



## Improved Peak Resolution and Shape for Antidepressants on a New Silica-Based Reversed-Phase Column

John Ezzell, Rosanne Slingsby, and Andre Bordunov,  
Dionex Corporation, Sunnyvale, California

*The increased efficiency and resolution of Dionex Acclaim™ silica-based HPLC columns provide for better peak resolution of tailing compounds and enhanced resolution of complex mixtures. This brief note highlights the significant technical aspects of this new column line.*

Peak tailing in RP-HPLC is a problem that affects peak resolution and integration. It is primarily a result of the ion exchange interaction of positively charged, basic analytes with negatively charged, exposed silanol groups. Above pH ~4, the surface silanols begin to deprotonate and interact with basic analytes. This effect can be reduced by optimizing the surface coverage of the bonded phase, exhaustively endcapping the residual silanol groups, and minimizing metal contaminants that increase the acidity of the free silanol groups.

The Acclaim 120 series is a new line of C18 and C8 silica-based reversed-phase columns manufactured by Dionex. Among the many reversed-phase columns on the market, retention behaviors of analytes differ significantly due to variations in both the raw silica and the ligand bonding chemistry and density. The Dionex Acclaim 120 columns address many of the performance issues encountered with traditional reversed-phase columns. Table I summarizes the technical specifications relevant to this stationary phase.

### Experimental Conditions

Dionex Summit HPLC system with P580 pump, ASI-100 Autosampler and PDA-100 photodiode array detector, controlled via Chromeleon chromatography workstation. Acclaim 120 150 × 4.6 mm, 5-μm C18 column.

**Table I:** Technical specifications of Acclaim stationary phases

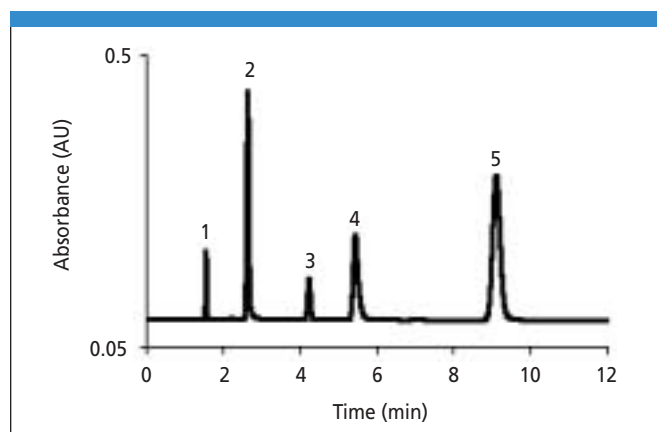
Particle size	3, 5 μm
Bonded phases	C8, C18
Total carbon content	11%, 18%
Average pore diameter	120 Å
Surface area	300 m <sup>2</sup> /g
Metal impurity	<10 ppm
Pore volume	0.90 cm <sup>3</sup> /g
pH range	2–8

### Results

Figure 1 shows the separation of a series of tricyclic antidepressants including amitriptyline. The tailing on amitriptyline is a very good indicator of the number of exposed silanol groups. The following peak asymmetry values were obtained: 1. uracil 1.04, 2. propranolol 1.10, 3. toluene 0.99, 4. doxepin 1.37, 5. amitriptyline 1.10.

### Conclusions

The new Acclaim 120 series is a line of C18 and C8 silica-based reversed-phase columns manufactured by Dionex. The Acclaim 120 line comprises four highly characterized C18 and C8 phases that have very high surface coverage and very low metal content. All the phases are LC-MS compatible with very low background and good capacity for use with high organic mobile phases. The C18 phases exhibit low polarity, high hydrophobicity, and good steric selectivity, which result in high-capacity columns with unique selectivity. All Dionex columns receive extensive testing to ensure column-to-column reproducibility, and are shipped with certificates of analysis detailing these tests.



**Figure 1:** Separation of tricyclic antidepressants. 80:20 MeOH-KH<sub>2</sub>PO<sub>4</sub> (30 mM, pH 6), 1 mL/min, 20 °C, UV, 220 nm, 5-μL injection volume.

### Dionex Corporation

1228 Titan Way, P.O. Box 3603, Sunnyvale, CA 94088-3603  
tel. (408) 737-0700, fax (408) 730-9403  
<http://www.dionex.com>