

# NANOVA

2010-2016

多维气相色谱(MDGC)研究与应用论文摘要集

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**STUDIES AND APPLICATIONS OF  
MULTI-DIMENTIONAL GAS  
CHROMATOGRAPHY (MDGC)**

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**ABSTRACTS**



**Nanova Environmental Inc**

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# **Technical Articles**

Liu, J., Khaing Oo, M.K., Reddy, K., Gianchandani, Y.B., Schultz, J.C., Appel, H.M., Fan, X.

**Adaptive two-dimensional microgas chromatography  
(2012) Analytical Chemistry.**

**ABSTRACT:** We proposed and investigated a novel adaptive two-dimensional (2-D) microgas chromatography system, which consists of one 1st-dimensional column, multiple parallel 2nd-dimensional columns, and a decision-making module. The decision-making module, installed between the 1st- and 2nd-dimensional columns, normally comprises an on-column nondestructive vapor detector, a flow routing system, and a computer that monitors the detection signal from the detector and sends out the trigger signal to the flow routing system. During the operation, effluents from the 1st-dimensional column are first detected by the detector and, then, depending on the signal generated by the detector, routed to one of the 2nd-dimensional columns sequentially for further separation. As compared to conventional 2-D GC systems, the proposed adaptive GC scheme has a number of unique and advantageous features. First and foremost, the multiple parallel columns are independent of each other. Therefore, their length, stationary phase, flow rate, and temperature can be optimized for best separation and maximal versatility. In addition, the adaptive GC significantly lowers the thermal modulator modulation frequency and hence power consumption. Finally, it greatly simplifies the postdata analysis process required to reconstruct the 2-D chromatogram. In this paper, the underlying working principle and data analysis of the adaptive GC was first discussed. Then, separation of a mixture of 20 analytes with various volatilities and polarities was demonstrated using an adaptive GC system with a single 2nd-dimensional column. Finally, an adaptive GC system with dual 2nd-dimensional columns was employed, in conjunction with temperature ramping, in a practical application to separate a mixture of plant emitted volatile organic compounds with significantly shortened analysis time. © 2012 American Chemical Society.

Liu, J., Seo, J.H., Li, Y., Chen, D., Kurabayashi, K., Fan, X.

**Smart multi-channel two-dimensional micro-gas chromatography for rapid workplace hazardous volatile organic compounds measurement  
(2013) Lab on a Chip - Miniaturisation for Chemistry and Biology.**

**ABSTRACT:** We developed a novel smart multi-channel two-dimensional (2-D) micro-gas chromatography ( $\mu$ GC) architecture that shows promise to

significantly improve 2-D µGC performance. In the smart µGC design, a non-destructive on-column gas detector and a flow routing system are installed between the first dimensional separation column and multiple second dimensional separation columns. The effluent from the first dimensional column is monitored in real-time and decision is then made to route the effluent to one of the second dimensional columns for further separation. As compared to the conventional 2-D µGC, the greatest benefit of the smart multi-channel 2-D µGC architecture is the enhanced separation capability of the second dimensional column and hence the overall 2-D GC performance. All the second dimensional columns are independent of each other, and their coating, length, flow rate and temperature can be customized for best separation results. In particular, there is no more constraint on the upper limit of the second dimensional column length and separation time in our architecture. Such flexibility is critical when long second dimensional separation is needed for optimal gas analysis. In addition, the smart µGC is advantageous in terms of elimination of the power intensive thermal modulator, higher peak amplitude enhancement, simplified 2-D chromatogram re-construction and potential scalability to higher dimensional separation. In this paper, we first constructed a complete smart  $1 \times 2$  channel 2-D µGC system, along with an algorithm for automated control/operation of the system. We then characterized and optimized this µGC system, and finally employed it in two important applications that highlight its uniqueness and advantages, i.e., analysis of 31 workplace hazardous volatile organic compounds, and rapid detection and identification of target gas analytes from interference background. © 2013 The Royal Society of Chemistry.

**Chen, D., Seo, J.H., Liu, J., Kurabayashi, K., Fan, X.**  
**Smart three-dimensional gas chromatography**  
**(2013) Analytical Chemistry.**

**ABSTRACT:** We developed a complete computer-controlled smart 3-dimensional gas chromatography (3-D GC) system with an automation algorithm. This smart 3-D GC architecture enabled independent optimization of and control over each dimension of separation and allowed for much longer separation time for the second- and third-dimensional columns than the conventional comprehensive 3-D GC could normally achieve. Therefore, it can potentially be employed to construct a novel GC system that exploits the multidimensional separation capability to a greater extent. In this Article, we introduced the smart 3-D GC

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concept, described its operation, and demonstrated its feasibility by separating 22 vapor analytes. © 2013 American Chemical Society.

Fan, X.

**Smart multi-dimensional gas chromatography  
(2013) IEEE SENSORS 2013 - Proceedings.**

**ABSTRACT:** A novel gas chromatography (GC) design, called smart multi-channel multi-dimensional GC, is first introduced and then demonstrated in 2- and 3-dimensional configurations by our lab recently. In this work, its performance is analyzed in comparison with the conventional GC design, showing distinct advantages in terms of total peak capacity and peak capacity efficiency. This smart GC provides a promising technology platform for portable GC devices that are potentially capable of carrying out rapid in-situ analysis of hundreds of volatile organic compounds. © 2013 IEEE.

**Collin, W.R., Scholten, K.W., Paul, D., Kurabayashi, K., Fan, X., Zellers, E.T.  
μGC × μGC microsystem with resistive and optical detection  
(2015) 2015 Transducers - 2015 18th International Conference on Solid-State  
Sensors, Actuators and Microsystems, TRANSDUCERS 2015.**

**ABSTRACT:** The integration of microfabricated chemiresistor (CR) and micro-optofluidic ring resonator (μOFRR) sensors with other microfabricated components to yield a microsystem capable of performing comprehensive two dimensional gas chromatography ( $\mu\text{GC} \times \mu\text{GC}$ ) is described. Two CRs with different monolayer protected Au nanoparticle (MPN) interface layers were tested along with one μOFRR with a PDMS interface layer. All three provided modulation numbers similar to those of a reference flame ionization detector (FID). Modulated peak widths (full width at half maximum, fwhm) for the CR detectors ranged from 0.31 to 1.9 sec, and those for the μOFRR ranged from 0.24 to 1.2 sec. Although these fwhm values were greater than those from the FID and they increased with the vapor pressure of the analyte, most were < 1 sec and all peaks were narrow enough to permit efficient  $\mu\text{GC} \times \mu\text{GC}$  separations. Analyses of mixtures of 6-18 volatile organic compounds are demonstrated. © 2015 IEEE.

**Zhou, M., Lee, J., Zhu, H., Nidetz, R., Kurabayashi, K., Fan, X.  
A fully automated portable gas chromatography system for sensitive and rapid  
quantification of volatile organic compounds in water**

## (2016) RSC Advances.

**ABSTRACT:** This paper presents the design, assembly and characterization of a fully automated portable gas chromatography system coupled with a purge-and-trap system for the sensitive and rapid field analysis of volatile organic compounds (VOCs) in water samples. The VOCs were firstly purged by helium gas in the micro-fabricated preconcentrator/injector and then injected into the downstream capillary column and photoionization detector for separation and detection. The purge-and-trap conditions were optimized to efficiently extract VOCs from water samples. The calibration of 6 VOCs with concentrations ranging from 1 µg L<sup>-1</sup> to 500 µg L<sup>-1</sup> showed excellent linearity ( $R^2 > 0.99$ ). Detection limits (3 σ) of sub-µg L<sup>-1</sup> (or sub-parts-per-billion level) were achieved, which are orders of magnitude lower than the maximum contaminant level (MCL) established by the US Environmental Protection Agency (EPA). The separation of 26 analytes (in a vapor pressure range from 0.087 Torr to 180 Torr) in a water sample in less than 15 minutes was also demonstrated. Finally, the optimized system was applied to field analysis of a groundwater sample in an environmental remediation site. The quantified results agreed well with those obtained by an analytical lab using standard analytical methods and instruments. Our system offers a lab-on-a-chip solution for sensitive and rapid water analysis compliant with the EPA sample collection method. It will have a wide range of applications in environmental monitoring, industries and healthcare. © 2016 The Royal Society of Chemistry.

**Collin, W.R., Scholten, K.W., Fan, X., Paul, D., Kurabayashi, K., Zellers, E.T.  
Polymer-coated micro-optofluidic ring resonator detector for a comprehensive  
two-dimensional gas chromatographic microsystem: µgC × µgC-µOFRR**

## (2016) Analyst.

**ABSTRACT:** We describe first results from a micro-analytical subsystem that integrates a detector comprising a polymer-coated micro-optofluidic ring resonator (µOFRR) chip with a microfabricated separation module capable of performing thermally modulated comprehensive two-dimensional gas chromatographic separations (µGC × µGC) of volatile organic compound (VOC) mixtures. The 2 × 2 cm µOFRR chip consists of a hollow, contoured SiO<sub>x</sub> cylinder (250 µm i.d.; 1.2 µm wall thickness) grown from a Si substrate, and integrated optical and fluidic interconnection features. By coupling to a 1550 nm tunable laser and photodetector via an optical fiber taper, whispering gallery mode

(WGM) resonances were generated within the  $\mu$ OFRR wall, and shifts in the WGM wavelength caused by transient sorption of eluting vapors into the PDMS film lining the  $\mu$ OFRR cylinder were monitored. Isothermal separations of a simple alkane mixture using a PDMS coated 1st-dimension (1D)  $\mu$ column and an OV-215-coated 2nd- dimension (2D)  $\mu$ column confirmed that efficient  $\mu$ GC  $\times$   $\mu$ GC- $\mu$ OFRR analyses could be performed and that responses were dominated by film-swelling. Subsequent tests with more diverse VOC mixtures demonstrated that the modulated peak width and the VOC sensitivity were inversely proportional to the vapor pressure of the analyte. Modulated peaks as narrow as 120 ms and limits of detection in the low- $\text{ng}$  range were achieved. Structured contour plots generated with the  $\mu$ OFRR and a reference FID were comparable. © The Royal Society of Chemistry.

**Lee, J., Zhou, M., Zhu, H., Nidetz, R., Kurabayashi, K., Fan, X.  
In situ calibration of micro-photoionization detectors in a multi-dimensional  
micro-gas chromatography system**

**(2016) Analyst.**

**ABSTRACT:** A photoionization detector (PID) is widely used as a gas chromatography (GC) detector. By virtue of its non-destructive nature, multiple PIDs can be used in multi-dimensional GC. However, different PIDs have different responsivities towards the same chemical compound with the same concentration or mass due to different aging conditions of the PID lamps and windows. Here, we carried out a systematic study regarding the response of 5 Krypton  $\mu$ PIDs in a  $1 \times 4$ -channel 2-dimensional  $\mu$ GC system to 7 different volatile organic compounds (VOCs) with the ionization potential ranging from 8.45 eV to 10.08 eV and the concentration ranging from  $\sim$ 1 ng to  $\sim$ 2000 ng. We used one of the PIDs as the reference detector and calculated the calibration factor for each of the remaining 4 PIDs against the first PID, which we found is quite uniform regardless of the analyte, its concentration, or chromatographic peak width. Based on the above observation, we were able to quantitatively reconstruct the coeluted peaks in the first dimension using the signal obtained with a PID array in the second dimension. Our work will enable rapid and *in situ* calibration of PIDs in a GC system using a single analyte at a single concentration. It will also lead to the development of multi-channel multi-dimensional GC where multiple PIDs are employed. © 2016 The Royal Society of Chemistry.

# **External Publications**

## 1. 石油化工—Petrochemical Industry

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### 原油与天然气分析—Crude Oil & Natural Gas Analysis

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Ventura, G.T., Hall, G.J., Nelson, R.K., Frysinger, G.S., Raghuraman, B.,  
Pomerantz, A.E., Mullins, O.C., Reddy, C.M.

**Analysis of petroleum compositional similarity using multiway principal components analysis (MPCA) with comprehensive two-dimensional gas chromatographic data**

(2011) **Journal of Chromatography A**, 1218 (18), pp. 2584-2592.

**ABSTRACT:** The accurate establishment of oil similarity is a longstanding problem in petroleum geochemistry and a necessary component for resolving the architecture of an oil reservoir. Past limitations have included the excessive reliance on a relatively small number of biomarkers to characterize such complex fluids as crude oils. Here we use multiway principal components analysis (MPCA) on large numbers of specific chemical components resolved with comprehensive two-dimensional gas chromatography-flame ionization detection (GC×GC-FID) to determine the molecular relatedness of eight different maltene fractions of crude oils. MPCA works such that every compound eluting within the same first and second dimension retention time is quantitatively compared with what elutes at that same retention times within the other maltene fractions. Each maltene fraction and corresponding MPCA analysis contains upwards of 3500 quantified components. Reservoir analysis included crude oil sample pairs from around the world that were collected sequentially at depth within a single well, collected from multiple depths in the same well, and from different depths and different wells but thought to be intersected by the same permeable strata. Furthermore, three different regions of each GC×GC-FID chromatograms were analysed to evaluate the effectiveness of MPCA to resolve compositional changes related to the source of the oil generating sediments and its exposure to biological and/or physical weathering processes. Compositional and instrumental artefacts introduced during sampling and processing were also quantitatively evaluated. We demonstrate that MPCA can resolve multi-molecular differences between oil samples as well as provide insight into

the overall molecular relatedness between various crude oils. © 2011 Elsevier B.V.

Tipler, A., Marotta, L., Disanzo, F., Grecsek, H.

**Determination of low levels of methanol in crude oils by multi-dimensional gas chromatography (MDGC) using novel micro channel flow technology**

(2012) **Journal of Chromatographic Science**, 50 (3), pp. 184-189.

**ABSTRACT:** The accurate and precise determination of methanol in crude oils at concentrations less than 10 ppm is of economic value to the petroleum industry. This report presents the optimization, results and long term performance of a flow switching device MDGC hardware, the Swafer™, for the rapid and precise analysis of methanol from approximately 0.4 ppm (w/w) to 1000 ppm. The use of low temperature injection and backflush technique decreases maintenance and increases sample throughput. The short term quantitative percent relative standard deviations at 1, 30 and 1000 ppm (w/w) methanol in crude oils are 5, 3 and 0.3, respectively. The MDGC procedure follows closely that described in ASTM D7059 for determination of crude oils; however, D7059 previously has not been evaluated at concentrations of less than 10 ppm. This work further extends the application of D7059 to concentrations of <10 ppm. © 2012 The Author.

## 多维气相色谱法测定石脑油族组成

《石化技术与应用》2011 年 第 5 期 | 陆海萍

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**摘要：**建立了一种采用具有多阀、多柱和吸附阱的多维气相色谱测定石脑油族组成的分析方法。通过 5 个阀对 7 根选择性不同的色谱柱进行准确切换控制，使样品分别进入不同的色谱柱中，分离出正构、异构烷烃，正构、异构烯烃，环烷烃，环烯烃和芳烃，然后利用程序升温，使各族组分再按碳数分离后进行定量分析。对标样的测定结果表明，组分质量分数可精确至 0.01%，加标回收率为 99.7%~108.6%，相对标准偏差为 0.24%~1.40%。

## 气相色谱新技术在油田天然气组成分析中的应用

《吉林广播电视台大学学报》2011 年 第 10 期 | 董娟

大庆油田有限责任公司勘探开发研究院 黑龙江大庆 163453

**摘要：**检测天然气的组成一般采用气相色谱法，随着色谱分析技术的日益成熟，

天然气分析检测技术也相应提高。本文研究了多维气相色谱分析技术和反吹技术在检测油田天然气的组成中的应用。通过实验，考察了分流比、载气流量、阀切换时间等因素对实验结果的影响，确定了最佳实验条件，同时对该方法的准确度和精密度进行验证。

## 凝析油全二维气相色谱分析

《石油勘探与开发》2012 年 第 1 期 | 王汇彤 张水昌 翁娜 李伟 秦胜飞 马文玲  
中国石油勘探开发研究院 中国石油天然气股份有限公司油气地球化学重点实验室  
提高石油采收率国家重点实验室

**摘要：**用全二维气相色谱对四川盆地 22 个凝析油样品进行族组分和化合物定性、定量分析。凝析油中各化合物得到很好的分离，有效消除了常规色谱分析时的共馏峰干扰，在 nC3—nC8 色谱段有效定性出 67 个化合物，比常规的色谱轻烃分析结果多 13 个化合物；在全二维图谱上可把凝析油中的各类化合物根据其结构特征分成 12 类组分。与氢火焰离子化检测器联用，过去在色谱上无法定量分析的异构烃烃、烷基环戊烷、烷基环己烷、其他单环烷烃、非甾萜类的多环烷烃、烷基苯、多环芳烃等系列化合物被同时检测，解决了凝析油族组分难以定量的问题。12 类组分、180 多种主要化合物的定量分析数据可为凝析油的成熟度判识、油源对比等油气地球化学研究提供有效数据。

## 采用二维气相色谱及微板流路控制技术分析石脑油中的微量含氧化合物

《石化技术与应用》2013 年 第 4 期 | 童玲 郭星  
中国石油兰州石化公司质检部 甘肃兰州 730060

**摘要：**采用微板流路控制技术(DeansSwitch)实现中心切割，将非极性 PDMS-1 柱与极性 Lowox 柱双柱串联，建立了分析石脑油中微量含氧化合物的二维气相色谱新方法。结果表明，采用外标法定量，30min 内可完成 14 种含氧化合物的分离与分析；标准样品 5 次重复测定的相对标准偏差均小于 2.00%；样品的加标回收率为 96.95%—102.86%；分析方法的最小检出限为 0.1 μg/g；这种方法适合测定石脑油中的微量含氧化合物。

## 一种分析天然气组分的多维色谱法

《新疆石油天然气》2014 年 第 1 期 | 刘会平  
中国石化股份有限公司胜利油田分公司地质科学研究院 东营 257015

**摘要:**介绍了一种快速分析天然气组成的多维气相色谱分析方法。通过确定色谱分析条件和阀切换时间,实现了一次进样完成样品中所有组分的分离检测。数据处理时采用了标准气体进行定性,并用面积校正归一化法进行定量计算。该方法重复性好,准确度高,并可用于炼厂气,热模拟产物的分析。

## 多维气相色谱法分析天然气组成与轻烃

《化工管理》2014年 第35期 | 黄合庭 稅蕾蕾 黎燕凌 冯燕娴  
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中海油中国有限公司湛江分公司研究院

**摘要:**本文研究了一种用 Agilent 6890N 气相色谱仪来分析天然气组成和天然气轻烃的方法。利用 4 个转换阀将 3 个定量管、4 根微填充柱和 1 根毛细管柱联接到 1 台色谱仪中,由色谱工作站控制。通过转换阀的自动切换,实现由一台色谱仪完成天然气轻烃和组分的分析工作。经验证,利用该方法所得的分析结果重复性好,准确度高,整个系统具有操作简便,省时等特点。

## 全二维气相色谱在石油地质样品分析中的应用进展

《色谱》2014年 第10期 | 高偎博 常振阳 代威 童婷 张万峰 何生 朱书奎  
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**摘要:**对石油地质样品的化学组成进行全面准确的剖析,可以获得丰富的地球化学信息,为油气勘探工作提供科学 依据。然而,该类样品除了组成复杂之外,还易受到各种物理(如蒸发、乳化、扩散、溶解和吸附)、化学(如光降解)和生物(如微生物降解)过程的影响。这些特点给样品的分析研究工作带来了极大的困难,传统的一维气相色/质谱技术很难对其进行理想的分离。全二维气相色谱(GC×GC)作为新发展起来的一种分离技术,在复杂样品分 析方面具有独特的优势,虽然在石油地质样品分析中的应用相对较晚,但也日益受到关注。本文主要综述了近 5 年来 GC×GC 在石油地质方面国内外的研究进展以及存在的主要问题,并对今后的研究进行了展望。

## 气流吹扫-注射器微萃取-全二维气相色谱法用于原油组分的表征

《色谱》2014年 第10期 | 童婷 张万峰 李东浩 赵锦花 常振阳 高偎博 代威 何生 朱书奎  
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延边大学 长白山生物资源与功能分子教育部重点实验室 吉林延吉 133002  
**摘要:**建立了气流吹扫-注射器微萃取(GP-MSE)与全二维气相色谱/飞行时间质谱

(GC×GC/TOFMS) 联用分析原油成分的方法。为了找到适用于原油样品分析的 GP-MSE 条件, 用饱和烃混合标准溶液和 15 种芳烃的混合标准溶液进行了条件优化, 得到的最佳条件如下: 取样量 5 mg、萃取溶剂正己烷 20 μL、载气流速 2 mL/min、加热时间 3 min、加热温度 300°C、冷凝温度 -2°C。处理后的样品在全二维色谱/飞行时间质谱上进样分析, 得到了满意结果。方法的检出限为 34~93 μg/L, 线性相关系数 (R<sup>2</sup>) > 0.99, 对 50 种烃类化合物的回收率在 82.0%~107.3% 之间, 相对标准偏差 < 10% (n=5)。结果表明 GP-MSE-GC×GC/TOFMS 法是一种新型绿色、高效、灵敏的分析方法, 非常适合原油中挥发性与半挥发性组分的分析。

## 全二维色谱飞行时间质谱分析法如何应用于石油地质样品分析

《中国新技术新产品》2015 年 第 6 期 | 朱庆华

大庆油田有限责任公司勘探开发研究院岩芯资料室 黑龙江大庆 163712

**摘要:** 随着我国经济的快速发展, 对于石油的需求量也越来越多, 在石油的勘测过程中使用全二维气相色谱—飞行时间质谱技术来对开采出来的石油地质样品进行了分析研究, 通过使用此项技术来对原油进行分析能够直接显示原油中的组分, 各种烃类物质的组成分布在特征区域, 此项技术在石油勘探研究中有着非常广泛的应用。

## 三检测通道-气相色谱法快速分析天然气的组成

《理化检验-化学分册》2012 年 | 肖细炼 李季 张彩明 蒋启贵 梁舒 张芳

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214151

**摘要:** 采用配有五阀(2 个十通阀和 3 个六通阀)、七柱(2 根毛细管柱和 5 根填充柱)和三检测器(氢火焰离子化检测器 A、热导检测器 B 和 C)的气相色谱法测定了天然气的组分。借助阀的切换系统及设置的分析程序, 一次进样便可实现天然气常规组分的测定。检测器 A 用于烃类气体的检测, 检测器 B 用于永久气体的检测, 检测器 C 用于氢气检测。根据标准样品组分的保留时间对未知样品作定性检测, 用外标法进行定量测定。方法的精密度符合国家标准 GB/T13610-2003 中的规定, 本方法所测得的由标准气体所混合组成的标准样品中, 各组分的测定值与标准值之间的相对误差均小于 5%。

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炼油体系与技术分析—Refinary System & Technology Analysis

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Ventura, G.T., Simoneit, B.R.T., Nelson, R.K., Reddy, C.M.

The composition, origin and fate of complex mixtures in the maltene fractions of hydrothermal petroleum assessed by comprehensive two-dimensional gas chromatography

(2012) *Organic Geochemistry*, 45, pp. 48-65.

**ABSTRACT:** Sedimentary organic matter in hydrothermal systems can be altered by high temperature fluids to generate petroleum. The saturated and aromatic fractions of these hydrothermal oils are compositionally similar to conventional oil with the exception that they often contain higher concentrations of polycyclic aromatic hydrocarbons (PAH) as well as substantial mixtures of coeluting organic compounds that produce dramatically rising signal on the baseline of gas chromatograms termed unresolved complex mixtures (UCMs). Little is known about the compounds that compose UCMs and why or how they form. This is in part due to an inability to discriminate between *in situ* and migrated components that characterize the petroleum generated in hydrothermal systems. However, UCMs are also a product of the limitations imbedded in analytical separation techniques. With the advent of comprehensive two-dimensional gas chromatography (GC.  $\times$ . GC), a revision of what should constitute molecular complexity needs to be considered. We address these problems by comparing the molecular compositions of the maltene fractions of three previously published hydrothermal petroleum samples using time of flight-mass spectrometry (GC.  $\times$ . GC-ToF-MS) and 12 hydrothermal petroleum samples in cores from three locales using comprehensive two-dimensional gas chromatography with flame ionization detection (GC.  $\times$ . GC-FID). The sediment cores were collected from Middle Valley, located off the axis of the Juan de Fuca Ridge, and the Escanaba Trough, along the Gorda Ridge, both in the NE Pacific Ocean, as well as from the Guaymas Basin in the Gulf of California. We define a UCM in GC.  $\times$ . GC data to be a condition in which  $\geq 25\%$  of the detected peaks within a chromatographic area coelute in either the first or second dimension. In turn, complex (CM) and simple mixtures (SM) are defined as having 5-24% and <5% coelution, respectively. All CM and UCMs were dominated by an array of configurational isomers, which becomes increasingly aromatic with higher molecular weight. We relate this to a multi-molecular complexity metric (MCM) by quantitatively

comparing the difference in total peak variance and peak density for a GC. ×. GC chromatogram. MCM values correlate with biomarker thermal maturity ratios for the Escanaba Trough and Guaymas Basin samples indicating that molecular complexity in these hydrothermal environments is in part a function of burial temperatures. Partial Least Squares (PLS) linear regression was applied to the total number of peak retention times as a proxy for the bulk molecular differences between each hydrothermal oil sample. Differences in the sample regressions correlate with the thermal maturity and the degree of PAH alkylation, indicating that this technique can be used to assess the degree of oxidative weathering due to dehydrogenation and hydrocarbon cracking. Subtracted chromatograms were then used to quantitatively track all of the individual molecular changes within the pyrolytic regime at Escanaba Trough. These subtracted chromatograms indicate that high molecular weight PAHs are highly mobile in hydrothermal fluids and may represent a phase partitioning that is occurring at greater depths. This phase condenses just below the seafloor to form an UCM in the near surface sediments. Saturated hydrocarbon biomarkers, such as hopanes, steranes and biphytanes are less mobile and more prone to being cracked and/or aromatized prior to migration toward the ocean floor. Together these techniques suggest that the molecular complexity of hydrothermal petroleum maximizes during the early stages of thermal maturation. The diversity of compounds forming these UCMs then decreases with increasing dehydrogenation, dealkylation and condensation reactions associated with elevated thermal stress and exposure to oxidants within the hydrothermal fluids.

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### 多维气相色谱法分析羰基合成反应气

《齐鲁石油化工》2010 年 第 3 期 | 葛育金  
中国石化齐鲁分公司第二化肥厂 山东淄博 255400

**摘要：**利用 Agilent 6890 气相色谱仪，采用阀切换以及柱反吹技术，实现了羰基合成反应气中 H<sub>2</sub>、N<sub>2</sub>、Ar、CO、CO<sub>2</sub>、C<sub>1</sub>-C<sub>3</sub> 烃类以及丁醛组分的快速分离，利用校正面积归一法对各组分进行定量。该方法通过一次进样就能实现丁醇羰基合成反应气各组分的分离和检测，试验结果表明该方法具有操作简便、分离效果好、分析速度快和定量准确的优点。

### 多维色谱技术配合三氧化二铝毛细柱检测溶解乙炔中烃类杂质

《低温与特气》2011年 第3期 | 邹震 邱彬

福州大学化学化工学院 福州 350108

国家化学工业气体产品质量监督检验中心，福建福州 350025

摘要：利用多维色谱的中心切割技术与三氧化二铝毛细柱配合检测溶解乙炔中的烃类杂质，此法也适于检测高浓度气体中的微量杂质。

## 全二维气相色谱-飞行时间质谱联用技术分析重馏分油中芳烃组

### 成

《色谱》2012年 第2期 | 郭琨 周建 刘泽龙

石油化工科学研究院 北京 100083

摘要：通过对升温速度、二维补偿温度、调制周期等关键实验参数的优化，建立了全二维气相色谱-飞行时间质谱(GC×GC-TOF MS)分析重馏分油中芳烃组分的方法，得到了重馏分油芳烃组分按环数分布的二维点阵图。根据谱库检索、标准化合物对照及文献报道，对重馏分油芳烃组分中菲、甲基菲及芘、苯并蒽等常见多环芳烃(PAH)进行了准确定性，并将该方法应用到重馏分油加氢处理工艺研究中，对菲、芘的加氢处理产物进行了定性分析。该研究为重馏分油芳烃组分的准确定性提供了新的技术手段，为加深对油品加氢规律的认识提供了技术支持。全二维气相色谱与普通一维色谱对比，在重馏分油的芳烃组分分析上体现了极大优势。

## 全二维气相色谱-飞行时间质谱法分析表征重馏分油中多环芳烃

### 化合物

《石油炼制与化工》2012年 第10期 | 周建 郭琨 田松柏 刘涛

中国石化石油化工科学研究院 北京 100083

摘要：建立全二维气相色谱-飞行时间质谱分析方法，利用该方法对重馏分油中多环芳烃进行详细表征，通过标准化合物的保留时间、质谱图和NIST谱库定性和半定量分析重馏分油中的多环芳烃和烷基取代多环芳烃，并研究结构和烷基取代基对多环芳烃加氢转化的影响。结果表明，不同结构、不同烷基取代位置和不同烷基取代数量的多环芳烃的加氢转化率有很大区别。全二维气相色谱-飞行时间质谱具有高分辨能力和高灵敏度，是分析表征复杂样品中目标化合物的强有力工具，将在石油分子水平表征领域发挥重要的作用。

## 采用多维气相色谱快速分析碳二烃加氢除炔尾气的组成

《石化技术与应用》2012 年 第 4 期 | 李赫 高源 梁玉龙 韩伟 车春霞 谭都平 梁琨

中国石油兰州化工研究中心 甘肃兰州 730060

**摘要：**采用反吹-7 柱联用-双检测器-多通道切换多维气相色谱技术定性、定量分析碳二烃加氢除炔尾气中的各组分。结果表明，建立的分析方法准确度和精密度均较高，平行测试 6 次，14 种组分的相对标准偏差范围为 0.07%~1.98%，加标回收率为 99%~103%。

## 多维气相色谱快速分析苯乙烯尾气的组成

《分析仪器》2014 年 第 3 期 | 王晓莉

中国石油独山子石化公司乙烯厂中心化验室 独山子 833600

**摘要：**采用 4 柱联用-双检测器-多通道切换气相色谱仪定性、定量分析苯乙烯尾气中的各组分，结果表明，准确度和精密度均较高，平行测定 6 次，11 种组分的相对标准偏差范围为 0.04%~18%。

## 多维气相色谱技术在乙腈法抽提丁二烯装置技术的应用

《技术应用》2015 年 | 郭星 李虞 颜宁

中国石油兰州石化公司质检部

四川石化生产监测部

吉林大学珠海学院

**摘要：**利用二维色谱的中心切割技术，通过压力切换分别将不同极性的物质切换到不同性质的两根色谱柱中进行分离，完成烃类组分及乙腈含量的分析。

## 二维气相色谱在乙烯装置分析中的研究与应用

《兰州大学》2015 年 | 戴小明

**摘要：**针对目前多维色谱的普及应用，结合兰州石化乙烯装置分析需求，本文研究了多维色谱的原理、构成以及多维色谱技术手段，本文依据作者实验室现有条件对色谱进行了二维改造，用于优化乙烯装置裂解气的分析，改造后很好的解决了裂解气分析需要两台设备的情况。同时针对现有的中心切割技术的二维分析色谱建立应用方法，并在此基础上起草行业标准。多维色谱的使用可以用于分析不同性质的样品，能够一机多用，在乙烯装置分析过程中带来了极大的便利，并优化了分析工作，为乙烯装置的平稳运行和控制奠定了基础。本论文所进行的试验值得推广，在化工行业色谱分析的应用中有良好的借鉴价值。

## 全二维气相色谱在重馏分油分析中的应用

《现代化工》2015年 第2期 | 安谧 曹青 马晨菲 杨晓燕 陈泱 林骏 肖占敏

中国石油天然气股份有限公司石油化工研究院 北京 100195

**摘要：**重馏分油组成的复杂性对其分子水平标准工作提出了巨大的挑战。全二维气相色谱(GC×GC)作为分析多成分复杂混合物极为有效的方法，在重馏分油分析中已有越来越多的应用，显示了其在族组成定性定量上的巨大优势。笔者主要对全二维气相色谱在重馏分油分析上的应用进行综述。

## 多维气相色谱在丁二烯装置乙腈组分分析中的应用

《工业》2016年 第02期 | 李庆毅

四川大学化工学院化学工程(工程硕士 2012 级)，四川成都 610065

**摘要：**用多维气相色谱仪中的多维切割单元的中心切割技术，通过压力分别将不同极性的物质切换到不同性质的两根色谱柱中进行分离，完成烃类组分及乙腈组分含量的分析。该方法的两个通道均采用了外标法进行定量，标准样品 5 次重复测定的相对标准偏差均小于 1.00%。此方法是丁二烯装置混合碳四物料测定乙腈组成的一种快捷准确、详细可靠的分析方法。

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## 炼厂气分析—*Refinery Gas Analysis*

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Qi, Z., Xu, L., Yi, Y.

The application of multi-dimensional gas chromatography in refinery gas analysis

(2013) Speciality Petrochemicals, 30 (5), pp. 83-86.

**ABSTRACT:** Multi-dimensional gas chromatograph with valve column switching techniques has been widely applied in the refinery gas analysis. According to the type of samples, refinery gas analyzer were divided into the following categories: three-valve-four-column system with two detectors, four-valve-five-column system with two detectors, four-valve-six-column system with three detectors, four-valve-five-column system with two detectors and five-valve-seven column system with three detectors. Features of each analyzer

system were compared and analyzed, which would be beneficial to know about the chromatographic analysis of refinery gas and other industrial gas.

## 多维气相色谱在炼厂气分析中的应用与发展

《广东化工》2012 年 第 4 期 | 康之军 吴碧涛

湖南长岭石化科技开发公司 湖南岳阳 414012

**摘要：**介绍了气相色谱的工作原理以及气相色谱系统在炼厂气分析中的应用与发展。探讨了多维气相色谱分析技术检测炼厂气的方法。考察了阀切换时间，柱温，检测器灵敏度的选择对实验结果的影响，并确定了最佳实验条件；该方法具有很好的精密度，单次样品分析周期为 42 min。由此可见，采用多维气相色谱分析技术，可以快速、准确地测定炼厂气的各种组分。

## 三通道气相色谱法测定炼厂气组成

《化学分析计量》2012 年 第 5 期 | 原雯

乌鲁木齐石化公司研究院 乌鲁木齐 830019

**摘要：**炼厂气是炼油工艺产生的各种气体的混合物，采用四阀六柱将炼厂气分离分解为 3 部分，以双 TCD+FID 检测器 3 通道气相色谱法快速分析炼厂气。FID 通道用于分析烃类，一个 TCD 通道分析永久性气体和硫化物，另一个 TCD 通道分析氢气，采用面积归一化法定量计算分析结果。用该法测定了 3 种标准气体，测定值与标准值基本一致，测定结果的相对标准偏差小于 8%。该法适用于测定包括液化气、烟气、裂解气等组分相近的样品组成。

## 多维气相色谱技术用于炼厂气组成的定性和定量分析

《中国新技术新产品》2012 年 第 8 期 | 郭为民 张晓辉 岳爱范 朱海燕 李毅梅

呼和浩特石化公司质检部 内蒙古呼和浩特 010070

**摘要：**介绍了多维气相色谱法一次进样同时测定炼厂气组成的分析方法，其中包括烃类(C<sub>1</sub>~C<sub>6</sub>+ )和无机气体(H<sub>2</sub>、O<sub>2</sub>、N<sub>2</sub>、CO<sub>2</sub>、CO、H<sub>2</sub>S)等全部组分。与传统炼厂气组成分析方法进行对比，该方法具有速度快、准确度高等特点，完全符合炼厂气的分析要求。

## 多维气相色谱技术在炼厂气分析中的应用

《精细石油化工》2013 年 第 5 期 | 张齐 徐立英 乐毅

中国石油化工股份有限公司北京化工研究院 北京 100013

**摘要：**基于阀柱切换的多维气相色谱技术在炼厂气分析中的应用十分广泛，概括介绍了针对不同气体类型开发的三阀四柱双通道分析系统、四阀五柱三通道分析系统、四阀六柱三通道分析系统、四阀五柱双通道分析系统、五阀七柱三通道分析系统等，对比分析了每种系统的特性，从而对炼厂气或其他工业气体的色谱分析形成系统认识。

## 小分子组分多维气相分析系统在气体分析中的应用

《化工管理》2013年 第14期 | 张京慧 王书梅

烟台巨力精细化工股份有限公司 山东莱阳 265200

**摘要：**在企业气体分析系统中，传统的方法采用六通阀进行进样操作，然后通过分析柱后，通过检测器对分离后的各组分进行分析；这样就需要进行多次进样和多台气相色谱仪。而小分子组分多维气相分析系统采用十通阀的中心切割，通过四通阀进行通道选择，可以以 H<sub>2</sub> 或者 He 为载气，一次进样，完成对 O<sub>2</sub>，N<sub>2</sub>，CH<sub>4</sub>，CO，CO<sub>2</sub> 等组分的分离分析，保持系统稳定，应用效果良好。

## 多维气相色谱双阀五柱快速分析炼厂气中的干气组成

《2015 年现场检测仪器前沿技术研讨会论文集》2015 年 | 李洪宽

山东省化工研究院色谱技术开发中心

**摘要：**正炼厂气中的干气主要含有 H<sub>2</sub>、N<sub>2</sub>、CO、CO<sub>2</sub> 及甲烷、乙烷、乙烯，以及少量的 O<sub>2</sub>、丙烷、丙烯。C4 及以上的烃类很少，其中 C4 烃含量约 1%，C5 烃含量约 0.1%；有时含有少量 H<sub>2</sub>S。干气主要是炼厂气液化制液化气时不液化的尾气，干气用来制氢气，进一步用于裂化加氢上；也用于抽提乙烯、以及用于制取环氧乙烷、乙苯等化工产品[1, 2]。从干气的组成和各组分的浓度。

## 模块化阀柱多维色谱系统用于分析炼厂气的技术

《石油与天然气化工》2015 年 第 2 期 | 薛青松 薛腾 王一萌

华东师范大学 化学与分子工程学院 上海市绿色化学与化工过程绿色化重点实验室

**摘要：**在调研了应用于测定复杂气体组分的 6 个阀柱模块及其等价关系的基础上，概括归纳了常用的 7 个以阀柱模块为基础的测定炼厂气特定组分的分析通道和 18 种测定炼厂气组分的多维气相色谱系统，并按色谱柱数量将其分为 4 柱、5 柱、6 柱及 7 柱系统，讨论了其优缺点及互变规律。调研发现，阀柱模块中两个六通阀可同一个十通阀互换，多维气相色谱系统创建的一般过程为：设计阀柱模块——搭配分析通道——组建色谱系统，多维气相色谱系统完成相同的分析任务仅与柱的数量

有关，同阀的数量无直接对应关系。

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## 石油产品分析—*Petroleum Product Analysis*

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Toussaint, G., Lorentz, C., Vrinat, M., Geantet, C.

**Comprehensive 2D chromatography with mass spectrometry: A powerful tool  
for following the hydrotreatment of a Straight Run Gas Oil**

(2011) *Analytical Methods*, 3 (12), pp. 2743-2748.

**ABSTRACT:** Comprehensive two-dimensional gas chromatography ( $\text{GC} \times \text{GC}$ ) is a technique which is rapidly gaining importance for the analysis of complex samples, especially within petrochemical matrixes. Until recently  $\text{GC} \times \text{GC}$  has been a technique used within analytical laboratories to characterize complex multi-component mixtures such as fuels, perfumes and organic aerosol extracts, which are difficult to analyze using conventional GC or GC-MS techniques. In the present study we will illustrate how  $\text{GC} \times \text{GC-qMS}$  can be used as an effective tool to accurately monitor the conversion of a Straight Run Gas Oil (SRGO) on a Cobalt Molybdenum (CoMo) supported on an alumina sulfide catalyst. A SRGO was hydrotreated at various temperatures and liquid hourly space velocities (LHSV). The products were analyzed and attention was paid to the transformation of sulfur and aromatic compounds. Quantitative analyses using  $\text{GC} \times \text{GC-qMS}$  were performed and compared to the "Simulated Distillation" and total sulfur analysis illustrating that this technique can provide a comprehensive view of the entire matrix, family of products and their distribution as well as single molecule evolution upon catalytic conversion. © 2011 The Royal Society of Chemistry.

Loegel, T.N., Cramer, J., Johnson, K., Morris, R.E.

**A novel multiplexed two dimensional gas chromatographic approach for  
improved fuel analysis**

(2015) IASH 2015 - 14th International Symposium on Stability, Handling and  
Use of Liquid Fuels.

**ABSTRACT:** Comprehensive GCxGC-MS couples two chromatographic columns with orthogonal selectivities to a fast Time-Of-Flight (TOF) mass analyzer. This approach enables direct analysis of complex organic mixtures without the need for liquid-liquid extraction pretreatment. The increased peak capacity and selectivity afforded by GCxGC are highly applicable to chemometric analyses. Like all separation techniques, the analysis of complex mixtures using GCxGC-MS is a compromise between high resolution and speed. In order to maximize chromatographic resolution to extract detailed information from a fuel sample, long primary columns are typically used with slow temperature programming. However, as with all chromatographic separations, there are limits to how well co-eluting compounds can be resolved, and this situation is exacerbated by the complexity of hydrocarbon fuels. In order to overcome these limitations, a novel parallel column GCxGC-MS configuration is being developed that will take advantage of multivariate analysis to extract discrete analyte peaks from poorly resolved chromatograms. To accomplish this goal, a LECO GCxGC-TOFMS has been modified to provide two parallel complementary sets of secondary GCxGC columns connected by a capillary flow splitter between the primary and secondary columns. The effluent from both column sets is then combined at the mass detector. In this manner, each analyte in the two-dimensional chromatogram will be represented by two peaks, thus providing a multiplex advantage that can be used in the multivariate analysis to not only isolate poorly resolved compounds, but improve compound identification and quantitation.

## 多维气相色谱法测定车用汽油含氧化合物方法改进研究

《广东化工》2010 年 第 9 期 | 黄河柳 招阳 伍海波 赫小琦

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**摘要：**一直以来车用汽油中含氧化合物分析基本均采用 ASTMD4815 或 SH/T0663 的方法，但随着车用汽油产品标准 GB/T17930-2006 版的出台，ASTMD4815 和 SH/T0663 两种方法存在着部分含氧化合物分离效果较差、甲醇检测限达不到要求的问题，因此该分析方法迫切需要改进。该文提出采用多维气相色谱，利用经过硅烷化处理后的约 1m TCEP 微填柱和 30mHP. 1 弹性毛细管柱，TCD 和 FID 双检测器进行样品分离及检测，通过对柱系统和操作条件的优化，可准确地分析车用汽油中 C1~C4 醇类和醚类含氧化合物，甲醇检测限达到 <0.05%。方法重复性的相对标准偏差 (RSD) ≤ 0.827%，标准样品回收率在 90%~110% 之间。

## 多维气相色谱法测定汽油烃类的方法改进

《内蒙古石油化工》2010 年 第 11 期 | 侯湘丽 韩斌香 张颖  
中国石油大港石化公司

**摘要:**随着环保意识的加强,国家车用汽油标准对汽油中烯烃、芳烃和苯含量提出明确限值要求,有些地方例如北京对汽油的烃类组成要求更加严格。目前使用的汽油烃类组成的测定方法(荧光指示剂吸附法),虽然经典,但存在一些问题。而多维气相色谱法的出现,在分析准确性、精密度、经济效益方面都有了很大提高。本文根据多维气相色谱法在试验中的应用,对这种方法进行了详细评述。

## 全二维气相色谱分析直馏柴油中含硫化合物

《分析化学》2010 年 第 12 期 | 杨永坛 王征 Yong-Tan  
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**摘要:**采用全二维气相色谱-硫化学发光检测器,以直馏柴油为研究对象,考察了一维色谱柱初始温度、升温速率及两维柱温温差等条件对含硫化合物分离的影响,建立了直馏柴油中含硫化合物的分析方法。本方法对基质复杂的直馏柴油中含硫化合物的分离,并定性分析或归类了直馏柴油中的主要含硫化合物。以苯并噻吩为测试样,以峰面积对浓度作图,硫的浓度在 1~100mg/kg 范围内,峰面积与硫的浓度呈线性关系,相关系数大于 0.999。与传统一维气相色谱相比,全二维气相色谱技术除可检测到苯并噻吩类、二苯并噻吩类等含硫化合物外,还可检测到直馏柴油中的硫醚类化合物;苯并噻吩类和二苯并噻吩类化合物也可得到较好分离。

## 两种分析汽油族组成的多维气相色谱法及比较

《广东化工》2011 年 第 3 期 | 汪公望  
中海油新能源(海南)生物能源化工有限公司 海南东方 572600

**摘要:**介绍目前汽油中烃组成和含氧化合物含量分析的多维气相色谱法,从仪器流程、适用对象、测试范围、测试结果等多方面进行比较。SH/T0741 方法仪器构造简单,分析简捷,特别适合装置馏出口等分析,ASTM D6839 方法在分析复杂的成品汽油和未知的调合组分时,具有适用性广,分析结果准确性好的特点。结论可供相关人员在选择试验标准、采购设备和分析时参考。

## 汽油烃类族组成检测方法的比较

《兰州石化职业技术学院学报》2011 年 第 4 期 | 袁贵锁 薛华

兰州石化公司质检部 甘肃兰州 730060

**摘要：**对几种汽油族组成的测定方法及其适用范围和优劣进行了探讨，实验结果表明：使用多维气相色谱仪采用 EN14517 测定方法，一次就能检测出烯烃、芳烃、苯含量和氧含量。在与 GB/T11132、SH/T0713、SH/T0663 方法比对后，结果均符合比对方法再现性的要求，它可以作为 GB/T11132、SH/T0713、SH/T0663 方法的替代分析方法。

### 多维气相色谱法测定汽油的组成

《当代化工》2012 年 第 8 期 | 甘露 程仲莘

中国石油化工股份有限公司洛阳分公司质检中心 河南洛阳 471012

中国石油化工股份有限公司抚顺石油化工研究院 辽宁抚顺 113001

**摘要：**在 AC 公司生产的多维色谱仪上建立了两种分析含不同烯烃浓度汽油组成的分析方法—PIONA 和 PHONA，该方法适合国内汽油组成分析的要求。经研究得到结论：当烯烃含量高时，应该采用带有二段烯烃吸附温度的 PHONA 分析模式；烯烃含量在体积分数 20% 以下的汽油最好使用 PIONA 的分析模式。

### 多维气相色谱法快速测定汽油烃族组成

《广东化工》2012 年 第 2 期 | 郭惠娟

中国石油化工集团有限公司洛阳分公司 河南洛阳 471012

**摘要：**介绍了多维气相色谱法在汽油烃族组成分析中的应用，并将多维气相色谱法和荧光指示剂吸附法、毛细管色谱法等方法进行了比较，该方法在方法准确性、精密度、分析时间和经济效益上都优于其他分析方法。

### 多维气相色谱技术对不同组分汽油烃族组成测定的适宜性探讨

《分析仪器》2013 年 第 1 期 | 王海青

中国石化九江分公司质管中心 九江 332004

**摘要：**以荧光指示剂法为基准，利用模型建立流程、模型准确性检验修正程序模式和准确分析谱图技术，建立起各种不同组分汽油烃族组成分析程序模型。采用配对 t 检验方法判断色谱法和指示剂法不存在显著性差异；色谱法和指示剂法伴随时间推移比对，其方法差值小于方法再现性；色谱法重复测试区间极差低于方法重复性。本文建立起来的分析程序模型，保证了多维气相色谱法对不同成分汽油烃族组成测定的适宜性。色谱法投用后，分析数据反馈加快，馏出口控制参数得到及时调

整，成品出厂速度加快，工作效率提高，生产成本降低。

## 多维气相色谱法测定汽油组成的研究

《西安石油大学》 2014 年 | 甘露

**摘要：**为提高汽油品质监控，本文在国外先进的新配方汽油分析仪上建立了用多维色谱法分析国产汽油组成的两种分析模式 PIONA 和 PHONA，系统分析了影响测试结果的因素，通过与现行的 FIA 标准分析方法对比得出以下结论：烯烃含量小于 20% 的情况下，多维气相色谱法分析模式 PIONA 的分析结果与标准分析方法 FIA 有较好的一致性，并且得到的信息量大，对生产监控及科研能够起到很大的帮助；多维气相色谱法分析模式 PHONA 适用于高烯烃的分析，非常适合国内汽油烯烃含量高的状况，其独特的二段吸附方法可以兼顾烯烃的吸附和保证没有重饱和烃的吸附，减少误判。多维色谱法有非常好的重复性，差值全在方法的允许误差范围内。该方法适用的样品范围宽、自动化程度高。可用于计算汽油的平均分子量、密度和 C/H 比，避免因样品量过少造成汽油的其他物性不能分析的困扰。影响因素考察表明烯烃吸附阱性质、吸附阱温度、样品进样体积、醇醚吸附阱分离温度、5A 分子筛分离温度、阀切换时间等工艺参数需要规范和确定。

## 二维气相色谱法测定车用汽油中的甲缩醛、碳酸二甲酯、乙酸仲丁酯、乙酸异丁酯和 N-甲基苯胺

《石油学报(石油加工)》2014 年 第 4 期 | 高枝荣 李继文 王川

中国石化上海石油化工研究院 上海 201208

**摘要：**针对常规车用汽油中非常规添加物的难分离问题，采用二维气相色谱分析技术，将在非极性毛细管色谱柱中与汽油组分难分离的物质——甲缩醛、碳酸二甲酯、乙酸仲丁酯、乙酸异丁酯和 N-甲基苯胺切换至极性毛细管色谱柱进行分离，并采用双氢离子火焰检测器(FID)检测，内标法定量。结果表明，采用二维气相色谱分析技术，甲缩醛、碳酸二甲酯、乙酸仲丁酯、乙酸异丁酯和 N-甲基苯胺 5 个组分均能得到较好的分离，回收率在 97.9%~102.2%，6 次重复测定的相对标准偏差小于 2.0%，定量数据准确可靠；在实验范围内，各组分线性响应良好，检测限为质量分数 0.01%。该方法简单可靠，重复性和再现性均能满足常规分析要求。

## 全二维气相色谱-飞行时间质谱分析焦化柴油中饱和烃的分子组成

《色谱》2014年 第11期 | 牛鲁娜 刘泽龙 周建 蔡新恒 田松柏

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**摘要:**建立了全二维气相色谱-飞行时间质谱(GC×GC-TOF MS)分析柴油馏分中饱和烃的分子组成的方法。结合谱库检索、质谱图解析、沸点与分子结构关系和全二维谱图特征,定性(或归类)了焦化柴油饱和烃组分中1057个化合物单体,其中正构烷烃排列规律性最强,一环-三环环烷烃按照极性和沸点的差异呈瓦片状分布在其上方。另外,还准确区分了在一维气相色谱上共流出的正构烷基环己烷和正构烷基环戊烷,以及正构 $\alpha$ 单烯烃。根据质谱采集的总离子流色谱图,采用峰面积归一化法得到了饱和烃组分的碳数分布结果,并将该方法应用于研究不同类型柴油馏分饱和烃的分子组成特点。结果表明,催化裂化和焦化柴油馏分饱和烃组分的化合物类型和分布各不相同。分子组成分析能为油品加工工艺机理的研究提供方法支持。

## 二维中心切割气相色谱法测定车用汽油中醚类、酯类和甲缩醛含量

《现代化工》2015年 第7期 | 费旭东 魏宇锋 邱丰 吴颖

上海出入境检验检疫局工业品与原材料检测技术中心 上海 200135

东华大学化学化工与生物工程学院 上海 201620

**摘要:**采用二维中心切割气相色谱法测定了车用汽油中醚类、酯类和甲缩醛的含量。目标化合物经强极性TCEP色谱柱预分离后,再由非极性DB1色谱柱进一步分离,确定了车用汽油中甲基叔丁基醚(MTBE)、乙基叔丁基醚(ETBE)、乙酸仲丁酯、碳酸二甲酯(DMC)和甲缩醛在柱切换和不切换时的保留时间,建立了双柱定性和定量分析方法。各待测物在10~10000mg/kg范围内呈良好的线性关系,相关系数均大于0.999;样品中各待测物的平均加标回收率在88.41%~114.84%之间,相对标准偏差(RSD,n=7)在0.04%~3.69%之间,方法检出限(LOD)在0.3803~21.1967mg/kg之间。该方法中样品不需要进行前处理,操作简便,灵敏度高,具有良好的重复性和再现性。

## 中心切割二维气相色谱法测定汽油中的甲缩醛含量

《石油化工》2015年 第5期 | 闻环 徐玲 吕焕明 胡江涌 温佛钱 李嘉欢

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**摘要:**采用微板流路控制(Deans Switch)中心切割技术,建立了分析汽油中甲缩醛含量的二维气相色谱法。该方法将两根不同极性色谱柱串联,汽油试样先经非极性DB-1毛细管色谱柱进行初步分离,通过优化设置中心切割时间,将汽油试样中的极性含氧化合物组分切换至强极性CP-LOWOX毛细管色谱柱进行进一步分离。以乙二

醇二甲醚为内标物，可实现甲缩醛与汽油中常见的甲醇、乙醇等13种含氧化合物的完全分离。实验结果表明，甲缩醛含量(w)在0.01%~5.00%范围内时标准工作曲线呈良好的线性关系，线性相关系数达0.999 9，试样加标回收率为97.83%~104.11%，6次重复测定的相对标准偏差小于2.00%，甲缩醛检出限为0.003% (w)。该方法准确可靠，适合测定车用汽油中的甲缩醛含量。

## 二维气相色谱技术测定汽油中甲缩醛含量

《理化检验：化学分册》2016年第2期 | 徐董育 赵彦 陈晓燕 林浩学 陈泽勇  
深圳市计量质量检测研究院 深圳 518131

**摘要：**提出了二维气相色谱技术测定汽油中甲缩醛含量的方法。利用非极性填充预柱将汽油中沸点大于正己烷的重组分反吹放空，轻组分和甲缩醛经一个装填有Carbowax-1500[15%(m/m)]固定相的色谱柱分离分析。甲缩醛的质量浓度在0.040~80.0g·L<sup>-1</sup>范围内与其峰面积呈线性关系，检出限(3S/N)为10mg·L<sup>-1</sup>。方法用于汽油样品的分析，加标回收率在102%~114%之间，测定值的相对标准偏差(n=6)在0.28%~0.91%之间。

## 二维气相色谱法测定汽油中甲缩醛、甲醇、乙醇和苯的含量

《南京工业大学学报：自然科学版》2016年第1期 | 高记 秦金平 王建 陈名浪 余鹏程

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**摘要：**利用二维气相色谱的原理和反吹技术，将预柱中的待测组分切换到分离柱中，建立汽油中甲缩醛、甲醇、乙醇和苯含量的检测方法。在进样状态下，待样品中被测组分(甲缩醛、甲醇、乙醇和苯)从预柱(SE-30)中流出后，将六通阀切换到反吹状态，重组分被反吹出预柱放空，被测组分(甲缩醛、甲醇、乙醇和苯)进入强极性的PEG-20M毛细管色谱柱中进行分析。采用氢焰离子化检测器(FID)、外标法定量进行分析。结果表明：在检测范围内线性关系良好，甲缩醛、甲醇、乙醇和苯4种组分的相关系数( $r^2$ )分别为0.998 7、0.999 2、0.999 5和0.999 4；对标准样品进行6次重复实验，相对标准偏差(RSD)均小于2%，回收率为95.5%~105.3%。该方法操作简单，灵敏度高，检测结果准确、可靠。

## 全二维气相色谱-飞行时间质谱(GC×GC/TOFMS)对比研究不同燃料油中烷烃和芳烃的图谱特征

《环境化学》2016年第5期 | 押淼磊 李昱茜 王怡程 陈宇 郑少艳 李永玉 王新

红

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**摘要:**采用全二维气相色谱-飞行时间质谱(GC×GC-TOFMS)方法对比研究不同沸程燃料油(93号和97号汽油、0号柴油和重柴油)中烃类组分的全二维谱图特征,通过对色谱和质谱条件的优化,较好地实现了不同汽油和柴油中烷烃、规则类异戊二烯烃、十氢化萘系、四氢化萘系、萘系、菲系等族组分分离和目标化合物分离,各族化合物均呈现显著的“瓦片效应”的特征。4类燃料油的烃类碳骨架分布、正构烷烃与姥鲛烷和植烷的比值以及不同族化合物的相对丰度等存在较显著的差异。

## 全二维气相色谱法测定柴油和生物柴油混合燃料中7种脂肪酸甲酯含量

《现代化工》2016年 第2期 | 林海 关剑锋 毛容妹 余建龙 李丹 李展江

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**摘要:**建立了全二维气相色谱法测定柴油和生物柴油混合燃料中7种常见脂肪酸甲酯(月桂酸甲酯、肉豆蔻酸甲酯、软脂酸甲酯、硬脂酸甲酯、油酸甲酯、亚油酸甲酯、亚麻酸甲酯)含量的方法。采用外标法定量,7种脂肪酸甲酯的标准曲线的线性相关系数r为0.99913~0.99997,检出限为2.2~5.2 mg,加标回收率在92.0%~104.0%之间,相对标准偏差为2.3%~4.7%(n=8)。方法操作简单、快速、重复性好,适用于柴油和生物柴油混合燃料中脂肪酸甲酯的测定。

## 2. 煤炭及钢铁产业—Coal and Steel Industry

Omais, B., Charon, N., Courtiade, M., Ponthus, J., Thiébaut, D.  
A novel analytical approach for oxygen speciation in coal-derived liquids  
(2013) Fuel, 104, pp. 805-812.

**ABSTRACT:** Since the production of crude oil may not meet the increasing demand in the next future, coal liquefaction products have sparked great interest

as one of the possible substitutes of petroleum in the transportation field. Speciation of oxygen in direct coal liquefaction products is essential considering the important role of oxygenated compounds in coal conversion reactions. This study attempts to characterize them as fully as possible. The originality of this approach is based on the development of complementary analytical tools to describe the composition of two coal-derived distillates: a naphtha cut and a gasoil cut. Two-dimensional gas chromatography (GC × GC), high resolution mass spectrometry (FT-ICR/MS), nuclear magnetic resonance (NMR), and UV-visible spectroscopy were applied to these two matrices. This analytical scheme shows that among 2.89% w/w of elemental oxygen present in the naphtha cut, 1.78% w/w O corresponds to phenols, 0.08% w/w O to alcohols and 0.21% w/w O to ketones. Concerning carboxylic acids, they are negligible (<0.01% w/w). Thus, a total of 2.07% w/w O is quantified what represents 72% of the oxygenated compounds contained in the naphtha cut. Similarly, in the gasoil cut, among the 0.80% w/w O of elemental oxygen, 0.62% w/w O are attributed to phenols, 0.07% w/w O to alcohols, and 0.015% w/w O to ketones. Benzo and dibenzofurans may represent the species which have not been quantified. Quantification of alcohols and phenols by carbon atom number is also allowed by GC × GC-FID using response factors. It shows that the carbon atom number varies from 6 to 11 for phenols and from 4 to 9 for alcohols. Similarly, carboxylic acids distribution by alkylation degree can be obtained by combining FT-ICR/MS and NMR results. This unique multi-technical approach offers a detail level which was never reached so far in terms of oxygenated compounds characterization for such products. This information is crucial to evaluate the potential of these liquids as a substitute for fuel and envisage their upgrading. © 2010 Elsevier Ltd. All rights reserved.

## 多维气相色谱在钢铁企业气体成分分析中的应用

《冶金环境保护》2010 年 第 5 期 | 杨四川

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**摘要：**各大钢铁企业均需分析煤气的热值成分，并计算煤气热值以满足不同生产部门的需要，同时，为了保证产品质量，也需要分析各加热炉内高纯保护气组分含量。多维气相色谱同时使用毛细管柱和填充柱混合色谱柱，兼备毛细管柱和填充柱的优点；采用自动切换载气的办法，实现对钢铁企业煤气中烃类和永久性气体的分析。文章分析了多维气相色谱检测煤气热值组分的准确度和精密度，并对气体组分

多点校正标准曲线进行了研究。结果表明，多维气相色谱分析煤气组分时具有很高的准确度和精密度，多点校正曲线也具有很好的回归线性，完全可以应用于煤气的热值及高纯炉气成分分析。

## 3. 食品工业—Food Industry

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### 食品分析—Food Analysis

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Miyazato, H., Hashimoto, S., Hayashi, S.

**First identification of the odour-active unsaturated aliphatic acid  
(E)-4-methyl-3-hexenoic acid in yuzu (Citrus junos Sieb. ex Tanaka)**  
(2013) Flavour and Fragrance Journal, 28 (1), pp. 62-69.

**ABSTRACT:** We have investigated the aroma of yuzu (*Citrus junos* Sieb. ex Tanaka). Application of the aroma extract dilution analysis technique allowed us to determine a sequence of the odour-active compounds in yuzu volatile oil. In this study, we confirmed the presence of an unknown odorant that possesses a sweaty odour and a relatively high flavour dilution factor. In order to identify the unknown compound, the concentrated volatile acid fraction of yuzu volatile oil was obtained, using an enrichment procedure consisting of the combination of the sub-extraction of an acidic component with a basic aqueous solution and subsequent affinity chromatography with an Oasis MAX cartridge. Gas chromatography-olfactometry and multi-dimensional gas chromatography-mass spectrometry analyses revealed that the unknown compound was (E)-4-methyl-3-hexenoic acid [(E)-4M3H]. (E)-4M3H was identified for the first time as an odour-active unsaturated aliphatic acid in yuzu. Additionally, both (Z)- and (E)-4M3H were stereoselectively synthesised over two steps comprised of the iron-catalysed carbometalations of homopropargylic alcohols with Grignard reagents, followed by oxidation. On the basis of sensory evaluation, the orthonasal odour detection threshold of the (E)-isomer was 10-fold to 100-fold lower than that of the (Z)-isomer. © 2012 John Wiley & Sons, Ltd.

**Purcaro, G., Cordero, C., Liberto, E., Bicchi, C., Conte, L.S.**  
**Toward a definition of blueprint of virgin olive oil by comprehensive**  
**two-dimensional gas chromatography**  
**(2014) Journal of Chromatography A, 1334, pp. 101-111.**

**ABSTRACT:** This study investigates the applicability of an iterative approach aimed at defining a chemical blueprint of virgin olive oil volatiles to be correlated to the product sensory quality. The investigation strategy proposed allows to fully exploit the informative content of a comprehensive multidimensional gas chromatography (GC. ×. GC) coupled to a mass spectrometry (MS) data set. Olive oil samples (19), including 5 reference standards, obtained from the International Olive Oil Council, and commercial samples, were submitted to a sensory evaluation by a Panel test, before being analyzed in two laboratories using different instrumentation, column set, and software elaboration packages in view of a cross-validation of the entire methodology. A first classification of samples based on untargeted peak features information, was obtained on raw data from two different column combinations (apolar. ×. polar and polar. ×. apolar) by applying unsupervised multivariate analysis (i.e., principal component analysis-PCA). However, to improve effectiveness and specificity of this classification, peak features were reliably identified (261 compounds), on the basis of the MS spectrum and linear retention index matching, and subjected to successive pair-wise comparisons based on 2D patterns, which revealed peculiar distribution of chemicals correlated with samples sensory classification. The most informative compounds were thus identified and collected in a "blueprint" of specific defects (or combination of defects) successively adopted to discriminate Extra Virgin from defected oils (i.e., lampante oil) with the aid of a supervised approach, i.e., partial least squares-discriminant analysis (PLS-DA). In this last step, the principles of sensomics, which assigns higher information potential to analytes with lower odor threshold proved to be successful, and a much more powerful discrimination of samples was obtained in view of a sensory quality assessment.

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**Špánik, I., Pažitná, A., Šiška, P., Szolcsányi, P.**  
**The determination of botanical origin of honeys based on enantiomer**  
**distribution of chiral volatile organic compounds**

(2014) *Food Chemistry*, 158, pp. 497-503.

**ABSTRACT:** The enantiomer ratios of chiral volatile organic compounds in rapeseed, chestnut, orange, acacia, sunflower and linden honeys were determined by multi-dimensional gas chromatography using solid phase microextraction (SPME) as a sample pre-treatment procedure. Linalool oxides, linalool and hotrienol were present at the highest concentration levels, while significantly lower amounts of  $\alpha$ -terpineol, 4-terpineol and all isomers of lilac aldehydes were found in all studied samples. On the other hand, enantiomer distribution of some chiral organic compounds in honey depends on their botanical origin. The significant differences in enantiomer ratio of linalool were observed for rapeseed honey that allows us to distinguish this type of honey from the other ones. The enantiomer ratios of lilac aldehydes were useful for distinguishing of orange and acacia honey from other studied monofloral honeys. Similarly, different enantiomer ratio of 4-terpineol was found for sunflower honeys. © 2014 Elsevier Ltd. All rights reserved.

**Capobiango, M., Mastello, R.B., Chin, S.-T., Oliveira, E.D.S., Cardeal, Z.D.L.,  
Marriott, P.J.**

**Giri, A., Khummueang, W., Mercier, F., Kondjoyan, N., Tournayre, P.,  
Meurillon, M., Ratel, J., Engel, E.**

**Relevance of two-dimensional gas chromatography and high resolution  
olfactometry for the parallel determination of heat-induced toxicants and  
odorants in cooked food**

(2015) *Journal of Chromatography A*, 1388, pp. 217-226.

**ABSTRACT:** The assessment of the dual impact of heating treatments on food safety and aroma is a major issue for the food sector. The aim of the present study was to demonstrate the relevance of multidimensional GC techniques, olfactometry and mass spectrometry for the parallel determination of process-induced toxicants and odorants in food starting with cooked meat as a food model. PAHs were analyzed by comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry after extraction by accelerated solvent extraction (ASE-GC $\times$ GC-TOF/MS). Odour-active compounds were determined by dynamic headspace-GC hyphenated with eightbooth olfactometry and mass spectrometry (DH-GC-MS/8O) and DH-heart-cutting multidimensional GC hyphenated with olfactometry and mass spectrometry (DH-GC-GC-MS/O). For PAH determination, the GC $\times$ GC conditions consisted

of a combination of a primary non-polar BPX-5 column and a secondary polar BPX-50 column, and a modulation period of 5s. In terms of linearity ( $R^2$  ranging from 0.985 to 0.997), recovery rate (84-111%) and limit of detection (5-65ng/kg of cooked meat), the ASE-GC $\times$ GC-TOF/MS method was found consistent with the multiresidue determination of 16 PAHs including benzo[a]pyrene in cooked meat. For aroma compounds, DH-GC-MS/8O and DH-GC-MS/O revealed 53 major meat odour-active compounds. A customized heart-cutting GC-GC-MS/O enabled the coeluting odour zones with high odour-activity to be resolved and revealed 15 additional odour-active compounds. Finally, these developments of multidimensional approaches were used to investigate the balance between 16 PAHs and 68 odour-active compounds generated with different cooking techniques. © 2015 Elsevier B.V.

**Mastello, R.B., Capobiango, M., Chin, S.-T., Monteiro, M., Marriott, P.J.  
Identification of odour-active compounds of pasteurised orange juice using  
multidimensional gas chromatography techniques  
(2015) Food Research International, 75, pp. 281-288.**

**ABSTRACT:** Odour-active compounds present in pasteurised orange juice were identified by gas chromatography-olfactometry (GC-O) employing heart-cut multidimensional GC techniques with olfactometry (O) and mass spectrometry (H/C MDGC-O/MS) and comprehensive two-dimensional gas chromatography-accurate mass time-of-flight MS (GC $\times$ GC-accTOFMS). Headspace solid phase microextraction sampling proved to be qualitatively adequate for the analysis of pasteurised orange juice. The GC-O approach distinguished 13 potent odour regions (detection frequency  $\geq 3$ ) in the orange juice extract, in which 7 regions were then subjected to detailed identification of the compounds that contribute to the odour, by using higher resolution H/C MDGC-O/MS. This analysis permitted the odour-active peaks to be better resolved on the 2D column, with removal from background matrix, for the seven regions. GC $\times$ GC-FID and GC $\times$ GC-accTOFMS reveal the overall complexity of the volatile compounds in the product and assisted in assignment of the isolated peaks of the odour-active compounds, confirming the identification in a number of cases. Four aldehydes (hexanal, heptanal, octanal, citral), 2 esters (ethyl butanoate, methyl hexanoate), and 4 monoterpenes ( $\alpha$ -pinene, D-limonene, linalool,  $\alpha$ -terpineol) were confirmed in accordance with olfactometry assessment in the processed juice. This multi-assessment instrument approach of GC-O,

GC $\times$ GC, and H/C MDGC provided an effective insight into the processed orange juice aroma. © 2015 Elsevier Ltd.

**Bertrand, E., Meyer, X.-M., Machado-Maturana, E., Berdagué, J.-L., Kondjoyan, A.**

**Modelling the Maillard reaction during the cooking of a model cheese**  
**(2015) Food Chemistry, 184, pp. 229-237.**

**ABSTRACT:** During processing and storage of industrial processed cheese, odorous compounds are formed. Some of them are potentially unwanted for the flavour of the product. To reduce the appearance of these compounds, a methodological approach was employed. It consists of: (i) the identification of the key compounds or precursors responsible for the off-flavour observed, (ii) the monitoring of these markers during the heat treatments applied to the cheese medium, (iii) the establishment of an observable reaction scheme adapted from a literature survey to the compounds identified in the heated cheese medium (iv) the multi-responses stoichiokinetic modelling of these reaction markers.

Systematic two-dimensional gas chromatography time-of-flight mass spectrometry was used for the semi-quantitation of trace compounds. Precursors were quantitated by high-performance liquid chromatography. The experimental data obtained were fitted to the model with 14 elementary linked reactions forming a multi-response observable reaction scheme. © 2015 Elsevier Ltd. All rights reserved.

## 全二维气相色谱-飞行时间质谱在香精分析中的应用进展

《香料香精化妆品》2011 年 第 6 期 | 王凯 蔡炳彪 侯春 李智宇

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**摘要：**全二维气相色谱是一种新型的功能强大的分离分析技术。介绍了全二维气相色谱-飞行时间质谱在香精香料研究领域的研究进展,该技术在香精香料的成分分析方面具有广阔的应用前景。

## 非正常食用油鉴别新方法(二): 特征奇数碳脂肪酸的多维气相色谱-质谱检测

《色谱》2012 年 第 11 期 | 金静 王龙星 陈吉平 田玉增 邹黎黎 张保琴 王淑秋

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**摘要:**通过对脂肪酸的非靶标/靶标筛查,确立了非正常食用油(俗称地沟油)的内源性特征指示物:两种奇数碳脂肪酸,包括源自动物油的13-甲基十四烷酸和源自加热植物油的十一烷酸。并借助多维气相色谱-质谱技术,依据不同极性气相色谱柱的保留作用,不仅实现了不同碳数烷酸以及同碳数烷酸异构体之间的有效分离,达到了对13-甲基十四烷酸和十一烷酸准确定量的目的;而且实现了对目标化合物的在线净化、富集。凭借该项检测方法,参加了国家食品安全风险评估中心组织的第四、五批地沟油盲样考核。经过不断完善,该方法阴性样品的正确率提高到100%,阳性样品的正确率分别达到71%和75%。再结合辣椒碱指标,从内、外源指示物两方面全面、准确地对食用油样品进行判定,使得阳性样品的正确率分别提高至89%和100%。目前,该方法已经入选国家卫生部公布的四大地沟油鉴定仪器分析方法,正等待权威部门的协同性验证。

## 全二维气相色谱/飞行时间质谱法定性筛查鱼肉组织中含卤有机 污染物

《分析化学》2012年 第8期 | 汪洋 于志强 罗湘凡 冯加良 张东平 任国发 盛国英 傅家谟

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**摘要:**采用全二维气相色谱/飞行时间质谱(GC×GC-TOFMS),建立了鱼肉样品中含卤有机污染物的定性和定量分析方法。鱼肉样品用正己烷-丙酮(1:1, V/V)提取,凝胶色谱和复合硅胶柱净化,浓缩富集,全二维气相色谱联用飞行时间质谱(DB-5MS毛细管色谱柱联HT-8色谱柱)检测。软件自动识别后,经三步筛查,共鉴定出含氯或溴化合物72种,其中包括33种多氯联苯,9种有机氯农药,4种多溴联苯醚,4种DDT代谢产物,2种氯代茴香醚,2种氯苯乙烯,1种氯代茴香硫醚及1种甲基三氯生。另外,从质谱信息上看,有16种化合物明显含氯或含溴,但是因为缺少必要的谱库信息不能准确识别。采用外标定量法,对鱼肉样品中检出的主要的10种多氯联苯和1种多溴联苯醚进行了准确定量分析。

## 全二维气相色谱-四极杆质谱法检测植物油脂中脂肪酸

《色谱》2012年 第11期 | 郑月明 冯峰 国伟 储晓刚 潘家荣 贾玮

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**摘要:**建立了植物油脂中 31 种脂肪酸成分的全二维气相色谱-四极杆质谱(GC×GC-qMS)分析方法。样品经甲酯化衍生后,以 DB-1 柱(30 m×0.25 mm×0.25 μm)作为一维柱、DB-Wax 柱(3.2 m×0.1 mm×0.1 μm)作为二维柱组成柱系统进行分离,在调制周期为 3.5 s、四极杆质量扫描范围为 m/z 40~350 的条件下,植物油脂中 31 种脂肪酸成分可以在 50 min 内得到准确和灵敏的检测。将本方法应用于实际样品的分析,灵敏度较传统的气相色谱-质谱法提高了 100 倍以上,一些植物油中微量的脂肪酸成分也因此被检出。该研究不仅为植物油中脂肪酸成分的分析提供了新的技术手段,同时对于确保食用植物油的质量安全、消除食用植物油的掺假伪劣等均有重要意义。

## 滁菊挥发性成分的全二维气相色谱/飞行时间质谱研究

《食品科学》2013 年 第 2 期 | 韩婷 毛健 姬中伟 黄桂东

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**摘要:**建立滁菊挥发性成分的全二维气相色谱/飞行时间质谱检测方法。通过使用全二维气相色谱/飞行时间质谱,以非极性柱 Rxi-5MS(30m×0.25mm, 0.2 μm)作为第一维柱,中等极性柱 Rxi-17(1.75m×0.1mm, 0.1 μm)作为第二维柱,对滁菊的挥发油成分进行定性分析。结合飞行时间质谱谱图库检索与全二维特有的包含结构信息的二维谱图,鉴定出滁菊的挥发性成分中相似度大于 850 的 156 种组分。

## 在线凝胶渗透色谱-二维气相色谱/质谱法测定鲫鱼样品中的 14 种农药残留

《色谱》2014 年 第 2 期 | 李淑静 董梅 许泓 宓捷波 陈其勇 葛宝坤 翟自芹

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**摘要:**建立了在线凝胶渗透色谱-二维气相色谱/质谱(GPC-MDGC/MS)测定鲫鱼样品中 14 种农药残留的方法。样品用环己烷/乙酸乙酯(1:1, v/v)提取两次,合并提取液。提取液冷冻、过膜后,经在线凝胶渗透色谱净化后直接进行 MDGC/MS 分析,通过中心切割的方式将农药组分选择性切割进入二维色谱柱进行进一步分离分析,采用内标法进行定量。实验结果表明:14 种农药在 0.01~0.9 mg/L 范围内具有较好的线性关系,相关系数均大于 0.99。14 种农药在 3 个添加水平(0.01、0.05、0.1 mg/kg)

的加标回收率为 83.0%~112.9%，相对标准偏差为 3.2%~12.0%。该方法前处理简单，实现了在线 GPC 和 MDGC 的有效结合，准确度好、精密度高，具有很好的推广性。

## 全二维气相色谱-飞行时间质谱法分析糟带鱼挥发性风味成分

《现代食品科技》2014 年 第 2 期 | 谢诚 欧昌荣 曹锦轩 汤海青

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**摘要：**采用固相微萃取结合全二维气相色谱-飞行时间质谱萃取、分析糟带鱼挥发性风味成分。比较了不同萃取头的萃取效果，研究萃取温度、萃取时间、搅拌速度和氯化钠等因素对萃取效果的影响。结果表明：在相同条件下，50/30CAR/PDMs/DvB 对糟带鱼挥发性成分的萃取效果最好，萃取温度、时间、搅拌速度和盐的添加量对萃取效果有不同程度的影响，最适萃取条件为：萃取温度 50℃，萃取时间 40min，搅拌速度 300r/min，氯化钠添加量 20%。共鉴定出 295 种挥发性成分，其中醛类 58 种，占 33.62%；酮类 58 种，占 15.52%；酯类 65 种，占 24.12%；醇类 53 种，占 17.49%；其它 61 种，占 9.25%。和一维气相色谱相比，全二维气相色谱飞行时间质谱具有高分辨率和高灵敏度特性，可用于食品特征风味分析，风味产生机理等相关领域研究，为食品的品质控制提供理论依据。

## 全二维气相色谱-飞行时间质谱法分析肉味香精挥发性风味成分

《核农学报》2016 年 第 7 期 | 张喆 钟莺莺 汤海青 欧昌荣 赵海英 俞雪钧

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**摘要：**为明确肉味香精的风味组成和特征风味成分，并为其品质评价及掺伪鉴别提供信息，采用固相微萃取结合全二维气相色谱-飞行时间质谱分析肉味香精中的顶空挥发性成分。结果表明，最适的萃取条件为：用 50/30 μm CAR/PDMS/DVB 萃取头，在 60 ℃温度下萃取 30 min。此条件下萃取该肉味香精中的挥发性成分，共有 167 个化合物通过全二维气相色谱-飞行时间质谱分析得到鉴定，分别为酯类 20 种，醇类 32 种，酮类 25 种，醚类 8 种，醛类 32 种，杂环类 26 种，烃类 24 种，其中对肉味香精风味贡献较大的是醛类、杂环类物质及各类物质中的含硫化合物，它们是形成该肉味香精特征风味的主要成分。此外，全二维气相色谱-飞行时间质谱法具有的高分辨率、高灵敏度和高峰容量的特性，使分离鉴定肉味香精这类复杂食品基质样品的风味成分、深入表征其风味特征成为可能。本研究为肉味香精及同类产品的

挥发性成分的研究、品质及安全性评价等提供了研究方法和科学依据。

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## 造酒业—Wine Industry

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Botezatu, A., Pickering, G.J., Kotseridis, Y.

**Development of a rapid method for the quantitative analysis of four methoxypyrazines in white and red wine using multi-dimensional Gas Chromatography - Mass Spectrometry**  
(2014) Food Chemistry, 160, pp. 141-147.

**ABSTRACT:** Alkyl-methoxypyrazines (MPs) are important odour-active constituents of many grape cultivars and their wines. Recently, a new MP - 2,5-dimethyl-3- methoxypyrazine (DMMP) - has been reported as a possible constituent of wine. This study sought to develop a rapid and reliable method for quantifying DMMP, isopropyl methoxypyrazine (IPMP), secbutyl methoxypyrazine (SBMP) and isobutyl methoxypyrazine (IBMP) in wine. The proposed method is able to rapidly and accurately resolve all 4 MPs in a range of wine styles, with limits of detection between 1 and 2 ng L<sup>-1</sup> for IPMP, SBMP and IBMP and 5 ng L<sup>-1</sup> for DMMP. Analysis of a set of 11 commercial wines agrees with previously published values for IPMP, SBMP and IBMP, and shows for the first time that DMMP may be an important and somewhat common odorant in red wines. To our knowledge, this is the first analytical method developed for the quantification of DMMP in wine. © 2014 Elsevier Ltd. All rights reserved.

**Identification of aroma-active volatiles in banana Terra spirit using multidimensional gas chromatography with simultaneous mass spectrometry and olfactometry detection**

(2015) Journal of Chromatography A, 1388, pp. 227-235.

**ABSTRACT:** Fruit spirits have been produced and consumed throughout the world for centuries. However, the aroma composition of banana spirits is still poorly characterised. We have investigated the aroma-impact compounds of the banana Terra spirit for the first time, using multidimensional gas chromatography (MDGC and GC × GC) in a multi-hyphenated system - i.e.,

coupled to flame ionisation detection (FID), mass spectrometry (MS), and olfactometry (O). Solid-phase microextraction (SPME) was used to isolate the headspace aroma compounds of the banana spirit. The detection frequency (DF) technique was applied and aroma regions, detected in the first column separation at >60% Nasal Impact Frequency (NIF), were screened as target potent odour regions in the sample. Using a polar/non-polar phase column set, the potent odour regions were further subjected to MDGC separation with simultaneous O and MS detection for correlation of the aroma perception with MS data for individual resolved aroma-impact compounds. GC-O analysis enabled 18 aroma-impact regions to be located as providing volatiles of interest for further study; for example, those comprising perceptions of flower, whisky, green, amongst others. Compounds were tentatively identified through MS data matching and retention indices in both first and second dimensions. The principal volatile compounds identified in this work, which are responsible for the characteristic aroma of the banana spirit, are 3-methylbutan-1-ol, 3-methylbutan-1-ol acetate, 2-phenylethyl acetate and phenylethyl alcohol. This is the first such study to reveal the major aroma compounds that contribute to banana spirit aroma. © 2015 Elsevier B.V.

**Chin, S.-T., Eyres, G.T., Marriott, P.J.**

**Application of integrated comprehensive/multidimensional gas chromatography with mass spectrometry and olfactometry for aroma analysis in wine and coffee**

**(2015) Food Chemistry, 185, pp. 355-361.**

**ABSTRACT:** Component coelution in chromatographic analysis complicates identification and attribution of individual odour-active volatile molecules in complex multi-component samples. An integrated system incorporating comprehensive two-dimensional gas chromatography ( $\text{GC} \times \text{GC}$ ) and multidimensional gas chromatography (MDGC), with flame ionisation, olfactometry and mass spectrometry detection was developed to circumvent data correlation across different systems. Identification of potent odorants in Shiraz wine and the headspace of ground coffee are demonstrated as selected applications. Multiple solid-phase microextraction (SPME) sampling with GC-O located odour-active regions;  $\text{GC} \times \text{GC}$  established the complexity of odour-active regions; MDGC provided high-resolution separation for each region; simultaneous 'O' and MS detection completed the analysis for target

resolved peaks. Seven odour regions in Shiraz were analysed with MDGC-O/MS detection, revealing 11 odour volatiles through matching of mass spectrometry and retention indices from both separating dimensions, including acetic acid; octen-3-ol; ethyl octanoate; methyl-2-oxo-nonanoate; butanoic acid, 2-methylbutanoic acid, and 3-methylbutanoic acid; 3-(methylthio)-1-propanol; hexanoic acid;  $\beta$ -damascenone; and ethyl-3-phenylpropanoate. A capsicum odour in ground coffee was identified as 2-methoxy-3-isobutylpyrazine with a 5-fold increase in S/N of the odorant when acquired using a 6-time cumulative SPME sampling approach. © 2015 Elsevier Ltd. All rights reserved.

## 多维气相色谱法检测酒中甲醇

《分析化学》2011年 | 伊雄海 郭德华 邓晓军 朱坚 樊祥 施婧嫣  
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**摘要：**建立了多维气相色谱检测酒中甲醇含量的方法。采用自动进样器将酒类样品直接注入气相色谱进样口，依次经过非极性 RTX-5ms 色谱柱和极性 HP-INNOWax 色谱柱，由气相色谱质谱选择离子监测技术进行测定与确证。甲醇在 1~10000 mg/L 范围内呈线性相关；方法检出限为 1 mg/L。各种酒类样品在 5, 50, 250, 400 和 800 mg/L 添加水平时，平均回收率为 91.3% ~ 101.9%，相对标准偏差为 4.5% ~ 8.6%。本方法无需前处理，灵敏度高，选择性好，定性准确。

## 利用全二维气质联用技术和吸附搅拌萃取技术对中国名酒剑南春 酒体风味质量特色的研究

《酿酒》2012年第5期 | 徐占成 陈勇 王双  
四川剑南春集团有限责任公司 四川绵竹 618200

**摘要：**白酒是中国特有的一种蒸馏酒，其独特的生产工艺赋予了它独特的滋味特色，其中包括了数量众多的微量香味成分。随着科技的进步，研究人员对传统固态白酒的风味成分分析越来越深入透彻。而针对白酒这类复杂体系的分离分析需要，多维联用技术已经成为近年来国际上研究的热点。其中全二维气相色谱(GC×GC)及其与飞行时间质谱(TOFMS)联用即是新近发展起来的一种高分辨、高灵敏度的分离鉴定技术。

## 利用 SBSE 和全二维气质联用 (GC×GC-TOFMS) 新技术解析白酒香

## 味物质的研究

《酿酒科技》2012年 第7期 | 徐占成 陈勇 王双

四川剑南春集团有限责任公司 四川绵竹 618200

**摘要：**将搅拌吸附萃取(SBSE)和全二维气相色谱/飞行时间质谱联用技术(GC×GC/TOFMS)联合应用，对中国传统名优白酒中的香味成分展开了全面细致的研究，结果表明，该法可从不同名优白酒中分离分析出上千种微量成分，远远多于其他分析技术。

## 基于全二维气相色谱-飞行时间质谱分析技术同时测定白酒中 10

### 种吡嗪类物质

《安徽农业科学》2015年 第26期 | 周庆伍 徐祥浩 汤有宏 刘国英 高江婧 姜利

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**摘要：**[目的]利用全二维气相色谱-飞行时间质谱(GC×GC-TOFMS)分析技术同时测定白酒中10种吡嗪类物质。[方法]吡嗪类物质是白酒中的一类重要健康功能成分，应用全二维气相色谱-飞行时间质谱联用仪结合液液萃取样品前处理过程同时检测白酒中10种吡嗪类物质。[结果]试验表明，标准曲线线性相关系数R<sup>2</sup>均大于0.99，相对标准偏差RSD在3.1%-11.3%，加标回收率在79%-96%，该方法满足白酒中吡嗪类痕量物质的定量要求。[结论]该试验方法分析结果准确，可用于白酒中吡嗪类物质的定性与定量分析。

## 全二维气相色谱/飞行时间质谱用于露酒微量成分的分析

《酿酒》2016年 第4期 | 许和强 熊含鸿 花振新

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**摘要：**用全二维气相色谱/飞行时间质谱(GC×GC/TOFMS)研究露酒中微量成分，并对我国具有典型性露酒中微量成分进行了分析。在相同条件下，露酒1分出512个峰，露酒2分出686个峰，露酒3分出615个峰。其中包括酯类、醇类、酸类、醛酮类、N类、S类、萜烯类等。定性分析了多种有利于人体健康的氨基酸类、酮类、萜烯类等物质。分析结果表明，GC×GC/TOFMS在产品微量成分分析、风格形成机理及对人类饮食健康研究等领域有很大的优势。

## 基于全二维气相色谱-飞行时间质谱分析对古井贡酒健康功能成

## 分的研究

《酿酒科技》2016年 第1期 | 李安军 徐祥浩 汤有宏 刘国英 高江婧 姜利

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**摘要:**应用全二维气相色谱-飞行时间质谱联用仪(GC×GC-TOFMS)结合顶空固相微萃取技术(HS-SPME)全面分析古井贡酒中的风味成分。结果表明,古井贡酒中含有吡嗪类、萜烯类、内酯类以及不饱和烯烃、烯酸、烯酯等一系列与人体健康密切相关的健康功能成分。

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## 制茶业—Tea Industry

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Jia, W., Chu, X., Zhang, F.

**Multiresidue pesticide analysis in nutraceuticals from green tea extracts by comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry**

(2015) *Journal of Chromatography A*, 1395, pp. 160-166.

**ABSTRACT:** A new analytical method was developed and validated for simultaneous analysis of 423 pesticides, isomers, and pesticide metabolites in nutraceutical products obtained from green tea (*Camellia sinensis*) extract. Response surface methodology was employed to optimize a generic extraction method. The automated extraction procedure was achieved in a simple disposable pipet extraction. Comprehensive two-dimensional gas chromatography with time-of-flight mass spectrometry was used for the separation and detection of all the analytes. The method was validated by taking into consideration the guidelines specified in European SANCO/12571/2013 Guideline 2013 and Commission Decision 2002/657/EC. The extraction recoveries were in a range of 81.6-113.0%, with coefficient of variation <6.4%. The limits of decision for the analytes are in the range 0.04-4.15 $\mu\text{g kg}^{-1}$ . The detection capabilities for the analytes are in the range 0.07-6.92 $\mu\text{g kg}^{-1}$ . The 423 compounds behave dynamic in the range 0.1-200 $\mu\text{g kg}^{-1}$  concentration, with correlation coefficient >0.99. This validated method has been successfully applied on

screening of pesticide residues in one hundred and twenty-four different commercial nutraceutical products from green tea extract, and methamidophos, resmethrin, propoxur, tridemorph, ethiofencarb, flamprop isopropyl, furalaxyd, bifenthrin and fenpropathrin were detected in a few samples tested in this study.  
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## 西湖龙井茶香气成分的全二维气相色谱-飞行时间质谱分析

《中国农业科学》2015 年 第 20 期 | 朱荫 杨停 施江 余方林 戴伟东 谭俊峰 郭

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**摘要:** 【目的】香气是评价茶叶品质优劣的最重要因子之一，探明茶叶香气的化学组成可进一步丰富茶叶香气化学理论，为改善和提高茶叶香气品质提供重要的科学理论依据。【方法】采用全二维气相色谱-飞行时间质谱联用技术(GC×GC-TOFMS)与气相色谱-质谱联用技术(GC-MS)分析西湖龙井茶的香气成分，比较两种分析技术在分离性能上的差异性；结合质谱数据库匹配、化合物保留时间、结构谱图及峰面积等对通过 GC×GC-TOFMS 分离得到的香气成分进行定性及相对定量分析；进而结合相对高含量化合物( $\geq 0.5\%$ )的气味特征分析西湖龙井茶的特征性香气成分。【结果】通过与 GC-MS 的总离子流图及色谱峰对比，GC×GC-TOFMS 在分离性能上显示出了强大的优越性；定性分析及相对定量分析表明，采用 GC×GC-TOFMS 技术鉴定出西湖龙井茶样品中存在的 522 种共性香气成分，归为烯醇、烯、胺、烷烃、醛、烯醛、醚、醇、酯、内酯、烯酯、烯酮、酮、酚、酸、含硫化合物、氮杂环化合物、氧杂环化合物、芳香烃及炔等 20 类化合物，其中芳香烃的数量最多(77 个)，烷烃(50 个)、烯(43 个)、酯(43 个)、酮(41 个)次之，炔类最少(3 个)；戊烯-3-醇、顺-3-己烯醇、芳樟醇、 $\alpha$ -松油醇、香叶醇、丁烷、甲基环戊烷、2, 2, 4, 6, 6-五甲基庚烷、十二烷、二十一烷、二十四烷、三十一烷、乙醛、戊醛、己醛、糠醛、庚醛、苯乙醛、壬醛、乙丙醚、2-乙氧基丁烷、乙基丁基醚、1, 2-二乙氧基乙烷、戊基乙基醚、正戊醇、叔丁醇、苄醇、苯乙醇、植醇、邻苯二甲酸二异丁酯、酞酸二丁酯、乙基-2-(5-甲基-5-乙烯基四氢呋喃-2-烯)丙基-2-烯碳酸酯、顺-己酸-3-己烯酯、棕榈酸甲酯、乙偶姻、2, 4-二叔丁基苯酚、异戊酸、壬酸、棕榈酸、亚油酸、硬脂酸、二甲基亚砜、苯并噻唑、吲哚、咖啡碱、芳樟醇氧化。

## 全二维气相色谱-飞行时间质谱结合聚类分析与 Fisher 判别分析

## 对铁观音品质等级的评价研究

《分析测试学报》2015 年 第 5 期 | 程权 杨方 李捷 卢声宇 蓝锦昌 江锦彬  
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**摘要:**采用丙酮超声萃取铁观音样品,以全二维气相色谱-飞行时间质谱分析丙酮提取物。经筛选,在 24 份不同产季与等级的铁观音中获得 68 种共有组分,并结合质谱数据库、保留指数与结构谱图等进行了初步鉴定。在此基础上以基于 Ward 法的聚类分析将所有样品分为 3 个类别,获得了与感官审评基本相似的结果。通过逐步判别获得 5 种对分类结果有显著影响的组分,以此为变量通过 Fisher 判别法建立了 4 个判别函数,其对样品等级分类的结果与感官审评结果的符合率达到 95.8%。证实了通过分析茶叶生化成分进行品质评判的可能。

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## 烟草业—Tobacco Industry

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Wang, Y., Sun, W., Su, Q., Liu, B.

**Determination of main neutral aroma components in tobacco by solvent extraction with heart-cut multidimensional gas chromatography-mass spectrometry**

(2013) **Tobacco Science and Technology**, (7), pp. 43-47.

**ABSTRACT:** A method for determining the main neutral aroma components in tobacco was developed by combining solvent extraction with heart-cut multidimensional gas chromatography-mass spectrometry (MDGC-MS). Tobacco sample(0.5 g)was extracted by ethyl ether(5 mL), the extract was washed with 5% H<sub>2</sub>SO<sub>4</sub>(2 mL)subsequently, then analyzed by MDGC-MS after standing overnight. The neutral aromatic components, including solanone, β-damascenone, geranylacetone, β-ionone, megastigmatrienone and dihydroactinidiolide were determined qualitatively and quantitatively by using methyl eugenol as the internal standard. Further, this method was compared with simultaneous distillation extraction(SDE)-gas chromatography-mass spectrometry(GC/MS). The results showed that: The detection limit of the analytes ranged from 0.02 to 0.18 μg/mL, the recoveries were between 80.0% to

123.4% with the intra-day relative standard deviation (RSD)≤7.5%. The developed method is simple, fast, accurate and suitable for the analysis of neutral aroma compounds in batches of tobacco samples.

**Botezatu, A., Kotseridis, Y., Inglis, D., Pickering, G.J.**

## A survey of methoxypyrazines in wine

(2016) **Journal of Food, Agriculture and Environment, 14 (1), pp. 24-29.**

**ABSTRACT:** Alkyl-methoxypyrazines (MPs) elicit green aroma and flavour in many wines, and are generally considered detrimental to wine quality. Grapes and select members of the Coccinellidae (ladybeetle) family contribute MPs to wine, however, relatively little is known about the relative contribution of each source or the prevalence of the recently identified 2,5-dimethyl-3-methoxypyrazine (DMMP) in commercial wines. Here, we determined the concentrations of DMMP, isopropyl methoxypyrazine (IPMP), secbutyl methoxypyrazine (SBMP) and isobutyl methoxypyrazine (IBMP) in 187 wines from 14 countries, nine vintages and seven wine varietals using Multi Dimensional Gas Chromatography coupled with Mass Spectrometry and headspace solid phase microextraction. Results generally confirm previous findings, including the range of IBMP and SBMP values, and the lack of a clear relationship between MP concentrations in wines and any one geographical, climatic, or vintage factor. DMMP was detected in a large number of wines for the first time, particularly reds, and at levels that indicate it may play an active role in wine aroma. IPMP concentrations in Chardonnay wines were higher than previously reported. A new parameter – Total Impact Factor (TIF) - is proposed as a metric of the combined sensorial impact of MPs on wine aroma. We also conclude that IPMP:IBMP ratios may be useful as a possible diagnostic of Coccinellidae contamination in some varietal wines, and thus potential for ladybug taint (LBT). © 2016, World Food Ltd. and WFL Publishers. All Rights Reserved.

## 中心切割二维气相色谱法测定卷烟主流烟气中的硝基苯

《烟草科技》2010 年 第 5 期 | 尚静静 郭吉兆 张晓兵 蔡君兰 王冰 谢复炜  
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**摘要：**建立了环己烷萃取、硅胶固相萃取柱净化和中心切割二维气相色谱法测定卷烟主流烟气粒相物中硝基苯的方法，并采用该法测定了 6 种卷烟样品主流烟气中

的硝基苯释放量。结果表明:1. 中心切割技术有效提高了色谱的分离能力,使得痕量硝基苯可以从复杂的烟气基质中分离出来,分离效果明显优于一维色谱; 2. 方法的检测限为 1.28 mg/mL, 相对标准偏差(RSD) 6.82%, 回收率 87.6%; 3. 6 种卷烟主流烟气中硝基苯的释放量为 0.091~1.66 ng/支。

## 薄荷卷烟中香味成分的全二维气相色谱/飞行时间质谱分析

《化学研究》2010 年 第 5 期 | 郑晓云 熊晓敏 万敏 沈晓晨

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**摘要:**采用全二维气相色谱/飞行时间质谱法(GC×GC/TOFMS),以较长的非极性柱DB-5MS(30m×0.25mm×0.25 μm)作为第一维柱,较短的中等极性柱DB-17MS(2m×0.1mm×0.1 μm)作为第二维柱,利用固相微萃取法作为香味成分的萃取方法,对薄荷型ESSE卷烟的核心香味成分进行了定性分析,TOFMS谱图库检索结合全二维特有的包含结构信息的二维谱图,通过族分离和结构谱图鉴定,共鉴定了187种挥发性成分,其中对香气有贡献的成分118种。

## 中心切割二维 GC/MS 定性分析卷烟烟气中的杂环烃

《烟草科技》2011 年 第 9 期 | 张艳芳, 谢复炜, 黄琪, 苏国岁, 杨运红, 陈伟华

河北中烟工业公司技术中心 石家庄 050051

中国烟草总公司郑州烟草研究院 郑州 450001

黑龙江烟草工业有限责任公司 哈尔滨 150001

**摘要:**建立了二氯甲烷超声萃取、碱性氧化铝固相萃取柱净化和中心切割二维气相色谱质谱联用法定性分析卷烟主流烟气中杂环烃类化合物的方法,并采用该方法与一维气相色谱质谱联用法的分析结果进行了比较。结果表明:1. 中心切割技术能有效提高色谱的分离能力,分离效果明显优于一维色谱; 1. 显著增加目标化合物的质谱匹配度,进而为质谱的准确定性定量提供了良好条件。

## 全二维气相色谱技术及其在烟草行业中的应用

《云南化工》2011 年 第 1 期 | 伊奥尔 孟昭宇

红塔烟草集团有限责任公司技术中心 云南玉溪 653100

**摘要:**全二维气相色谱(GC×GC)是近些年来发展起来的一门新技术,它允许所有的样品进入到两根完全不同的柱子中进行分离,使其峰容量约等于两根柱各自峰容量的乘积,而且灵敏度显著提高,非常适合用于复杂样品的分析。介绍了全二维气

相色谱的工作原理、特点及仪器构成，主要总结了其最关键部分调制器的发展，并综述了该技术在烟草行业中的应用。

## 溶剂萃取-中心切割多维色谱-质谱法测定烟草主要中性香味成分

《烟草科技》2013年 第7期 | 王晔 孙文梁 苏庆德 刘百战

上海烟草集团有限责任公司技术中心 上海 200082

中国科学技术大学烟草与健康中心 合肥 230022

**摘要：**采用溶剂萃取-中心切割多维气相色谱-质谱技术(MDGC/MS)建立了一种烟草主要中性香味成分的分析方法。0.5 g烟样经5 mL乙醚萃取和2 mL 5% H<sub>2</sub>SO<sub>4</sub>酸洗，静置过夜后萃取液用多维气相色谱-质谱分析；以甲基丁子香酚为内标，对茄酮、β-大马酮、香叶基丙酮、β-紫罗兰酮、巨豆三烯酮和二氢猕猴桃内酯等中性香味成分进行了定性定量测定，并与同时蒸馏萃取(SDE)-气质联用(GC/MS)法进行比较。结果表明：各待测组分检出限在0.02~0.18 μg/mL之间，回收率为80.0%~123.4%，日内相对标准偏差≤7.5%。该方法快速、简便、准确，适用于烟草中性香味成分的批量测试。

## 基于顶空固相微萃取-全二维气相飞行时间质谱快速检测烟草挥发性及半挥发性生物碱

《分析试验室》2014年 第11期 | 向章敏 蔡凯 张婕 葛永辉 周淑平 郭玉双 耿召良

贵州省烟草科学研究院 贵阳 550081

**摘要：**通过正交试验设计与单因素试验设计相结合，建立了顶空固相微萃取-全二维气相飞行时间质谱快速分析烟草挥发性及半挥发性生物碱的方法。烟叶样品在70℃条件下平衡20 min，然后采用PDMS/DVB/CAR纤维头在顶空瓶中平衡15 min，最后在250℃进样口解析3 min效果最好。以喹啉为内标，分别对烟草中烟碱、麦斯明和二烯烟碱3种生物碱的挥发性成分进行定性定量分析，其方法的线性相关系数( $r^2$ )为0.9944~0.9992，检出限为0.60~150 pg，添加回收率为86.7%~98.3%，相对标准偏差为1.8%~4.2%。通过实际烟草样品检测表明，不同生物碱的挥发性与总量具有显著相关性。

## 在线液相-气相二维色谱测定卷烟主流烟气中的苯并[a]芘

《分析化学》2016年 第2期 | 刘春波 申钦鹏 张凤梅 张安丰 何沛 司晓喜 刘志华 杨光宇 缪明明

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**摘要：**建立了在线液相-气相二维色谱测定卷烟主流烟气中的苯并[a]芘方法。采用剑桥滤片捕集烟气粒相物，环己烷萃取，以 D12-苯并[a]芘为内标，然后用在线液相-气相二维色谱测定：样品直接进样进入液相色谱，经微型硅胶柱分离，含苯并[a]芘的部位切割进入气相色谱，排干溶剂后启动气相色谱升温，经毛细管柱进行分离，用质谱检测。本方法将烟气苯并[a]芘测定中的硅胶柱层析和气相色谱-质谱分析在线连接起来，可不经样品前处理净化直接进样分析；而且每次进样可达 40#L，与常规气相色谱-质谱分析最大进样 2.0#L 相比，分析灵敏度提高了 20 倍。方法线性范围达 0.08~50 ng/L，相关系数为  $r^2=0.999$ ，回收率为 94.2%~105.5%；检出限和定量限分别为 0.09 和 0.30 ng/支，应用本方法对 14 个不同类型市售卷烟和 2R4F 标准烟进行了测定，结果与 GB/T21130-2007 测定结果相符合。

## 4. 环境业—Environmental Industry

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### 环境监测—Environment Monitoring

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Wardlaw, G.D., Nelson, R.K., Reddy, C.M., Valentine, D.L.

Biodegradation preference for isomers of alkylated naphthalenes and benzothiophenes in marine sediment contaminated with crude oil

(2011) *Organic Geochemistry*, 42 (6), pp. 630-639.

**ABSTRACT:** Contamination of coastal marine sediments with polycyclic aromatic hydrocarbons is pervasive, with major sources including anthropogenic activity and natural seepage. Biodegradation serves as a major hydrocarbon sink with evaporation and dissolution responsible for the removal of low boiling range compounds and photo-oxidation acting on many multi-substituted aromatic compounds. In this work, first-order rate constants for aerobic biodegradation were quantified for naphthalene (N), benzothiophene (BT) and their alkylated congeners (1-4 carbon substituents (C1-C4)) in laboratory experiments with oil laden marine sediments from a natural seep. Rate constants were used as proxies for microbial preference, which follows the order:

naphthalene > C1N > C2N > C1BT > C2BT > benzothiophene > C3BT > C3N > C4BT > C4N, with some overlap. The application of comprehensive two dimensional gas chromatography further enabled separation and quantification of multiple structural isomers for C2N-C4N and C2BT-C4BT, with 7-12 isomers resolved for each C2N-C4N and 4-7 isomers resolved for each C2BT-C4BT. A strong isomeric biodegradation preference was noted within each of these compound classes, with rate constants varying as much as a factor of 2 for structural isomers of the same compound class. Each isomer was consumed to a low, but non-zero concentration, suggesting that their residual load in sediment may be proportional to the number of structural isomers originally present, in addition to the pattern and the number of alkyl substitutions. The simultaneous first-order biodegradation rates observed for 52 aromatic hydrocarbons resolved in this study, along with reference compounds such as n-alkanes, lends support to the concept of broad scale metabolic specificity during aerobic biodegradation of petroleum. © 2011 Elsevier Ltd.

**Blase, R.C., Patrick, E.L., Mitchell, J.N., Libardoni, M.**

**Analysis of cave atmospheres by comprehensive two-dimensional gas chromatography (GC $\times$ GC) with flame ionization detection (FID)**

**(2015) Analytical Chemistry Research, 3, pp. 54-62.**

**ABSTRACT:** In this paper, we describe a simple method for sampling, pre-concentrating, and separating volatile and semi-volatile components from two different cave atmospheres. Sampling is performed by capturing a volume of cave atmosphere in a Tedlar bag or Suma canister for sample storage and transport back to the laboratory. Loading a portion of the sample on a multi-bed sorption trap allows for sample pre-concentration prior to separation and detection of components on a comprehensive two-dimensional gas chromatograph (GC $\times$ GC). Comparison of two Texas caves reveals the power of comprehensive two-dimensional gas chromatography (GC $\times$ GC) for volatile separation and detection, and to our knowledge marks the first use of GC $\times$ GC for the analysis of cave atmospheres. Analysis of the results revealed 138 and 146 chromatographic signals over an S/N threshold of 500 and direct comparison of the two samples revealed 50 identical chromatographic signals. This study is a first step toward demonstrating the ability of GC $\times$ GC to separate the complex volatiles and semi-volatiles in the cave atmosphere as a fingerprinting tool. © 2014 The Authors.

## 二维气相色谱法分析天然气水合物区沉积物间隙水中示踪气体的浓度

《色谱》2011年 第1期 | 王虎 杨群慧 季福武 周怀阳 薛翔

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**摘要：**利用微流路控制技术中心切割装置(Deans Switch)、两根色谱柱(PoraPLOT Q 和 Molsieve5A)和 3 个检测器(脉冲氦离子化检测器、火焰光度检测器、热导检测器)，建立了一种二维气相色谱分析系统，实现了海洋中多种示踪气体组分(氢气、甲烷、二氧化碳、硫化氢)的同时分析和精确测定。氢气、甲烷、二氧化碳、硫化氢的含量分别在 2–1 030、0.6–501、120–10 500 和 0.2–49.1  $\mu\text{mol/mol}$  范围内的校正曲线线性关系良好，检出限分别为 0.51、0.17、82 和 0.08  $\mu\text{mol/mol}$ ，10 次重复测定含量的相对标准偏差均小于 10%。通过对南海天然气水合物区沉积物间隙水顶空气的测定，表明该方法方便、灵敏、可靠，易于实现海上现场测定；与以往采用多种分析方法分别测定示踪气体相比，大大节省了样品量。该方法适用于海洋天然气水合物、海底热液等资源的调查和海洋溶解态气体的研究等。

## 全二维气相色谱技术在痕量污染物分析中的应用进展

《色谱》2011年 第4期 | 郑明辉，吴嘉嘉

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## 二维气相色谱法测定空气中的总烃及非甲烷总烃

《污染防治技术》2011年 第2期 | 於香湘 吴鹏 缪建军

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**摘要：**采用单一进样口六通阀进样、毛细管柱二维气相色谱仪三通路分离塔柱分离系统、采用氢离子化检测器分别产生信号，测定环境空气中的非甲烷总烃。通过柱分离系统将样品平均分配到不同类型的两个毛细管色谱柱，分别测定总烃及甲烷。方法简单、快速，最大程度地保证了进样的一致性，从而保证了分析结果的准确性。

## 大气有机物热脱附-全二维气相色谱-火焰离子化分析方法

《中国科学：化学》2012 年 第 2 期 | 王瑛 徐晓斌 毛婷 张康平 程红兵 印丽媛  
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**摘要：**全二维气相色谱(GC×GC)是 20 世纪 90 年代发展起来的具有高分辨率、高灵敏度、高峰容量等优势的分离技术，在我国将其用于大气挥发性有机物(VOCs)研究方面才刚刚起步。本文将 GC×GC 与氢火焰离子化检测器(FID)联用，构建了用于测量大气有机物的热脱附-全二维气相色谱-氢火焰离子化分析系统(TD-GC×GC-FID)。采用 HP-5MS 和 HP-INNOWAX 色谱柱，建立了 C<sub>5</sub>~C<sub>15</sub> 大气有机物分析方法，实现了一次分析过程同时分离非甲烷烃(NMHCs)、含氧挥发性有机物(OVOCs)和卤代烃等多种组分。利用标准物质和四级杆质谱(qMS)进行定性，外标法结合 FID 质量校正因子定量。目标物在 GC×GC 谱图中第一和第二维保留时间变化分别小于 0.6 s 和 0.02 s，峰体积平均相对标准偏差为 14.3%，其中烷烃和芳香烃为 4.5%。标准曲线  $r^2$  均值大于 0.99，平均检出限为 6.04 ng，平均回收率为 111%。利用该方法检测到 2010 年 1 月北京市区大气中 400 多种有机物(信噪比大于 50)，鉴定了其中的 103 种物质，包括烷烃、烯烃、芳香烃、卤代烃、醛、酮、酯、醇和醚等。所测定有机物平均总浓度为  $51.3 \times 10^{-9}$  V/V，其中 OVOCs 约占 51%，芳香烃约占 30%，烷烃约占 15%，卤代烃和烯烃分别占 3% 和 1%。平均浓度最高的前 3 个组分是乙醇( $9.84 \times 10^{-9}$  V/V)、丙酮( $6.72 \times 10^{-9}$  V/V)和甲苯( $3.48 \times 10^{-9}$  V/V)。

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## 地质分析—Geological Analysis

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Li, S., Cao, J., Hu, S., Luo, G.

Characterization of compounds in unresolved complex mixtures (UCM) of a  
Mesoproterzoic shale by using GC×GC-TOFMS

(2015) Marine and Petroleum Geology, 66, pp. 791-800.

**ABSTRACT:** Unresolved complex mixtures (UCM) in shales have important implications for organic matter formation and diagenesis. However, their origin is not well understood, one important reason for this is lacking the knowledge of their molecular compositions. Here, using the comprehensive two-dimensional gas chromatography (GC×GC) coupled with time-of-flight mass spectrometry

(TOFMS), we conduct a pilot study based on a Mesoproterozoic black shale from North China. Detailed identification of compounds was made and a possible new origin was suggested. Results show that the separation of UCM hydrocarbons by a reversed-phase column system (polar/non-polar) is efficient for the identification of molecular composition of UCM. The UCM in the shale is composed mainly of C11-C27 compounds, including paraffin hydrocarbons, cycloalkanes and aromatics, in particular 1-3 cyclic paraffins with a short-chain alkyl group, multi-branched isoparaffins and their homologous series, which are indicative of isomerization of molecules intensively. This implies that the origin of the UCM might include the isomerization of organic molecules in addition to the commonly-thought microbial activities. This study improves the understanding of the geochemical compositions and origin of UCM in shales. © 2015 Elsevier Ltd.

## 中国石化无锡石油地质研究所实验地质技术之全二维色谱飞行时间质谱分析技术

《石油实验地质》2010年 第4期 | 蒋启贵

**摘要:** 分析仪器为美国力可公司(LECO)的全二维气相色谱-飞行时间质谱联用系统(GC×GC—TOFMS)，由Agilent 7890气相色谱仪和LECO公司的PEGASUS4D飞行时间质谱仪组成。全二维色谱飞行时间质谱的分析原理为：将分离机理不同而又互相独立的两根色谱柱以串联的方式结合成二维色谱，经第一支色谱柱分离后的馏分，经调制器聚焦后以脉冲方式进入第二支色谱柱中进行进一步分离。

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## 挥发性有机物分析—VOCs Analysis

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**Liu, J., Seo, J.H., Li, Y., Chen, D., Kurabayashi, K., Fan, X.**  
**Smart multi-channel two-dimensional micro-gas chromatography for rapid workplace hazardous volatile organic compounds measurement**  
**(2013) Lab on a Chip - Miniaturisation for Chemistry and Biology, 13 (5), pp. 818-825.**

**ABSTRACT:** We developed a novel smart multi-channel two-dimensional (2-D) micro-gas chromatography ( $\mu$ GC) architecture that shows promise to significantly improve 2-D  $\mu$ GC performance. In the smart  $\mu$ GC design, a

non-destructive on-column gas detector and a flow routing system are installed between the first dimensional separation column and multiple second dimensional separation columns. The effluent from the first dimensional column is monitored in real-time and decision is then made to route the effluent to one of the second dimensional columns for further separation. As compared to the conventional 2-D µGC, the greatest benefit of the smart multi-channel 2-D µGC architecture is the enhanced separation capability of the second dimensional column and hence the overall 2-D GC performance. All the second dimensional columns are independent of each other, and their coating, length, flow rate and temperature can be customized for best separation results. In particular, there is no more constraint on the upper limit of the second dimensional column length and separation time in our architecture. Such flexibility is critical when long second dimensional separation is needed for optimal gas analysis. In addition, the smart µGC is advantageous in terms of elimination of the power intensive thermal modulator, higher peak amplitude enhancement, simplified 2-D chromatogram re-construction and potential scalability to higher dimensional separation. In this paper, we first constructed a complete smart  $1 \times 2$  channel 2-D µGC system, along with an algorithm for automated control/operation of the system. We then characterized and optimized this µGC system, and finally employed it in two important applications that highlight its uniqueness and advantages, i.e., analysis of 31 workplace hazardous volatile organic compounds, and rapid detection and identification of target gas analytes from interference background. © 2013 The Royal Society of Chemistry.

**Gaquerel, E., Baldwin, I.T.**

**Honing in on phenotypes: Comprehensive two-dimensional gas chromatography of herbivory-induced volatile emissions and novel opportunities for system-level analyses**  
**(2013) AoB PLANTS, 5, art. no. plt002, .**

**ABSTRACT:** Plant volatile organic compound (VOC) production requires a complex network of biochemical pathways, which, although well mapped from a biochemical point of view, remains only partly understood with regard to its physiological and genetic regulation. Additionally, although analytical procedures for plant VOC measurement have become increasingly faster and more sensitive in recent years, pinpointing relevant shifts in VOC production from the thousands of molecular fragments that are generated by modern mass

spectrometer instruments remains challenging. Here we discuss novel opportunities for system-wide analysis provided by the implementation of non-targeted data processing and multivariate statistics in VOC analysis. We illustrate the value of implementing non-targeted data processing with examples of recent findings from our group on the interactive control exerted by salivary components of a lepidopteran herbivore, *Manduca sexta*, on herbivory-induced VOC emissions in the wild tobacco *Nicotiana attenuata*. Finally, we briefly discuss the use of multi-platform data integration for probing the nature of metabolic and regulatory systems underlying VOC emissions. © The Authors 2013.

### 全二维气相色谱-飞行时间质谱法分离复杂体系中 7 种指示剂多氯

#### 联苯

《中国环境监测》 2015 年 01 期 | 周伟峰 黄旭锋 郑浩 姚林江 王林

郑州市环境监测中心站

北京博赛德科技有限公司

**摘要：**采用全二维气相色谱-飞行时间质谱法(GC×GC TOF-MS)分析了混标样品Aroclor 1260、Aroclor 1254、Aroclor 1242中的多氯联苯(PCBs)单体，考察了在复杂体系下对7种PCBs指示剂的分离能力。结果表明，与优化柱系统的分离效果相比，136种单体的混标样品中共分离出121种单体，7种指示剂经一维和二维的保留时间及质谱定性，在121种单体的2D斑点图中清晰可辨。全二维对于复杂体系的PCBs 和指示剂的分离表明其强大的分离能力和对于检测复杂体系中指示剂的分辨能力，对该类物质的定性和定量检测具有重要意义。

## 5. 农业—Agricultural Industry

Khan, Z.S., Ghosh, R.K., Girame, R., Utture, S.C., Gadgil, M., Banerjee, K.,  
Reddy, D.D., Johnson, N.

**Optimization of a sample preparation method for multiresidue analysis of pesticides in tobacco by single and multi-dimensional gas chromatography-mass spectrometry**

(2014) **Journal of Chromatography A**, 1343, pp. 200-206.

**ABSTRACT:** A selective and sensitive multiresidue analysis method, comprising 47 pesticides, was developed and validated in tobacco matrix. The optimized sample preparation procedure in combination with gas chromatography mass spectrometry in selected-ion-monitoring (GC-MS/SIM) mode offered limits of detection (LOD) and quantification (LOQ) in the range of 3-5 and 7.5-15. ng/g, respectively, with recoveries between 70 and 119% at 50-100. ng/g fortifications. In comparison to the modified QuEChERS

(Quick-Easy-Cheap-Effective-Rugged-Safe method: 2. g tobacco. +. 10. ml water. +. 10. ml acetonitrile, 30. min vortexing, followed by dispersive solid phase extraction cleanup), the method performed better in minimizing matrix co-extractives e.g. nicotine and megastigmatrienone. Ambiguity in analysis due to co-elution of target analytes (e.g. transfluthrin-heptachlor) and with matrix co-extractives (e.g. δ-HCH-neophytadiene, 2,4-DDE-linolenic acid) could be resolved by selective multi-dimensional (MD)GC heart-cuts. The method holds promise in routine analysis owing to noticeable efficiency of 27 samples/person/day. © 2014 Elsevier B.V.

固相萃取-全二维气相色谱/飞行时间质谱同步快速检测蔬菜中 64

**种农药残留**

《分析化学》2011 年 第 1 期 | 姜俊 李培武 谢立华 丁小霞 李英 王秀嫔 汪雪芳  
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农业部油料及制品质量监督检验测试中心 武汉 430062

**摘要:**建立了全二维气相色谱/飞行时间质谱法(GC×GC-TOFMS)快速定性筛查食品样品中 19 种防腐剂和抗氧化剂、13 种超范围使用的非法添加物的方法。以 40 mL 乙腈为溶剂, 对 20 g 均质样品进行液体萃取, 萃取液经过离心过滤后, 使用无水 MgSO<sub>4</sub> 脱水(含油样品使用 40 mL 正己烷去除油脂); 萃取液再用乙腈稀释 5 倍进样。本方法采用 GC×GC 二维特征谱图、TOFMS 谱图库检索的定性手段, 能在 20.5 min 内快速分离和定性分析食品中 19 种防腐剂和抗氧化剂。使用 GC×GC-TOFMS 技术对

含量大于 0.3 mg/kg 的样品有较好的灵敏度，而在食品添加剂领域，添加量一般都大于 100 mg/kg。因此，本方法可以作为一种快速定性筛查的手段。

## 全二维气相色谱-电子捕获检测器法分析土壤中毒杀芬同类物的 残留

《分析化学》2012 年 第 8 期 | 张兵 郑明辉 刘国瑞 李成 高丽荣  
中国科学院生态环境研究中心 北京 100085

**摘要：**建立了全二维气相色谱-电子捕获检测器(GC×GC- $\mu$ ECD)检测土壤中毒杀芬同类物的分析方法。以非极性的 DB-XLB(20 m×0.25 mm×0.25  $\mu$ m)为第一色谱柱，中等极性的 BPX-50(2 m×0.1 mm×0.1  $\mu$ m)为第二色谱柱，对土壤中 23 种高关注毒杀芬同类物进行了分离鉴定，并采用基质曲线外标法进行定量分析。本方法在 1~200  $\mu$ g/L 浓度范围内，毒杀芬同类物的线性相关系数( $r^2$ )均大于 0.99，方法检出限(S/N=3)为 0.039~0.482  $\mu$ g/L，基质加标毒杀芬同类物的回收率为 55%~115%，相对标准偏差(RSD)均小于 30%(n=5)。利用本方法对毒杀芬污染的土壤样品进行了测定，获得了较好的分离效果。

## 全二维气相色谱-质谱法定性分析土壤中有机氯农药、酞酸酯和多 氯联苯

《分析试验室》2013 年 第 2 期 | 李燕群 张渝 钱蜀 但德忠 杨坪  
四川大学建筑与环境学院 成都 610065  
四川省环境监测中心站 成都 610041

**摘要：**建立了土壤中 20 种有机氯农药、16 种酞酸酯和 18 种多氯联苯的全二维气相色谱-质谱分离和定性方法。土壤样品与无水 Na<sub>2</sub>SO<sub>4</sub>(1:1, m/m)混匀后，用丙酮、正己烷(1:1, V/V)在微波快速溶剂萃取仪上以 1600W 的功率，在 20min 内从室温升至 110℃，并保持 20min 的条件进行萃取。通过优化升温速率、调制周期及热喷时间等参数，该方法能够在 40.5min 内分离目标化合物及土壤萃取杂质，并通过二维谱图和质谱检索对 54 种目标化合物准确定性。

## 固相萃取-多维气相色谱-串联质谱法测定粮谷中 31 种农药残留

《环境化学》2016 年 第 1 期 | 吴岩 姜冰 张辛博 赵伟 潘仲乐 李丽丽 韩  
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东北林业大学森林植物生态学教育部重点实验室

黑龙江出入境检验检疫局  
黑龙江省环境监测中心站  
岛津企业管理(中国)有限公司

**摘要:**建立了粮谷中六六六、滴滴涕、硫丹、氯丹、稻瘟灵及氯菊酯等31种有机氯农药和拟除虫菊酯农药残留的多维气相色谱-串联质谱(MDGC-MS/MS)分析方法。样品加适量水溶胀后,以乙腈为提取剂,经高速匀浆方法提取2次,提取液经浓缩后进行净化处理。比较了固相萃取与凝胶渗透色谱两种不同净化技术的净化效果,最终确定采用Carbon-NH<sub>2</sub>固相萃取为净化手段,有效除去样品中大部分的脂肪和大分子蛋白质等干扰基质,再经MDGC-MS/MS分析,通过中心切割的方式将农药组分选择性切割进入多维色谱柱进行进一步分离,有效地降低了样品中的复杂基质所带来的背景干扰。加标水平为5、10、20 μg·kg<sup>-1</sup>时,大部分农药的回收率为66%-108%,相对标准偏差小于15%。3种农药的检出限为0.5-5 μg·kg<sup>-1</sup>。采用GC-MS/MS定性分析、外标法定量分析,线性关系和回收率结果均满意。

## 6. 药物学—Pharmacology

### 二维气相色谱-飞行时间质谱分析不同产地的川芎挥发油

《色谱》2010年 第4期 | 王楠 张艺 李响 童应鹏 孔宏伟 许国旺  
中国科学院大连化学物理研究所 中国科学院分离分析重点实验室 辽宁大连  
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**摘要:**建立了川芎挥发油的全二维气相色谱/飞行时间质谱(GC×GC/TOFMS)指纹图谱,并结合聚类分析评价了川芎药材的质量。4种萜类和苯酞类物质在DB-Petro×DB-17柱系统上实现了明显的族组分分离。从新都县的1个样品中分离出375种组分,其中相似度、反相似度皆大于800的化合物215个;根据质谱库检索和保留指数验证,或参照标准化合物及文献报道,从中定性了43个化合物。结合偏最小二乘法-判别分析,可以很好地将4个产区的挥发油样品加以区分,并找出20种差异最大的化合物,其中包括4种苯酞类物质。在此基础上,进一步应用正交偏最小二乘法,关联化学成分和抗氧化活性,结果发现,苯酞类物质(如藁本内酯、川芎内酯A和新蛇床内酯)对川芎挥发油样品地区差异的影响最大,其中彭州产川芎

样品中苯酞类物质的含量最高。本文通过高分辨色谱技术研究了相邻产地的药物化学组成差异，并将化学指纹及生物学活性相关联，建立了系统的中药质量评价及活性化合物筛选研究模式。

### 多维气相色谱及其在中药领域中的研究进展

《中国农学通报》2010 年 第 15 期 | 吴剑威 匡莹 赵润怀 陈波  
中国医学科学院北京协和医学院药用植物研究所 北京 100193  
中国药材集团公司科技研发部 北京 102600

**摘要：**多维气相色谱是近年来发展较为迅速的一种色谱分离技术，其高分辨、高效、高峰容量以及易与质谱等其他技术联用等优势正成为研究应用的热点。对多维气相色谱技术及其近年来在中药研究中的应用进行了综述，并对多维气相色谱的发展趋势和前景进行了展望。

### 全二维气相色谱技术及其在药物分析中的应用

《心理医生》2012 年 第 7 期 | 吴军凯 谢海龙 于丹  
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**摘要：**全二维气相色谱(GC×GC)是近年发展起来的一种多维色谱分离技术。本文对全二维气相色谱的原理、特点及其在药物分析领域的应用进行了综述，该技术在中药及天然药物挥发性成分分析领域具有广阔的应用前景。

### 多维气相色谱及其在中药领域中的研究

《求医问药：下半月刊》2012 年 第 6 期 | 刘敏  
江苏省丹阳市人民医院 江苏丹阳 212300

**摘要：**多维气相色谱是近年来发展较为迅速的一种色谱分离技术，其高分辨、高效、高峰容量以及易与质谱等其他技术联用等优势正成为研究应用的热点。对多维气相色谱技术及其近年来在中药研究中的应用进行了综述，并对多维气相色谱的发展趋势和前景进行了展望。

### 二维色谱技术及其在中药领域中的应用

《中国科学：化学》 2013 年 第 11 期 | 沈保家 秦昆明 刘启迪 蔡皓 刘晓 蔡宝昌  
南京中医药大学国家教育部中药炮制规范化及标准化工程研究中心  
国家中医药管理局中药炮制标准重点研究室

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**摘要:**二维色谱是近年发展起来的多维色谱分离技术,因其高分辨率、高峰容量、高灵敏度等优点,在复杂样品的分离分析中发挥了巨大作用。中药是一个复杂的化学体系,包含多种类化学成分,因此,分析和阐明中药中的化学成分是中药现代研究的一个关键问题。二维色谱技术在中药等复杂体系的应用中显示了重要的应用价值,也具有广阔的发展前景。本文对二维色谱的分类、主要部件以及在中药领域中的应用进行综述,重点介绍二维气相色谱和二维液相色谱在中药研究中的应用。

## 全二维气相色谱-飞行时间质谱法分析广藿香浸膏中挥发性有机物

《理化检验: 化学分册》2015 年 第 3 期 | 邹西梅 金晶 万强 刘剑 杨靖

贵州中烟工业有限责任公司技术中心

郑州轻工业学院食品与生物工程学院

**摘要:**应用全二维气相色谱-飞行时间质谱法(GC×GC-TOFMS)分析广藿香浸膏的挥发性有机物。采用超临界 CO<sub>2</sub> 流体萃取广藿香样品获得广藿香浸膏,浸膏经分子蒸馏分离,所得的轻馏分经 GC×GC-TOFMS 分析。二维色谱由 Rxi-5sil MS 柱和 Rtx-200 柱串联而成,通过 TOFMS 软件结合质谱分析对化合物进行定性定量分析。分析得到匹配度大于 750 的组分有 323 个,含量占总量的 89.45%,其中酮类 67 个、醇类 52 个、烯类 40 个、酸类 37 个、烷类 36 个、酯类 34 个、醛类 17 个,其它类 40 个。相对含量大于 0.1% 的组分 69 个,其中相对含量大于 1% 的组分 11 个,广藿香酮和广藿香醇为主要成分,质量分数分别为 21.75% 和 18.93%。

## 全二维 GC×GC-qMS 用于紫苏叶挥发油的成分分析

《中国化学会第二届全国质谱分析学术报告会会议摘要集》2015 年 | 孙谦 范

军 黄涛宏 端裕树

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**摘要:**紫苏叶为唇形科紫苏属植物紫苏 *Perilla frutescens* (L.) Brit. 的干燥叶,是一种常用的辛温解表药,具有解表散寒、行气和胃等功效,用于治疗风寒感冒、咳嗽呕恶、妊娠呕吐、鱼蟹中毒等症[1]。紫苏叶中含有丰富的挥发油,不仅具有药用价值,而且可以作为香料添加到食品、烟草、化妆品中[2]。紫苏叶挥发油成分一般采用气相色谱-质谱(GC-MS)法,但由于紫苏叶挥发油是一个复杂的分析体系,其中性质相似的组分保留时间较近,色谱重叠严重,在色谱峰的识别和定量上常会遇到较大困难[3]。全二维气相色谱(GC×GC)是 20 世纪 90 年代发展起来的一种分离复

杂混合物的全新手段,它把分离机理不同而又相互独立的两根色谱柱通过调制器(或称调制解调器)以串联方式连接在一起的二维气相色谱柱系统。全二维气相色谱比普通一维气相色谱具有分辨率更高、峰容量大、灵敏度好、分析速度快等优点[4]。目前,全二维气相色谱已在食品[5]、石化产品[6]、香精[7]、环境研究[8]等多个领域得到应用。本实验将全二维气相色谱质谱联用法(GC×GC-q MS)应用于紫苏叶挥发油成分的分析。结果表明,GC×GC-q MS为紫苏叶挥发油成分的分析提供了很好的手段(图 1),这对于紫苏叶挥发油的药用成分及食用成分的研究提供借鉴,同时也可对不同产地的紫苏叶挥发油进行分类。

## 7. 生物化学—Biochemistry

Almstetter, M.F., Oefner, P.J., Dettmer, K.

Metabolic fingerprinting using comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry  
(2012) Methods in Molecular Biology, 815, pp. 399-411.

**ABSTRACT:** Comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry (GC × GC-TOF-MS) is applied to the comparative metabolic fingerprinting of physiological fluids. Stable isotope-labeled internal standards plus norvaline serve as extraction standards and are added to the blanks, controls and patient samples prior to protein precipitation with methanol. The extracts are evaporated to complete dryness and derivatized in two steps using methoximation with methoxylamine hydrochloride (MeOx) and silylation with N-methyl-N-trimethylsilyl-trifluoroacetamide (MSTFA). Between derivatization steps a second internal standard containing odd-numbered, saturated straight chain fatty acids is added for quality control and to normalize retention time shifts. After GC × GC-TOF-MS analysis raw data are processed, aligned, and combined in one data matrix for subsequent statistical evaluation. Both a custom-made and the NIST 05 library are used to preliminarily identify significant metabolites. For verification purposes, commercial standards are run individually. Absolute

quantification of selected metabolites is achieved by using a multi-point calibration curve and isotope-labeled internal standards.

**Caldeira, M., Perestrelo, R., Barros, A.S., Bilelo, M.J., Morête, A., Câmara, J.S., Rocha, S.M.**

**Allergic asthma exhaled breath metabolome: A challenge for comprehensive two-dimensional gas chromatography**

(2012) *Journal of Chromatography A*, 1254, pp. 87-97.

**ABSTRACT:** Allergic asthma represents an important public health issue, most common in the paediatric population, characterized by airway inflammation that may lead to changes in volatiles secreted via the lungs. Thus, exhaled breath has potential to be a matrix with relevant metabolomic information to characterize this disease. Progress in biochemistry, health sciences and related areas depends on instrumental advances, and a high throughput and sensitive equipment such as comprehensive two-dimensional gas chromatography-time of flight mass spectrometry ( $\text{GC} \times \text{GC-TOFMS}$ ) was considered.  $\text{GC} \times \text{GC-TOFMS}$  application in the analysis of the exhaled breath of 32 children with allergic asthma, from which 10 had also allergic rhinitis, and 27 control children allowed the identification of several hundreds of compounds belonging to different chemical families.

Multivariate analysis, using Partial Least Squares-Discriminant Analysis in tandem with Monte Carlo Cross Validation was performed to assess the predictive power and to help the interpretation of recovered compounds possibly linked to oxidative stress, inflammation processes or other cellular processes that may characterize asthma. The results suggest that the model is robust, considering the high classification rate, sensitivity, and specificity. A pattern of six compounds belonging to the alkanes characterized the asthmatic population: nonane, 2,2,4,6,6-pentamethylheptane, decane, 3,6-dimethyldecane, dodecane, and tetradecane. To explore future clinical applications, and considering the future role of molecular-based methodologies, a compound set was established to rapid access of information from exhaled breath, reducing the time of data processing, and thus, becoming more expedite method for the clinical purposes. © 2012 .

**Dolejšová, K., Krasulová, J., Kutalová, K., Hanus, R.**

**Chemical alarm in the termite *Termitogeton planus* (Rhinotermitidae)**

(2014) *Journal of Chemical Ecology*, 40 (11-12), pp. 1269-1276.

**ABSTRACT:** Effective defense is a common characteristic of insect societies. Indeed, the occurrence of specialized defenders, soldiers, has been the first step toward eusociality in several independent lineages, including termites. Among the multitude of defensive strategies used by termite soldiers, defense by chemicals plays a crucial role. It has evolved with complexity in advanced isopteran lineages, whose soldiers are equipped with a unique defensive organ, the frontal gland. Besides direct defense against predators, competitors, and pathogens, the chemicals emitted by soldiers from the frontal gland are used as signals of alarm. In this study, we investigated the chemical composition of the defensive secretion produced by soldiers of the termite *Termitogeton planus* (Isoptera: Rhinotermitidae), from West Papua, and the effects of this secretion on the behavior of termite groups. Detailed two-dimensional gas chromatography/mass spectrometry analyses of the soldier defensive secretion revealed the presence of four linear and nine monoterpene hydrocarbons. Soldier head extracts, as well as synthetic mixtures of the monoterpenes found in these extracts, elicited alarm behavior in both soldiers and pseudergates. Our results suggest that the alarm is not triggered by a single monoterpene from the defensive blend, but by a multi-component signal combining quantitatively major and minor compounds. © 2014, Springer Science+Business Media New York.

**Lamani, X., Horst, S., Zimmermann, T., Schmidt, T.C.**

**Determination of aromatic amines in human urine using comprehensive multi-dimensional gas chromatography mass spectrometry (GCxGC-qMS)**  
**(2015) Analytical and Bioanalytical Chemistry, 407 (1), pp. 241-252.**

**ABSTRACT:** Aromatic amines are an important class of harmful components of cigarette smoke. Nevertheless, only few of them have been reported to occur in urine, which raises questions on the fate of these compounds in the human body. Here we report on the results of a new analytical method, in situ derivatization solid phase microextraction (SPME) multidimensional gas chromatography mass spectrometry (GCxGC-qMS), that allows for a comprehensive fingerprint analysis of the substance class in complex matrices. Due to the high polarity of amino compounds, the complex urine matrix and prevalence of conjugated anilines, pretreatment steps such as acidic hydrolysis, liquid-liquid extraction (LLE), and derivatization of amines to their corresponding aromatic iodine compounds are necessary. Prior to detection, the derivatives were enriched by

headspace SPME with the extraction efficiency of the SPME fiber ranging between 65 % and 85 %. The measurements were carried out in full scan mode with conservatively estimated limits of detection (LOD) in the range of several ng/L and relative standard deviation (RSD) less than 20 %. More than 150 aromatic amines have been identified in the urine of a smoking person, including alkylated and halogenated amines as well as substituted naphthylamines. Also in the urine of a non-smoker, a number of aromatic amines have been identified, which suggests that the detection of biomarkers in urine samples using a more comprehensive analysis as detailed in this report may be essential to complement the approach of the use of classic biomarkers. © The Author(s) 2014.

## 血清中二噁英类物质的同位素稀释-全二维气相色谱-串联质谱法 研究

《食品安全质量检测学报》2015年 第3期 | 尹帅星 吕冰 张磊 李敬光 吴永宁  
武汉轻工大学食品科学与工程学院 农产品加工湖北省协同创新中心 国家食品安全  
风险评估中心卫生部食品安全风险评估重点实验室

**摘要：**目的建立高灵敏度和高选择性的全二维气相色谱-串联质谱法(GC×GC-MS/MS)测定17种多氯代二苯并二噁英和多氯代二苯并呋喃(PCDD/Fs)同系物的痕量分析方法。方法将牛血清样品经过加速溶剂萃取，全自动净化仪净化后，通过配备DB-5MS和BPX-50柱全二维气相色谱-串联质谱进行样品分析。结果17种PCDD/Fs同系物的标准曲线在范围内显示良好线性( $R^2 > 0.99$ )。方法的检出限为0.1~0.8 pg/g。结论本方法满足血清样品中PCDD/Fs的痕量分析需求。

## 采用 GC×GC-TOFMS 结合顶空固相微萃取技术检测枯草芽孢杆菌 发酵液中吡嗪类物质

《食品科学》2016年 第6期 | 周庆伍 汤有宏 徐祥浩 刘国英 高江婧 姜利  
安徽古井贡酒股份有限公司 安徽亳州 236820  
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**摘要：**应用全二维气相色谱-飞行时间质谱联用仪结合顶空固相微萃取技术同时检测枯草芽孢杆菌发酵液中吡嗪类物质。结果表明：标准曲线线性关系良好，相关系数 $r^2$ 均大于0.99，相对标准偏差在4.2%-10.9%之间，加标回收率在84%-116%之间，满足发酵液样品中吡嗪类物质定量要求。

## 8. 化妆品工业—Cosmetics Industry

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### 精油成分分析—*Essential Oil Compounds Analysis*

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Yang, S.-O., Kim, Y., Kim, H.-S., Hyun, S.-H., Kim, S.-H., Choi, H.-K., Marriott, P.J.

#### Rapid sequential separation of essential oil compounds using continuous heart-cut multi-dimensional gas chromatography-mass spectrometry

(2011) Journal of Chromatography A, 1218 (18), pp. 2626-2634.

**ABSTRACT:** A method for separation and identification of peaks in essential oil samples based on rapid repetitive heart-cutting using multidimensional gas chromatography (MDGC)-mass spectrometry (MS) coupled with a cryotrapping interface is described. Lavender essential oil is analyzed by employing repetitive heart-cut intervals of 1.00 and 1.50. min, achieved in a parallel MDGC-MS/GC-FID experiment. The number of peaks that were detected in 1D GC operation above a given response threshold more than tripled when MDGC-MS employing the cryotrapping module method was used. In addition, MDGC-MS enabled detection of peaks that were not individually evident in 1D GC-MS, owing to effective deconvolution in time of previously overlapped peaks in 1D GC. Thus separation using the cryomodulation approach, without recourse to using deconvolution software, was possible. Peaks widths decreased by about 5-7-fold with the described method, peak capacity increased from about 9 per min to 60 per min, and greater sensitivity results. Repeatability of retention times for replicate analyses in the multidimensional mode was better than 0.02% RSD. The present study suggests that the described heart-cutting technique using MDGC-MS can be used for general improvement in separation and identification of volatile compounds. © 2011 Elsevier B.V.

Bonaccorsi, I., Sciarrone, D., Schipilliti, L., Dugo, P., Mondello, L., Dugo, G.

**Multidimensional enantio gas chromatography/mass spectrometry and gas chromatography-combustion-isotopic ratio mass spectrometry for the authenticity assessment of lime essential oils (*C. aurantifolia* Swingle and *C. latifolia* Tanaka)**

**(2012) Journal of Chromatography A, 1226, pp. 87-95.**

**ABSTRACT:** This article focuses on the genuineness assessment of Lime oils (*Citrus aurantifolia* Swingle and *C. latifolia* Tanaka), by Multi Dimensional Gas Chromatography (MDGC) to determine the enantiomeric distribution of  $\alpha$ -thujene, camphene,  $\beta$ -pinene, sabinene,  $\alpha$ -phellandrene,  $\beta$ -phellandrene, limonene, linalool, terpinen-4-ol,  $\alpha$ -terpineol and by gas chromatography-combustion isotope ratio mass spectrometry (GC-C-IRMS) to determine the isotopic ratios of  $\alpha$ -pinene,  $\beta$ -pinene, limonene,  $\alpha$ -terpineol, nerol, geranal,  $\beta$ -caryophyllene, trans- $\alpha$ -bergamotene, germacrene B. To the author's knowledge this is the first attempt to assess the authenticity and differentiate Persian Lime from Key lime oils by GC-C-IRMS. The results of the two analytical approaches were compared. The simultaneous use of the two techniques provides more reliable capability to detect adulteration in Citrus essential oils. In fact, in some circumstance only one of the two techniques allows to discriminate adulterated or contaminated oils. In cases where only small anomalies are detected by the two techniques due to subtle adulterations, their synergic use allows to express judgments. The advantage of both techniques is the low number of components the analyst must evaluate, reducing the complexity of the data necessary to deal with. Moreover, the conventional analytical approach based on the evaluation of the whole volatile fraction can fail to reveal the quality of the oils, if the adulteration is extremely subtle. © 2011 Elsevier B.V.

**Van Der Wat, L., Dovey, M., Naudé, Y., Forbes, P.B.C.  
Investigation into the aroma of rosemary using multi-channel silicone rubber  
traps, off-line olfactometry and comprehensive two-dimensional gas  
chromatography-mass spectrometry**

**(2013) South African Journal of Chemistry, 66, pp. 21-26.**

**ABSTRACT:** Multi-channel polydimethylsiloxane rubber traps were used to sample the headspace of rosemary samples (two essential oils from different sources, one oleoresin and one dried herb) followed by comprehensive two-dimensional gas chromatography - time of flight mass spectrometry(GCxGC-TOFMS)orGC-MSanalyses. The aromaof different

headspace samples was characterized using a custom-built olfactory apparatus. The differences between the aroma profiles were evident from bubble plots of the perceived aroma at different temperatures. The samples were heat-treated to simulate cooking of food products, and were then reassessed to determine any changes in the aroma profile. It was found that the intense menthol and cooling aromas subsided in all the samples with heating. GCxGC-TOFMS allowed for separation of the numerous components in the headspace samples. Many terpenes and aliphatics were thus tentatively identified and the relative peak areas were compared to better understand the mixture that contributes to the rosemary aroma.

## 二维气相色谱/质谱分离分析中国苦水玫瑰精油中的复杂天然产物

《化学通报》2011年 第9期 | 周围 张雅珩 刘红卫

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**摘要：**运用二维气相色谱/质谱联用技术对天然植物萃取精油—中国苦水玫瑰精油中难分离的复杂成分进行了分离分析，同时对色谱共流出组分的切割方式和切割时间进行了研究探讨。玫瑰精油组分首先在预毛细管柱 J & W Scientific DB-1701 (60m×250 μm×0.5 μm) 上得到初步分离，通过 FID 信号确定了目标组分的切割时间段 (46.5~47.8min)，目标组分被有选择性的切换到主柱 Varian CP-Wax 57CB (25m×250 μm×0.2 μm) 实现进一步分离，并通过质谱检测器检测。通过以上方法成功分离确证了以往单独使用多根不同极性色谱柱难以分离确证的玫瑰精油组分香茅醇和橙花醇，同时还考察了该二维气相色谱/质谱联用仪切割的重复性。通过以上实验证实了二维气相色谱/质谱联用技术用于复杂天然产物成分研究的可靠性和良好前景。

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## 挥发性分析—Volatility Analysis

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## 全二维气相色谱-飞行时间质谱分析香紫苏油中的挥发性成分

《香料香精化妆品》2011年 第6期 | 李智宇 冒德寿 徐世娟 刘强

红塔烟草(集团)有限责任公司 云南玉溪 653100

**摘要:**建立了香紫苏油的全二维气相色谱-飞行时间质谱指纹图谱,根据质谱库检索、保留指数验证,或参照标准化合物及文献报道,共定性了183个物质,占峰面积的96.75%,其中正构烷烃、芳香环物质和非芳香环物质在DB-Petro×DB-17ht柱系统上实现了明显的族组分分离,主要成分为乙酸芳樟酯(15.11%)、芳樟醇(9.32%)、乙酸橙花酯(6.02%)、橙花醇(5.89%)、香紫苏醇(4.85%)、顺式-β-罗勒烯(4.14%)、α-月桂烯(3.52%)等。

## 全二维气相色谱-飞行时间质谱分析赖百当油的挥发性成分

《光谱实验室》2012年 第4期 | 徐世娟 冒德寿 李智宇 蔡炳彪 刘强

红塔烟草集团有限责任公司技术中心香精香料研究室 云南 653100

**摘要:**建立了赖百当油的全二维气相色谱-飞行时间质谱(GC×GC/TOF-MS)指纹图谱,通过质谱库检索、保留指数比对、标准品同时进样验证与文献比较,共鉴定了108个组分,占挥发油总量的94.81%,主要成分为:苯丙酸乙酯(30.92%)、绿花白干层醇(9.85%)、苯丙酸(7.91%)、乙酸龙脑酯(2.87%)、乙酸(2.81%)、杜香醇(2.41%)、喇叭烯(2.14%)等。本实验首次用GC×GC/TOF-MS对赖百当油进行了分析,并初步进行了致香机理的阐释,为该植物的开发、调香应用和品控提供了技术支撑。

## 9. 其他应用—Other Applications

**De Godoy, L.A.F., Pedroso, M.P., Hantao, L.W., Poppi, R.J., Augusto, F.**  
**Quantitative analysis by comprehensive two-dimensional gas chromatography**  
**using interval Multi-way Partial Least Squares calibration**  
**(2011) Talanta, 83 (4), pp. 1302-1307.**

**ABSTRACT:** A new approach for target quantitative analysis for comprehensive two-dimensional gas chromatography (GC × GC), interval Multi-way Partial Least Square (iNPLS) is presented and evaluated in this paper. In iNPLS, the two-dimensional chromatogram is split in small sections; each of these pieces is treated as an independent new chromatogram. Separated conventional NPLS calibration models for the concentration of the target analyte are built for each of

the pieces of the whole chromatogram, and the best model is selected for quantitative analysis. An algorithm for iNPLS running on MatLab platform was written, preliminarily evaluated with using solutions of model compounds with different chemical properties and subsequently applied to quantify some allergens in perfume samples. The results were found to be adequate, and good precision and accuracy was obtained even for poorly resolved peaks. © 2010 Elsevier B.V. All rights reserved.

**Parshintsev, J., Lai, C.K., Hartonen, K., Kulmala, M., Riekkola, M.-L.**

**A new approach to determine vapor pressures of compounds in multicomponent systems by comprehensive two-dimensional gas chromatography coupled to time-of-flight mass spectrometry**

**(2014) Talanta, 124, pp. 21-26.**

**ABSTRACT:** A method is described to determine vapor pressures of compounds in multicomponent systems simultaneously. The method is based on temperature-gradient analysis by comprehensive two-dimensional gas chromatography coupled to time-of-flight mass spectrometry (GCxGC-TOFMS). Vapor pressures are determined with the aid of known vapor pressure values of reference compounds eluting before and after the analytes. Reference compounds with the same functionalities as the analytes are preferred, but when these are not available, the alkane series can be utilized. The number of compounds whose vapor pressures can be determined is limited only by the peak capacity of the chromatographic system. Although the lowest subcooled vapor pressure determined was 0.006 Pa, for tetrahydroarauacarolone in an atmospheric aerosol sample, vapor pressures as low as 10<sup>-6</sup> Pa can be measured with the described set-up. Even lower values can be measured with higher GC temperatures and longer analysis times. Since only a few picograms of compound is required, in a mixture of any complexity, the GCxGC-TOFMS method offers unique sensitivity, rapidity, and comprehensiveness. © 2014 Elsevier B.V.

## 多维气相色谱分析技术在多元气体分析中的应用

《中国计量》2013年 第8期 | 张群

中国计量科学研究院

**摘要：**在气体标准物质制备过程中。利用气相色谱仪分析气体组分是一项重要的

测试手段。由于多元气体标准物质的气体组分较为复杂。既有组分间沸点范围大或某些有机官能团的性质相近的轻烃类气体。又有永久性气体并且某些组分间浓度差异较大。因此采用单通道色谱系统很难对一个复杂的气体混合物进行完全分离。

## 10. 科技前沿—Technique Development

Ji, Y., Xia, B., Luan, F., Zhang, X., Zhang, H.

Quantitative structure-retention relationship model of organic compounds on comprehensive two-dimensional gas chromatography by heuristic method  
(2010) Chinese journal of chromatography / Zhongguo hua xue hui, 28 (9), pp. 826-832.

ABSTRACT: The descriptors of organic compounds were calculated by means of semi-empirical PM3 method and the quantitative structure-retention relationship model of organic compounds on three columns of comprehensive two-dimensional gas chromatography were set by heuristic method. The good stability and predictive power of the multi-linear model were tested and the results showed the model had good linear relationships with all squared correlation coefficients more than 0.88 and each standard deviation less than 0.105. The leave-one-out cross-validated squared correlation coefficients were corresponding with the squared correlation coefficients from the model.

Zapadlo, M., Krupčík, J., Májek, P., Armstrong, D.W., Sandra, P.

Use of a polar ionic liquid as second column for the comprehensive two-dimensional GC separation of PCBs

(2010) Journal of Chromatography A, 1217 (37), pp. 5859-5867.

ABSTRACT: The orthogonality of three columns coupled in two series was studied for the congener specific comprehensive two-dimensional GC separation of polychlorinated biphenyls (PCBs). A non-polar capillary column coated with poly(5%-phenyl-95%-methyl)siloxane was used as the first (1D) column in both series. A polar capillary column coated with 70% cyanopropyl-polysilphenylene-siloxane or a capillary column coated with the

ionic liquid 1,12-di(tripropylphosphonium)dodecane bis(trifluoromethane-sulfonyl)imide were used as the second (2D) columns. Nine multi-congener standard PCB solutions containing subsets of all native 209 PCBs, a mixture of 209 PCBs as well as Aroclor 1242 and 1260 formulations were used to study the orthogonality of both column series. Retention times of the corresponding PCB congeners on 1D and 2D columns were used to construct retention time dependences (apex plots) for assessing orthogonality of both columns coupled in series. For a visual assessment of the peak density of PCBs congeners on a retention plane, 2D images were compared. The degree of orthogonality of both column series was, along the visual assessment of distribution of PCBs on the retention plane, evaluated also by Pearson's correlation coefficient, which was found by correlation of retention times  $t_{R,i,2D}$  and  $t_{R,i,1D}$  of corresponding PCB congeners on both column series. It was demonstrated that the apolar+ionic liquid column series is almost orthogonal both for the 2D separation of PCBs present in Aroclor 1242 and 1260 formulations as well as for the separation of all of 209 PCBs. All toxic, dioxin-like PCBs, with the exception of PCB 118 that overlaps with PCB 106, were resolved by the apolar/ionic liquid series while on the apolar/polar column series three toxic PCBs overlapped (105+127, 81+148 and 118+106). © 2010 Elsevier B.V.

**Gardner, J.Y., Brillhart, D.E., Benjamin, M.M., Dixon, L.G., Mitchell, L.M.,  
Dimandja, J.-M.D.**

**The use of GC $\times$  GC/TOF MS with multivariate analysis for the  
characterization of foodborne pathogen bacteria profiles  
(2011) Journal of Separation Science, 34 (2), pp. 176-185.**

**ABSTRACT:** The cellular fatty acid profiles of eight strains of *Bacillus*, *Staphylococcus*, and *Enterobacteriaceae* (*Escherichia coli* and *Salmonella*) were analyzed by comprehensive two-dimensional gas chromatography/time-of-flight mass spectrometry. A novel template method was developed to standardize the raw two-dimensional gas chromatography retention data through the use of a chemical indexing mixture. Analyte retention coordinates were normalized in the primary dimension with respect to a series of n-alkanes (Kovats index) and in the secondary dimension with respect to a series of aromatic hydrocarbons (Lee index). Fatty acid profiles extracted from the templates were compared by multidimensional scaling and principal component analysis. Differences in the profiles of Gram-positive and Gram-negative bacteria

were observed, and a series of heterogeneous mixtures comprising different fractions (containing one Gram-positive and one Gram-negative bacteria strain) were also distinguished from their homogeneous constituents. Copyright © 2011 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

**Marriott, P.J., Chin, S.-T., Maikhunthod, B., Schmarr, H.-G., Bieri, S.**

**Multidimensional gas chromatography**

**(2012) TrAC - Trends in Analytical Chemistry, 34, pp. 1-20.**

**ABSTRACT:** Analytical multidimensional gas chromatography (MDGC) and the excellent separation efficiency it achieves serve advanced characterization of complex volatile and semi-volatile samples, which is unlikely to be accomplished by single-dimensional chromatography. Here, we provide a technical overview of recent method implementation in MDGC, for both the classical sense (i.e. conventional heart-cut MDGC), including recent approaches to MDGC, and the comprehensive two-dimensional gas chromatography ( $GC \times GC$ ) variant. We summarize selected applications in diverse fields that best typify the role of these methods. We also draw attention to concepts (e.g., orthogonality of separation mechanisms and recently introduced microfluidic technology), and briefly comment on compatibility of detection systems. As a guide to potential opportunities for continued innovation in multidimensional applications, we highlight the capabilities of GC platforms that either combine various  $GC \times GC$  and MDGC arrangements or offer alternative operational modes for implementation of these methods. © 2012 Elsevier Ltd.

**Zeng, Z.-D., Hugel, H.M., Marriott, P.J.**

**Component correlation between related samples by using comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry with chemometric tools**

**(2012) Journal of Chromatography A, 1254, pp. 98-106.**

**ABSTRACT:** A chemometric strategy has been developed to discover component difference and similarity between two chromatograms (correlation) by using comprehensive two-dimensional (2D) gas chromatography with time-of-flight mass spectrometry ( $GC \times GC$ -TOFMS). It allows for rapid determination of the presence or absence of analytes of interest in both pure and overlapping peak clusters, and then locates elution windows of target components. First, representative elution windows of analytes are extracted from the 2D  $GC \times GC$

map to characterize the spectral space and further construct an orthogonal projection matrix for analysis. Next, multi-component spectral correlative chromatography (MSCC) is employed to scan the whole or pre-selected GC×GC-TOFMS data range to obtain component features. An auto-correlative projection curve is proposed to assess the projection residual from MSCC by defining a new evaluation index as reference, based on fixed-size moving window evolving factor analysis. In principle, the method can also be utilized to locate specific compounds whose known spectra are available. It is not restricted by data with high homoscedastic and heteroscedastic noise. Simulated GC-MS data and an extremely complicated herbal product mixture comprising 9 herbs demonstrates that the two-dimensional correlative distribution graph is effective for chemical interpretation between GC×GC-TOFMS data. It allows discovery of information buried in this type of highly complex dataset, especially for rapid and effective data comparison, where specific molecular identity might otherwise be hidden. © 2012 Elsevier B.V.

Lommen, A., van der Kamp, H.J., Kools, H.J., van der Lee, M.K., van der Weg, G., Mol, H.G.J.

**MetAlignID: A high-throughput software tool set for automated detection of trace level contaminants in comprehensive LECO two-dimensional gas chromatography time-of-flight mass spectrometry data**  
**(2012) Journal of Chromatography A, 1263, pp. 169-178.**

**ABSTRACT:** A new alternative data processing tool set, metAlignID, is developed for automated pre-processing and library-based identification and concentration estimation of target compounds after analysis by comprehensive two-dimensional gas chromatography with mass spectrometric detection. The tool set has been developed for and tested on LECO data. The software is developed to run multi-threaded (one thread per processor core) on a standard PC (personal computer) under different operating systems and is as such capable of processing multiple data sets simultaneously. Raw data files are converted into netCDF (network Common Data Form) format using a fast conversion tool. They are then preprocessed using previously developed algorithms originating from metAlign software. Next, the resulting reduced data files are searched against a user-composed library (derived from user or commercial NIST-compatible libraries) (NIST = National Institute of Standards and Technology) and the identified compounds, including an indicative

concentration, are reported in Excel format. Data can be processed batch wise. The overall time needed for conversion together with processing and searching of 30 raw data sets for 560 compounds is routinely within an hour. The screening performance is evaluated for detection of pesticides and contaminants in raw data obtained after analysis of soil and plant samples. Results are compared to the existing data-handling routine based on proprietary software (LECO, ChromaTOF). The developed software tool set, which is freely downloadable at [www.metalign.nl](http://www.metalign.nl), greatly accelerates data-analysis and offers more options for fine-tuning automated identification toward specific application needs. The quality of the results obtained is slightly better than the standard processing and also adds a quantitative estimate. The software tool set in combination with two-dimensional gas chromatography coupled to time-of-flight mass spectrometry shows great potential as a highly-automated and fast multi-residue instrumental screening method. © 2012 Elsevier B.V.

**Chen, B.-X., Hung, T.-Y., Jian, R.-S., Lu, C.-J.**  
**A multidimensional micro gas chromatograph employing a parallel separation**  
**multi-column chip and stop-flow  $\mu$ G<sub>C</sub> ×  $\mu$ G<sub>Cs</sub> configuration**  
**(2013) Lab on a Chip - Miniaturisation for Chemistry and Biology, 13 (7), pp.**  
**1333-1341.**

**ABSTRACT:** A dual-chip, multidimensional micro gas chromatographic module was designed, built and evaluated. Column chips were fabricated on a silicon wafer with an etched rectangular channel 100  $\mu$ m (width) × 250  $\mu$ m (depth) using a deep reactive ion etching (DRIE) process. The column chip for the first GC dimension was 3 m long and was coated with polydimethylsiloxane (DB-1) as the stationary phase. The columns on the second dimensional chip were etched with the same width and depth as the first chip, but the flow channel was split into three parallel columns, 1 m long, on the same sized silicon chip (i.e., 3 cm × 3 cm). These three parallel columns on the second chip were coated with polyethylene oxide (DB-Wax), trifluoropropylpolymethylsilicone (OV-210) and cyanopropylmethylphenylmethyldopolysilicone (OV-225), accordingly, in order to provide diversified chromatographic retention. These two chips were connected via a stop-flow configuration to simultaneously generate multiple two-dimensional gas chromatograms for every analysis. This stop-flow  $\mu$ GC ×  $\mu$ G<sub>Cs</sub> design allowed the first column to function as a pre-separator and as a sequencing injector for the second parallel-separation chip. Fifteen volatile

organic compounds with boiling points that ranged from 80-131 °C with various functional groups were tested using this  $\mu$ GC ×  $\mu$ GCS module. Three discrete 2-D chromatograms were generated simultaneously, which demonstrated the advantages of simultaneously combining GC × GC with parallel separation GCs in microchip chromatography. The total traveling length in the column was only 4 m for each eluted peak and fully resolved separation was achieved through the cross reference among triplet 2-D chromatograms. © 2013 The Royal Society of Chemistry.

**Marney, L.C., Christopher Siegler, W., Parsons, B.A., Hoggard, J.C., Wright, B.W., Synovec, R.E.**

**Tile-based Fisher-ratio software for improved feature selection analysis of comprehensive two-dimensional gas chromatography-time-of-flight mass spectrometry data**

(2013) *Talanta*, 115, pp. 887-895.

**ABSTRACT:** Comprehensive two-dimensional (2D) gas chromatography coupled with time-of-flight mass spectrometry (GC $\times$ GC-TOFMS) is a highly capable instrumental platform that produces complex and information-rich multi-dimensional chemical data. The data can be initially overwhelming, especially when many samples (of various sample classes) are analyzed with multiple injections for each sample. Thus, the data must be analyzed in such a way as to extract the most meaningful information. The pixel-based and peak table-based Fisher ratio algorithmic approaches have been used successfully in the past to reduce the multi-dimensional data down to those chemical compounds that are changing between the sample classes relative to those that are not changing (i.e., chemical feature selection). We report on the initial development of a computationally fast novel tile-based Fisher-ratio software that addresses the challenges due to 2D retention time misalignment without explicitly aligning the data, which is often a shortcoming for both pixel-based and peak table-based algorithmic approaches. Concurrently, the tile-based Fisher-ratio algorithm significantly improves the sensitivity contrast of true positives against a background of potential false positives and noise. In this study, eight compounds, plus one internal standard, were spiked into diesel at various concentrations. The tile-based F-ratio algorithmic approach was able to "discover" all spiked analytes, within the complex diesel sample matrix with thousands of potential false positives, in each possible concentration comparison,

even at the lowest absolute spiked analyte concentration ratio of 1.06, the ratio between the concentrations in the spiked diesel sample to the native concentration in diesel. © 2013 Elsevier B.V.

**Shu-Jiang, L., Zhan-Ying, C., Yin-Zhong, C., Shi-Lian, W., Qi, L., Yuan-Qing, F.**  
**Inference and analysis of xenon outflow curves under multi-pulse injection in two-dimensional chromatography**

(2013) *Journal of Chromatography A*, 1311, pp. 183-187.

**ABSTRACT:** Multidimensional gas chromatography is widely applied to atmospheric xenon monitoring for the Comprehensive Nuclear-Test-Ban Treaty (CTBT). To improve the capability for xenon sampling from the atmosphere, sampling techniques have been investigated in detail. The sampling techniques are designed by xenon outflow curves which are influenced by many factors, and the injecting condition is one of the key factors that could influence the xenon outflow curves. In this paper, the xenon outflow curves of single-pulse injection in two-dimensional gas chromatography has been tested and fitted as a function of exponential modified Gaussian distribution. An inference formula of the xenon outflow curve for six-pulse injection is derived, and the inference formula is also tested to compare with its fitting formula of the xenon outflow curve. As a result, the curves of both the one-pulse and six-pulse injections obey the exponential modified Gaussian distribution when the temperature of the activated carbon column's temperature is 26°C and the flow rate of the carrier gas is 35.6mLmin<sup>-1</sup>. The retention time of the xenon peak for one-pulse injection is 215min, and the peak width is 138min. For the six-pulse injection, however, the retention time is delayed to 255min, and the peak width broadens to 222min. According to the inferred formula of the xenon outflow curve for the six-pulse injection, the inferred retention time is 243min, the relative deviation of the retention time is 4.7%, and the inferred peak width is 225min, with a relative deviation of 1.3%. © 2013 Elsevier B.V.

**Peroni, D., Sampat, A.A.S., van Egmond, W., de Koning, S., Cochran, J., Lautamo, R., Janssen, H.-G.**

**Comprehensive two-dimensional gas chromatography with a multi-capillary second dimension: A new column-set format for simultaneous optimum linear velocity operation**

(2013) *Journal of Chromatography A*, 1317, pp. 3-11.

**ABSTRACT:** Comprehensive two-dimensional gas chromatography (GC×GC) suffers from the impossibility to operate both dimensions at their optimum carrier gas velocity at the same time due to the different inner diameters of the columns typically employed. The use of multiple parallel capillary columns in the second dimension (GC×multi-GC) is studied as a means to achieve simultaneous optimum-velocity operation. A programme written in Microsoft Excel® was developed to calculate the efficiency of the two dimensions in GC×multi-GC for different numbers of columns in the second dimension. With the aid of this programme the appropriate number of columns was selected. Columns with maximum repeatability were specifically manufactured to grand suitable performance, i.e. to avoid band broadening effects caused by inter-column variations. 1D-GC experiments were carried out on the columns separately and combined in parallel. The performance of the parallel column set was consistent with that of the individual columns, with over 9100 plates generated (approximately 10,000 plates/m). A GC×multi-GC set-up was successfully installed. Model experiments proved the possibility to operate both dimensions at their optimum linear velocity simultaneously. The suitability of the novel second dimension column format to perform multidimensional separations was also shown for a number of selected applications. © 2013 Elsevier B.V.

**Blase, R.C., Llera, K., Luspay-Kuti, A., Libardoni, M.**

**The Importance of Detector Acquisition Rate in Comprehensive  
Two-Dimensional Gas Chromatography (GC×GC)**

**(2014) Separation Science and Technology (Philadelphia), 49 (6), pp. 847-853.**

**ABSTRACT:** The use of comprehensive two-dimensional gas chromatography (GC×GC) is becoming more popular for complex sample analysis and the optimization of this technique is paramount for accurate qualitative and quantitative reporting. In this work, the effect of detector data acquisition rate (or detector sampling speed) for GC×GC is investigated and summarized. Effects on chromatographic signal such as peak area, height, and width, as well as analytical figures of merit such as resolution and limit of detection, are investigated at four data acquisition rates using a multi-component test mixture. Adjusting the data acquisition rate of the detector has a profound effect on peak areas, heights, and peak widths. In addition, the increase in detected signal and decreased peak widths led to improved chromatographic resolution and limits of

detection (LOD) under optimized conditions. Copyright © Taylor & Francis Group, LLC.

**Kouremenos, K.A., Jones, O.A.H., Morrison, P.D., Marriott, P.J.  
Development of An Online LC-LVI-GC × GC System: Design and Preliminary  
Applications**

**(2016) Chromatographia, 79 (1-2), pp. 79-87.**

**ABSTRACT:** The identification of unknown compounds is of fundamental importance for a range of applications in chromatography including, but not limited to, environmental pollution, food/natural product analysis, metabolomics, sports testing, petrochemicals and biofuel analysis. Critical to the success of each application is the ability to separate the compounds of interest, both from each other and the sample matrix, which may be present at concentrations orders of magnitude higher than the analyte(s). Selectivity and sensitivity are key to such analyses and both may be increased by the use of multiple dimensions of separation. Here, we report on the construction of an online, liquid chromatography hyphenated large volume injection, cryogenically modulated, multi-dimensional gas chromatography (LC-LVI-GC × GC) system for the characterisation of complex matrices using existing instruments that are common in most analytical laboratories. We detail the design of the instrument and demonstrate its performance and potential on a range of sample types. The combination of the LC, and large volume injection cryogenic GC × GC, was found to lead to a high selectivity and peak capacity with little sample preparation needed, but with a trade off of a large sample run time of ~40 (but up to ~60) minutes in each case. The system therefore has great potential for the targeted and untargeted analysis of very complex sample types. © 2015, Springer-Verlag Berlin Heidelberg.

**Kulsing, C., Nolvachai, Y., Rawson, P., Evans, D.J., Marriott, P.J.  
Continuum in MDGC Technology: From Classical Multidimensional to  
Comprehensive Two-Dimensional Gas Chromatography**

**(2016) Analytical Chemistry, 88 (7), pp. 3529-3538.**

**ABSTRACT:** Recent advances in multidimensional gas chromatography (MDGC) comprise methods such as multiple heart-cut (H/C) analysis and comprehensive two-dimensional gas chromatography (GC × GC); however, clear approaches to evaluate the MDGC results, choice of the most appropriate method, and

optimized separation remain of concern. In order to track the capability of these analytical techniques and select an effective experimental approach, a fundamental approach was developed utilizing a time summation model incorporating temperature-dependent linear solvation energy relationship (LSER). The approach allows prediction of optimized analyte distribution in the 2D space for various MDGC approaches employing different experimental variables such as column lengths, temperature programs, and stationary phase combinations in order to evaluate separation performance (apparent 1D, 2D, total number of separated peaks, and orthogonality) for simulated MDGC results. The methodology applied LSER to generate results for nonpolar-polar and polar-nonpolar 2D column configurations for separation of 678 compounds in an oxidized kerosene-based jet fuel sample. Three-dimensional plots were generated in order to illustrate the dependency of separation performance on 2D column length and number of injections for different stationary phase combinations. With a given limit of analysis time, a MDGC approach to obtain an optimized total separated peak number for a particular column set was proposed depending on 1D and 2D analyte peak distribution. This study introduces fundamental concepts and establishes approaches to design effective GC × GC or multiple H/C systems for different column combinations, to provide the best overall separation outcomes with the highest separated peak number and/or orthogonality. © 2016 American Chemical Society.

## 多维色谱研究的最新进展

《色谱》2011 年 第 2 期 | 许国旺, 石先哲

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## 焚烧炉飞灰和烟道气中-2, 3, 4, 7, 8-PeCDF 的检测方法

专利 CN201310422421.9 | 陈吉平 王雪丽 倪余文

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**摘要:** 本发明提供了焚烧炉飞灰和烟道气中-2, 3, 4, 7, 8-PeCDF 的检测方法, 采用多维气相色谱与质谱联用的方法进行定量分析。是将净化后样品通过一维气相色谱进行初步分离达到进一步净化的目的, 分离柱为石英毛细管色谱柱 DB-5, 检测器为电子捕获检测器; 再将目标物所在峰通过安装在一维色谱柱温箱内的切割装置

切割进入第二维色谱进行进一步的分离检测，通过质谱检测，离子源为负化学源 (NCI)，质量分析器为四级杆质量分析器。采用同位素内标法进行定量。基于多维气相串联质谱法实现二噁英毒性指示物-2, 3, 4, 7, 8-PeCDF 的定量。实现焚烧炉飞灰及烟道气中二噁英含量的快速分析，分析仪器简单，谱图及定量方法简单，检测结果准确。