

application note

accelerated thermal degradation of indomethacin drug product

thermal degradation study highlights the expressLC™ system's throughput and selectivity

introduction

Whether it is degradation studies to challenge the specificity of a drug substance or drug product assay, analysis of toxicological dosing solutions, or long term stability studies (LTSS)-the number of samples can be overwhelming. For specificity and LTSS studies, selectivity is a crucial aspect of analysis: low level impurities need to be detected before they become regulatory issues. The high throughput and excellent selectivity of the ExpressLC systems is expressly designed to address these time and resource intensive activities. Do you need to re-inject some samples due to instrumental failure during a long instrumental run? The ExpressLC system can make several injections with the sample volume required for one conventional LC analysis. No more embarrassing "insufficient sample for analysis" data entries. ExpressLC systems transform routine analyses into consistent, high-quality data.

figure 1. chromatogram of thermally-degraded indomethacin

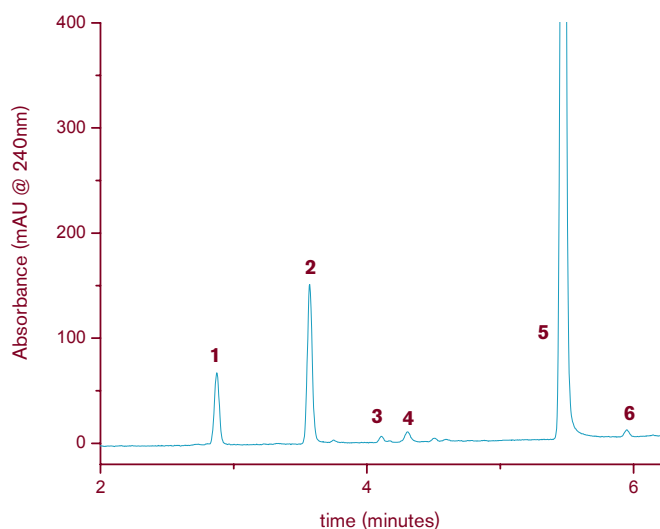


figure 2. experimental conditions and area percent table

Instrument: ExpressLC system
 Column: Zorbax 300SB-C18 50 x 0.32 mm, 3.5µm particle
 Mobile phase: A/B water/acetonitrile with 0.1% TFA
 Flow rate: 5 µL/min
 Gradient: 10 to 90 % acetonitrile in 8 minutes
 90% acetonitrile hold for 2 minutes
 Sample: 40 nL injection of thermally-degraded indomethacin

species	peak	area %
thermal a	1	4.6
thermal b	2	9.9
thermal c	3	0.39
thermal d	4	0.80
indomethacin	5	83.8
thermal e	6	0.45

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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