100. The fast furnace ETAAS analysis was performed using a pyrolytic platform coated with titanium carbide. No matrix interference was registered for either of the analytes in slurries containing 4 to 10 mg mL<sup>-1</sup> of RTP, which permitted simple calibration against aqueous solutions. The precision was about 2%

RSD. The results for sodium and iron content in RTP were in good agreement with those obtained by flame AAS after sample digestion.

**14. G. Gentscheva**, P. Tzvetkova, P.Vassileva, L.Lakov, O.Peshev, E. Ivanova (2006) "Analytical characterization of a silica gel sorbent with thioetheric sites", *Microchim. Acta*, **156** (3-4) 303-306 IF=1.237

## Abstract.

The sorption behavior of a newly synthesized silica gel sorbent with thioetheric sites (STS) towards microgram levels of Au(III), Pt(IV) and Pd(II) was studied. Au(III) is quantitatively (>95%) sorbed in the pH region of 1–9. The sorption of Pt(IV) starts at pH 1 and does not exceed 25% in the entire pH region examined. The sorption of Pd(II) starts at pH 7 and reaches 80% at pH 9. The sorption of Au(III) on STS at pH 1 is not affected by milligram amounts of Ni(II), Zn(II), Fe(III), Cu(II), Pb(II), Cd(II) or Co(II). Au(III) is quantitatively eluted with a 5% aqueous solution of thiourea. The adsorption capacity of STS towards Au(III) is 195 mg g<sup>-1</sup>. The detection limit (DL) of Au(III) ( $3\sigma$ , n=9) is 25 ng mL<sup>-1</sup>. The RSD at a level of 10×DL is about 2%. Solid-phase extraction of trace amounts of Au(III) on the STS sorbent, followed by its flame AAS determination in the eluate was applied to the determination of gold in geological samples. The results obtained for the gold content in the samples were in good agreement with those of the ICP-AES analysis

**15. G. Gentscheva**, P. Vassileva, P. Tzvetkova, L. Lakov, O. Peshev, and E. Ivanova, (2008) "Activated carbon sorbent with thioetheric sites – preparation and characterization", *Journal of Porous Materials*, **15** (3) 331-334 **IF=0.959** 

#### Abstract

An activated carbon sorbent containing thioetheric sites (ACTS) was prepared by modification of the activated carbon with 2,2'-thiodiethanol. The specific surface area, pore volume, concentration of oxygen-containing groups and sulfur content of the sorbent were determined. The sorption behavior towards ions of some precious metals—Au(III), Pt(IV), Pd(II) and heavy metals—Ni(II), Zn(II), Fe(III), Cu(II), Pb(II), Cd(II) and Co(II) was studied. Selectivity towards gold, palladium and platinum in the pH range 1–9 was observed. The capacity for gold was 80 mg g<sup>-1</sup>. The sorption of Au(III) at pH 1 is not affected by milligram amounts of Ni(II), Zn(II), Fe(III), Cu(II), Pb(II), Cd(II) and Co(II). The sorbed gold species is Au(0).

**16.** A. Petrov, **G. Gentscheva**, I. Havezov, E, Ivanova (**2009**) "Determination of the Uncertainty of the Flame Atomic Absorption Spectrometer for Copper, Cobalt, Cadmium and Nickel", *Analytical Letters*, **42** (16) 2509-2519 **IF=1.317** 

#### Abstract

The sources of uncertainty in the determination of copper, cobalt, cadmium, and nickel, originating from the flame atomic absorption spectrometer, were studied. They were identified and divided in three main groups according to their origin—from the flame, from the light source (hollow-cathode lamp), and from the optics. The combined uncertainty of the flame atomic absorption determination was experimentally determined.

**17.** Kr. Nikolova, I. Panchev, S. Sainov, G. Gentscheva, E. Ivanova (2012)"Selected Physical Properties of Lime Bee Honey in Order to Discriminate between Pure Honey and Honey Adulterated with Glucose", *International Journal of Food Properties*, **15** (6) 1358-1368 **IF=0.877** 

#### Abstract

The refractive index, conductivity, optical rotation, thermophysical parameters, and infrared spectra of bee honey samples, as well as the content of  $\beta$ -carotene and potassium, were determined with a view to discriminate between pure honey and honey adulterated with glucose. The glass transition temperature and the optical rotation may be used for qualitative assessment of honey adulteration with glucose. Another indication of lime honey adulterated with glucose was the  $\beta$ -carotene content below 20 ppm and the potassium content below 200 ppm. The minimum detectable amount of glucose adulterant was 25 g in 100 g honey.

**18.** St. Gyurov, N. Marinkov, Y. Kostova, D. Rabadjieva, D. Kovacheva, Ch. Tzvetkova, G. Gentscheva, I. Penkov (2017) "Technological scheme for copper slag processing", *International Journal of Mineral Processing*, **158**, 1-7 **IF=1.617** 

#### Abstract

A technological scheme for copper slag processing is proposed. It comprises 5 stages, namely: (i) air oxidation of the copper slag at a temperature above 800 °C for 2 h; (ii) hydrothermal treatment of the oxidized slag with sodium hydroxide solution (140 g/l) at 190°C for 3 h; (iii) separation of the solid from the liquid phase by hot filtration; (iv) gel formation through hydrolysis of the liquid silicate phase by changing pH; (v) obtaining of amorphous SiO<sub>2</sub> (silica gel) by drying at 80°C. The processes used for slag manipulation were elucidated and optimized for silicon extraction. It was established that the increase in the oxygen partial pressure in the oxidizing gas does not change the mechanism nor significantly intensifies the oxidizing process. A decisive factor for the extraction of SiO<sub>2</sub> during hydrothermal treatment was the concentration of NaOH. Its increase from 60 to 140 g/l reduced the amount of residual SiO<sub>2</sub> more than half and significantly decreased the formation of analcime (NaAlSi<sub>2</sub>O<sub>6</sub>·H<sub>2</sub>O) in the solid phase. Hydrolysis of the liquid silicate phase by changing pH is an appropriate process for gel formation.

**19.** M. O. Marychev, I. Koseva, **G. Gencheva**, R. Stoyanova, R. Kukeva, V. Nikolov, "Cr doped  $Ca_2GeO_4$ ,  $Ca_5Ge_3O_{11}$  and  $Li_2CaGeO_4$  single crystals grown by the flux method", *Journal of Crystal Growth*, **461**, 46–52 (**2017**) **IF=1.462** 

#### Abstract

Pure and Cr doped  $Ca_2GeO_4$ ,  $Ca_5Ge_3O_{11}$  and  $Li_2CaGeO_4$  single crystals were grown by the flux method. The  $Ca_2GeO_4$  crystals were grown at significantly more proper conditions than used up to now, while  $Ca_5Ge_3O_{11}$  and  $Li_2CaGeO_4$  single crystals were grown for the first time. The macroscopic defects are characterized and discussed in relation to the growth conditions. The distribution coefficients of Cr were established being very different for the three crystals. EPR investigation shows that as in the case of  $Ca_2GeO_4$ , in  $Ca_5Ge_3O_{11}$  and  $Li_2CaGeO_4$  the Cr occupied the tetrahedral position. The Cr doped crystals show broad band emissions from 1000 to 1600 nm. The dependence of the integrated intensity of emission on the Cr concentration in the crystals is discussed.

#### Б. Публикации в български научни списания

**20.** E. Ivanova, **G. Gentscheva**, M. Stoimenova, I. Havezov (**1995**) "Atomic absorption determination of arsenic, cadmium and thallium in cultivated soils", *Analytical Laboratory*, **4**, 14-17

#### Abstract

The total and the pseudototol of As, Cd and Tl in various representatives of cultivated Bulgarian soils are determined by AAS after sample digestion using appropriate acid mixtures and separation/preconcentration of the analyzed elements from the matrix. The total contents of As, Cd and Tl exceed their mean natural abundances in soils, which is related to the prolonged agricultural treatment of these soils with fertilizers, pesticides, etc. The pseudototal contents of As, Cd and Tl are within 75-90% from the total one. The method is characterized by an RSD of 4-10%.

**21.** E. Ivanova, **G. Gentscheva**, M. Stoimenova, I. Havezov (**1996**\**97**) "Atomic absorption spectrometric determination of arsenic, cadmium and thallium in couples of atmospheric particulate matter and soil collected from the same spot", *Bulg. Chem. Comm.* **29**, 117-122

#### Abstract

The content of the toxic pollutants As, Cd and Tl has been determined in pairs of atmospheric particles and soil collected from several spots in Bulgarian. The AAS determination has been performed after sample digestion with *aqua regia*, The content of elements in the atmospheric particles has been found to be higher than in the corresponding soil samples, They were however, far below the threshold levels prescribed in the Bulgarian legislation for admissible emissions of harmful substances in the atmosphere. The method is characterized by an relative standard deviation of 4-10%

22. E. Ivanova, G. Gentscheva (2000) "Determination of platinum metals in environmental and biological samples" (a review), *Bulg. Chem. Comm.*, 32, 191-201

#### Abstract

The determination of trace amounts of platinum metals (PMs) is of marked interest for geochemical prospecting, medicine and recently for environmental research. The latter is related to the use of car exhaust catalysts containing PMs, which leads to gradual environmental pollution with these metals. The data on the environmental pollution with PMs in Europe and USA are reviewed. An overview is given of the analytical methods used for PMs determination in geological, environmental and biological samples. To this purpose spectroscopic, radiochemical and electrochemical techniques are employed, which are most often coupled with preconcentration and separation procedures.

**23.** Г. Генчева, С. Величков (2001) "ICP-AES анализ на оптични монокристали от калиево гадолиниев волфрамат и β-бариев борат", Пловдивски университет "Паисий Хилендарски", *Научни трудове*, **30**, 5, 35-38

#### Abstract

Optical crystals of potassium gadolinium tungstate /KGW/ and  $\beta$ -barium borate /BBO/ doped with rare earth elements are analysed by means of ICP-AES. The spectral interferences are studied using the Q-concept of Boumans and Vrakking. The detection limits of Nd, Ho and Er in KGW are determined. The analytical lines for the determination of the major component gadolinium in KGW are chosen. The precision of the analysis is characterized by 1-6% RSD.

**24. G. Gentscheva**, E. Ivanova, J. Jordanov, V. Petrova (**2003**) "Use of atomic spectrometry for the determination of Li, Na, K and Al in novel optical materials based on double alkalialuminium tungstates, *Bulg. Chemistry and Industry*, **74**, 73-75

#### Abstract

Double alkali–aluminium tungstates are promising new materials for the preparation of tuneable solidstate lasers. With a view to identifying the phases crystallising from the ternary oxide system  $M_2O-Al_2O_3-WO_3$ , where M is Li, Na or K, the content of the corresponding alkali metal and Al in the single crystals of double alkali–aluminium tungstates was determined by means of flame atomic absorption spectrometry (FAAS) and X-ray fluorescence spectrometry (XRF). As the solubility of the crystal materials decreases on increasing their tungsten content, some of them were dissolved in water, others – in acid mixture, or sodium or potassium tetra borate melt was used. Under the optimum conditions of the FAAS analysis, no matrix interference was registered. The XRF analysis of the solid samples was carried out after pelletising. WO<sub>3</sub>- based standards were used for calibration. The precision of the obtained results is characterised by an RSD of 1.5–4% for FAAS and 4–7% for XRF. Their accuracy was confirmed by the good agreement with the data from the comparative microprobe analysis. Based on the analytical information the phases crystallising in the system M<sub>2</sub>O–Al<sub>2</sub>O<sub>3</sub>–WO<sub>3</sub>, (M = Li, Na or K) were identified.

**25.** G. D. Gentscheva, E. H. Ivanova (2003) "Flame atomic absorption spectrometric determination of chromium dopants in optical crystals of sodium aluminium tungstate", *Bulg. Chem. Comm.*, 35, 4, 224-226

#### Abstract

Flame atomic absorption spectrometry (AAS) was applied to the determination of chromium dopants in single crystals of sodium aluminium tungstate ((NaAl(WO<sub>4</sub>)<sub>2</sub>). The single crystals of NaAl(WO<sub>4</sub>)<sub>2</sub> were dissolved by means of hydrofluoric acid, sulphuric acid and hydrogen peroxide. Owing to matrix effects on the absorbance of chromium, calibration by standard additions was performed. The detection limit ( $3\sigma$ ) of chromium in the crystal material is 17 µg.g<sup>-1</sup>. The precision of the analysis of single crystals doped with different amounts of chromium is characterized by a relative standard deviation (RSD) of 2–8%. The accuracy of the obtained results was checked by comparison to those of an independent method – alternating current (AC) arc–atomic emission spectrometry (AES) with photographic registration. The analytical results pointed to a constant distribution coefficient of chromium in a broad

concentration range (0.24 to 2.17 at. % Cr/Al), which is an important characteristic of optical single crystals, testifying their homogeneity

**26. G. Gentscheva**, J. Jordanov, E. Ivanova, V. Petrova, L. Vladeva (**2005**) "X-ray fluorescence spectrometric determination of trace amounts of Bi, Sb, Co, Ni, Pb, Cu, Fe and Zn in mineral waters after precipitation with ammonium pyrrolidinedithiocarbamate", *Bulg. Chem. Comm.*, **37**, 69-72

## Abstract

A fast and efficient X-ray fluorescence spectrometric method is developed for the determination of micro trace amounts of Bi, Sb, Co, Ni, Pb, Cu, Fe and Zn in mineral waters after precipitation with ammonium pyrrolidinedithiocarbamate. The metal dithiocarbamates are filtered through a Millipore filter of 0.22  $\mu$ m pore size. The detection limits are from 0.05 to 4.75  $\mu$ g l<sup>-1</sup> depending on the analysed element. Waters with total degree of mineralization of up to 0.25 g l<sup>-1</sup> can be analyzed by direct calibration, while those with higher matrix content require calibration by standard additions. The accuracy of the method is checked by spike recovery from the mineral water and by analysis of synthetic fresh water (NIST SRM 1643d). The RSD of the results at a level of approx. 10×DL is 3–5%. The simple preconcentration procedure may be applied in mobile laboratories with subsequent XRF detection

**27.** Kr. Nikolova, I. Panchev, S. Sainov, G. Gentscheva, E. Ivanova (2008) "Physical and chemical characteristics of South Bulgarian bee honeys", *Bulgarian Chemistry and Industry* **79**, 26–30

## Abstract

The quality of six bee honey varieties harvested from different locations in South Bulgaria was assessed by several physical and chemical parameters: refractive index, electric conductivity, viscosity, color, pH, water content, sugar,  $\beta$ -carotene, some essential and toxic trace elements. Obtained results reveal that examined South Bulgarian bee honeys are a high-quality product with characteristics close to those of bee honeys from different European countries.

**28**. A. Petrov, **G. Gentscheva**, I. Havezov, E. Ivanova (**2009**) "Instrumental Contribution to Uncertainty Measurement in Flame Atomic Absorption Spectrometry", *Compt. rend. Acad. bulg. Sci.*, **62** (10) 1235-1240 **IF=0.204** 

## Abstract

The sources of uncertainty in the flame atomic absorption determination of copper, cobalt, cadmium and nickel, originating from the spectrometer, were studied. They were identified and divided in three main groups according to their origin – from the flame, from the light source (hollow-cathode lamp) and from the optics. The combined uncertainty of the flame atomic absorption determination was experimentally determined.

**29.** P. Vassileva, **G. Gentscheva**, E. Ivanova, P. Tzvetkova, D. Voykova, M. Apostolova, (**2011**) "Characterization of natural diatomites from Bulgaria", *Compt. rend. Acad. bulg. Sci.*, **64** (6) 823-830 **IF=0.210** 

#### Abstract

Several characteristics of natural diatomites from Dragovishtitsa and Ignatievo deposits (Bulgaria) such as chemical composition, surface area, pore volumes, XRD and IR spectra were studied. The sorption properties of the diatomites towards  $Fe^{3+}$ ,  $Pb^{2+}$ ,  $Cu^{2+}$ ,  $Cd^{2+}$ ,  $Tl^+$ ,  $Mn^{2+}$ ,  $Ni^{2+}$  and  $Co^{2+}$  were investigated and the sorption capacities towards  $Fe^{3+}$ ,  $Pb^{2+}$ ,  $Cu^{2+}$  and  $Cd^{2+}$  ions were determined. The natural Bulgarian diatomites from Dragovishtitsa and Ignatievo were found as suitable low cost sorbents for the removal of milligram amounts of  $Fe^{3+}$ ,  $Pb^{2+}$ ,  $Cu^{2+}$  and  $Cd^{2+}$  from contaminated waters.

**30. G. Gentscheva**, A. Petrov, E. Ivanova, I. Havezov (**2012**) "Flame AAS determination of trace amounts of Cu, Ni, Co, Cd and Pd in waters after preconcentration with 2-Nitroso-1-Naphthol", *Bulg. Chem. Comm.*, **44** (1) 52-56 **IF=0.320** 

#### Abstract

Flame AAS was applied to the determination of micro trace amounts of Co, Ni, Cu, Cd and Pd in waters after precipitation of their complexes with 2-nitroso-1-napthol. The precipitate was separated from the sample solution by filtration through Millipore filter of 0.22  $\mu$ m pore size under suction. It was dissolved with a minimum amount of ethanol and was subjected to flame AAS analysis. The detection limits of Co, Ni, Cu, Cd and Pd were 0.61, 0.64, 0.89, 0.10 and 0.60  $\mu$ g l<sup>-1</sup>, respectively. The method was validated using the reference material SPS-WW2 – Spectrapure Standards, Norway, and was applied for analysis of capture water and waste water.

**31.** Kr. Nikolova, **G. Gentscheva**, E. Ivanova (**2013**) "Survey of the mineral content and some physico-chemical parameters of Bulgarian bee honeys", *Bulg. Chem. Comm.*, **45** (2) 244-249 **IF=0.349** 

#### Abstract

The quality control of honey requires a number of physical and chemical parameters to be determined in order to provide evidence of the origin and environmental purity of the product. 14 types of Bulgarian bee honeys were analyzed and the following parameters were determined: refraction index, thermophysical characteristics, color characteristics, lightness  $L^*$  and chroma  $C^*_{ab}$ . The contents of water,  $\beta$ -carotene, glucose, fructose, saccharose, oligosaccharides, essential and toxic trace elements were also found. The correlation between the refractive index and the water content of honey was determined as a criterion for the quality of honey. The fructose-glucose ratio was determined as a parameter related to the crystallization of honey. The criteria used were applied for the first time to Bulgarian honeys.

Relatively high content of potassium was found in the analyzed Bulgarian bee honeys, which makes them an important source of this essential element. No traces of the toxic elements As, Cd, Ni and Pb were found.

**32. G. Gentscheva**, I. Uzunov, I. Karadjova, A. Predoeva (**2014**) "Inorganic components, IR, XRD and TG/DTA characterization of *Triticum monococcum* L. and modern cultivated cereals", *Compt. rend. Acad. bulg. Sci.*, 67 (5) 647-654 **IF=0.284** 

#### Abstract

In this study mineral content (K, Mg, Ca, Zn, Fe, Mn, Na, Cu, Al, Ba, Sr, B, V, Mo, Ni, Co, Cr, Cd, Pb, As, Hg, Tl) of different einkorn samples *Triticum monococcum*, limez (in Bulgarian) grown in Eastern Rhodope mountains and local varieties of cultivated crops: wheat and barley was determined and discussed from the view point of healthy eating. Additionally ash content of the hulls, and phase composition of the ashes were defined using IR, XRD and TG/DTA analysis.

**33. G. Gentscheva**, I. Karadjova, Dr. Buhalova, A. Predoeva, Kr. Nikolova, I. Aleksieva (**2014**) "Determination of essential and toxic elements in berries from Bulgaria (Plovdiv region)", *Compt. rend. Acad. bulg. Sci.*, 67 (9) 1241-1248 **IF=0.284** 

#### Abstract

The objective of this study was to investigate the level of 17 trace elements in wildharvested fruits (*Crataegus monogyna, Cornus mas, Vaccinium vitisi-daea* and *Vaccinium myrtillus*) grown in Plovdiv region in order to define their significance for healthy nutrition. The essential (Mg, Mn, Fe, Zn, Cu, Co and Se), toxic elements (Pb, Cd, Hg, As and Tl) and Cr, Ni, Mo, Sr and Ba contents were determined as total amount in dried fruits, as well as bioavailable fraction (Mg, Fe, Mn, Zn) in fresh fruits. The trace elements in each dried fruit sample were determined by ICP-MS and FAAS after microwave digestion. The toxic trace element analysis demonstrated that their content was safe for human consumption. The content of bioavailable fraction especially for essential elements is another important aspect for food evaluation for healthy nutrition. *Vaccinium vitis-idaea* and *Vaccinium myrtillus* are very good suppliers of Mn, around 100 g of fruits ensure 50–60% of its daily allowance. Berries are relatively good sources of Fe 5–10%; Zn 1–3% and Mg 1.5–4.0% as well.

**34.** A. Chatzis, P. Tzvetkova, L. Manoilova, **G. Gentscheva**, R. Nickolov (**2016**) "Modified activated carbons as materials for a decontamination of Tl poisoned water", *International scientific journal Science. Business. Society.*, **5**, 18-21

#### Abstract

Removal of large monovalent cations, as highly toxic thallium (Tl), from the waters is a subject of significant interest due to the hazards its pose. Active materials on the basis of activated carbons intended for removal of Tl ions from drinking water was synthesized and characterized in two stages. During the first, deposition and stabilization of the Fe(3+) phase in the internal surface of activated carbon samples (AC/Fe(3+)) was carry out. During the second, deposition on the AC/Fe(3+) of K<sub>4</sub>[Fe(CN)<sub>6</sub>] phase and subsequent chemical reaction were realized. The removal performance of the samples prepared for Tl ions in aqueous solution was investigated by adsorption process. Increased sorption possibilities were observed toward Tl ions as compare to initial carbons.

**35.** G. Gentscheva, A. Petrov, I. Havezov, E. Ivanova (2016) "Uncertainty of FAAS determination of Co, Ni, Cu and Cd in waters after preconcentration with 2-nitroso-1-naphthol", *Ann. Sofia Univ., Fac. Chem. and Pharm.*, 107/108, 55-63

#### Abstract

A procedure for estimating the measurement uncertainty of flame AAS determination of Cu, Co, Ni and Cd in waters, after their simultaneous separation and preconcentration with 2-nitroso-1-naphtol, was developed. The concentrations of Ni, Cu and Co were determined using linear calibration and that of Cd – by standard additions. The procedure was based on the ISO GUM approach. The mathematical model was defined and the uncertainty sources were identified and evaluated. The uncertainty budget of the proposed method was very good – the relative uncertainty was less than 10%

## ПУБЛИКАЦИИ (РАЗПИСАНИ В ПЪЛЕН ТЕКСТ С КНИГОПИС И РЕЗЮМЕ) В РЕЦЕНЗИРАНИ СБОРНИЦИ НА НАУЧНИ ЗВЕНА ИЛИ В СБОРНИЦИ ОТ ПРОВЕДЕНИ НАУЧНИ ФОРУМИ

**36.** A. Detcheva, I. Havezov, **G. Gentscheva**, E. Ivanova (**2003**) "Studies on the ETAAS determination of thallium in different matrices using a graphite atomizer with a refractory metal platform", Proc. 3-rd International conference "Instrumental methods of analysis modern trends and applications, pp. 405- 408, 23-27 September, Thessaloniki, Greece

#### Abstract

The electrothermal atomization of thallium in different matrices from a refractory metal platform consisting of a tungsten coil on tantalum foil with the dimensions of a conventional graphite platform was studied. Two chemical modifiers - ammonium tungstate, added as a solution or iridium as a permanent modifier were used. Best performance as regards both thermal stabilization and sensitivity was obtained with iridium as a permanent modifier. This platform proved to be particularly appropriate for the analysis of thallium in refractory matrices such as tungstate and borate which destroy the conventional pyrolytic graphite platform by formation of refractory carbides of the matrix components.

**37.** Y. I. Kouzmanova, I. V. Dimitrova, **G. D. Gentscheva**, L. I. Aleksandrov, M. G. Markova-Velichkova, D. G. Kovacheva (**2015**) "Comparative study of the phase formation and interaction with water of calcium-silicate cements with dental applications", *Bulg. Chem. Comm.*, 47 (1) 239–244 **IF=0.229** 

#### Abstract

Mineral trioxide aggregate (MTA) is based on plain Portland cement and is composed of tricalcium silicate, dicalcium silicate, tricalcium aluminate, and tetracalcium alumoferrite. This cement is used for various dental clinical applications. The degree of solubility of calcium-silicate based cements is an object of debate among investigators. The present study was designed to compare the phase formation and evolution, as well as solubility of five different commercial calcium-silicate cements. X-ray diffraction (XRD) analysis was applied to determine the phase composition of the initial powder mix, cement compositions, and cement compositions evolution after aging the material in distilled water. Thermal analyses (TG-DTA) were also performed to confirm the XRD results. The concentrations of Ca, Na, Mg, Al, K, Fe, Ti, Bi, Ta

and Zr ions passed into the solution were determined by means of ICP-MS and FAAS. The changes of elemental and phase composition were discussed. Biodentine seems an alternative to MTA. It releases significant amounts of Ca ions and therefore stimulates tissue mineralization. The concentration of radiopaquer elements in the soaking water is at trace levels in all materials studied, which makes them safe for dental applications. The obtained good results do not cancel regular safety checks.

**38.** G. Uzunova, Kr. Nikolova, M. Perifanova, G. Gentscheva, M. Marudova, G. Antova, (2016) "Physicochemical characterization of chia (*Salvia hispanica*) seed oil from Argentina", *Bulg. Chem. Comm.*, **48**, *Special Issue G*, 131 - 135 **IF=0.229** 

## Abstract

The physicochemical characteristics of chia oil from Argentina, which is one of the most efficient omega-3 (n-3) sources for enriching foods, have been studied. The results from analysis show that the chia oil has a relative density of 0.9288, refractive index 1.4810 and yellow color component that dominates over the red one. Its acidity index is 1.68 mg KOH/g, its saponification index is 197.9 mg KOH/g, iodine index is 208.3 g I<sub>2</sub>/ 100g and the peroxide index is 1.95 meq O<sub>2</sub>/kg. The fluorescence spectra for excitation wavelength 350 nm contain 3 peaks at about 472 nm, 503 nm and 670 nm, which are attributed to pigments, vitamins and oxidation products. Besides, the spectra in visible and UV range are used for determination of chlorophyll content, content of  $\beta$ -carotene, oxidation products and oxidant stability. Phase transition is observed at - 36.9 °C. The content of some essential, non essential and toxic elements in the solution obtained after microwave-assisted (MW) digestion of the examined oil were determined by Inductively Coupled Plasma Mass Spectrometry (ICP-MS). This method could be useful for quality control of the oil when used in food industry, medicine and cosmetics

## СПИСЪК НА РЕЗЮМЕТА ОТ НАУЧНИ ФОРУМИ В ЧУЖБИНА (СВЪРЗАНИ И НЕ СВЪРЗАНИ С ДОКТОРСКАТА ДИСЕРТАЦИЯ) ПУБЛИКУВАНИ В СЪОТВЕТНИТЕ СБОРНИЦИ С РЕЗЮМЕТА

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Congress of Chemists and Technologists of Macedonia, pp. 131, 8-11 October, 2014, Ohrid, Macedonia

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## СПИСЪК НА РЕЗЮМЕТА ОТ НАУНИ ФОРУМИ В БЪЛГАРИЯ (СВЪРЗАНИ И НЕ СВЪРЗАНИ С ДОКТОРСКАТА ДИСЕРТАЦИЯ) ПУБЛИКУВАНИ В СЪОТВЕТНИТЕ СБОРНИЦИ С РЕЗЮМЕТА

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