教学研究

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凝胶渗透色谱-光散射联用表征聚合物摩尔质量的实验教学改革

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摘要:凝胶渗透色谱-光散射(gel permeation chromatography-light scatting, GPC-LS)联用是目前最常用的表征聚合物摩尔质量的方法之一,具有灵敏度高、结果准确等特点,在科学研究与生产实践中得到了广泛应用。"凝胶渗透色谱-光散射联用表征聚合物摩尔质量"是《高分子物理实验》课程中一个重要的教学内容。然而,目前的 GPC-LS 实验教学内容简单,缺乏深度。本文在该实验项目原有内容的基础上进行扩充,重新设计出多套实验项目:(1)选取商品化的聚苯乙烯为实验样品,利用 GPC-LS 对其摩尔质量、摩尔质量分布以及回转半径等分子结构参数进行表征;(2)选取两种分子结构参数接近的聚丙烯腈样品,借助质量微分分布曲线揭示这两种样品在摩尔质量分布中的细微差别;(3)选取一系列不同摩尔质量的聚乙二醇为实验样品,通过比较其色谱图分析摩尔质量的高低对色谱峰的影响;(4)选取3种不同的聚合物(聚丙烯腈、聚甲基丙烯酸甲酯、聚β-环糊精)样品,借助构象图对其分子链的构象进行分析。此外,本文对实验教学方法进行了改革,将被动学习转变为主动学习,提高了学生的自主学习能力。通过本实验项目的教学改革探索,使学生能更加全面地理解凝胶渗透色谱-光散射联用的原理及应用,开拓了学生的知识视野,激发了学生的学习热情,提升了实验教学效果。

关键词:凝胶渗透色谱-光散射联用:验证性实验;设计性实验;教学改革

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Experimental teaching reform of polymer molar mass determination using gel permeation chromatography coupled with light scattering

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Abstract: Gel permeation chromatography coupled with light scattering (GPC-LS) is among the most common methods for determining the molar masses of polymers. GPC-LS is widely used in polymer science research and has been adopted for many industrial applications owing to its high sensitivity, accuracy, and precision. The determination of polymer molar masses using GPC-LS is an important experimental component of the "Polymer Physics Experiments" course. However, the present GPC-LS experimental teaching content tends to be overly simplistic and lacking in depth. Herein, the original experimental content is expanded and multiple sets of experiments are redesigned: (1) Using commercial polystyrene as an experimental sample, the molar mass, molar mass distribution, radius of gyration, and other molecular structure parameters are determined using GPC-LS; (2) Using two polyacrylonitriles with similar molecular structure parameters, subtle differences in the molar mass distributions of the samples are explored using differential mass distribution curves; (3) By comparing the chromatograms of a series of polyethylene glycols with different molar masses, the effect of molar mass on chromatographic peaks is investigated; and (4) For three different polymers (polyacrylonitrile, poly (methyl methacrylate), and poly (β -cyclodextrin)), the polymer chain conformations are

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analyzed using conformation plots (i.e., radius of gyration vs. molar mass). In addition, the experimental teaching method is modified to convert passive learning into active learning, thereby improving the students' self-directed learning ability. This experimental teaching reform will help students obtain a more comprehensive understanding of GPC-LS principles and applications, stimulate their enthusiasm for learning, and improve the teaching quality of the experimental course.

Key words: gel permeation chromatography coupled with light scattering (GPC-LS); confirmatory experiment; designing experiment; teaching reform

凝胶渗透色谱(gel permeation chromatography, GPC)是一种利用由惰性多孔凝胶装填而成的 色谱柱将溶液中的高分子按照其流体力学体积大小 进行分离的液相色谱技术[1]。分子尺寸大即摩尔 质量高的组分先洗脱出来,分子尺寸小即摩尔质量 低的组分后洗脱出来。其分离机理是基于熵驱动的 高分子在多孔凝胶内外分配达到平衡的体积排除机 理,因此人们将这类方法统称为体积排除色谱(size exclusion chromatography, SEC)。传统的 GPC 只能提供样品的摩尔质量信息,且受标样的影响较 大[2]。而激光光散射能够准确表征聚合物样品的 绝对摩尔质量,是研究高分子聚合反应的有力手 段[3]。凝胶渗透色谱-光散射(GPC-LS)联用是表征 聚合物摩尔质量与摩尔质量分布的最常用手段之 一,具有快速、准确、灵敏度高、重复性好等特点,能 够提供聚合物的数均摩尔质量、重均摩尔质量及摩 尔质量分布等信息[4-7]。此外,光散射还可以提供 高分子在溶液中的构象以及聚合物与溶剂分子间的 相互作用信息。因此 GPC-LS 联用是研究聚合物分 子结构的有效工具,已成为在分子水平研究聚合物 结构与性能关系的常用手段[8]。

然而,在目前的《高分子物理实验》教学项目中,GPC-LS 联用通常仅被用于表征聚合物样品的摩尔质量与摩尔质量分布,实验教学内容单一,缺乏创新性。本文围绕该技术在高分子结构表征中的应用,推出了多套可供学生选做的设计性实验项目,弥补了这方面的缺陷。通过对该实验教学项目中原有内容的扩展,使学生在掌握 GPC-LS 联用实验技术基础上,能够利用该技术深入地研究高分子的远程

结构,即高分子链的构象及聚合物的摩尔质量,在分子水平上加强了对高分子结构与性能之间复杂关系的理解。此外,通过对实验教学方法的改革,开拓了学生的知识视野,提高了实验教学效果,加深了学生对 GPC-LS 联用技术原理的理解和应用,培养了学生的团队合作意识与创新能力。

1 GPC-LS 联用实验教学内容设计

本文对该实验项目原有的教学内容进行了优化 与扩充,围绕该技术在高分子结构表征中的应用,借 助仪器操作软件 ASTRA 强大的数据分析功能,推 出了多套设计性实验项目。其主要内容如下:1)选 取商品化的聚苯乙烯为实验样品,利用 GPC-LS 对 其摩尔质量、摩尔质量分布以及回转半径等分子结 构参数进行表征:2)选取两种分子结构参数接近的 聚丙烯腈(polyacrylonitrile, PAN)样品,借助质量 微分分布曲线揭示这两种样品在摩尔质量分布中的 细微差别:3)选取一系列不同摩尔质量的聚乙二醇 (PEG)为实验样品,通过比较其色谱图分析摩尔质 量的高低对色谱峰的影响;4)选取3种不同的聚合 物(PAN、聚甲基丙烯酸甲酯(polymethyl methacrylate, PMMA)、聚 β-环 糊 精 (poly (β-cyclodextrin), $poly(\beta-CD)$))样品,借助构象图对其分子链 的构象进行分析。

2 实验案例分析

围绕上述实验内容,本文设计了4个实验教学案例并进行了分析,如下:(1)聚苯乙烯分子结构参数的表征;(2)PAN摩尔质量分布的表征;(3)不同

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摩尔质量 PEG 的表征;(4)聚合物分子链构象的表 征。通过对这些实验案例的分析,有助于深入理解 摩尔质量、分子链尺寸及分子链构象等这些远程结 构的物理意义及其相互关系。

2.1 仪器与试剂

凝胶渗透色谱-激光光散射联用(Wyatt,美 国);包括 Optilab T-rEX 示差检测器与 DAWN HE-LEOS-Ⅱ激光检测器。

聚苯乙烯、PEG与硝酸钠均购自国药集团化学 试剂有限公司(上海); PAN、PMMA、聚 β -CD 与高 纯水均由实验室自制;二甲基甲酰胺(DMF)、溴化 锂与叠氮钠均由 Sigma 公司(美国)提供。

2.2 色谱条件

实验案例(1)(2)(4) 色谱柱: PLgel 5 μm MIXED-C+PLgel 5 µm MIXED-D 柱(300 mm×7.5 mm, Agilent 公司);柱温:60 ℃;样品溶液质量浓 度:3~5 mg/mL;流动相:含 0.05 mol/L LiBr 的 DMF;流动相流速:1.0 mL/min;进样量:100 μL。

实验案例(3) 色谱柱: OHpak SB-803HQ 和 OHpak SB-804HQ (300 mm×8.0 mm, Shodex); 柱温:35 ℃;样品溶液质量浓度:3~5 mg/mL;流动 相:含 0.1 mol/L NaNO,和 0.2 g/L NaN,的水溶 液:流动相流速:0.5 mL/min;进样量:100 μL。

2.3 结果与讨论

2.3.1 聚苯乙烯分子结构参数的表征

图 1a 给出了某聚苯乙烯样品的 GPC-LS 色谱 图,可以看出,光散射检测与示差检测均在洗脱时间 10~17 min 出现一个峰。由于样品的多分散性及管 路延迟等原因,这两个峰通常不会重合。此外,示差 检测在洗脱时间 18~20 min 出现的小峰与倒峰均 代表溶剂峰,在洗脱时间 20 min 后,样品全部从色 谱柱中洗脱出来。找到样品峰后,需要借助一个模 型拟合计算摩尔质量。对于常见的聚合物而言,最 常用的模型为 **Zimm** 模型^[9],见式(1)。

$$\frac{Kc}{R(\theta)} = \frac{1}{M} \left(1 + \frac{16\pi^2 n^2}{3\lambda^2} R_{\rm g}^2 \sin^2 \frac{\theta}{2} \right)$$
 (1)

其中光学常数 $K=4\pi^2n^2/N_{\Lambda}\lambda^4(dn/dc)^2$, n 和 λ 分 别为溶剂的折光指数和入射光在真空中的波长,c为高分子的浓度, $R(\theta)$ 是散射角为 θ 时测得的瑞利 因子, R_a 为高分子的回转半径,M 为摩尔质量。

由于散射峰的任何一点,其浓度都非常低(c约 为 10^{-4} g/mL),因此以 $Kc/R(\theta)$ 对 $\sin^2\frac{\theta}{2}$ 作图,其

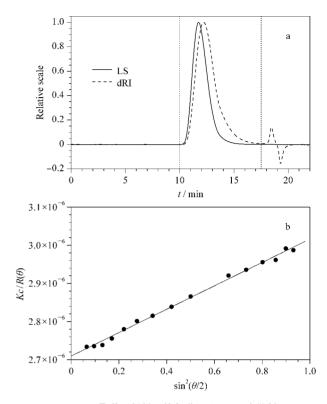


图 1 (a)聚苯乙烯样品的色谱图以及(b)光散射 峰中某一级分的 Zimm 模式图

Fig. 1 (a) Chromatograms of polystyrene sample, and (b) Zimm plot for one data slice from the LS

LS: light scatting detection; dRI: refractive index detection. Conditions: solvent, 0.05 mol/L LiBr in DMF; flow rate, 1.0 mL/min; column temperature, 60 °C.

Results; slice index, 354; model, Zimm; fit degree, 1; abscissa position, 11.725 min; mass concentration, 0.1565 mg/mL; molar mass, $(4.691\pm0.007)\times10^5$ g/mol; radius of gyration, (22.5 ± 0.2) nm; dn/dc, 0.159 mL/g; $R^2 = 0.9957$.

截距的倒数即为摩尔质量M,由斜率可得到分子的 回转半径 R_{σ} 。图 1b 给出了样品峰中一个点(级分) 的拟合计算结果,峰级分:354,拟合模型:Zimm,拟 合度: 1, 洗脱时间: 11.725 min, 级分质量浓度: 0.1565 mg/mL,级分的摩尔质量:(4.691±0.007) $\times 10^{5}$ g/mol,级分的回转半径:(22.5±0.2) nm,样 品的折光指数增量(dn/dc): 0.159 mL/g,拟合相 关性:0.9957。

因此借助 Zimm 模型拟合可以得到样品光散射 信号峰中每个级分的摩尔质量和回转半径。在此基 础上就可以得到完整的样品摩尔质量-洗脱时间及 回转半径-洗脱时间曲线(图2)。在图2数据的基 础上,可以得到该聚苯乙烯样品的平均摩尔质量、摩 尔质量分布系数以及回转半径等结构参数(表1)。

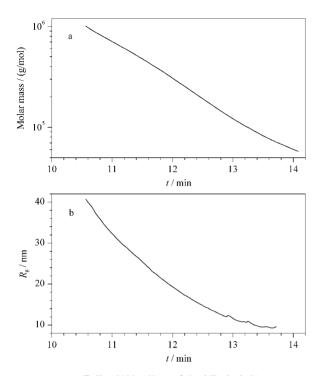


图 2 聚苯乙烯样品的(a)摩尔质量-洗脱时间及(b)回转半径-洗脱时间曲线

Fig. 2 Curves of (a) molar mass versus elution time and (b) radius of gyration versus elution time for polystyrene sample

Conditions: solvent, 0. 05 mol/L LiBr in DMF; flow rate, 1. 0 mL/min; column temperature, 60 $^\circ\!\! C.$

表 1 聚苯乙烯样品的 GPC-LS 测试项目及结果 Table 1 GPC-LS test items and results of polystyrene sample

Content	Result	Uncertainty
$M_{\rm n}$	1.686×10 ⁵	0.2%
$M_{ m p}$	3.045×10^{5}	0.1%
$M_{ m w}$	2.959×10^{5}	0.1%
M_{z}	4.381×10^{5}	0.3%
$M_{ m w}/M_{ m n}$	1.755	0.2%
$M_{\rm z}/M_{\rm n}$	2.599	0.4%
$R_{ m gn}$	13.7	4%
$R_{ m gw}$	17.9	2%
$R_{ m gz}$	22.2	1%
	$M_{ m p}$ $M_{ m w}$ $M_{ m z}$ $M_{ m w}/M_{ m n}$ $M_{ m z}/M_{ m n}$ $R_{ m gn}$	$\begin{array}{ccc} M_{\rm n} & 1.686\times10^5 \\ M_{\rm p} & 3.045\times10^5 \\ M_{\rm w} & 2.959\times10^5 \\ M_{\rm z} & 4.381\times10^5 \\ M_{\rm w}/M_{\rm n} & 1.755 \\ M_{\rm z}/M_{\rm n} & 2.599 \\ R_{\rm gn} & 13.7 \\ R_{\rm gw} & 17.9 \\ \end{array}$

通过这个实验,学生可熟悉聚合物摩尔质量、摩尔质量分布以及回转半径等分子结构参数的测试方法及其物理意义,理解这些结构参数之间的关系及影响规律,加深对聚合物分子远程结构的理解,并初步掌握聚合物分子结构参数的表征及数据处理方法。

2.3.2 PAN 摩尔质量分布的表征

摩尔质量分布是聚合物分子结构的一大特色,聚合物本质上是由摩尔质量大小不同的同系物组成的混合物。该混合物的性能主要与某些摩尔质量级

分的含量有关。因此,摩尔质量分布是聚合物最基 本的结构参数之一,对其性能具有重要的影响[10]。 聚合物摩尔质量分布的宽窄可以通过摩尔质量分布 系数 $(M_{\rm w}/M_{\rm p})$ 进行表征。然而,单纯的摩尔质量分 布系数只能给出摩尔质量分布的范围,并不能给出 不同摩尔质量组分的含量。例如图 3a 中两种 PAN 样品的色谱图非常相似,其重均摩尔质量分别为 90 000 g/mol(PAN-1)与 93 000 g/mol(PAN-2), 另外两种样品的摩尔质量分布也比较接近(PAN-1: 2.1; PAN-2: 2.2)。从平均摩尔质量与摩尔质量分 布系数看,这两种 PAN 样品差别不大,然而二者的 纺丝性能却显著不同。此时,需要借助质量微分分 布曲线才能揭示这两种样品在摩尔质量分布中的细 微差别。如图 3b 所示, PAN-1 中摩尔质量为 70 000 g/mol 左右的组分占的比例较高,而 PAN-2 中含量较高的组分的摩尔质量在 90 000 g/mol 左 右。由此可以推断, PAN-2 的纺丝性能要优于 PAN-1。因此借助质量微分分布曲线可以直观地显 示出样品的摩尔质量分布差异。

通过该实验,学生可借助质量微分分布曲线更

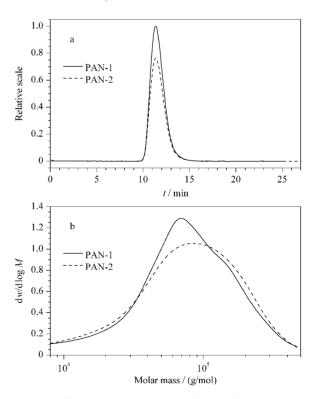


图 3 两种聚丙腈样品的(a)色谱图及其(b)质量微分分布曲线 Fig. 3 (a) Chromatograms and (b) differential weight fraction curves of two polyacrylonitrile (PAN) samples

Conditions; solvent, 0.05 mol/L LiBr in DMF; flow rate, 1.0 mL/min; column temperature, 60 $^{\circ}\mathrm{C}.$

好地理解聚合物的摩尔质量分布,包括摩尔质量分布的宽度、分布的对称性以及摩尔质量集中的范围等。聚合物的摩尔质量分布与材料的力学性能如拉伸强度、冲击强度以及加工性能都有密切的关系。因此控制与改进聚合物的摩尔质量分布是高分子材料改性的重要涂径。

2.3.3 不同摩尔质量 PEG 的表征

学生在学习 GPC 机理的时候,很容易将组分的 洗脱顺序与其摩尔质量的关系弄反。基于这种情况,为了能更直观地看出样品摩尔质量与其洗脱时间的关系,本文设计了该实验。如图 4 所示,不同摩尔质量的 PEG 样品的色谱峰位置显著不同。摩尔质量为 20 000 g/mol 的 PEG 洗脱时间是 30 min 左右,随着样品摩尔质量的降低,其洗脱时间逐渐增加,当样品的摩尔质量降低至 1 000 g/mol 时,其洗脱时间增加到 38 min 左右。此外,本实验选用的PEG 是一种常用的水溶性高分子[11],其生产厂家标注的摩尔质量与实验测定的摩尔质量存在一定的差异。不同摩尔质量 PEG 的测试结果见表 2,从中可以看出,样品的重均摩尔质量与厂家标注的摩尔质量基本一致。

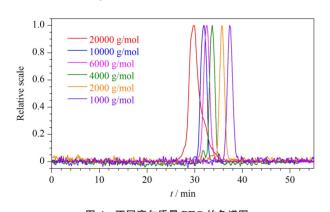


图 4 不同摩尔质量 PEG 的色谱图
Fig. 4 Chromatograms of polyethylene glycol
with different molar masses

Conditions: solvent, 0.1 mol/L NaNO $_3$ and 0.2 g/L NaN $_3$ in water; flow rate, 0.5 mL/min; column temperature, 35 $^\circ\!\! C.$

表 2 不同摩尔质量 PEG 的测试结果

Table 2 Determination results of polyethylene
glycol with different molar masses

Label $M_{\rm w}/({\rm g/mol})$ Determined $M_{\rm w}/({\rm g/mol})$ Uncertainty 1000 13% 1260 2000 2210 12% 4000 4450 12% 6000 7760 9% 10000 8130 10%

17430

6%

20000

通过对不同摩尔质量的 PEG 色谱图的分析,学 生可更直观地了解聚合物摩尔质量与其洗脱时间之 间的关系,巩固对凝胶渗透色谱分离原理的认识,加 深对高分子稀溶液理论的理解。

2.3.4 聚合物分子链构象的表征

对于摩尔质量分布较宽的样品,通过测量经 GPC 分离后所得各级分的 M 与 R_a , 借助 ASTRA 软 件中的构象图(RMS conformation plot)建立 R_{σ} 与 M之间的标度关系 $R_{\sigma} \sim M^{\nu}$, 从标度关系指数 ν 即可 获得聚合物分子链构象的信息。研究表明,标度关 系指数 $\nu = 1$ 时大分子呈现棒状构象 $\nu = 0.5 \sim 0.6$ 对应着无规线团构象, v=1/3则表明大分子紧密堆 砌采取球形构象^[12]。图 5 为 PMMA、PAN 和聚 β -环糊精的构象图,拟合得到的标度关系指数分别为 $\nu = 0.56 \pm 0.03 \text{ (PMMA)} \ 0.58 \pm 0.02 \text{ (PAN)} \ \text{$10.63 \pm$}$ 0.05 (poly(β-CD))。这一结果表明: PMMA、PAN 和聚 β-CD 这 3 种不同化学结构的高分子在含 0.05 mol/L LiBr 的 DMF 中均呈现无规线团构象。与同 为乙烯基聚合物的 PMMA 相比,相同摩尔质量下 PAN 的回转半径更大,这可由两者结构重复单元摩 尔质量的差异来解释。相较于 PMMA 与 PAN,相 同摩尔质量下聚 β -CD 的回转半径最小,这是因为 聚 β-CD 的结构重复单元 β-环糊精具有紧凑的环状 结构,整个聚合物链段所占的空间更小。

通过对聚合物构象图的分析,学生可加深对聚合物分子链回转半径的概念及其测试方法的了解,熟悉高分子线团尺寸的变化规律及影响因素,促进

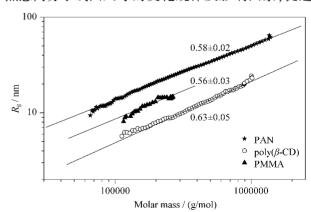


图 5 不同聚合物样品的回转半径与摩尔质量间的标度关系 Fig. 5 Scaling relationship between radius of gyration and molar masses for different polymer samples

PMMA: polymethyl methacrylate; poly ($\beta\text{-CD}$): poly ($\beta\text{-cyclodextrin})$.

Conditions: solvent, 0.05 mol/L LiBr in DMF; flow rate, 1.0 mL/min; column temperature, 60 °C.

对聚合物分子链构象的理解,初步掌握聚合物分子 链构象的表征方法。

3 GPC-LS 实验教学方法改革

在目前的 GPC-LS 联用实验教学中,由于教学方法单一,学生对 GPC与 LS 的原理及其应用认识不足。学生通常只是在课堂上听老师讲述仪器的工作原理与操作规程,虽然对实验教学内容有一些了解,但通常都是处于一知半解的状态;实验结束后并不能及时复习巩固,而是在仓促提交实验报告后迅速转入其他课程的学习,直到下一次开始新的实验课程,从而导致教学效果不佳。针对这种大型测试设备而言,留给学生动手操作的时间很短,整个实验过程仿佛"过眼云烟",难以保证教学效果。针对上述问题,本文创新教学模式,将被动学习转变为主动学习,提高了学生的自主学习能力。

实验开始前,老师通过布置预习报告作业提出若干问题,引导学生查阅相关资料,了解 GPC-LS 联用测聚合物摩尔质量的原理,影响 GPC-LS 测试的因素,色谱柱、溶剂,样品浓度的选择以及温度对摩尔质量的影响等。让同学们带着问题来做实验,做到"有备而来",激发了学生的学习兴趣,提高了学习的主观能动性。

由于受到实验设备与实验学时的限制,每人只能完成一部分实验内容。基于这种现实情况,为提高课堂教学效率,我们将学生分成若干小组,每个小组完成不同条件下的实验项目。在实验教学中,对学生实验过程进行全程跟踪,引导学生进行正确的实验操作以及对实验现象的观察与分析。实验结束后对所有实验数据进行共享,实验教学内容得到了丰富。

实验结束后以小组为单位提交实验报告,并对整个实验项目包括实验目的、实验原理、实验设备、实验过程、数据处理、实验结果分析等进行论述,并推选代表展示 PPT 以汇报答辩的形式交流讨论各种实验因素对 GPC-LS 联用测试的影响。

4 教学效果评价

这种创新研究型实验的设计极大丰富了实验教学内容。在实验实施过程中,将多个知识点进行有机整合,使学生对 GPC-LS 联用的测试原理理解得

更加形象生动。通过本实验项目的教学改革探索, 开拓了学生的知识视野,激发了学生对 GPC-LS 联 用技术的学习兴趣,加深了学生对于高分子溶液基 本理论以及摩尔质量、摩尔质量分布、回转半径与构 象等基本概念的理解。此外,丰富了高分子物理知 识体系,提高了学生实验技能以及综合运用高分子 物理基本理论分析和解决复杂高分子工程问题的能 力,为学生的进一步学习奠定了基础。

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《色谱》论文中可直接使用的缩略词

Abbreviations that may be used without definition in Chinese Journal of Chromatography

A. C. A. activation activation and entire, cytidine, guantine, thymine Ac, OAC ACN ACN APP, AMP ATP ARR ARR AND, AMP ATP Arrivation and I-letter codes ACN AU	Abbreviation	Definition
Ac, OAC ACN ACN ACN ACN ACN ACN ACN ACN ACN A		
ACN ADP, AMP, ATP adenosine 5'-di-, mono-, -triphosphate, etc. and similar nucleoside phosphates AR analytical grade bp base pair Standard 3- and 1-letter codes amino acids AU absorbance units BET Brunauer-Emmett-Teller CE, CEC capillary electrophoresis, capillary electrochromatography in nucleosides' decoxy, messenger, phosphate, recombinant/ribosomal, transfer mucleosides' decoxy, messenger, phosphate, recombinant/ribosomal, transfer decombinant/ribosomal, recombinant/ribosomal, transfer fell electrom inpart (soutation) ESI electrom inpart (soutation) ESI electrom protection decore gas chromatography in decore electros protection decore protection decore protection decore protection messenger, phosphate, edition decore protection decore protection messenger, phosphate, edition decore protection decore pro		
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AR analytical grade bp base pair Standard 3- and 1-letter codes AU absorbance units BET Brunauer-Emmett-Teller CE, CEC capillary electrophoresis, capillary electrochromatography in nucleosides/decoxy, messenger, phosphate, recombinant/ribosomal, transfer nucleosides/dec	ADP, AMP, ATP	adenosine 5'-di-, -mono-, -triphosphate, etc. and similar nucleoside phosphates
Slandard 3- and 1-letter codes AU BET BUDAN BET Brunauer-Emmett-Teller CE, CEC capillary electrophoresis, capillary electrochromatography in nucleosides/decoxy, messenger, phosphate, recombinant/ribosomal, transfer nucleotides/nucleic acids DAD diode-array detector bEAE diethylaminoethyl DNA, DNase deoxyribonucleic acid, deoxyribonuclease EDTA ethylene diamine tetraacetic acid electron impact (ionization) ESI electrospray ionization FID flame ionization detector FT FOurier transform GC gas chromatography GCB graphitized carbon black Hilb hydrophilic-lipophilic blanee HPR high performance high performance lid internal diameter innunoglobulin G internal diameter innunoglobulin G internal diameter innunoglobulin G internal diameter innunography internal diameter innunogra		analytical grade
BET Bruaner-Emmett-Teller CE, CEC capillary electrophoresis, capillary electrochromatography in uncleosides/electory DAD diode-array detector DEAE diethylaminoethyl DNA, DNase deoxyribonucleic acid, deoxyribonuclease EIDTA ethylene diamine tetraacetic acid EI electron impact (inization) ESI electrosipray (inization) ESI electrosipray (inization) FID flame ionization detector FID flowers transform GC gas chromatography GCB graphitized carbon black HLB hydrophilic-lipophilic balance HP		
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CEC capillary electrophoresis, capillary electrochromatography in uncleosides/ decoyn, messenger, phosphate, recombinant/ribosomal, transfer nucleotides/nucleic acids array detector diethylaminethyl doe array detector diethylaminethyl decoyn; homoleic acid, deoxyribonuclease ethylene diamine tetraacetic acid ethylene ethylene diamine tetraacetic acid ethylene ethylen		
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DAD diode-array detector DEAE diethylaminoethyl DNA, DNase dexyribonucleic acid, dexyribonuclease EDTA ethylene diamine tetraacetic acid EI electron impact (i onization) ESI electron impact (i onization) ESI electron grapat (i onization) ESI electron e		
DAD DEAE dichylaminoethyl DNA, DNase deoxyribonucleic acid, deoxyribonuclease EDTA ethylene diamine tetraacetic acid EI electron impact (ionization) ESI electrospry ionization FID flame ionization detector FT FOUTH The ionization GCB gas chromatography GCB graphitized carbon black HLB hydrophilic-lipophilic balance HP	d, m, p, r, t	
DEAE DNA, DNase deoxyribonucleic acid, deoxyribonuclease EDTA ethylene diamine tetraacetic acid electron impact (ionization) ESI electron impact (ionization) FID flame ionization detector FLD florescence detector FLD florescence detector FT Fourier transform GC gas chromatography GCB graphitized carbon black IIIB hydrophilic-lipophilic balance lip- ligh performance ligh performance ligh internal diameter ligh detection light detection light detection light detection light detection light detection monitoring matrix effect method light detection monitoring light detection light detection light detection light detection light detection light matrix defect light detection ligh	DAD	
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EDTA ethylene diamine tetraacetic acid EI electron impact (ionization) ESI electron impact (ionization) ESI electron impact (ionization) FID flame ionization detector FID fluorescence detector FT Pourier transform GC gas chromatography GCB graphitized carbon black HLB hydrophilic hipophilic balance HP		
ESI electrospray ionization FID flame ionization detector FID fluorescence detector FT Fourier transform GC gas chromatography GCB graphitized carbon black HLB hydrophilic-lipophilic balance HHP		
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FID flame ionization detector FLD fluorescence detector FT Fourier transform GC gas chromatography GCB graphitized carbon black HLB hydrophilic-lipophilic balance HP		
FLD FIT FOURIE TRANSFORM GC GC GS		
FOURTH Fourier transform GC gas chromatography GCB graphitized carbon black HLB hydrophilic-lipophilic balance HP··· high performance ··· HR high resolution IC ion chromatography i.d. internal diameter IgG immunoglobulin G IR infrared IS internal standard IC liquid chromatography I.OD limit of detection III limit of detection III matrix-assisted laser desorption ionization-time of flight ME matrix effect MeOH methanol M, relative molecular mass MRM multiple reaction monitoring MS. MS/MS mass spectrometry, tandem mass spectrometry NAD, NADH (NADP, NADPH) nicotinamide-adenine dinucleotide (phosphate) NMR nuclear magnetic resonance PCR polymerase chain reaction PDA photodiode array PSA primary secondary amine QuEChERS quick, easy, cheap, effective, rugged and safe correlation coefficient RNA, RNase ribonucleic acid, ribonuclease RP··· reversed-phase ··· relative standard deviation (preferred over coefficient of variation) SPA, SPME scandard deviation SPA, SPME scandard deviation (preferred over coefficient of variation) SPE, SPME solid phase extraction, solid phase microextraction SPA, SPME signal/noise SPE, SPME solid phase extraction, solid phase microextraction SPM signal/noise SPE, SPME solid phase extraction, solid phase microextraction SPM signal/noise SPM signal/noise transmission electron microscopy title transmission electron microscopy title transmission electron microscopy title		
GCB graphitized carbon black III.B hydrophilic-lipophilic balance HP··· high performance ··· high performance ··		
GCB HILB hydrophilic-lipophilic blance HP··· high performance HR high resolution IC ion chromatography i.d. internal diameter IgG immunoglobulin G IR infrared IS internal standard IC liquid chromatography LOD limit of detection IImit of quantification MALDI-TOF matrix-assisted laser desorption ionization-time of flight ME matrix effect MeOH methanol methanol Mr, relative molecular mass MRM multiple reaction monitoring MS, MS/MS MS/MS MADP, NADPH) nuclear magnetic resonance PCR polymerase chain reaction PCR polymerase chain reaction photodiode array pSA primary secondary amine QuEChETS quick, easy, cheap, effective, rugged and safe reversed-phase ·· reversed-phase ·· reversed-phase ·· RSD relative standard deviation (preferred over coefficient of variation) SPE, SPME solid phase extraction, solid phase microextraction SPF, SPME solid phase extraction, solid phase microextraction SRM selected ion monitoring TC total ion (current) chromatography Ire tention time tris (hydroxymethyl) aminomethane UPLC ultra performance liquid chromatography ultraviolet ultr		gas chromatography
HP high performance HR high resolution IC ion chromatography i.d. internal diameter IgG immunoglobulin G IR IR infrared IS internal standard LC liquid chromatography LOD limit of detection LOQ limit of quantification MALDL*TOF matrix-assisted laser desorption ionization-time of flight ME matrix effect MeOH methanol Mr, relative molecular mass MRM multiple reaction monitoring MS, MS/MS mass spectrometry, tandem mass spectrometry NAD, NADH (NADP, NADPH) nicotinamide-adenine dinucleotide (phosphate) NMR nuclear magnetic resonance PCR polymerase chain reaction PDA photodiode array PSA primary secondary amine QuEChERS quick, easy, cheap, effective, rugged and safe r correlation coefficient RNA, RNase ribonucles are RP reversed-phase -	GCB	
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$\begin{array}{c} \operatorname{MRM} & \operatorname{multiple reaction monitoring} \\ \operatorname{MS}, \operatorname{MS/MS} & \operatorname{mass spectrometry}, \operatorname{tandem mass spectrometry} \\ \operatorname{NAD}, \operatorname{NADH} \left(\operatorname{NADP}, \operatorname{NADPH}\right) & \operatorname{nicotinamide-adenine dinucleotide} \left(\operatorname{phosphate}\right) \\ \operatorname{NMR} & \operatorname{nuclear magnetic resonance} \\ \operatorname{PCR} & \operatorname{polymerase chain reaction} \\ \operatorname{PDA} & \operatorname{photodiode array} \\ \operatorname{PSA} & \operatorname{primary secondary amine} \\ \operatorname{QuEChERS} & \operatorname{quick}, \operatorname{easy}, \operatorname{cheap}, \operatorname{effective}, \operatorname{rugged} \operatorname{and safe} \\ r & \operatorname{correlation coefficient} \\ \operatorname{RNA}, \operatorname{RNase} & \operatorname{ribonucleic acid}, \operatorname{ribonuclease} \\ \operatorname{RP} \cdots & \operatorname{reversed-phase} \cdots \\ \operatorname{RSD} & \operatorname{relative standard deviation} \left(\operatorname{preferred over coefficient of variation}\right) \\ \operatorname{SD} & \operatorname{standard deviation} \\ \operatorname{SEM} & \operatorname{scanning electron microscopy} \\ \operatorname{SIM} & \operatorname{selected ion monitoring} \\ \operatorname{S/N} & \operatorname{signal/noise} \\ \operatorname{SPE}, \operatorname{SPME} & \operatorname{solid phase extraction}, \operatorname{solid phase microextraction} \\ \operatorname{SRM} & \operatorname{selected reaction monitoring} \\ \operatorname{SEM} & \operatorname{selected reaction monitoring} \\ \operatorname{TEM} & \operatorname{transmission electron microscopy} \\ \operatorname{TIC} & \operatorname{total ion} \left(\operatorname{current}\right)\operatorname{chromatogram} \\ \operatorname{TLC} & \operatorname{thin-layer chromatography} \\ t_{\mathbb{R}} & \operatorname{retention time} \\ \operatorname{Tris} & \operatorname{tris}\left(\operatorname{hydroxymethyl}\right)\operatorname{aminomethane} \\ \operatorname{UPLC} & \operatorname{ultra violet} \\ & \operatorname{ultra violet} \\ \end{array}$		
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$\begin{array}{c} \text{NAD, NADH (NADP, NADPH)} & \text{nicotinamide-adenine dinucleotide (phosphate)} \\ \text{NMR} & \text{nuclear magnetic resonance} \\ \text{PCR} & \text{polymerase chain reaction} \\ \text{PDA} & \text{photodiode array} \\ \text{PSA} & \text{primary secondary amine} \\ \text{QuEChERS} & \text{quick, easy, cheap, effective, rugged and safe} \\ \text{r} & \text{correlation coefficient} \\ \text{RNA, RNase} & \text{ribonucleic acid, ribonuclease} \\ \text{RP}\cdots & \text{reversed-phase} \cdots \\ \text{RSD} & \text{relative standard deviation (preferred over coefficient of variation)} \\ \text{SD} & \text{standard deviation} \\ \text{SEM} & \text{scanning electron microscopy} \\ \text{SIM} & \text{selected ion monitoring} \\ \text{S/N} & \text{signal/noise} \\ \text{SPE, SPME} & \text{solid phase extraction, solid phase microextraction} \\ \text{SRM} & \text{selected reaction monitoring} \\ \text{TEM} & \text{transmission electron microscopy} \\ \text{TIC} & \text{total ion (current) chromatogram} \\ \text{TLC} & \text{thin-layer chromatography} \\ t_{\text{R}} & \text{retention time} \\ \text{Tris} & \text{tris(hydroxymethyl) aminomethane} \\ \text{UPLC} & \text{ultra performance liquid chromatography} \\ \text{UV} & \text{ultraviolet} \\ \end{array}$		
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$ \begin{array}{ccc} \text{TLC} & \text{thin-layer chromatography} \\ t_{\text{R}} & \text{retention time} \\ \\ \text{Tris} & \text{tris (hydroxymethyl) aminomethane} \\ \\ \text{UPLC} & \text{ultra performance liquid chromatography} \\ \text{UV} & \text{ultraviolet} \\ \end{array} $		
$ \begin{array}{ccc} t_{\mathbb{R}} & \text{retention time} \\ \hline \text{Tris} & \text{tris(hydroxymethyl) aminomethane} \\ \hline \text{UPLC} & \text{ultra performance liquid chromatography} \\ \hline \text{UV} & \text{ultraviolet} \\ \hline \end{array} $	TLC	
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