Rapid Simultaneous Analysis of Valerenic Acids Using the Agilent 1290 Infinity LC System and Sub-2 Micron Columns

Application Note

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Introduction

Advancements in LC instrumentation and column design have drastically increased the potential efficiency of many LC analyses. The instrumental quantification of valerenic acids can be completed approximately 4x faster than the current USP instrument method using a sub-2 µm (STM) column, the Agilent 1290 Infinity LC System, and an alternative instrument method developed at Schwabe North America. It also reduces solvent consumption significantly.

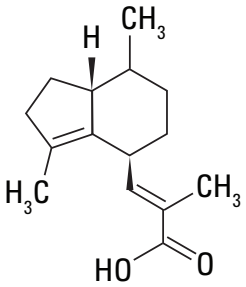
Agilent Technologies

Valerenic acids

Valerenic acid and the analogs hydroxyvalerenic and acetoxyvalerenic acid are the active components of the herb *Valeriana officinalis*. It has been used to treat insomnia and other sleep disorders. Though not fully understood, there are some basic theories about the physiological pathways in which the herb works. Powdered valerian root extracts typically have a distinct unpleasant odor not characteristic of the valerenic acids themselves.

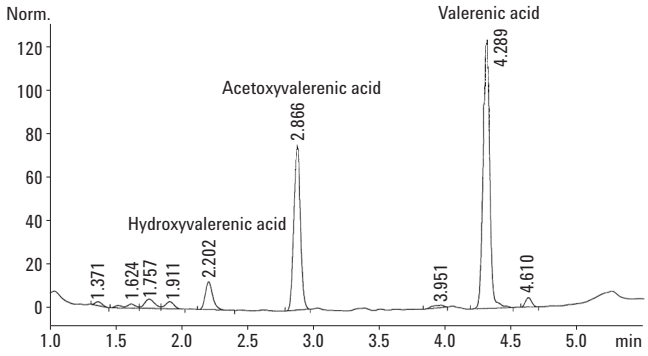
Extraction procedure

USP Method: Sonicated in 25/75 aqueous 0.1 % *o*-phosphoric acid/methanol.



Results and Discussion

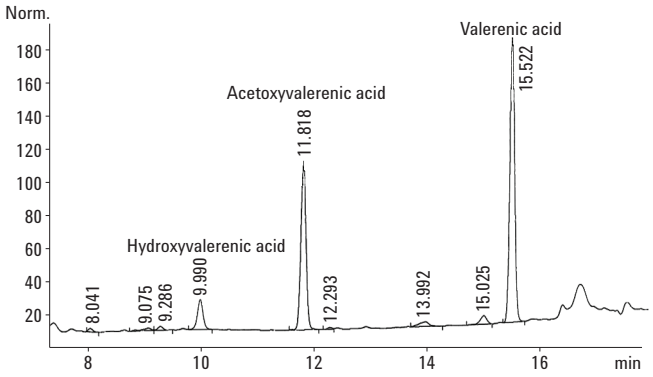
The new method and instrumentation can separate valerenic acid and its analogs for quantification in 10 minutes (Figure 1). This is faster than the USP analysis, which takes 37 minutes (Figure 2). The USP analysis uses a 4.6 × 250 mm, 5 µm C-18 column, while the new Schwabe North America method uses an Agilent ZORBAX Eclipse C-18, 2.1 × 50 mm, 1.8 µm column.



Instrument parameters

Temperature	40 °C		
Injection amount	2 µL		
Detection	UV (DAD), 225 nm		
Flow rate	0.33 mL/minute		
Mobile Phases	A) Water with 0.3 % *o*-phosphoric acid B) Methanol with 0.3 % *o*-phosphoric acid		
Gradient	Time	% A	% B
	0.0	40	60
	6.0	5	95
	8.5	5	95
	9.5	40	60
	10.0	40	60

Figure 1. Valerenic acids in a multicomponent matrix using an Agilent 1290 Infinity LC System with an Agilent ZORBAX Eclipse C-18, 2.1 × 50 mm, 1.8 µm column.



Instrument parameters

Temperature	40 °C
Injection amount	15.0 µL
Detection	UV (DAD), 225 nm
Flow rate	1.0 mL/minute
Mobile phases	A) Water with 0.3 % *o*-phosphoric acid B) Methanol with 0.3 % *o*-phosphoric acid

Gradient	Time	% A	% B
	0.0	40	60
	15.0	5	95
	25.0	5	95
	30.0	40	60
	37.0	40	60

Figure 2. Valerenic acids in a multicomponent matrix using the USP method with an Agilent ZORBAX Eclipse C-18, 4.6 × 250 mm, 5.0 µm column.

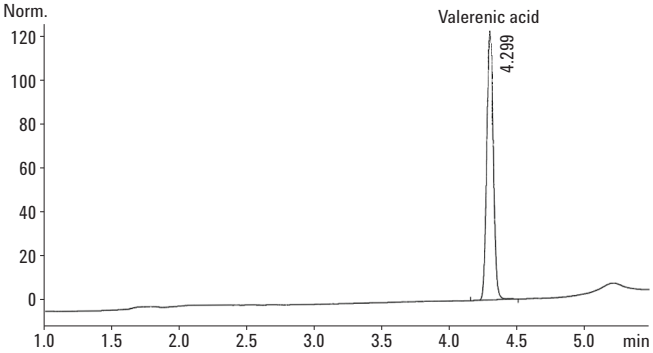


Figure 3. Reference standard, valerenic acid: Indofine (023801S) – Lot# 12121035, concentration: 20.7 µg/mL, % RSD (five injections): 0.048 %.

Conclusion

In analyzing complex herbal blends, the Agilent 1290 Infinity LC System coupled with STM columns can speed up analysis time, thus saving cost in labor and solvent use. This is done without any loss in resolution.

Reference

1. United States Pharmacopeia, Valerian, USP36/NF31 p. 5886. Wikipedia contributors, 'Valerenic acid', Wikipedia, The Free Encyclopedia, 2 October 2013, 02:52 UTC, http://en.wikipedia.org/w/index.php?title=Valerenic_acid&oldid=575382648

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