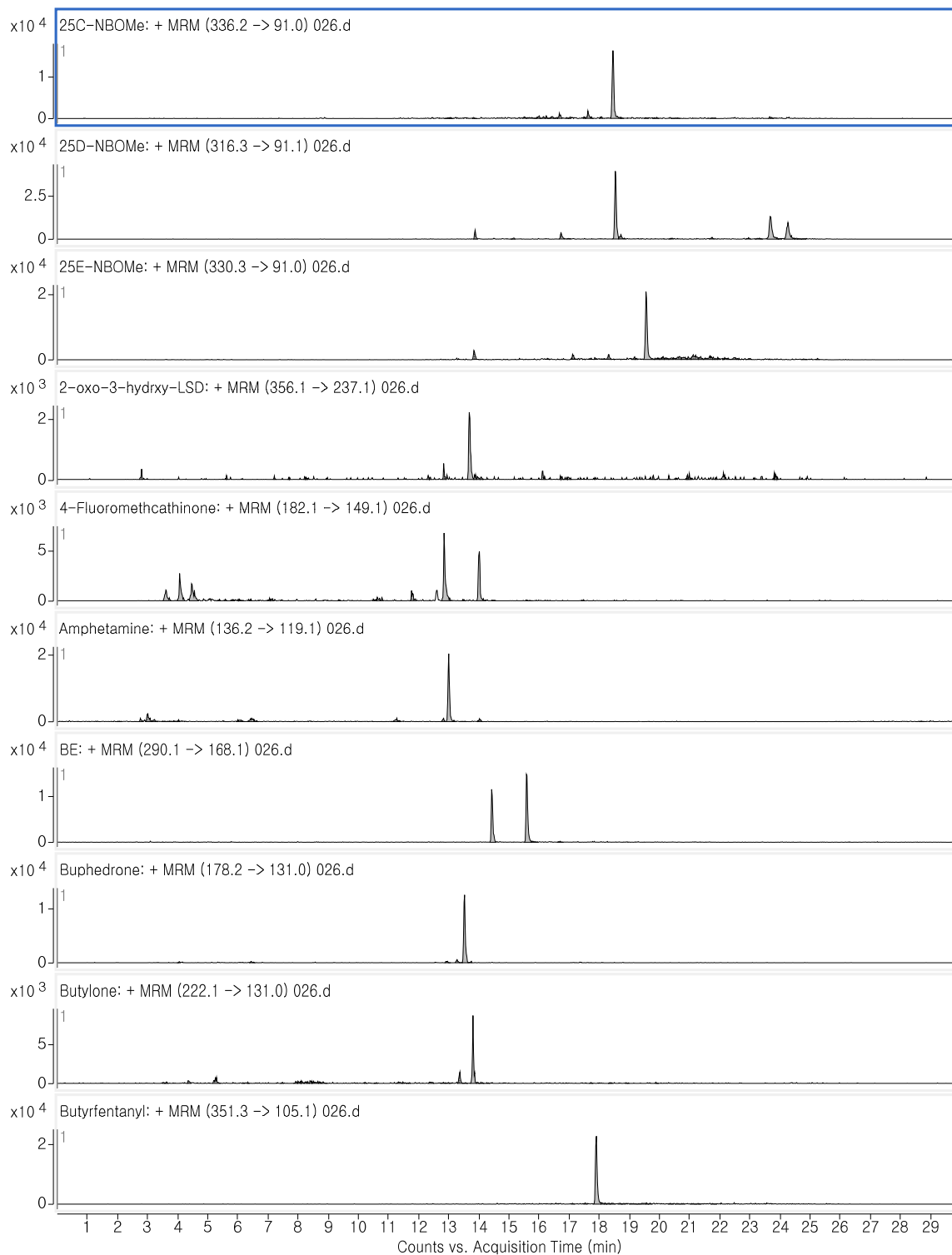




## Supplementary Materials



**Fig. S1.** MRM chromatogram of 37 NPS in wastewater.

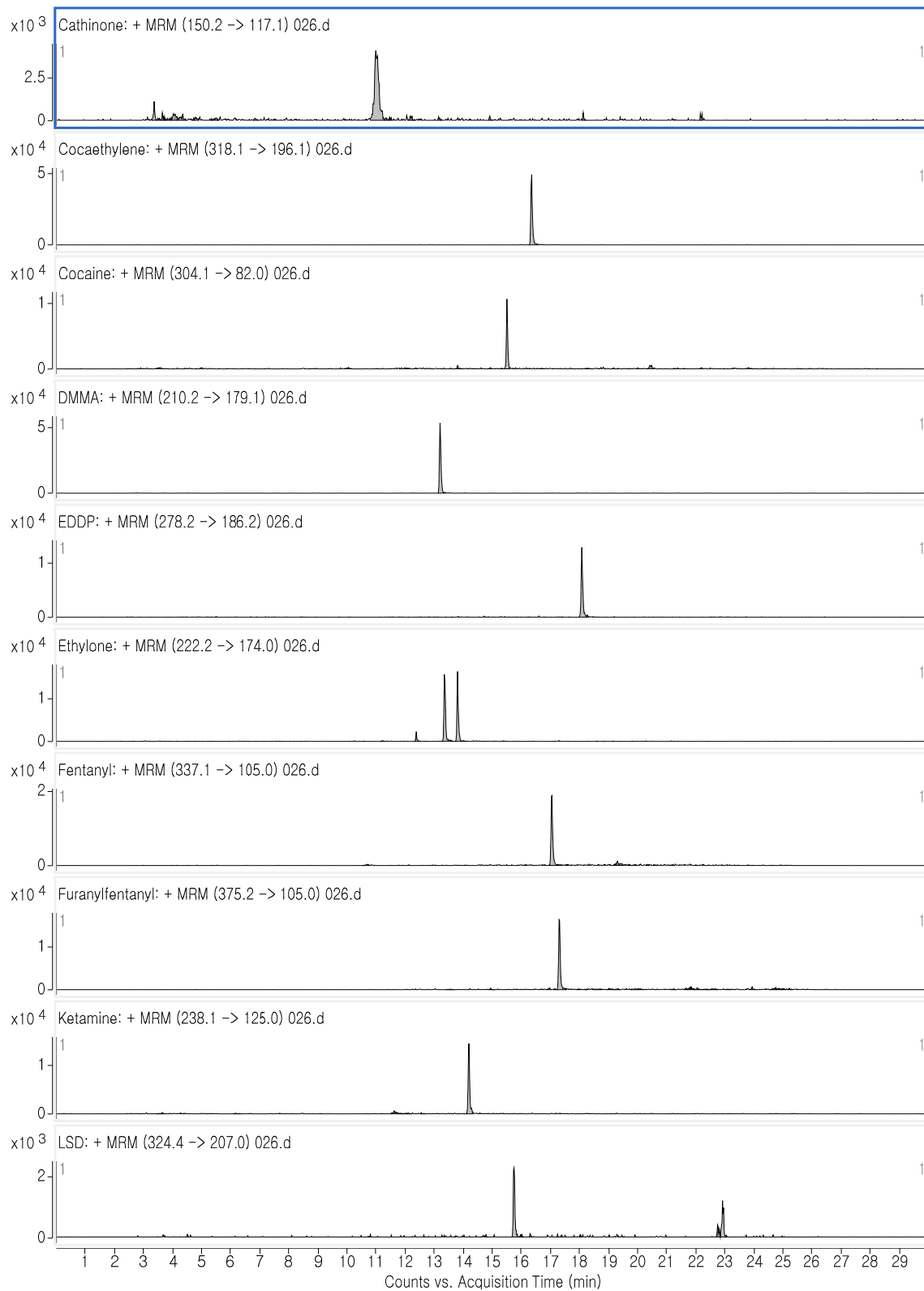
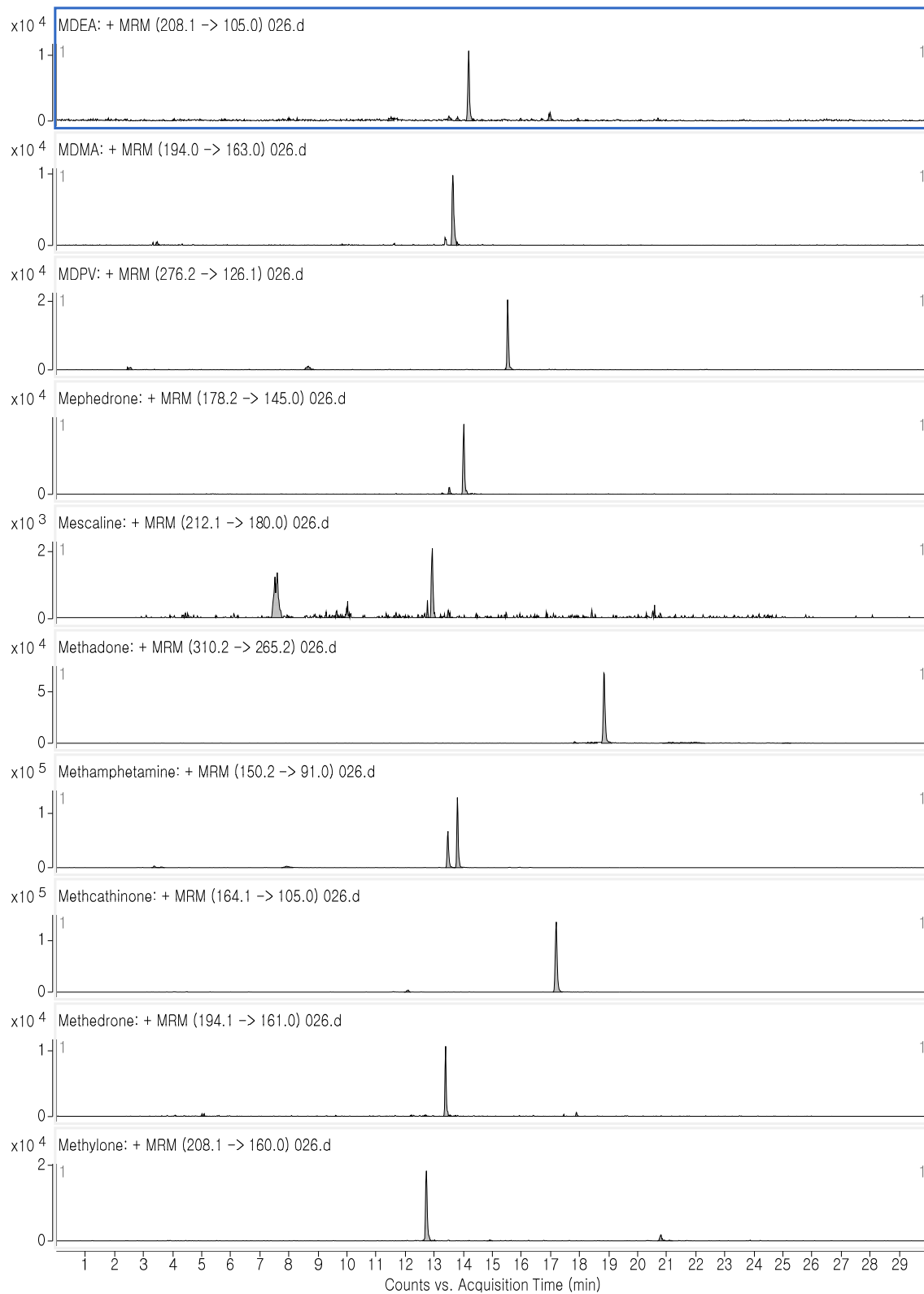
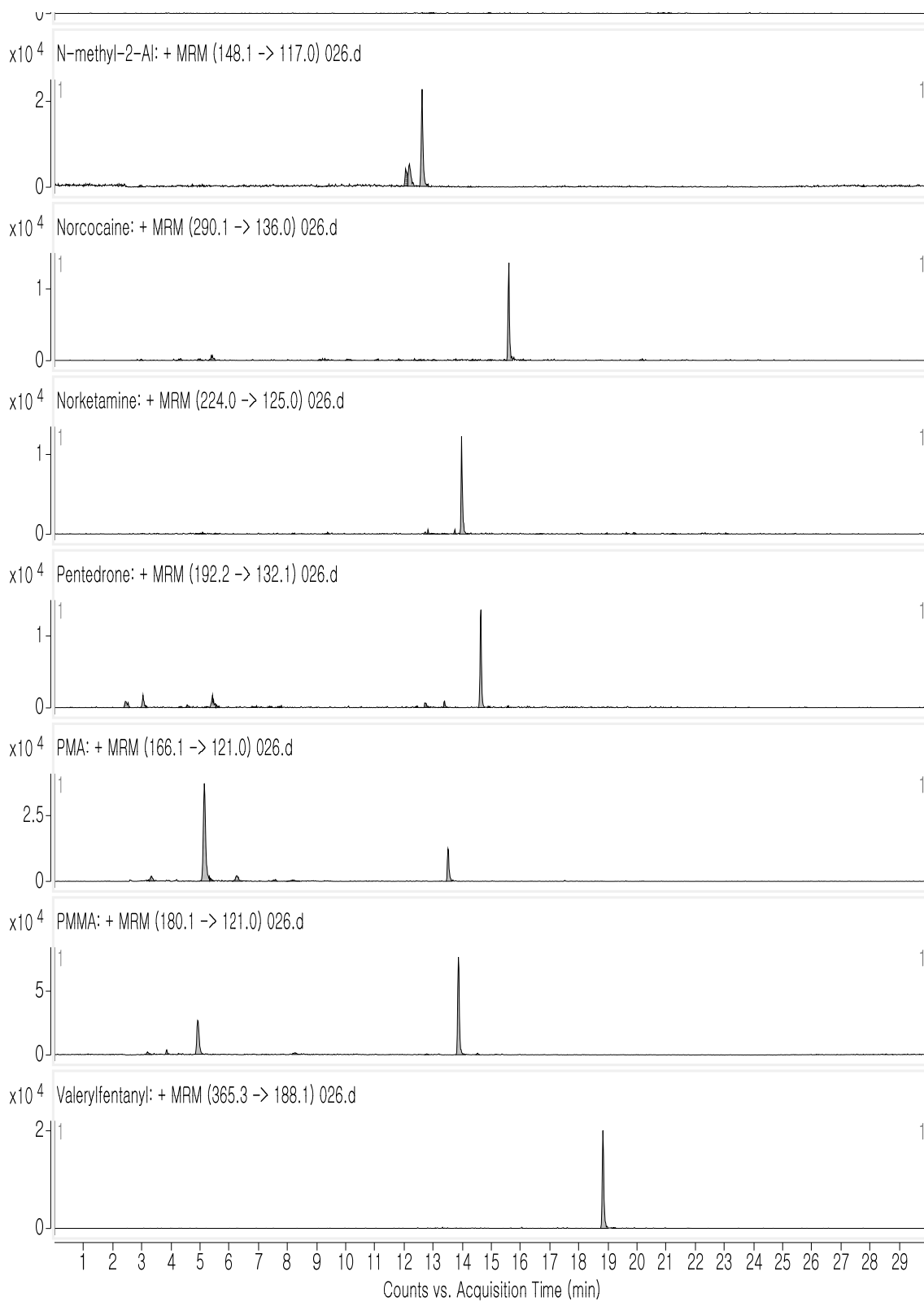


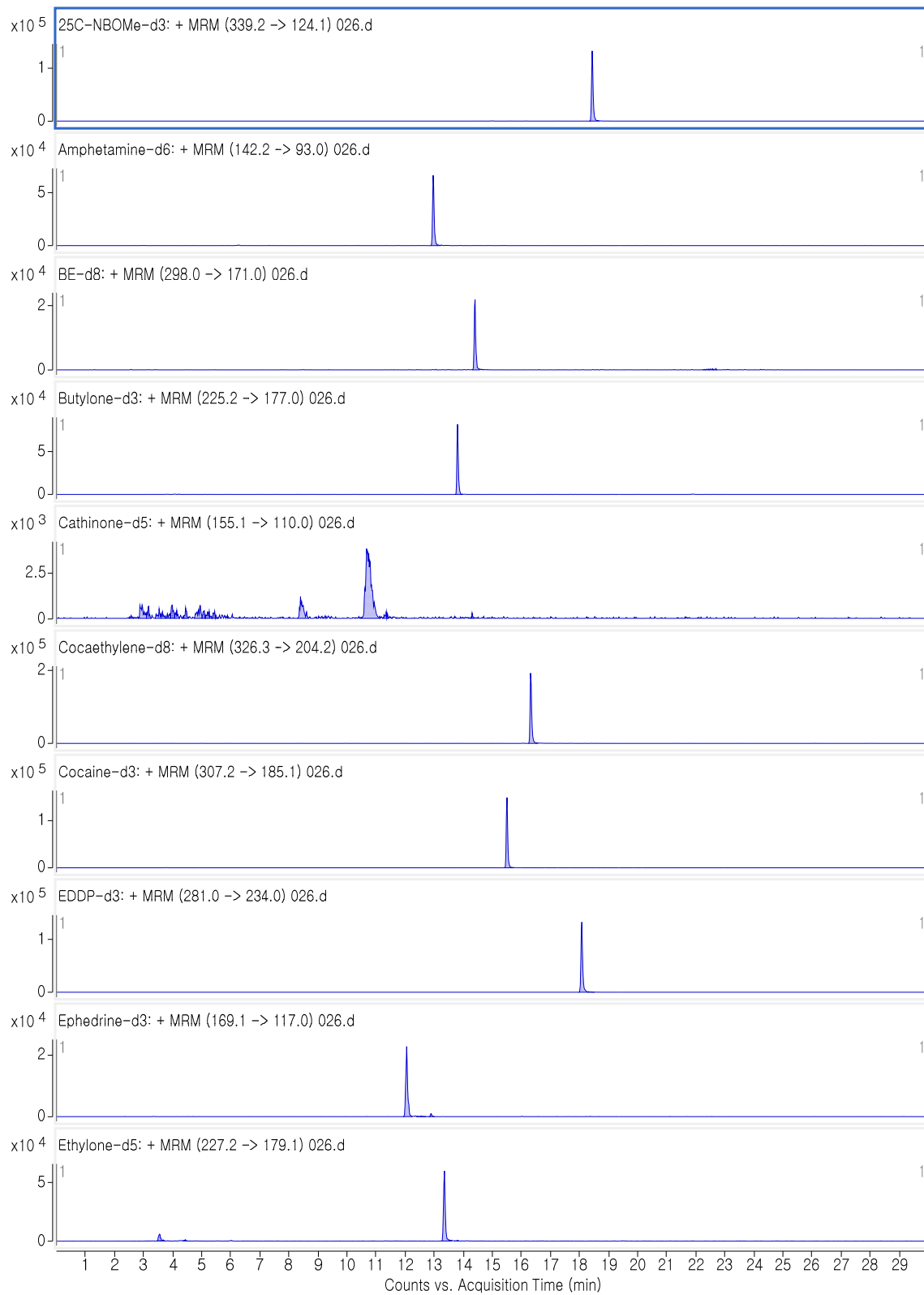
Fig. S2. MRM chromatogram of 37 NPS in wastewater (continued).



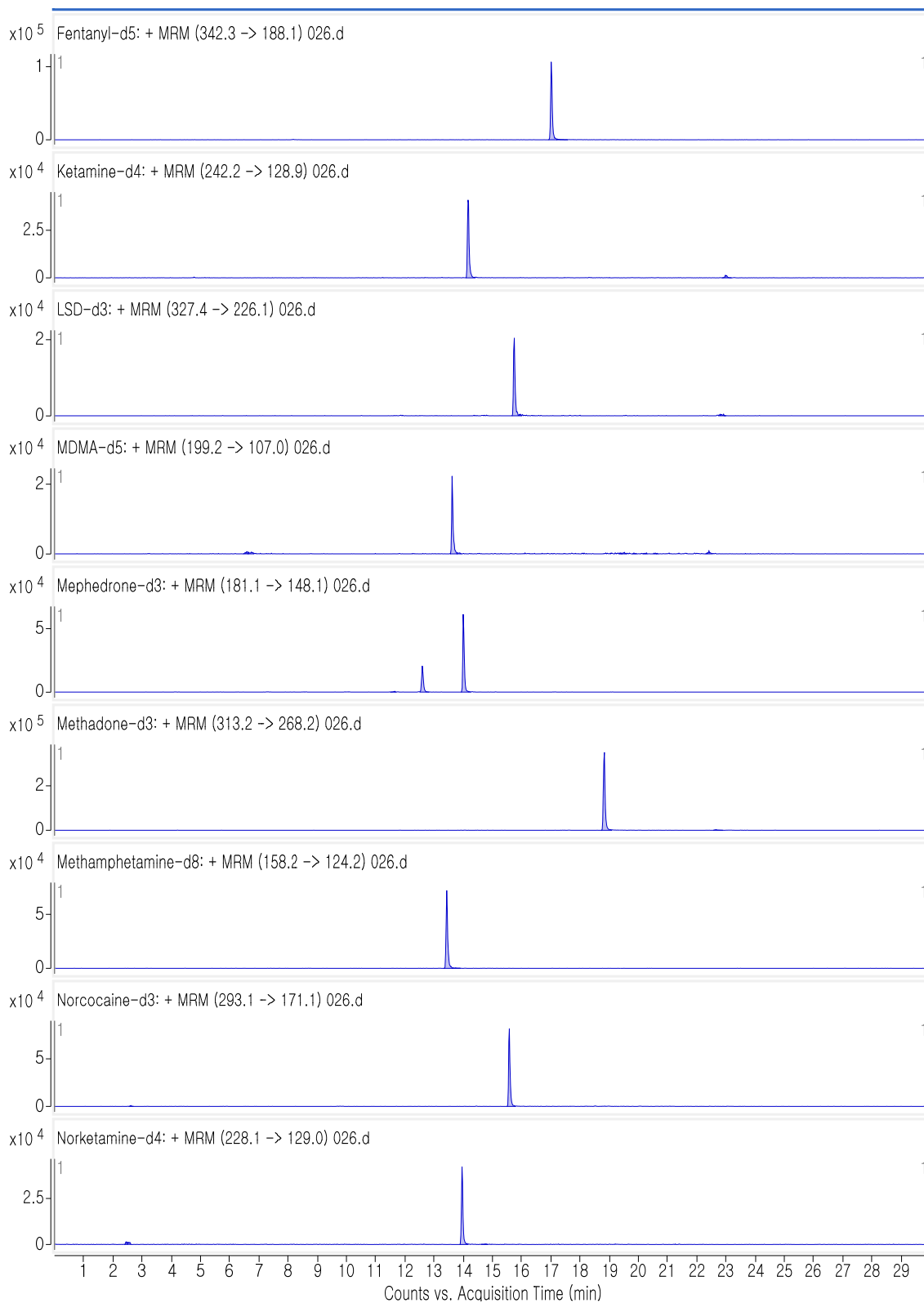
**Fig. S3.** MRM chromatogram of 37 NPS in wastewater (continued).



**Fig. S4.** MRM chromatogram of 37 NPS in wastewater (continued).



**Fig. S5.** MRM chromatogram of internal standards in wastewater.



**Fig. S6.** MRM chromatogram of internal standards in wastewater (continued).

**Table S1.** MRM condition of target chemicals

No	Compound name	Molecular weight (g/mol)	Precursor ion (m/z)	Product ion1 (m/z)	Collision energy1 (V)	Product ion2 (m/z)	Collision energy2 (V)	Ionization mode	RT (min)	ISTD
1	25C-NBOMe	335.8	336.2	121.1	10	91	40	Positive(+)	18.5	25C-NBOMe-d3
2	25D-NBOMe	315.4	316.3	121.1	14	91.1	40	Positive(+)	18.6	MDMA-d5
3	25E-NBOMe	329.4	330.3	120.9	16	91	37	Positive(+)	19.6	25C-NBOMe-d3
4	Amphetamine	135.2	136.2	119.1	2	91	13	Positive(+)	13.0	Amphetamine-d6
5	Dimethoxymethamphetamine (DMMA)	209.3	210.2	179.1	3	151.1	19	Positive(+)	13.2	Amphetamine-d6
6	MDEA	207.3	208.1	163.1	6	105	24	Positive(+)	14.2	MDMA-d5
7	MDMA	193.2	194	163	6	105	22	Positive(+)	13.7	MDMA-d5
8	Methamphetamine	149.2	150.2	119.1	5	91	17	Positive(+)	13.5	Methamphetamine-d8
9	N-methyl-2-AI	147.2	148.1	117	10	115	30	Positive(+)	12.7	Amphetamine-d6
10	p-Methoxymphetamine (PMA)	165.2	166.1	121	20	91	30	Positive(+)	13.5	Amphetamine-d6
11	para-Methoxy-N-methylamphetamine (PMMA)	179.1	180.1	149	6	121	17	Positive(+)	13.9	Amphetamine-d6
12	Cocaine	303.4	304.1	182.1	15	82	32	Positive(+)	15.5	Cocaine-d3
13	BE	289.3	290.1	168.1	14	77.2	46	Positive(+)	14.5	BE-d8
14	Cocaehtylene	317.4	318.1	196.1	16	82.1	28	Positive(+)	16.4	Cocaehtylene-d8
15	Norcocaine	289.3	290.1	168	10	136	19	Positive(+)	15.6	Norcocaine-d3
16	Ketamine	237.7	238.1	220	9	125	26	Positive(+)	14.2	Ketamine-d4
17	Norketamine	223.7	224	207	4	125	21	Positive(+)	14.0	Norketamine-d4
18	LSD	323.4	324.4	223.1	20	207	44	Positive(+)	15.8	LSD-d3
19	2-oxo-3-hydroxy-LSD	355.4	356.1	237.1	20	221.9	40	Positive(+)	13.7	Methamphetamine-d8
20	Mescaline	211.3	212.1	195	3	180	14	Positive(+)	13.0	Butylone-d3
21	Cathinone	149.2	150.2	132.2	5	117.1	23	Positive(+)	11.1	Cathinone-d5
22	3,4-Methylenedioxypropyvalerone (MDPV)	275.2	276.2	135	27	126.1	27	Positive(+)	15.6	Methamphetamine-d8
23	4-Fluoromethcathinone (Flephedrone)	181.1	182.1	149.1	20	103	30	Positive(+)	12.9	Ethylone-d5
24	Buphedrone	177.1	178.2	131	20	91	30	Positive(+)	13.6	Amphetamine-d6
25	Butylone	221.1	222.1	174.1	10	131	37	Positive(+)	13.8	Butylone-d3
26	Ethylone	221.1	222.2	174	20	146	27	Positive(+)	13.4	Ethylone-d5
27	Mephedrone	177.1	178.2	145	19	119	20	Positive(+)	14.0	Mephedrone-d3
28	Methcathinone	163.1	164.1	146	7	131	20	Positive(+)	12.2	Mephedrone-d3
29	Methedrone	193.1	194.1	161	20	146	29	Positive(+)	13.4	Mephedrone-d3
30	Methylone	207.1	208.1	160	10	132	25	Positive(+)	12.8	Butylone-d3
31	Pentadron	191.1	192.2	132.1	15	91.1	25	Positive(+)	14.7	Methamphetamine-d8
32	Butyrfentanyl	350.5	351.3	188.2	20	105.1	40	Positive(+)	17.9	Fentanyl-d5
33	Fentanyl	336.5	337.1	118.2	20	105	46	Positive(+)	17.1	Fentanyl-d5
34	Furanylfentanyl	374.5	375.2	188.1	20	105	40	Positive(+)	17.3	Fentanyl-d5
35	Valerylfentanyl	364.5	365.3	188.1	20	105.1	40	Positive(+)	18.9	LSD-d3
36	Methadone	309.5	310.2	285.1	9	105	29	Positive(+)	18.9	Methadone-d3
37	EDDP	277.2	278.2	234.1	29	186.2	37	Positive(+)	18.1	EDDP-d3

Table S2. MRM condition of internal standards

No	Compound name	Molecular weight (g/mol)	Precursor ion (m/z)	Production1 (m/z)	Collision energy1 (V)	Production2 (m/z)	Collision energy2 (V)	Ionization mode	RT (min)
1	25C-NBOMe-d3	338.1	339.2	124.1	15	92.1	50	Positive(+)	18.5
2	Amphetamine-d6	141.1	142.2	125.1	1	93	10	Positive(+)	13.0
3	BE-d8	297.4	298	171	16	109.9	29	Positive(+)	14.5
4	Butylone-d3	224.1	225.2	177	20	149.1	30	Positive(+)	13.8
5	Cathinone-d5	154.1	155.1	122.2	30	110	20	Positive(+)	11.1
6	Cocaehtylene-d8	325.2	326.3	204.2	21	85.1	30	Positive(+)	16.4
7	Cocaine-d3	306.2	307.2	185.1	18	85.1	32	Positive(+)	15.5
8	EDDP-d3	280.4	281	234	33	189	36	Positive(+)	18.1
9	Ephedrine-d3	168.2	169.1	151.1	8	117	17	Positive(+)	12.2
10	Ethylone-d5	226.1	227.2	179.1	20	151.1	30	Positive(+)	13.4
11	Fentanyl-d5	341.2	342.3	188.1	23	105.1	44	Positive(+)	17.1
12	Ketamine-d4	241.7	242.2	224.1	12	128.9	28	Positive(+)	14.2
13	LSD-d3	326.2	327.4	226.1	20	210	42	Positive(+)	15.8
14	MDMA-d5	198.3	199.2	165	7	135	20	Positive(+)	13.7
15	Mephedrone-d3	180.1	181.1	163.1	6	148.1	20	Positive(+)	14.0
16	Methadone-d3	312.2	313.2	313.2	1	268.2	10	Positive(+)	18.9
17	Methamphetamine-d8	157.2	158.2	124.2	6	93.2	18	Positive(+)	13.5
18	Norococaine-d3	292.1	293.1	171.1	10	136.1	17	Positive(+)	15.6
19	Nonketamine-d4	227.7	228.1	211.1	6	129	21	Positive(+)	14.0