SUPPLEMENTARY MATERIALS

Development and Validation of a LC-MS/MS Method for Determination of Multi-class Antibiotic Residues in Aquaculture and River Waters, and Photocatalytic Degradation of Antibiotics by TiO_2 Nanomaterials.

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Contents of supplementary materials

Figure S1-S10: Optimization of MS-MS parameters for antibiotic analysis.

S1. The result autotune of optimum MS-MS parameters of amoxicillin (AMOX), ampicillin (AMPI), lincomycin (LCM), trimethoprim (TMTP).

Results

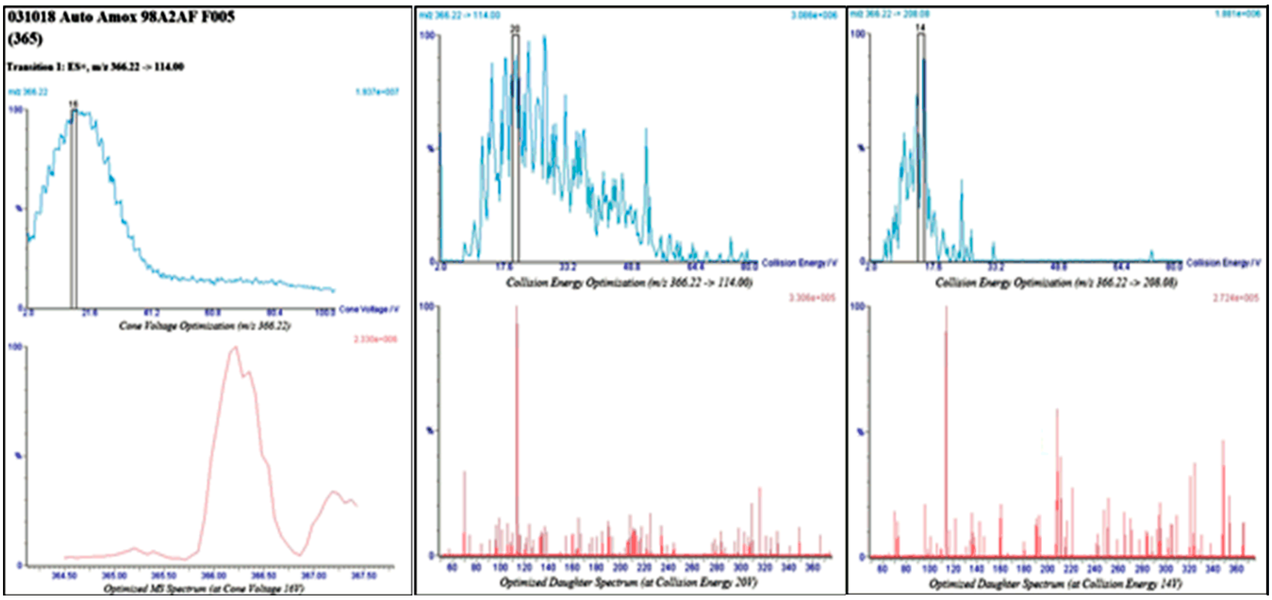
IntelliStart generated the following experiments:

MRM Experiment	E:\MASSLYNX PROJECT\QUOC DUY.PRO\AUTOTUNE\Van-Co-Ate\031018 Auto Function 2 Amox-Ampi-Linco-Trime 98A2AF F005 L2.exp

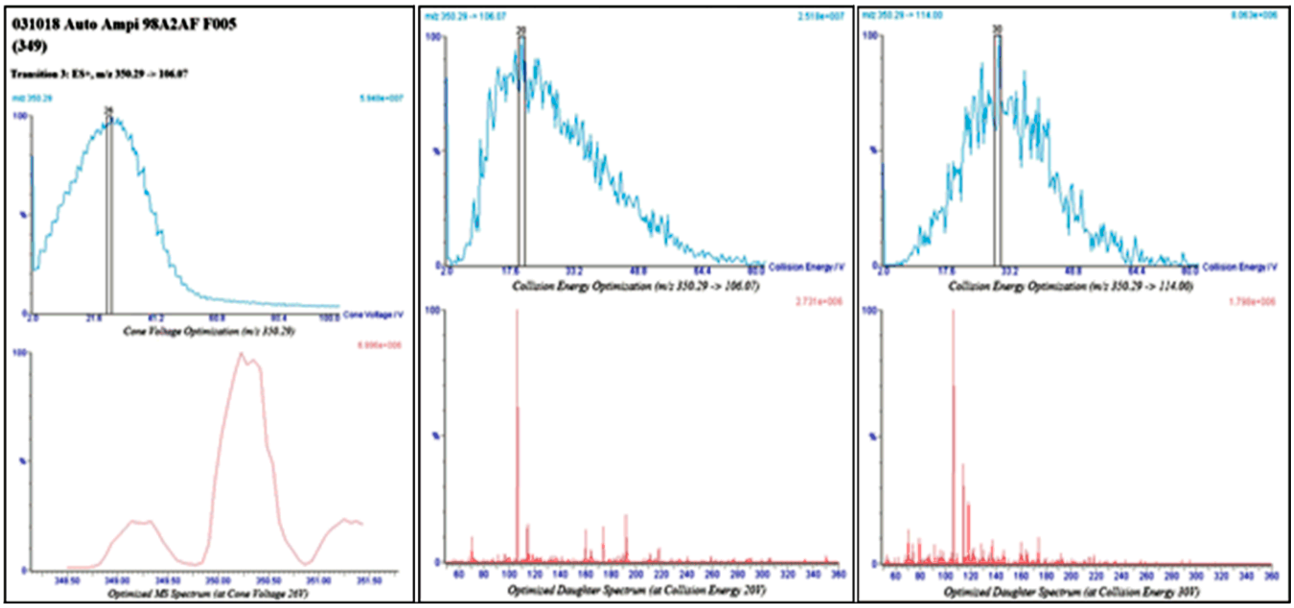
IntelliStart found the following compounds:

Compound	Formula/Mass		Parent m/z	Cone Voltage	Daughters	Collision Energy	Ion Mode
031018 Auto Amox 98A2AF F005	365	1 2	366.22 366.22	16 16	114.00 208.08	20 14	ES+ ES+
031018 Auto Ampi 98A2AF F005	349	1 2	350.29 350.29	26 26	106.07 114.00	20 30	ES+ ES+
031018 Auto Linco 407 98A2AF F005	406	1 2	407.35 407.35	42 42	126.16 359.20	28 18	ES+ ES+
031018 Auto Trime 98A2AF F005	290	1 2	291.29 291.29	48 48	230.13 123.05	22 24	ES+ ES+

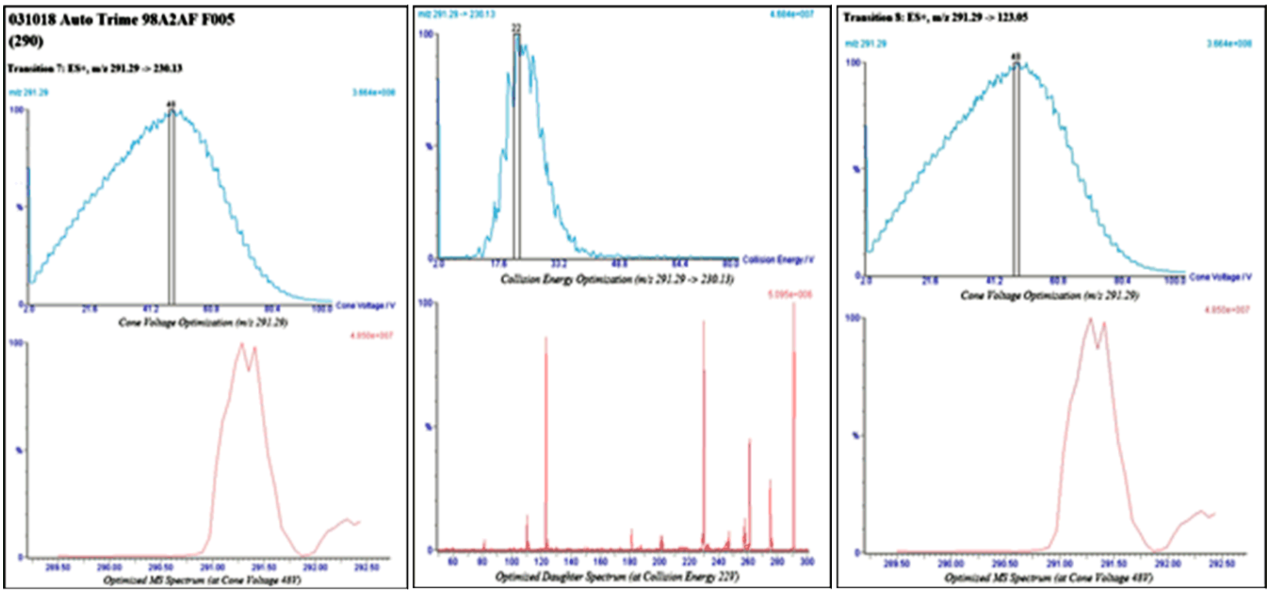
S2. Optimum MS-MS parameters of ampicilline (AMPI).



S3. Optimum MS-MS parameters of ampicilline (AMPI).



S4. Optimum MS-MS parameters of trimethoprim (TMTP).



S5. The result autotune of optimum MS-MS parameters of sulfamethazine (SMZ), sulfamethoxazole (SMZX).

Results

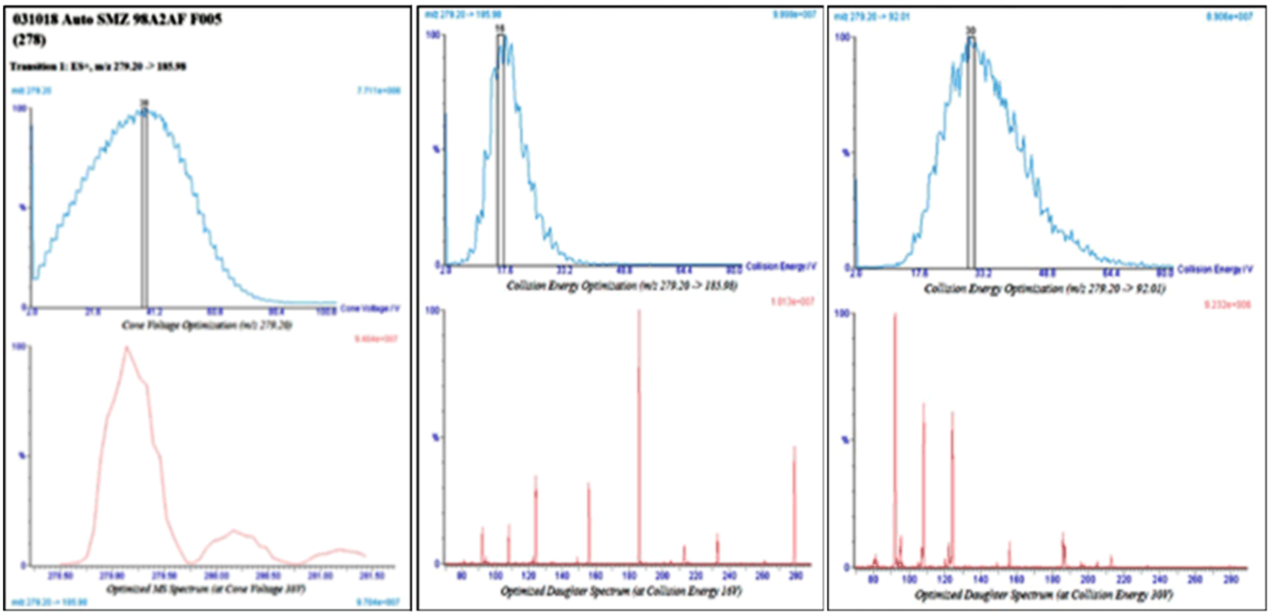
IntelliStart generated the following experiments:

MRM Experiment	E:\MASSLYNX PROJECT\QUOC DUY.PRO\AUTOTUNE\SMZ-SMZX\031018 SMZ-SMZX 98A2AF F005.exp

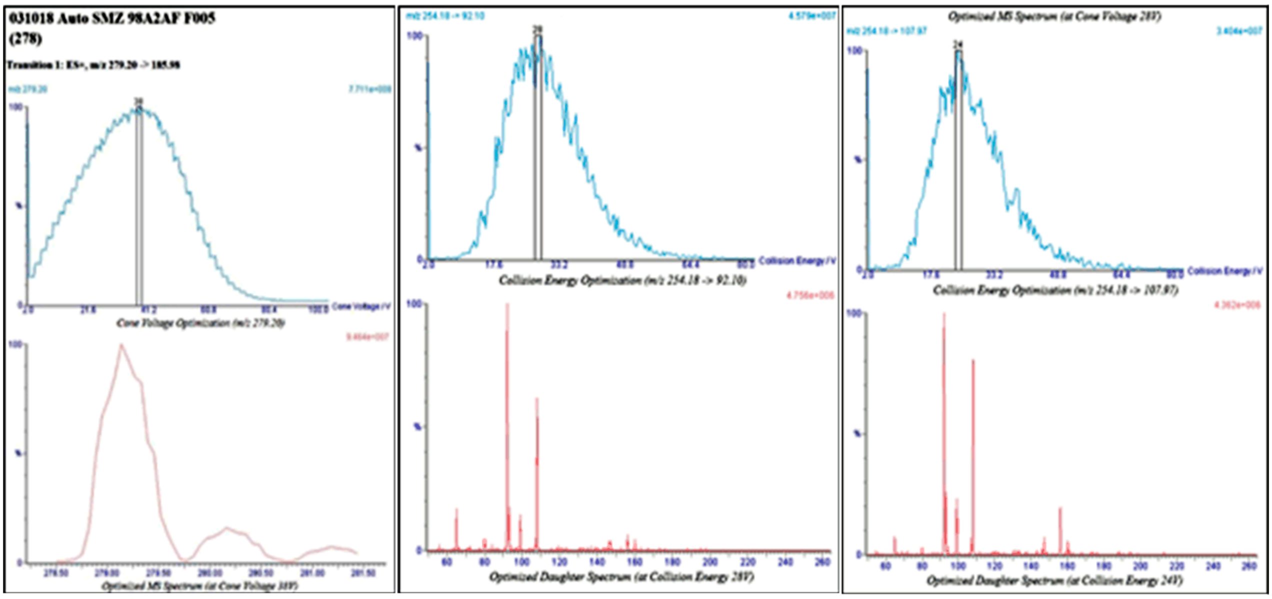
IntelliStart found the following compounds:

Compound	Formula/Mass		Parent m/z	Cone Voltage	Daughters	Collision Energy	Ion Mode
031018 Auto SMZ 98A2AF F005	278	1 2	279.22 279.22	36 36	186.06 92.04	16 30	ES+ ES+
031018 Auto SMZX 98A2AF F005	253.4	1 2	254.18 254.18	30 30	92.10 107.97	28 22	ES+ ES+

S6. Optimum MS-MS parameters of sulfamethazine (SMZ).



S7. Optimum MS-MS parameters of Sulfamethoxazole (SMZX).



S8. The result autotune of optimum MS-MS parameters of vancomycin (VCM), atenolol-IS (ATN).

Results

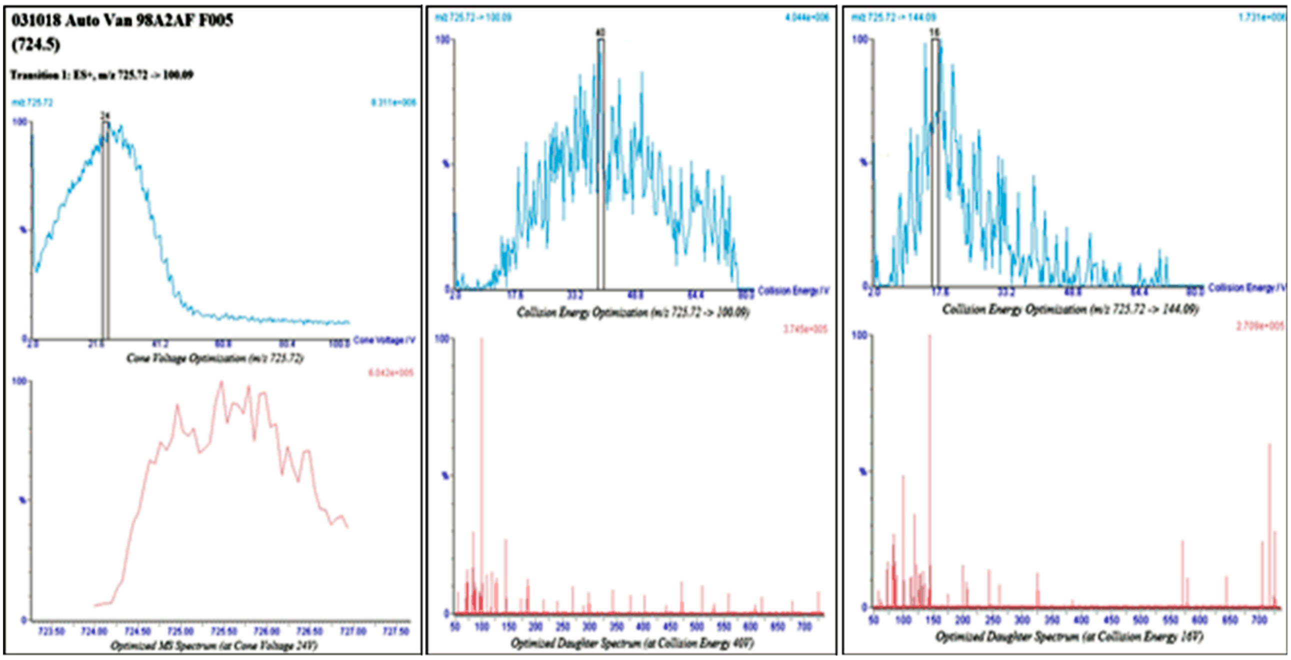
IntelliStart generated the following experiments:

MRM Experiment	E:\MASSLYNX PROJECT\QUOC DUY.PRO\AUTOTUNE\Van-Ate\031018 Auto Function 1 Van-Ate 98A2AF F005.exp

IntelliStart found the following compounds:

Compound	Formula/Mass		Parent m/z	Cone Voltage	Daughters	Collision Energy	Ion Mode
031018 Auto Van 98A2AF F005	724.5	1 2	725.72 725.72	24 24	100.09 144.09	40 16	ES+ ES+
031018 Auto Ate 98A2AF F005	266	1 2	267.29 267.29	38 38	71.62 56.01	22 32	ES+ ES+

S9. Optimum MS-MS parameters of Vancomycin (VCM).



S10. Optimum MS-MS parameters of Atenolol (ATN).

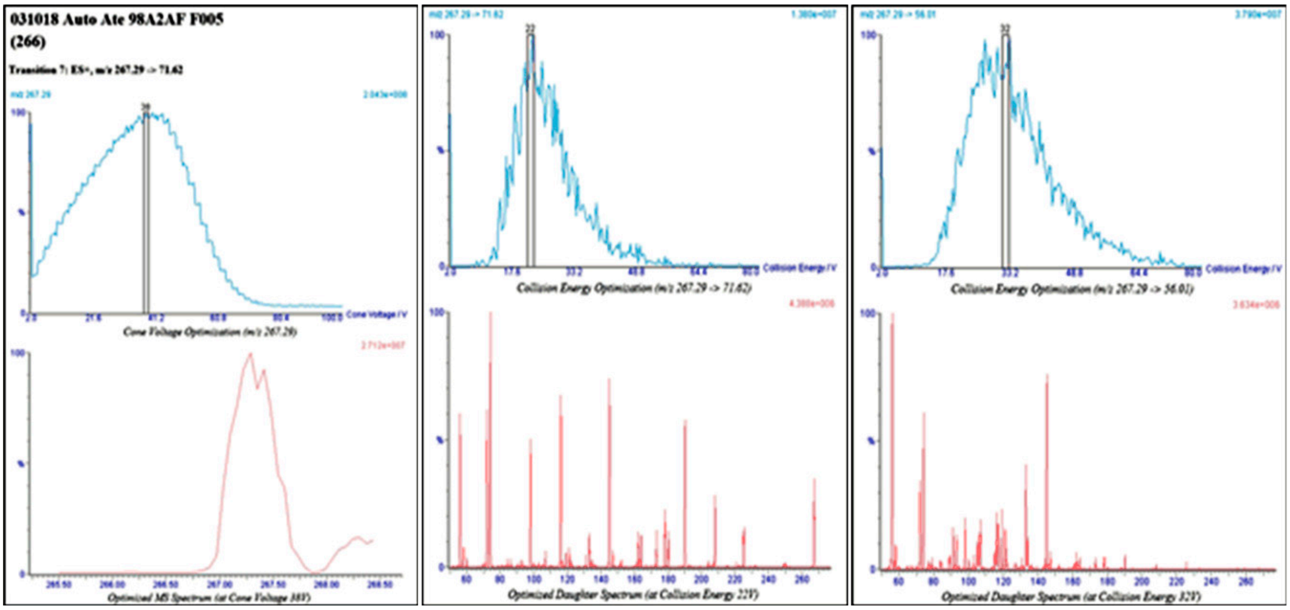
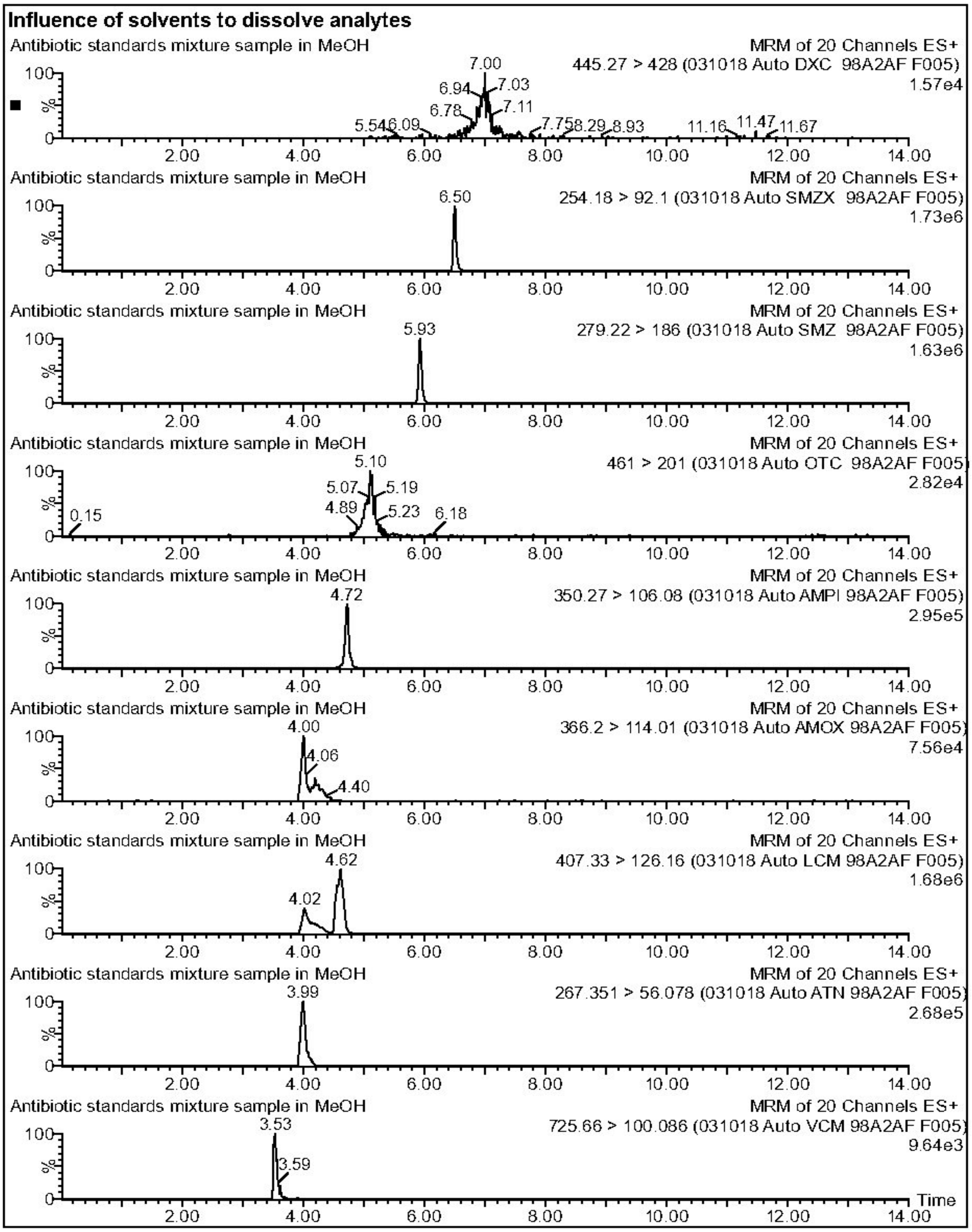
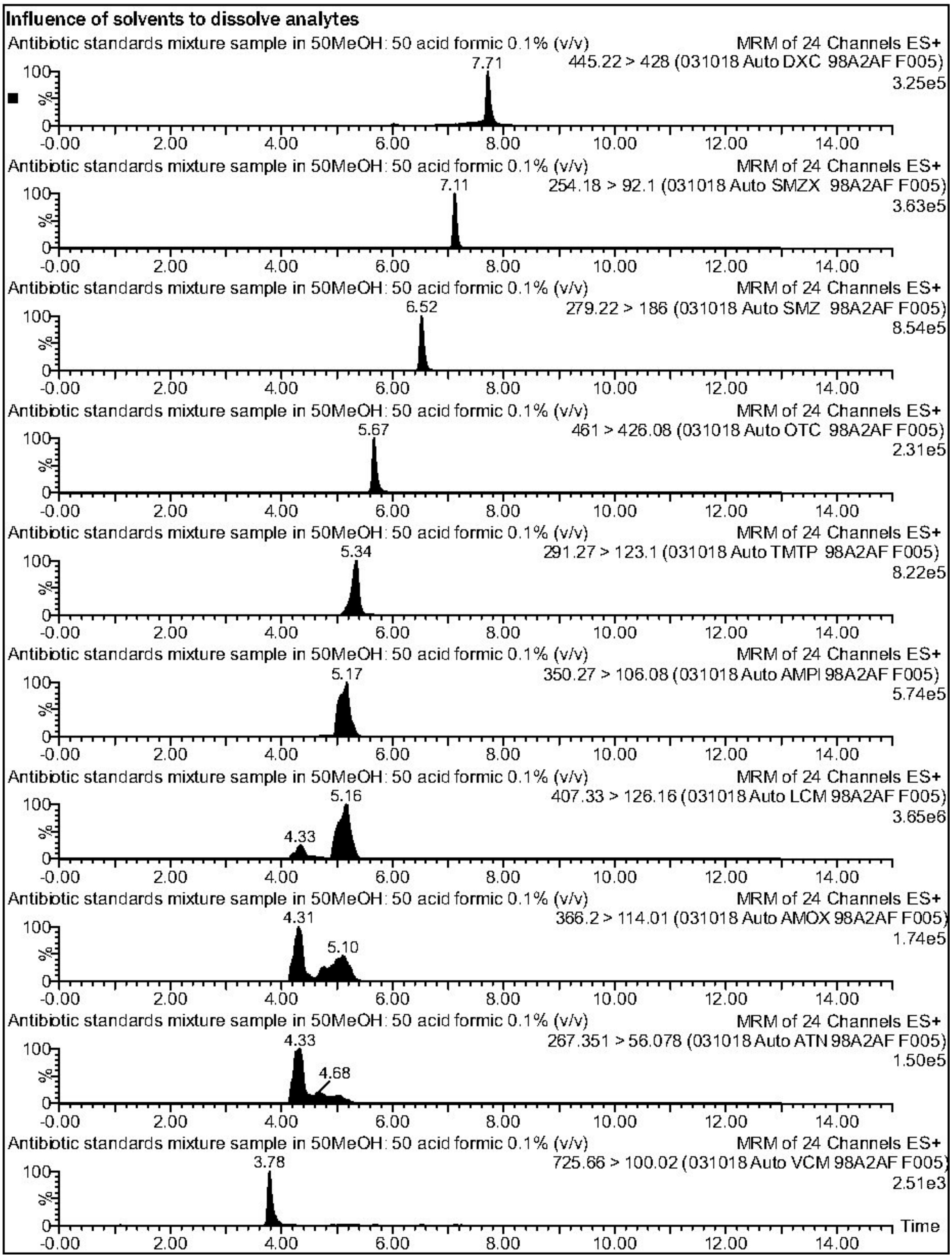


Figure S11-S14: Chromatograms indicated the effects of solvents on dissolve analytes.

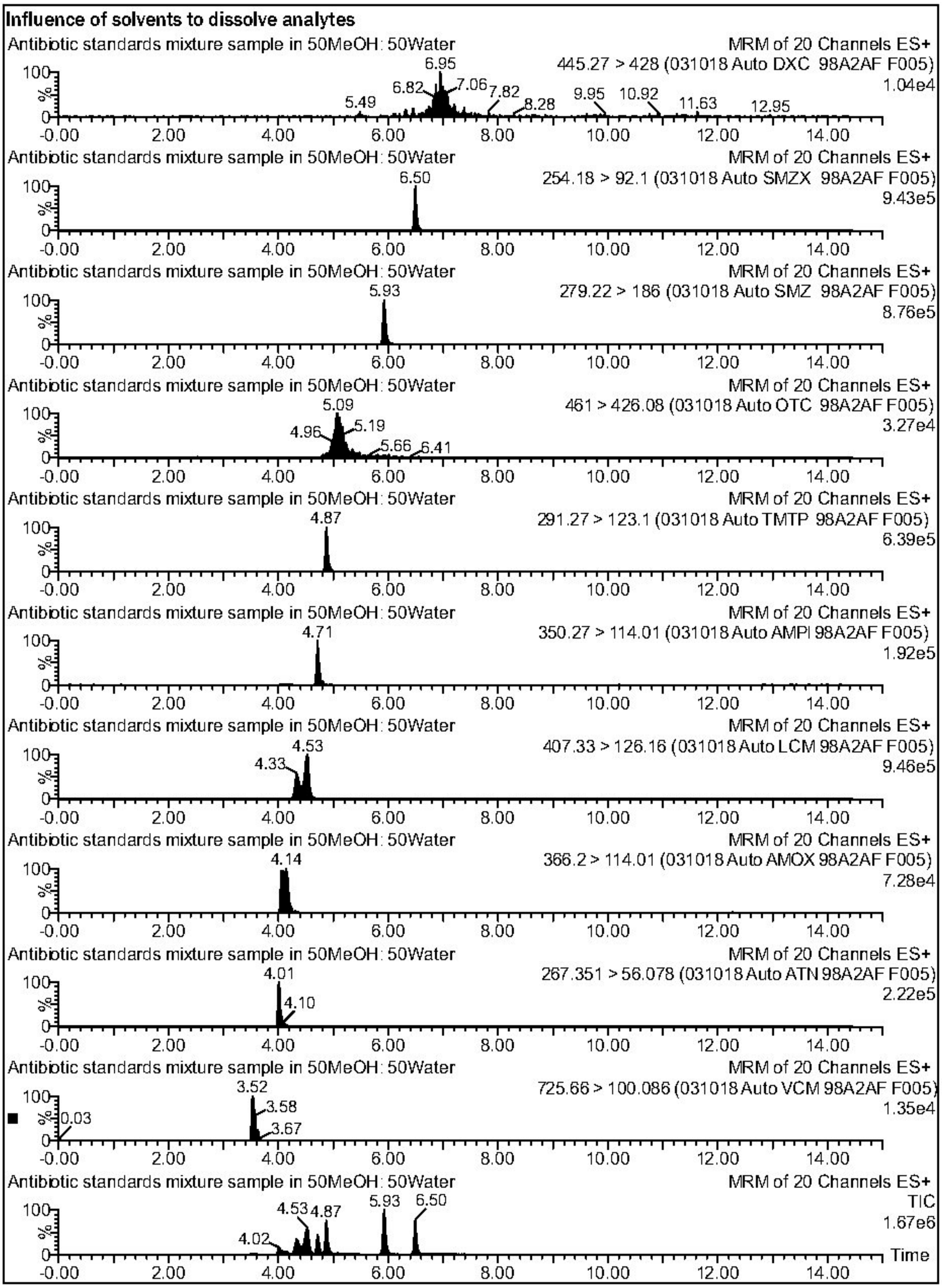
S11. Standard antibiotic mixture in MeOH.



S12. Standard antibiotic mixture in 50 MeOH: 50 formic acid 0.1% in water (v/v).



S13. Standard antibiotic mixture in 50 MeOH: 50 water.



S14. Standards antibiotic mixture in water- formic acid (999:1).

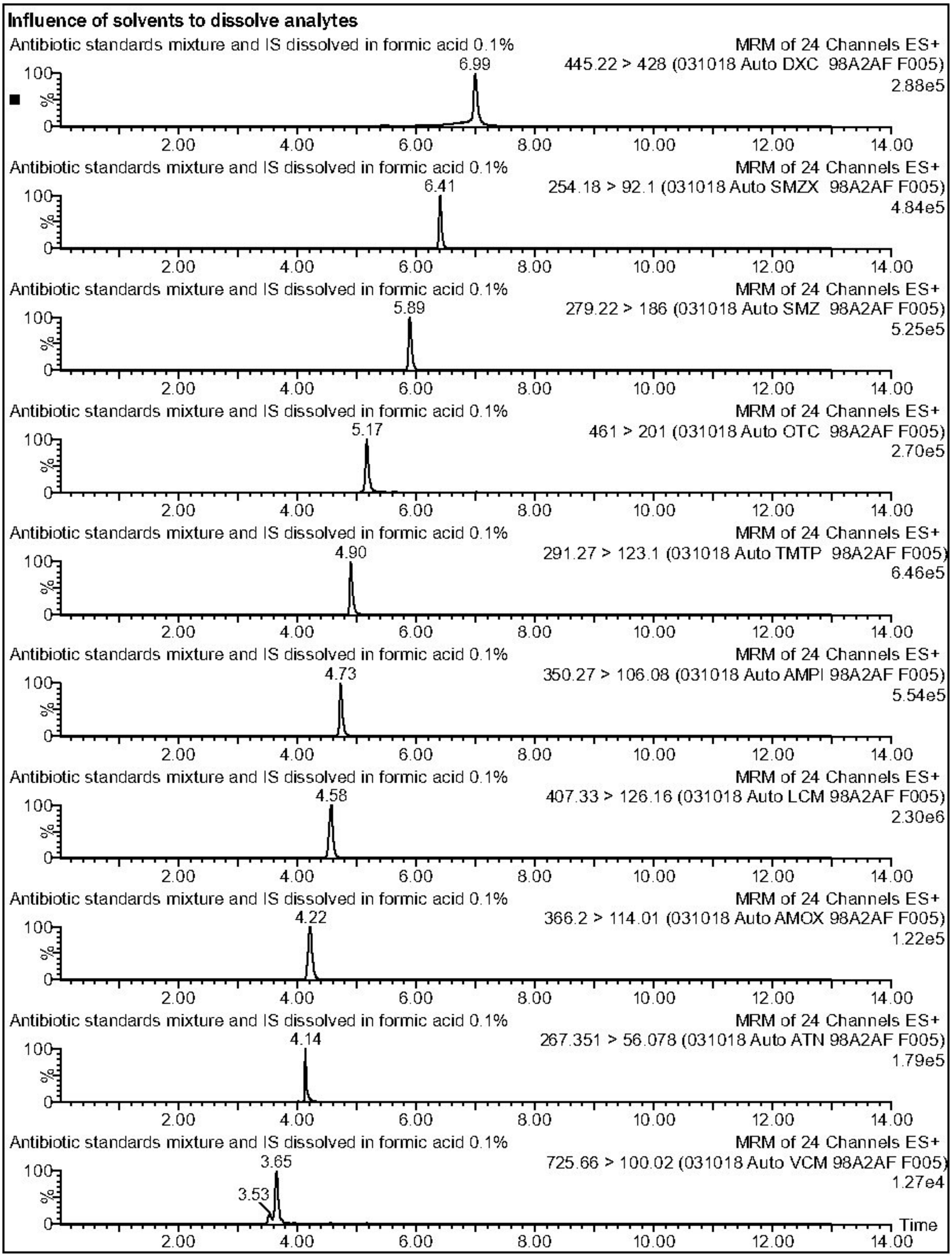
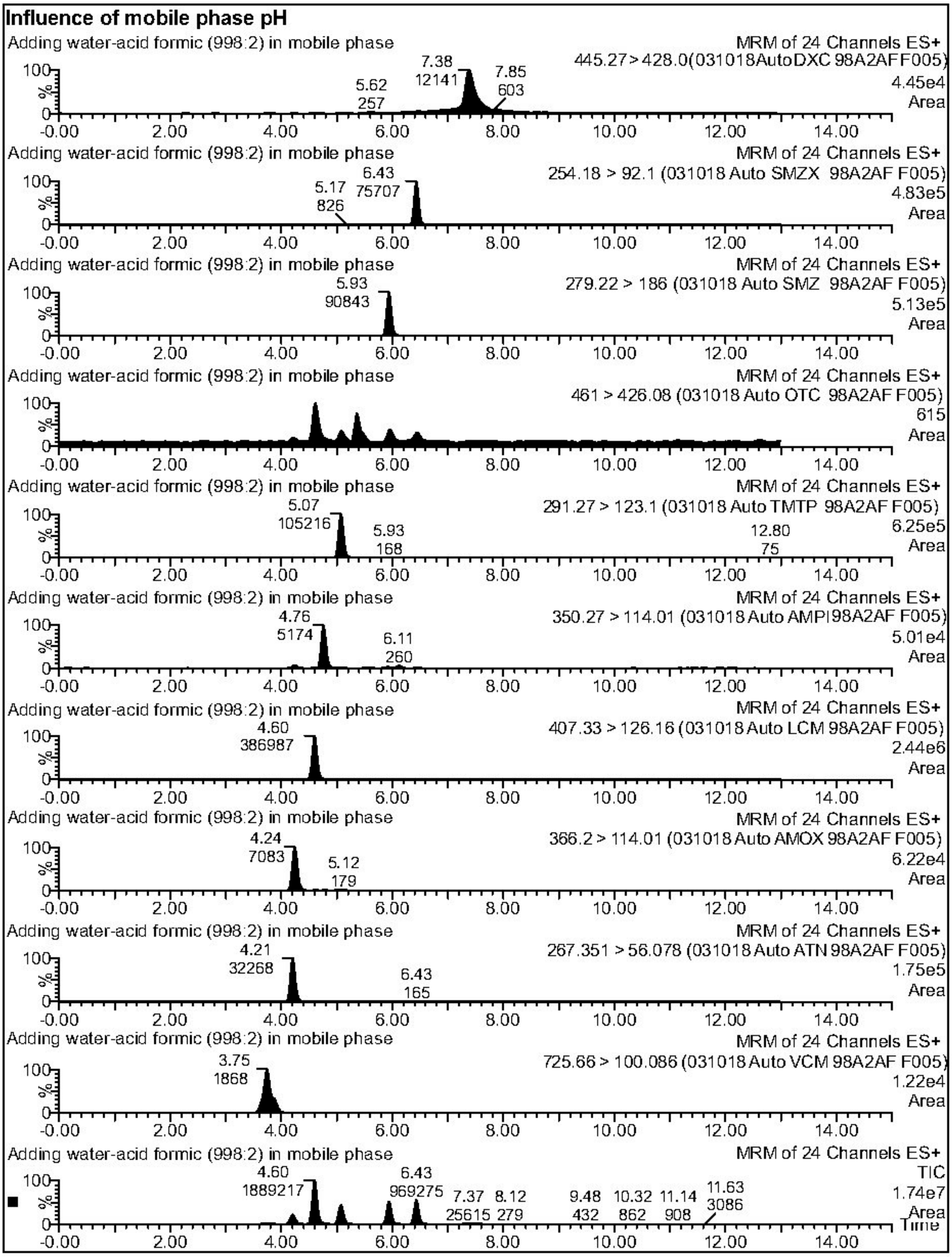
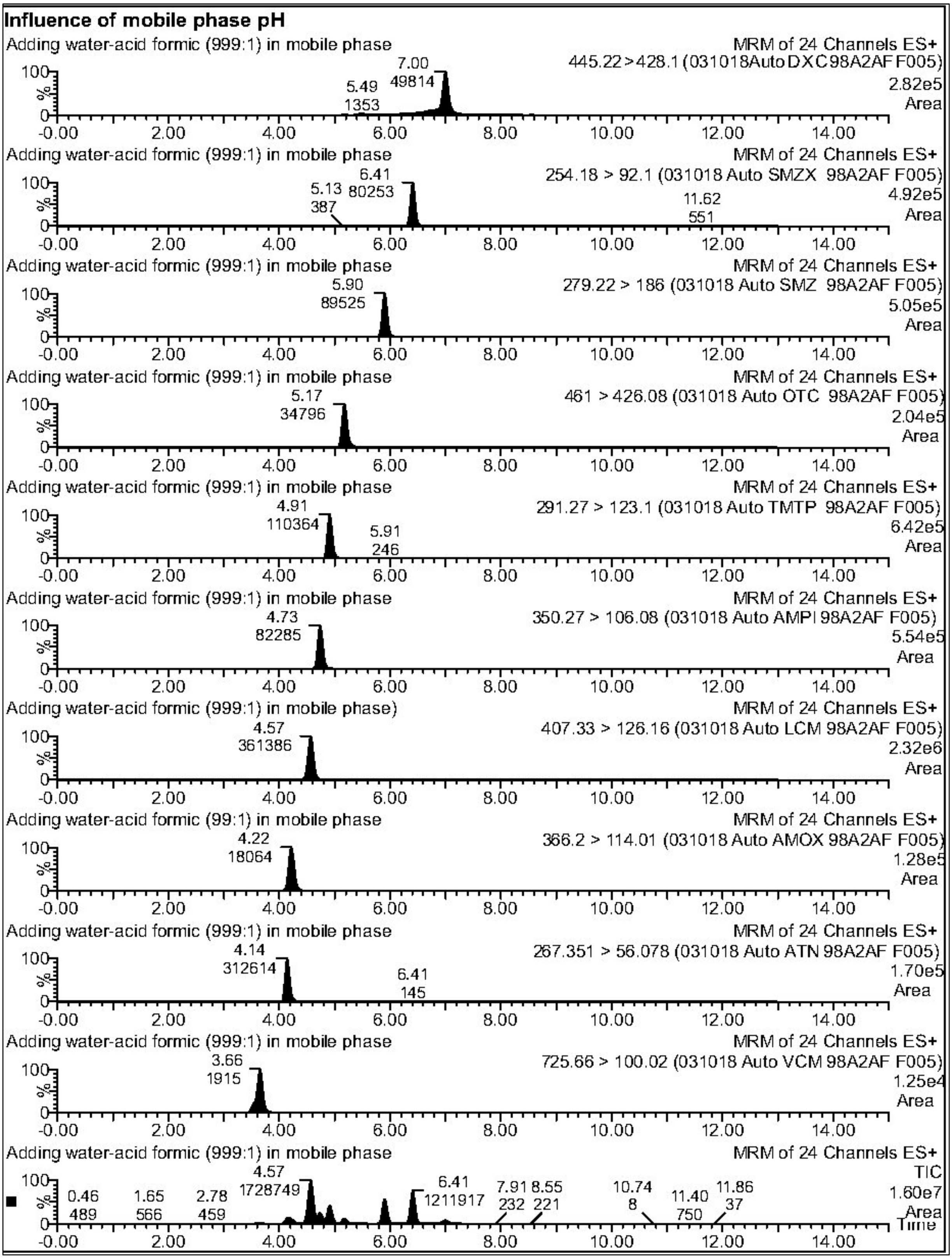


Figure S15-S16: Chromatograms indicated influence of mobile phase pH.

S15. Adding water-acid formic (998:2) in mobile phase.

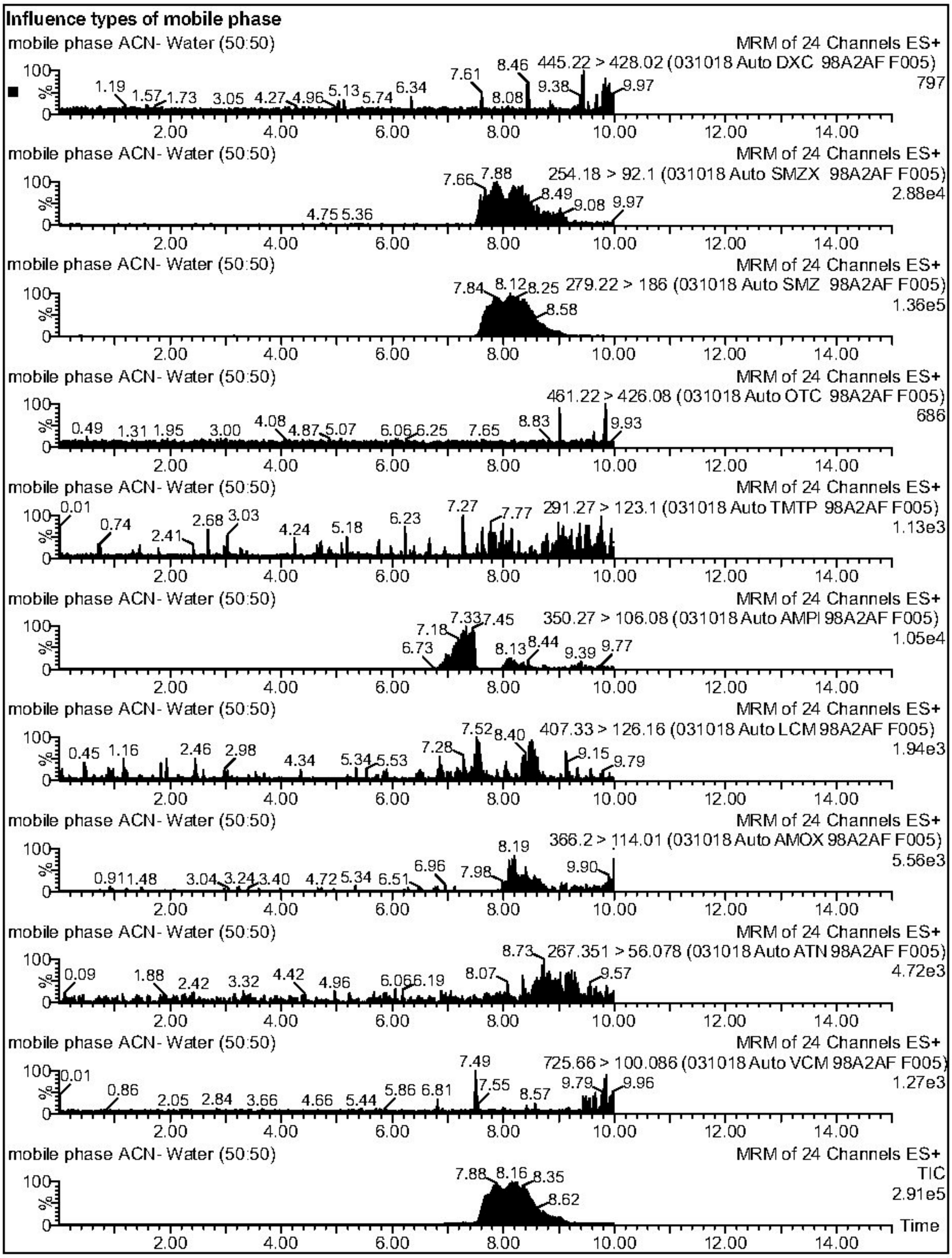


S16. Adding water-acid formic (999:1) in mobile phase.

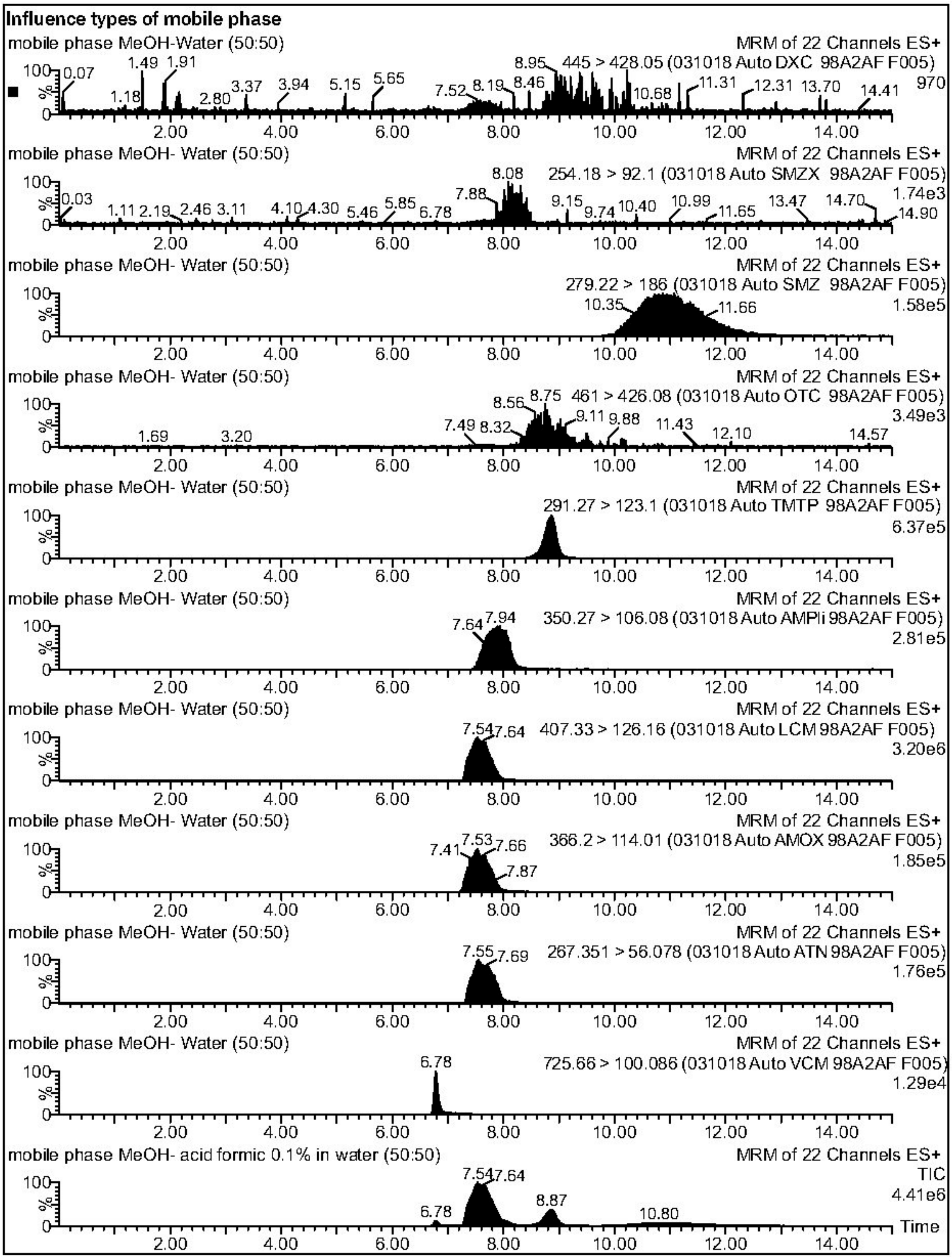


Figures S17-S19: Chromatograms indicated influence of mobile phase types

S17. Mobile phase of ACN-water (50:50).



S18. Mobile phase of MeOH-water (50:50).



S19. Mobile phase of MeOH-acid formic 0.1% (50:50).

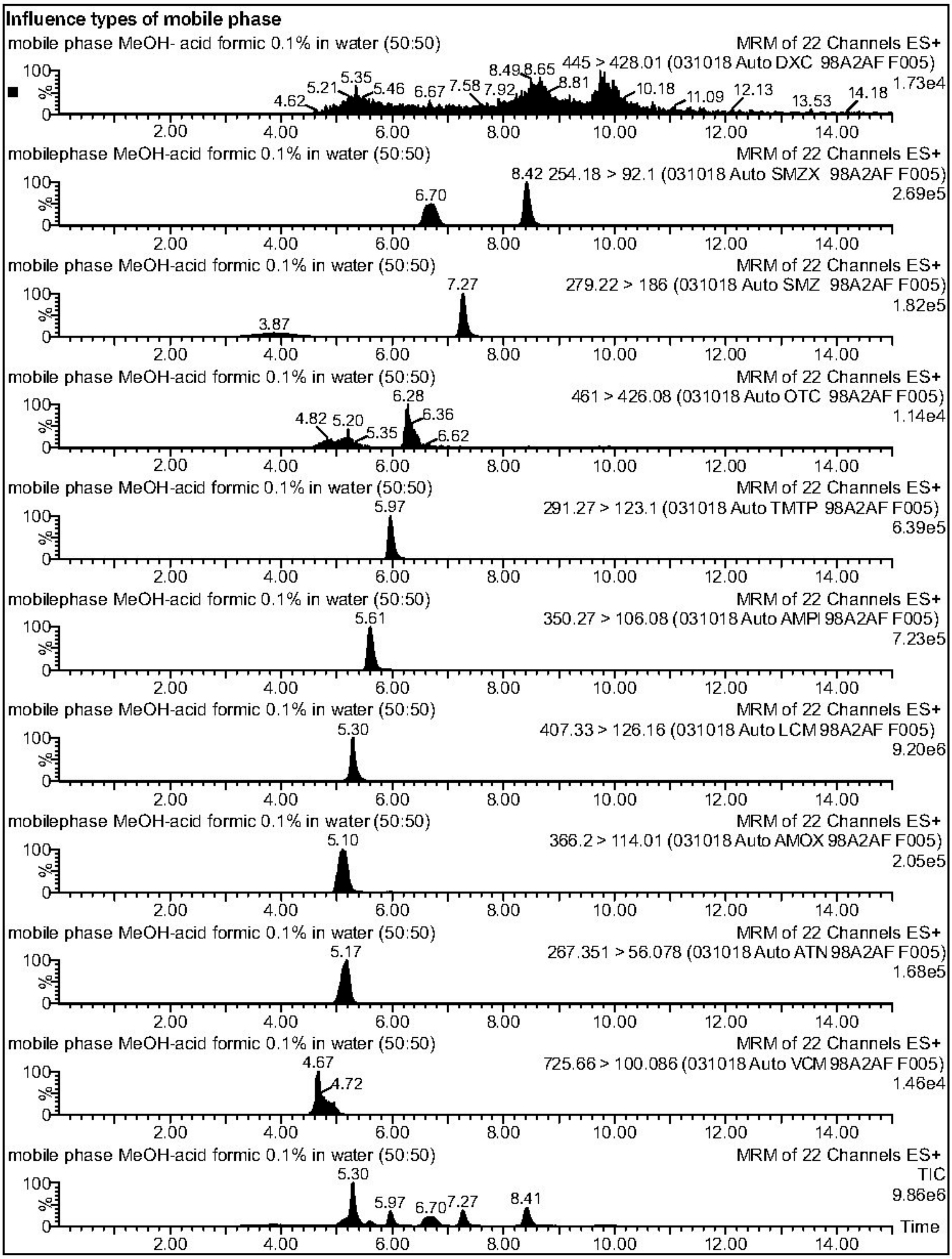
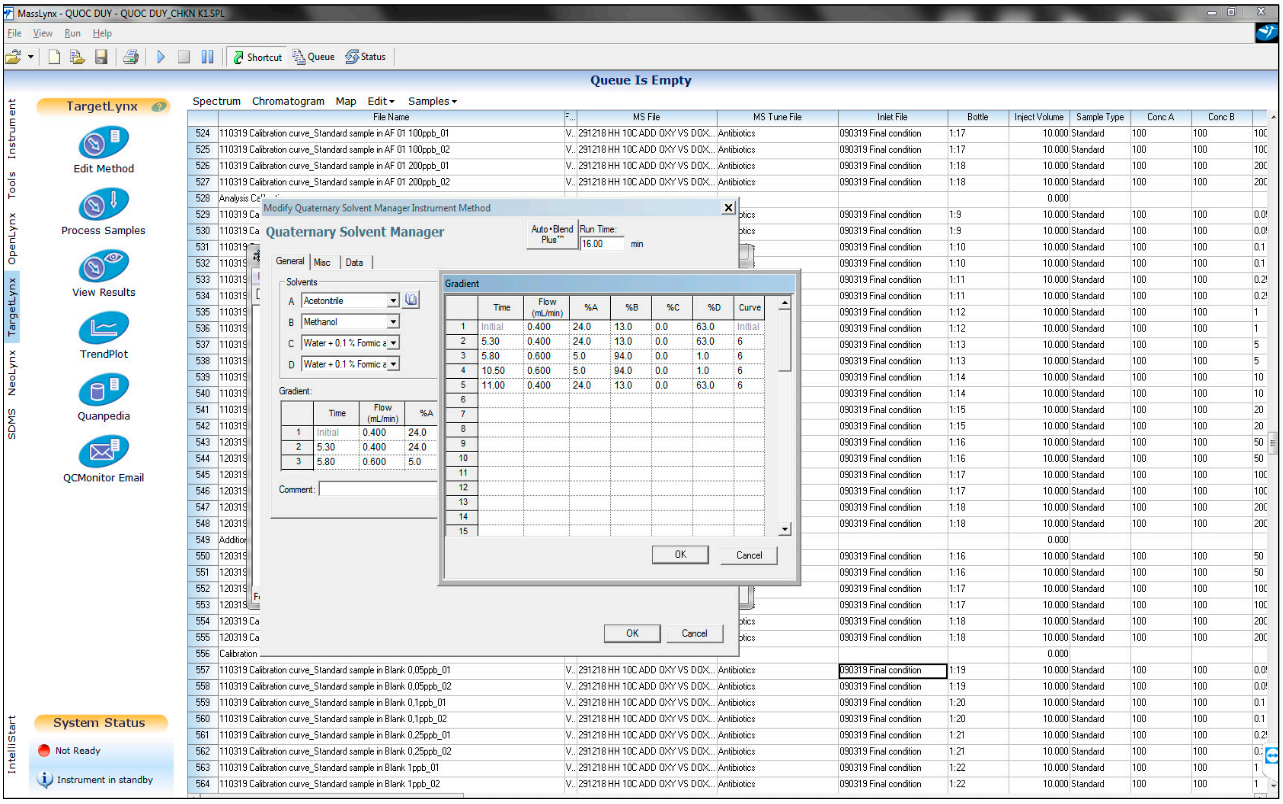
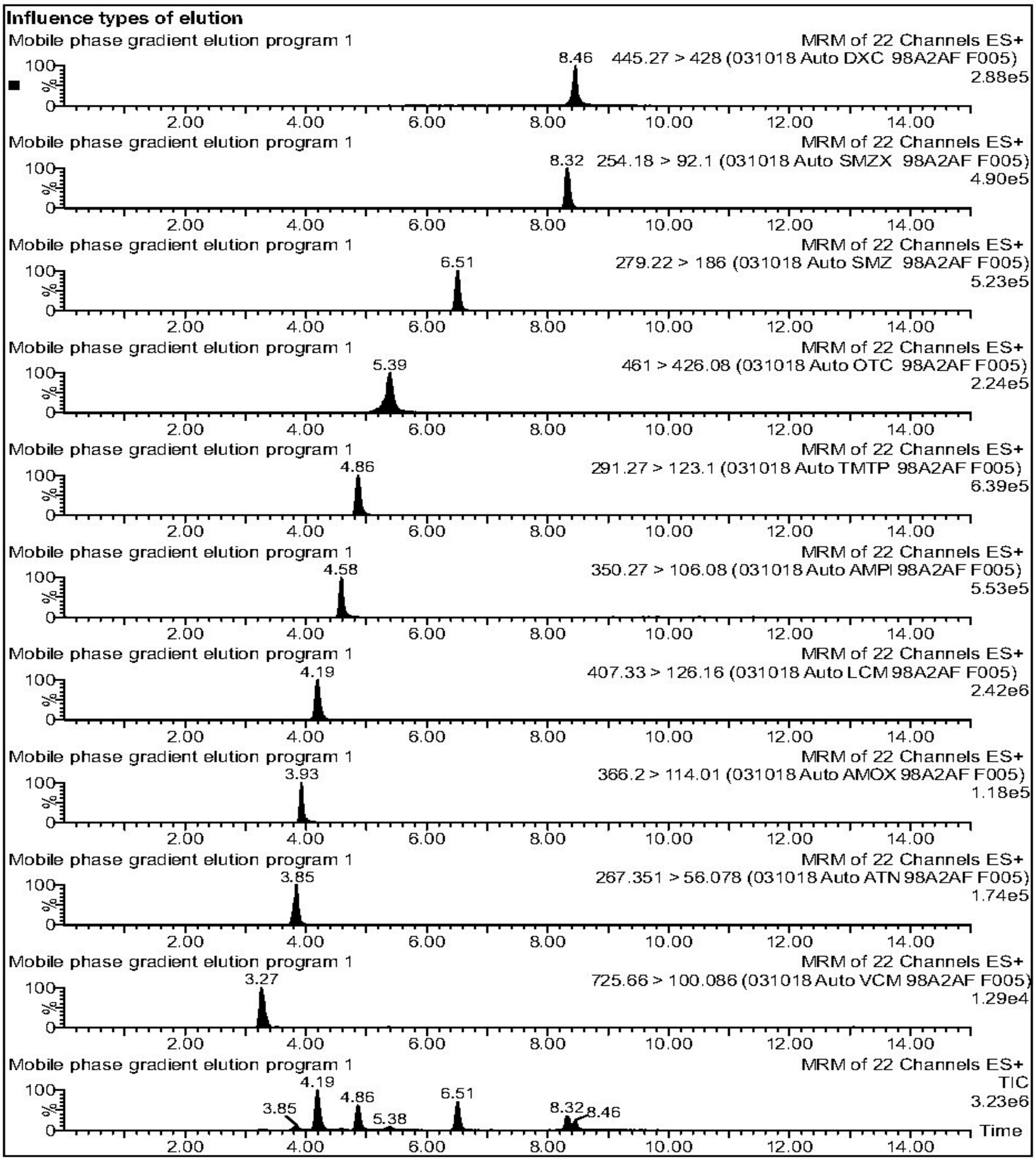


Figure S20-S21: Chromatograms indicated influence of elution program

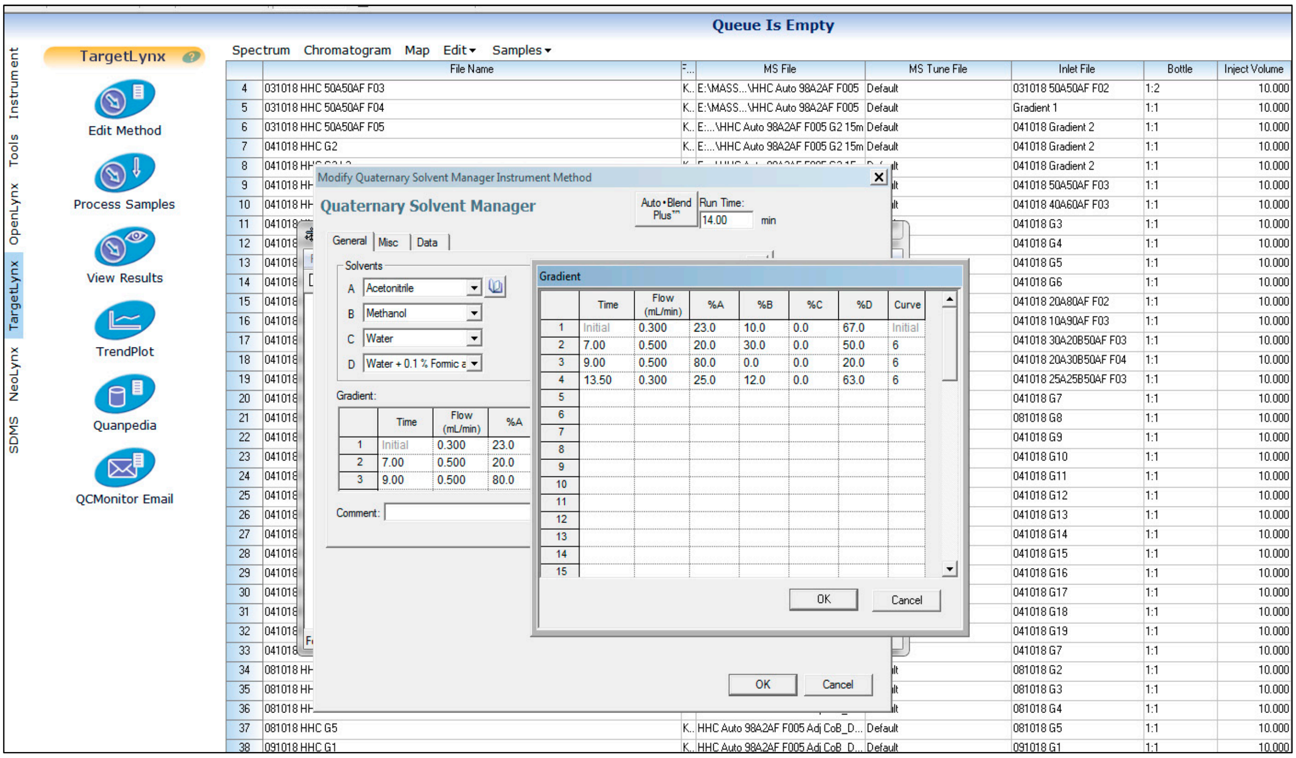
S20A. Gradient program 1 of mobile phase.



S20B. LC-MS/MS chromatogram at gradient program 1 of mobile phase.



S21A. Gradient program 2 of mobile phase.



S21B. LC-MS/MS chromatogram at gradient program 2 of mobile phase.

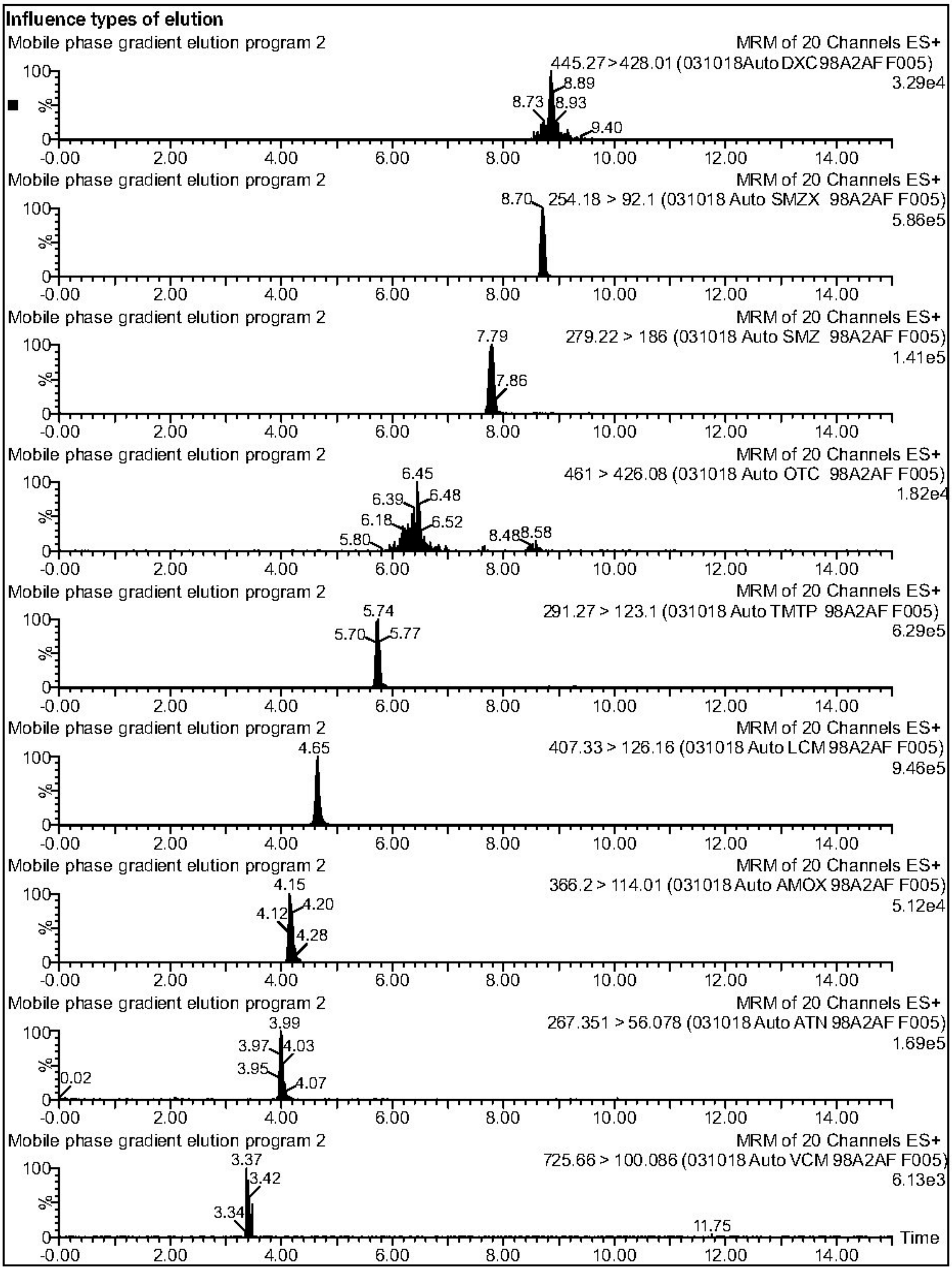
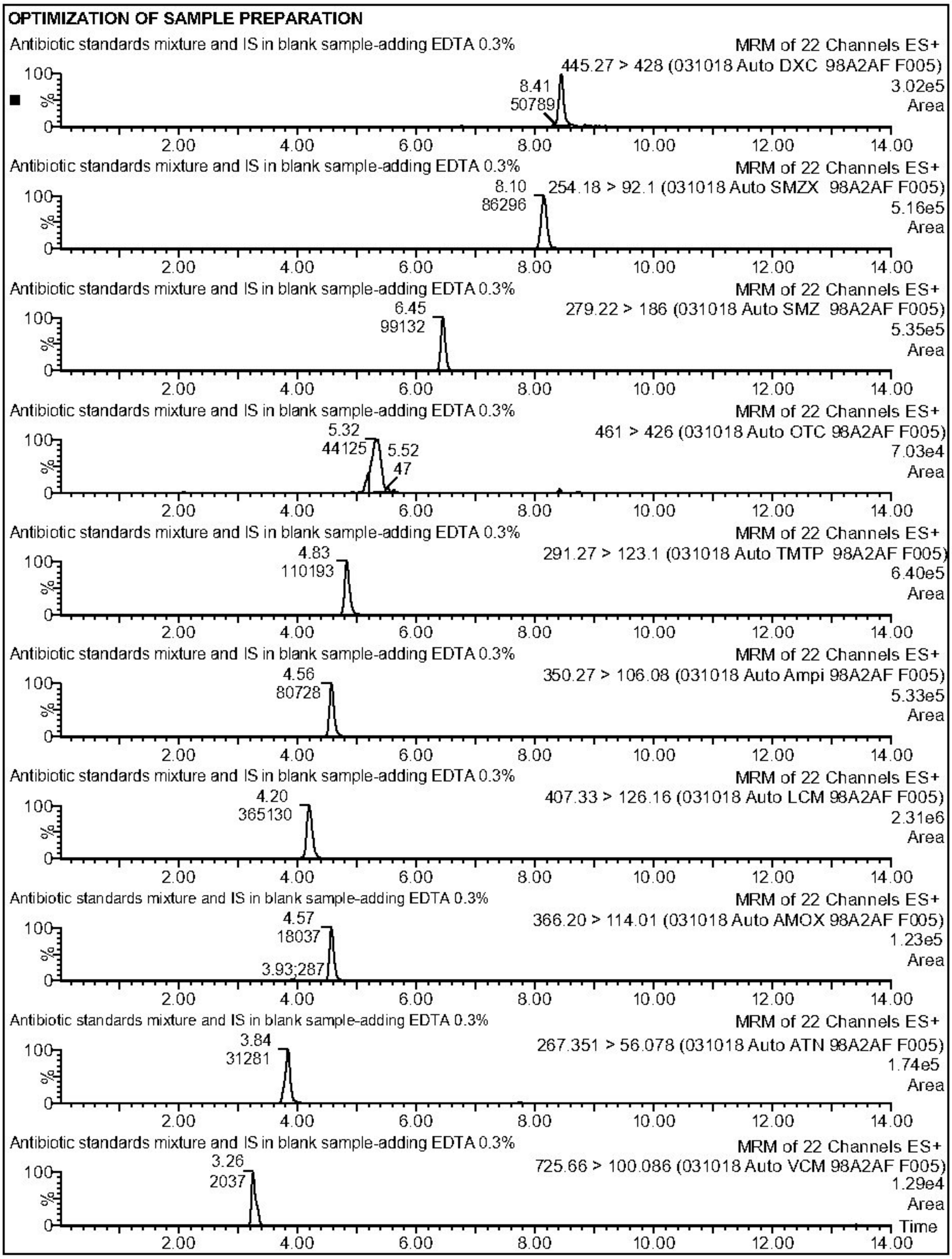
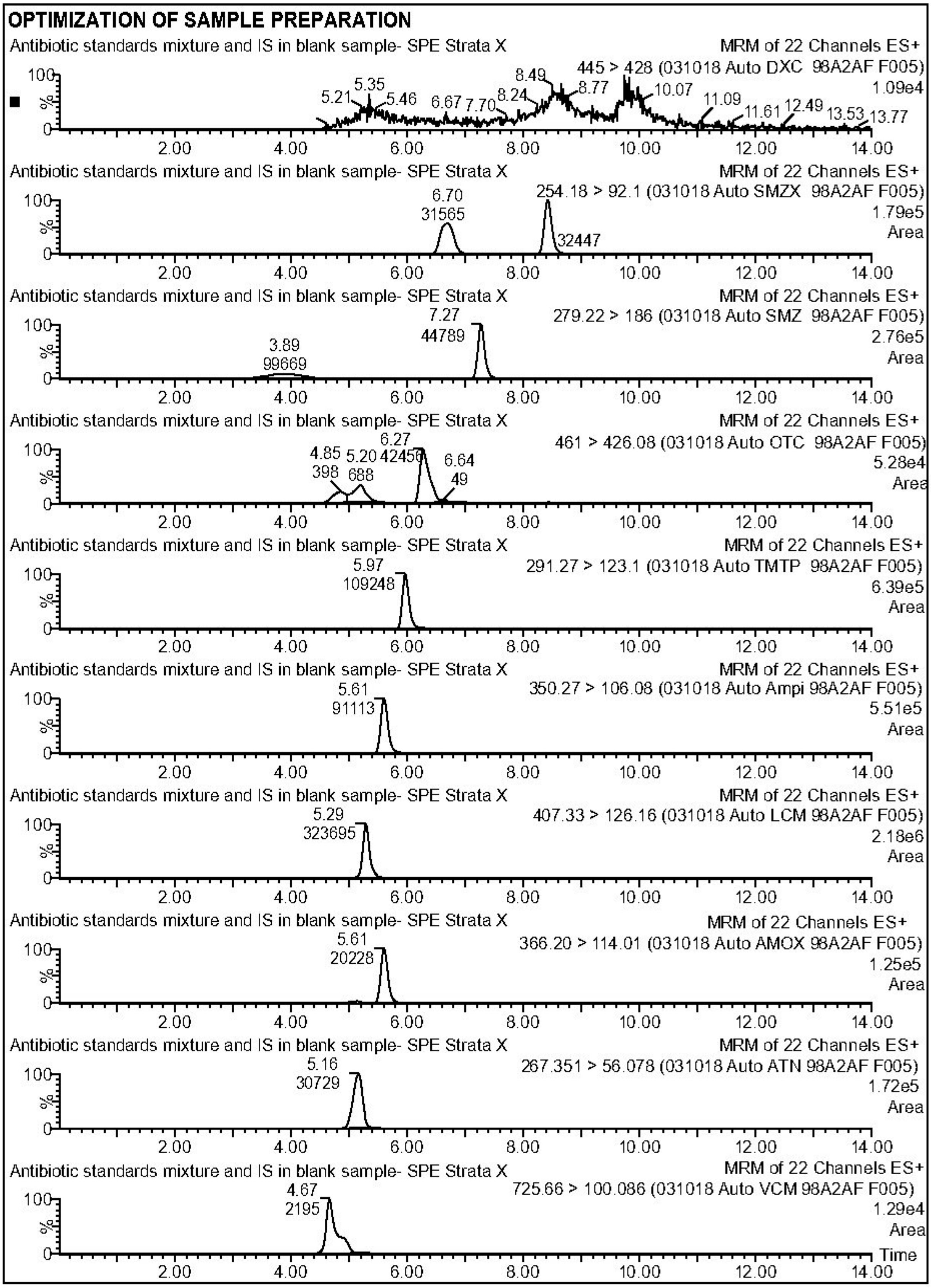


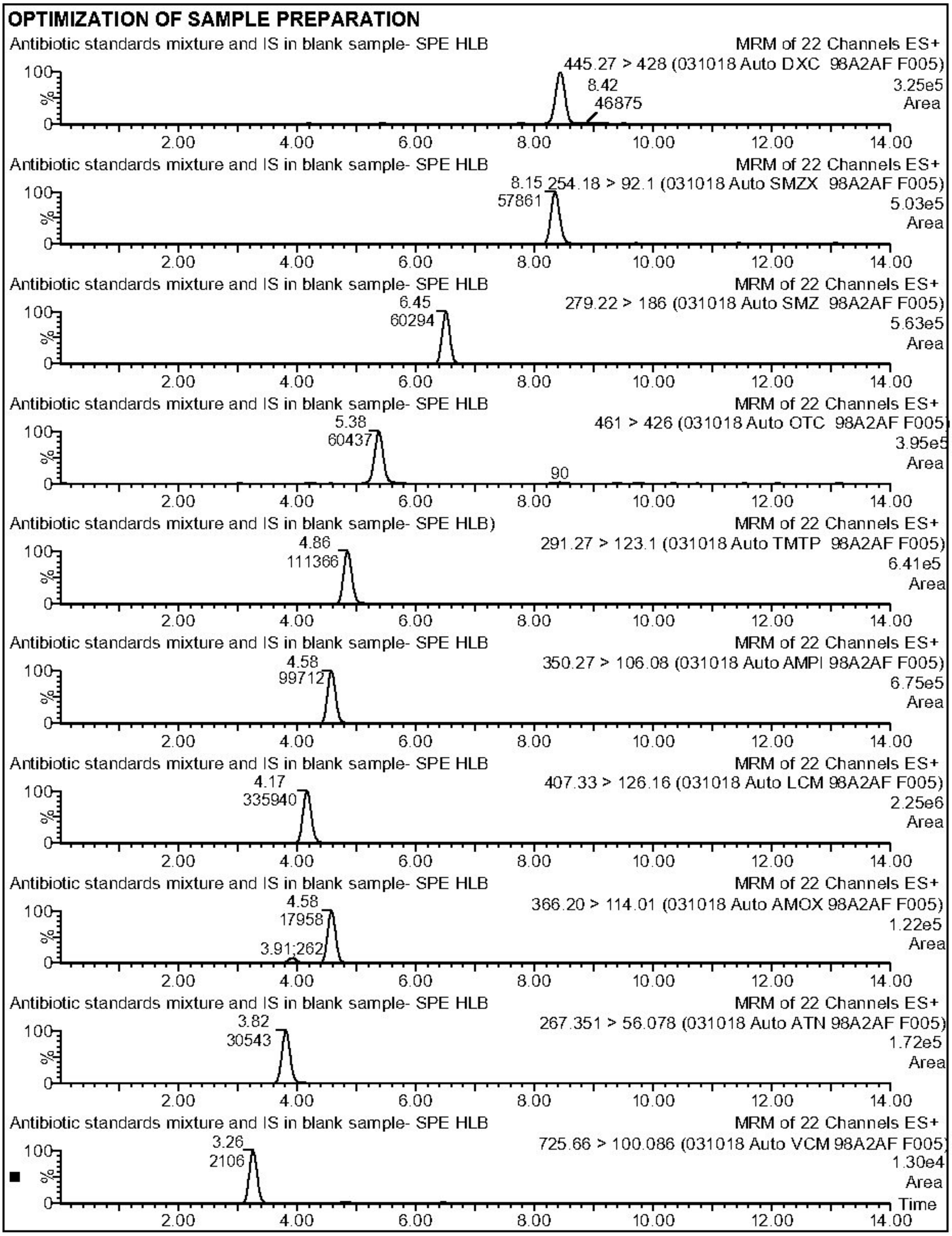
Figure S22-S25: Chromatograms present the investigation of sample preparation
S22. LC-MS/MS chromatogram of sample preparation by adding EDTA 0.3%.



S23. LC-MS/MS chromatogram of sample preparation by using SPE-Strata X.



S24. LC-MS/MS chromatogram of sample preparation by using SPE-Oasis HLB.



S25. LC-MS/MS chromatogram of sample preparation by adding EDTA 0.3% + SPE-HLB.

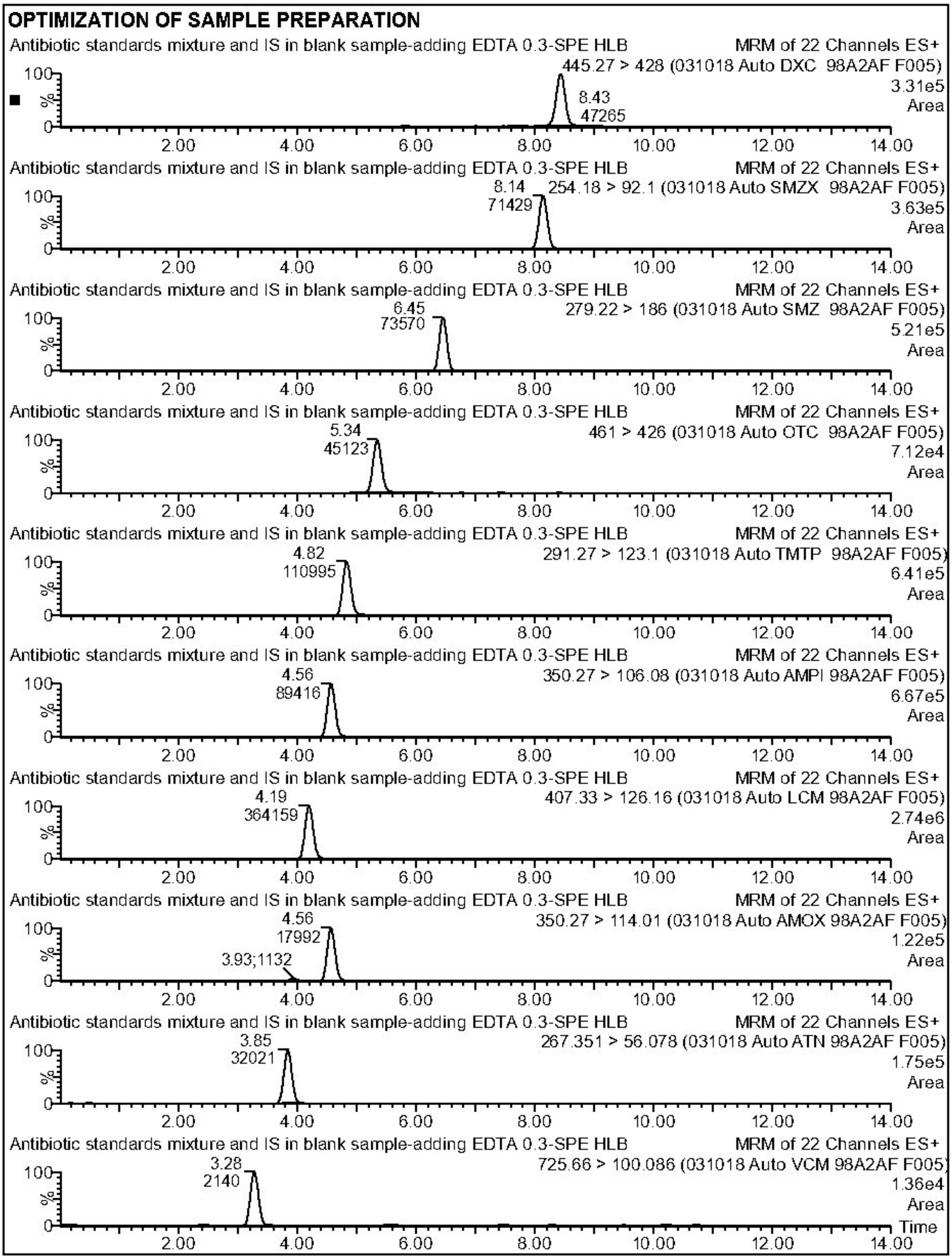
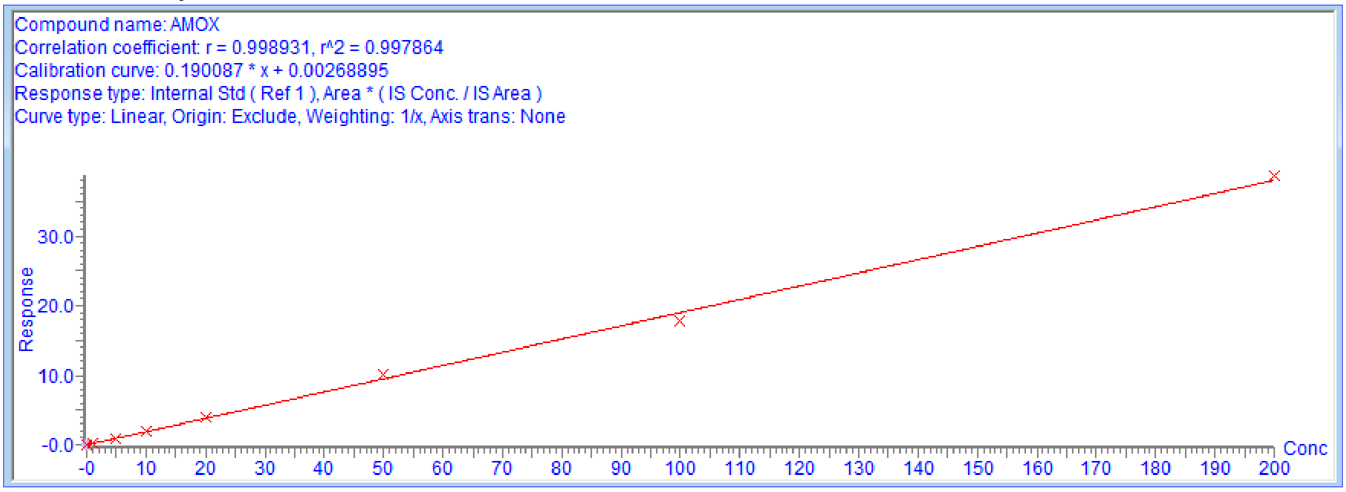
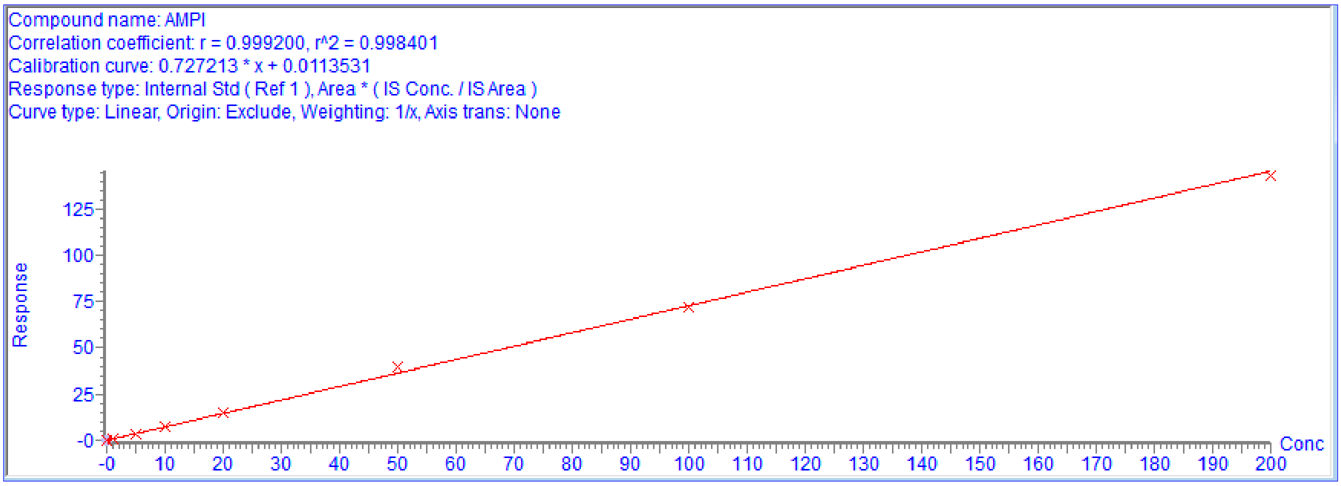


Figure S26-S33: Method validation-Linearity of target antibiotics

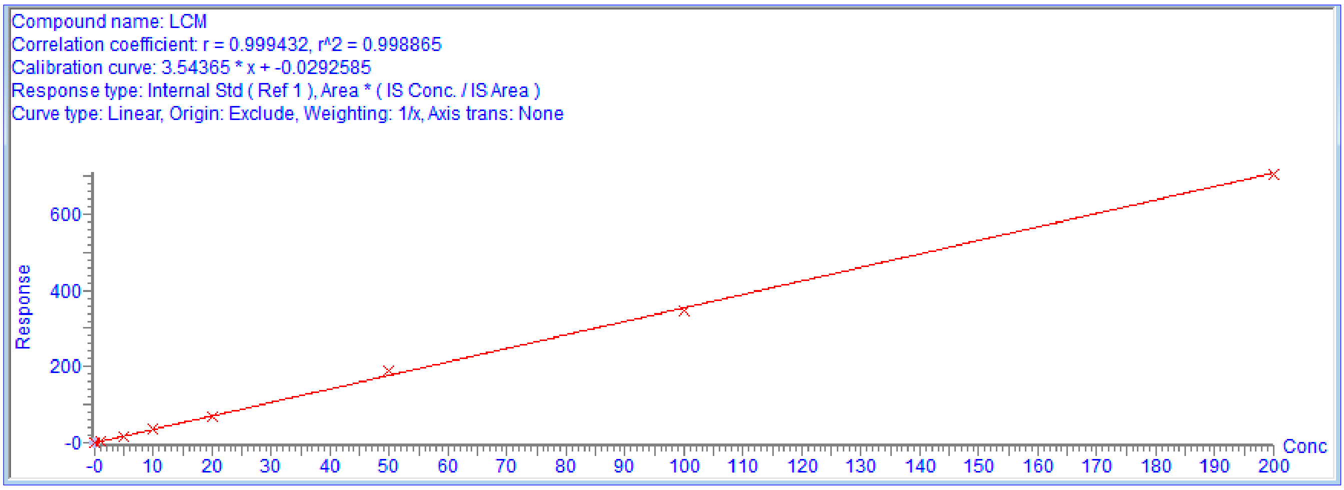
S26. Linearity of amoxicilline.



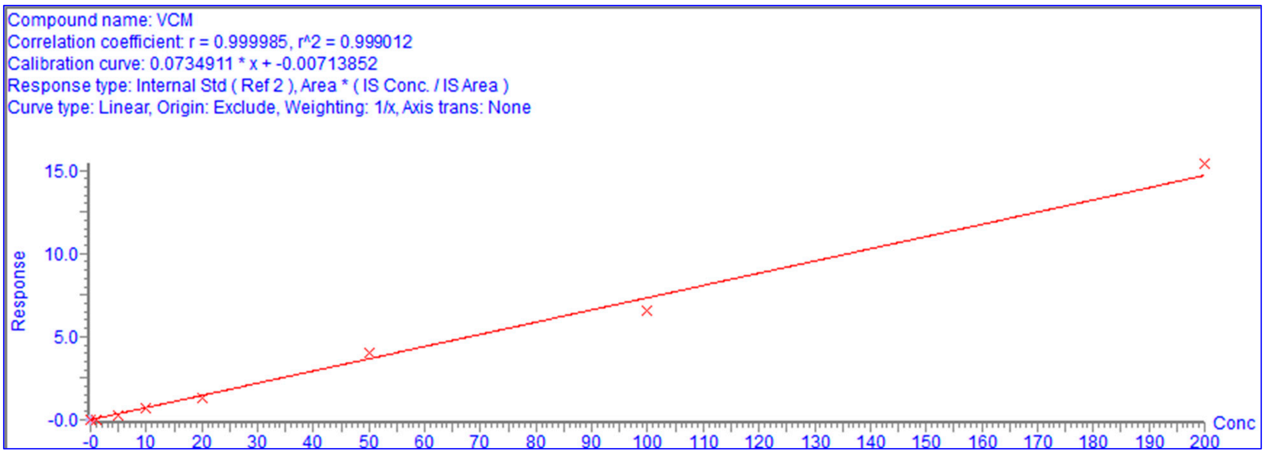
S27. Linearity of ampicilline.



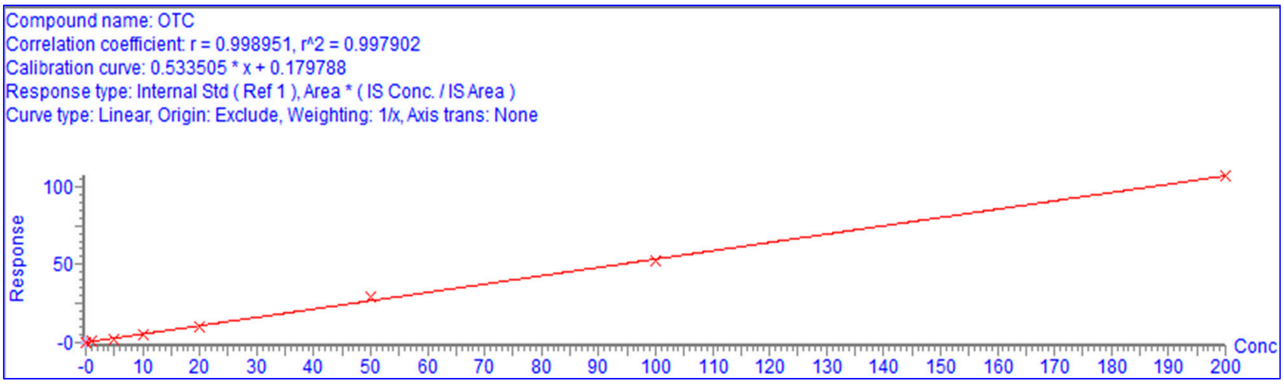
S28. Linearity of lincomycin (LCM).



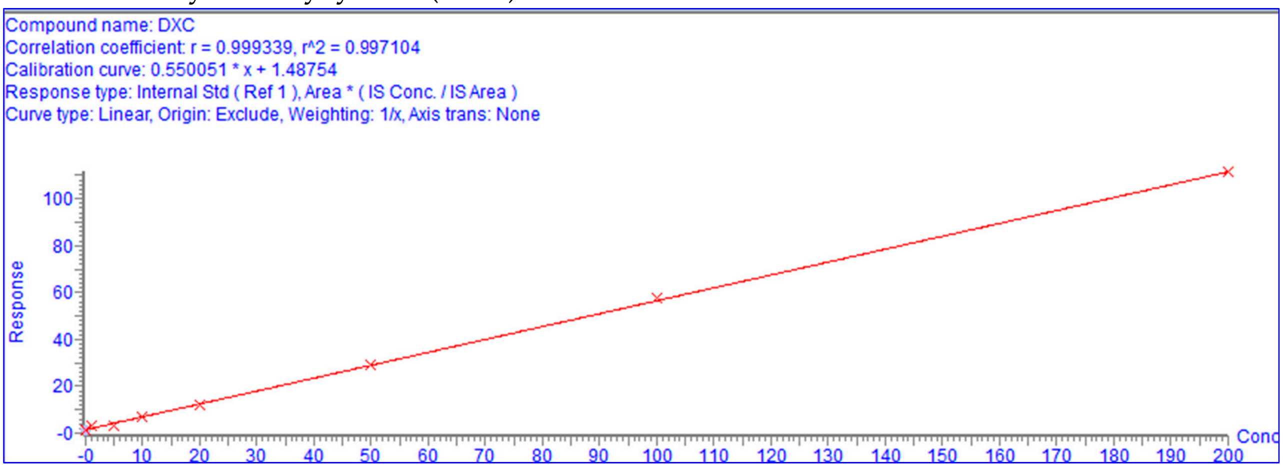
S29. Linearity of vancomycin (VCM).



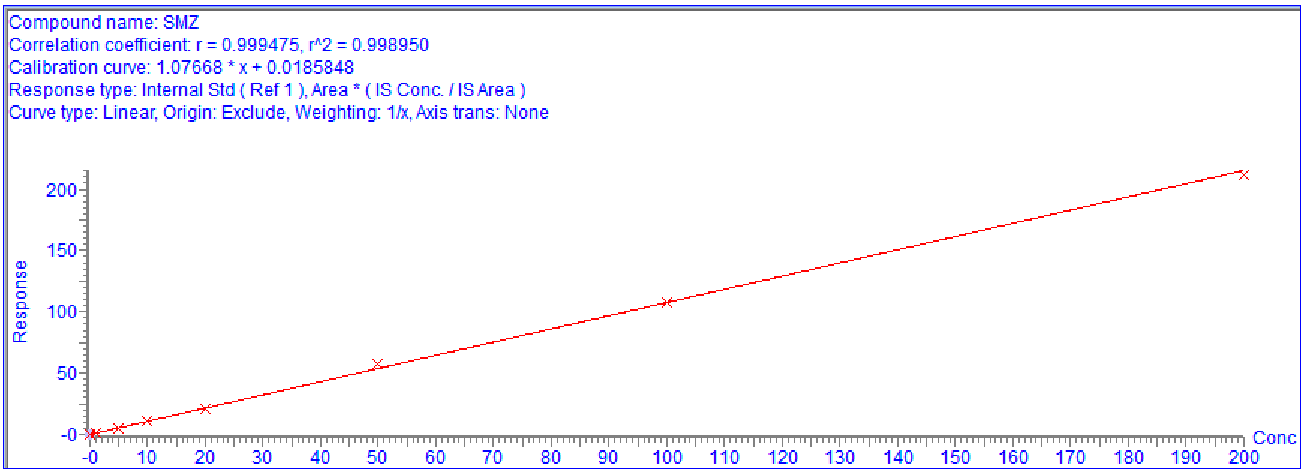
S30. Linearity of oxytetracycline (OTC).



S31. Linearity of doxycycline (DXC).



S32. Linearity of sulfamethazine (SMZ).



S33. Linearity of sulfamethoxazole (SMZX).

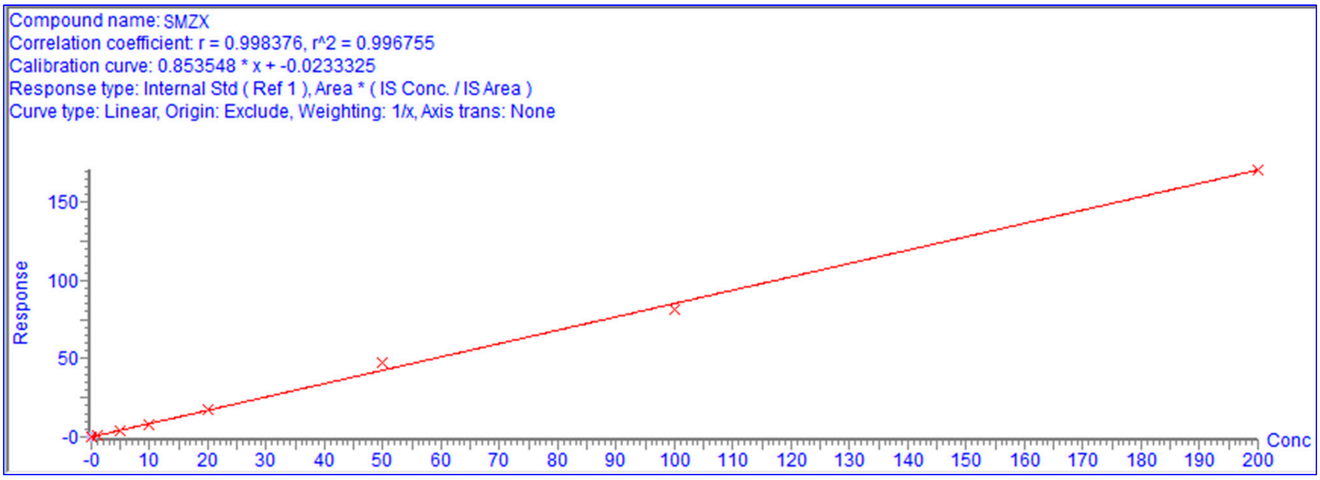
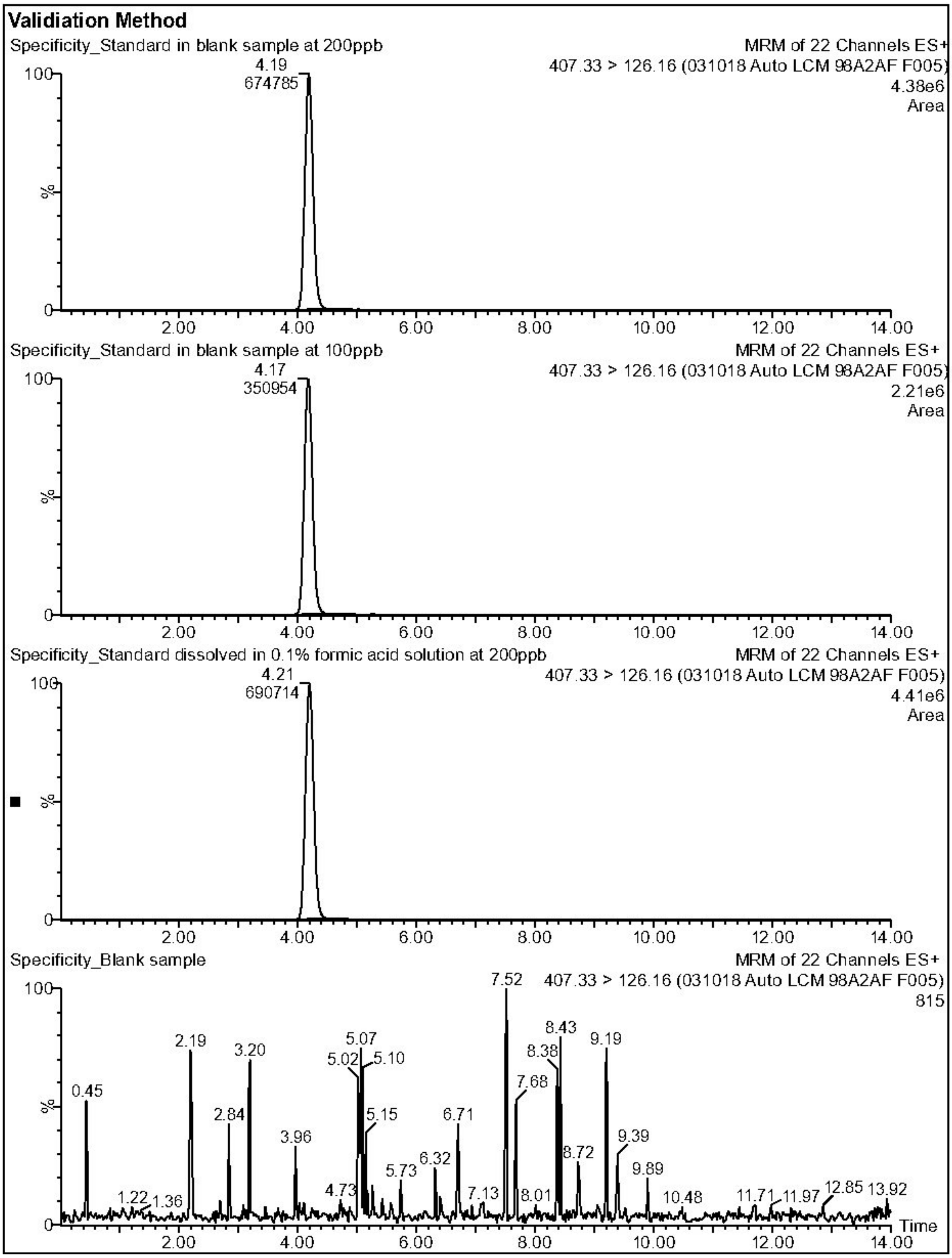
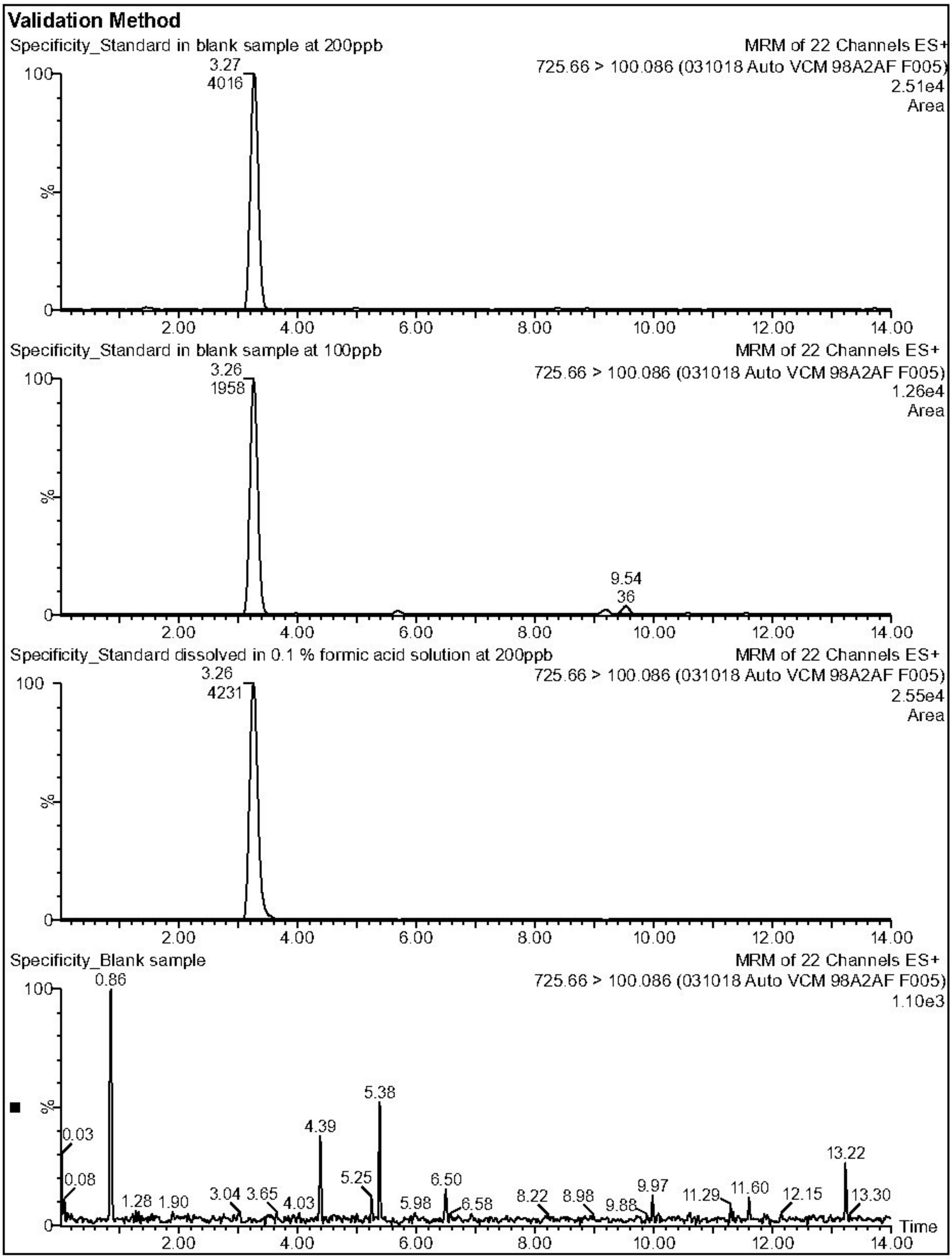


Figure S34-S36: Method validation- LC-MS/MS chromatograms for specificity of target antibiotics.

S34. LC-MS/MS chromatogram of specificity of lincomycin (LCM).



S35. LC-MS/MS chromatogram of specificity of vancomycin.



S36. LC-MS/MS chromatogram of specificity of doxycycline (DXC).

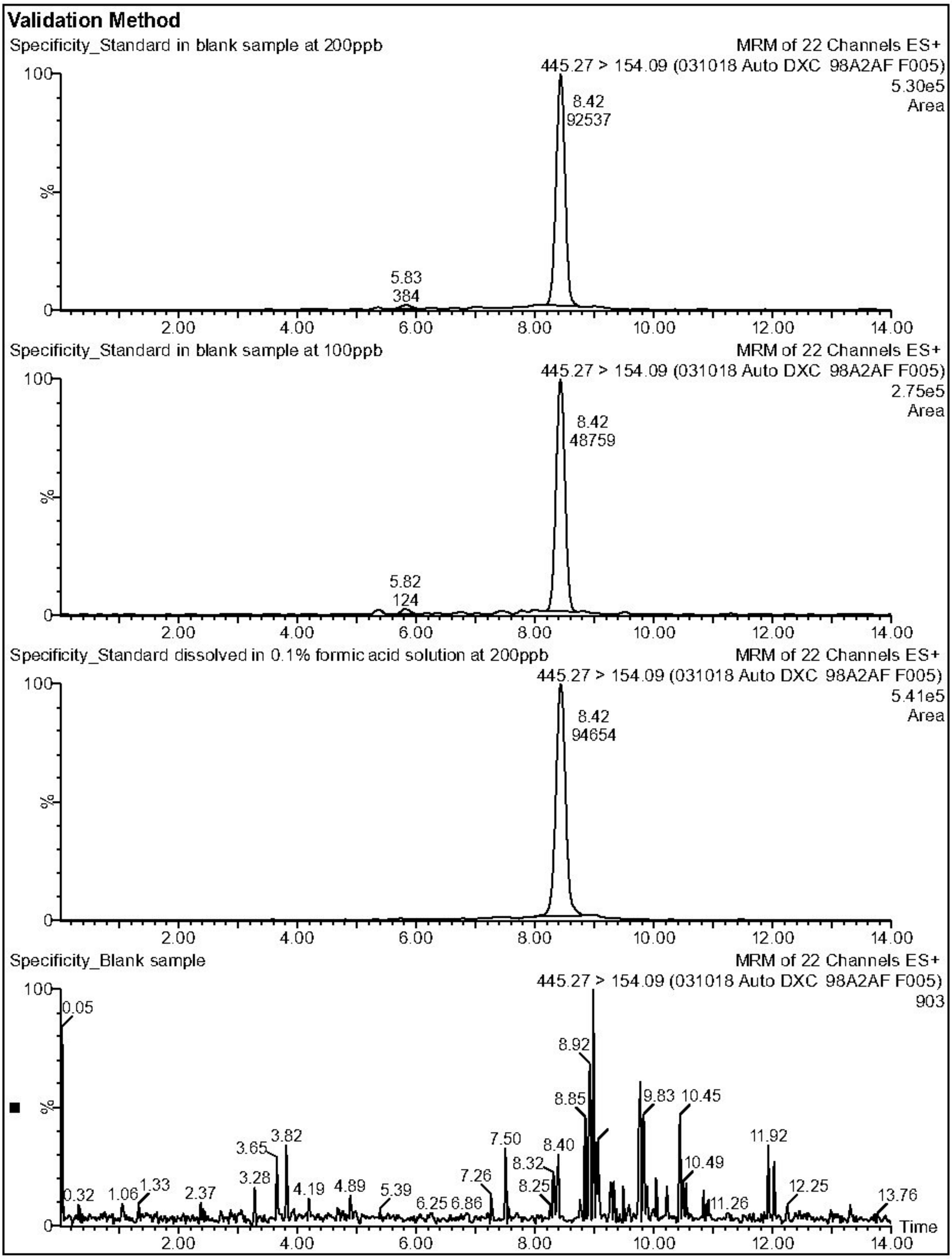
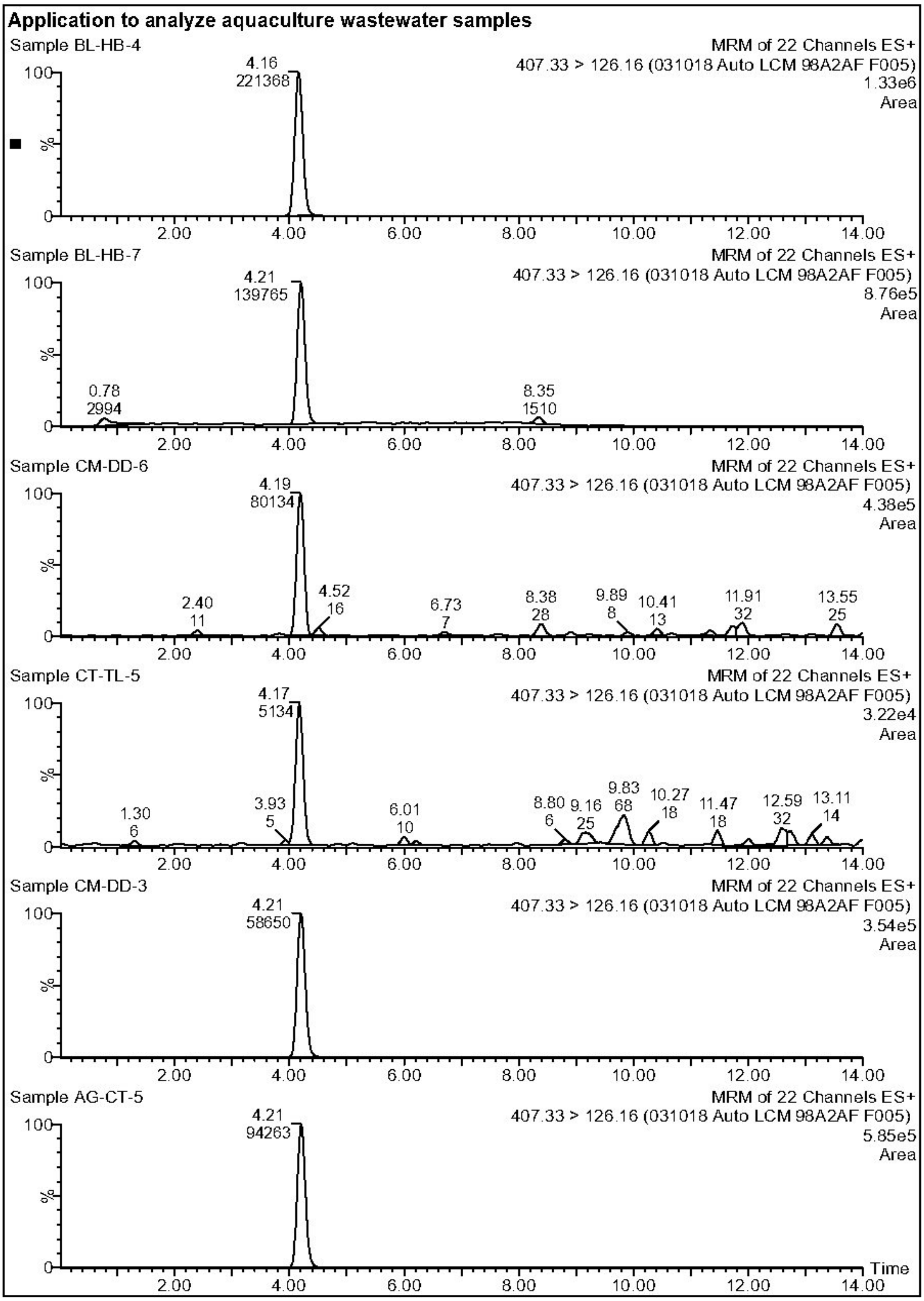
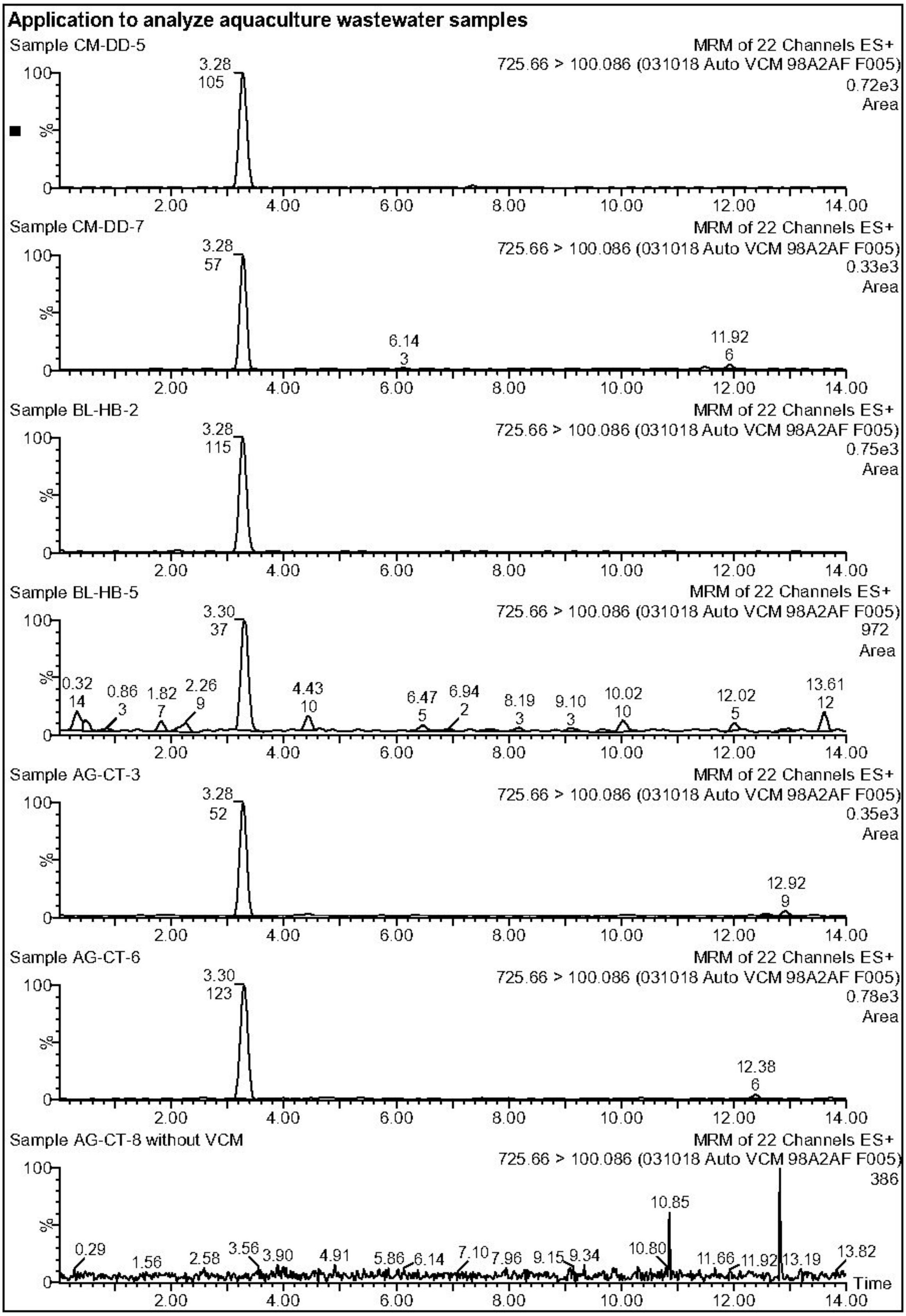


Figure S37-S41: LC-MS/MS chromatograms of analyzing aquaculture and river/canal water samples.

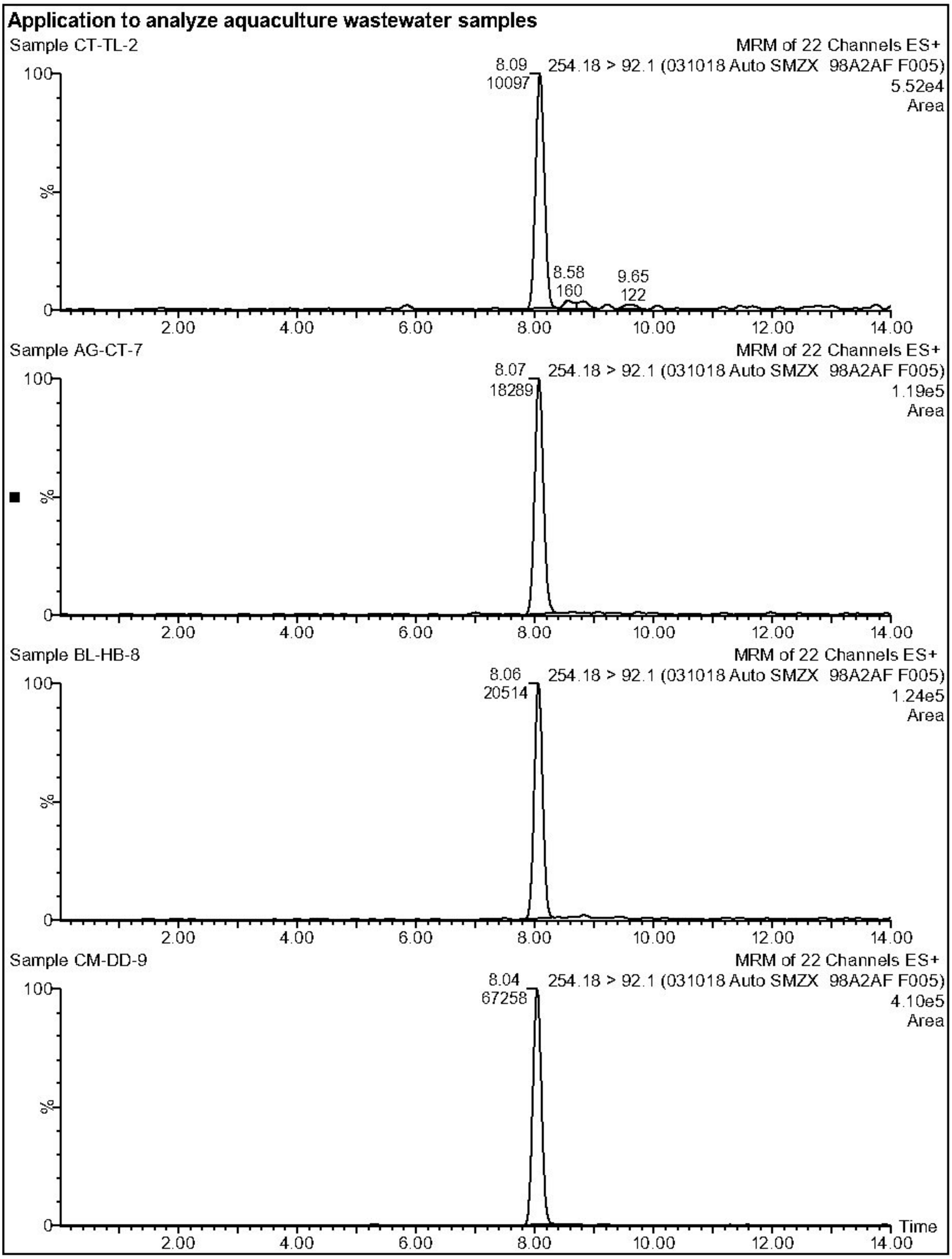
S37. LC-MS/MS chromatogram of lincomycin detection in aquaculture wastewater samples.



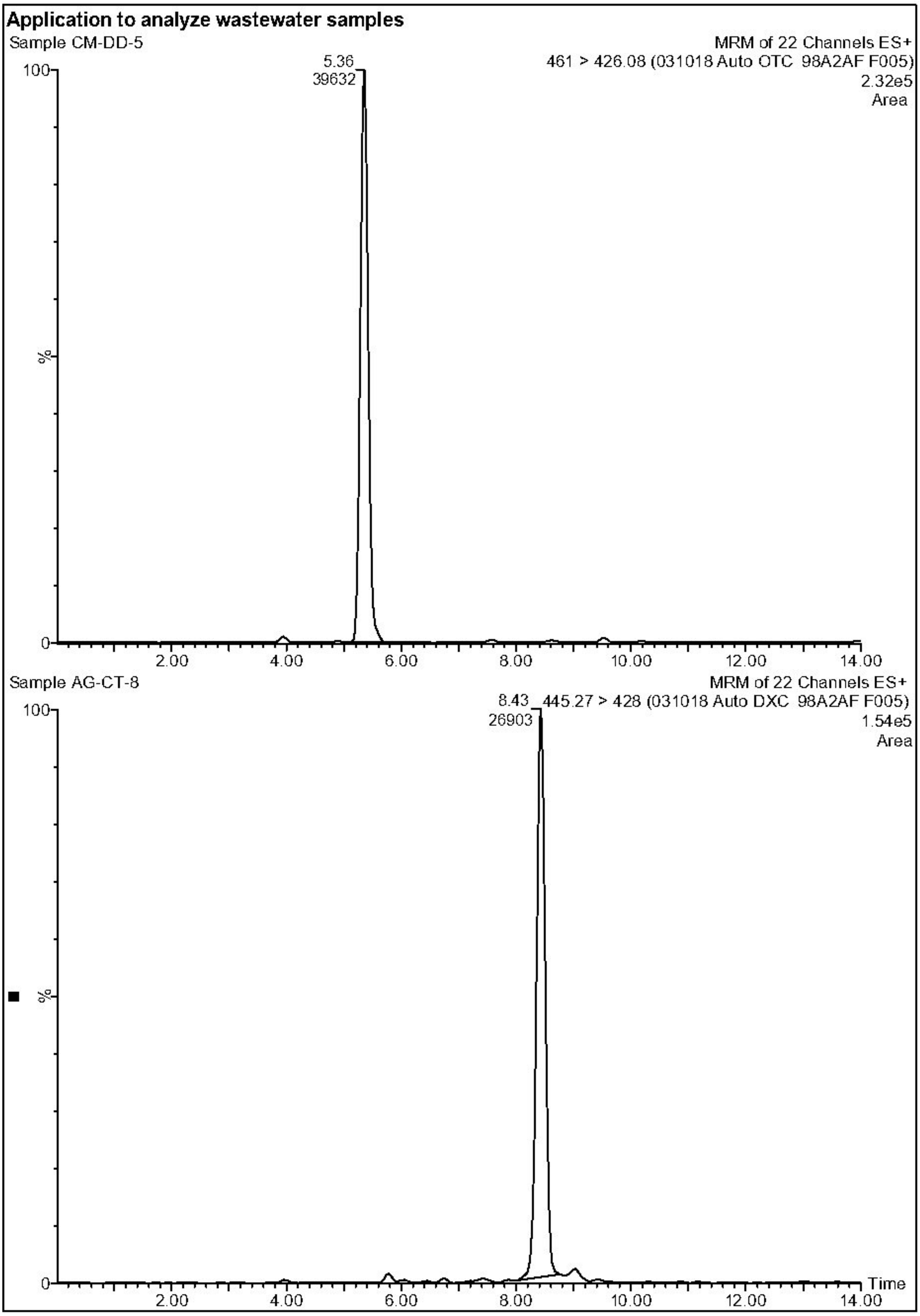
S38. LC-MS/MS chromatogram of vancomycin (VCM) detection in aquaculture wastewater samples.



S39. LC-MS/MS chromatogram of sulfamethoxazole (SMZX) detection in aquaculture wastewater samples.



S40. LC-MS/MS chromatograms show the high concentrations of oxytetracycline and doxycycline in aquaculture wastewater samples.



S41. LC-MS/MS chromatogram of doxycycline detection in river/canal water samples.