



APMP-APLAC Joint Proficiency Testing Programme
(APLAC PT T094)
Pesticide residues in cabbage



**Asia Pacific Metrology Program (APMP) -
Asia Pacific Laboratory Accreditation Cooperation (APLAC)
Joint Proficiency Testing Programme
<APLAC PT T094>**

Pesticide residues in cabbage

Draft Final Report

Jointly Coordinated by:

Korea Research Institute of Standards and Science (KRISS)

and

Korea Laboratory Accreditation Scheme (KOLAS)

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Summary of Results

1. This proficiency testing programme (APLAC PT T094) was jointly organized by Korea Research Institute of Standards and Science (KRISS) and Korea Laboratory Accreditation Scheme (KOLAS). The aim of this proficiency testing programme is to demonstrate the capability of participating laboratories in measuring the amount of p,p' -DDE and α -endosulfan in kimchi cabbage by various analytical techniques.
2. A total of sixty six laboratories were nominated for p,p' -DDE and seventy laboratories were nominated for α -endosulfan from thirty four economies enrolled in the APLAC PT T094 programme. Fifty three laboratories for p,p' -DDE and fifty seven laboratories for α -endosulfan returned the analytical results within the scheduled deadline. Participants were confidentially assigned with unique laboratory codes (T094-01 to T094-70) as per ISO/IEC 17043 and the codes were used throughout the program.
3. The values certified by KRISS, the national metrology institute (NMI), were used as the assigned values for the APLAC PT T094 programme. The Horwitz Equation serves as a general mode to determine the standard derivations for proficiency assessment. The performance of participating laboratories would be assessed using z-scores against the assigned values in the program.
4. The z-scores of the participants are summarized as follows:

z-score	Number of Participants	
	p,p' -DDE	α -endosulfan
$ z \geq 3.0$	8 (15 %)	14 (25 %)
$2.0 < z < 3.0$	2 (4%)	4 (7 %)
$ z \leq 2.0$	43 (81 %)	39 (68 %)
Total	53 (100 %)	57 (100 %)



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1. Introduction

Kimchi cabbage is extensively used in fermented Asian dish and the most popular Brassica vegetable in the Asia-Pacific region. However, it is prone to be contaminated with various pesticides. Even though many countries set the regulation for pesticide residues, contamination by variety kinds of pesticides in agricultural products is always hot issues in food safety. Codex Alimentarius Commission (CAC) set maximum residue levels (MRLs) of DDE at 0.2 mg/kg for vegetable, and α -endosulfan at 1 mg/kg for Brassica vegetables [1]. European Commission (EC) set MRLs of both DDE and α -endosulfan at 0.05 mg/kg for Brassica vegetables [2]. In Korea, MRLs of both DDE and α -endosulfan is 0.2 mg/kg [3]. In addition, increase of international trade for food and agricultural products has brought the importance of traceable measurement of pesticide residues in agricultural products for ensuring food safety.

With the aim of enhancing the quality and traceability of measurements in various economies of the Asia-Pacific region through a better regional scientific infrastructure, the Asia-Pacific Metrology Programme (APMP) and the Asia Pacific Laboratory Accreditation Cooperation (APLAC) agreed to strengthen bilateral cooperation and established the APMP-APLAC Joint Proficiency Testing Working Group (PTWG). APLAC PT T094 is one of the initial two programmes conducted under the auspices of the PTWG both of which, were jointly organized by KRISS as a member of APMP and KOLAS as a member of APLAC.

The purpose of APLAC PT T094 programme was to demonstrate the capability of participating laboratories in measuring pesticide residues, *p,p'*-DDE and α -endosulfan, in a test sample of dried kimchi cabbage. The study material was freeze-dried kimchi cabbage powder fortified with two pesticides (*p,p'*-DDE and α -endosulfan). Each sample bottle contains 15 g of study material and each participant received one bottle of sample. Participants were asked to report results of both pesticides based on dry mass base (instruction for dry mass correction was provided on the study protocol). Samples were shipped to participants in packages with ice packs and participants were asked to store the sample at or below -20 °C before analysis.

The values certified by KRISS, the national metrology institute (NMI), using isotope dilution gas chromatography mass spectrometry (ID-GC/MS) as a primary method were used as the assigned values for the APLAC PT T094 programme. The Horwitz Equation serves as a general mode to determine the standard derivations for proficiency assessment and participants' performance was assessed using the z-score.



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APLAC PT T094 programme was conducted in accordance with the following schedule:

Event	Schedule
Call for participation	July 14, 2014
Deadline for registration	July 24, 2014
Distribution of samples	July 31, 2014
Deadline for submission of results	September 5, 2014
Issue of the interim report	January 7, 2015
Issue of the final report	February 28, 2015

2. Summary of participants

A total of sixty six laboratories were nominated for *p,p'*-DDE and seventy laboratories were nominated for α -endosulfan from thirty four economies enrolled in the APLAC PT T094 programme. Participants were confidentially assigned with unique laboratory codes (T094-01 to T094-70) as per ISO/IEC 17043 and the codes were used throughout the program. Fifty three laboratories for *p,p'*-DDE and fifty seven laboratories for α -endosulfan returned the analytical results to the proficiency testing provider within the scheduled deadline.

Six from total nominated laboratories (Lab code: T094-7, T094-15, T094-16, T094-28, T094-53, T094-62) did not submit both sample receipt forms and result report forms after sample distribution. Four laboratories (Lab code: T094-32, T094-33, T094-40, T094-50) replied to give up submitting the results in this PT programme. Three laboratories (Lab code: T094-11, T094-14, T094-70) did not received the study material because of some problems such as custom or wrong mail address. Lab code T094-44, T094-45 and T094-66 were accredited for α -endosulfan only, and T094-2 submitted the result of α -endosulfan only because of a problem of *p,p'*-DDE standard. Finally, fifty three results for *p,p'*-DDE and fifty seven results for α -endosulfan were submitted and used for the performance assessment as following table:

Analytes	Total number of nominated laboratories	Number of valid results submitted by participants
<i>p,p'</i> -DDE	66	53
α -endosulfan	70	57



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The list of participant laboratories and accreditation bodies:

No.	AB Name (Economies)	Participants nominated	Returned results
1	IANZ(New Zealand)	3	3
2	IARM(Macedonia)	1	1
3	OAA(Argentina)	1	1
4	Standards Malaysia (Malaysia)	3	3
5	Akkreditierung Austria (Austria)	1	1
6	RENAR (Romania)	1	0
7	ISRAC (Israel)	1	1
8	TUNAC (Tunisia) - ARAC	1	1
9	CAI (Czech)	2	2
10	UKAS (United Kingdom)	2	2
11	TAF (Taiwan)	3	3
12	HKAS (Hong Kong)	3	3
13	OSA (El Salvador)	2	1
14	ATCG (Montenegro)	1	1
15	COFRAC (France)	2	1
16	JAB (Japan)	2	2
17	KAN (Indonesia)	1	1
18	BELAC (Belgium)	2	2
19	OGA (Guatemala)	1	0
20	SCC (Canada)	1	1
21	CNAS (China)	3	2
22	RvA (Netherlands)	2	2
23	PAO (Philippine)	3	3
24	PCA (Poland)	2	2
25	DANAK (Denmark)	1	1
26	CAA (Croatia)	2	2
27	NABL (India)	4	3
28	HAB (Hungary)	1	1
29	IPAC (Portugal)	1	1
30	PAU (Palestine) - ARAC	3	0
31	DAC (United Arab Emirates) – ARAC	1	0
32	SEMAC (Morocco) - ARAC	1	0
33	EGAC (Egypt) - ARAC	1	0
34	KOLAS (Korea)	10	10
Total		70	57



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3. Assigned values and standard deviation for proficiency assessment

3.1 Assigned values

In accordance with the guidance provided by *ISO/IEC 17043:2010* [4] *Conformity Assessment – General Requirements for Proficiency Testing*, certified values as determined by definitive test or measurement methods can be used as the assigned values for proficiency test. Therefore, the values certified by KRISS, the national metrology institute (NMI), using isotope dilution gas chromatography mass spectrometry (ID-GC/MS) as a primary method were used as the assigned values for the APLAC PT T094.

3.2 Standard deviation for performance assessment

The Horwitz Equation [5] serves as a general mode to determine the standard derivations for proficiency assessment (σ_{pt}).

$$\sigma_{pt} = 0.02c^{0.8495}$$

where c is the assigned value of the analyte expressed as a dimensionless mass ratio (e.g. $1 \mu\text{g/g} = 1 \text{ ppm} = 10^{-6}$)

3.3 The assigned values and σ_{pt} are summarized as follows:

	Analytes	
	<i>p,p'</i> -DDE	α -endosulfan
Assigned value (mg/kg)	0.668	2.405
Standard uncertainty(mg/kg)	0.007	0.010
Coverage factor (k)	2.26	2.13
Expanded uncertainty (mg/kg)	0.017	0.021
Standard deviation for performance assessment, σ_{pt} (mg/kg)	0.114	0.337

4. Performance assessment

4.1 Participants' performance was assessed using the z-score, which is calculated as follows:



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$$Z = \frac{X_i - X}{\sigma_{pt}}$$

Where x_i = the reported result of the i^{th} participant
 X = the assigned value of the test material
 σ_{pt} = the standard deviation for proficiency assessment (in mg/kg) estimated from the Horwitz Equation

4.2 The z-Score is commonly interpreted as:

- (i) $|z| \leq 2.0$ Satisfactory
- (ii) $2.0 < |z| < 3.0$ Questionable
- (iii) $|z| \geq 3.0$ Unsatisfactory

Participants having $|z| \geq 3.0$ should thoroughly investigate their results. Participants having z-scores in the range $2.0 < |z| < 3.0$ are also encouraged to review their results.

5. Participants' results and Discussions

5.1. An overview of participants' results is summarized in the following table:

	<i>p,p'</i> -DDE	α -endosulfan
No. of Results	50	54
Assigned values	0.668 mg/kg	2.405 mg/kg
Median of Participants' Results	0.590 mg/kg	2.220 mg/kg
Average of Participants' Results	0.584 mg/kg	2.152 mg/kg
Robust average	0.591 mg/kg	2.179 mg/kg
Standard Deviation of Participants' Results	0.183 mg/kg	0.925 mg/kg
Robust Standard Deviation	0.099 mg/kg	0.506 mg/kg
Relative Standard Deviation of Participants' Results	31 %	43 %
Relative Robust Standard Deviation of Participants' Results	17 %	23 %
Relative Standard Deviation Estimated from the Horwitz Equation	17 %	14 %
Difference of the average from PTRV	-0.084 mg/kg	-0.253 mg/kg
Relative Difference of the average from PTRV	-13 %	-11 %



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Three participants (Lab code: T094-4, T094-60, T094-61) submitted reversed values of two pesticides, p,p' -DDE to α -endosulfan, and α -endosulfan to p,p' -DDE. These values were not included for the standard deviations for proficiency assessment and performance assessment (including Figure 3 and 4), that is why the number of results is fifty for p,p' -DDE, and fifty four for α -endosulfan in the table. The difference of the average from PTRV is -0.084 mg/kg (-12.6 %) for p,p' -DDE and -0.253 mg/kg (-10.5 %) for α -endosulfan. A negative bias of average of participant results from the assigned value was observed for both analytes.

5.2. Performance of participants, in terms of z-scores, is summarized as follows:

z-score	Number of Participants	
	p,p' -DDE	α -endosulfan
$ z \geq 3.0$	8 (15 %)	14 (25 %)
$2.0 < z < 3.0$	2 (4%)	4 (7 %)
$ z \leq 2.0$	43 (81 %)	39 (68 %)
Total	53 (100 %)	57 (100 %)

Results from thirty five participants were satisfactory with $|z| \leq 2.0$ for both analytes, and five participants were identified as having unsatisfactory results with $|z| \geq 3.0$ on both analytes.

5.3 Figures and tables for participants' results

Participants' results including mass fraction (mg/kg), combined standard uncertainty, coverage factor and expanded uncertainty and z-scores of p,p' -DDE and α -endosulfan are given in Tables 1 to 2 respectively. z-Scores underlined were considered as unsatisfactory (i.e. $|z| \geq 3.0$). The summaries of participants' methods are listed in Tables 3 and 4.

Figures 1 and 2 show the assigned values and the participants' reported results with associated expanded uncertainties, if provided. Figures 3 and 4 show the pattern of z-score distribution in ascending order for all participants. Scatter plot in figure 5 shows the distribution of participants' results. In figure 5,



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most sky blue diamond spots are around $|z| \leq 3$ square zone. However, six spots are far from $|z| = 3$ square zone. Four green triangle spots of z-score < -3 for both analyte, p,p' -DDE and α -endosulfan, could be suspicious for the recovery problem in their analysis procedure for pesticide residues. There are two curious spots. One orange square spot is in z-score < 2 for p,p' -DDE with satisfactory but in z-score > 5 for α -endosulfan with unsatisfactory, and the other red round spot is z-score > 4 for p,p' -DDE and in z-score < -3 for α -endosulfan with both unsatisfactory. These participants should inspect their validation system of the analysis procedure for pesticide residues.

5.4 Summary of participants' experiment methods

Most participants used one of three extraction methods, solid/liquid extraction (16 %), liquid/liquid extraction (32 %), and QuEChERS (40 %). One participant used a soxhlet method and the other described just shaking. All participants using QuEChERS and some participants using liquid/liquid extraction employed dispersive solid phase extraction (dSPE) as clean-up of matrix from cabbage. Fourteen participants adapted a florisil SPE cartridge for cleaning-up and the rest of them used other cartridges like GPC column, graphite SPE and amine SPE.

All participants used GC for separation of target pesticides. For quantifying the level of analyte pesticides, twenty two participants (37 %) used GC/ECD. Seventeen participants (30 %) and twelve participants (21 %) employed GC/MS/MS and GC/MSD. Three participants used both GC/ECD and GC/MSD and one participant used GC/ITD. Two participants described just GC.

Over half of participants (thirty two, 60 %) did not apply an internal standard for quantifying the analyte pesticide. Among participants using an internal standard, ten participants applied triphenylphosphate and the rest of them used diverse internal standards.

Most participants were accredited and only three participants were not accredited for both analyte. As described, three participants were accredited with α -endosulfan thus submitted the α -endosulfan result only.

6. Additional observations

- Lab code-20 did not submit the result with result form but just sent an email including two values



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without any other information.

- Lab code-58 submitted the result with the other result form.
- Lab code-2 nominated with both analyte but submitted only α -endosulfan because of a standard problem of p,p' -DDE.
- According to the results, many participants confused the water contents and corresponding correction factor for dry mass correction.

7. References

- [1] CODEX Alimentarius, Pesticide Residues in Food online database (36th Session, July 2013)..
- [2] EU Pesticides database
http://ec.europa.eu/sanco_pesticides/public/?event=homepage&language=EN
- [3] Korea Food and Drugs Administration, MRLs for Pesticides in Foods, April 2012.
- [4] ISO/IEC 17043:2010. Conformity Assessment – General Requirements for Proficiency Testing, International Standards Organization, Geneva, Switzerland, 2010.
- [5] Horwitz, W. Evaluation of analytical methods used for regulations of food and drugs, Anal. Chem., 1982, 54: 67A-76A.



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Table 1: Participants' results for *p,p'*-DDE

Lab. code	Mass fraction (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	z-Score	Accreditation status
T094-1	0.530	---	---	---	-1.22	N
T094-2	N/A					Y
T094-3	0.589	0.034	2	0.068	-0.70	Y
T094-4	3.44	0.08	2	0.16	<u>24.41</u>	<u>Y</u>
T094-5	0.51	0.00505	2	0.01	-1.47	Y
T094-6	0.536	0.0526	2	0.105	-1.16	Y
T094-7	***	***	***	***	***	Y
T094-8	0.39	0.06	2	0.12	-2.45	Y
T094-9	0.752	0.028	2	0.046	0.74	Y
T094-10	0.486	0.065	2.3	0.15	-1.60	Y
T094-11	***	***	***	***	***	Y
T094-12	0.215	0.015	2	0.014	<u>-3.99</u>	<u>Y</u>
T094-13	0.55	0.077	2	0.15	-1.04	Y
T094-14	***	***	***	***	***	Y
T094-15	***	***	***	***	***	Y
T094-16	***	***	***	***	***	Y
T094-17	0.668	0.045	2.140	0.097	0	N
T094-18	0.6	0.09	2	0.19	-0.60	Y
T094-19	0.65	---	---	---	-0.16	Y
T094-20	0.53	---	---	---	-1.22	Y
T094-21	0.581	0.036	2	0.071	-0.77	Y
T094-22	0.59	0.129	2	0.258	-0.69	Y
T094-23	0.552	0.031	2	0.061	-1.02	Y
T094-24	0.040	0.003	2	0.006	<u>-5.53</u>	<u>N</u>
T094-25	0.610	0.027	2	0.06	-0.51	Y
T094-26	0.56	---	2	0.11	-0.95	Y
T094-27	0.6	0.01863	1.96	0.028	-0.60	Y
T094-28	***	***	***	***	***	N
T094-29	0.5	---	---	---	-1.48	N
T094-30	0.89	0.08	2	0.16	1.96	N
T094-31	0.787	0.028	2	0.056	1.05	Y
T094-32	***	***	***	***	***	Y
T094-33	***	***	***	***	***	Y
T094-34	0.595	0.029	2.776	0.08	-0.64	Y
T094-35	0.616	0.087	2	0.175	-0.46	Y
T094-36	0.468	0.082	2	0.164	-1.76	Y
T094-37	0.7	0.1341	2	0.268	0.28	Y

Table 1: Participants' results for *p,p'*-DDE (Cont'd)

Lab. code	Mass fraction (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor <i>k</i> (95% level of confidence)	Expanded uncertainty (mg/kg)	z-Score	Accreditation status
T094-38	0.591	0.036	2	0.072	-0.68	Y
T094-39	0.136	0.018	2	0.037	-4.69	Y
T094-40	***	***	***	***		Y
T094-41	0.732	0.058	2	0.037	0.56	Y
T094-42	0.586	0.011	2	0.117	-0.72	Y
T094-43	0.723	0.043	2	0.087	0.48	Y
T094-44	N/A					Y
T094-45	N/A					N
T094-46	0.6	---	---	0.18	-0.60	Y
T094-47	1.14	---	---	---	4.16	
T094-48	0.58	0.0306	2	0.061	-0.78	Y
T094-49	0.897	0.224	2	0.448	2.02	Y
T094-50	***	***	***	***		Y
T094-51	0.83	0.16	2	0.32	1.43	Y
T094-52	0.515	0.082	---	---	-1.35	N
T094-53	***	***	***	***		Y
T094-54	0.630	0.027	2.380	0.06	-0.33	Y
T094-55	0.570	0.039	2.069	0.046	-0.86	Y
T094-56	0.50	0.125	2	0.25	-1.48	Y
T094-57	0.578	0.032	2	0.064	-0.79	Y
T094-58	0.184	0.01	2	0.027	-4.26	N
T094-59	0.476	0.043	2	0.086	-1.69	Y
T094-60	2.34	---	---	---	14.73	Y
T094-61	2.51	0.1	2	0.19	16.22	Y
T094-62	***	***	***	***		Y
T094-63	0.598	0.007	2	0.014	-0.62	Y
T094-64	0.550	---	---	---	-1.04	Y
T094-65	0.638	0.008	2	0.016	-0.26	N
T094-66	N/A					N
T094-67	0.61	0.034	2	0.067	-0.51	Y
T094-68	0.72	0.05	2	0.1	0.46	N
T094-69	0.711	0.178	2	0.356	0.38	Y
T094-70	***	***	***	***		Y

“***” The registered participant did not submit the results.

“---” Data or information was not provided.

N/A: Not applicable

Gray color lab codes did not included in performance assessment.



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Table 2: Participants' results for α -endosulfan

Lab. code	Mass fraction (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor k (95% level of confidence)	Expanded uncertainty (mg/g)	z-Score	Accreditation status
T094-1	2.510	---	---	---	0.31	N
T094-2	0.84	0.0273	2	0.055	-4.64	Y
T094-3	2.594	0.193	2	0.387	0.56	Y
T094-4	1.39	0.07	2	0.14	-3.01	<u>Y</u>
T094-5	1.399	0.0022	2	0.004	-2.98	Y
T094-6	1.88	0.306	2	0.612	-1.56	Y
T094-7	***	***	***	***	***	Y
T094-8	1.4	0.2	2	0.4	-2.98	Y
T094-9	4.14	0.1	2	0.2	5.15	Y
T094-10	3.090	0.599	2.4	1.44	2.03	Y
T094-11	***	***	***	***	***	Y
T094-12	0.54	0.034	2	0.043	-5.53	<u>Y</u>
T094-13	2.2	0.26	2	0.52	-0.61	Y
T094-14	***	***	***	***	***	Y
T094-15	***	***	***	***	***	Y
T094-16	***	***	***	***	***	Y
T094-17	2.433	0.036	2.14	0.078	0.08	N
T094-18	1.8	0.14	2	0.28	-1.79	Y
T094-19	2.75	---	---	1.38	1.02	Y
T094-20	1.97	---	---	---	-1.29	Y
T094-21	2.128	0.131	2	0.262	-0.82	Y
T094-22	2.219	0.567	2	1.133	-0.55	Y
T094-23	2.546	0.171	2	0.341	0.42	Y
T094-24	0.175	0.016	2	0.032	-6.61	<u>N</u>
T094-25	2.150	0.091	2	0.19	-0.76	Y
T094-26	2.1	---	2	0.42	-0.90	Y
T094-27	2.33	0.01905	1.96	0.105	-0.22	Y
T094-28	***	***	***	***	***	N
T094-29	2.3	---	---	---	-0.31	N
T094-30	2.618	0.13	2	0.26	0.63	N
T094-31	2.83	0.12	2	0.24	1.26	Y
T094-32	***	***	***	***	***	Y
T094-33	***	***	***	***	***	Y
T094-34	2.388	0.046	3.182	0.147	-0.05	Y
T094-35	1.719	0.13	2	0.26	-2.03	Y
T094-36	2.27	0.397	2	0.795	-0.40	Y
T094-37	2.0	0.1296	2	0.259	-1.20	Y



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Table 2: Participants' results for α -endosulfan (Cont'd)

Lab. code	Mass fraction (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor k (95% level of confidence)	Expanded uncertainty (mg/kg)	z-Score	Accreditation status
T094-38	2.476	0.151	2	0.302	0.21	Y
T094-39	0.364	0.048	2	0.096	-6.05	Y
T094-40	***	***	***	***	***	Y
T094-41	2.632	0.208	2	0.416	0.67	Y
T094-42	2.392	0.034	2.776	0.094	-0.04	Y
T094-43	1.90	0.079	2	0.158	-1.5	Y
T094-44	1.08	0.06	2	0.12	-3.93	Y
T094-45	2.027	0.2925	2	0.585	-1.12	Y
T094-46	2.24	---	---	0.67	-0.49	Y
T094-47	1.14	---	---	---	-3.75	
T094-48	2.22	0.09245	2	0.185	-0.55	Y
T094-49	2.773	0.693	2	1.387	1.09	Y
T094-50	***	***	***	***	***	Y
T094-51	3.9	0.82	2	1.6	4.43	Y
T094-52	2.14	0.43	---	---	-0.79	N
T094-53	***	***	***	***	***	Y
T094-54	2.380	0.088	2.26	0.2	-0.07	Y
T094-55	2.410	0.073	2.13	0.37	0.01	Y
T094-56	1.87	0.468	2	0.94	-1.60	Y
T094-57	1.842	0.068	2	0.136	-1.67	Y
T094-58	0.388	0.019	2	0.024	-5.98	N
T094-59	2.038	0.173	2	0.346	-1.09	Y
T094-60	0.89	---	---	---	-4.49	Y
T094-61	0.68	0.04	2	0.08	-5.12	Y
T094-62	***	***	***	***	***	Y
T094-63	2.406	0.022	2	0.043	0	Y
T094-64	1.970	---	---	---	-1.29	Y
T094-65	2.47	0.05	2	0.1	0.19	N
T094-66	0.903	0.016	2	0.032	-4.46	Y
T094-67	2.3	0.15	2	0.78	-0.31	Y
T094-68	5.92	0.39	2	0.78	10.43	N
T094-69	2.703	0.676	2	1.351	0.88	Y
T094-70	***	***	***	***	***	Y

“***” The registered participant did not submit the results.

“---” Data or information was not provided.

N/A: Not applicable

Gray color lab codes did not included in performance assessment.



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Table 3: Summary of participants' sample preparation methods

Lab. code	Extraction solvent(s)	Extraction technique	Clean up procedure
T094-1	Acetonitrile	Solid/Liquid (Shaking)	SPE(Florisil)
T094-2	Ethyl acetate	Solid/Liquid (Shaking)	SPE(PSA)
T094-3	Acetonitrile	QuEChERS	SPE
T094-4	Toluen	QuEChERS	Syringe filter
T094-5	Ethyl acetate	Liquid/Liquid (Vortex)	PSA & GCB
T094-6	Acetonitrile	QuEChERS	dSPE(PSA)
T094-7	***	***	***
T094-8	Ethyl acetate	Liquid/Liquid	dSPE
T094-9	Petroleum ether	Liquid/Liquid	Column with Florisil
T094-10	Acetonitrile	QuEChERS	No
T094-11	***	***	***
T094-12	Petroleum ether	Liquid/Liquid	Florisil
T094-13	Ethyl acetate	Solid/Liquid (Water bath)	SPE
T094-14	***	***	***
T094-15	***	***	***
T094-16	***	***	***
T094-17	Acetonitrile	Solid/Liquid (Shaking)	SPE(Florisil)
T094-18	Ethyl acetate, acetone & hexane	Liquid/Liquid	SAX/PSA
T094-19	Acetonitrile	QuEChERS	No
T094-20	Acetonitrile	---	---
T094-21	Acetonitrile	QuEChERS	dSPE(with amino sorbent PSA)
T094-22	Acetone/dicloromethane	Liquid/Liquid	GPC
T094-23	Ethyl acetate	Solid/Liquid (Vortex)	Dispersive Chromatography with PSA
T094-24	Hexane	Solid/Liquid (Shaking)	Florisil column
T094-25	Acetonitrile/dicloromethane	Liquid/Liquid	SPE(Florisil)
T094-26	Acetonitrile	QuEChERS	dSPE(PSA)
T094-27	Acetonitrile with 1% acetic acid	QuEChERS	PSA and MgSO ₄
T094-28	***	***	***
T094-29	Acetonitrile	QuEChERS	dSPE
T094-30	Acetonitrile/Hexane	Liquid/Liquid	No
T094-31	Acetonitrile	QuEChERS	MgSO ₄ , PSA, C18 and GCB
T094-32	***	***	***
T094-33	***	***	***

Table 3: Summary of participants' sample preparation methods (cont'd)

Lab. code	Extraction solvent(s)	Extraction technique	Clean up procedure
T094-34	Acetonitrile/acetone	Liquid/Liquid	SPE(Florisil)
T094-35	Acetonitrile	QuEChERS	dSPE(PSA)
T094-36	Acetonitrile	QuEChERS	dSPE(PSA:MgSO ₄ :C18)
T094-37	Acetonitrile	QuEChERS	dSPE
T094-38	Acetonitrile	QuEChERS	dSPE(PSA:MgSO ₄)
T094-39	Petroleum ether:dicloromethane (50:50)	Liquid/Liquid	GPC
T094-40	***	***	***
T094-41	Acetonitrile	Solid/Liquid (Shaking)	SPE(Florisil)
T094-42	Acetonitrile	Solid/Liquid (Shaking)	SPE(Florisil)
T094-43	Acetonitrile	QuEDhERS	dSPE(PSA:MgSO ₄)
T094-44	Acetonitrile/ Petroleum ether	Liquid/Liquid	Florisil
T094-45	Acetonitrile	Liquid/Liquid	SPE(Graphite : NH ₂)
T094-46	Dichloromethane, acetone and methylene chloride	Liquid/Liquid	None
T094-47	Dichloromethane	Liquid/Liquid	SPE
T094-48	Acetonitrile with 1% acetic acid	Solid/Liquid (Shaking)	SPE(PSA:MgSO ₄)
T094-49	Acetonitrile	QuEChERS	dSPE(Na ₂ SO ₄ :MgSO ₄)
T094-50	***	***	***
T094-51	Dichloromethane, Petroleum ether, acetone	Liquid/Liquid	None
T094-52	Hexane/dicloromethane	Soxhlet	SPE(Florisil)
T094-53	***	***	***
T094-54	Acetonitrile	Solid/Liquid (Shaking)	SPE(Amine)
T094-55	Acetonitrile/acetone	Liquid/Liquid	SPE(Florisil)
T094-56	Acetonitrile	QuEChERS	dSPE(PSA)
T094-57	Acetone, petroleum ether, dicloromethane	Liquid/Liquid	45 µm filter
T094-58	Acetonitrile	QuEChERS	dSPE(MgSO ₄ , PSA)
T094-59	---	---	---
T094-60	Acetonitrile with 1 % acetic acid	QuEChERS	dSPE(MgSO ₄ , PSA)
T094-61	Acetone : hexane (3:7)	Solid/Liquid	Florisil column
T094-62	***	***	***



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Table 3: Summary of participants' sample preparation methods (cont'd)

Lab. code	Extraction solvent(s)	Extraction technique	Clean up procedure
T094-63	Acetonitrile	Solid/Liquid	SPE(Florisil)
T094-64	Acetonitrile	Liquid/Liquid	SPE(Florisil, Aminopropyl))
T094-65	Acetonitrile	Shaking	SPE
T094-66	Acetonitrile	QuEChERS	dSPE with amino-sorbent and MgSO ₄
T094-67	Acetonitrile with 1 % acetic acid	QuEChERS	dSPE with amino-sorbent and MgSO ₄
T094-68	Acetonitrile	QuEChERS	SPE
T094-69	Acetone	Solid/Liquid	Filtration + liquid/liquid with isopropylether
T094-70	***	***	***



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Table 4: Summary of participants' methods

Lab. code	Analytical Instrument(s)	Name of Internal standard	Moisture content (%)	Method accredited	Methods of quantification
T094-1	GC/ECD	Not used	0.7960	N	Five point
T094-2	GC/ μ ECD	NIL	1.012	Y	Three point
T094-3	GC/MS/MS	Atrazine-d5	16.9	Y	Eight point
T094-4	GC/MS/MS	Not used	3.16	Y	Five point
T094-5	GC/MS/MS	Not used	0	Y	Three point
T094-6	GC/MS/MS	Triphenyl phosphate	1.110	Y	Three point
T094-7	***	***	***	***	***
T094-8	GC/MS/MS	Dimethachlor	0	Y	Multi-point
T094-9	GC	Not used	13.32	Y	Single point
T094-10	GC/ECD	Not used	0.737	Y	Three point
T094-11	***	***	***	***	***
T094-12	GC/ECD	Not used	98.84	Y	Six point
T094-13	GC/MSD	Triphenyl phosphate	0.61	Y	Three point
T094-14	***	***	***	***	***
T094-15	***	***	***	***	***
T094-16	***	***	***	***	***
T094-17	GC/ECD & GC/MSD	Not used	0.223	N	Three point
T094-18	GC/ μ ECD	Not used	8.0	Y	Five point
T094-19	GC/MS/MS	Triphenyl phosphate	---	Y	Six point
T094-20	---	---	---	---	---
T094-21	GC/MS	Triphenyl phosphate	4.7	Y	Three point, Matrix-matched
T094-22	GC/ μ ECD, GC/MS	Not used	6.5	Y	Two point
T094-23	GC/MS/MS	Not used	0.93	Y	Three point
T094-24	GC/ECD	Not used	2	Y	Five point
T094-25	GC/ECD	Not used	0.9556	Y	Seven point
T094-26	GC/MS	Triphenyl phosphate	11.4	Y	Three point
T094-27	GC/MS/MS	Triphenyl phosphate	11	Y	Six point
T094-28	***	***	***	***	***
T094-29	GC/MSD	Phenanthrene-d10 And Crysene-d12	---	Y	Five point
T094-30	GC/MS & GC/ECD	Not used	0.7182	Y	Seven point
T094-31	GC/MS/MS	Not used	4.40	Y	Single point
T094-32	***	***	***	***	***
T094-33	***	***	***	***	***
T094-34	GC/ECD/NPD	Not used	0.9570	Y	Six point
T094-35	GC/MS/MS	Triphenyl phosphate	0	Y	Matrix match multipoint
T094-36	GC/ITD	Not used	7.7	Y	Single point



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Table 4: Summary of participants' methods (cont'd)

Lab. code	Analytical Instrument(s)	Name of Internal standard	Moisture content (%)	Method accredited?	Methods of quantification
T094-37	GC/MS	ANA-d ₁₀ , PHE-d ₁₀ , CHR-d ₁₂ , PER-d ₁₂	12.6	Y	Five point
T094-38	GC/MS/MS	Triphenyl phosphate	0.6	Y	Five point
T094-39	GC/ECD	Not used	8	Y	Five point
T094-40	***	***	***	***	***
T094-41	GC/ECD	Not used	1.05	Y	Six point
T094-42	GC/ECD	Not used	0.0140	Y	Five point
T094-43	GC/MS	Not used	1.28	Y	Matrix matched, Four point
T094-44	GC/ECD	Not used	4.3	Y	Three point
T094-45	GC/MS	Not used	7.073	Y	Three point Matrix-matched
T094-46	GC/MS	Not used	1.3	Y	Single point
T094-47	GC	Not used	7.1	Y	Two point
T094-48	GC/MS/MS	Triphenyl phosphate	0.13	Y	Five point Matrix-matched
T094-49	GC/MS	Parathion-ethyl-d ₁₀	0	Y	Five point
T094-50	***	***	***	***	***
T094-51	GC/MS(Ion Trap)	Anthracene	16	Y	Standard addition
T094-52	GC/ECD	PCB29, PCB198, &Endo I-d ₄	4.8	Y	Three point
T094-53	***	***	***	***	***
T094-54	GC/MS/MS	Not used	1.01	Y	Five point
T094-55	GC/ECD/NPD	Not used	0.73	Y	Five point
T094-56	GC/MS/MS	Caffein	0	Y	Bracket, six point
T094-57	GC/MS/MS	PCB 153	16.1	Y	Seven point
T094-58	GC/ECD	Not used	No	---	---
T094-59	GC/ECD	---	10.85	Y	---
T094-60	GC/MS/MS	Not used	1.09	Y	Single point
T094-61	GC/ECD	---	0.4	Y	Single point, matrix matched
T094-62	***	***	***	***	***
T094-63	GC/ECD	Not used	0.92	Y	Five point
T094-64	GC	---	0.3290	Y	Three point
T094-65	GC/ECD	Not used	1.09	Y	Three point
T094-66	GC/MS	Tris(2-chloro-1-(chloro methyl)ethyl)phos	0	Y	Six point
T094-67	GC/ECD & GC/MSD	PCB 209 Triphenylphosphate	0.84	Y	Bracketing and five point
T094-68	GC/ECD	---	23.03	Y	Bracketing
T094-69	GC/MS/MS	Not used	1.85	Y	Three point
T094-70	***	***	***	***	***



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p,p'-DDE in Kimchi Cabbage

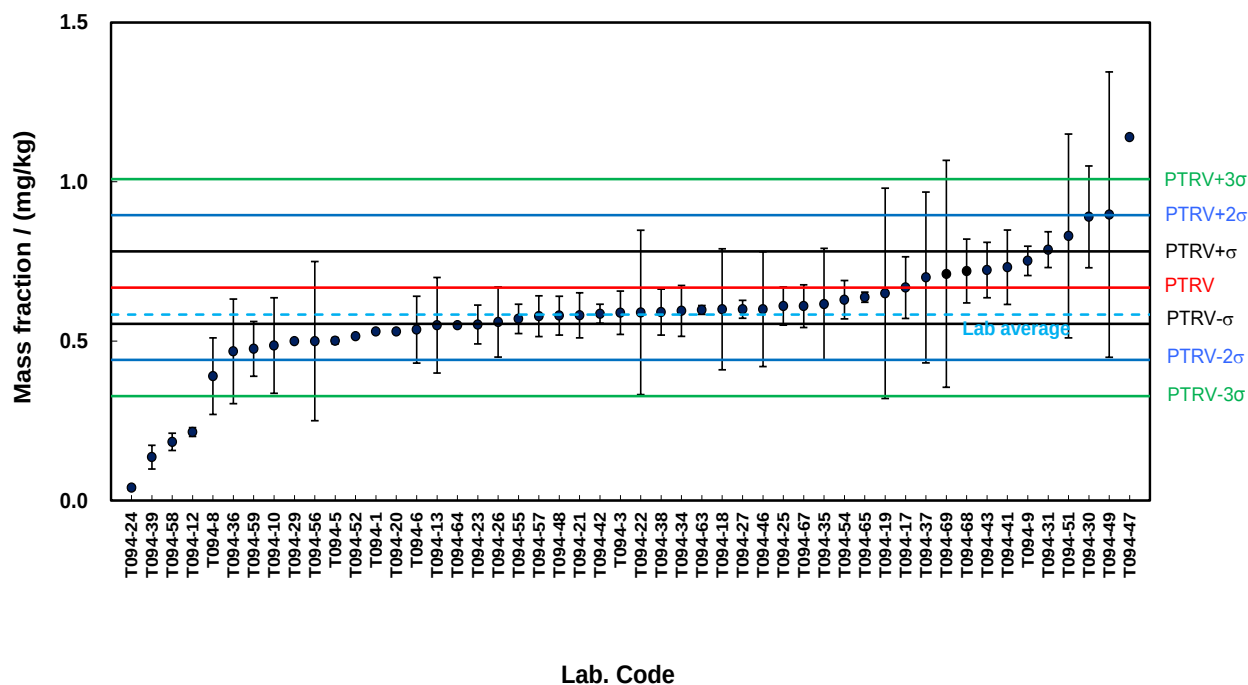


Figure 1 Participants' results of *p,p'*-DDE



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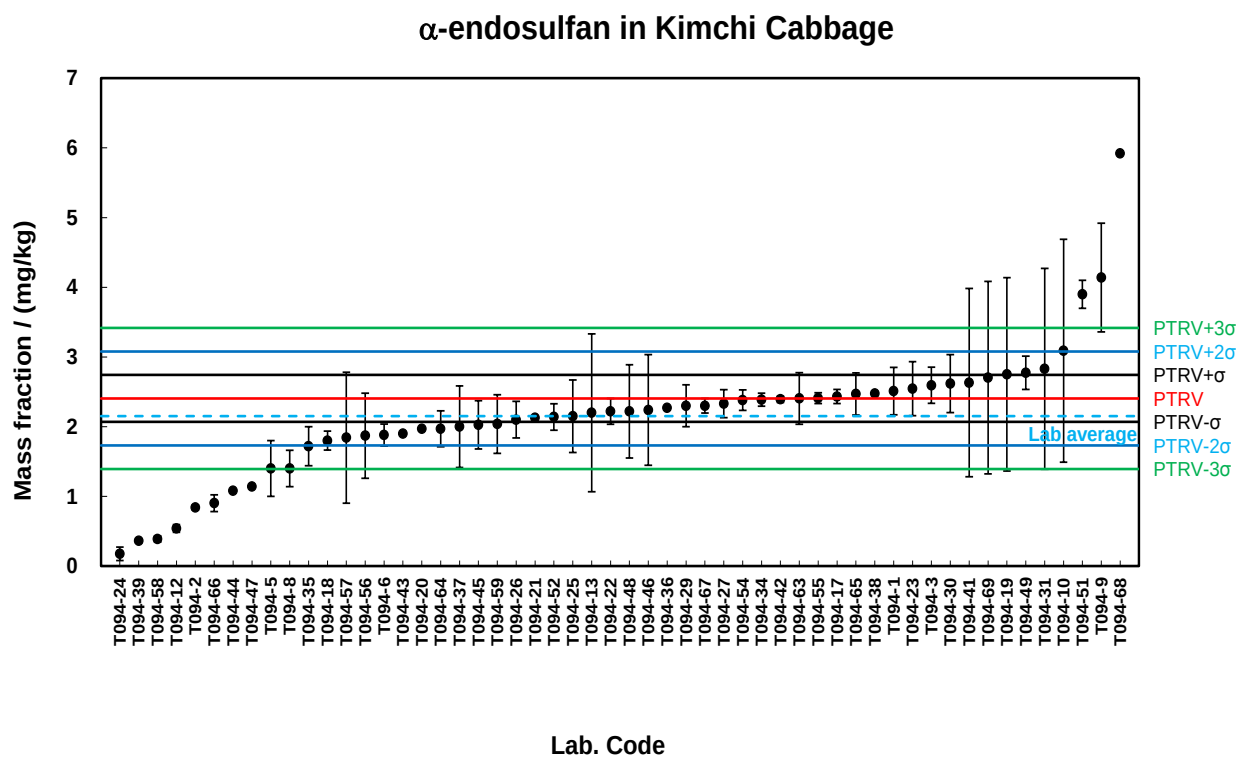


Figure 2 Participants' results of α -endosulfan



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Plot of z-scores of participants results on p,p' -DDE
(σ estimated from Horwitz equation)

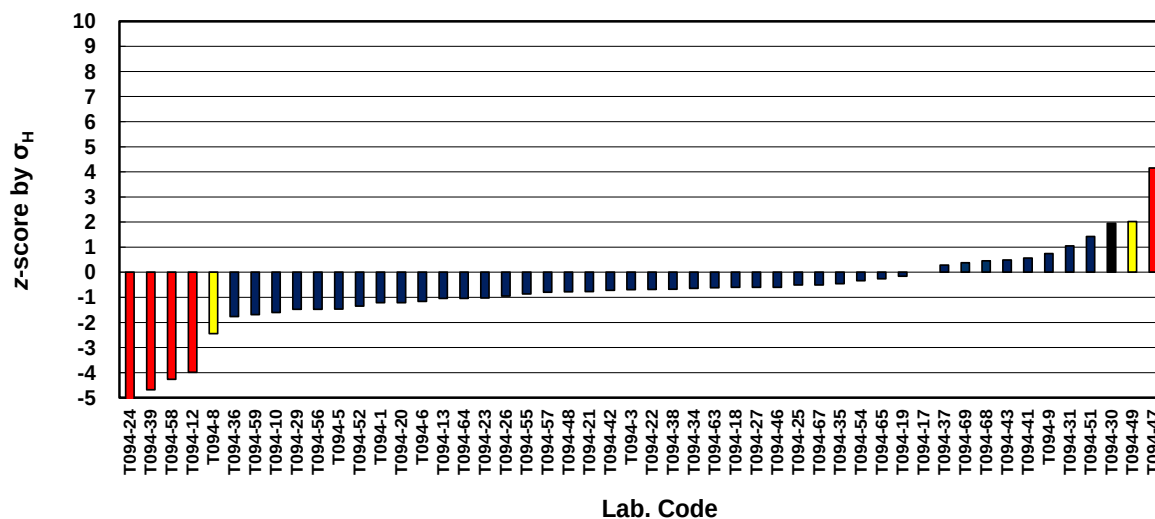


Figure 3 Plot of z-scores of participants' results on p,p' -DDE

Plot of z-scores of participants on α -endosulfan results
(σ estimated from Horwitz equation)

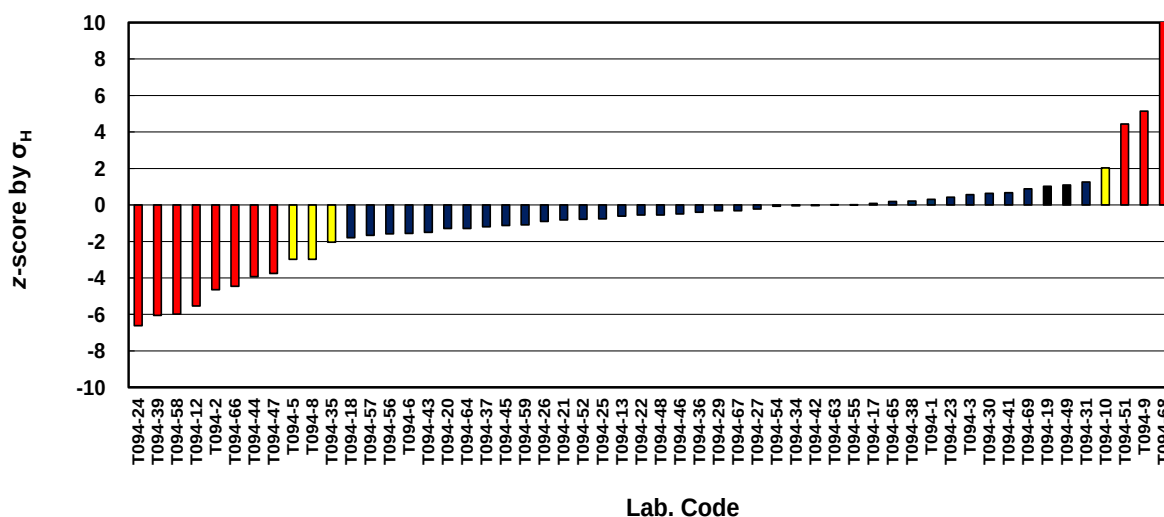


Figure 4 Plot of z-scores of participants' results on α -endosulfan

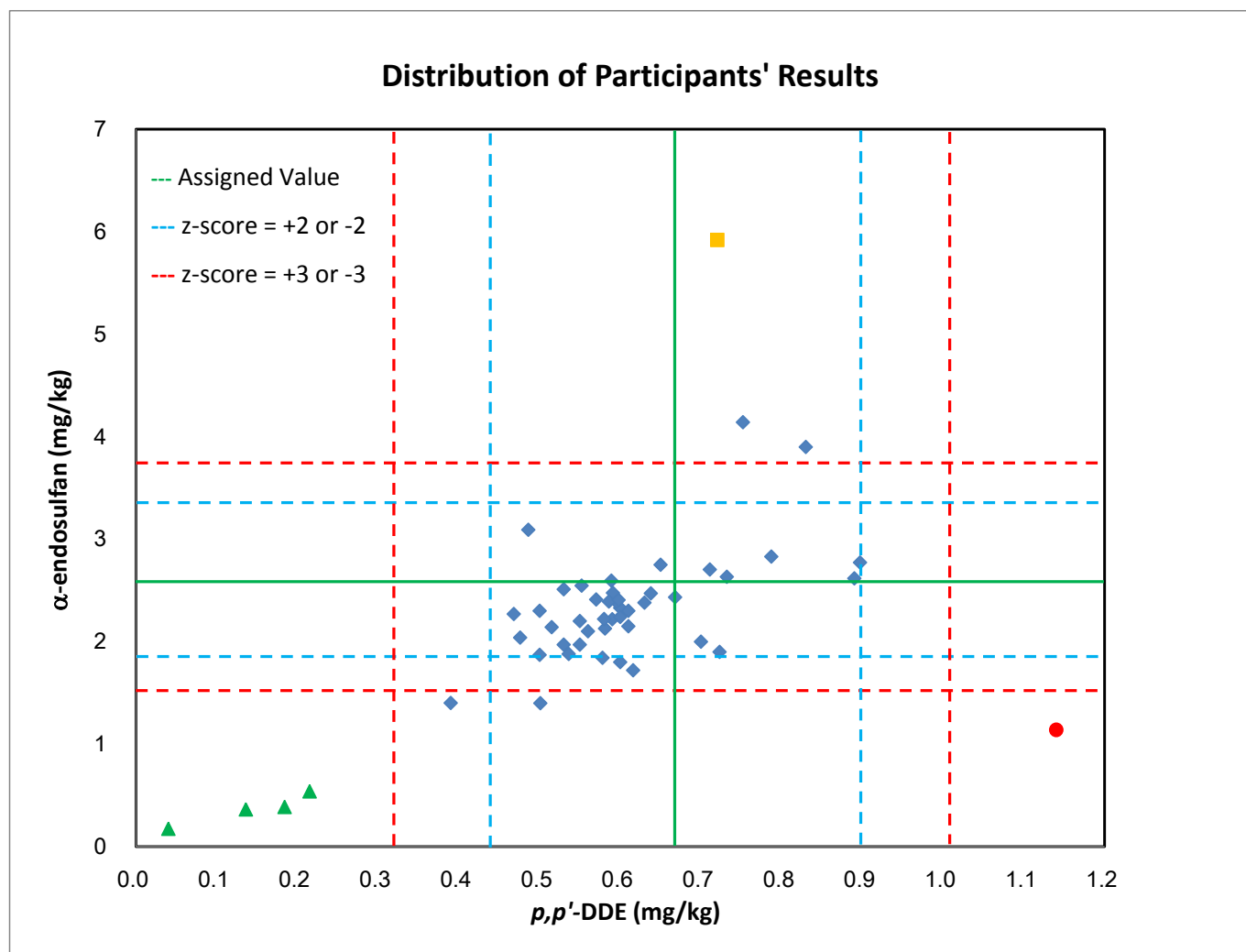


Figure 5 Scatter plot of mass fraction of p,p' -DDE and α -endosulfan



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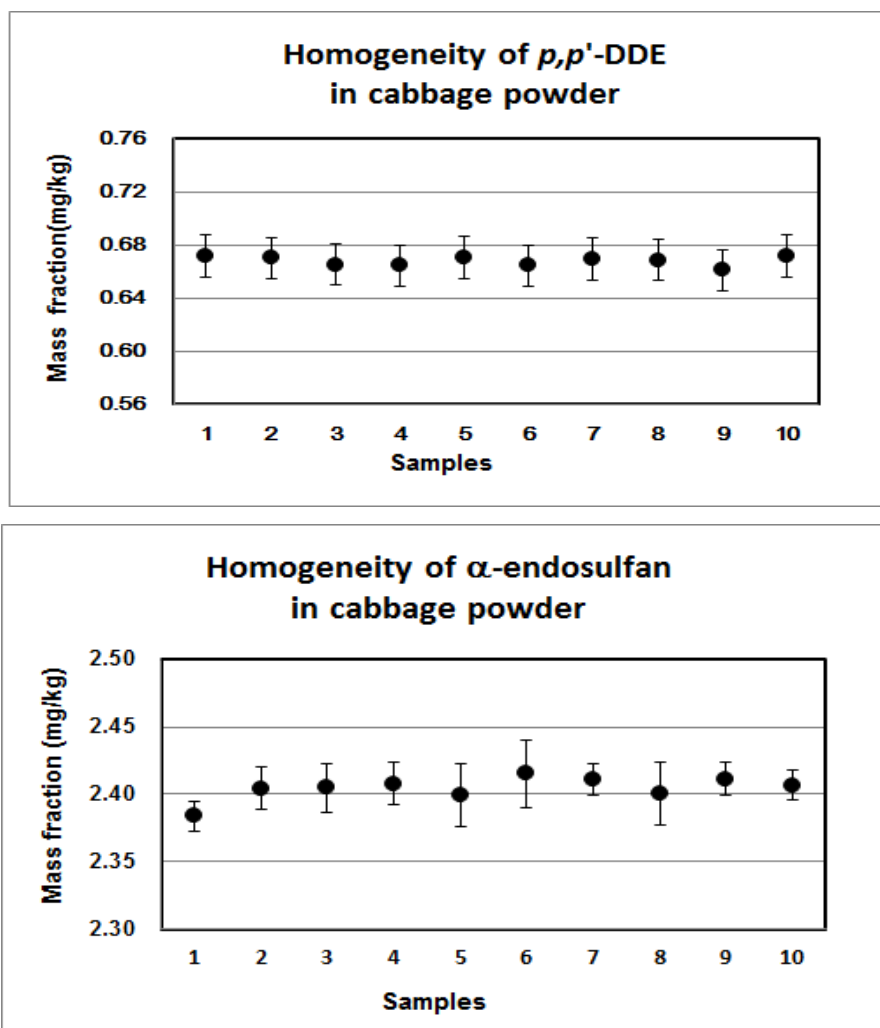
Appendix I. Homogeneity Test

The homogeneity study of the proficiency test sample was carried out using Isotope dilution Gas Chromatography Mass Spectrometry (ID-GC/MS) analysis after sample preparation. At least ten samples were taken with even interval following the bottling order and one subsample from each bottle was analysed with minimum sampling size of 1.0g. Cabbage powder sample was spiked with an isotope standard solution and then reconstitute with adding ten mL of water, equilibrated for two hours and extracted by shaking with ten mL of acetonitrile and ten mL of *n*-hexane. The *n*-hexane layer was further cleaned-up with a Florisil solid phase extraction cartridge. The extract was then analyzed by GC/MS. The GC/MS consists of a gas chromatography (Hewlett Packard 6890) and a double focusing magnetic sector mass spectrometer (Jeol JMS 700). The GC was equipped with a DB-5MS column (60cm long, 0.32 mm id, 0.25 μ m film thickness). One μ L of sample extract was loaded through its on-column injector. The relative standard deviations of *p,p'*-DDE and α -endosulfan among bottles were 0.5 % and 0.4 %, respectively, which are satisfactory for the present proficiency testing.

Samples for this PT program were produced 378 bottles from one batch cabbage powder; therefore, ANOVA test has not been done for homogeneity test. The relative standard deviations of *p,p'*-DDE and α -endosulfan among bottles were 0.5 % and 0.4 %, respectively, indicating good homogeneity of whole batch, thus it is not necessary to test for homogeneity of in bottle. This issue was published in the earlier literature of our laboratory as following: Kim, B., Park, S., Lee, I., Lim, Y., Hwang, E., So, H.-Y. (2010). Development of a certified reference material for the determination of acrylamide in potato chips. *Analytical Bioanalytical Chemistry*, 398, 1035-1042.

The between-samples standard deviation (S_s) was compared with the standard deviation for the proficiency assessment (σ_{pt}) and found to meet the requirement, $S_s \leq 0.3 \sigma_{pt}$, both for *p,p'*-DDE and α -endosulfan. Therefore, the samples may be considered to be adequately homogeneous based on ISO 13528 (International Standards Organization. ISO 13528:2005, Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons, ISO, Geneva, Switzerland, 2005). The plots showing the results of homogeneity assessments for analytes (*p,p'*-DDE and α -endosulfan) are shown in the following figures.

The plots for showing the homogeneity study for both analytes are shown in the following figures.



Appendix II. Stability Test

Based on stability tests of several batches of kimchi cabbage CRMs and PT materials produced by KRISS, p,p' -DDE and α -endosulfan were stable for more than three years at $-70\text{ }^{\circ}\text{C}$ and more than 6 months at $-20\text{ }^{\circ}\text{C}$ (storage condition recommended in the instruction for participants), for at least two months at $4\text{ }^{\circ}\text{C}$, and for at least two weeks at room temperature. The study materials for this PT were prepared in the same way as the previous batches and should be stable throughout the study period if participants followed the given instructions. Stability test for pesticide residue in cabbage CRM was published in literature, *Bulletin of the Korean Chemical Society*, (2011), 32, 1365-1367, "Stability Monitoring of Pesticide Residues in a Chinese Cabbage Certified Reference Material".



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Appendix III. Instruction for Participants

Instructions for Participants

1. Analysis of the proficiency test sample

(1) Sample information

- Participating laboratories will be provided with **ONE** bottle containing about **15g** of dried Kimchi cabbage powder. The date of dispatching of sample will be informed to participants. Participants are required to confirm the receipt of the samples(including the serial number of the sample, date of receipt, any damages found in the sample package) by e-mail(aplacpt.org@kriss.re.kr) using the sample receipt form attached in annex B. If any damage on the sample is observed on arrival, please contact us immediately.
- Analytes and their approximate mass fractions are given as follows:

Compound	Mass fraction (expected range of values)
<i>p,p'</i> -DDE	0.5 –10mg/kg
α -endosulfan	0.5 –10mg/kg

- Homogeneity: ID-GC/MS analysis of one subsample taken from each of 10 bottles was carried out. The relative standard deviations of *p,p'*-DDE and α -endosulfan among bottles were 0.5 % and 0.4 %, respectively, which are satisfactory for the present proficiency testing.
- Several other pesticides can be detected in the test sample, but please report only the mass fractions of the two target measurands (*p,p'*-DDE and α -endosulfan).

(2) Sample storage

- The proficiency test sample should be kept sealed in its original bottle and stored at -20 °C or lower temperature.
- According to our stability test, storing the sample at room temperature for a week did not change the level of the target analytes. However, storing at room temperature for a long term period should be avoided.
- Opening of the sample bottle should be carefully planned to avoid contamination and deterioration of the sample.
- For safety considerations, the proficiency test sample should be handled with care to prevent inhaling the sample powder and getting into eyes. In the case of accidental exposure to sample, wash the exposed areas with plenty of water and consult physicians when necessary.



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- For this proficiency testing programme, it is not required to return remaining sample.

(3) Guidelines for sample preparation and dry-mass correction

- Sample preparation:
 - Sample handling in a laboratory with well-controlled humidity and temperature is recommended and any kinds of contamination should be avoided.
 - Sample bottle should be equilibrated to ambient temperature prior to opening the cap (by placing the sample bottle to the weighing room at least overnight before sampling).
 - Sample in the bottle should be mixed thoroughly before taking a subsample from a bottle.
 - It is recommended that the minimum subsampling size for a single analysis is 1.0g (Homogeneity test was carried out with 1.0g of sampling).
 - Sample taken for analysis should be reconstituted with water before further sample preparation (equivalent to 10mL of water for 1g of dried kimchi cabbage powder).
 - Participants are recommended to perform at least triplicate measurements and report the mean and associated measurement uncertainty. A single result with its associated measurement uncertainty for each analyte shall be reported as specified in the Result Report. Participants should estimate the measurement uncertainty using their own practice.
- Dry-mass correction:
 - Participants should also carry out the dry mass correction.
 - Sampling for dry mass correction should be carried out at the same time with sampling for analysis.
 - At least three separate portions (with a recommended sample size of about 0.5g for each portion) of the sample should be taken and placed over P_2O_5 in a desiccator at room temperature for 7 days (168 hours).
 - Calculate the moisture content from the mass change observed in the three aliquots and use it for dry-mass correction.

(4) Measurement method

- Participants are expected to use the test method of their choice, which should be consistent with their routine procedures. If the laboratory is accredited, it is recommended to use the method listed in the scope of its accreditation.

2. Reporting and submission of results

Participants should complete the Result Report (Annex C). The manners of reporting test results are as followings:



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- For each analyte, the mean value of at least three independent measurements and its associated expanded uncertainty with 95% level of confidence should be reported on a dry mass basis;
- Report the mass fractions of analytes in mg/kg for *p,p'*-DDE and α -endosulfan; and
- Participants should provide information about the methods of analysis.

Participants should be aware that any submitted results are considered final and accordingly such results and units should be thoroughly checked before submission. Participants should submit the Result Report electronically to the coordinator of the proficiency testing programme (E-mail: aplacpt.org@kriss.re.kr) before the deadline. Results submitted after the deadline will not be accepted. Participants are reminded that the ability to report results in the specified unit and within the given time scale are part of the proficiency test.

The proficiency testing programme is conducted in the belief that participants will perform the analysis and report results with scientific rigour. Collusion and falsification of results are clearly against the spirit of the proficiency testing programme.

3. Contact

Participants may wish to contact the coordinator of the proficiency testing programme for any enquires, (E-mail: aplacpt.org@kriss.re.kr).

Dr. Seonghee Ahn
Principle Research Scientist
Center for Organic Analysis
Division of Metrology for Quality of Life
Korea Research Institute of Standards and Science (KRISS)
267 Gajeong-Ro, Yuseong-Gu, Daejeon 305-340, Republic of Korea
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Fax: +82-42-868-5800



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Appendix IV. Sample Receipt Form and Result Report

**Sample Receipt Form
(Participating Laboratories)**

Institute/
Laboratory:

Postal address:

Contact person:

Title

Given name

Surname

E-mail:

Print name /

Signature:

Date of receipt:

Confirmation of Package Content

Please choose the state of the sample: ☐ intact

☐ broken

☐ others: _____

Please write the serial numbers of the sample bottle: _____

Please complete this form and return it to Dr. Seonghee Ahn, KRIS(E-mail: aplacpt.org@kriss.re.kr) by email after receipt of the sample.



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Result Report

Institute/
Laboratory:

Postal address:

Contact person:

Title

Given name

Surname

E-mail:

Laboratory
accreditation:

YES (based on ISO 17025/ ISO 9000 series/ GLP/ under
the law of your economy(please specify)) /NO

If yes, please specify the name of accreditation body. _____

1. Analytical results

1) Mass fractions of p,p' -DDE and α -endosulfan in Kimchi cabbage

Analyte	Analytical result (mg/kg)	Combined standard uncertainty (mg/kg)	Coverage factor k (95% level of confidence)	Expanded uncertainty (mg/kg)
p,p' -DDE				
α -endosulfan				

Notes: (i) Report the analytical results and associated uncertainties in the unit mg/kg; (ii) Report the analytical results on a dry mass basis.



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2) Results from individual aliquots (dry-mass corrected)

Aliquot number	<i>p,p'</i> -DDE (mg/kg)	α -endosulfan(mg/kg)
1		
2		
3		
Average		
Standard deviation		
Standard deviation of the mean		

Please insert more lines for more determinations.

3) Dry Mass Correction

Moisture contents (weight %) : _____

Correction factor: _____

2. Methods of analysis

1) Sample size used for analysis _____g

2) Extraction method (e.g., Liquid/Liquid extraction, Soxhlet, etc.)

(Please briefly describe the extraction procedures)

3) Solvent used for extraction _____

4) Duration of extraction _____

5) Post extraction clean-up method and the transformation procedure, if any.

(e.g. SPE, GPC, etc, please briefly describe the clean-up and transformation procedures)



APMP-APLAC Joint Proficiency Testing Programme
(APLAC PT T094)
Pesticide residues in cabbage



6) Analytical instrument(s) used _____
(Specify the model)

7) Chromatographic column used _____
(Specify the dimensions)

8) The chromatographic conditions
(Please describe GC oven temperature programme, LC Mobile phase gradient, etc)

9) Methods of quantification(s)
(e.g., single-point, bracketing, three-point calibration curves, etc.)

10) Internal standard (if used)
(Specify the compounds)

11) Sources of calibration standards (with the purity/concentration and associated uncertainties)



Date(dd/mm/yyyy): / /