

# Streamlined Sample Preparation Methodology to enable Higher Recovery, and minimize loss of Pesticides, Fungicides and Antibiotics by LC/MS or GC/MS

**THOMSON** Solutions™  
At Work  
INSTRUMENT COMPANY

Presented by: Lisa Wanders, Joe Machamer

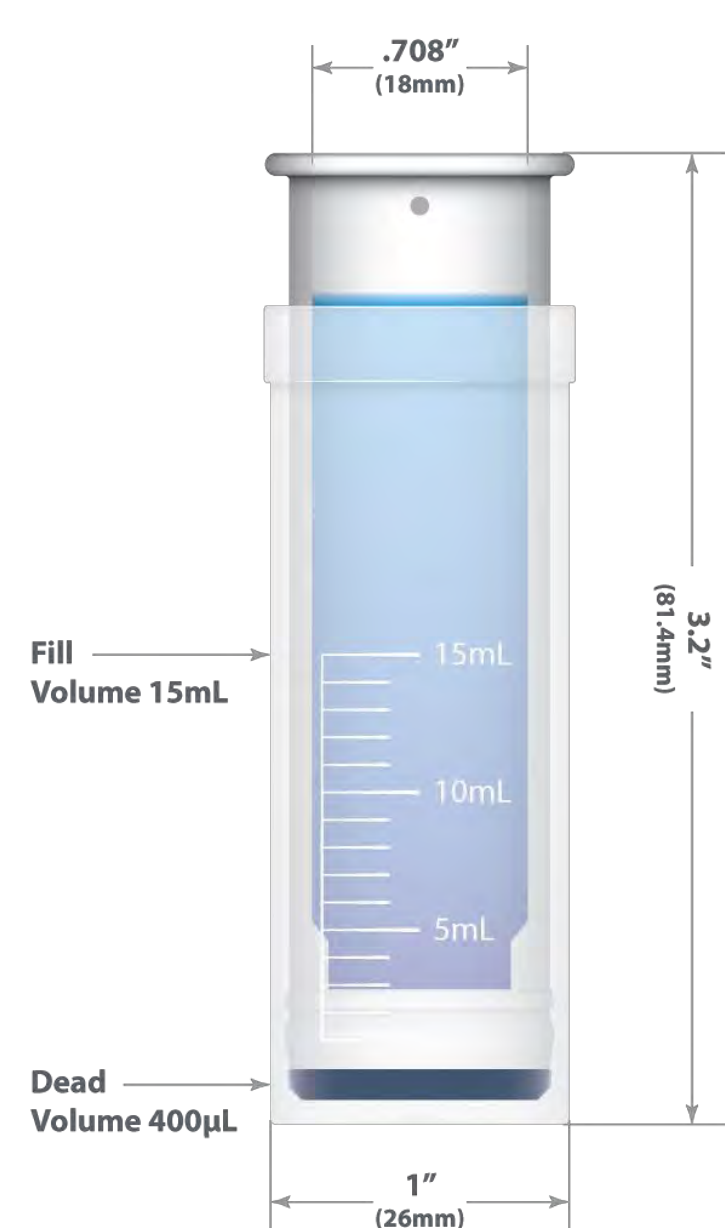
## MEGA|FV

### Filter Once for Multiple Applications

The MEGA|FV™ (patented) with 15mL working volume, from Thomson Instrument Company, performs similarly to our other Filter Vials. Available in multiple membranes (PVDF, PTFE & Nylon) and pore sizes (0.45µm & 0.2µm), the MEGA|FV™ can be used for many applications from biologics to chemistry to food science.

The MEGA|FV™ is a valuable tool for fast filtration of large sample volumes in your daily applications. It allows for filtration of up to 15mL in one vial, letting you move quickly from sample clean-up to analysis or storage, and providing clean, filtered samples ready for multiple applications.

The Multi-Use Press (p/n # 35015) can accommodate up to 8 MEGA|FV™s at a time. The Multi-Use Press 8-Position Rack fits most standard robots for automated sample applications.



### Key Features

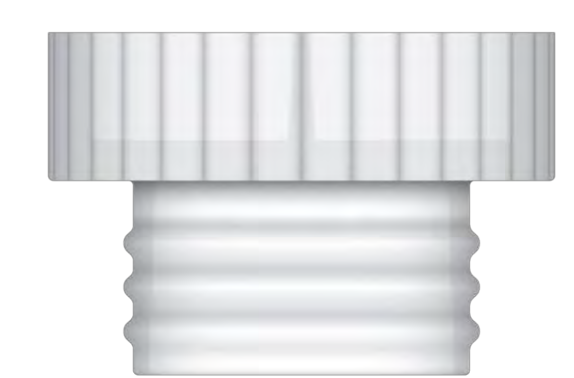
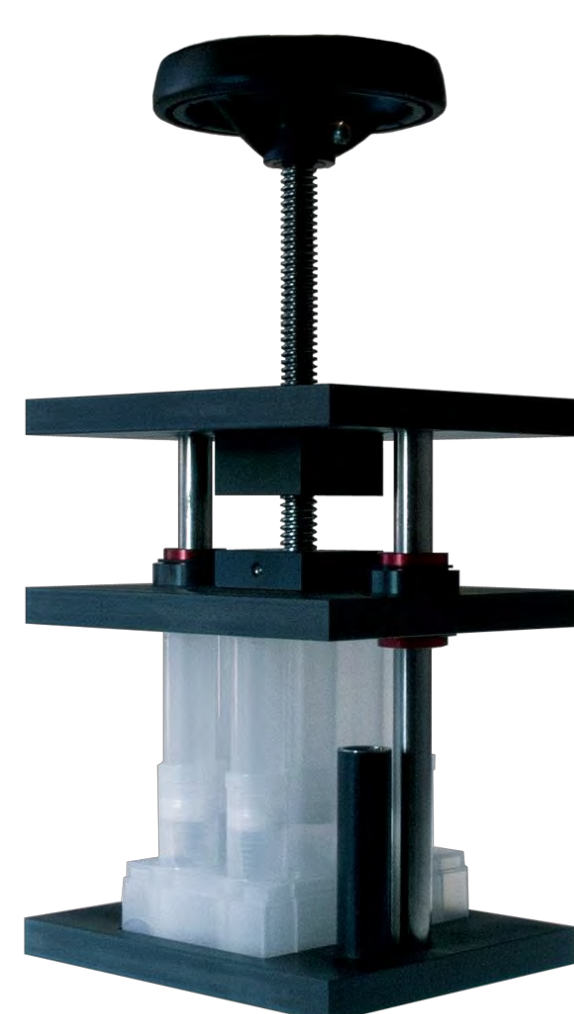
- Fast Filtration for large volumes, up to 15mL
- Minimum sample loss
- Long-term storage cap available
- Automation-friendly with 8-Position Rack



### Multi-Use Press

Case Qty: 1 | Part # 35015

8 Position for MEGA|FV / Optimum Growth™ Tube  
48 Position for Autosampler Ready Filter Vials



### MEGA Storage Cap

Storage Cap for use with MEGA|FV™



## Analysis of Antibiotics in Honey by an Integrated On-Line Extraction UHPLC-MS/MS System

Zicheng Yang and Louis Maljers  
Bruker Daltonics Inc., 3500 West Warren Ave, Fremont, CA 94538  
NACRW 2015

### UHPLC Conditions

Trap Column: YMC-Pack ODS-AQ, 10 µm, 10 mm x 3.0 mm I.D.  
Mobile Phase C: 0.1% FA in water  
Equilibration flow: 1000 µL (4.0 min)  
Loading Flow: 500 µL  
Analytical Column: YMC- UltraHT Pto C18, 2 µm, 100 mm x 2.0 mm I.D.  
Column Temperature: 40 °C  
Injection Volume: 10 µL (100 µL Loop)  
Mobile Phase A: 0.1% FA in water  
Mobile Phase B: MeOH

LC Gradient			
Time min.	Mobile Phase A (%)	Mobile Phase B (%)	Flow Rate µL/min.
0.0	80	20	200
0.2	80	20	200
4.0	0	100	200
6.0	0	100	200
6.1	80	20	200
8.0	80	20	200

### Bruker EVOQ MS Conditions

Source parameters	
Source:	HESI
Spray Voltage (+)	4000 V
Cone Gas Flow	20
Cone Temperature	350 °C
Heated Probe Gas Flow	45
Heated Probe Temperature	400 °C
Nebulizer Gas Flow	55
Exhaust Gas	On

Compound Name	Retention Time	Q1 First Mass	Q3 First Mass	Q3 Resolution	Collision Energy	Dwell Time (ms)	Quantifier Ions	Qual Mass
Ciprofloxacin	3.168	332.2	230.9	0.7	-18	86.4	230.90	314
		245	0.7	-18	49.4			
Enrofloxacin	3.201	360.3	286	0.7	-29	74.1		286
		316	0.7	-15	74.1			316
Tetracycline	3.169	445.2	154	1	-24	74.1	410.0	154
		427.1	0.7	-9	74.1			427.1
Erythromycin	4.370	734	158.1	2	-27	166.7	158.1	
		576.3	2	-16	166.7			576.3

### Sample Preparation

- Weigh approximately 50 mg of honey into the outer shell of the eXtreme|Filter Vial (p/n 85531, Thomson Instrument Company).
- Add solvent (MeOH/water, 50/50, v/v) to make 100 mg/mL solution.
- Mix by pipet and press the filter plunger, 0.2 µm PVDF, of the eXtreme|Filter Vial (p/n 85531) completely to filter.
- Solution is ready for injection.

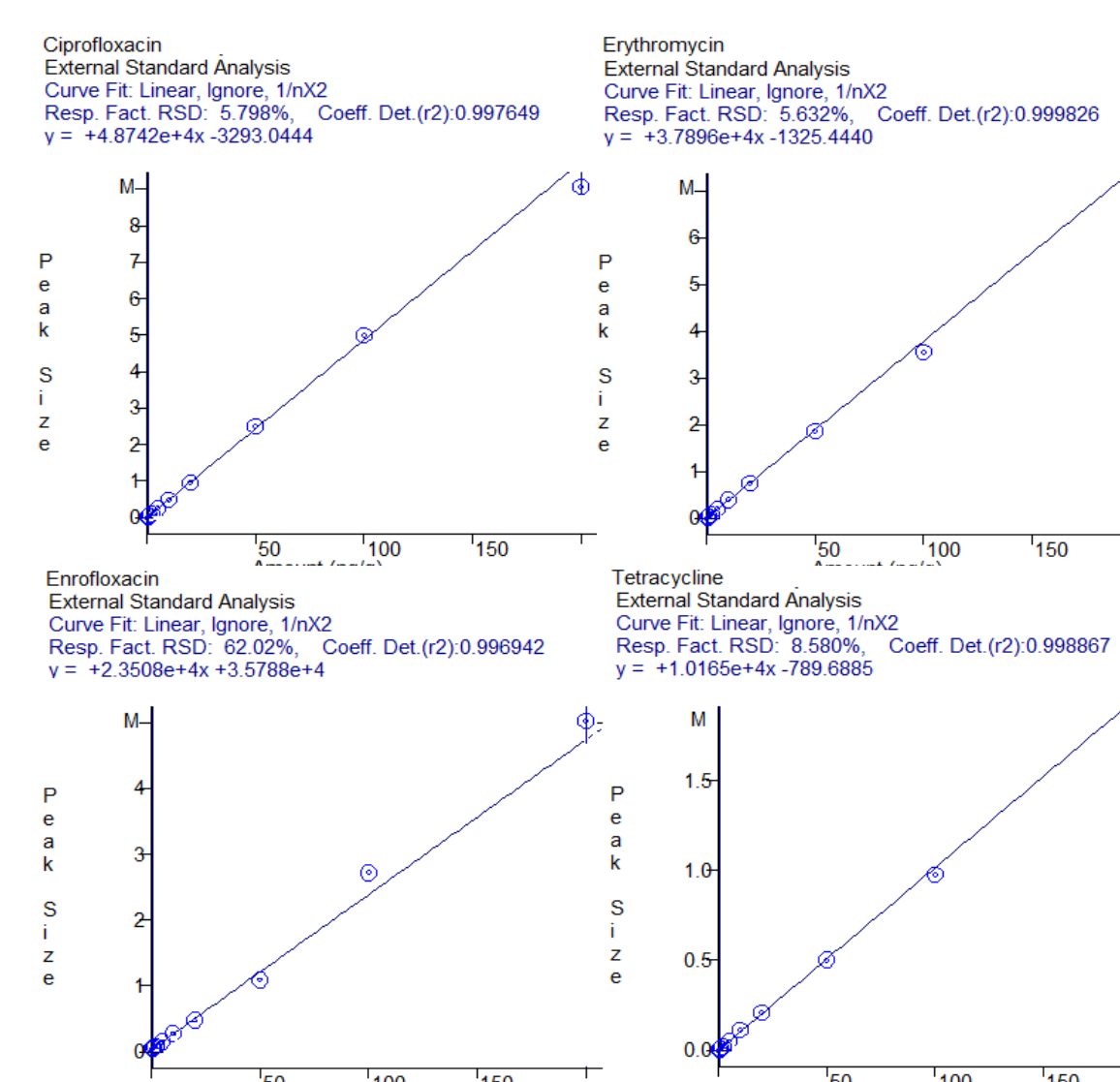


### Antibiotics

Name	Structure	Fomular	Tolerance Limit (ug/kg, ppb)
Erythromycin		C <sub>37</sub> H <sub>67</sub> NO <sub>13</sub>	5
Ciprofloxacin		C <sub>17</sub> H <sub>18</sub> FN <sub>3</sub> O <sub>3</sub>	5
Enrofloxacin		C <sub>19</sub> H <sub>22</sub> FN <sub>3</sub> O <sub>3</sub>	5
Tetracycline		C <sub>22</sub> H <sub>24</sub> N <sub>2</sub> O <sub>8</sub>	5



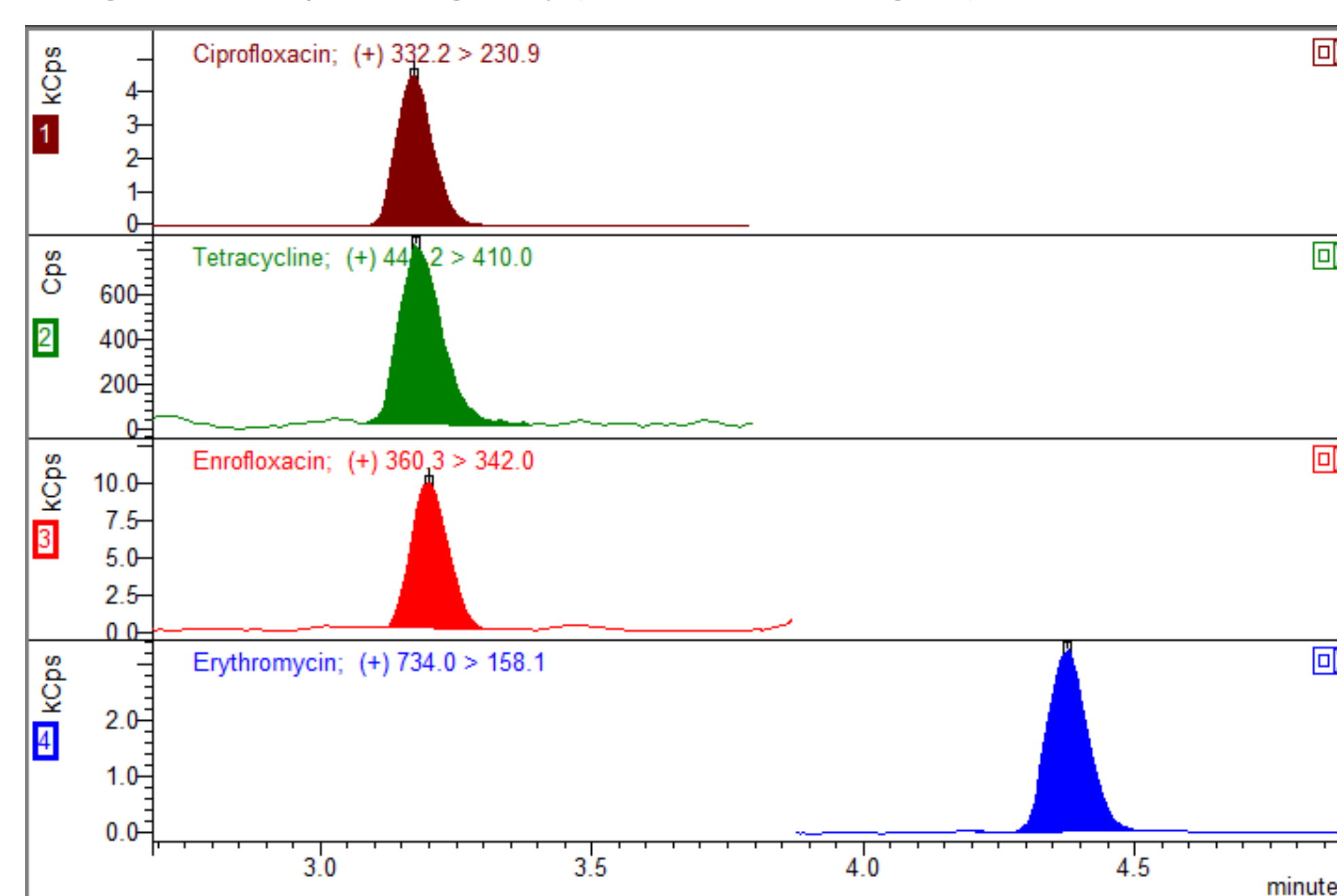
### Calibration Curves -Antibiotics in Honey



Calibration Level	ng/g	ng/mL
1	0.5	0.05
2	1	0.1
3	2	0.2
4	5	0.5
5	10	1
6	20	2
7	50	5
8	100	10
9	200	20

### Chromatograms

0.5ng antibiotics spiked in 1g honey (concentration: 0.05ng/mL)



### Test Result

Tolerance Limit for all four antibiotics is 5 µg/kg.

Antibiotics	Ciprofloxacin	Enrofloxacin	Erythromycin	Tetracycline
Honey Source	ng/g			
USA-1	ND	ND	ND	ND
USA-2	ND	ND	ND	ND
USA-3	ND	ND	ND	ND
Canada	ND	ND	ND	ND
China	ND	ND	ND	ND
India	ND	ND	ND	3.8

ND: Not Detected or <0.5ng/g. test result based on calibration curve of antibiotics in honey. The antibiotics was spiked in Honey USA-1. Tolerance Limit for all four antibiotics is 5 µg/kg.

### Recovery

- Calculations are based on matrix matched calibration curves = 100/(detected amount/spiked amount)
- The recovery for Ciprofloxacin and Erythromycin looks consistent across all levels. The Enrofloxacin signal is enhanced in matrix and Tetracycline signal is enhanced at low concentration.

Recovery				
Standard(ng/g)	Ciprofloxacin	Enrofloxacin	Erythromycin	Tetracycline
0.5	109.6	-	85.5	191
1	121.1	-	85.7	278
2	114.2	6666.7	88.2	233
5	158.9	511.8	83.8	335
10	93.2	207.4	86.1	116
20	111.3	202.8	88.9	169
50	103.3	180.7	93.4	132
100	109.8	179.1	96.2	127
200	133.4	190.5	99.3	120