「应用说明」

根据USP 621用ChroZen HPLC和UHPLC分析布洛芬

- HPLC & UHPLC 应用

描述

制药行业的方法验证在确保产品质量和评估系统对其预期目的的适用性方面起着非常关键的作用。分析方法验证包括几个参数，如特异性、准确性、精密度、系统适用性等。

最重要的是，基本上需要系统适用性来验证配置有仪器、操作参数和参考标准的分析方法是否在指南下进行，并得到适当的认可。为了确认这一点，评估了重复注射的再现性以及分析物和内标之间的分辨率，并在允许的范围内应用了一些分析条件的变化以满足要求。

在这项研究中，根据USP<621>，通过ChroZen HPLC和ChroZen UHPLC对布洛芬片剂进行分析，以验证系统对应用的适用性。布洛芬作为一种非甾体抗炎药被广泛使用，其分析在药物中非常常规。

仪器和软件

· ChroZen HPLC System

Item	Description	Part No.
Pump	ChroZen HPLC Quaternary Gradient Pump with Vacuum degasser	9421011020
Autosampler	ChroZen HPLC Autosampler	5421011020
Column oven	ChroZen HPLC Column oven for Analytical scale	3421011020
Detector	ChroZen HPLC UV/Vis Detector with dual wavelength	7411011020
Install. Option	HPLC Performance Kit (Without LC C18 Column)	1601011890
CDS	YL-Clarity software for single instrument of YCM HPLC	5301011000
	Autosampler control of YL-Clarity	5301011040
	System Suitability Test of YL-Clarity	5301011050
Column	C18 (4.6 mm x 250 mm, 5 μm)	-

· ChroZen UHPLC System

Item	Description	Part No.
Pump	ChroZen UHPLC Pump	9431011012
Autosampler	ChroZen UHPLC Cool/Heat Autosampler	9451011013
Column oven	ChroZen UHPLC Column oven for Analytical scale	3531011030
Detector	ChroZen UHPLC UV/Vis Detector	7521011020
Install. Option	UHPLC Performance Kit (Without LC C18 Column)	9361011150
CDS	YL-Clarity software for single instrument of YCM HPLC	5301011000
	Autosampler control of YL-Clarity	5301011040
	System Suitability Test of YL-Clarity	5301011050
Column	Poroshell C18 (2.1 mm x 100 mm, 2.7 μm)	-

[ChroZen HPLC]

[ChroZen UHPLC]

试剂和标准品

- 乙腈, HPLC 级别
- 氢氧化铵溶液，28.0-30.0%
  氯乙酸，99.0%
- 布洛芬，药物性继发性性病
  缬苯酮，99.0%
  超纯水，电阻率18.2 MΩ-cm

准备

1. 流动相

- ① 将4.0克氯乙酸溶解在400毫升水中。
  ② 用氢氧化铵将pH值调节至3.0。
  ③ 向②溶液中加入600mL乙腈，混匀。然后，在使用前过滤并脱气混合物。

2. 内标溶液

将缬草酮溶解在流动相中，使浓度达到0.35 mg/mL

3. 标准制剂

将布洛芬标准品溶解在内标溶液中，使其浓度达到12mg/mL

4. 化验准备

- ① 研磨20多片布洛芬，精确称量1200毫克。
  ② 加入100 mL内标溶液，搅拌10分钟。
  ③ 将溶液离心，分离顶层以备使用。

仪器条件和色谱图

ChroZen HPLC System	
Mobile phase	10% Chloroacetic acid(pH 3, NH_4OH) : ACN = 40 : 60
Flow rate	2.0 mL/min
Column	C18 (4.6 mm x 250 mm, 5 µm)
Temperature	30°C
Injection volume	5 µL
Detection	UV/Vis detector 254 nm

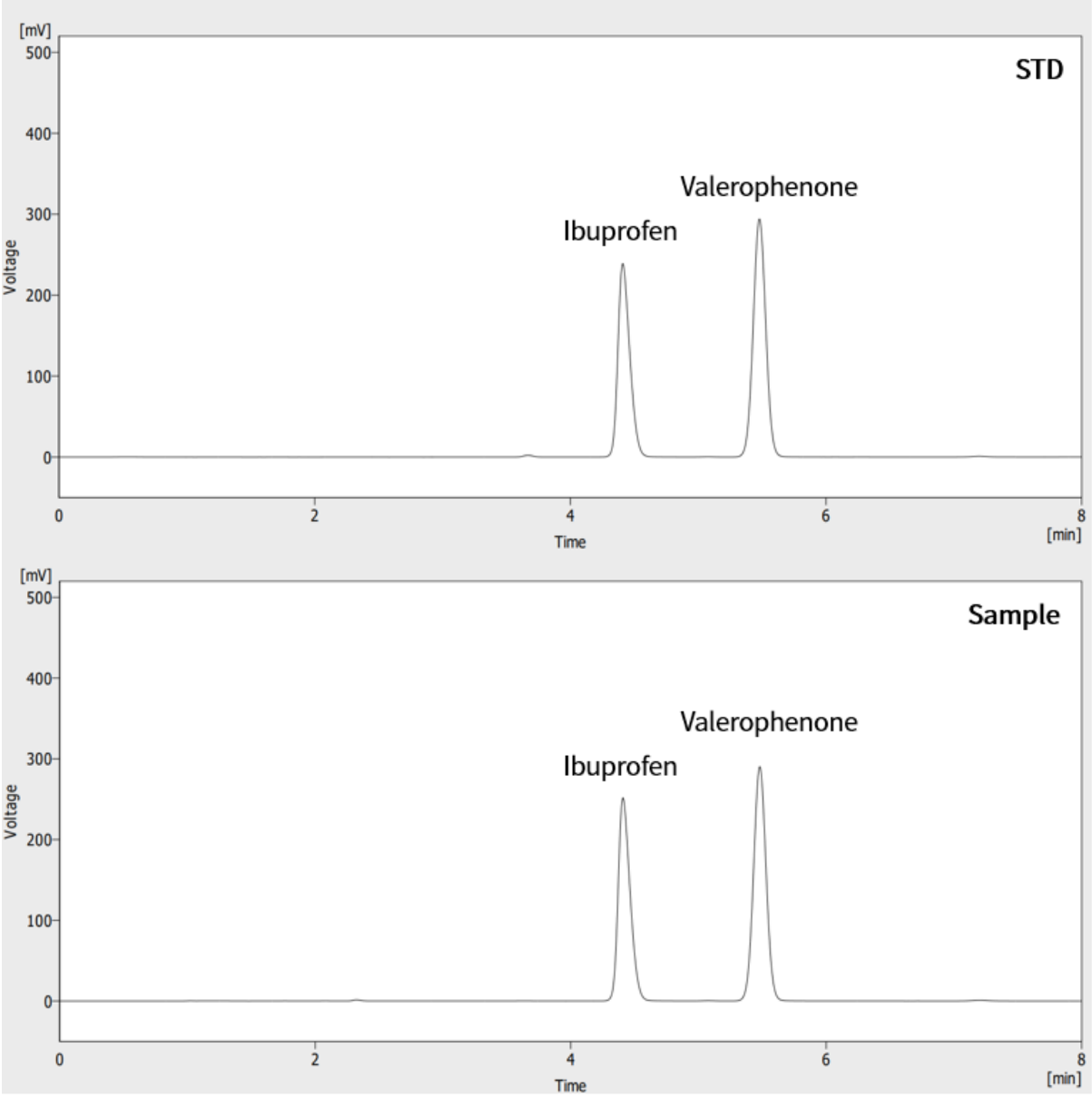

图1. 布洛芬和戊烯酮（ISTD）的ChroZen HPLC标准和样品色谱图

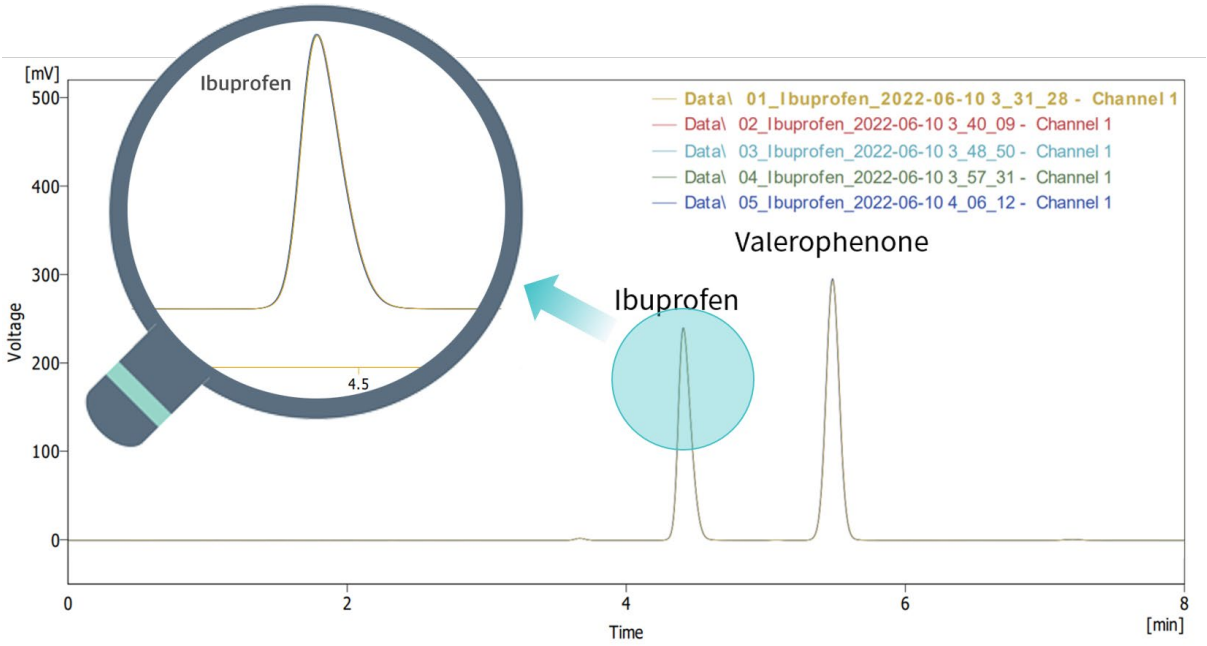

图 2. 通过ChroZen HPLC叠加5次重复注射的标准色谱图

表 1. ChroZen HPLC法测定5次重复注射试验方法的有效性

5 replicate injections	Resolution	Area RSD (%)	
		Ibuprofen	Valerophenone
USP Regulation	≥ 2.5	≤ 2.0	≤ 2.0
Result	6.04	0.293	0.267
	Pass	Pass	Pass

表2.ChroZen HPLC法测定20次重复注射试验方法的有效性

20 replicate injections	Resolution	Area RSD (%)	
		Ibuprofen	Valerophenone
Result	6.04	0.323	0.319

ChroZen UHPLC System	
Mobile phase	10% Chloroacetic acid(pH 3, NH_4OH) : ACN = 40 : 60
Flow rate	0.6 mL/min
Column	Proshell C18 (2.1 mm x 100 mm, 2.7 µm)
Temperature	30°C
Injection volume	1 µL
Detection	UV/Vis detector 254 nm

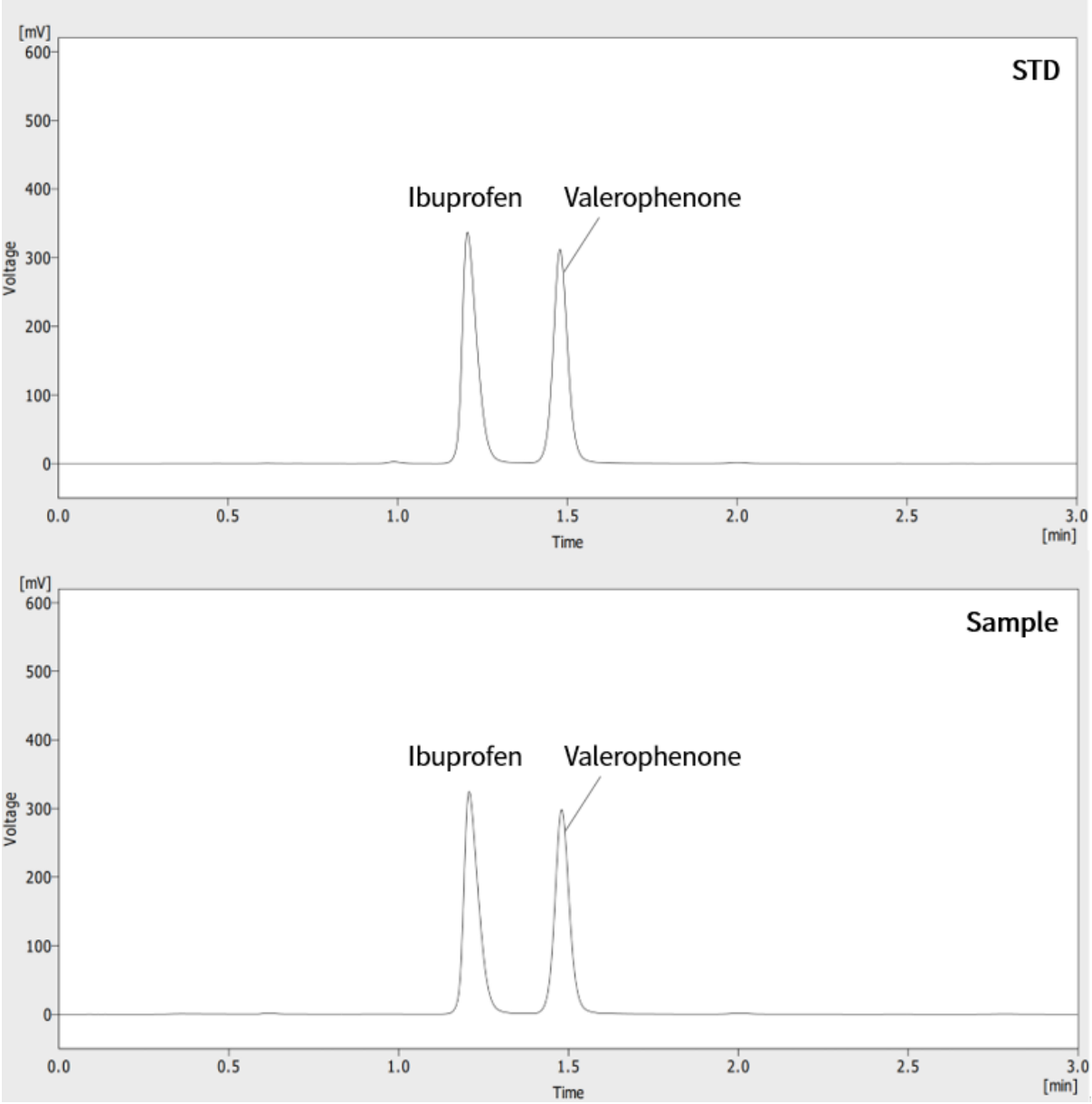

表3. 通过ChroZen UHPLC绘制布洛芬和戊烯酮（ISTD）的标准和样品色谱图

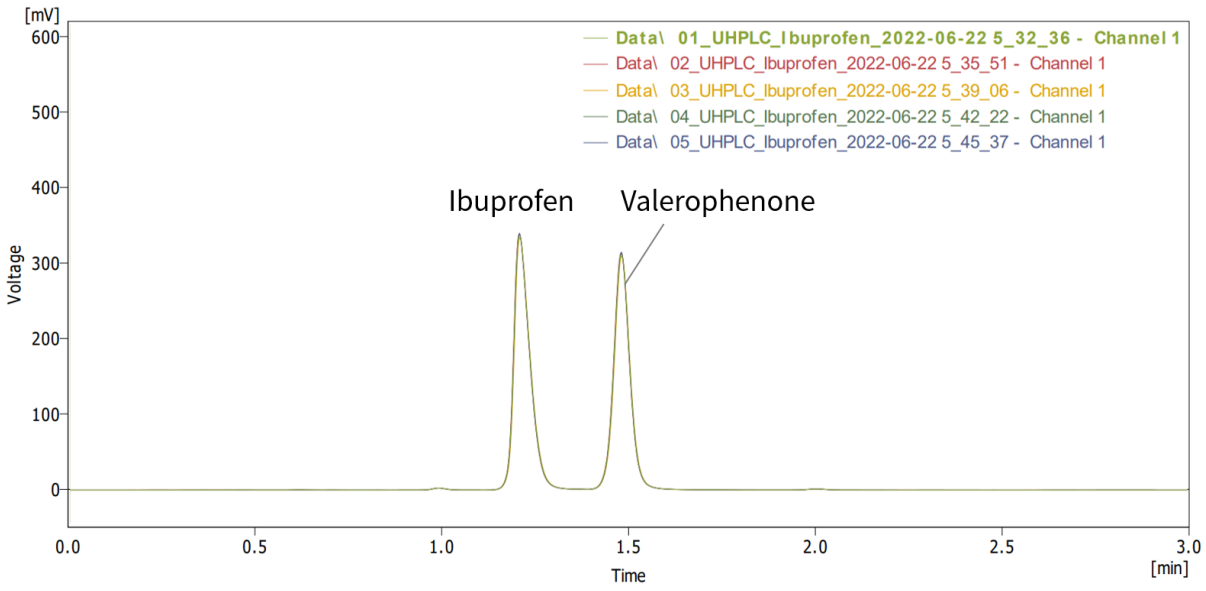

图4. ChroZen UHPLC对标准色谱图的5次重复进样进行叠加

表3. ChroZen UHPLC 5次重复注射试验方法的有效性

5 replicate injections	Resolution	Area RSD (%)	
		Ibuprofen	Valerophenone
USP Regulation	≥ 2.5	≤ 2.0	≤ 2.0
Result	3.42	0.638	0.582
	Pass	Pass	Pass

系统适用性

	USP <621>	HPLC Condition	UHPLC Condition
pH of mobile phase	± 0.2	As specified	As specified
Concentration of salts in buffer	± 10%	As specified	As specified
Ratio of components in mobile phase	± 30% Relative Cannot exceed ± 10%	As specified	As specified
Wavelength of UV-visible detector	± 3 nm	As specified	As specified
Column length	*Isocratic separation* ; Column length(L) to particle size diameter(dp) ratio can be adjusted between -25% and 50% *Gradient separation* ; Not allowed	As specified	As specified
Column inner diameter	Can be adjusted if the linear velocity maintained	As specified	2.1 mm (As specified)
Particle size	*Isocratic separation* ; Column length(L) to particle size diameter(dp) ratio can be adjusted between -25% and 50% *Gradient separation* ; Not allowed	As specified	2.7 µm (As specified)
Flow rate	± 50%(Isocratic only)	As specified	0.6 mL/min (As specified)
Injection volume	Can be adjusted as far as it is consistent with aceepted precision, linearity, and detection limit	As specified	1 µL (As specified)
Column temperature	± 10°C	As specified	As specified

结论

本研究旨在验证该系统是否适用于通过ChroZen HPLC和ChroZen UHPLC分析布洛芬。按照USP 621的规定，采用ChroZen HPLC的分析方法。布洛芬和戊烯酮之间的分辨率为6.04，布洛芬和戊炔酮的面积再现性（%RSD）为0.293和0.267，达到了USP要求的满意结果[表1]。为了确认系统的可靠性，进行了20次重复注射，结果与5次重复注射中的一次相似[表2]。

布洛芬的分析也由ChroZen UHPLC按照USP 621的规定进行。在分析条件的允许变化范围内调整柱和流动相的条件。布洛芬和戊烯酮之间的分辨率为6.04，布洛芬和戊炔酮的面积再现性（%RSD）分别为0.638和0.582。这也符合USP的要求[表2]。使用ChroZen UHPLC，流动相和溶液的消耗大大减少，分析时间也大大缩短，从而提高了生产率。

因此，ChroZen HPLC和ChroZen UHPLC都完全适用于布洛芬的分析，需要根据用户的偏好和条件选择系统。

参考

- USP <621> CHROMATOGRAPHY

- USP Ibuprofen, Ibuprofen tablets

- Korean Pharmaceuticals

YOUNG IN Chromass

60, Anyangcheondong-ro, Dongan-gu,
Anyang-si, Gyeonggi-do, 14042, Korea
TEL: +82-31-428-8700
FAX: +82-31-428-8787
E-mail: export@youngincm.com
Homepage: https://www.youngincm.com/