



Application phare : Antibiotiques vétérinaires de différentes classes sur Raptor C18 par LC-MS/MS

Une seule analyse, une seule colonne, et moins de 9 minutes pour plus de 60 antibiotiques de différentes classes

- Excellente séparation des composés et temps d'analyse rapides.
- Polyvalente et applicable à tout type d'analyse de résidus d'antibiotiques — et optimisation possible pour la quantification individuelle de panels de différentes classes :

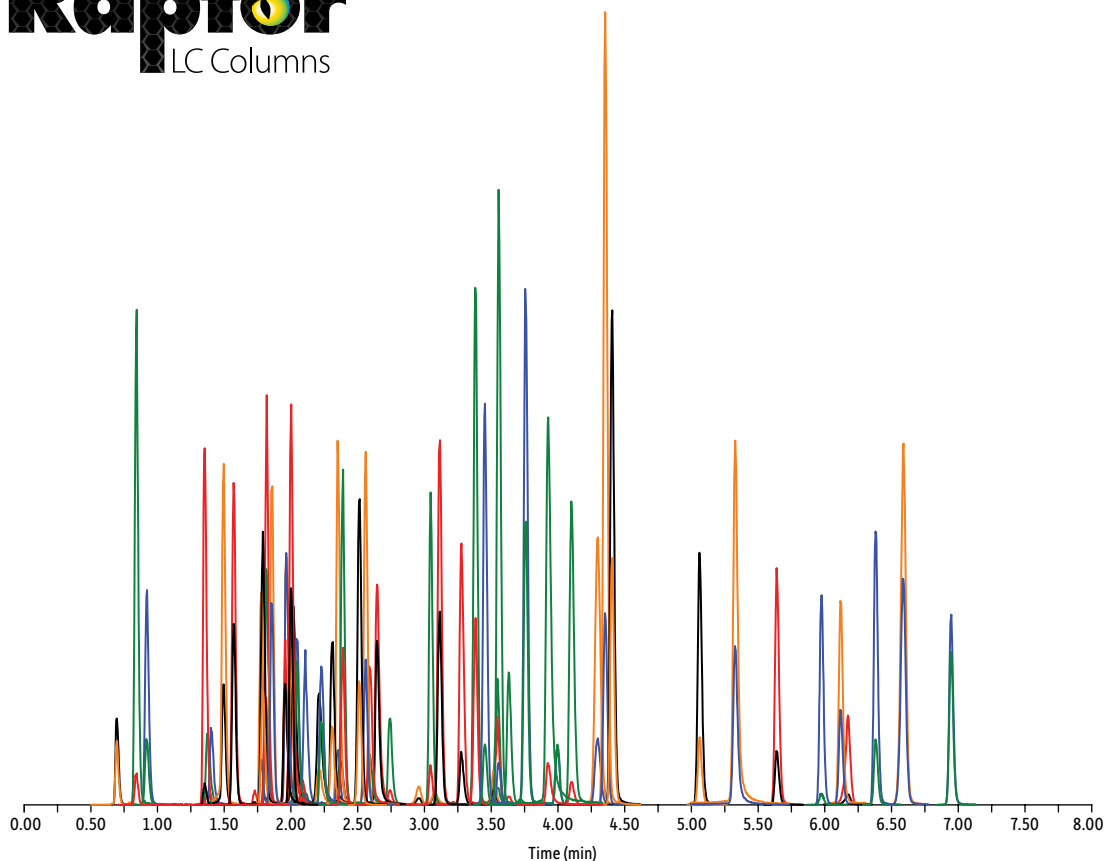
- Macrolide, Lincosamide, et Streptogramine (Figure 1)
 - Amphénicol et Tétracycline (Figure 2)
 - Quinolone (Figure 3)
 - Pénicilline, Céphalosporine, et Tétracycline (Figure 4)
 - Sulfonamide (Figure 5)
- (pour les ionophores, utiliser la colonne Raptor Biphenyl. [Figure 6])

L'utilisation d'antibiotiques sur les animaux élevés pour la consommation humaine est une préoccupation de Santé Publique et de sécurité en raison de la génération potentielle de bactéries résistantes aux médicaments. De nombreux pays dans l'Union Européenne ainsi que le Canada ont interdit l'utilisation d'antibiotiques à des fins non thérapeutiques, et les États-Unis définissent une politique visant à réduire l'utilisation d'antibiotiques médicalement importants pour stimuler la croissance. Afin de réglementer l'usage approprié des antibiotiques vétérinaires, la FDA aux États-Unis a fixé des limites maximales pour les résidus (MRL – « Maximum Residue Limits ») pour différents tissus animaux et produits alimentaires (21 CFR Part 556). Une méthode analytique sensible, efficace et fiable pour différentes classes d'antibiotiques est donc nécessaire pour répondre à ces exigences réglementaires, et la colonne LC Restek Raptor C18 est la colonne idéale pour cela.

Peaks	tR (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion	Peaks	tR (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion
1. Desacetyl cephalirin	0.70	150	382.03	111.92	124.21	31. Sarafloxacin	2.59	10	386.20	342.20	368.15
2. Sulfanilamide	0.85	200	172.98	93.07	75.23	32. Difloxacin	2.65	10	400.23	356.17	299.13
3. Amoxicillin	0.92	100	366.24	349.10	208.07	33. Cefazolin	2.75	100	455.10	323.06	295.09
4. Cephalirin	1.36	50	424.17	292.08	124.14	34. Spiramycin	2.96	200	843.64	540.36	699.48
5. Tildipirosin	1.38	200	734.59	561.45	204.15	35. Pirlimycin	3.05	20	411.32	363.18	327.21
6. Desfuroyl ceftiofur cysteine disulfide	1.40	300	549.16	183.02	126.00	36. Chlortetracycline	3.08	25	479.27	154.07	371.06
7. Lincomycin	1.50	50	407.32	359.23	389.28	37. Sulfachlorpyridazine	3.12	20	285.05	156.03	108.09
8. Sulfadiazine	1.57	20	251.18	156.04	92.08	38. Gamithromycin	3.28	100	777.63	619.52	601.45
9. Cefquinome	1.73	200	529.19	134.10	125.12	39. Sulfadoxine	3.39	10	311.17	156.03	108.09
10. Ampicillin	1.78	50	350.19	106.07	160.06	40. Sulfamethoxazole	3.46	20	254.18	155.98	147.06
11. Sulfathiazole	1.79	10	256.16	156.03	92.08	41. Cefoperazone	3.52	100	646.26	143.07	148.02
12. Marbofloxacin	1.81	10	363.20	72.11	320.10	42. Florfenicol*	3.55	200	356.10	336.02	184.98
13. Cefalexin	1.82	100	348.10	158.05	174.05	43. Sulfaethoxyypyridazine	3.56	20	295.17	267.07	156.03
14. Sulfapyridine	1.86	10	250.13	156.10	92.08	44. Tilimicosin	3.64	100	869.72	696.50	522.42
15. Norfloxacin	1.96	20	320.23	276.20	233.13	45. Sulfisoxazole	3.76	20	268.14	156.03	113.10
16. Ofloxacin	1.98	10	362.21	318.20	261.15	46. Oxolinic acid	3.94	5	262.10	244.06	215.96
17. Sulfamerazine	2.00	20	265.08	156.03	92.08	47. Chloramphenicol*	4.00	200	321.16	151.99	257.04
18. Cefalonium	2.01	100	459.16	337.03	123.10	48. Ceftiofur	4.11	50	524.14	241.08	125.24
19. Oxytetracycline	2.02	25	461.27	426.15	443.32	49. Erythromycin	4.31	25	734.64	576.40	558.38
20. Ciprofloxacin	2.04	20	332.18	288.22	245.15	50. Sulfadimethoxine	4.36	10	311.17	156.09	108.09
21. Cefacetrile	2.09	300	362.07	258.08	178.01	51. Sulfamethoxazole	4.42	20	301.18	156.04	108.02
22. Tulathromycin A	2.11	100	806.65	577.42	420.31	52. Tylosin**	4.67	100	916.62	772.49	598.36
23. Tetracycline	2.21	25	445.28	154.07	427.32	53. Penicillin G	5.07	100	335.18	176.07	160.07
24. Danofloxacin	2.23	20	358.22	340.16	314.21	54. Flumequine	5.34	5	262.15	244.11	202.03
25. Enrofloxacin	2.32	10	360.29	316.22	245.13	55. Penicillin V	5.56	100	351.10	160.06	114.07
26. Orfloxacin	2.35	10	396.22	352.17	226.12	56. Oxacillin	5.99	100	402.15	160.05	114.06
27. Thiamphenicol*	2.38	200	354.16	290.04	184.98	57. Virginiamycin M1	6.13	50	526.43	508.31	355.10
28. Sulfamethazine	2.39	10	279.23	186.08	124.08	58. Tylvalosin	6.19	50	1042.71	814.46	640.39
29. Sulfamethizole	2.52	10	271.17	156.02	108.02	59. Cloxacillin	6.39	100	436.15	277.06	160.05
30. Sulfamethoxyypyridazine	2.56	10	281.14	156.03	126.07	60. Nafcillin	6.60	25	415.19	199.09	171.06
						61. Dicloxacillin	6.96	100	470.11	160.05	311.02

*Acquired in negative ion mode

Voir le chromatogramme et les conditions analytiques en page 2



LC_FS0506

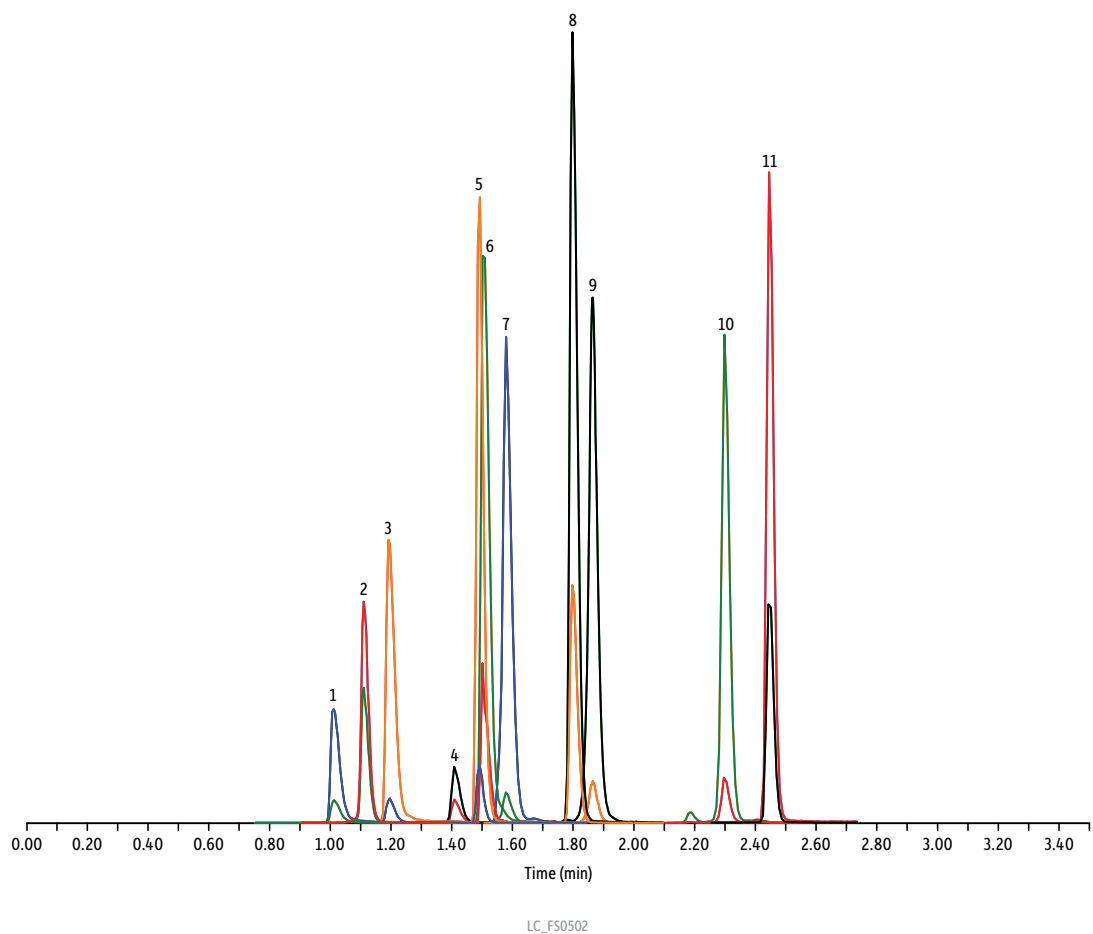
Column Raptor C18 (cat.# 9304A12)
Dimensions: 100 mm x 2.1 mm ID
Particle Size: 2.7 µm
Pore Size: 90 Å
Guard Column: Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)
Temp.: 35 °C
Sample
Diluent: Water
Conc.: 5–300 ng/mL
Inj. Vol.: 2 µL
Mobile Phase
A: 0.1% Formic acid in water
B: 0.1% Formic acid in acetonitrile

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	90	10
4.50	0.4	65	35
7.00	0.4	45	55
7.01	0.4	90	10
9.00	0.4	90	10

Detector MS/MS
Ion Mode: ESI+/ESI-
Mode: Scheduled MRM
Instrument UHPLC
Notes

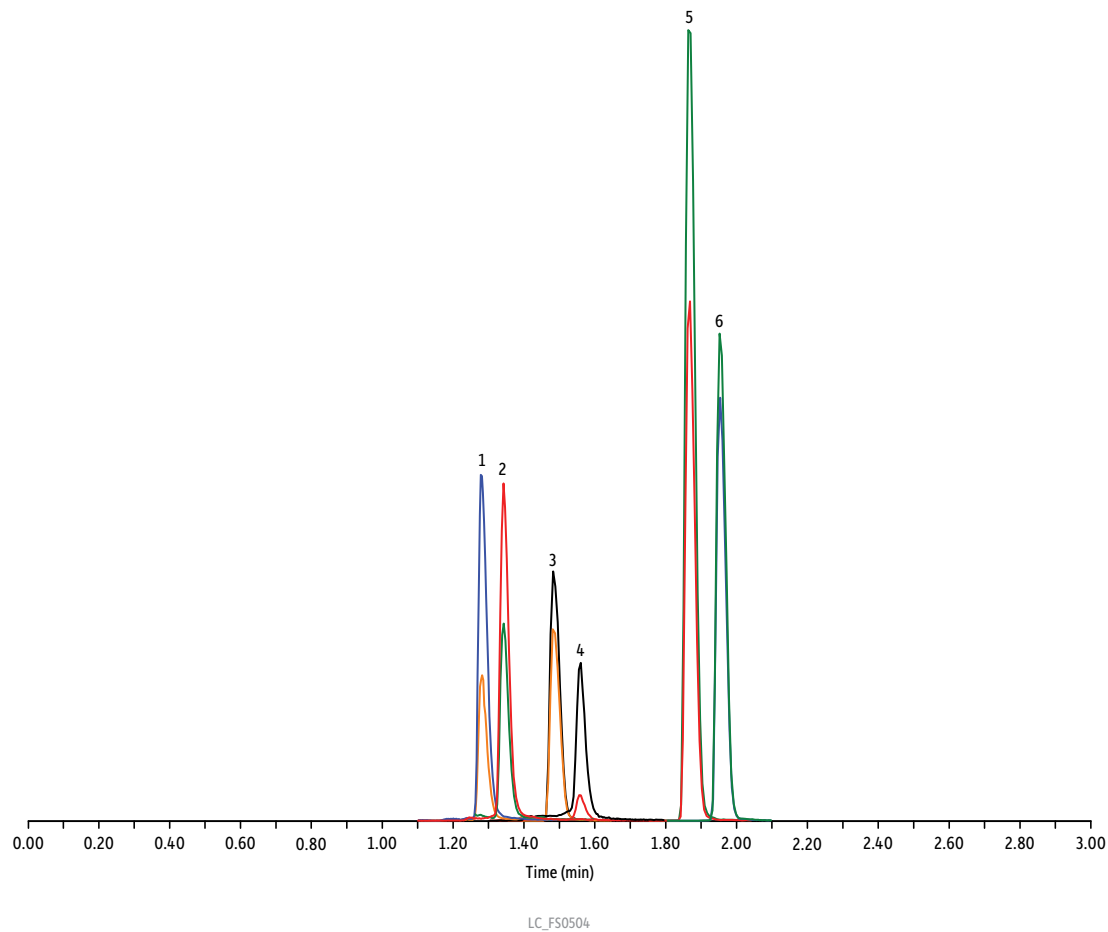
1. Positive and negative polarity data were collected simultaneously from a single injection.
 2. Amphenicol compounds (chloramphenicol, thiamphenicol, and florfenicol) were detected with negative polarity.
 3. The MRM was scheduled at +/- 20 to 30 seconds for each analyte.
 4. Multiclass antibiotics include penicillin, cephalosporin, tetracycline, sulfonamide, macrolide, lincosamide, streptogramin, amphenicol, and quinolone.
- **The retention time for Tylosin is noted in the peak list; however, it was not included in the chromatogram.

Figure 1 : Antibiotiques Macrolide, Lincosamide, et Streptogramine sur Raptor C18 par LC-MS/MS



Peaks	tr (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion	Column
1. Tildipirosin	1.01	200	734.59	561.45	204.15	Raptor C18 (cat.# 9304A12)
2. Lincomycin	1.11	50	407.32	359.23	389.28	Dimensions: 100 mm x 2.1 mm ID
3. Tulathromycin A	1.19	100	806.65	577.42	420.31	Particle Size: 2.7 µm
4. Spiramycin	1.41	200	843.64	540.36	699.48	Pore Size: 90 Å
5. Pirlmycin	1.49	20	411.32	363.18	327.21	Guard Column: Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)
6. Gamithromycin	1.50	100	777.63	619.52	601.45	Temp.: 40 °C
7. Tilmicosin	1.58	100	869.72	696.50	522.42	Sample
8. Erythromycin	1.80	25	734.64	576.40	558.38	Diluent: Water
9. Tylosin	1.87	100	916.62	772.49	598.36	Conc.: 20–200 ng/mL
10. Tyvalosin	2.30	50	1042.71	814.46	640.39	Inj. Vol.: 2 µL
11. Virginiamycin M1	2.45	50	526.43	508.31	355.10	Mobile Phase
						A: 0.1% Formic acid in water
						B: 0.1% Formic acid in acetonitrile
						Time (min) Flow (mL/min) %A %B
						0.00 0.4 90 10
						3.00 0.4 20 80
						3.01 0.4 90 10
						5.00 0.4 90 10
						Detector MS/MS
						Ion Mode: ESI+
						Mode: MRM
						Instrument UHPLC

Figure 2 : Antibiotiques Amphénicol et Tétracycline sur Raptor C18 par LC-MS/MS



Peaks	tr (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion	Column
1. Oxytetracycline	1.28	25	461.27	426.15	443.32	Raptor C18 (cat.# 9304A12)
2. Tetracycline	1.34	25	445.28	154.07	427.32	Dimensions: 100 mm x 2.1 mm ID
3. Thiamphenicol*	1.48	200	354.16	290.04	184.98	Particle Size: 2.7 µm
4. Chlortetracycline	1.56	25	479.27	154.07	371.06	Pore Size: 90 Å
5. Florfenicol*	1.86	200	356.10	336.02	184.98	Guard Column: Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)
6. Chloramphenicol*	1.95	200	321.16	151.99	257.04	Temp.: 40 °C

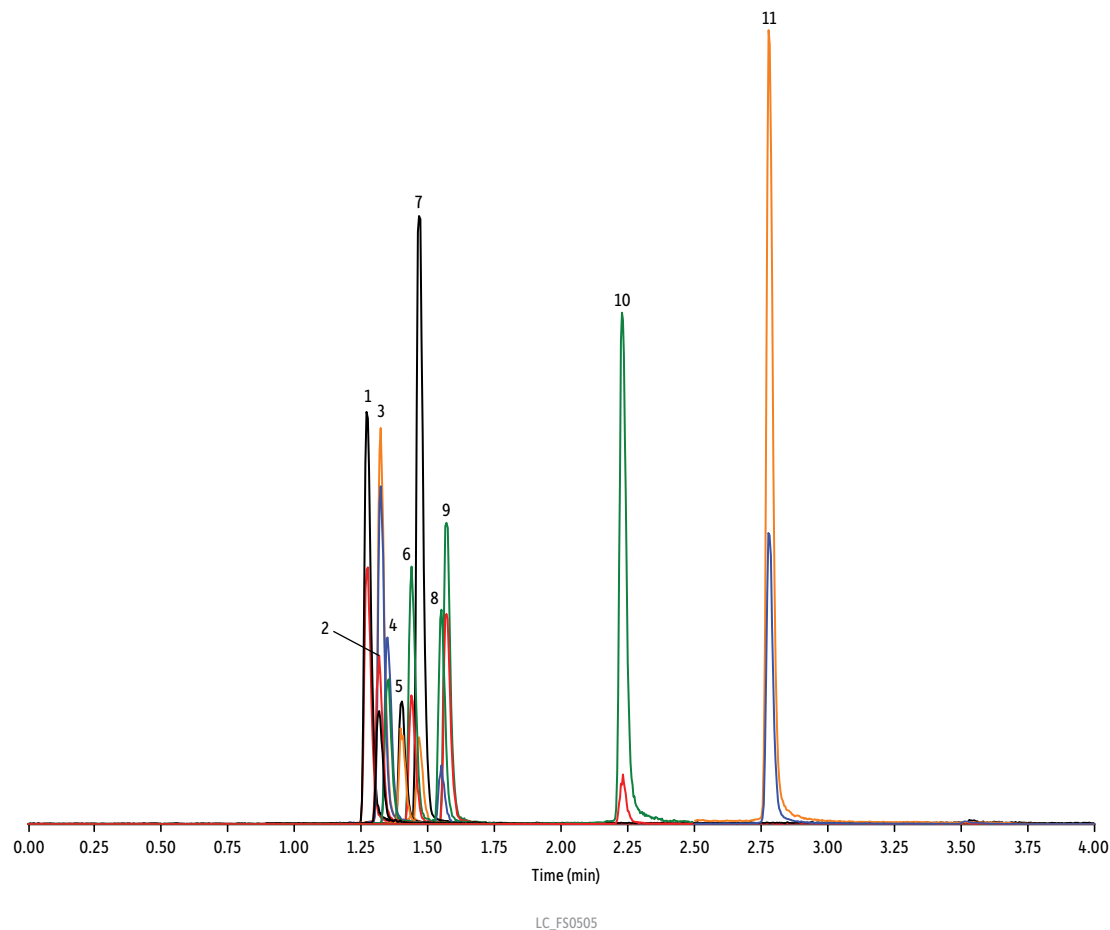
*Acquired in negative ion mode.

Sample	Water
Diluent:	25–200 ng/mL
Inj. Vol.:	2 µL
Mobile Phase	
A:	0.1% Formic acid in water
B:	0.1% Formic acid in acetonitrile

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	90	10
3.00	0.4	20	80
3.01	0.4	90	10
5.00	0.4	90	10

Detector	MS/MS
Ion Mode:	ESI+/ESI-
Mode:	MRM
Instrument	UHPLC
Notes	Tetracyclines and amphenicols were analyzed with ESI+ and ESI- mode, respectively.

Figure 3 : Antibiotiques Quinolone sur Raptor C18 par LC-MS/MS



Peaks	tr (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion
1. Marbofloxacin	1.28	10	363.20	72.11	320.10
2. Norfloxacin	1.32	20	320.23	276.20	233.13
3. Ofloxacin	1.32	10	362.21	318.20	261.15
4. Ciprofloxacin	1.35	20	332.18	288.22	245.15
5. Danofloxacin	1.40	20	358.22	340.16	314.21
6. Enrofloxacin	1.44	10	360.29	316.22	245.13
7. Orbifloxacin	1.47	10	396.22	352.17	226.12
8. Sarafloxacin	1.55	10	386.20	342.20	368.15
9. Difloxacin	1.57	10	400.23	356.17	299.13
10. Oxolinic acid	2.23	5	262.10	244.06	215.96
11. Flumequine	2.78	5	262.15	244.11	202.03

Column
Dimensions:
Particle Size:
Pore Size:
Guard Column:
Temp.:
Sample
Diluent:
Conc.:
Inj. Vol.:
Mobile Phase
A:
B:

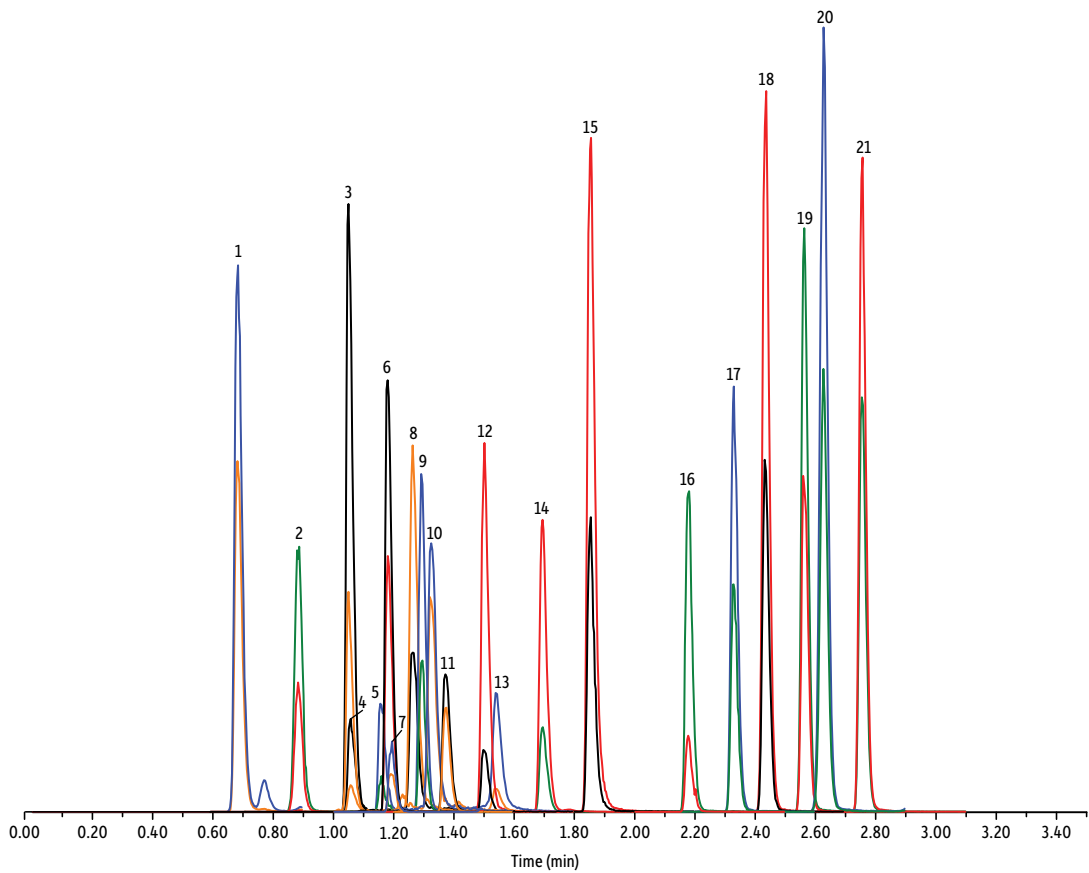
Raptor C18 (cat.# 9304A12)
100 mm x 2.1 mm ID
2.7 µm
90 Å
Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)
40 °C
Water
5–20 ng/mL
2 µL
0.1% Formic acid in water
0.1% Formic acid in acetonitrile

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	90	10
3.00	0.4	40	60
3.01	0.4	90	10
5.00	0.4	90	10

Detector
Ion Mode:
Mode:
Instrument

MS/MS
ESI+
MRM
UHPLC

Figure 4 : Antibiotiques Pénicilline, Céphalosporine et Tétracycline sur Raptor C18 par LC-MS/MS

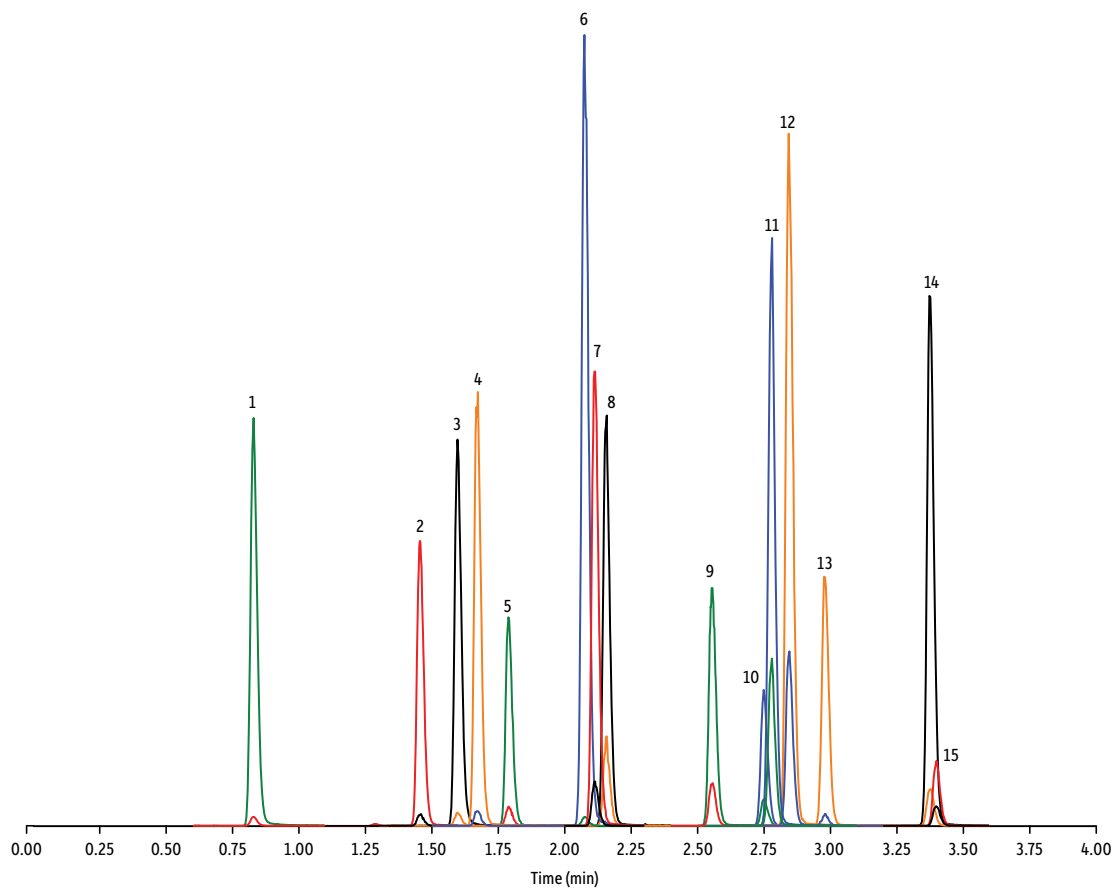


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Peaks	tr (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion
1. Desacetyl cephalirin	0.68	150	382.03	111.92	124.21
2. Amoxicillin	0.89	100	366.24	349.10	208.07
3. Cephalirin	1.05	50	424.17	292.08	124.14
4. Desfuroyl ceftiofur cysteine disulfide	1.06	300	549.16	183.02	126.00
5. Cefquinome	1.16	200	529.19	134.10	125.12
6. Ampicillin	1.18	50	350.19	106.07	160.06
7. Cefalexin	1.19	100	348.10	158.05	174.05
8. Oxytetracycline	1.26	50	461.27	426.15	443.32
9. Cefalonium	1.29	100	459.16	337.03	123.10
10. Tetracycline	1.32	50	445.28	154.07	427.32
11. Cefacetrile	1.37	300	362.07	258.08	178.01
12. Cefazolin	1.50	100	455.10	323.06	295.09
13. Chlortetracycline	1.54	50	479.27	154.07	371.06
14. Cefoperazone	1.69	100	646.26	143.07	148.02
15. Ceftiofur	1.85	50	524.14	241.08	125.24
16. Penicillin G	2.18	100	335.18	176.07	160.07
17. Penicillin V	2.33	100	351.10	160.06	114.07
18. Oxacillin	2.44	100	402.15	160.05	114.06
19. Cloxacillin	2.56	100	436.15	277.06	160.05
20. Nafcillin	2.63	25	415.19	199.09	171.06
21. Dicloxacillin	2.76	100	470.11	160.05	311.02

Column	Raptor C18 (cat.# 9304A12)		
Dimensions:	100 mm x 2.1 mm ID		
Particle Size:	2.7 µm		
Pore Size:	90 Å		
Guard Column:	Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)		
Temp.:	40 °C		
Sample			
Diluent:	Water		
Conc.:	25–300 ng/mL		
Inj. Vol.:	2 µL		
Mobile Phase			
A:	0.1% Formic acid in water		
B:	0.1% Formic acid in acetonitrile		
Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	90	10
3.00	0.4	20	80
3.01	0.4	90	10
5.00	0.4	90	10
Detector	MS/MS		
Ion Mode:	ESI+		
Mode:	MRM		
Instrument	UHPLC		

Figure 5 : Antibiotiques Sulfonamide sur Raptor C18 par LC-MS/MS



LC_FS0501

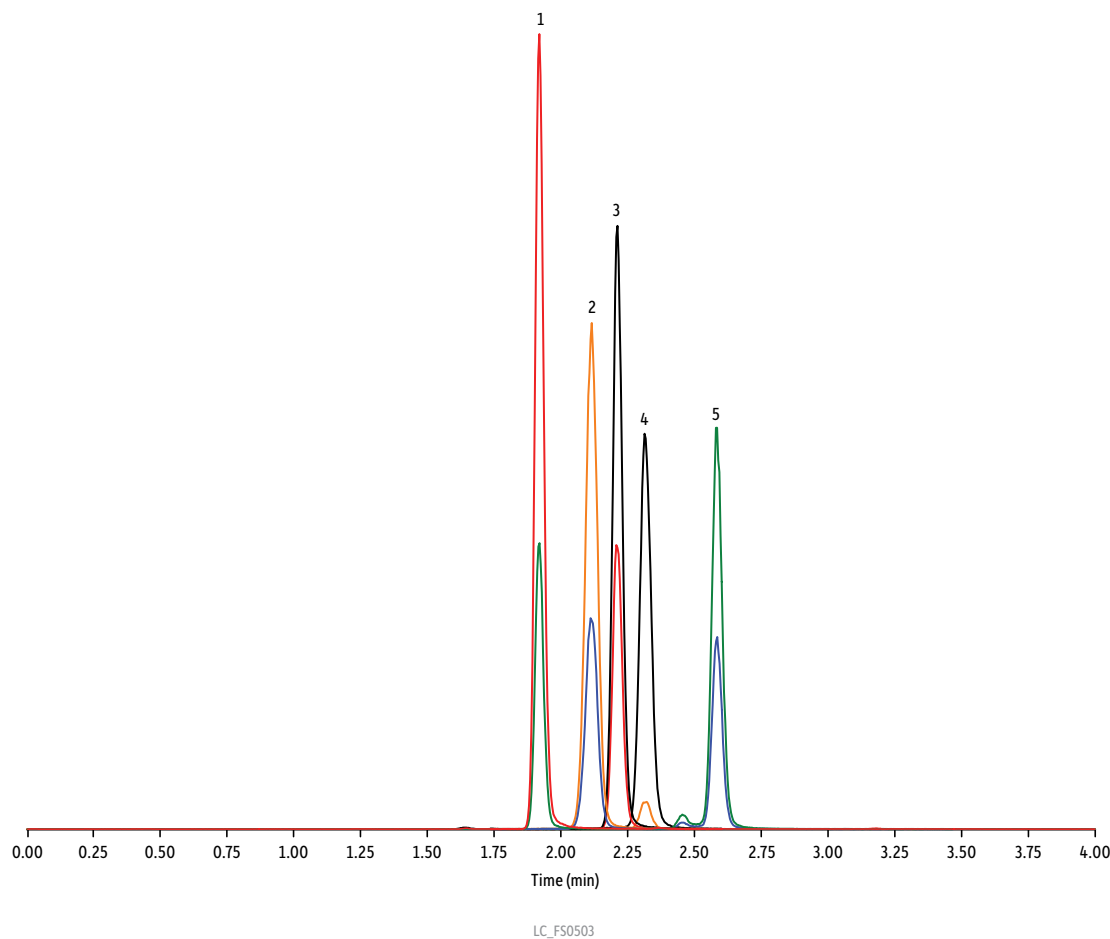
Peaks	tr (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion
1. Sulfanilamide	0.83	200	172.98	93.07	75.23
2. Sulfadiazine	1.45	20	251.18	156.04	92.08
3. Sulfathiazole	1.60	10	256.16	156.03	92.08
4. Sulfapyridine	1.67	10	250.13	156.10	92.08
5. Sulfamerazine	1.79	20	265.08	156.03	92.08
6. Sulfamethazine	2.07	10	279.23	186.08	124.08
7. Sulfamethizole	2.11	10	271.17	156.02	108.02
8. Sulfamethoxypyridazine	2.16	10	281.14	156.03	126.07
9. Sulfachlorpyridazine	2.55	20	285.05	156.03	108.09
10. Sulfadoxine	2.75	10	311.17	156.03	108.09
11. Sulfamethoxazole	2.78	20	254.18	155.98	147.06
12. Sulfathoxypyridazine	2.84	20	295.17	267.07	156.03
13. Sulfisoxazole	2.98	20	268.14	156.03	113.10
14. Sulfadimethoxine	3.37	10	311.17	156.09	108.09
15. Sulfaquinoxaline	3.40	20	301.18	156.04	108.02

Column Raptor C18 (cat.# 9304A12)
Dimensions: 100 mm x 2.1 mm ID
Particle Size: 2.7 µm
Pore Size: 90 Å
Guard Column: Raptor C18 EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9304A0252)
Temp.: 40 °C
Sample
Diluent: Water
Conc.: 10–200 ng/mL
Inj. Vol.: 2 µL
Mobile Phase
A: 0.1% Formic acid in water
B: 0.1% Formic acid in acetonitrile

Time (min)	Flow (mL/min)	%A	%B
0.00	0.4	90	10
3.50	0.4	60	40
3.51	0.4	90	10
5.00	0.4	90	10

Detector MS/MS
Ion Mode: ESI+
Mode: MRM
Instrument UHPLC

Figure 6 : Antibiotiques Ionophores sur Raptor Biphenyl par LC-MS/MS



Peaks	tg (min)	Conc. (ng/mL)	Precursor Ion	Product Ion	Product Ion	Column	
1. Lasalocid A	1.92	100	613.42	377.28	595.40	Dimensions:	Raptor Biphenyl (cat.# 9309A12)
2. Monensin	2.12	100	693.50	675.44	461.30	Particle Size:	100 mm x 2.1 mm ID
3. Salinomycin	2.19	100	773.57	431.24	531.39	Pore Size:	2.7 µm
4. Maduramicin	2.30	100	939.65	877.58	473.31	Guard Column:	90 Å
5. Narasin	2.58	100	787.59	431.27	531.35		Raptor Biphenyl EXP guard column cartridge 5 mm, 2.1 mm ID, 2.7 µm (cat.# 9309A0252)
						Temp.:	40 °C
						Sample	
						Diluent:	Water:methanol (10:90)
						Conc.:	100 ng/mL
						Inj. Vol.:	2 µL
						Mobile Phase	
						A:	0.5% Formic acid in water
						B:	0.5% Formic acid in acetonitrile
						Time (min)	Flow (mL/min)
						0.00	0.5
						3.00	0.5
						3.01	0.5
						5.00	0.5
						%A	%B
						35	65
						25	75
						35	65
						35	65
						Detector	MS/MS
						Ion Mode:	ESI+
						Mode:	MRM
						Instrument	UHPLC