

Preparation of a National Reference Material Organochlorine Pesticide Mixture for Residue Analysis

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Table S1. Some technical information of reference materials

No	Name of Compound	CAS	Product Code	Using
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		No		Solvent
1	4,4'-DDT	50-29-3	DRE-C12082000	AL
2	2,4'-DDT	789-02-6	N-12708-50MG	AL
3	4,4'-DDD (4,4-TDE)	72-54-8	N-10874-250MG	AL
4	Aldrin (HHDN)	309-00-2	N-11049-250MG	AL
5	Dieldrin	60-57-1	N-11688-100MG	AL
6	<i>alpha</i> -endosulfan	959-98-8	N-10979-100MG	AL
7	<i>beta</i> -endosulfan	33213-65-9	DRE-C13122000	AL
8	Endrin	72-20-8	N-11854-100MG	AL
9	<i>alpha</i> -HCH (Alpha BHC)	319-84-6	N-11194-100MG	AL
10	<i>gamma</i> -HCH (Lindane, Gamma BHC)	58-89-9	DRE-C14073000	AL
11	Heptachlor	76-44-8	N-12147-100MG	AL
12	Heptachlor- <i>endo</i> -epoxide (Isomer A) (Trans-Heptachloroepoxide)	28044-83-9	N-13618-50MG	ME

Table S2. The concentration of individual solutions

Name	GRAVIMETRIC (Mix A and B)	VOLUMETRIC (Mix C)
	Concentration (ppm)	Concentration (ppm)
HCH-alpha (Alpha BHC)	17682	14148
HCH-gamma (Lindane, Gamma BHC)	10880	8646
Aldrin	11858	9320
Dieldrin	17682	14148
Endrin	14778	11790
4,4-DDD (4,4-TDE)	9411	7467
4,4-DDT	17682	14148
2,4-DDT	12833	10218
Endosulfan-alpha	19608	15720
Endosulfan-beta	16716	13362
Heptachlor	17682	14148
Heptachlor Endoepoxide (Isomer A) (Trans-Heptachloroepoxide)	17682	-

Table S3. Operation Parameters of Gas Chromatography – Mass Spectrometry

GC									
Program: Column Oven Temperature						Column:			
		Final Temperature							
Column Oven Temperature	70°C	Rate		(°C)	Hold Time (min)	Name	HP 5 MS	Thickness	0.25 µm
Injection Temperature	240°C	0	-	70.0	2	Length	30 m	Diameter	0.25 mm
Injection Mode	Splitless	1	15	160.0	0				
Sampling Time	1.00 min	2	2	200.0	0				
Carrier Gas	Helium	3	10	250.0	0				
	Linear								
Flow Control Mode	Velocity	4	20	280.0	4				
Column Flow	1.00 mL/min								
Linear Velocity	36.7 cm/sec								
Purge Flow	3.0 mL/min								
MS									
Ion Source Temperature	230°C								
Interface Temperature	280°C								
Solvent Cut Time	6 min								

Table S4. The results of ANOVA

Name	F	F _{criteria}
<i>alpha</i> -HCH	4.29	5.32
<i>gamma</i> -HCH	0.18	5.32
Aldrin	0.46	5.32
Dieldrin	0.96	5.32
Endrin	0.20	5.32
4,4'-DDD	0.16	5.32
4,4'-DDT	0.81	5.32
2,4'-DDT	0.76	5.32
<i>alpha</i> -endosulfan	2.84	5.32
<i>beta</i> -endosulfan	0.93	5.32
Heptachlor	0.08	5.32
Heptachlor- <i>endo</i> -epoxide	2.01	5.32

Table S5. The results of homogeneity testing for mix A by gravimetrically

Name	SS _{within}	SS _{between}	MS _{within}	MS _{between}	u _{bb}	S _{bb}
<i>alpha</i> -HCH	0.005	0.003	0.001	0.003	0.009	0.02
<i>gamma</i> -HCH	0.004	0.000	0.001	0.000	0.009	-
Aldrin	0.005	0.000	0.001	0.000	0.009	-
Dieldrin	0.007	0.001	0.001	0.001	0.011	-
Endrin	0.007	0.000	0.001	0.000	0.011	-
4,4'-DDD	0.024	0.000	0.003	0.000	0.020	-
4,4'-DDT	0.032	0.003	0.004	0.003	0.024	-
2,4'-DDT	0.018	0.002	0.002	0.002	0.018	-
<i>alpha</i> -endosulfan	0.006	0.002	0.001	0.002	0.010	0.02
<i>beta</i> -endosulfan	0.005	0.001	0.001	0.001	0.009	-
Heptachlor	0.022	0.000	0.003	0.000	0.020	-
Heptachlor- <i>endo</i> -epoxide	0.004	0.001	0.000	0.001	0.008	0.01

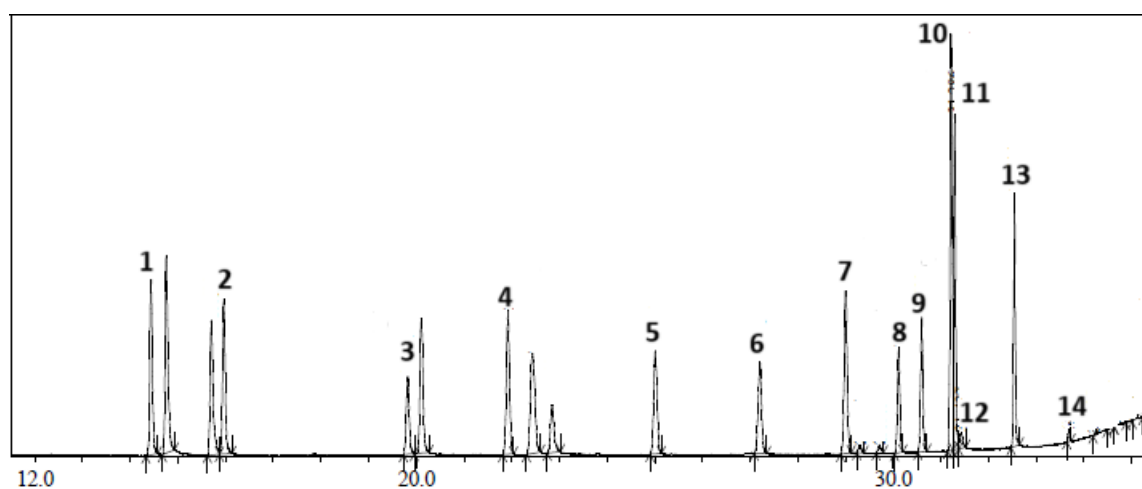


Fig. S1 The chromatogram of 2 ppm calibration standard (Peak 1: *alpha*-HCH, Peak 2: *gamma*-HCH, Peak 3: heptachlor, Peak 4: aldrin, Peak 5: heptachlor-*endo*-epoxide, Peak 6: *alpha*-endosulfan, Peak 7: dieldrin, Peak 8: endrin, Peak 9: *beta*-endosulfan, Peak 10: 4,4'-DDD, Peak 11: 2,4'-DDT, Peak 12: endrin aldehyde, Peak 13: 4,4'-DDT, Peak 14: endrin ketone)

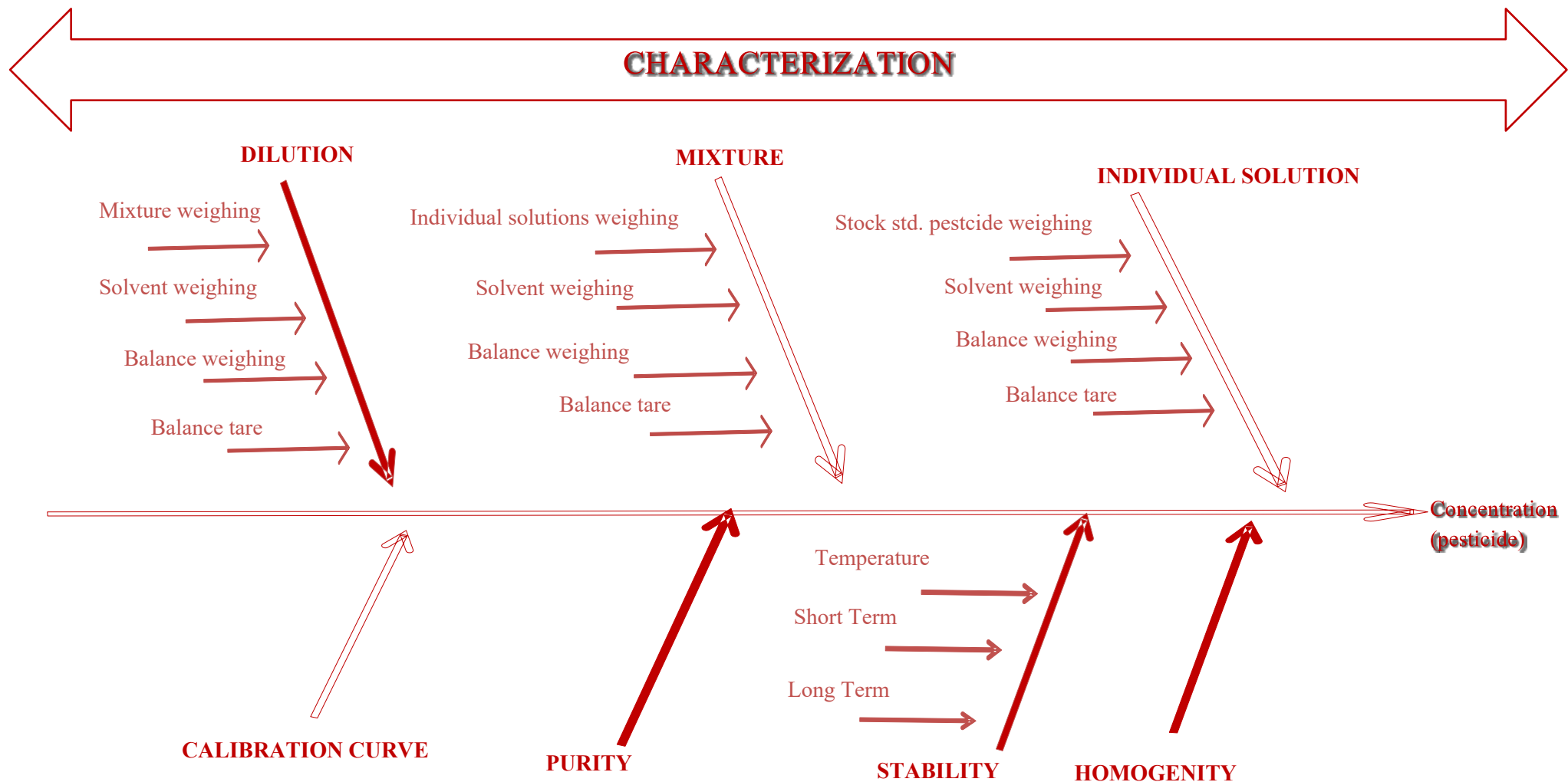


Fig. S2 Fish bone diagram of preparing CRM gravimetrically

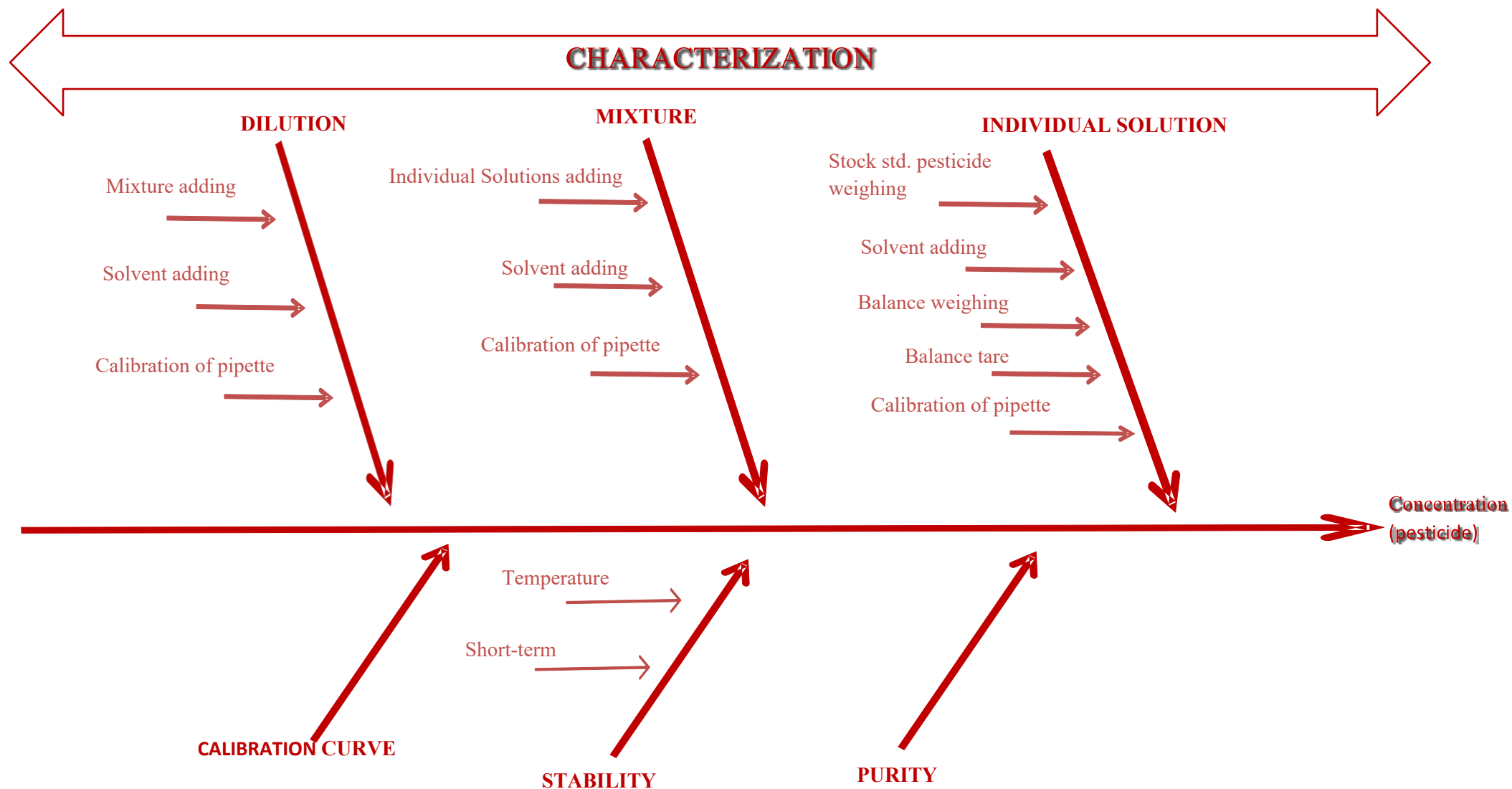
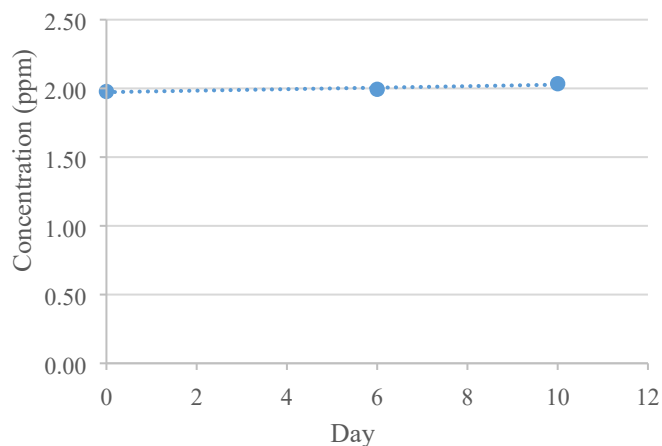
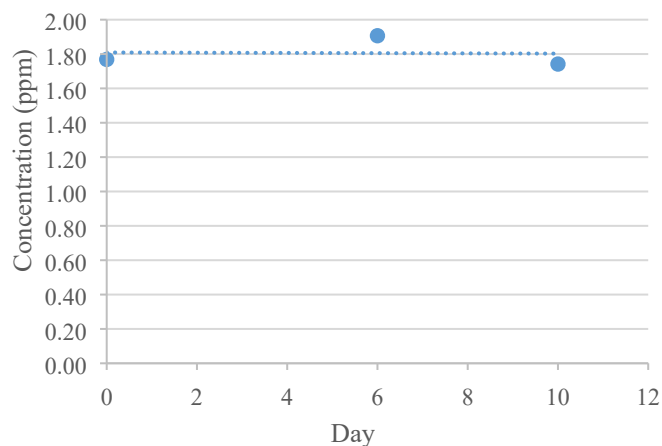


Fig. S3 Fish bone diagram of preparing CRM volumetrically

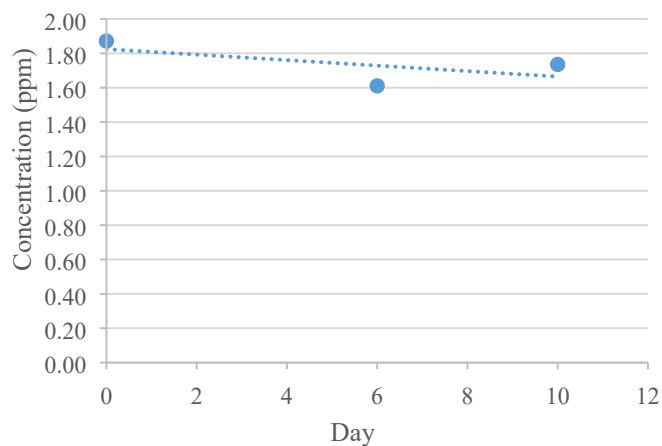
Stability of alpha-HCH in mix C
at -20°C for short-term



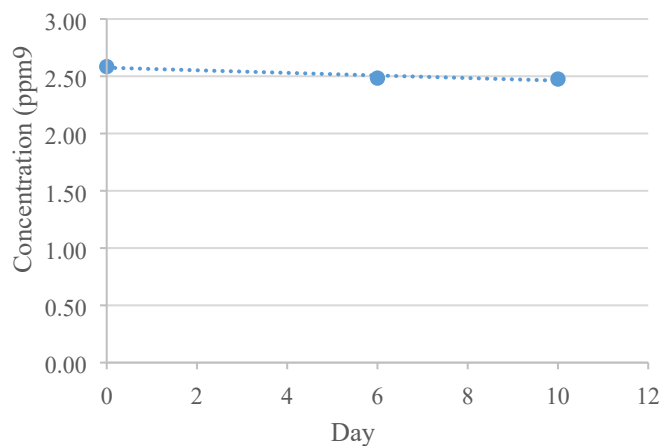
Stability of gamma-HCH in mix C
at -20°C for short-term



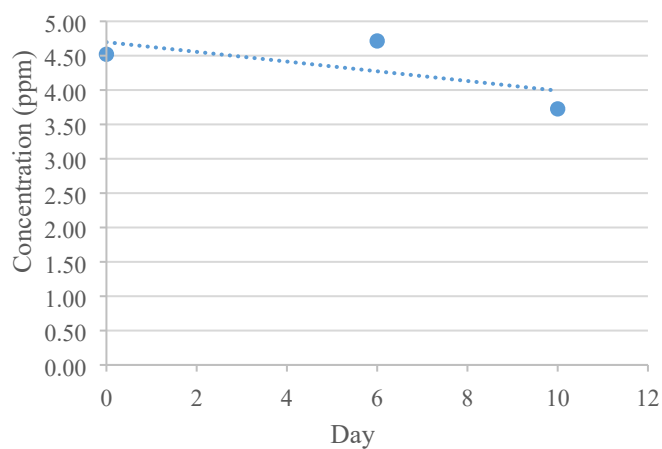
Stability of aldrin in mix C
at -20°C for short-term



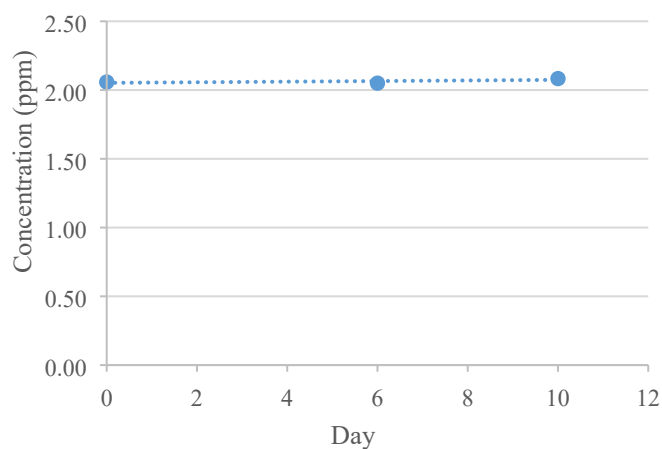
Stability of dieldrin in mix C
at -20°C for short-term



Stability of endrin in mix C at
-20°C for short-term



Stability of 4,4'-DDD in mix C
at -20°C for short-term



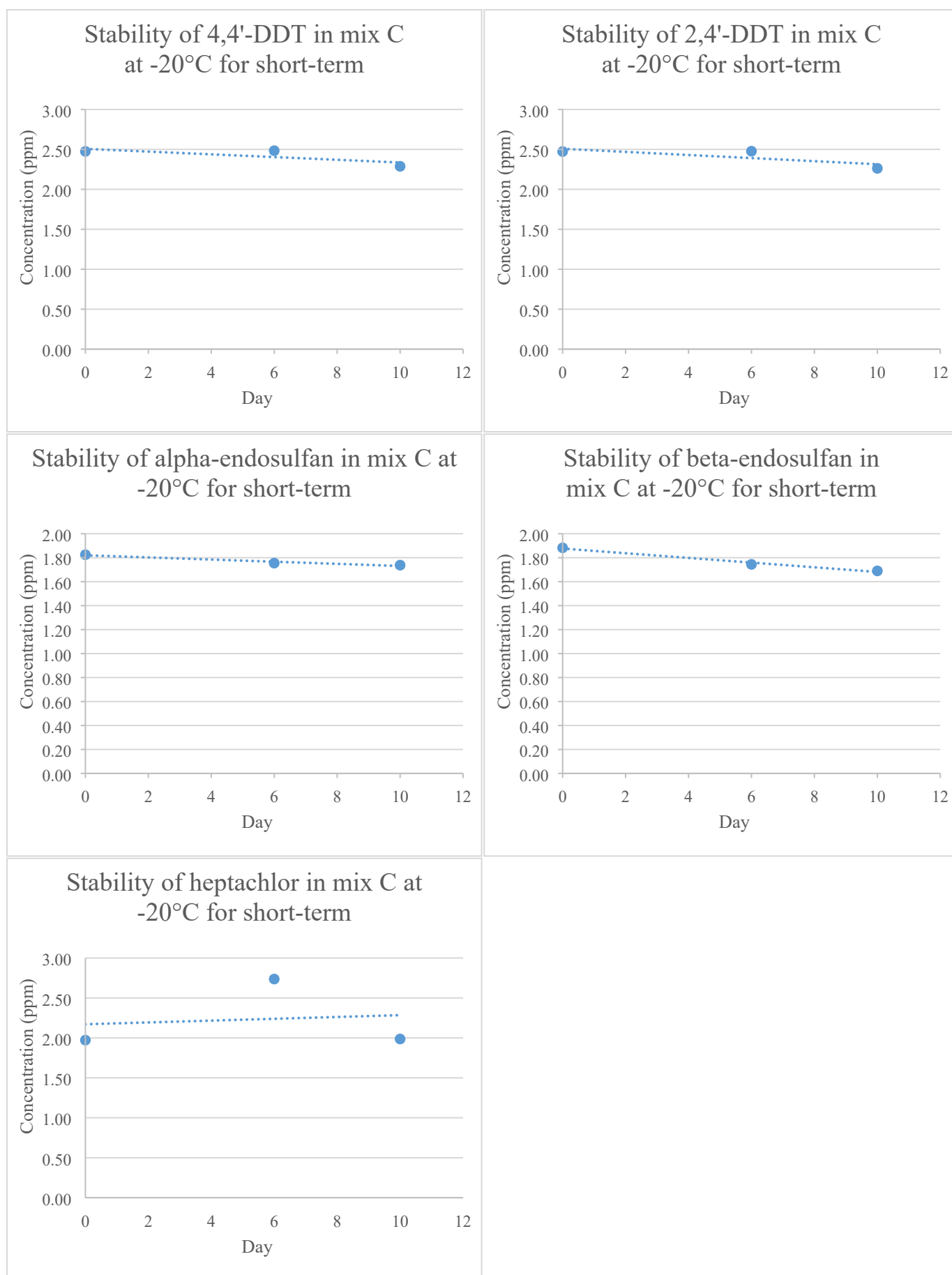
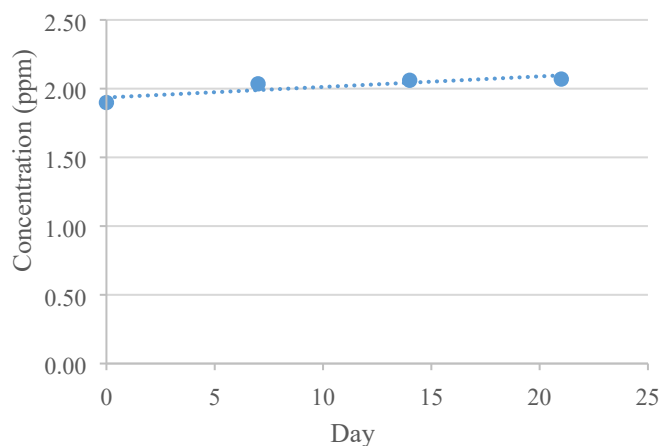
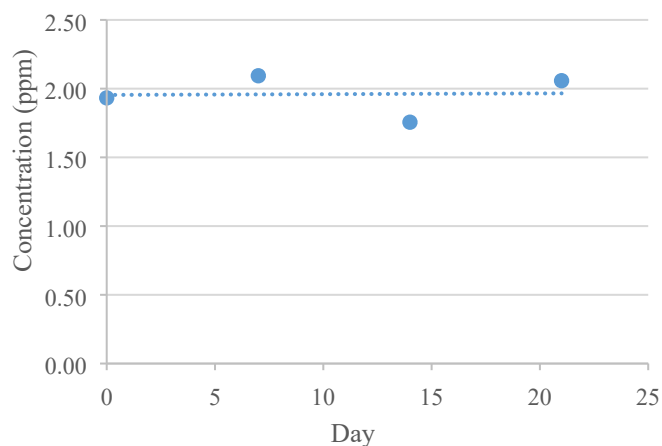


Fig. S4 The plots of volumetric mix C in short-term stability at -20°C for each pesticide

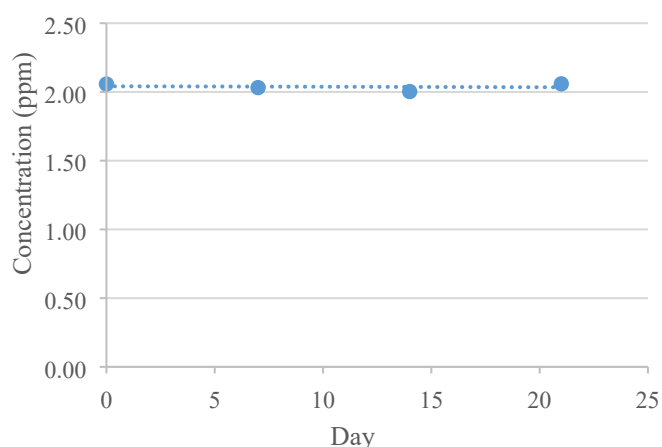
Stability of alpha-HCH in first bottle of mix A at -20°C for short-term



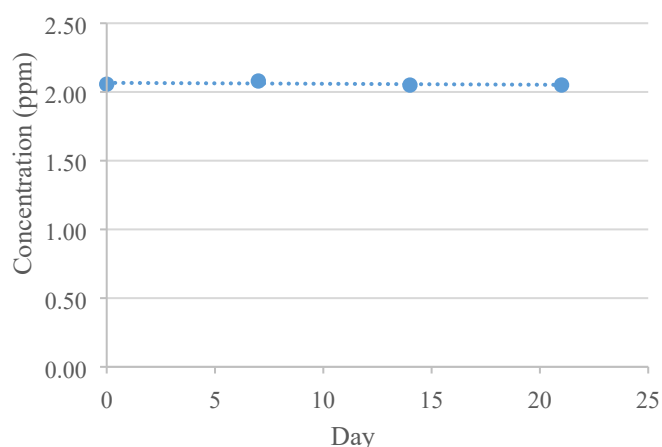
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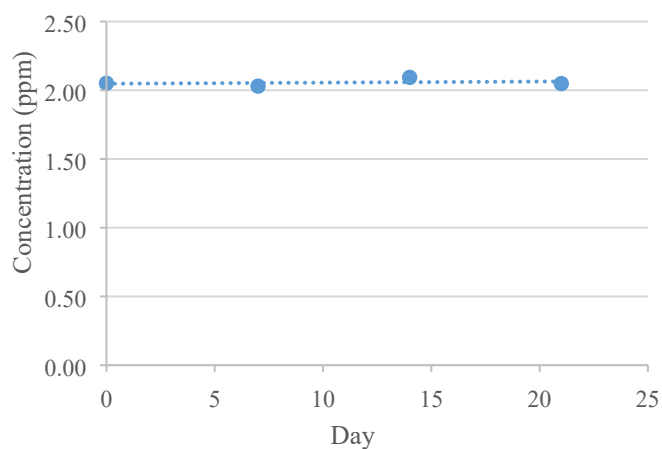
Stability of gamma-HCH in first bottle of mix A at -20°C for short-term



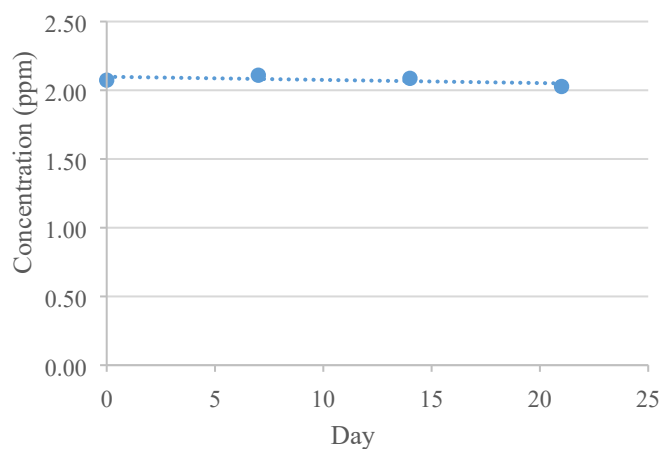
Stability of gamma-HCH in second bottle of mix A at -20°C for short-term



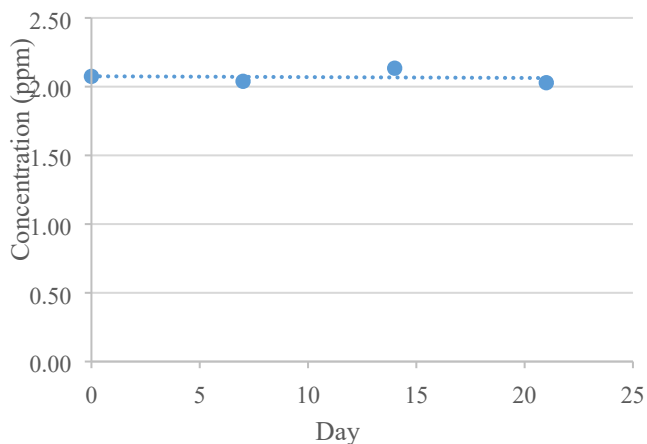
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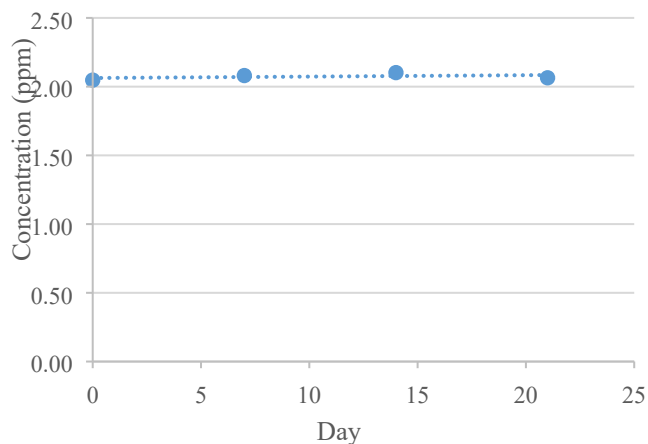
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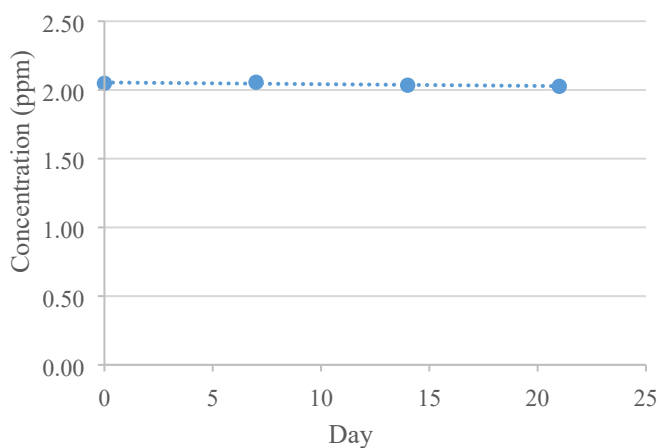
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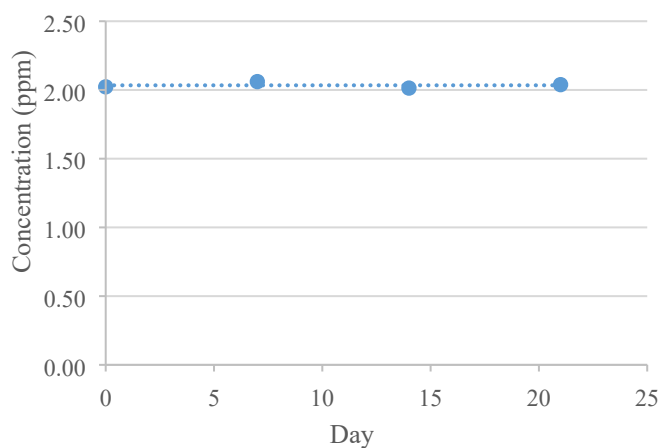
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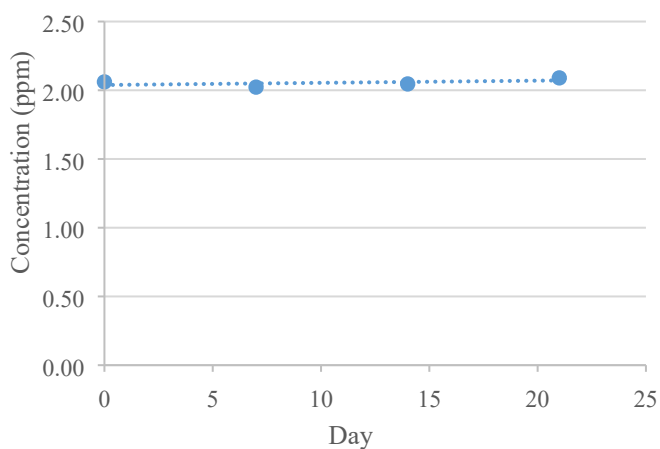
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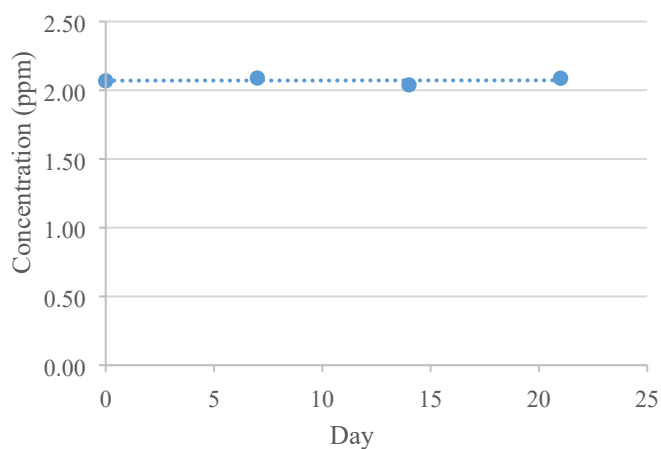
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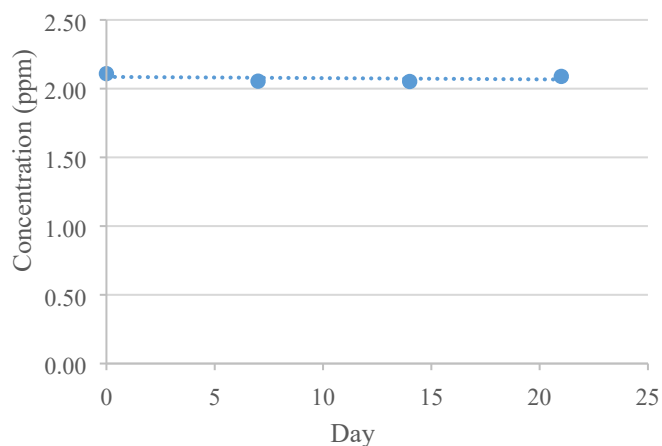
Stability of 4,4'-DDD in first bottle of mix A at -20°C for short-term



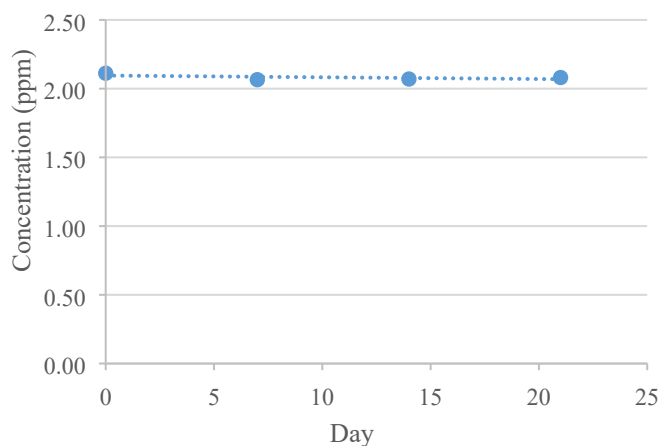
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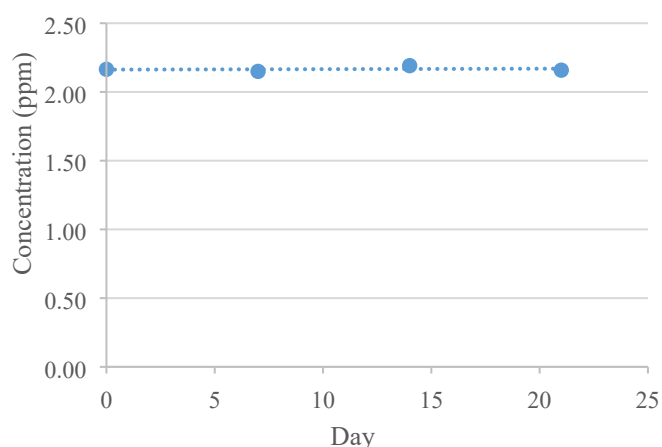
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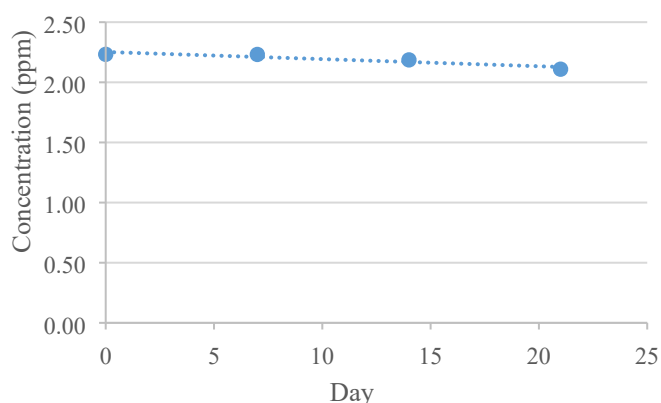
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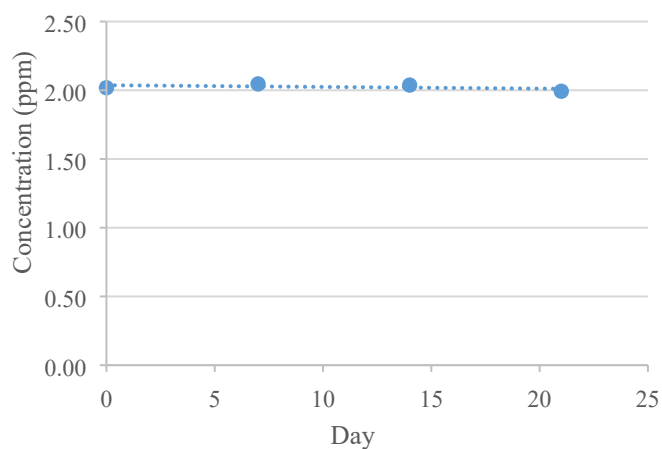
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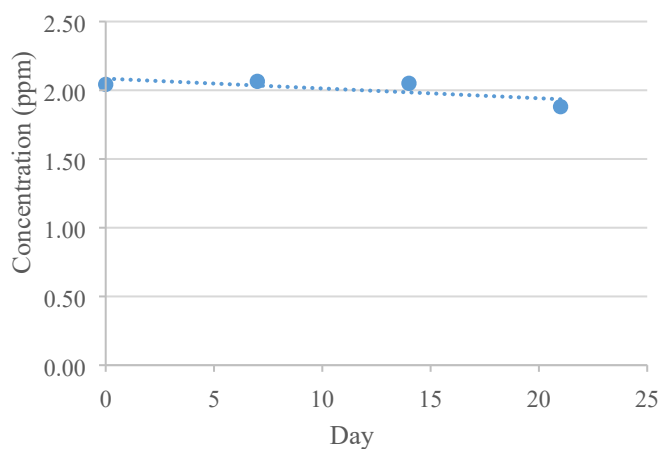
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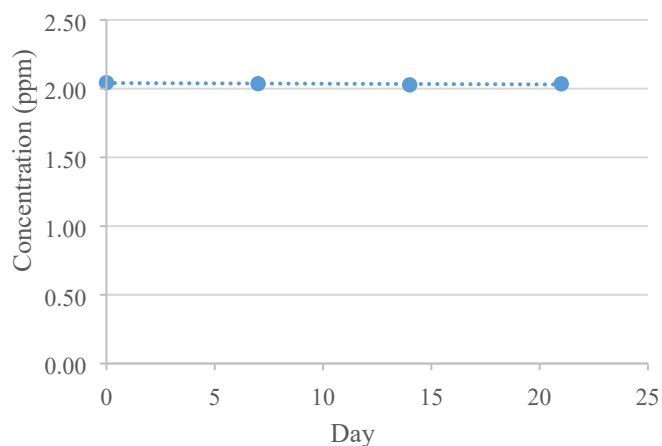
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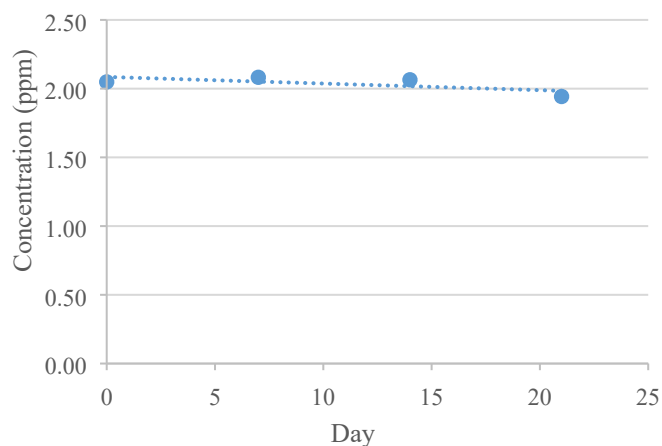
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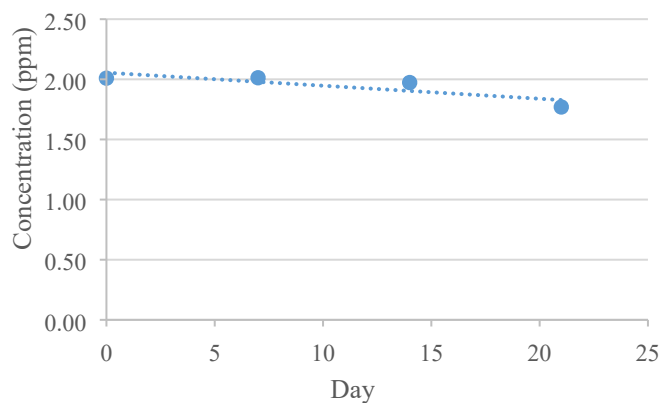
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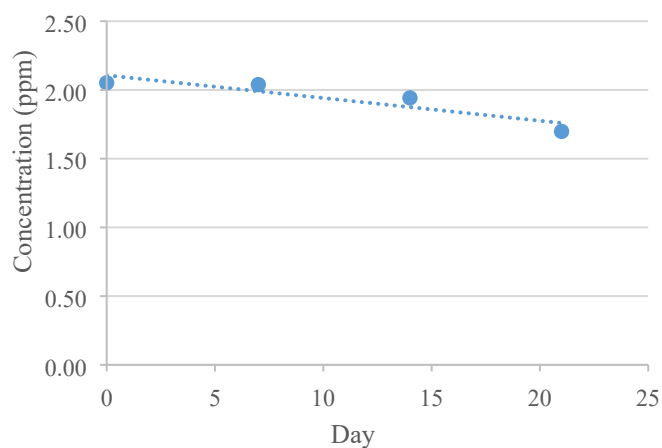
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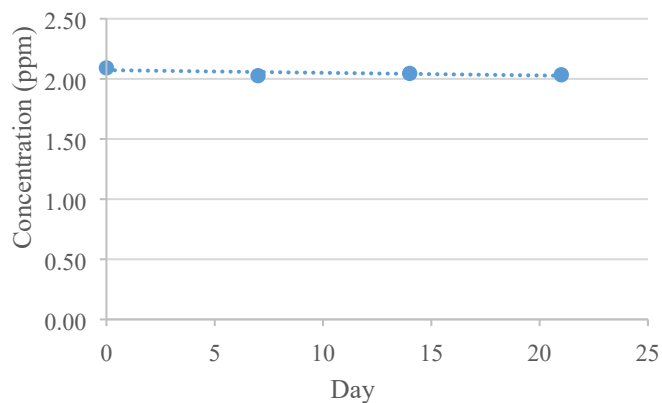
Stability of heptachlor in first bottle of mix A at -20°C for short-term



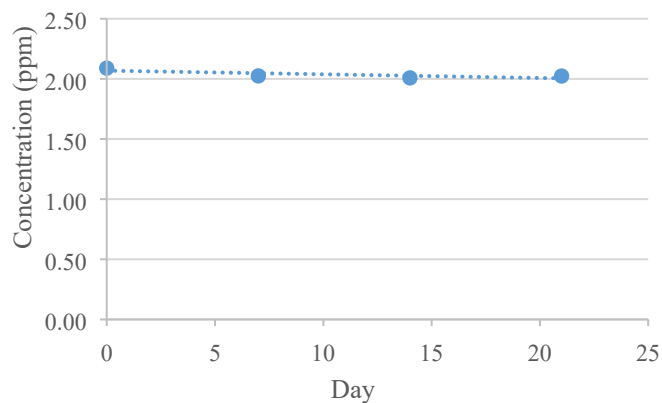
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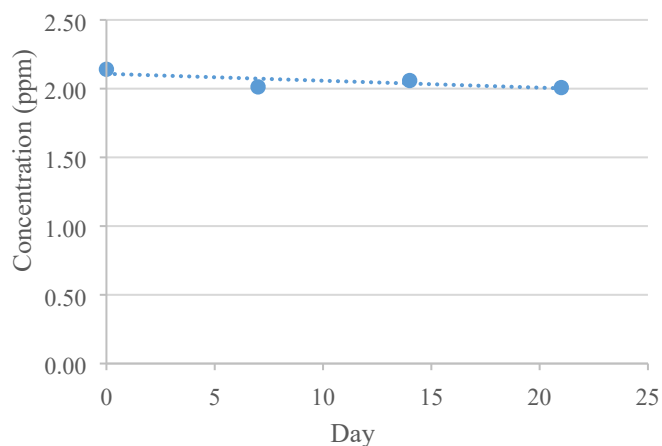
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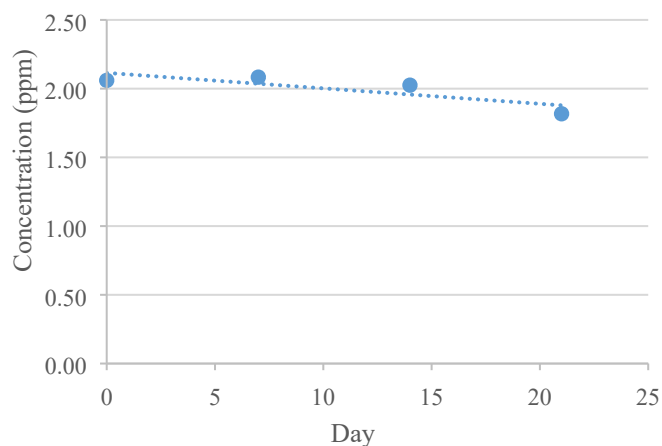
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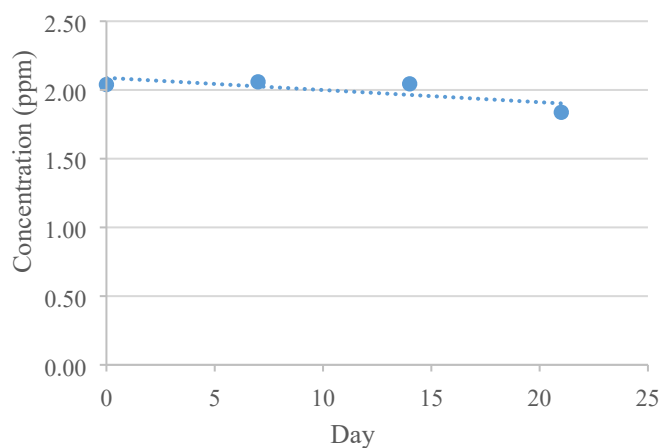
Stability of alpha-HCH in mix B at -20°C for short-term



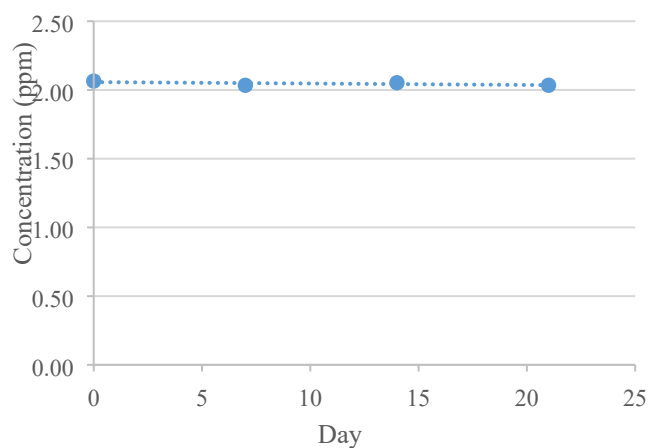
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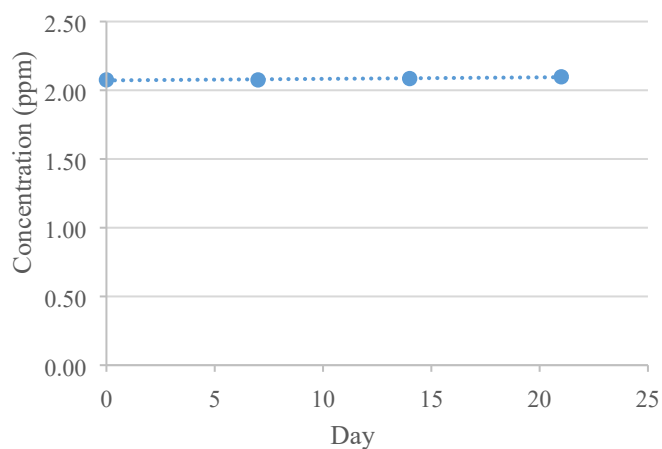
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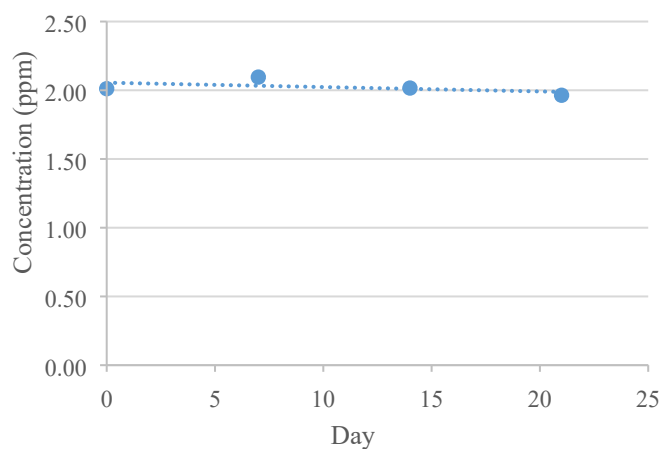
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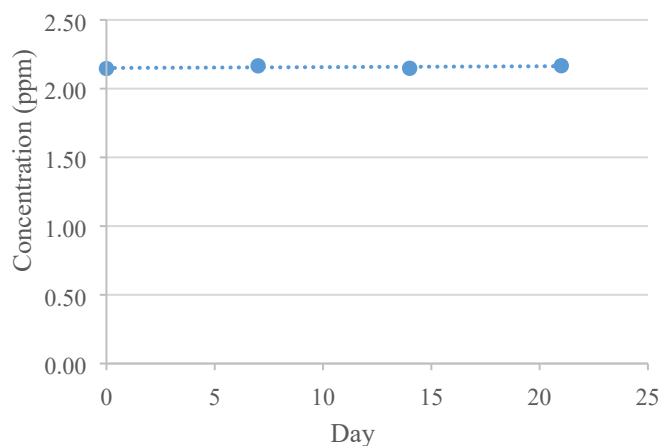
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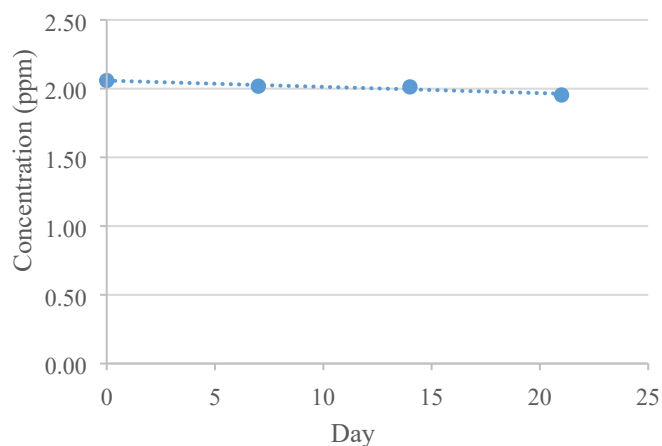
Stability of 4,4'-DDD in first bottle of mix B at -20°C for short-term



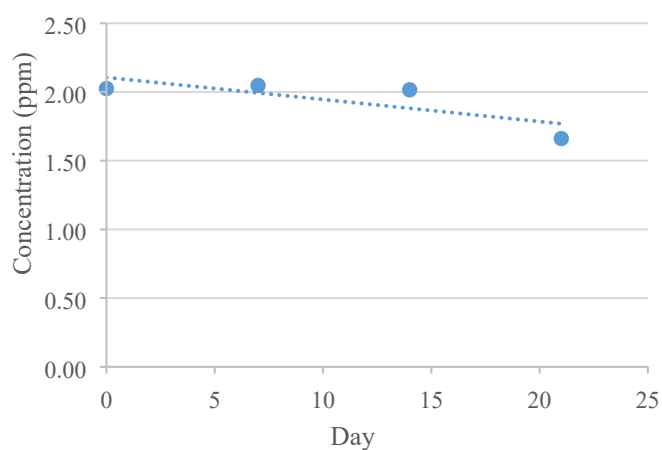
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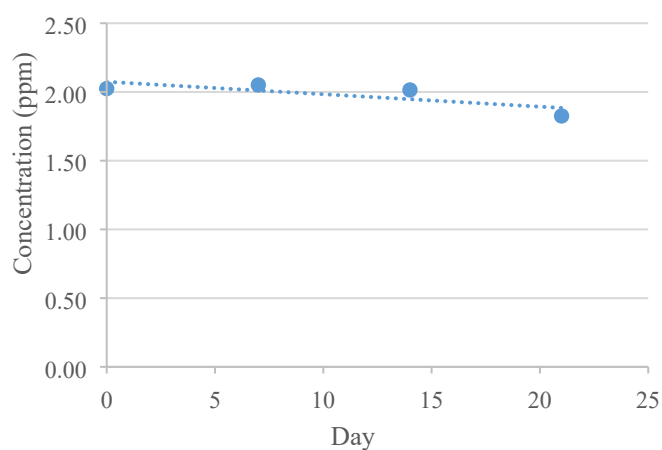
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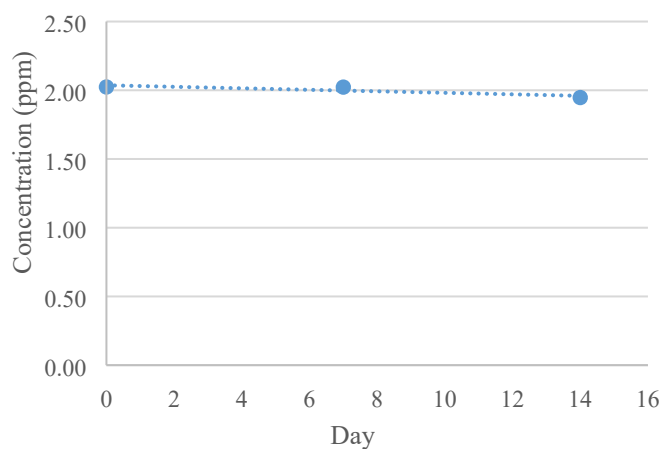
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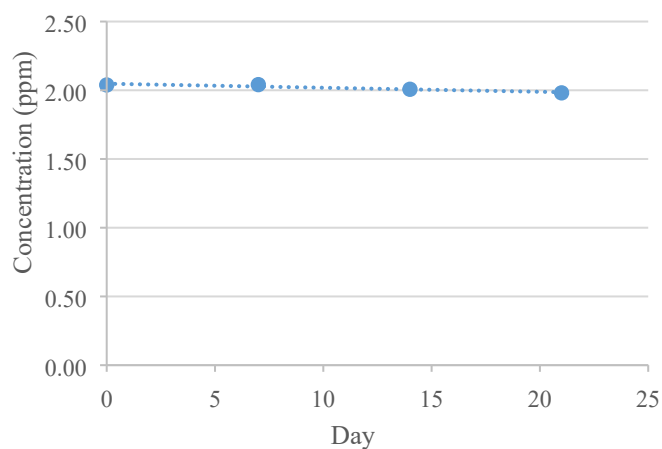
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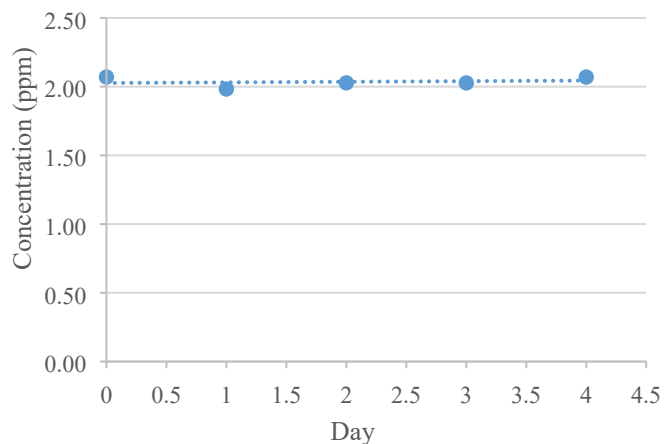
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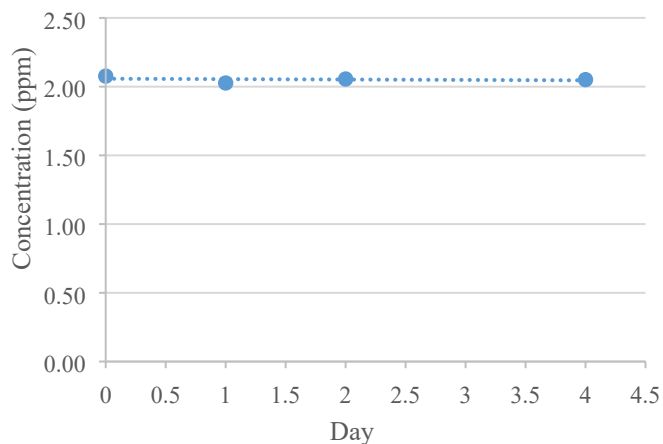
Stability of heptachlor-endo-epoxide in mix B at -20°C for short-term



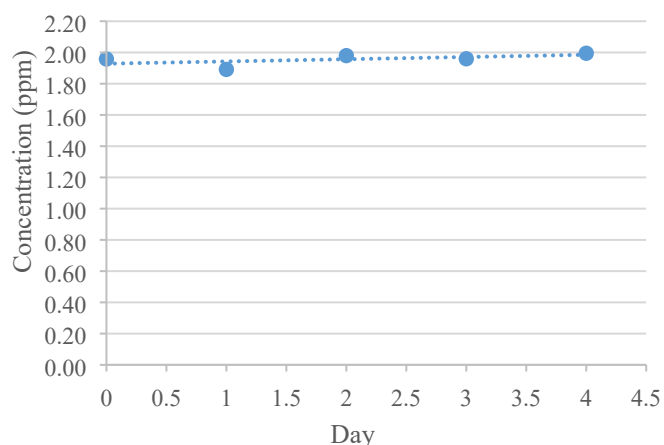
Stability of alpha-HCH in mix A at 20°C
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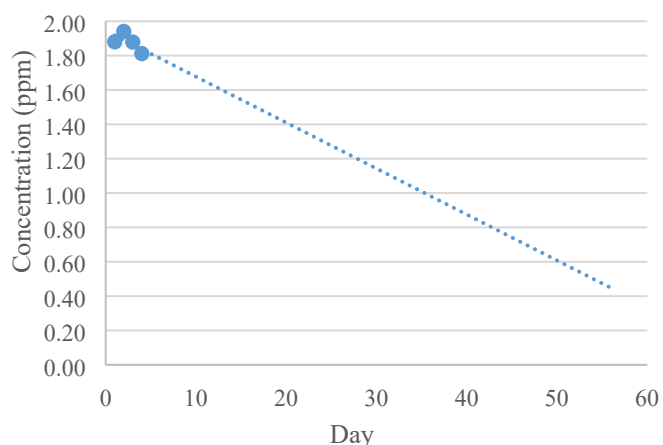
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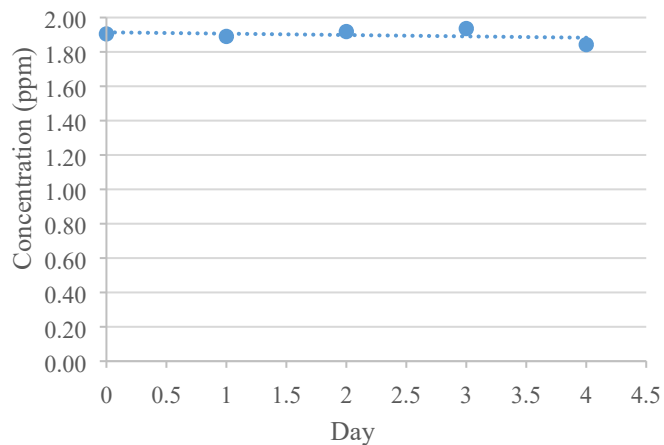
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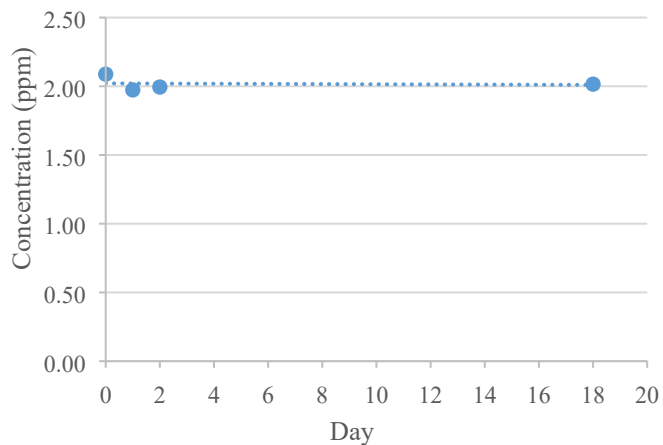
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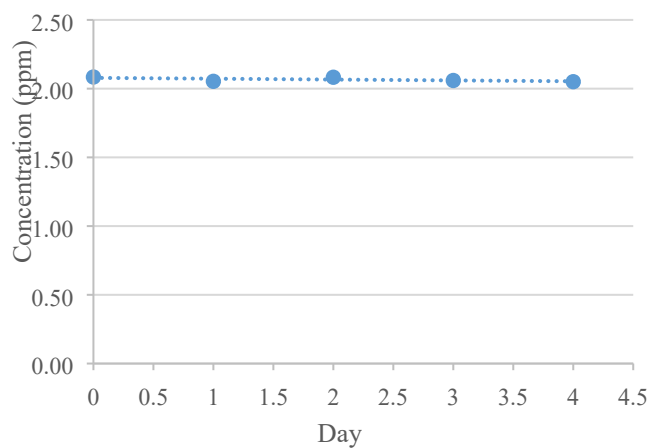
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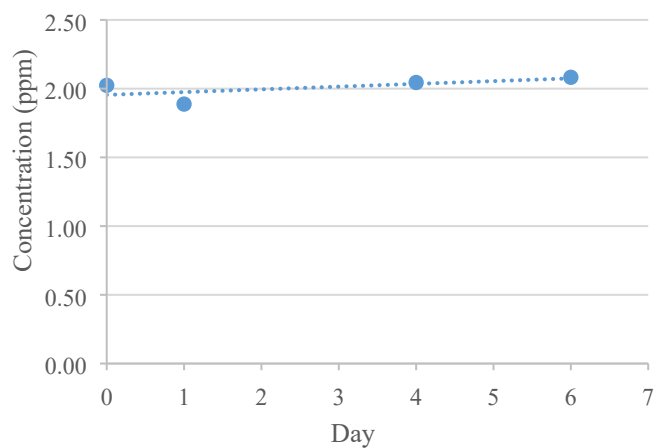
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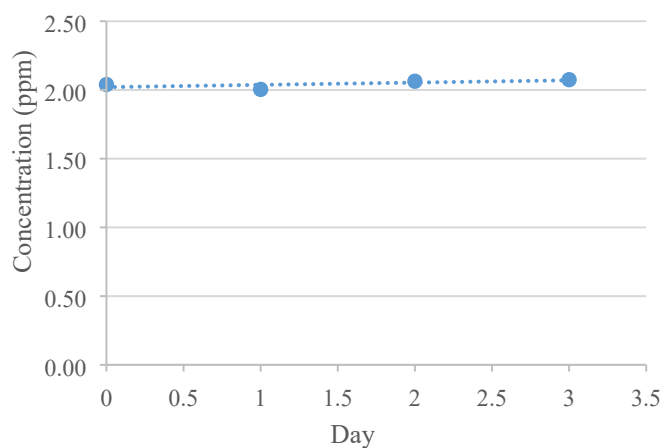
Stability of dieldrin in mix A at 20°C for short-term



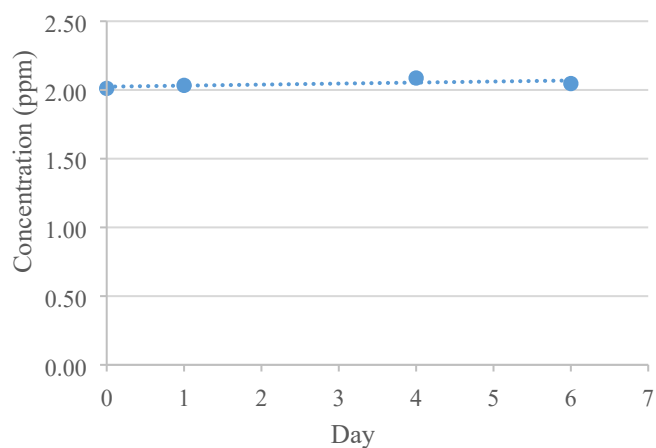
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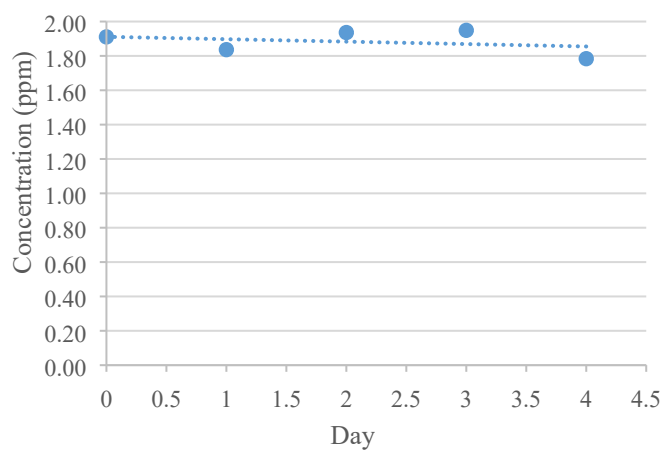
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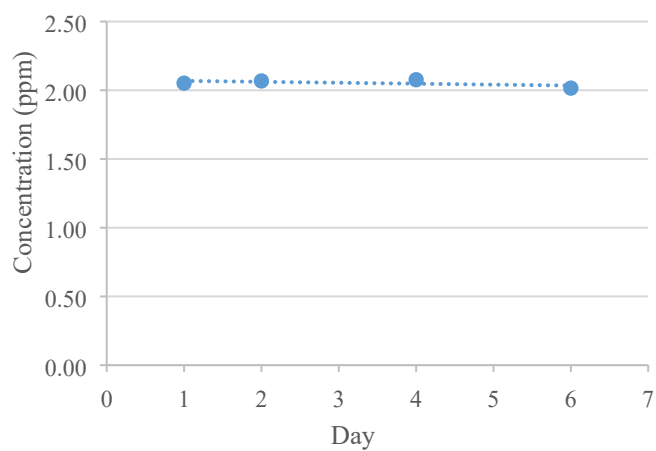
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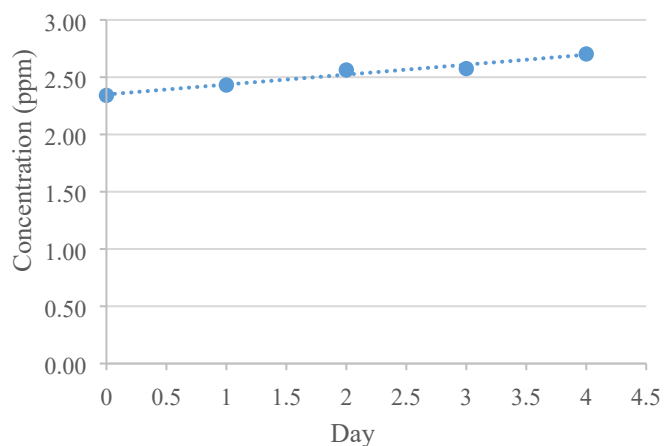
Stability of 4,4'-DDD in mix A at 20°C for short-term



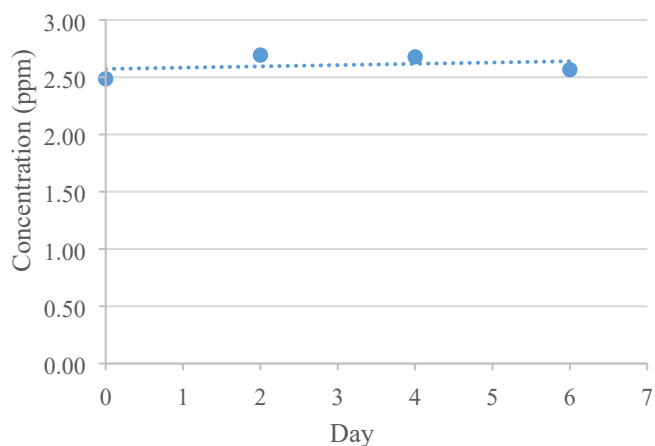
Stability of 4,4'-DDD in mix B at 20°C for short-term



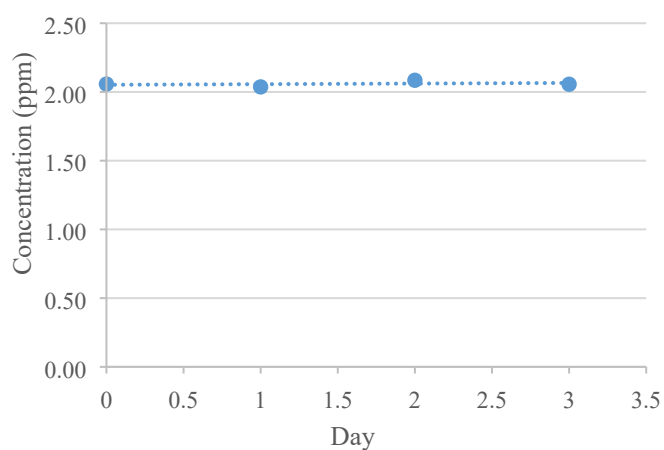
Stability of 4,4'-DDT in mix A at 20°C
for short-term



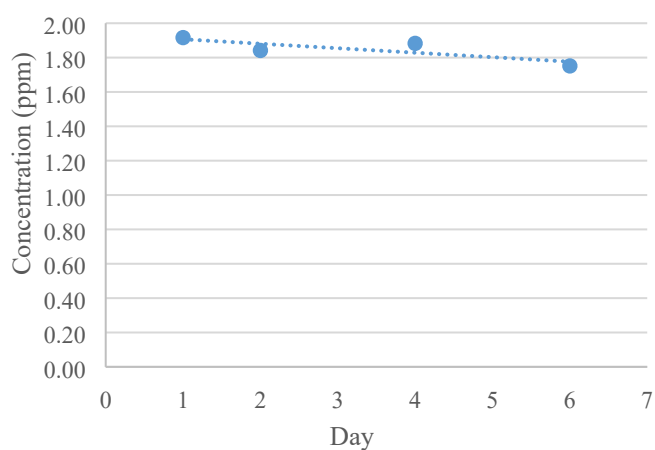
Stability of 4,4'-DDT in mix B at 20°C
for short-term



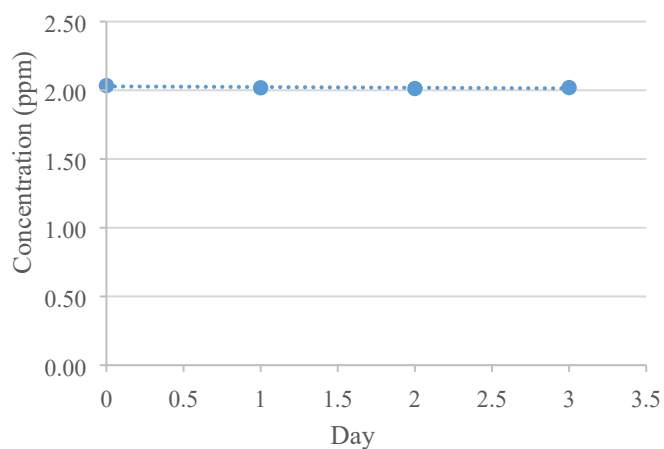
Stability of 2,4'-DDT in mix A at 20°C
for short-term



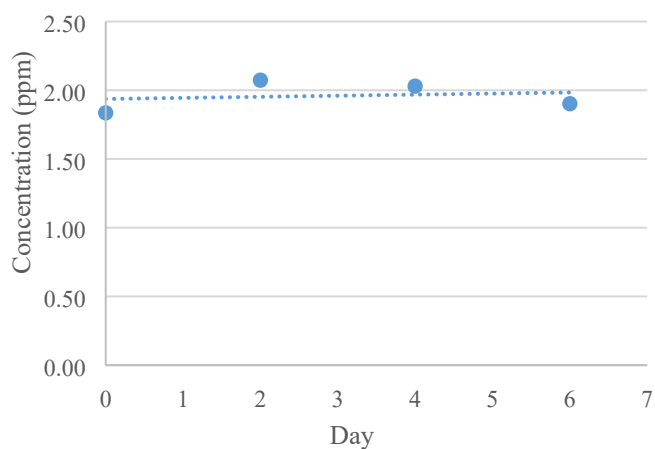
Stability of 2,4'-DDT in mix B at 20°C
for short-term



Stability of alpha-endosulfan in mix A at
20°C for short-term



Stability of alpha-endosulfan in mix B at
20°C for short-term



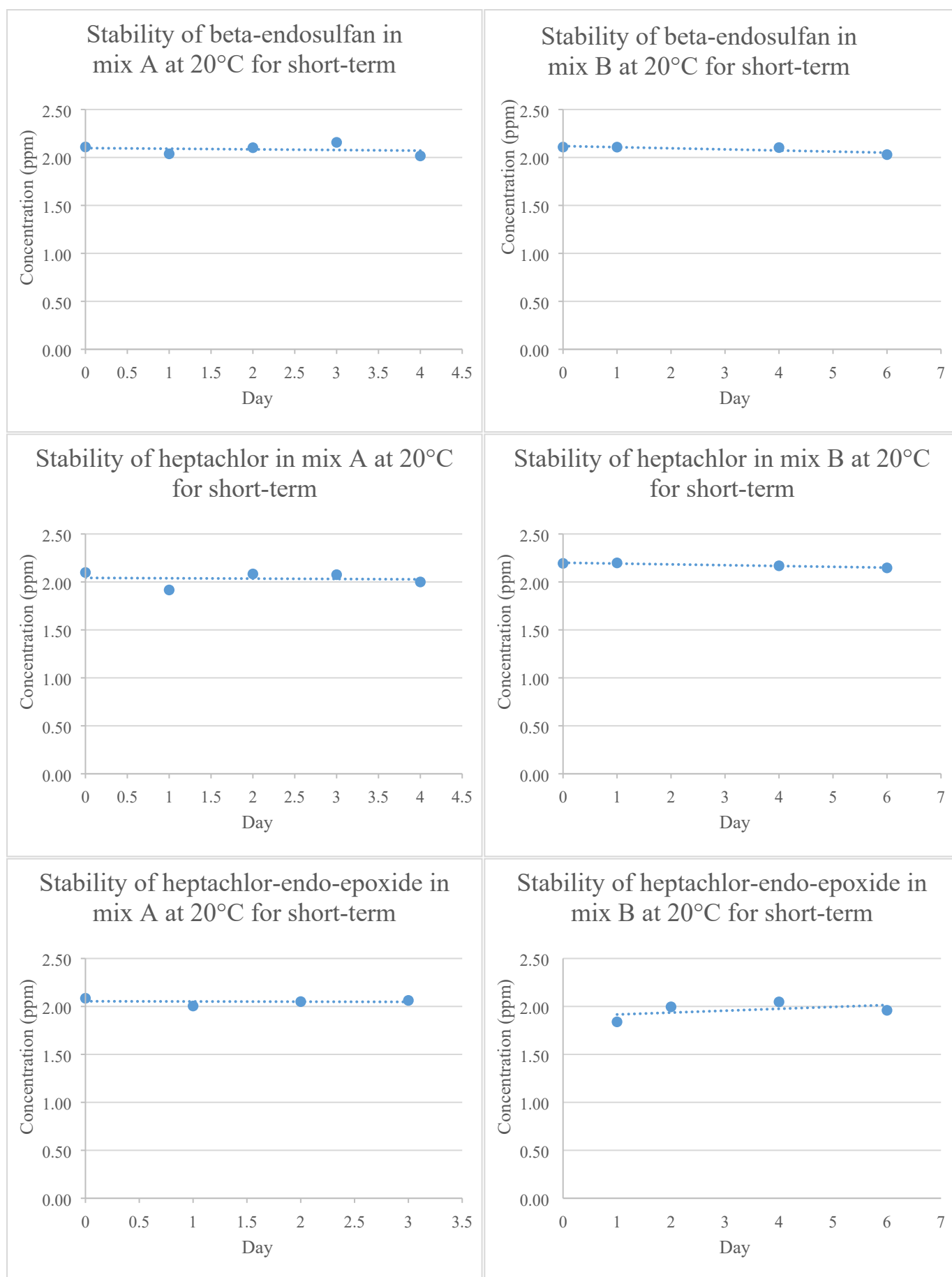
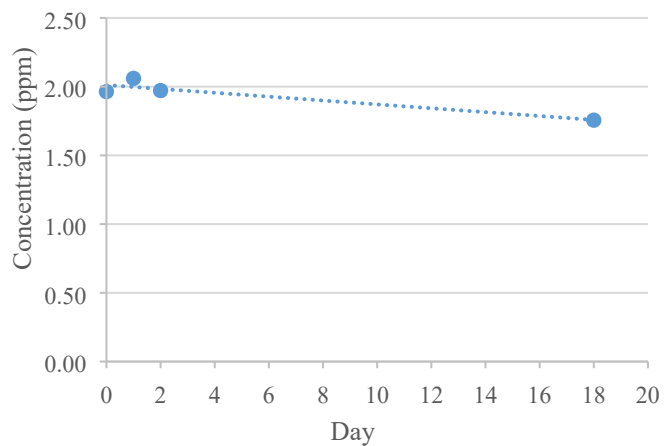
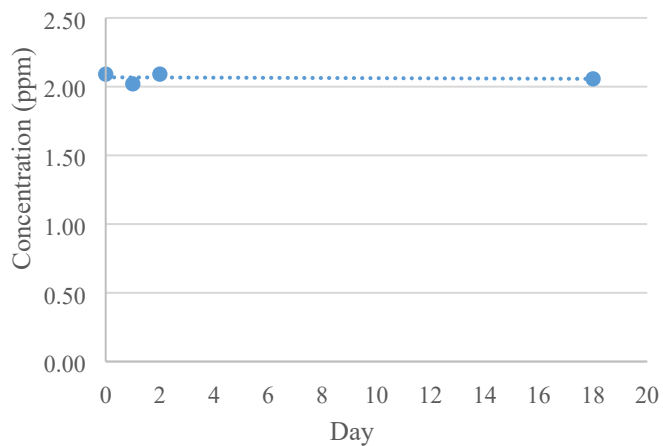


Fig. S5 The plots of gravimetric mixtures (A and B) in short term stability at 20°C for each pesticide

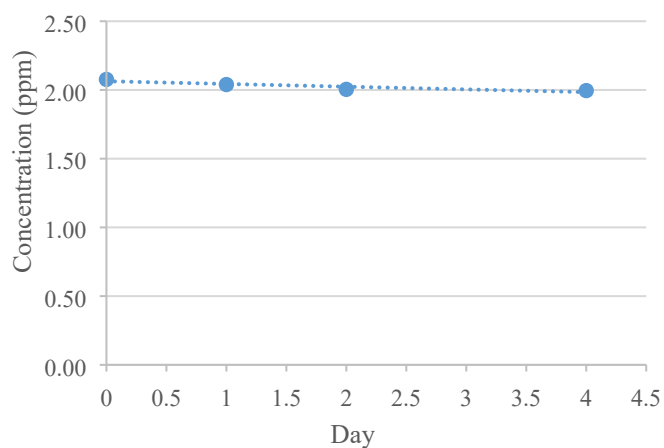
Stability of alpha-HCH in first bottle of mix A at +4°C for short-term



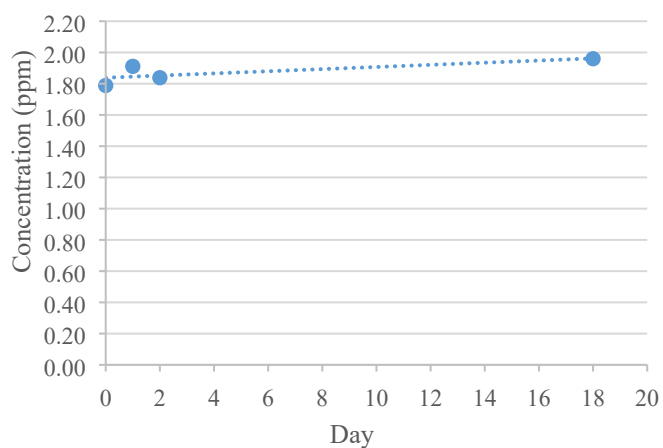
Stability of alpha-HCH in second bottle of mix A at +4°C for short-term



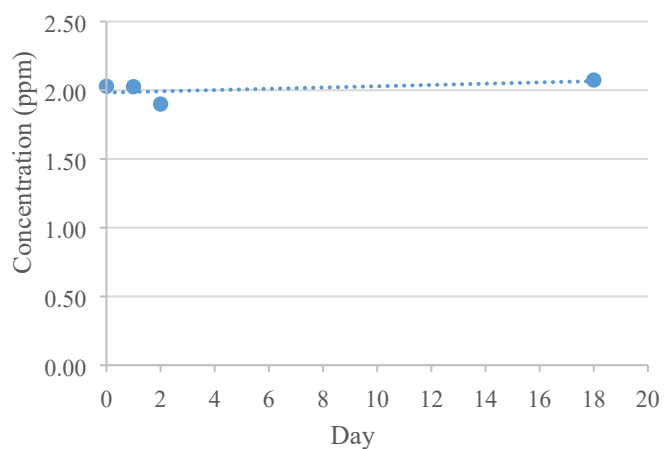
Stability of alpha-HCH in mix B at +4°C for short-term



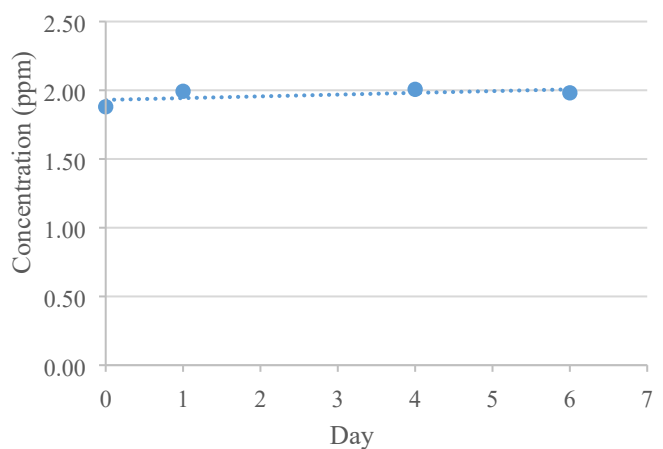
Stability of gamma-HCH in first bottle of mix A at +4°C for short-term



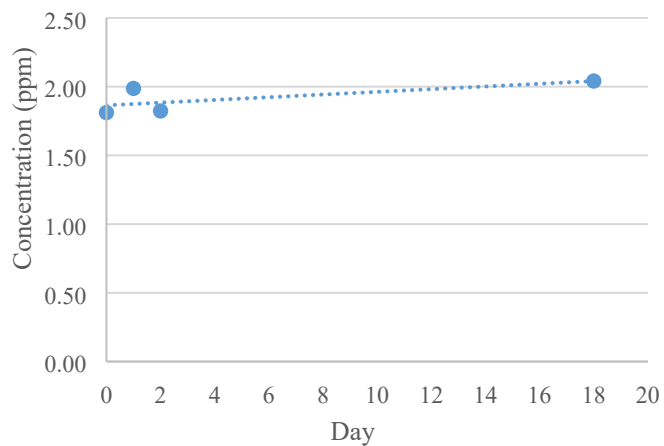
Stability of gamma-HCH in second bottle of mix A at +4°C for short-term



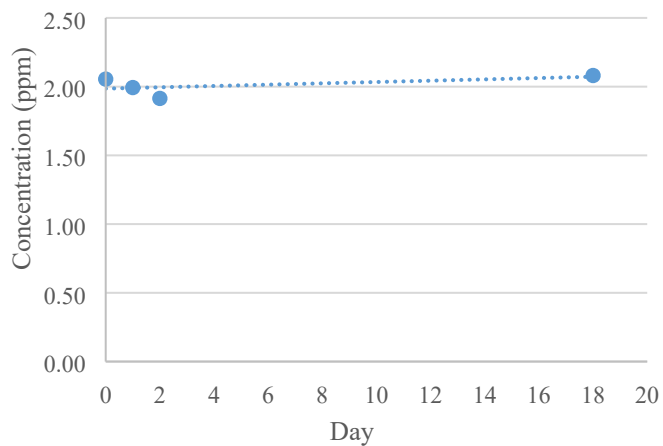
Stability of gamma-HCH in mix B at +4°C for short-term



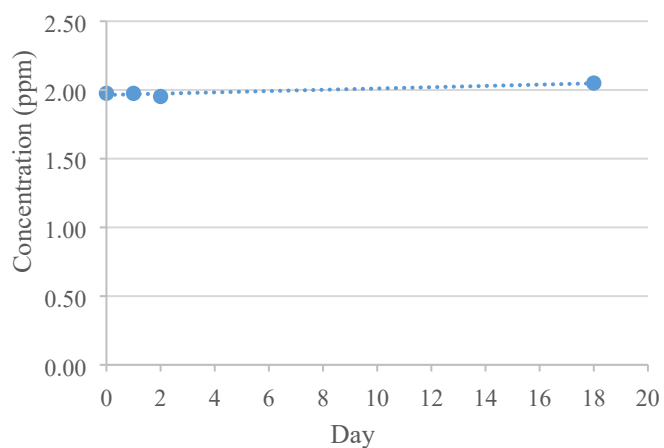
Stability of aldrin in first bottle of mix A at +4°C for short-term



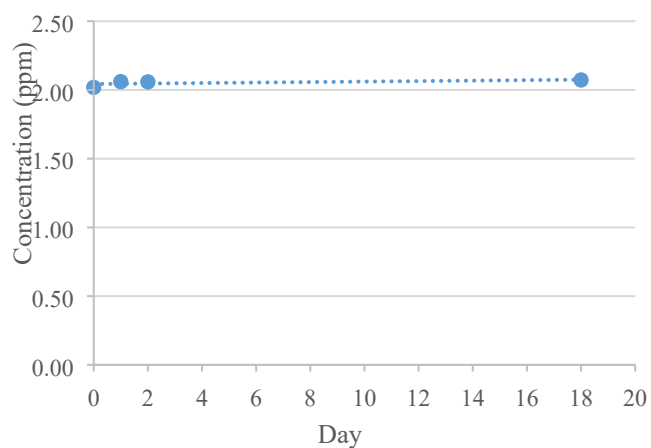
Stability of aldrin in second bottle of mix A at +4°C for short-term



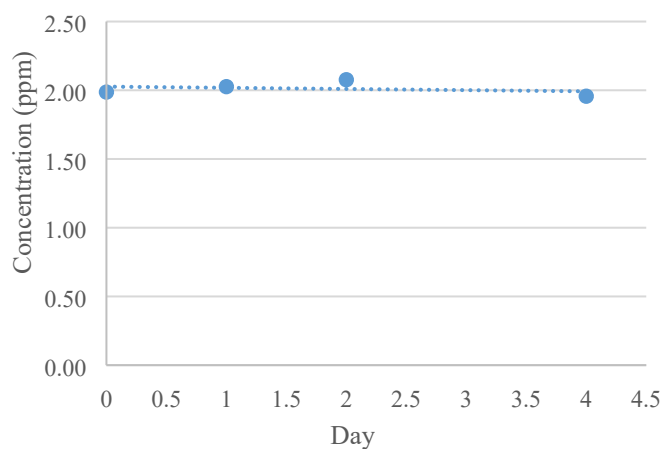
Stability of aldrin in mix B at +4°C for short-term



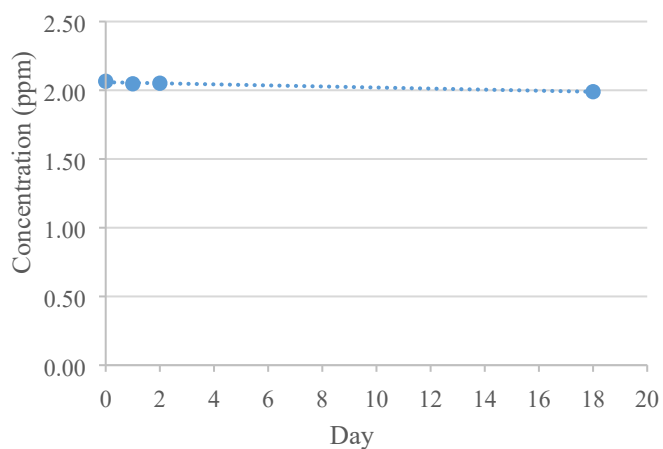
Stability of dieldrin in first bottle of mix A at +4°C for short-term



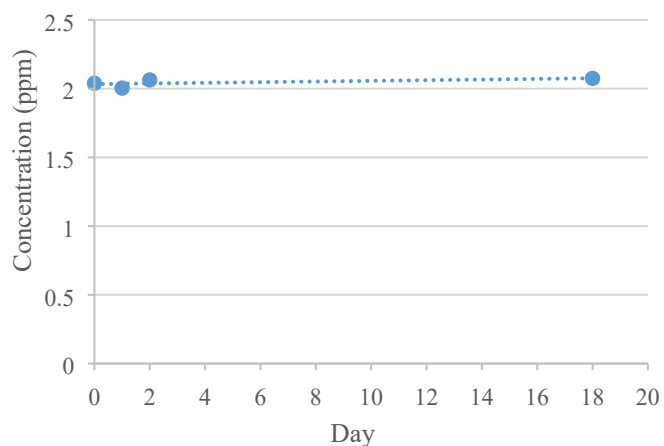
Stability of dieldrin in second bottle of mix A at +4°C for short-term



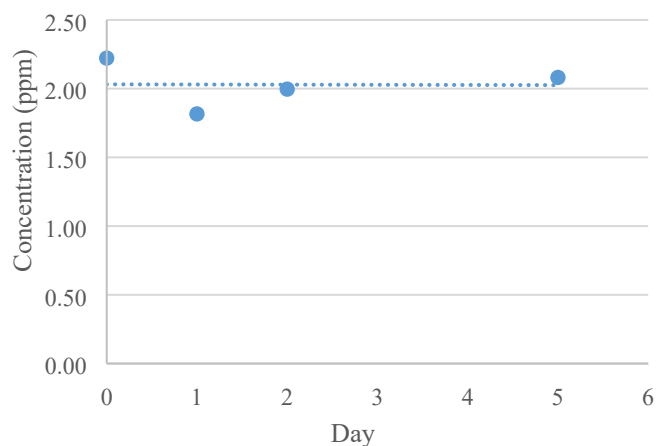
Stability of dieldrin in of mix B at +4°C for short-term



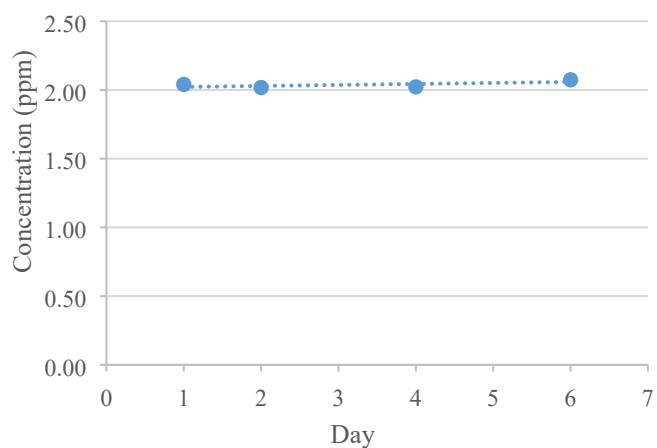
Stability of endrin in first bottle of mix A at +4°C for short-term



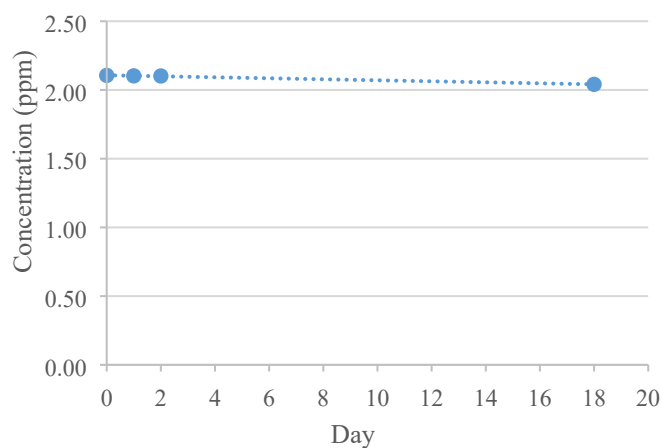
Stability of endrin in second bottle of mix A at +4°C for short-term



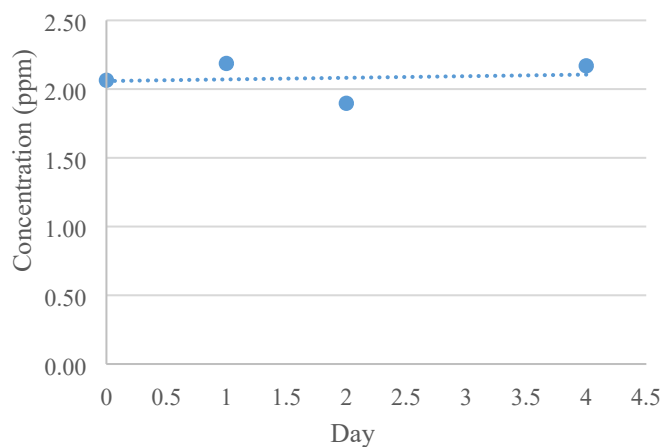
Stability of endrin in mix B at +4°C for short-term



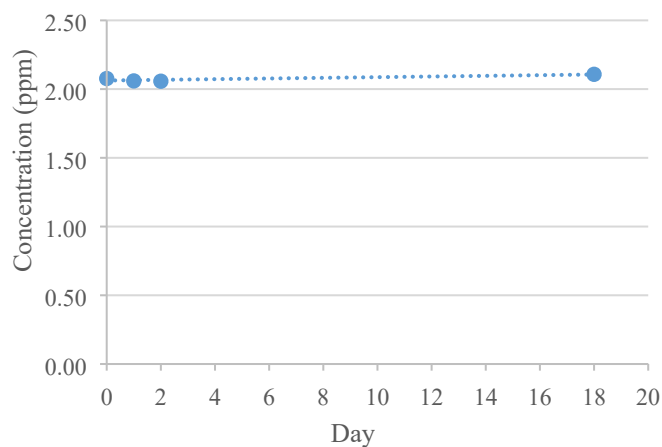
Stability of 4,4'-DDD in first bottle of mix A at +4°C for short-term



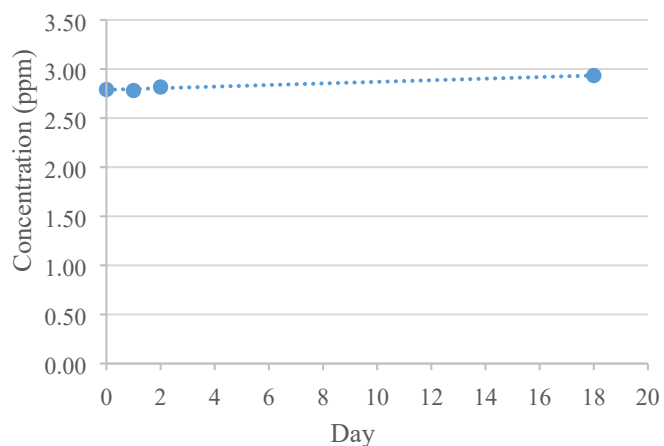
Stability of 4,4'-DDD in second bottle of mix A at +4°C for short-term



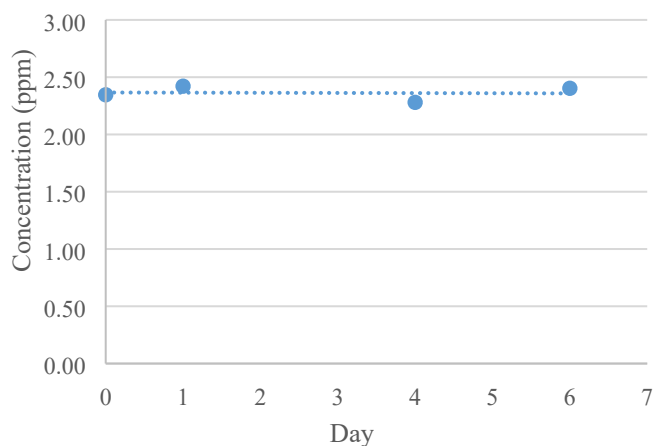
Stability of 4,4'-DDD in mix B at +4°C for short-term



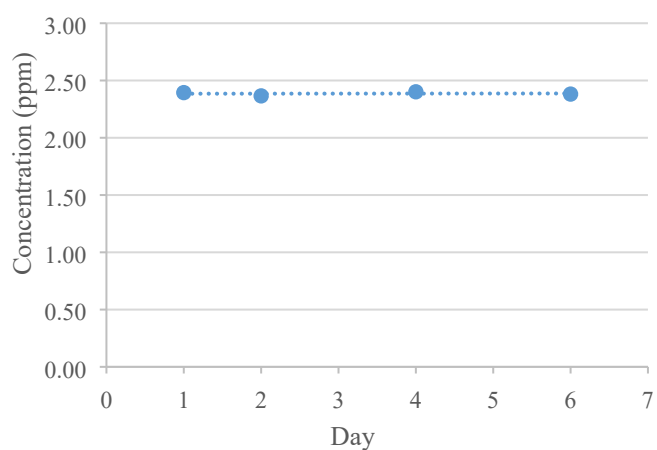
Stability of 4,4'-DDT in first bottle of mix A at +4°C for short-term



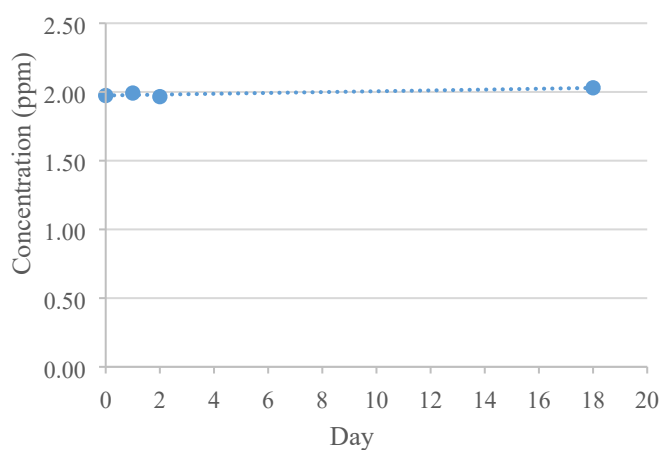
Stability of 4,4'-DDT in second bottle of mix A at +4°C for short-term



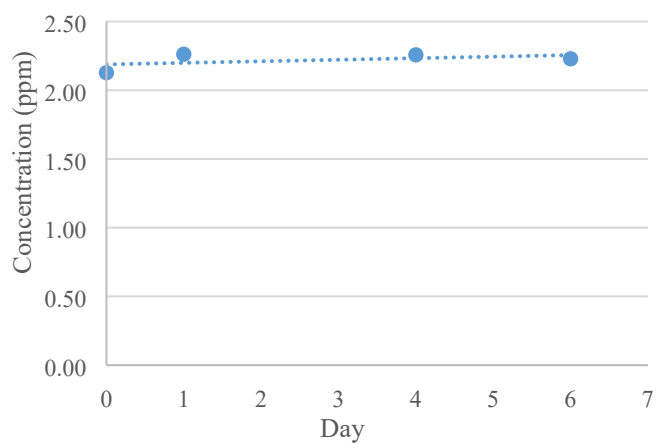
Stability of 4,4'-DDT in mix B at +4°C for short-term



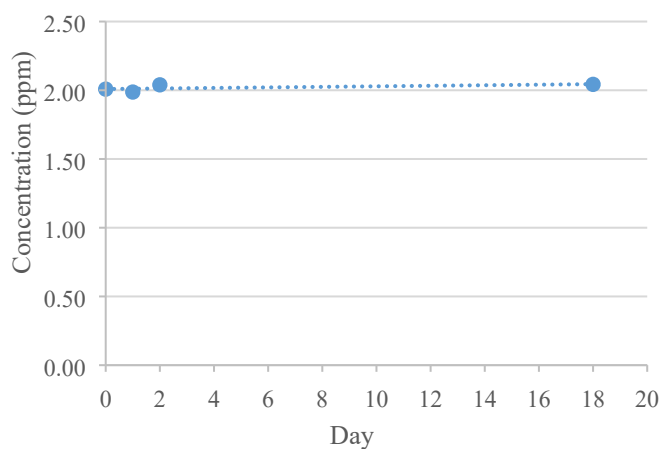
Stability of 2,4'-DDT in first bottle of mix A at +4°C for short-term



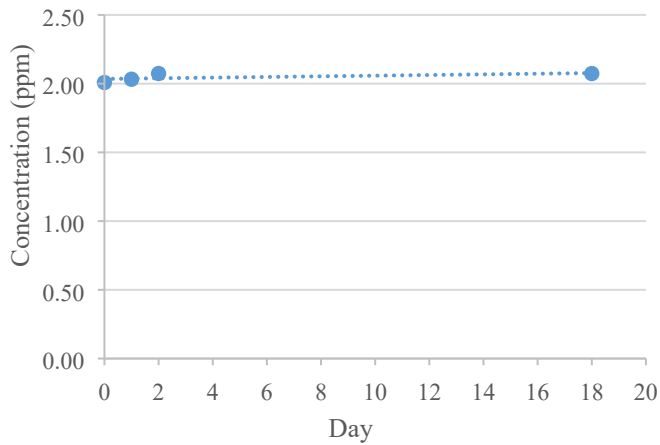
Stability of 2,4'-DDT in second bottle of mix A at +4°C for short-term



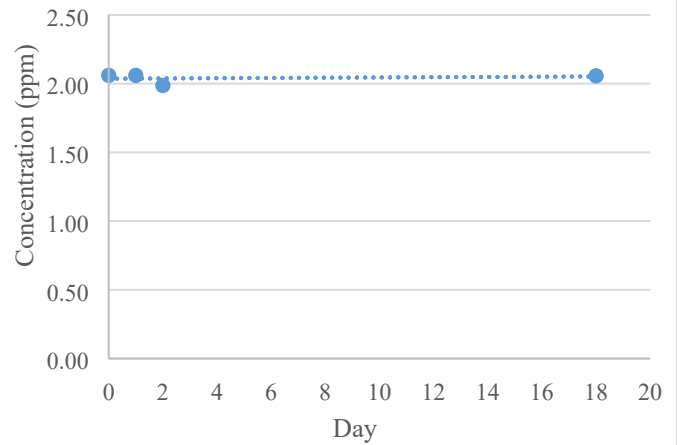
Stability of 2,4'-DDT in mix B at +4°C for short-term



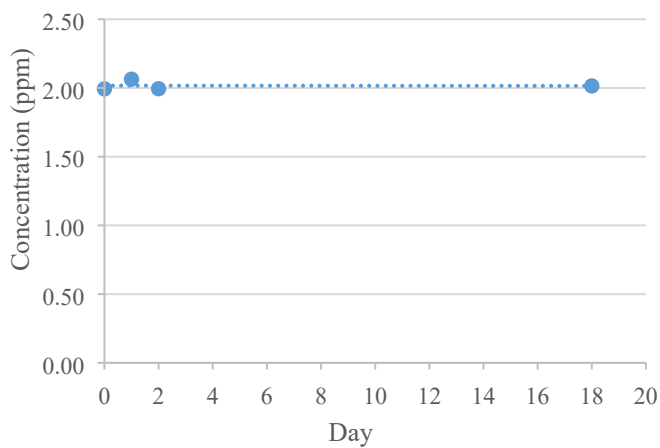
Stability of alpha-endosulfan in first bottle of mix A at +4°C for short-term



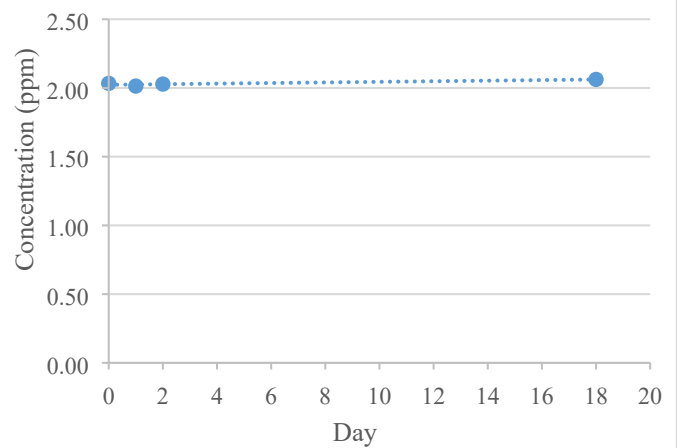
Stability of alpha-endosulfan in second bottle of mix A at +4°C for short-term



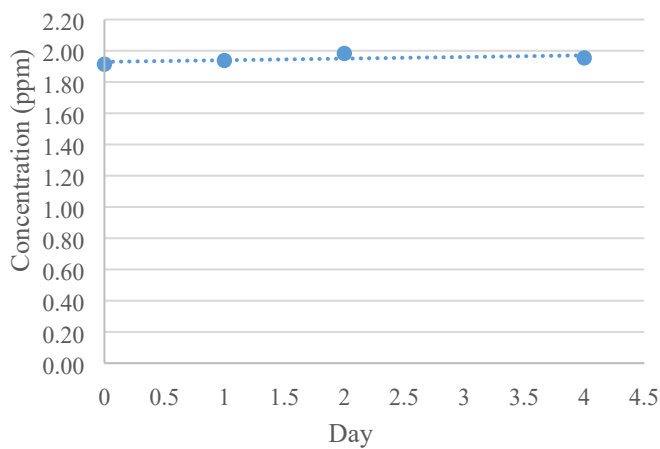
Stability of alpha-endosulfan in mix B at +4°C for short-term



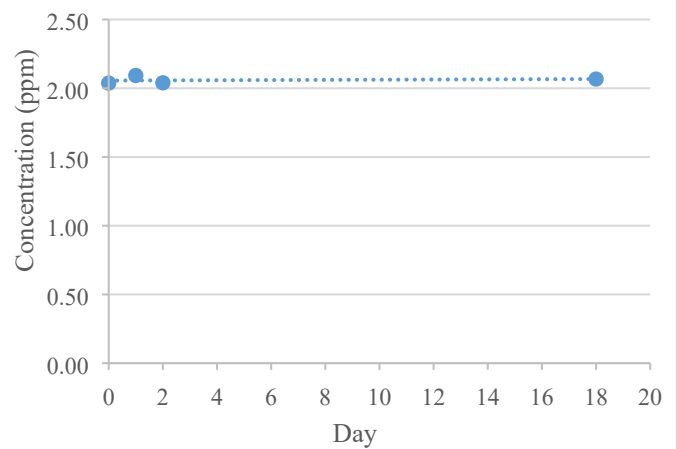
Stability of beta-endosulfan in first bottle of mix A at +4°C for short-term



Stability of beta-endosulfan in second bottle of mix A at +4°C for short-term



Stability of beta-endosulfan in mix B at +4°C for short-term



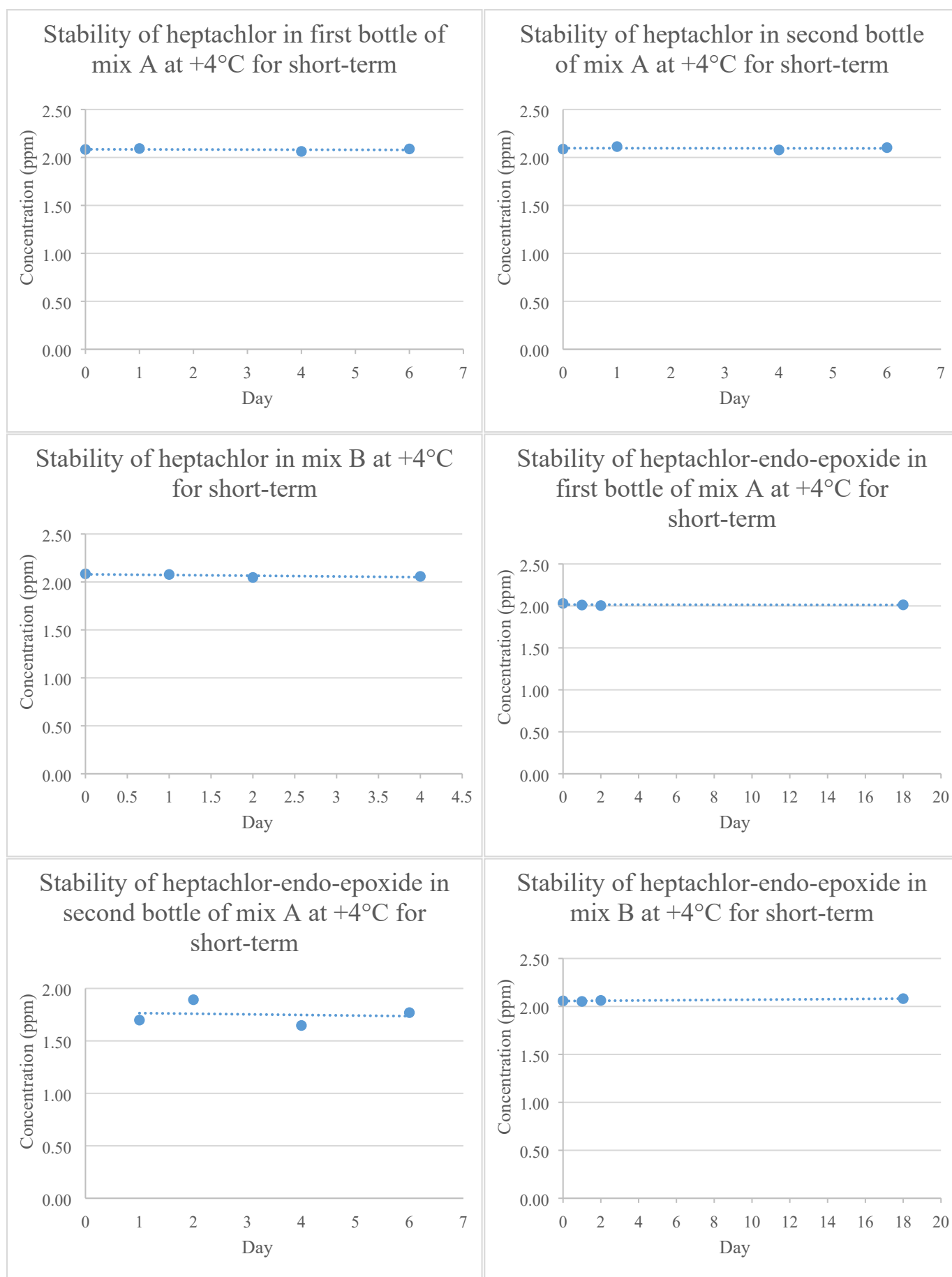
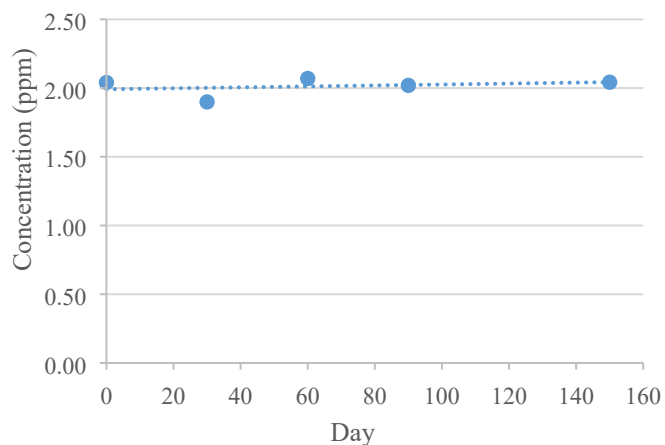
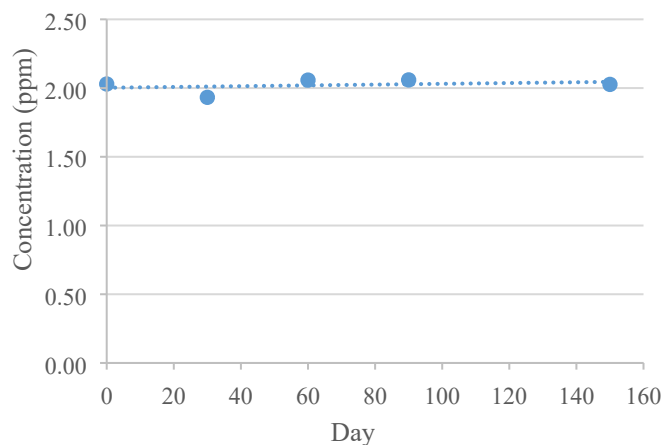


Fig. S6 The plots of gravimetric mixtures (A and B) in short term stability at +4°C for each pesticide

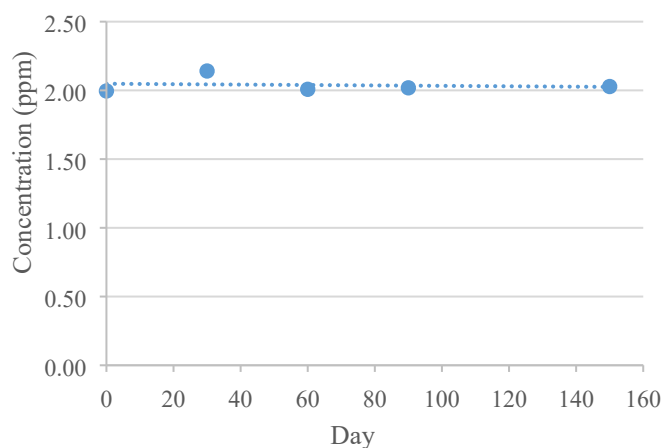
Stability of alpha-HCH in first bottle of mix A at -20°C for long-term



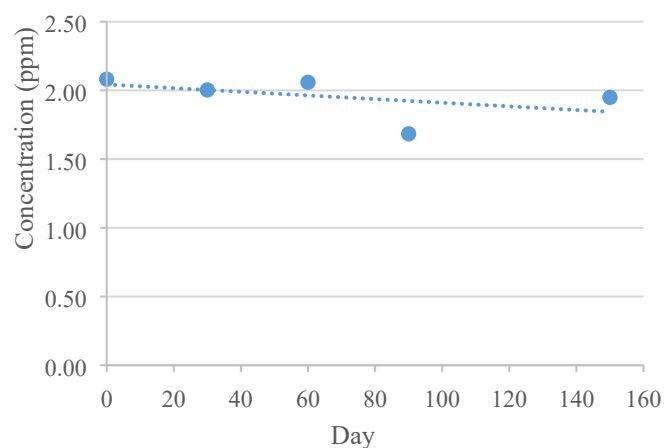
Stability of alpha-HCH in second bottle of mix A at -20°C for long-term



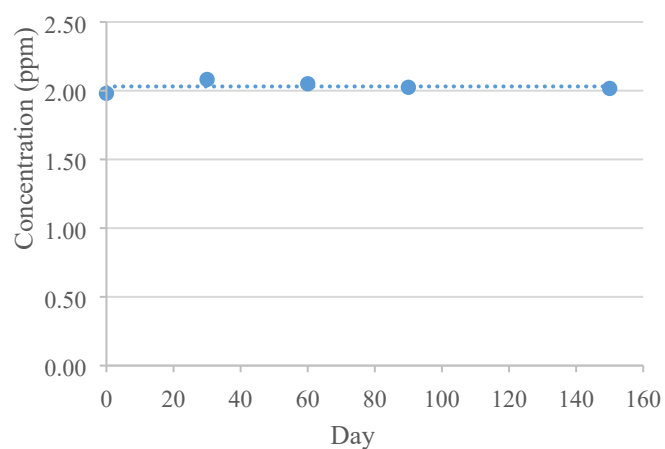
Stability of alpha-HCH in mix B at -20°C for long-term



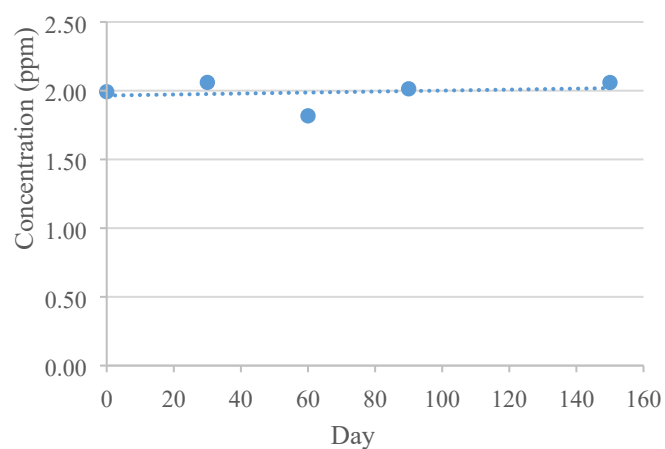
Stability of gamma-HCH in first bottle of mix A at -20°C for long-term



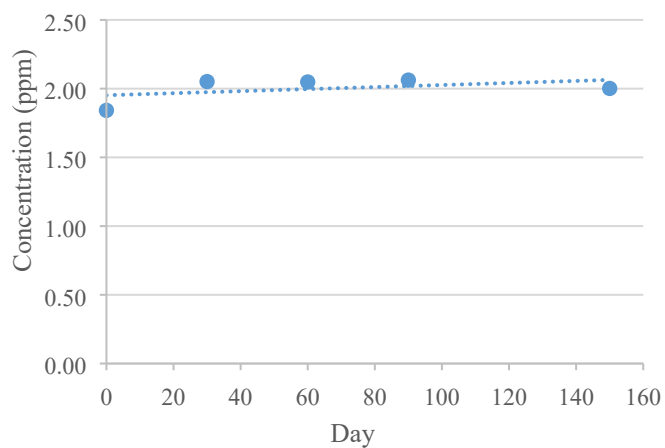
Stability of gamma-HCH in second bottle of mix A at -20°C for long-term



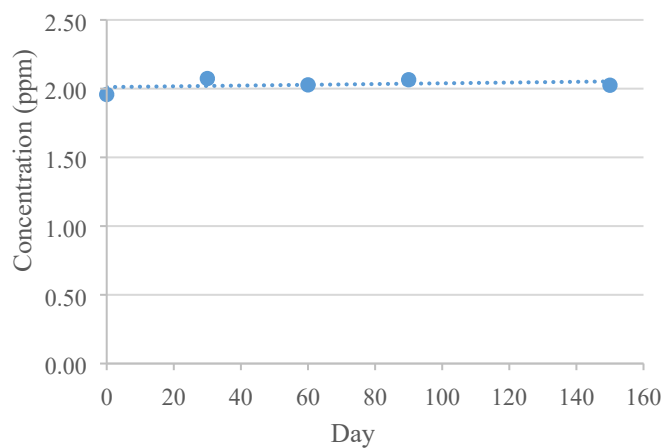
Stability of gamma-HCH in mix B at -20°C for long-term



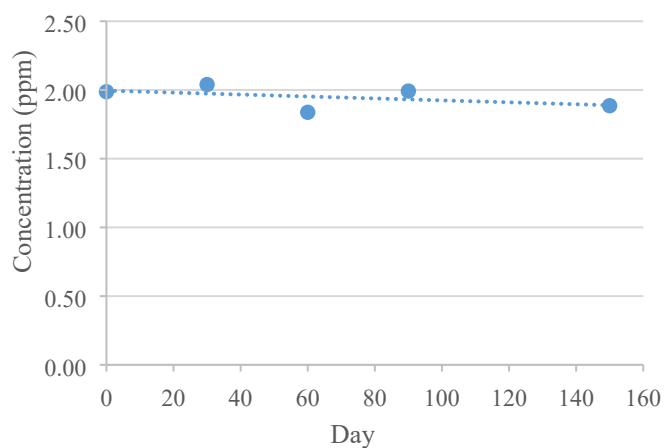
Stability of aldrin in first bottle of mix A at -20°C for long-term



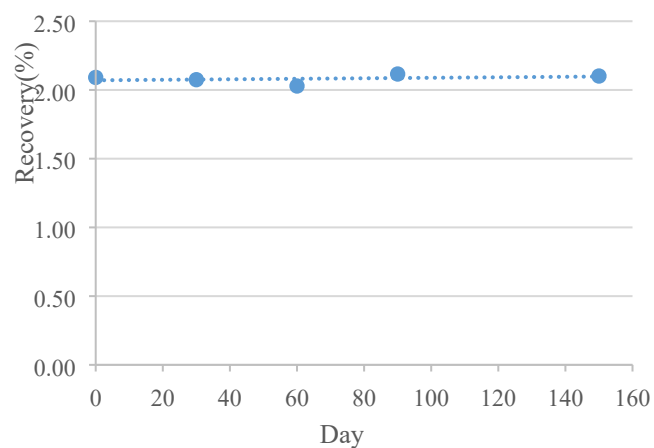
Stability of aldrin in second bottle of mix A at -20°C for long-term



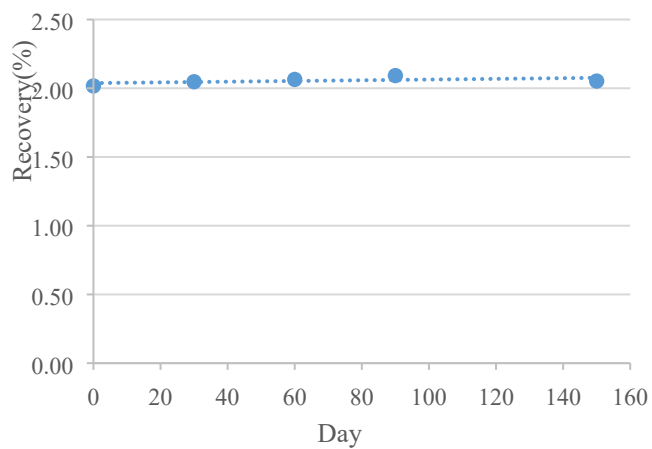
Stability of aldrin in mix B at -20°C for long-term



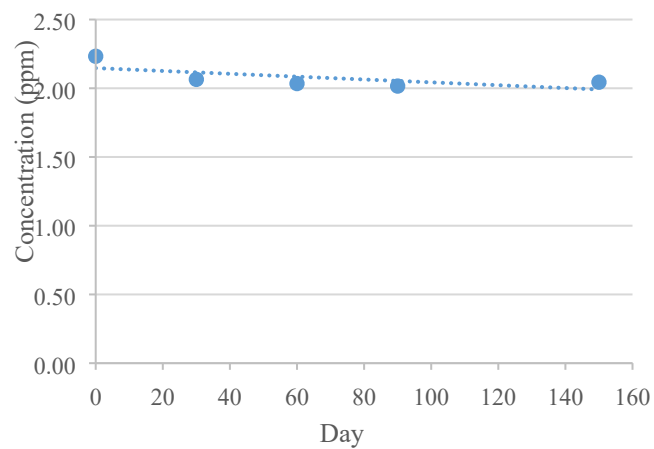
Stability of dieldrin in first bottle of mix A at -20°C for long-term



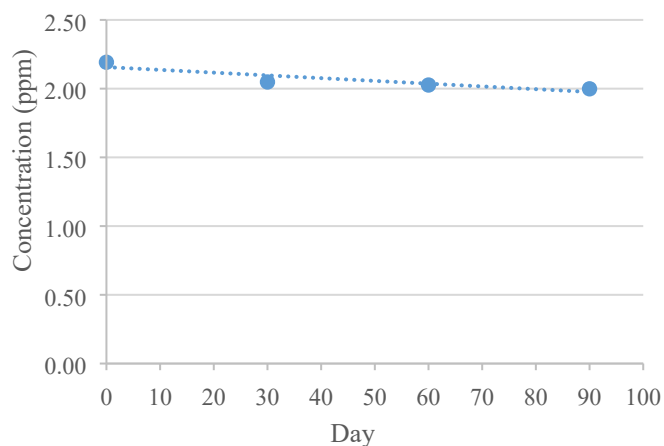
Stability of dieldrin in second bottle of mix A at -20°C for long-term



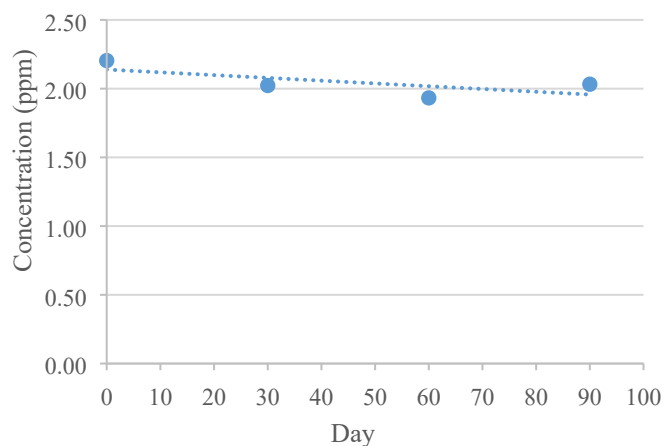
Stability of dieldrin in mix B at -20°C for long-term



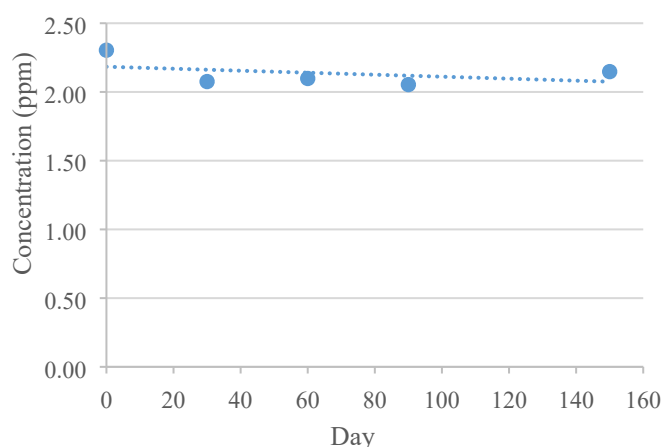
Stability of endrin in first bottle of mix A at -20°C for long term



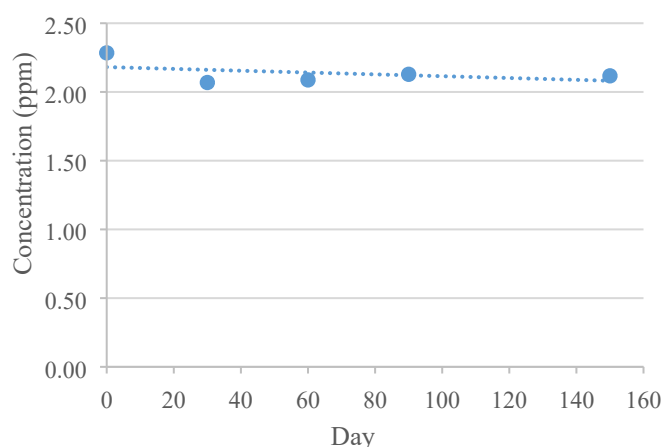
Stability of endrin in second bottle of mix A at -20°C for long-term



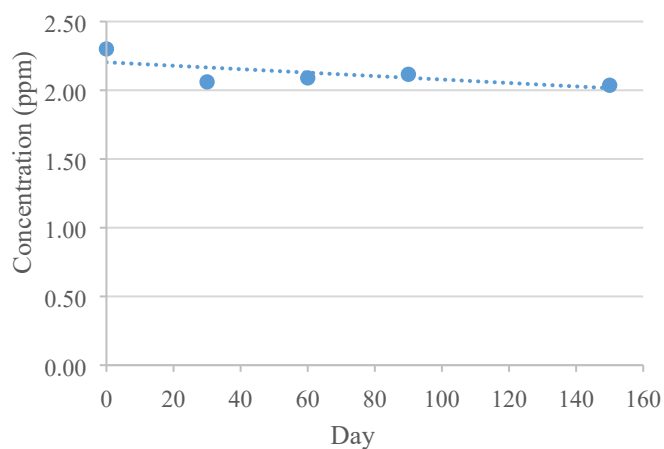
Stability of endrin in mix B at -20°C for long-term



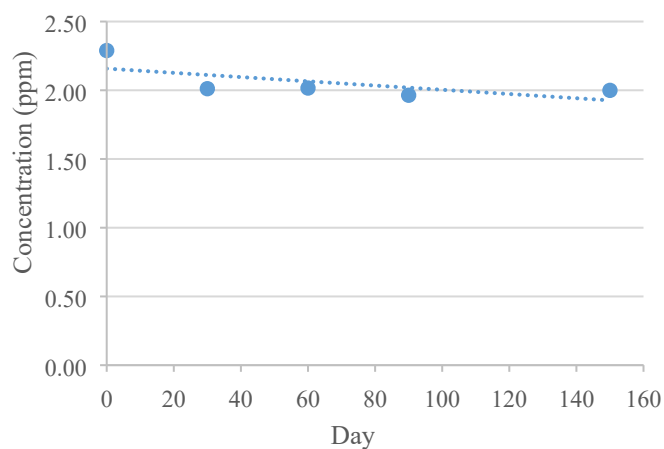
Stability of 4,4'-DDD in second bottle of mix A at -20°C for long-term



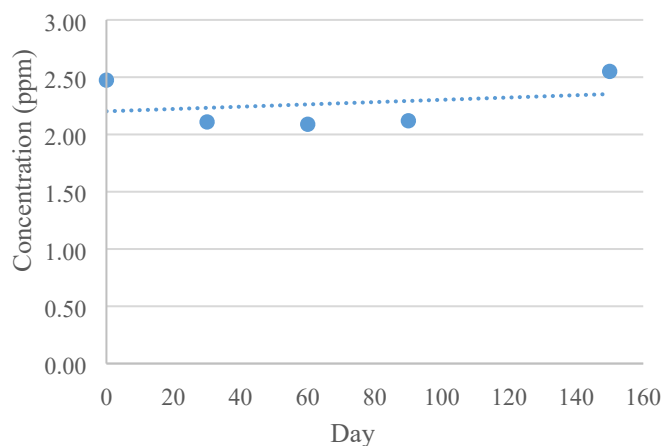
Stability of 4,4'-DDD in first bottle of mix A at -20°C for long-term



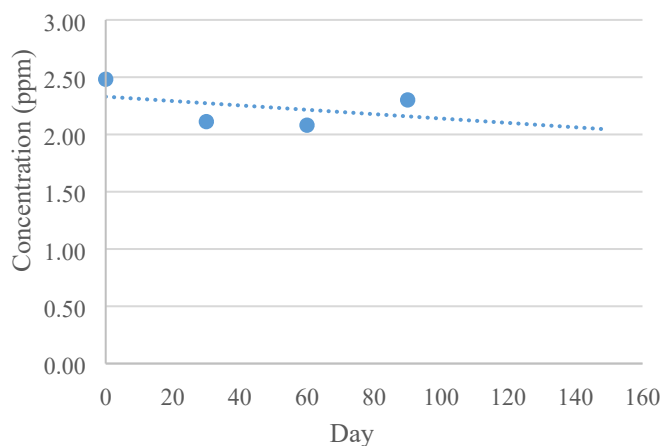
Stability of 4,4'-DDD in mix B at -20°C for long-term



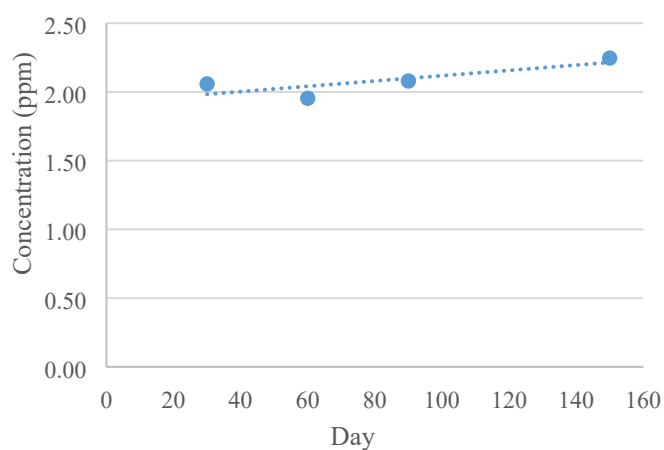
Stability of 4,4'-DDT in first bottle of mix A at -20°C for long-term



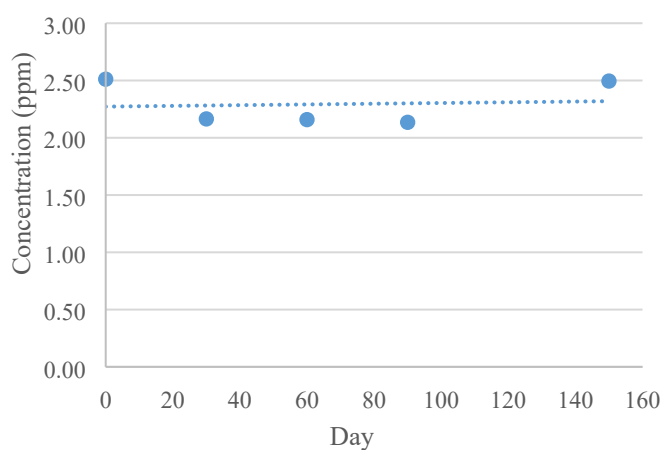
Stability of 4,4'-DDT in second bottle of mix A at -20°C for long-term



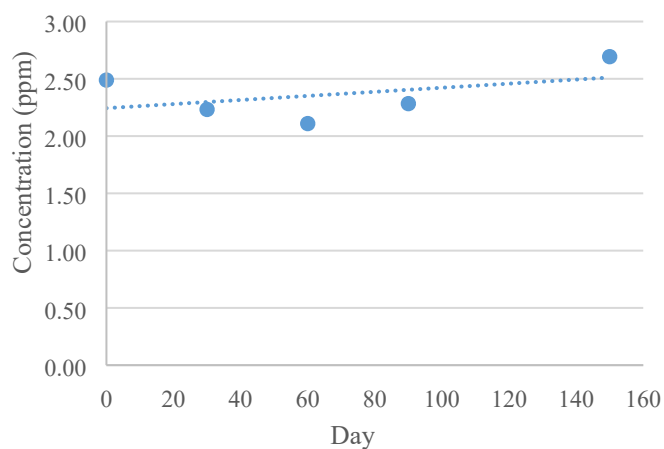
Stability of 4,4'-DDT in mix B at -20°C for long-term



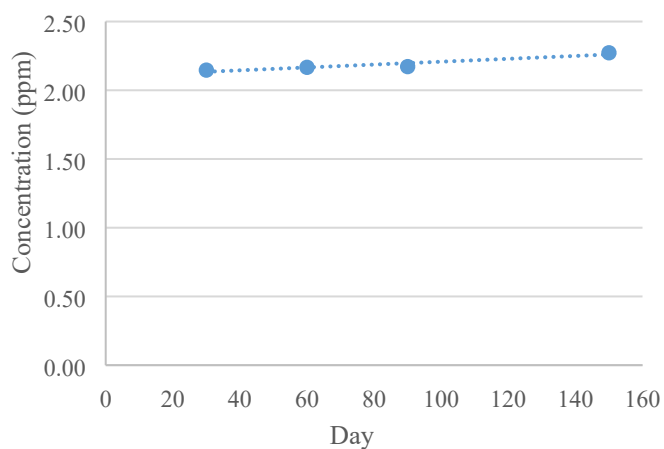
Stability of 2,4'-DDT in first bottle of mix A at -20°C for long-term



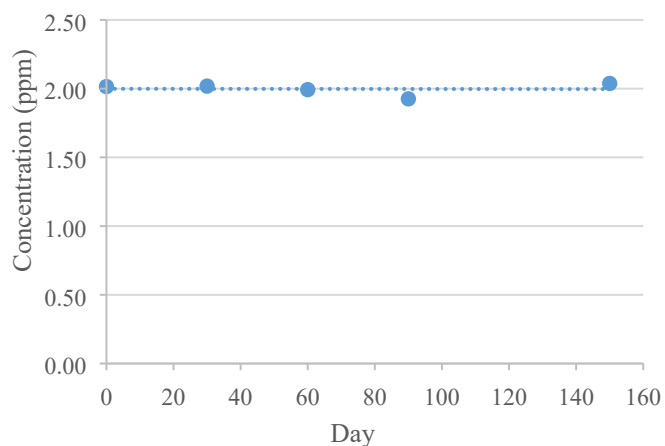
Stability of 2,4'-DDT in second bottle of mix A at -20°C for long-term



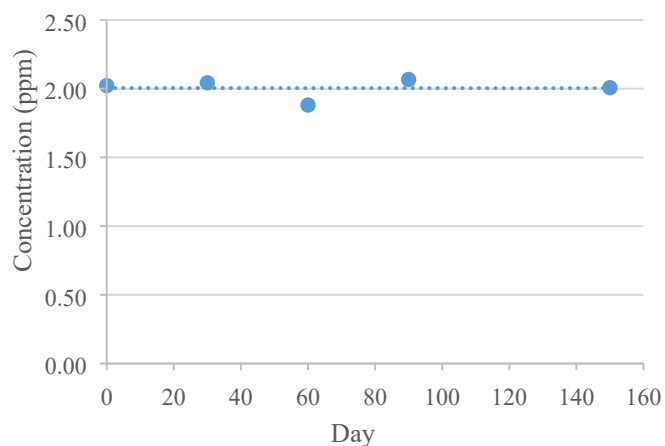
Stability of 2,4'-DDT in mix B at -20°C for long-term



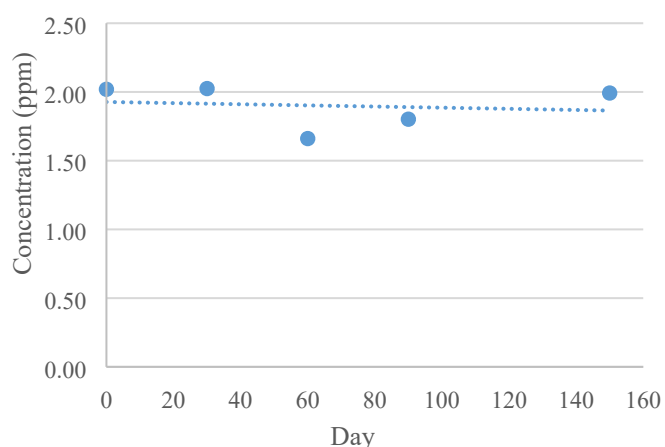
Stability of alpha-endosulfan in first bottle of mix A at -20°C for long-term



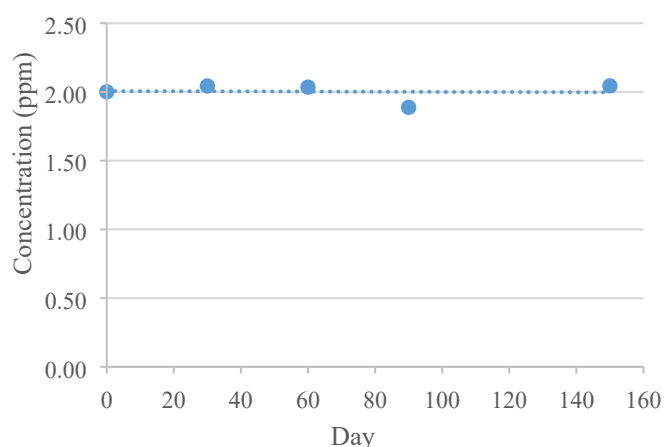
Stability of alpha-endosulfan in second bottle of mix A at -20°C for long-term



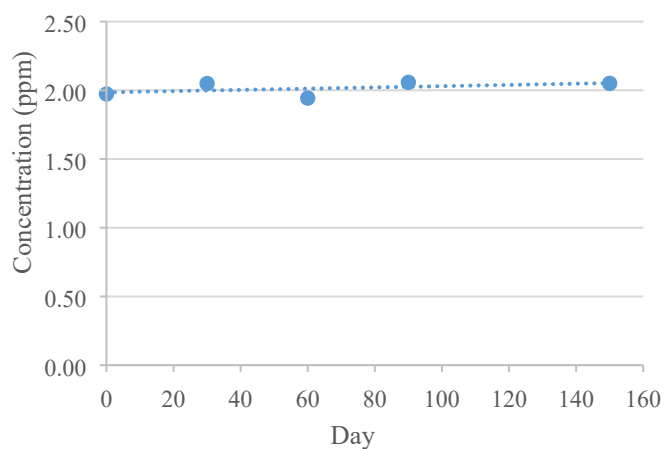
Stability of alpha-endosulfan in mix B at -20°C for long-term



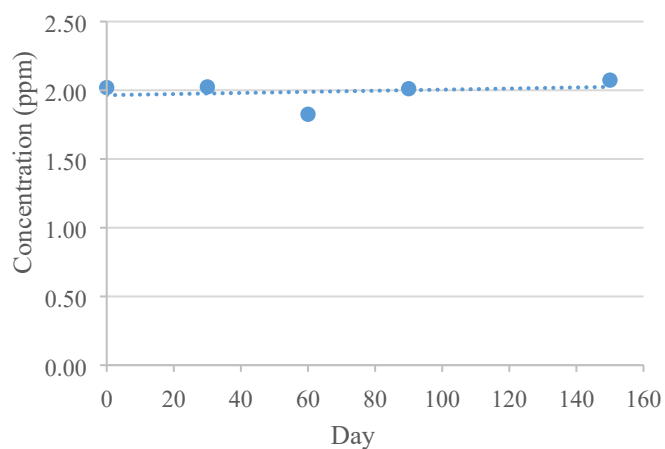
Stability of beta-endosulfan in first bottle of mix A at -20°C for long-term



Stability of beta-endosulfan in second bottle of mix A at -20°C for long-term



Stability of beta-endosulfan in mix B at -20°C for long-term



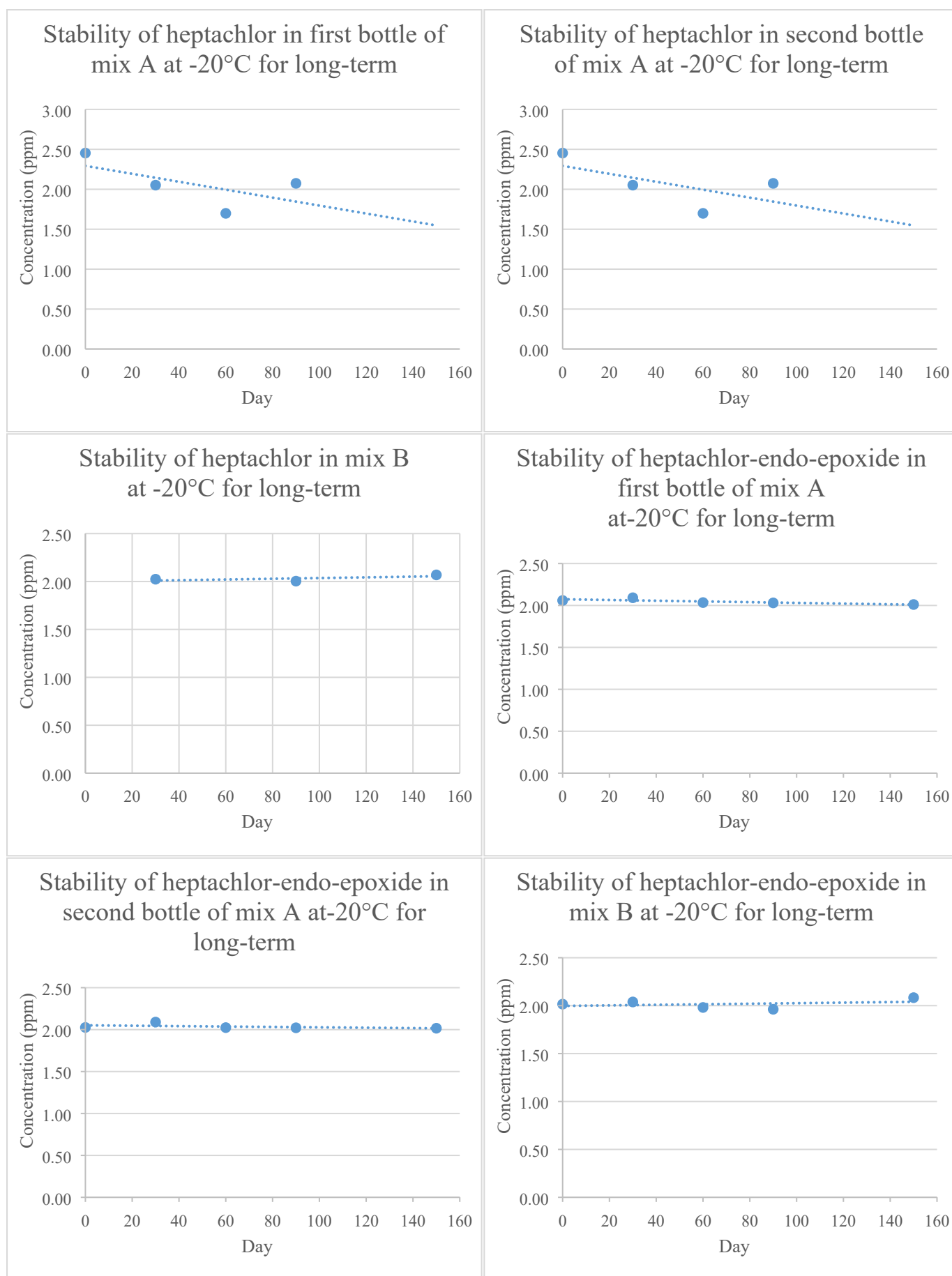


Fig. S7 The plots of gravimetric mixtures (A and B) in long-term stability at -20°C for each pesticide