HJ 1242 2022

6 -

Water quality Determination of 6 phthalate esters (PAEs) Liquid chromatography-triple quadrupole mass spectrometry

2022-04-15 2022-11-01

生 态 环 境 部

..... ii

1 1

2 1

3 1

4 1

5 2

6 3

7 3

8 4

9 7

10 8

11 10

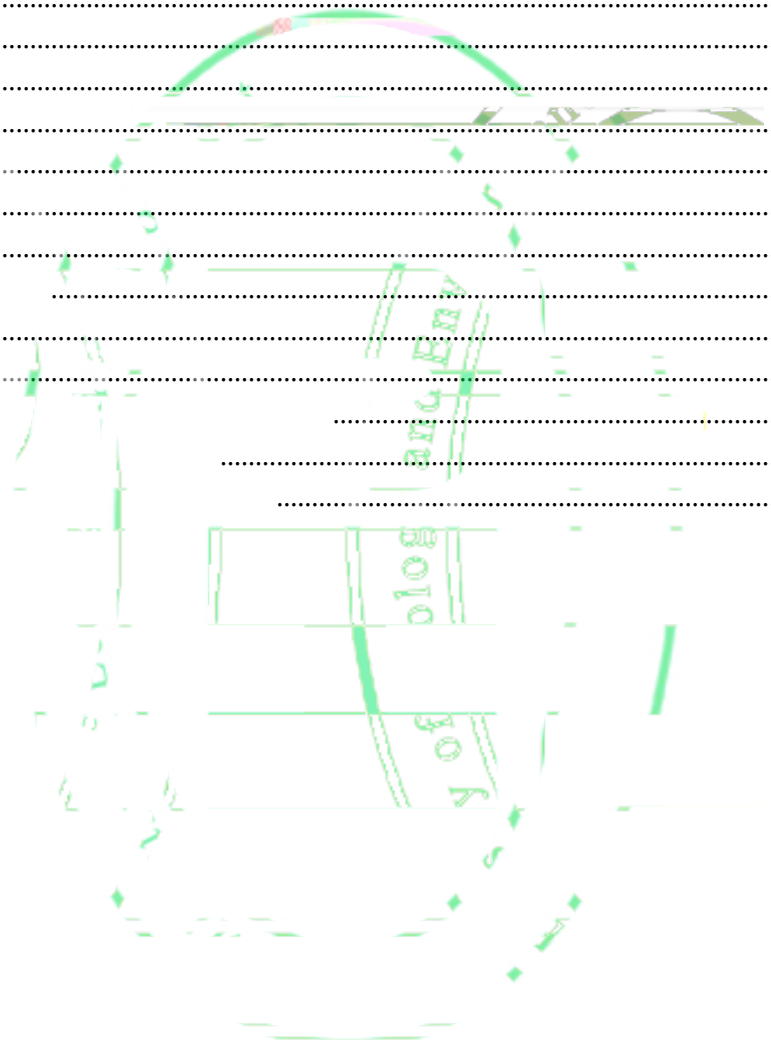
12 10

13 10

A 11

B 12

C 18



6

-

A B C

2022 4 15

2022 11 1

6

-

6 -

DMP

DEP BBP DBP 2-

DEHP DNOP 6

10.0 ml 5.0 ml 6

A

DBP DEHP 100 ml

2.0 ml DBP DEHP

A

GB 17378.3 3

GB 21749

HJ 91.1

HJ 91.2

HJ 164

HJ 442.3

-

DBP DIBP

DIBP

CH_3CN

CH_3OH

CH_3COCH_3

C_6H_{14}

$(CH_3)_2CHOH$

$HCOOH$

NaOH

HCl ρ=1.19 g/ml

NaCl

400 4 h

2

2 ml 5.6 1 L

ρ(NaOH) 0.4 g/ml

40 g 5.7 100 ml

5.8 1 1

ρ=100 mg/L 1000 mg/L

2-

ρ=20.0 mg/L

5.13 5.3 4

2

ρ=2.00 mg/L

5.14 5.3 4

2

ρ=100 mg/L 1000 mg/L

-d_4 DMP-d_4 -d_4 DEP-d_4

-d_4 BBP-d_4 -d_4 DBP-d_4 2-

-d_4 DEHP-d_4 -d_4 DNOP-d_4

ρ=10.0 mg/L

5.16 5.3 4

2

0.22 m

250 ml 500 ml 1000 ml

- ESI

50 mm 2.1 mm C_{18}

50 mm 2.1 mm C_{18}

0 3000 r/min

0 2500 r/min

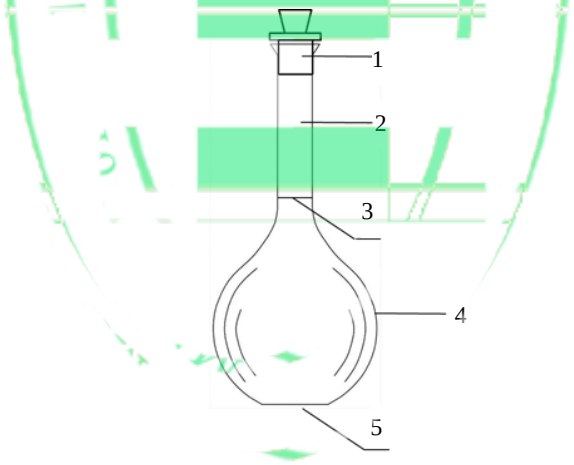
2.5 cm 3.0 cm

2 ml

100 ml

0.5 ml 12 ml 1

GB 21749



1 2 3 100 ml 0.5 ml 4 5

GB 17378.3 HJ 91.1 HJ 91.2 HJ 164 HJ 442.3

6.1 pH 5 7 5.11 5.12

pH 5 7 6.1 4 5 d
4 14 d
1 L
6.1 6.1

10.0 ml 7.1 25 ml 5.17
4 g 5.9 5.00 ml 5.1 1 min 5 min
6.8 1.0 ml 5.18

100 ml 7.1 6.11 5.17
5 g 5.9

ESI

MRM

a 5500 V

b 550

c 3.45 10^5 Pa 50 psi

d 3.45 10^5 Pa 50 psi

e 1.72 10^5 Pa 25 psi

f 4.14 10^4 Pa 6 psi

g 2

				V	V	s	
1		195.3	163.0*	40	12	0.03	-d_4
		195.3	77.1	40	46	0.03	
2		223.1	177.4*	50	12	0.03	-d_4
		223.1	149.3	50	25	0.03	
3		313.3	91.3*	68	27	0.03	
		313.3	205.2	68	12	0.03	-d_4
4		279.1	149.3*	72	20	0.03	-d_4
		279.1	205.2	72	12	0.03	
5	2-	391.1	167.0*	84	18	0.03	2-
		391.1	149.0	84	32	0.03	-d_4
6		391.3	261.1*	60	10	0.03	-d_4
		391.3	149.0	60	20	0.03	
7	-d_4	199.2	167.2*	60	14	0.03	
8	-d_4	227.2	181.2*	50	12	0.03	
9	-d_4	317.3					

6.9 5.15

5.17 5.1 5

1

2- 50.0

100

6.9 5.15

5.17 5.4 5

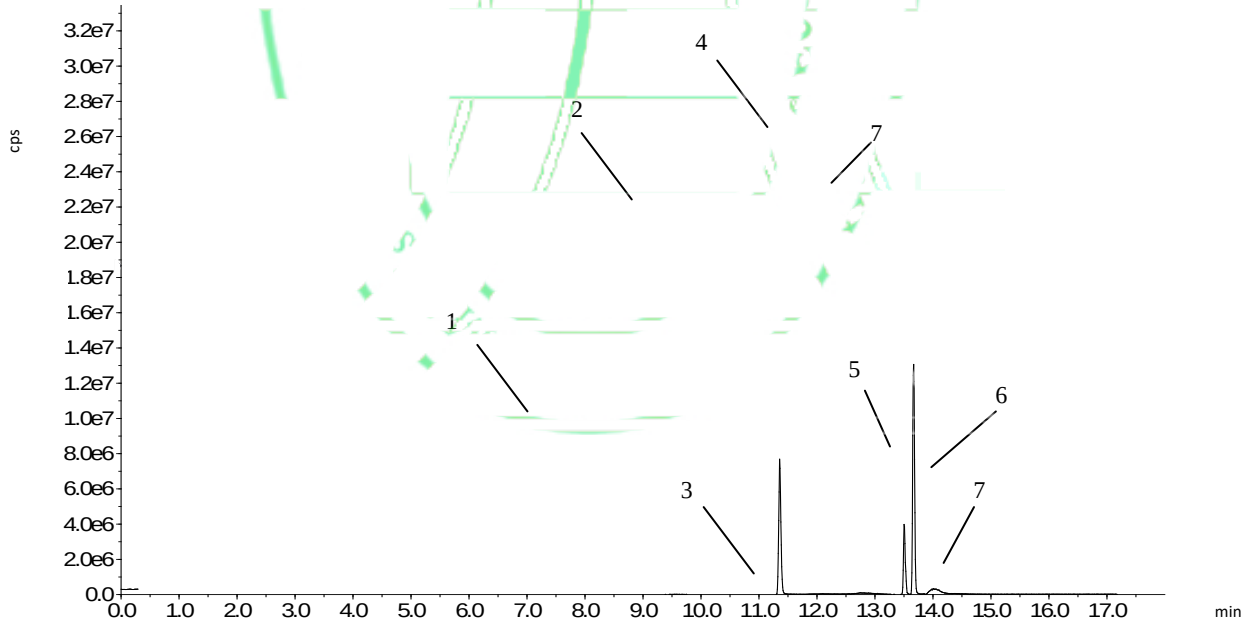
300 2-

500

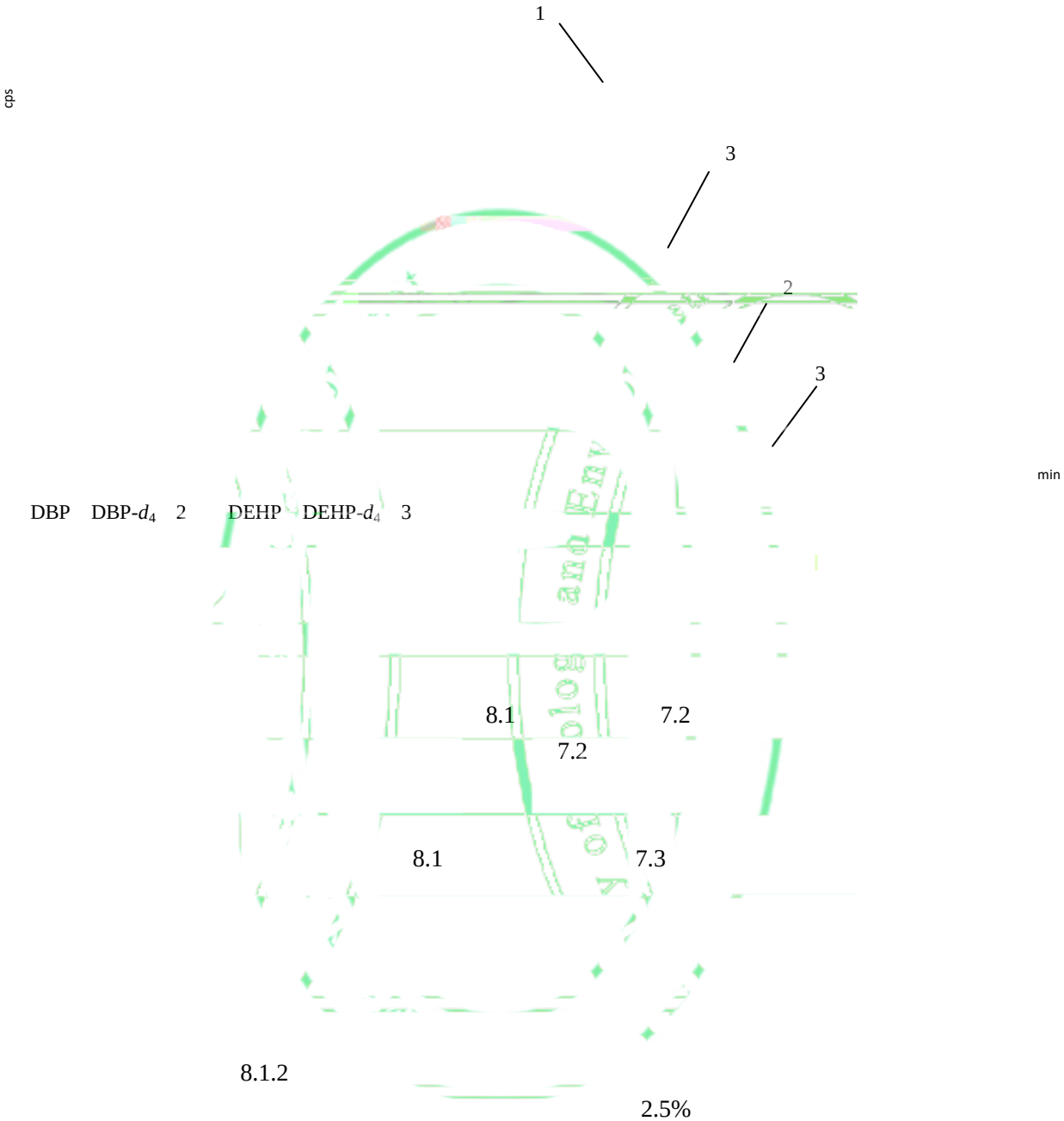
200

6 2

2 3



1 DMP DMP-d_4 2 DEP DEP-d_4 3 BBP BBP-d_4 4 DBP DBP-d_4 5 DEHP DEHP-d_4
6 DNOP DNOP-d_4 7



1 DBP DBP-d_4 2 DEHP DEHP-d_4 3

8.1 7.2

7.2

8.1 7.3

8.1.2

2.5%

K_{sam} K_{std}

3 K_{sam}

1

$$K_{sam} = \frac{A_2}{A_1} \times 100\% \quad (1)$$

K_{sam} %

A_2

A_1

K_{std} 2

$$K_{std} = \frac{A_{std2}}{A_{std1}} \times 100\% \qquad 2$$

K_{std} %

A_{std2}

A_{std1}

K_{std} %	K_{sam} %
K_{std} 50	20
20 K_{std} 50	25
10 K_{std} 20	30
K_{std} 10	50

3

$$\rho_i = \frac{\rho_I \times V_1}{V} \times D \qquad 3$$

ρ_i i

ρ_I i

V_1 ml

V ml

D

3

6

4.0 40.0 120 2-

40.0 80.0 120 6

4.5% 14% 3.1% 11% 2.5% 7.9% 9.6% 16%

5.5% 9.6% 4.3% 8.5% 0.9 11 7.9 14 14 18

1.5 19 9.8 20 18 31

6

103% 19% 108% 19% 99% 13% 100% 12% 98% 11% 98% 13%
6 2-
6
85% 116% 85% 129%
77% 122% 75% 130%
B

20 20 / 1

0.995

20 20 / 1
20%

20 20 / 1
30%

20 20 / 1 70%
130%

20%
3 5
3 5
3 5

A.1 10 ml
100 ml

			CAS No.				
						/L	
1		DMP	131-11-3	0.9	3.6		
2		DEP	84-66-2	1	4		
3		BBP	85-68-7				

B.1

				%	%		
1		4.00	4.1	4.5 11	9.9	1.0	1.5
		40.0	38.1	4.8 11	8.7	9.1	12
		120	117	2.9 5.7	8.5	16	31
2		4.00	3.8	6.1 12	16	1	2
		40.0	37.5	6.2 9.7	9.6	9	13
		120	110	2.8 5.8	4.3	14	18
3		4.00	4.1	5.5 12	12	1.0	1.6

B.2

				%
1			5.00	5.0 15
			50.0	2.7 6.9
			5.00	6.3 9.2
			50.0	4.3 11
			5.00	3.9 8.7
			80.0	1.3 8.3
			5.00	4.1 15
			80.0	3.7 6.4
2			5.00	9.0 14
			50.0	3.6 7.1
			5.00	3.5 11
			50.0	3.4 13
			5.00	3.9 9.6
			80.0	2.7 6.5
			5.00	2.9 11
			80.0	2.2 10
3			5.00	2.0 8.6
			50.0	5.7 14
			5.00	2.8 13
			50.0	4.9 10
			5.00	3.4 10
			80.0	2.8 6.9
			5.00	3.0 13
			80.0	2.2 7.2
4			50.0	3.7 10
			50.0	7.8 11
			80.0	1.5 7.5
			80.0	2.8 12
5	2-		50.0	2.3 15
			50.0	8.5 13
			80.0	2.7 13
			80.0	2.0 15
6			5.00	4.6 16
			50.0	5.4 15
			5.00	5.5 8.5
			50.0	1.3 10
			5.00	3.7 14
			80.0	4.3 14
			5.00	2.6 10
			80.0	3.0 6.6

B.3

				%	%
1		4.00	4.0	90 115	102 21
		40.0	37.7	86 108	95 17
		120	117	87 109	98 17

2

B.4

					%
1			ND	5.00	83 114
				50.0	87 113
			ND	5.00	83 117
				50.0	95 114
			ND	5.00	78 121
				80.0	88 105
			ND	5.00	79 111
				80.0	87 100
2			ND 1.1	5.00	97 125
				50.0	92 110
			ND	5.00	78 114
				50.0	88 108
			ND 0.8	5.00	77 116
				80.0	93 112
			ND 1.2	5.00	82 109
				80.0	94 106
3			ND	5.00	106 119
				50.0	99 114
			ND	5.00	92 109
				50.0	90 109
			ND	5.00	73 114
				80.0	91 104
			ND 1.0	5.00	81 98
				80.0	86 111
4			ND	50.0	89 111
			ND	50.0	92 107
			ND 9	80.0	89 109
			ND 9	80.0	85 112
5	2-		ND	50.0	81 111
			ND	50.0	83 107
			ND 10	80.0	86 103
			ND 13	80.0	84 111
6			ND	5.00	94 125
				50.0	88 110
			ND	5.00	98 119
				50.0	88 114
			ND 0.8	5.00	84 111
				80.0	91 110
			ND	5.00	79 103
				80.0	82 109
	ND				

B.5

				%	%		
1		1.60	1.7	7.1 11	9.1	0.4	0.6
		3.20	3.2	3.7 7.1	5.7	0.5	0.7
		4.80	4.7	2.5 5.3	5.3	0.5	0.9
2	2-	1.60	1.7	6.4 13	8.8	0.4	0.6
		4.80	4.8	2.8 7.0	6.5	0.7	1.1
		8.00	7.8	2.5 5.8	6.4	0.8	1.6

B.6

				%
1			1.6	4.9 15
			4.0	3.0 14
			1.6	5.2 13
			4.0	3.4 9.9
			1.6	2.9 15

2 2-

B.8

				L	%
1			ND 0.5	1.6	85 116
				4.0	85 113
			ND 0.4	1.6	77 115
				4.0	75 104
2	2-		ND 0.9	1.6	98 111
				8.0	92 129
			ND 0.6	1.6	92 122
				8.0	95 130
ND					

C.1