

application note

use of the expressLC™ system to monitor the acid-induced degradation of steroids in rat chow

degradation study highlights the ability of the expressLC system to perform determinations in exceptionally complex matrices

introduction

A sample matrix that is expected to be an important test of any HPLC system's ability to tolerate "dirty" samples would be rat chow. With fats, carbohydrates, and dozens of added vitamins and minerals, rat chow residues present even after sample preparation have the potential to clog the injector, column, and optical cell. Real-world samples are often more complex than "dilute and shoot" standards. As figure 1 demonstrates, there is no degradation of chromatographic efficiency of steroid peaks in the presence of less-than-pristine samples. Count on the ExpressLC systems to provide fast, high quality chromatography and data.

figure 1. chromatogram of acid-degraded steroids in rat chow

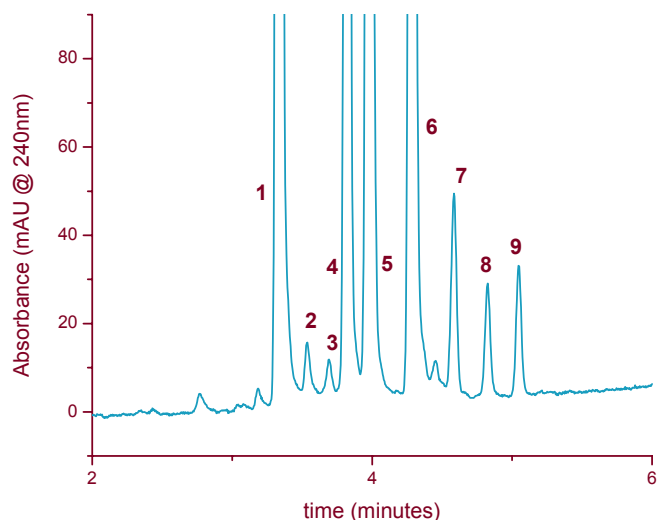


figure 2. experimental conditions and species studied

Instrument: ExpressLC-100 system
 Column: Zorbax 50 x 0.32 mm , 3.5 µm particle
 Mobile phase: A/B water/acetonitrile with 0.1% TFA
 Flow rate: 5 µL/min
 Gradient: 10 to 90 % acetonitrile in 8 min
 90% acetonitrile for 2 min
 Sample: 40 nL injection of steroid mix:
 120 µg/mL prednisone
 80 µg/mL dexamethasone
 70 µg/mL corticosterone
 290 µg/mL hydrocortisone acetate

species	peak	area %
prednisone	1	36.8
degradation 1	2	0.56
degradation 2	3	0.35
dexamethasone	4	18.4
corticosterone	5	18.5
hydrocortisone acetate	6	22.2
degradation 3	7	1.44
degradation 4	8	0.83
degradation 5	9	0.94

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expressLC system specifications**configuration**

expressLC-100 Single-channel System: Includes binary gradient pump, electronic injection valve, column temperature control, and array-based UV detection system. Optional high-speed autosampler available.

expressLC-800 8-channel Parallel System: Includes 8 binary gradient pumps, 8 electronic injection valves, 8 column temperature control compartments, an array-based UV detection system and high-throughput autosampler.

flow rate range

0.20–30 $\mu\text{L}/\text{min}$

pump type

Microfluidic direct pumping system with independent flow control feedback for each mobile phase. Retention time RSD < 0.5%.

gradient formation

High pressure gradient mixing. System can run full gradients as rapidly as 8 seconds. Maximum gradient length 2 hrs. at 5 $\mu\text{L}/\text{min}$.

delay volume

< 500 nL from mixer to column.

mobile phase compatibility

All mobile phases compatible with 316 stainless steel, PEEK, and silica.

injection valve

Eksigent Variable-Volume Injection System (software selectable). Standard injection volume 10–250 nL (larger injection volumes available).

columns

System optimized for 2.5–15 cm, 300 μm i.d. capillary LC columns

column temperature control

Software selectable from 27–40°C; stability within $\pm 0.1^\circ\text{C}$

detection

UV absorbance detection from 200–380 nm using linear CCD array detector. Detector drift $\leq 4 \times 10^{-4}$ AU/hr Non-linearity $\leq 5\%$ @ 2 AU.

flow cell

45 nL microfabricated flow cell with integral fiber optics, 4 mm path length

autosampler

High-throughput CTC autosampler available

system control

Computer with graphical user interface for control of all system parameters. Software allows import of run tables and creates CDF, text, and Excel files for data export and analysis. Tracking of instrument runtime, column usage, total injections, solvent usage, lamp hours, and error codes. System drivers available for Thermo Electron's Xcalibur and Applied Biosystems/MDS SCIEX Analyst 1.4.1 mass spectrometer software.

report features

Generates reports that include method conditions, chromatograms, peak retention times and areas, and spectral absorbance map.

dimensions**expressLC-100 System:**

21" (53 cm) wide, 20" (51 cm) deep, 18" (46 cm) high

expressLC-100 Autosampler:

Additional 14" (36 cm) high and 6" (15 cm) wide

expressLC-800 System:

30" (76 cm) wide, 34" (86 cm) deep, 40" (102 cm) high

expressLC-800 Autosampler:

Additional 16" (41 cm) high and 16" (41 cm) wide

computer

Additional lab space needed for keyboard, mouse and monitor