AN323　用加速溶剂萃取（ASE）技术提取环境样品中的多氯二苯二噁英和多氯二苯呋喃

PCDDs： 多氯二苯二噁英　　**OCPs：** 有机氯农药

PCDFs： 多氯二苯呋喃　　**OPPS：** 有机磷农药

样品基体：环境样品（烟囱砖灰，城市尘埃，飞尘和沉积物）

仪器： Dionex ASE200

GC/MS

低压 LC（样品清洗）

溶剂： 甲苯（农药级），乙酸（试剂级），HCl（试剂级）

推荐的萃取条件

压力：　13.8Mpa（2000psi）

温度：　175－200℃

静态时间：5－15min

溶剂：　甲苯，或甲苯/乙酸（5%，V/V）（如果用 HCl 作前处理）

冲洗体积：　60－70%

N_2 吹扫时间：　60－100 秒

静态循环次数：2 或 3 次

分析结果：

Table 2 Average Values (ng/kg) from Ground Chimney Brick—Comparison of Soxhlet vs. ASE

Group Totals	Soxhlet (n=1)	ASE® (n=2)
Total T_4CDD	440	530
Total P_5CDD	900	940
Total H_6CDD	1800	2000
Total H_7CDD	2000	2100
Total O_8CDD	2900	2600
Total T_4CDF	2300	2600
Total P_5CDF	4100	4300
Total H_6CDF	4700	4700
Total H_7CDF	2800	2600
Total O_8CDF	2000	2000
Congeners	**Soxhlet (n=1)**	**ASE® (n=2)**
2,3,7,8-T_4CDD	6.0	6.0
1,2,3,7,8-P_5CDD	52	57
1,2,3,4,7,8-H_6CDD	46	52
1,2,3,6,7,8-H_6CDD	120	130
1,2,3,7,8,9-H_6CDD	97	1000
1,2,3,4,6,7,8-H_7CDD	1000	820
2,3,7,8-T_4CDF	160	180
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	430	470
2,3,4,7,8-P_5CDF	390	390
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	1100	1100
1,2,3,6,7,8-H_6CDF	540	570
2,3,4,6,7,8-H_6CDF	400	360
1,2,3,7,8,9-H_6CDF	42	42
1,2,3,4,6,7,8-H_7CDF	2100	2000
1,2,3,4,7,8,9-H_7CDF	140	120
Toxicity equivalent (NATO)	540	540
Toxicity equivalent (BgVV)	490	510

Values are corrected for recovery of ^{13}C-labeled surrogates.
®Sum of two extractions of each sample.

Table 3 Average Values (ng/kg) from Urban Dust—Comparison of Soxhlet vs. ASE

Group Totals	Soxhlet (n=1)	ASE® (n=2)
Total T_4CDD	182	325
Total P_5CDD	175	221
Total H_6CDD	86.7	81.7
Total H_7CDD	221	217
Total O_8CDD	445	314
Total T_4CDF	333	419
Total P_5CDF	146	179
Total H_6CDF	65.9	122
Total H_7CDF	13.2	29.4
Total O_8CDF	n.d. (10)	n.d. (10)
Congeners	**Soxhlet (n=1)**	**ASE® (n=2)**
2,3,7,8-T_4CDD	3.3	3.2
1,2,3,7,8-P_5CDD	11.8	13.1
1,2,3,4,7,8-H_6CDD	9.8	8.0
1,2,3,6,7,8-H_6CDD	11.5	9.5
1,2,3,7,8,9-H_6CDD	n.d. (8)	n.d. (8)
1,2,3,4,6,7,8-H_7CDD	113	107
2,3,7,8-T_4CDF	12.5	18.6
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	9.9	12.0
2,3,4,7,8-P_5CDF	13.9	18.1
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	18.7	23.7
1,2,3,6,7,8-H_6CDF	10.7	15.8
2,3,4,6,7,8-H_6CDF	3.3	8.7
1,2,3,7,8,9-H_6CDF	n.d. (2)	n.d. (2)
1,2,3,4,6,7,8-H_7CDF	13.2	29.4
1,2,3,4,7,8,9-H_7CDF	n.d. (3)	n.d. (3)
Toxicity equivalent (NATO)	25.0	28.7
Toxicity equivalent (BgVV)	24.2	29.2

Values are corrected for recovery of ^{13}C-labeled surrogates.
n.d.=not detected. Detection limit, in ppt, given in parentheses.
®Sum of two extractions of each sample.

Table 4 Average Values (µg/kg) from Fly Ash[a] Comparison of Soxhlet vs. ASE (HCl Pretreatment)

Group Totals	Soxhlet (n=1)	ASE[b] (n=2)
Total T_4CDD	12.0	12.4
Total P_5CDD	16.6	20.5
Total H_6CDD	38.2	42.4
Total H_7CDD	15.0	19.8
Total O_8CDD	11.4	12.8
Total T_4CDF	60.5	67.5
Total P_5CDF	83.5	87.3
Total H_6CDF	65.2	73.5
Total H_7CDF	28.1	32.2
Total O_8CDF	13.5	15.8
Congeners	**Soxhlet (n=1)**	**ASE[b] (n=2)**
2,3,7,8-T_4CDD	0.32	0.36
1,2,3,7,8-P_5CDD	1.6	2.1
1,2,3,4,7,8-H_6CDD	1.2	1.4
1,2,3,6,7,8-H_6CDD	2.4	2.7
1,2,3,7,8,9-H_6CDD	2.4	2.3
1,2,3,4,6,7,8-H_7CDD	8.2	9.6
2,3,7,8-T_4CDF	3.7	4.3
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	4.2	4.6
2,3,4,7,8-P_5CDF	5.6	6.6
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	7.8	8.7
1,2,3,6,7,8-H_6CDF	7.2	8.5
2,3,4,6,7,8-H_6CDF	6.6	7.2
1,2,3,7,8,9-H_6CDF	0.43	0.56
1,2,3,4,6,7,8-H_7CDF	18.0	17.6
1,2,3,4,7,8,9-H_7CDF	2.3	2.4
Toxicity equivalent (NATO)	7.6	8.9
Toxicity equivalent (BgVV)	7.3	14.2

Values are corrected for recovery of ^{13}C-labeled surrogates.
[a]Pretreatment with HCl for 30 min followed by water rinse.
[b]Sum of two extractions of each sample.

Table 5 Average Values (µg/kg) from Fly Ash[a] Comparison of Soxhlet (HCl Pretreatment) vs. ASE (No HCl Pretreatment; Toluene/Acetic Acid Solvent)

Group Totals	Soxhlet (n=1)	ASE[b] (n=2)
Total T_4CDD	12.0	10.5
Total P_5CDD	16.6	16.2
Total H_6CDD	38.2	36.7
Total H_7CDD	15.0	16.0
Total O_8CDD	11.4	10.6
Total T_4CDF	60.5	56.1
Total P_5CDF	83.5	77.4
Total H_6CDF	65.2	46.1
Total H_7CDF	28.1	26.5
Total O_8CDF	13.5	13.9
Congeners	**Soxhlet (n=1)**	**ASE[b] (n=2)**
2,3,7,8-T_4CDD	0.32	0.28
1,2,3,7,8-P_5CDD	1.6	1.7
1,2,3,4,7,8-H_6CDD	1.2	1.2
1,2,3,6,7,8-H_6CDD	2.4	2.4
1,2,3,7,8,9-H_6CDD	2.4	2.2
1,2,3,4,6,7,8-H_7CDD	8.2	8.1
2,3,7,8-T_4CDF	3.7	3.4
1,2,3,7,8(+1,2,3,4,8)-P_5CDF	4.2	3.9
2,3,4,7,8-P_5CDF	5.6	5.8
1,2,3,4,7,8(+1,2,3,4,7,9)-H_6CDF	7.8	5.4
1,2,3,6,7,8-H_6CDF	7.2	5.3
2,3,4,6,7,8-H_6CDF	6.6	4.5
1,2,3,7,8,9-H_6CDF	0.43	0.30
1,2,3,4,6,7,8-H_7CDF	18.0	16.8
1,2,3,4,7,8,9-H_7CDF	2.3	2.0
Toxicity equivalent (NATO)	7.6	7.0
Toxicity equivalent (BgVV)	7.3	6.3

Values are corrected for recovery of ^{13}C-labeled surrogates.
[a]Pretreatment with HCl for 30 min followed by water rinse for Soxhlet samples.
[b]No HCl pretreatment, and toluene/acetic acid solvent used for ASE samples.
[c]Sum of two extractions of each sample.

Table 6 Average Values (ng/kg) from EC-2—Comparison of Soxhlet vs. ASE

	Soxhlet Extraction (n=10)			Accelerated Solvent Extraction (n=2)		
Group Totals	**Value**	**% RSD**	**Isomers**	**Value**	**% RSD**	**Isomers**
Total T_4CDD	430	9.7	8	370	1.9	9
Total P_5CDD	300	3.7	11	280	7.7	11
Total H_6CDD	720	5.8	7	690	2.0	7
Total H_7CDD	1300	7.0	2	1300	0.0	2
Total O_8CDD	4000	6.2	1	4200	0.0	1
Total T_4CDF	620	12	17	380	19	19
Total P_5CDF	820	9.4	14	710	7.0	17
Total H_6CDF	1900	5.7	12	1900	0.0	13
Total H_7CDF	3800	8.2	4	3900	3.6	4
Total O_8CDF	7800	8.3	1	7000	3.1	1
Congeners	**Value**	**% RSD**	**% Recovery[a]**	**Value**	**% RSD**	**% Recovery[a]**
2,3,7,8-T_4CDD	270	9.1	68	270	0.0	72
1,2,3,7,8-P_5CDD	24	12	74	22	3.3	81
1,2,3,4,7,8-H_6CDD	23	8.3	76	24	3.0	80
1,2,3,6,7,8-H_6CDD	83	3.6	78	87	0.8	54
1,2,3,7,8,9-H_6CDD	60	6.2	77	57	7.4	67
1,2,3,4,6,7,8-H_7CDD	720	6.7	81	720	1.0	79
2,3,7,8-T_4CDF[b]	100	7.3	68	82	2.6	70
1,2,3,7,8-P_5CDF	39	14	74	36	3.9	76
2,3,4,7,8-P_5CDF	62	5.5	79	60	0.0	75
1,2,3,4,7,8-H_6CDF	740	5.3	81	690	0.0	70
1,2,3,6,7,8-H_6CDF	120	6.2	81	120	0.0	50
2,3,4,6,7,8-H_6CDF	45	9.0	82	60	1.2	69
1,2,3,7,8,9-H_6CDF	4.9	31	84	5.3	15	70
1,2,3,4,6,7,8-H_7CDF	2600	6.7	85	2500	0.0	74
1,2,3,4,7,8,9-H_7CDF	160	5.5	83	160	0.0	72

Values are corrected for recovery of ^{13}C-labeled surrogates.
[a]Refers to recovery of corresponding ^{13}C-labeled surrogate.
[b]Maximum possible concentration due to potential chromatographic overlap.

Table 7 Average Values (ng/kg) from HS-2—Comparison of Soxhlet vs. ASE

	Soxhlet Extraction (n=4)			Accelerated Solvent Extraction (n=2)		
Group Totals	**Value**	**% RSD**	**Isomers**	**Value**	**% RSD**	**Isomers**
Total T_4CDD	3.9	14	2	2.5	34	5
Total P_5CDD	17	7.8	6	10	10	9
Total H_6CDD	510	5.6	8	570	1.3	7
Total H_7CDD	4700	8.3	2	5100	11	2
Total O_8CDD	6500	4.2	1	7100	0.0	1
Total T_4CDF	39	11	13	24	3.0	14
Total P_5CDF	33	13	8	28	0.0	11
Total H_6CDF	89	3.2	6	87	12	10
Total H_7CDF	293	3.3	4	310	0.0	4
Total O_8CDF	300	3.8	1	280	2.6	1
Congeners	**Value**	**% RSD**	**% Recovery[a]**	**Value**	**% RSD**	**% Recovery[a]**
2,3,7,8-T_4CDD	n.d. (1)		62	n.d. (1)		71
1,2,3,7,8-P_5CDD	1.6	4.6	69	n.d. (1)		75
1,2,3,4,7,8-H_6CDD	4.5	4.8	74	5.2	11	73
1,2,3,6,7,8-H_6CDD	19	4.3	75	21	0.0	50
1,2,3,7,8,9-H_6CDD	24	4.3	74	28	2.6	61
1,2,3,4,6,7,8-H_7CDD	1200	8.1	80	1300	0.0	93
2,3,7,8-T_4CDF[b]	8.5	11	62	6.6	5.4	65
1,2,3,7,8-P_5CDF	1.9	17	68	2.0	0.0	72
2,3,4,7,8-P_5CDF	3.7	7.9	71	3.7	3.8	59
1,2,3,4,7,8-H_6CDF	17	7.3	79	17	4.3	70
1,2,3,6,7,8-H_6CDF	3.7	5.6	80	4.0	5.4	49
2,3,4,6,7,8-H_6CDF	3.7	18	81	4.4	3.2	61
1,2,3,7,8,9-H_6CDF	n.d. (1)		83	n.d. (1)	0.0	75
1,2,3,4,6,7,8-H_7CDF	91	1.6	83	96	3.7	82
1,2,3,4,7,8,9-H_7CDF	5.2	6.7	84	5.3	6.7	83

n.d.=not detected. Detection limit, in ppt, given in parentheses. Values not used for statistical calculations.
Values are corrected for recovery of ^{13}C-labeled surrogates.
[a]Refers to recovery of corresponding ^{13}C-labeled surrogate.
[b]Maximum possible concentration due to potential chromatographic overlap.

Table 8 Average Values (ng/kg) from Highly Contaminated Sources—Comparison of Soxhlet vs. ASE

	Hamilton Harbor		Parrots Bay	
Group Totals	**Soxhlet**	**ASE**	**Soxhlet**	**ASE**
Total T_4CDD	50[6]	14[2]	39[3]	48[5]
Total P_5CDD	63[12]	15[5]	87[10]	66[10]
Total H_6CDD	220[7]	180[7]	230[6]	200[7]
Total H_7CDD	850[2]	810[2]	580[2]	530[2]
Total O_8CDD	3100	3100	1900	1600
Total T_4CDF	370[17]	130[11a]	400[14]	270[10]
Total P_5CDF	290[13]	110[11a]	180[8]	170[13]
Total H_6CDF	240[14]	160[11]	230[6]	230[10]
Total H_7CDF	350[4]	290[4]	400[4]	360[4]
Total O_8CDF	270	210	510	370
Congeners				
2,3,7,8-T_4CDD	3.7	3.1	19	19
1,2,3,7,8-P_5CDD	5.1	5.4	8.3	6.0
1,2,3,4,7,8-H_6CDD	6.4	7.2	8.6	6.7
1,2,3,6,7,8-H_6CDD	27	26	26	17
1,2,3,7,8,9-H_6CDD	20	28	24	18
1,2,3,4,6,7,8-H_7CDD	460	430	280	250
2,3,7,8-T_4CDF[b]	61	44[a]	80	48
1,2,3,7,8-P_5CDF	14	14	n.d. (20)	9.8
2,3,4,7,8-P_5CDF	26	25[a]	22	14
1,2,3,4,7,8-H_6CDF	27	37	79	59
1,2,3,6,7,8-H_6CDF	17	16	n.d. (20)	15
2,3,4,6,7,8-H_6CDF	14	14	21	11
1,2,3,7,8,9-H_6CDF	n.d. (2)	1.6	4.9	n.d. (1)
1,2,3,4,6,7,8-H_7CDF	130	130	270	220
1,2,3,4,7,8,9-H_7CDF	14	13	17	12

Values are corrected for recovery of isotopically labeled surrogate standards.
n.d.=not detected. Detection limit, in ppt, given in parentheses.
Superscripts indicate the number of isomers detected.
[a]Recoveries outside the range 25% to 150%; results are not to be used for regulatory compliance purposes.
[b]Maximum possible concentration due to potential chromatographic overlap.