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СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ

20 Май 2015 г.



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АСМ2 ЕООД е изключителен представител за България на **Thermo Scientific** - част от **Thermo Fisher Scientific**, водещ световен производител на инструментална лабораторна техника. Основните групи продукти на **Thermo Scientific**, са:

- ✓ **Хроматографска апаратура:** Газхроматографски (GC), Йоннохроматографски (IC) и Високоэффективни течно-хроматографски системи (HPLC) с различни конфигурации. Хроматографски колони и консумативи, системи за пробоподготовка и аксесоари;
- ✓ **Масспектрометрия:** Масспектрометри за хроматографски апарати (GC/MS и LC/MS), магнитносекторни, изотопни и мултиколекторни масспектрометри;
- ✓ **Апаратура за спектрален анализ:** UV/VIS спектрофотометри, Инфрачервени и Раман спектрометри;
- ✓ **Апарати за елементен анализ:** Атомно-абсорбционни спектрометри, ICP и ICP/MS спектрометри за определяне на елементен състав в различни матрици. Елемент анализатори, ЯМР спектроскопи, NanoDrop спектрофотометри;
- ✓ **Рентгено-структурен анализ:** XRF, XRD и OES;
- ✓ **Лабораторни информационни системи, LIMS.**

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АСМ2 ЕООД е представител за България и на следните фирми:

- **Erweka GmbH** - Тестери за фармацевтичен анализ и развойна дейност;
- **Elga LabWater** - Системи за пречистване на вода;
- **PEAK Scientific** - Газ генератори за азот, водород и въздух;
- **FEI** – Сканиращи и трансмисионни електронни и светлинни микроскопи;
- **LabTech** - Общо лабораторно оборудване;
- **Milestone Srl** - Микровълнови системи за пробоподготовка и живачни анализатори;

За времето на съществуването си фирмата се утвърди като един от основните доставчици на лабораторна аналитична апаратура за различни контролни, научно-изследователски, токсикологични и заводски лаборатории.

От началото на 2004 г. фирма АСМ2 ЕООД притежава сертификата за управление на качеството на предлаганите услуги съгласно изискванията на стандарт ISO 9001:2008.

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СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ

ПРОГРАМА:

8 ⁴⁵ – 9 ¹⁵	Регистрация
9 ¹⁵ – 09 ³⁰	Откриване на семинара <i>проф. Запрян Козлуджов, РЕКТОР на ПУ „П. Хилендарски”</i> <i>Андон Минков, АСМ2 ЕООД;</i> <i>доц. Веселин Кметов, ПУ „Паусий Хилендарски”</i>
09 ³⁰ – 10 ¹⁵	Нова Европейска директива за анализ на приоритетни вещества и специфични замърсители във води <i>Michal Godula, Thermo Fisher Scientific</i>
10 ¹⁵ – 10 ⁴⁵	Кафе пауза
10 ⁴⁵ – 11 ³⁰	Orbitrap базирани високоразделителни маспектрометри за рутинен анализ на многокомпонентни смеси и следови количества <i>Michal Godula, Thermo Fisher Scientific</i>
11 ³⁰ – 12 ¹⁵	Стандартизирани методи за анализ на води чрез Йонна хроматография <i>Roman Repas, Thermo Fisher Scientific</i>
12 ¹⁵ – 13 ¹⁵	Обяд
13 ¹⁵ – 14 ⁰⁰	Thermo Scientific Dionex Системи за пробоподготовка при анализ на храни и води <i>Roman Repas, Thermo Fisher Scientific</i>
14 ⁰⁰ – 14 ⁴⁵	Определяне на метали в храни и водни проби. Иновативни продукти на Milestone и съвети за подобряване процедурите за пробоподготовка <i>Giulio Colnaghi, Milestone Srl.</i>
14 ⁴⁵ – 15 ¹⁵	Кафе пауза
15 ¹⁵ – 15 ⁴⁵	Комбинирани методи за следови елементен анализ, базирани на съвременни екстракционни системи <i>доц. Виолета Стефанова, ПУ „Паусий Хилендарски”</i>
15 ⁴⁵ – 16 ¹⁵	Fitness for purpose - валидиране на методи за аналитичен контрол <i>доц. Веселин Кметов, ПУ „Паусий Хилендарски”</i>
14 ⁴⁵ – 17 ⁰⁰	Постерна сесия
17 ⁰⁰ –	Коктейл

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Организационен комитет:

Доц. д-р Веселин Кметов – Химически факултет ПУ

Д-р Христо Йорданов – АСМ2

Доц. д-р Виолета Стефанова – Химически факултет ПУ

Гл. ас. д-р Кирил Симитчиев – Химически факултет ПУ

Научна комисия :

Председател:

доц. д-р Мария Стоянова Зам. Ректор на ПУ

Членове:

Проф. д-р Магдален Златанов – Р-л катедра ХТ

Доц. д-р Кирил Гавазов – Р-л катедра ОНХМХ

Доц. д-р Веселин Кметов – Р-л катедра АХКХ

Доц. д-р Стела Статкова-Абегхе – Р-л катедра ОХ

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**СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015**

EU DIRECTIVE FOR WATER BODIES – KEY CHALLENGES AND INDUSTRY RESPONSE

Michal Godula, Ph.D.

*Manager, Special Solutions Center, Thermo Scientific,
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The European Water Framework directive is a directive which commits all member states to actively control and monitor all the water bodies in the various member states on a large list of environmental contaminants. The chosen contaminants and the desired levels are based on their toxicity and there are set limits for groundwater, surface water, marine water, shellfish water, drinking water and bathing water. The surface water directive is handling all rivers, streams, lakes and all these water bodies that are connected will be regarded as one water basin, in many cases crossing borders over various member states. The compound levels are expressed as Environmental Quality Standard or EQS and the annual average (AA) and the Maximum Allowable Concentration (MAC) is given.

In addition, the proposal (COM(2011)876) for the new limits for contaminants and the new analytes will be discussed; this will amend the 2008/105/EC directive. The new proposal contains even lower limits for some of those contaminants.

The presentation will summarize current situation in the analytical methodologies applied in various labs to fulfill the regulatory needs and requirements. Analytical aspects of water samples preparation, analysis with different chromatographic and mass spectrometric techniques as well as data evaluation and interpretation will be discussed.



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ПЛОВДИВ, 20.05.2015

STREAMLINING ROUTINE MULTI-CONTAMINANT AND RESIDUE ANALYSIS BY UHPLC-HRMS

Michal Godula, Ph.D.

Manager, Special Solutions Center, Thermo Scientific,

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The occurrence of pesticide residues, mycotoxins and other emerging chemical substances in foods has become a major concern both from public as well as regulatory agencies around the world. Effective monitoring of the large groups of possible contaminants and residues is therefore required. Although in most existing studies, multi-analyte methods were employed, typically only one group of the above hazardous compounds was targeted.

In the recent years the high resolution accurate mass spectrometry has gained popularity and started replacing traditional MS/MS technology. The main reasons are simple use, high analyte coverage and possible screening, quantitation and confirmation in one run. In the presentation, the principles of Orbitrap mass spectrometry will be discussed. The example of a generic method for analysis of more than 350 pesticide residues, mycotoxins and pyrrolizidine alkaloids will be given.

The method employs ultra-high performance liquid chromatography coupled with tandem high resolution mass spectrometric detection (UHPLC–HRAM-MS/MS) employing quadrupole-Orbitrap hybrid mass spectrometer Q Exactive™. Advanced data dependent MS/MS algorithm was used to trigger automatically MS/MS acquisition based on the fullscan MS survey scans. This allowed simultaneous screening, quantitation and confirmation of all analytes within a single analytical run.

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ПЛОВДИВ, 20.05.2015**

Standard methods for water analysis by Ion Chromatography

Roman Repas, Sales Manager,
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Drinking water quality is a universal health concern with global impact. The water discharged by municipal waste water treatment plants and industrial facilities must be monitored to ensure strict compliance with global regulatory requirements. These agencies have developed standards for water analysis to assure that the community is consuming only safe drinking water. In Europe, the Drinking Water Directive provides the essential quality standards. These quality standards were developed using guidelines from the World Health Organization (WHO) and the European Commission's Scientific Advisory Committee. Contaminant levels in drinking water are continuously subject to reassessment by the above regulatory bodies, both in regard to revised levels, as well as the addition of new contaminants to the list of existing regulated substances.

Ground and surface waters are a vital resource for a healthy environment. They are also the largest source of fresh water. These waters can comprise complex matrices that interfere with detection of analytes of interest. Another major challenge for qualifying drinking water is the analysis of disinfection byproducts (DBP). Drinking water is treated with disinfectants to remove potentially harmful bacteria. These disinfectants also react with ions and residual organic matter resulting in the formation of DBPs. DBPs are highly toxic, are regulated, and require mitigation of their concentrations prior to distribution of treated water.

Thermo Scientific™ Dionex™ ion chromatography (IC) instruments are used to provide solutions for environmental water testing for a wide range of regulated and emerging inorganic ions and organic compounds. Innovative techniques were developed to overcome common challenges in the analysis of drinking water contaminants and DBPs.

The presentation will summarize standard methods of water analysis by ion chromatography. Examples of the separation of anions and cations will be presented.

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**СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015**

Thermo Scientific Dionex Sample Preparation products for food and water analysis

Roman Repas, Sales Manager,
Alternate Channels, Thermo Scientific,
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Contaminants may originate from agricultural sources, such as pesticides, animal growth hormones, or antibiotics; environmental sources, such as water and air pollutants; or from food production processes, via contamination or adulteration. Food contaminants may cause acute effects ranging from mild rash, light headache, or vomiting to more serious diseases like cancer, auto-immune diseases, thyroid problems, and even death. Health effects of exposure to contaminants may become apparent only after years of prolonged exposure.

Food contaminant analysis poses several challenges. Usually, the contaminant must be isolated from the food matrix before it can be analyzed. The proper separation technique, medium, and mobile phase/gradient must be identified, depending on the suspected contaminants. Finally, a detection method sensitive to the suspected contaminants must be used.

The presentation will give the overview of Thermo Scientific Dionex sample preparation products. The method of Accelerated Solvent Extraction will be explained, ASE-150 and ASE-350 instruments will be introduced. Examples of extraction of various contaminants from food samples will be presented.

AutoTrace 280 instrument for automated solid phase extraction of large volume of liquid samples will be introduced. Examples of the extraction of organochloride pesticides, PAHs and phtalates from water samples will be presented.

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ПЛОВДИВ, 20.05.2015

METALS DETERMINATION IN FOOD AND WATER SAMPLES. MILESTONE'S INNOVATIONS AND GUIDELINES TO IMPROVE YOUR SAMPLE PREPARATION PROCEDURE

Giulio Colnaghi

Area Manager, Milestone Srl, e-mail: g.colnaghi@milestonesrl.com

Milestone has been widely recognized as the global leader in metals prep technology for the past 26 years. Committed to providing safe, reliable and flexible platforms to enhance your lab's productivity, customers worldwide look to Milestone for their metals digestion, mercury, organic extractions, analysis and clean chemistry processing needs.

The aim of the presentation is to give clear guidelines about the main parameters required to fully digest food and water samples and to address to a real solution for your needs. Power, number of vessels are important arguments to be considered, but not the only ones, sample type, temperature, pressure and acid mixture are key parameters to be considered.

Milestone's next generation rotor-based digestion system, Ethos UP, continues to improve in areas that are critical to today's lab – safety, throughput and connectivity. An all stainless steel construction accommodates the highest throughput rotors and includes an industry first – SafeVIEW - an integrated safety camera. EasyCONTROL software includes 300 pre-set digestion methods, which virtually eliminates method development. Additionally, our Milestone Connect web app offers remote system control, 24/7 technical support and direct access to a comprehensive library of content developed especially for lab professionals.

Alongside of the new Ethos UP, Milestone offers a revolutionary alternative to traditional closed vessel digestion, the UltraWAVE! It offers industrial and research labs greater digestion capability, at least double the sample throughput, improved workflow and significantly lower operating costs—all with a single, easy-to-use instrument.

The new Ethos UP and UltraWAVE are perfect solutions for food and environmental labs, which are looking to increase the throughput and the digestion quality.



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КОМБИНИРАНИ МЕТОДИ ЗА СЛЕДОВИ ЕЛЕМЕНТЕН АНАЛИЗ, БАЗИРАНИ НА СЪВРЕМЕННИ ЕКСТРАКЦИОННИ СИСТЕМИ

В. Стефанова^{1*}, К. Симитчиев¹, Д. Георгиева¹, Е. Върбанова¹, В. Кметов¹,
N. Kovachev², M. Hidalgo², A. Canals²

¹ Пловдивски университет „П. Хилендарски“, Катедра Аналитична химия и компютърна химия

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Определянето на следови съдържания от елементи в сложни матрици с променлив състав често изисква създаване на методи, съчетаващи предварителна обработка на пробите с високо-чувствителни инструментални методи за анализ.

Изборът на адекватна процедура за разделяне и концентриране на микроелементи и техни форми на присъствие в екологични обекти, храни и козметика пряко влияе върху точността, прецизността и границите на откриване на аналитичното измерване.

Представен е изследователският опит на екипа в областта на съвременните тенденции в развитието на екстракционни системи за следови елементен анализ, с акценти върху миниатюризация, автоматизация, висока производителност и намаляване консумацията на енергия, реактиви и проба в хода на аналитичната процедура.

Критично са оценени предимствата и ограниченията на три от водещите направления в методите за разделяне и концентриране като: Течно-течна микро-екстракция (екстракция в единична капка – SDME и дисперсивна екстракция - DLLME); Екстракция при температура на коагулация (CPE) и Твърдофазна екстракция (SPE).

Подобрена е ефективността на екстракционните процеси чрез агитация с микровълново лъчение при CPE и SPE.

Коментирани са предимствата и ограниченията на магнитни наночастици, като нови сорбенти за групова твърдофазна екстракция. Представени са аналитичните характеристики на екстракционни системи, комбинирани с различни инструментални методи като: FAAS, ETAAS, LIBS, ICP-OES и ICP-MS.

Показани са конкретни приложения на комбинираните методи при анализ на екологични и клинични обекти, храни и лекарства.



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Fitness for purpose - валидиране на методи за аналитичен контрол

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Наблюдават се две интересни посоки на развитие по отношение на изискванията към качеството на аналитичните изпитвания. От една страна интензивно нарастват броя на задачите в химичния анализ (обекти, компоненти, производителност и др.), търсят се възможности за определяне на все по-ниски нива (LOD) и постигане на по-добра прецизност. От друга страна се засилва тенденцията към обща хармонизация и консенсусно метрологично охарактеризиране (общоприета рамка за оценка) на качеството на резултатите от химичния анализ.

В настоящата лекция ще бъдат представени актуалните препоръки към процедурите за валидиране на аналитичните методи възприети от EURACHEM¹. Посочени са актуални Европейски регламенти и национални нормативи документи, касаещи изискванията към методи за анализ и контрол на тежки метали в природни обекти, храни и напитки. На тази база е представен критичен анализ на процедурите за верифициране и доказване на приложимост на даден аналитичен метод за целите на конкретно задание. Ще бъдат демонстрирани интерактивни модели, разработени на EXCEL, за оценяване на ключови индикатори на химичния анализ, предписани от системата на валидиране.

Ще бъдат илюстрирани възможности за инструментален анализ чрез апаратите на Thermo Scientific iCE AAS, iCAP ICP-OES, iCAPQ ICP-MS и ще се акцентува на оценката – „подходящ за целта“ (fit for purpose) при реализиране на задачи, касаещи методи за анализ и контрол на храни, напитки и води.

Разработените на EXCEL алгоритми, ще бъдат предоставени на участниците за трансфер и внедряване на know-how.

1. B. Magnusson and U. Örnemark (eds.) Eurachem Guide: The Fitness for Purpose of Analytical Methods – A Laboratory Guide to Method Validation and Related Topics, (2nd ed. 2014). ISBN 978-91-87461-59-0. Available from www.eurachem.org

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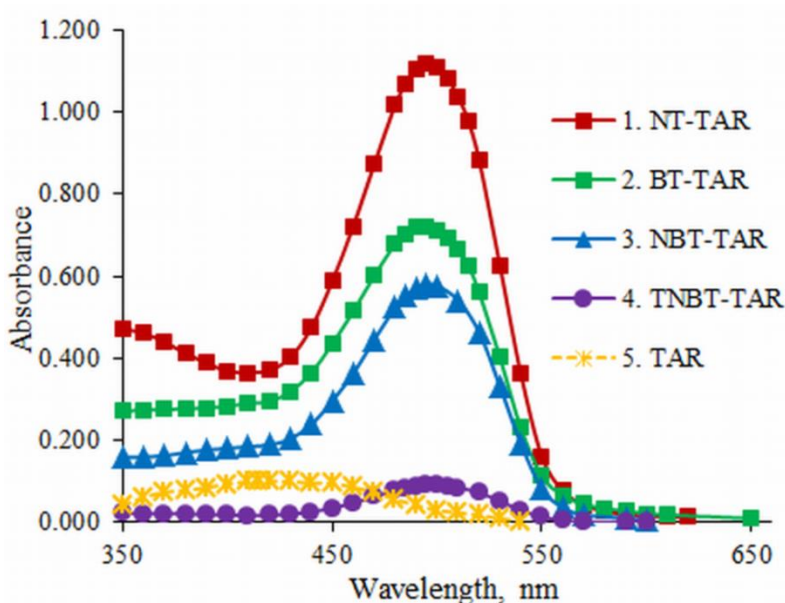
ION-ASSOCIATION BETWEEN SOME DITETRAZOLIUM CATIONS AND ANIONS ORIGINATING FROM 4-(2-THIAZOLYLAZO)RESORCINOL OR 4-(2-PYRIDYLAZO)RESORCINOL

Galya K. Toncheva* and Kiril B. Gavazov

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Ion-associates between cations originating from ditetrazolium salts {DT: Neotetrazolium chloride (NT), Blue Tetrazolium chloride (BT), Nitro Blue Tetrazolium chloride (NBT), and Tetranitro Blue Tetrazolium chloride (TNBT)} and anions originating from azo compounds {AC: 4-(2-pyridylazo)resorcinol (H₂PAR) and 4-(2-thiazolylazo)resorcinol (H₂TAR)} were studied by water-chloroform extraction and spectrophotometry. Some key characteristics of the extracted compounds were found. They can be expressed by different formulae depending on the ACs and DTs used: 1) (DT⁺)(HPAR⁻) (for DT = BT, NBT or TNBT); 2) (DT²⁺)(HTAR⁻)₂ (for DT = BT, NBT or TNBT); and 3) (DT⁺)₂(HAC⁻)₂ (for DT = NT).



The full text is available at the publisher's website:

<http://link.springer.com/article/10.1134/S1070363215010338>

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ПЛОВДИВ, 20.05.2015

EXTRACTION-SPECTROPHOTOMETRIC STUDIES ON THE COMPLEX FORMATION OF IRON(III) WITH 4-(2- THIAZOLYLAZO)RESORCINOL AND TETRAZOLIUM SALTS

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Four liquid-liquid extraction-chromogenic systems containing Fe^{III} , 4-(2-thiazolylazo)resorcinol (TAR), tetrazolium salt (TZS), water and chloroform were studied. 2,3,5-Triphenyl-2H-tetrazolium chloride (TTC), 3-(4,5-dimethyl-2-thiazol)-2,5-diphenyl-2H-tetrazolium bromide (MTT), 3-(2-naphtyl)-2,5-diphenyl-2H-tetrazolium chloride (TV), and 2-(4-iodophenyl)-3-(4-nitrophenyl)-5-phenyl-2H-tetrazolium chloride (INT) were the used TZSs. Optimization experiments for iron extraction were performed and some equilibrium constants and analytical characteristics were calculated. Under the optimum conditions, the molar ratio of the reacting Fe^{III} , TAR and TZS is 1:2:2. The suggested general formula of the extracted species is $(\text{TZ}^+)_2[\text{Fe}^{\text{II}}(\text{TAR}^{2-})_2]$.



The full text is available at the publisher's website:
<http://cirjac.com/ojs/index.php/jac/article/view/296>

ICP-MS DETERMINATION OF LANTHANIDES IN DRINKING WATER AFTER LIGANDLESS SPE WITH $\text{MnFe}_2\text{O}_4@ \text{SiO}_2$ MNPs

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The aim of the present study is to explore the application of silica modified manganese-ferrite nanoparticles as a sorbent for ligandless solid phase extraction of La, Ce, Eu, Gd and Er from drinking water. The main parameters of the extraction system as acidity of the water phase; mass of magnetic nanoparticles; extraction time and recovery conditions are varied on model solutions. The optimal conditions are determined: water phase pH=10, extraction with 40 mg MNPs for 20 minutes. Recovery of analytes in water solution is done by addition of 0.01 M HNO_3 and shaking for 1 hour. Enrichment factor EF=10 is achieved for the real sample analysis. The effectiveness and accuracy of the proposed procedure are demonstrated by the recovery of spiked lanthanides in drinking water under optimized conditions. The obtained recoveries of spikes are higher than 94%, with exception of La (R=85 \pm 3%). Despite the low selectivity of the ligandless system towards concomitant metal ions, $\text{MnFe}_2\text{O}_4@ \text{SiO}_2$ MNPs are proven as an effective sorbent for solid phase extraction of lanthanide ions, prior to ICP-MS analysis of drinking water.

Acknowledgements: НИ15-ХФ-001/24.04.2015



СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

CHARACTERIZATION OF GLASS FINDS EXCAVATED IN THE MEDIEVAL BULGARIAN CAPITAL PRESLAV BY TXRF

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This work presents the results of characterization of glass finds discovered in the medieval Bulgarian capital Preslav (893-972 AD). During the Middle Ages the town Preslav was one of the most beautiful and mighty cities of South-eastern Europe and significant cultural monuments have been preserved to nowadays. It is known from archaeological excavations that in the Inner city of Preslav a glass-workshop was located, which produced glass vessels, jewellery and ornamental window glass for decoration of the remarkable Preslav's buildings. In the south-eastern part of the Outer city of Preslav, the "Monastery of Mostich" was situated where the investigated glass artefacts were discovered.

The concentrations of 3d-transition elements (Ti, V, Cr, Mn, Fe, Co, Ni, Cu and Zn) which might be responsible for the colour of the glasses were determined by total reflection X-ray fluorescence (TXRF) analysis. Gallium and germanium were tested as internal standards. The validation of the method was performed using both certified reference materials BAM-S005 (Soda-Lime-Glass Type A and B). The experimental parameters were optimized. It was proved that the elements Mn, Fe, Co, Cu are responsible for colour generation in the investigated glass fragments. Resulting data confirm the suitability of TXRF for colouration studies of archaeological glasses.

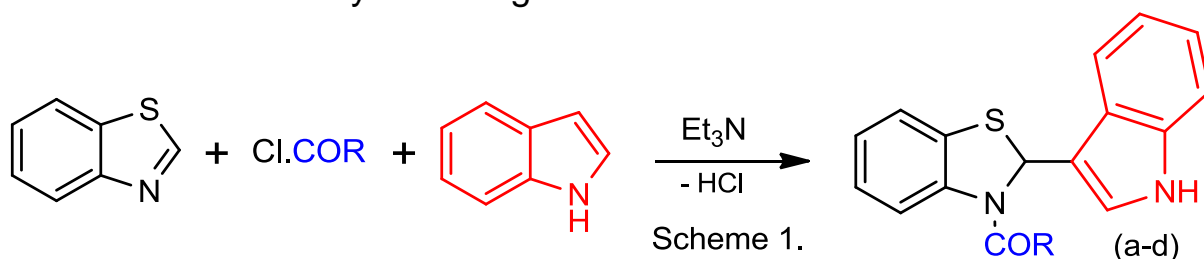
СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

THREE-COMPONENT MANNICH-TYPE REACTIONS FOR SYNTHESIS OF CAMALEXIN'S ANALOGS

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In response to pathogenic attack many plants rapidly induce the biosynthesis of low molecular weight antimicrobial compounds. Camalexin (2-(1H-indol-3-yl)thiazole) is a natural indole phytoalexin, produced in the leaves of *Camelina sativa* and *Arabidopsis thaliana* in response to infection by the fungus *Alternaria brassicae*. It exhibits antifungal activity similar to the systemic fungicide thiabendazole and also has antitumoral activity. In continuation of our interest in the synthesis of benzothiazole derivatives, we have decided to study obtaining of benzothiazoles linked to indole moiety.



(a) R= OMe, (b) R= OEt, (c) R= Ph, (d) R= Ac

Three-component Mannich-type reactions were successfully applied to the synthesis of new Camalexin's analogs in 60 - 85 % yield. The obtained products were purified by column chromatography on neutral alumina and spectral characterization by Mass spectrometry, using Thermo Scientific™ Q Exactive™ Orbitrap™, ¹H-NMR, ¹³C-NMR and IR spectroscopy.

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СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

REDUCTION OF 1-([1, 1'-BIPHENYL]-4-YL)-4-(3,4-DIMETHOXYPHENYL)-6,7-DIMETHOXY-3,4-DIHYDROISOQUINOLINE

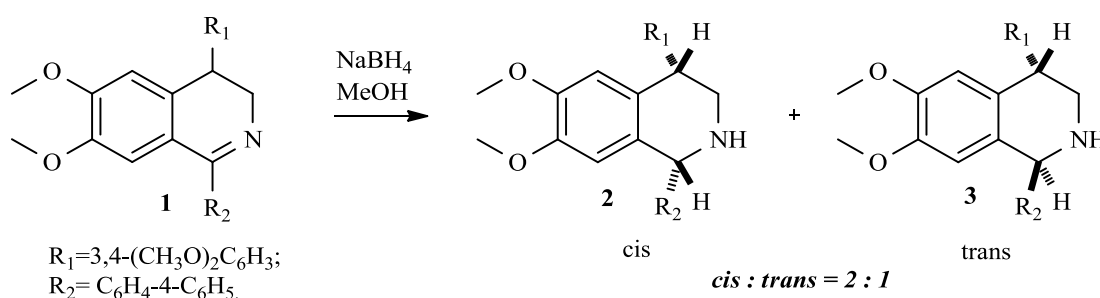
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Synthetic studies of 4-aryl-1,2,3,4-tetrahydroisoquinolines have attracted much attention from the synthetic community owing to the potential biological activities of this class of compounds and their increasing medicinal interest.

We applied simple reduction of 3, 4-dihydroisoquinoline **1** to corresponding 1,2,3,4-tetrahydroisoquinolines **2** and **3** using sodium borohydride. Sodium borohydride in methanol under normal conditions reduced the imine group in these compounds quite easily.



It was found that the reduction of 1, 4-disubstituted 3,4-dihydroisoquinoline **1** leads to formation of two compounds. Using HRMS analysis was found that both compounds have equal mass and element composition so they are diastereoisomers. The diastereoisomers are successfully separated by preparative column chromatography and 2:1 (cis:trans) ratio is determined. Further, the hypothesis was confirmed by ¹H, ¹³C NMR analysis. The relative configuration of the two diastereoisomers was determined using Nuclear Overhauser effect (NOE). The effect of the temperature on the diastereoselectivity of the reaction was checked with HPLC analysis. The ratio is not affected by the temperature at which the reaction is carried out.



СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

ORGANIC-INORGANIC MATERIALS AS NEW ADSORBENTS FOR REMOVAL OF METHYLENE BLUE AND INDIGO CARMINE FROM AQUEOUS SOLUTIONS

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Most of dyes are organic compounds used as colouring products in chemical, textile, paper, printing, leather, plastics and various food industries. The need for the treatment of dye contaminated waste water passed out from the industry. Several methods and techniques have been developed for the treatment of dye effluents. The chemical methods involve precipitation, oxidation, chemisorption and photocatalysis. Adsorption proved to be as well an efficient technique for rapid lowering the concentration of dissolved dyes in an effluent.

In the present study, several new organic-inorganic materials were studied for their potential use as adsorbents for the removal of methylene blue (MB) and indigo carmine (IC) from aqueous solutions. The materials were synthesized via sol-gel method, using tetraethyl orthosilicate (TEOS) as a source of SiO₂. The structure of the hybrid materials was studied by means of Fourier transform infrared spectroscopy (FTIR), X-ray diffractometry (XRD), scanning electron microscopy (SEM) and atomic force microscopy (AFM). The characterization of texture parameters of the investigated materials was carried out by means of low-temperature adsorption of nitrogen.

MB is a basic, while IC is an acid dye. Therefore they release in aqueous solution negative or positive charged coloured moieties respectively.

The adsorption of MB and IC was carried out by means of the batch method at initial dye concentration $C_0=100 \text{ mg L}^{-1}$. The concentrations of MB and IC were determined on a UV-VIS spectrophotometer by the wavelengths of 665 nm and 610 nm respectively. In most cases the adsorption capacities obtained for IC are higher than those for MB. Nevertheless all investigated materials could be used for the removal of MB and IC from contaminated aqueous solutions.

СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

ADSORPTION OF METHYLENE BLUE ON POLY-SULPHIDE-FUNCTIONALIZED HYBRID MATERIALS

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Industrial activities in the past few decades have caused uncontrolled contamination of aqueous streams. The methods for water purification differ in their technological design, efficiency and costs. Among these technologies adsorption seems to be one of the most appropriate and effective methods for removal of various contaminants from aqueous media.

In the present study, three sol-gel poly-sulphide-functionalized hybrid materials, denoted as A, B and C, were studied for their potential use as adsorbents for the removal of methylene blue (MB) from aqueous solutions. The effects of some experimental parameters, such as initial MB concentration, contact time and acidity of initial solutions, were evaluated. The concentrations of MB were determined on a UV-VIS spectrophotometer by the wavelength of 665 nm. The results showed that the adsorption was significantly affected by the pH value and the optimum pH range was found to be about 7.0. Pseudo-first order, pseudo-second order and intraparticle diffusion models were used to analyze kinetic data.

Equilibrium experimental data were fitted to linear Langmuir and Freundlich models. The Freundlich isotherm was found to be the most adequate in describing the adsorption processes. The highest equilibrium adsorption capacity was registered for the material, denoted as B. This fact cannot be explained only with the results obtained for texture parameters. Therefore it can be assumed that the adsorption is rather chemisorption than physical. Nevertheless, the three new materials could be used for the removal of MB from contaminated aqueous solutions. Possibilities for desorption of the investigated ions were estimated. The mixture ethanol/water could not be used for desorption of MB, while both diluted mineral acids HCl and HNO₃ showed higher recovery efficiency.

СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

Sulphate radical induced degradation of azo dye Acid Orange 7 in aqueous solution using $\text{Co}_3\text{O}_4\text{-Bi}_2\text{O}_3$ catalysts and peroxymonosulfate

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A series of $\text{Co}_3\text{O}_4\text{-Bi}_2\text{O}_3$ nanocomposite oxides were prepared by a conventional reverse co-precipitation method and post-calcination. The as-prepared catalysts were characterized by means of XRD, XPS and ICP-OES. The catalytic activity of hybrids for heterogeneous decomposition of peroxymonosulfate (PMS) to generate sulfate radicals targeting the degradation of refractory azo dye Acid Orange 7 (AO7) in aqueous solution was evaluated. The as-prepared catalysts exhibited strong PMS activation functionality and activity being much higher than bare Co_3O_4 under similar conditions. A significant improvement in the catalytic performance of Co_3O_4 after modifying with bismuth may be attributed to the basic nature of Bi_2O_3 , promoting the formation of surface Co (II) –OH complexes, playing a major role in the heterogeneous activation of PMS. The results obtained showed that 40 wt.% $\text{Co}_3\text{O}_4\text{-Bi}_2\text{O}_3$ exhibited the best catalytic activity among the composites tested and produced fast and full degradation of AO7 in 12 min. The stability of the synthesized samples and the effect of several operational parameters, such as catalyst loading, PMS concentration, solution pH and ionic strength on the AO7 degradation kinetics and removal efficiency was investigated.

Authors gratefully acknowledge financial support by the NSFB (DFNI T-02/4) and by the University of Plovdiv Research Fund (Project NI HF-2015).



**СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015**

APPLICATION OF ION CHROMATOGRAPHY FOR DETERMINATION OF TOTAL CONTENT OF SULFUR AND CHLORINE IN SOLID BIOFUELS

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The chemical composition of solid biofuels, as defined in [Directive 2000/76/EC of the European Parliament and the Council on the Incineration of Waste. In: European Commission, editor. Official Journal of the European Communities, vol. L 332; 2000. P. 91-111] and [CEN/TC 335-WG2 N94. Final draft. European Committee for standardization, editor. Solid biofuels-fuel specification and classes. Brussels, Belgium; 2003], has manifold effects on their thermal utilization. Two of the main parameters deciding whether particular type of biomass can be used in thermochemical process of biomass conversion are sulfur and chlorine content. Sulfur and chlorine are present in solid biofuels in varying concentration. During the combustion process they are usually converted to sulfur-oxides and chlorides. The presence of these elements and their reaction products may contribute significantly to corrosion and environmentally harmful emissions. S and Cl are responsible for deposit formation and corrosion. Furthermore, Cl causes HCl as well as PCDDs (polychlorinated dibenzodioxins) and PCDFs (polychlorinated dibenzofurans) that have strong carcinogenic and mutagenic properties. Many different analytical methods are used for the determination of sulfur and chlorine content in biomass, including titration, spectrometry, chromatography, X-Ray fluorescence and high-temperature oxidizing combustion with electrochemical detector.

The scope of this study is demonstration of modern automated analytical techniques for determination of sulfur and chlorine content in biomass samples, in two steps:

- decomposition of the biofuel (combustion in an oxygen bomb) and absorption of the acidic gas components in an absorption solution
- determination of sulphate and chloride in the receiving solution using ion chromatography

in accordance with BDS EN 15289:2011 "Solid biofuels. Determination of total content of sulfur and chlorine".

СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

DESIGN, SYNTHESIS AND APPLICATION OF ION- IMPRINTED SORBENTS FOR SEPARATION AND DETERMINATION OF MERCURY SPECIES IN WINE

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Ion-imprinted polymers are materials with remarkable properties the main of which is the extremely high selectivity toward the imprinted ion. Due to the memory effect introduced into the polymer matrix during the preparation process, these materials have a strong affinity toward the target analyte and could be successfully used for selective solid phase extractions.

In this study synthesis of three Hg-imprinted polymer layer coated silica gel particles and their application in speciation analysis by solid phase extraction of Hg²⁺ in wine samples are presented. To induce the selective occurrence of surface polymerization, the polymerizable double bonds were preliminary grafted at the surface of silica gel particles by silylation. Afterwards, the Hg²⁺ templates were imprinted into the polymer-coating layer through the non-covalent interaction between functional monomer (methacrylic acid) and Hg²⁺ complex with 1-pyrrolidinedithiocarboxylic acid (Hg-PDC), 1-(2-thiazolylazo)-2-naphthol (Hg-TAN) and dithizone (Hg-DTZ), respectively. After removal of templates by elution with 0.1 M thiourea in 0.1 M HCl, recognition sites of Hg²⁺ were exposed in the polymer layers. Due to the high stability of Hg²⁺ complexes with the incorporated ligands, it can be expected reliable separation of Hg²⁺ from CH₃Hg⁺ as well as the organic matrix of wine samples. The optimal conditions for quantitative sorption and elution were investigated and defined in a batch mode. Furthermore, Hg²⁺determination in white and red wine samples was achieved after its separation and preconcentration by column solid phase extraction using the prepared sorbents as a stationary phase.



СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
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Trace elements accumulation and distribution in the organs of *Phragmites australis* (common reed) from Lakes in Burgas region

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Due to its ability to accumulate metals, availability throughout the year and large biomass, *Phragmites australis* (common reed) is suitable for biomonitoring studies for the evaluation of load levels of trace metals in aqueous ecosystems. In this study, the content of toxic elements (Cd, Pb, As, Hg and Ni), main and essential elements (Na, K, Ca, Mg, Fe, Mn, Al, Cr, Co, Cu, Zn, Se, Mo and Ba) of *Phragmites australis* collected from Lake Mandra and p. 17 year 2013, were examined. For most elements, the concentrations in the inflorescence are higher than those of the stems. The concentrations of all elements of *Phragmites australis* were compared with concentrations obtained from rice hulls because they pertain to the same family *Poaceae*.

СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

CoSO₄ AS SUPPLEMENT IN BEE FEEDING – Co CONTENT IN BEE PRODUCTS AND BEE ORGANISM

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Honey is one of the most widely consumed bee products in a human diet. Both propolis and royal jelly have a long history of medicinal use and can be consumed fresh or taken in supplement form. Biological activities of honey, propolis and royal jelly, and their function as antibacterial, antioxidant, antitumor, anti-inflammatory and antiviral agents are attributed to the content of various chemical compounds such as flavonoids, essential amines and combination of essential elements. The actual chemical composition depends on many factors such as the bee feeding, pollen source, climate and environmental conditions. Salts of essential chemical elements have been studied as supplements in bee's food which showed positive effect on bees' productivity and Vitamin C exchange in the bee organism. The purpose of the work presented is to investigate the influence of CoSO₄, as supplement in bee feeding, on the Co content in the bee organism (bee heads, bee bodies and bee excrements) and bee products (honey, propolis, wax, royal jelly). CoSO₄ was added to the sugar syrup for bee feeding at three concentration levels – 4, 10 and 15 mg/L CoSO₄. Control experiment without CoSO₄ was carried out as well. Experiments were performed with two bee families, farmed in the Institute of Animal Science, Kostinbrod, Bulgaria. Co content in bee organism and bee products was determined after acid (HNO₃) digestion and ICP-MS measurement.



СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ ПЛОВДИВ, 20.05.2015

Оценка на комбинираната неопределеност на резултатите при определяне на активна субстанция цинеб получени чрез СІРАС 25 метод и ІСР-ОЕС метод

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Дитиокарбаматните фунгициди са един от най-често срещаните класове пестициди, използвани в селското стопанство. В Агрива АД на база етилен бис (дитиокарбамати) EBDS се произвеждат фунгициди от така наречена "група на манеба" - цинеб, манеб и манкозеп. Активната субстанция цинеб е дитиокарбаматен биоцид, за чиято пазарна реализация се изисква определяне и обявяване на наличната му в крайния продукт концентрация с висока точност и прецизност.

Класическият (СІРАС 25) метод за определяне на активното вещество цинеб е индиректен метод и се основава на титриметрично определяне на серовъглерода, получен след термичното разлагане на биоцида с използване на апарат на Кларк.

Предлагания от нас метод е инструментален и се основава на индиректно определяне на активното вещество чрез ІСР-ОЕС определяне на количеството на свързаната в цинеба сяра.

Инструменталният анализ е реализиран по оптимизиран метод ползващ оптико-емисионен спектрометър с индуктивно свързана плазма - Thermo Scientific - iCAP 6300 - ІСР-ОЕС CID.

Настоящата работа предлага оценка и сравнение на неопределеността на резултатите от двата метода.

Разгледани са моделните уравнения, включващи възможните величини с принос към неопределеността на двата метода. Изготвени са таблични модели за изчисляване на комбинираните неопределености на методите чрез подхода на Крагтен. Индентифицирани са приносите на входните величини към резултатната комбинирана неопределеност.

Сравнението на комбинираните неопределености на резултатите: 1.12% за СІРАС 25 метода и 0.28% за ІСР-ОЕС метода показва по-висока прецизност на инструменталния метод и го определят като подходяща алтернатива за определяне на активна субстанция в цинеб.

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СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

Изследване разделянето на Cr (III) от Cr (VI) чрез екстракция при температура на коагулация и течно-течна микроекстракция

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Проучена е възможността за селективна екстракция на Cr (III) в присъствие на Cr (VI) чрез две техники за разделяне и концентриране – екстракция при температура на коагулация (ЕТК) и течно-течна микроекстракция (ТТМЕ). В качеството на екстрагент при ЕТК е използван 1% m/v Triton X-114, а за провеждането на ТТМЕ е използвана смес от n-додекан-1-ол и n-декан-1-ол (3 : 1 m/m), в пропорция спрямо обема на водната фаза 1 : 50.

Установено е, че и при двете екстракционни техники може да се постигне селективна екстракция на Cr (III) само чрез поддържане на рН на водната фаза в диапазона от 6 до 9, без необходимост от внасяне на допълнителен лиганд. Оценена е ефективността на екстракция в зависимост от i) начинът за внасяне на енергия в системата (конвенционално загряване на водна баня и микровълново облъчване); ii) изходната концентрация на Cr (III) в екстракционната система (диапазон 0.01 – 1 mg/l); iii) присъствието на странични вещества в екстракционната система (NH₄Cl, CH₃COONa, буферни системи Na₂HPO₄/NaH₂PO₄ и H₃BO₃/Na₂B₄O₇); iv) подходът за диспергиране на фазите при ТТМЕ (ръчно разклащане; използване на магнитна бъркалка, клатачна машина или вортекс).

Азотна киселина с концентрация 3 mol/l е използвана за разтваряне на ПАВ-обогадената фаза и ре-екстракция на Cr (III) от органичната фаза, получени съответно след ЕТК и ТТМЕ. Крайните разтвори са анализирани чрез атомно емисионен спектрометър с микровълново генерирана плазма 4200 MP-AES Agilent. Отчетено е 20 % повишение на чувствителността за атомните линии на хром спрямо калибрация във водна среда (3 mol/l азотна киселина) в присъствие на ПАВ (4% Triton X-114).

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СЪВРЕМЕННИ МЕТОДИ ЗА ХИМИЧЕСКИ АНАЛИЗ И КОНТРОЛ
ПЛОВДИВ, 20.05.2015

НІІС – ЕДИН МАЛКО ПОПУЛЯРЕН РАЗПРЕДЕЛИТЕЛЕН МЕХАНИЗЪМ ПРИ ТЕЧНОХРОМАТОГРАФСКОТО РАЗДЕЛЯНЕ (СЪЩНОСТ, ВЪЗМОЖНОСТИ И ПРИЛОЖЕНИЕ)

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Най-популярният механизъм на разпределение при течнохроматографското разделяне е обратнофазовият (RP), при който в широк диапазон на хидрофобност на анализите има права зависимост на задържането им от полярността на подвижната фаза. Това позволява предвиждане на задържането и бързо оптимизиране на условията за разделяне. В някои случаи обаче той не позволява разделянето на близки по хидрофобност анализи, при които се прилагат обранофазово, йонообменно или молекулно-ситово разделяне. Има случаи и на анализи, при които никой от тези разпределителни механизми не позволява разделянето им.

Един относително малко популярен, но в някои случаи удачен за HPLC анализ на особено трудни за разделяне вещества, е т.н. НІІС разпределителният механизъм, който освен подобряване на разделянето, предлага и по-благоприятни условия за детекция и/или за разделяне на по-широк спектър от анализи.

В представяния постер, освен кратко описание и характеристика на НІІС хроматографията, са илюстрирани и примери за приложението ѝ в случаи на особено трудни за разделяне анализи, повишаване чувствителността при анализа им и едновременното определяне на много различни по полярност анализи за по-кратко време от необходимото при RP разделяне. Използването на масспектрометричен детектор с висока разделителна способност (HRMS) позволява ефективна експлоатация и атрактивно онагледяване на този ефект.



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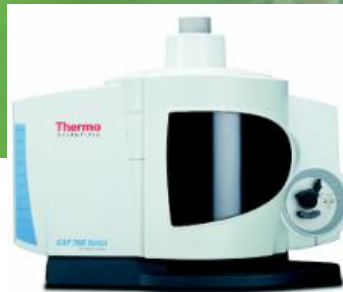
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