

Development and validation of an analytical method for the quantification of 2,6-diisopropylnaphthalene in agricultural products using GC-MS/MS

Han Sol Lee¹, Ji-Su Park¹, Su Jung Lee¹, Hye-Sun Shin¹, Yun mi Chung², Ha na Choi²,
Sang Soon Yun¹★, Yong-hyun Jung¹, and Jae-Ho Oh¹

¹Pesticide and Veterinary Drug Residues Division, Food Safety Evaluation Department, National Institute of Food and Drug Safety Evaluation, Ministry of Food and Drug Safety, Osong, Cheongju, Chungcheongbuk-do 28159, Korea

²Hazardous Substances Analysis Division, Gwangju Regional Food and Drug Administration, Gwangju 61012, Korea

(Received August 24, 2020; Revised October 8, 2020; Accepted October 9, 2020)

Abstract: An analytical method was developed and optimized for the quantification of a plant growth regulator, 2,6-diisopropylnaphthalene (2,6-DIPN), in agricultural products using gas chromatography-tandem mass spectrometry. The samples were extracted, partitioned, and were purified using a Florisil® cartridge. To validate the analytical method, its specificity, linearity, limit of detection (LOD) and limit of quantification (LOQ) of the instrument, LOQ of the analytical method (MLOQ), accuracy, and repeatability were considered. The method displayed excellent results during validation, and is suitable for the determination and quantification of the low residual levels of the analyte in the agricultural samples. All of the results with the optimized method were satisfactory and within the criteria ranges requested in the Codex Alimentarius Commission guidelines and the Ministry of Food and Drug Safety guidelines for pesticide residue analysis. The developed method is simple and accurate and can be used as a basis for safety management of 2,6-DIPN.

Key words: GC-MS/MS, 2,6-diisopropylnaphthalene, plant growth regulator, agricultural product, analytical method

1. Introduction

2,6-Diisopropylnaphthalene (2,6-Di(propan-2-yl)naphthalene; 2,6-DIPN, *Fig. 1*) is an odorless white crystalline solid. It is a hydrocarbon and a derivative of naphthalene. Theoretically, 10 positional isomers of diisopropylnaphthalene, such as 1,2-, 1,6-, and 2,7- are possible.¹ 2,6-DIPN is a synthetic plant growth regulator and has been classified as a biochemical pesticide. It acts through hormonal action to suppress

the sprouting of stored potatoes during their production and transport and is used to control their freshness and increase in their production rates. Further, this hydrocarbon is structurally and functionally similar to the other naturally occurring sprout inhibition agents present in plant tissues, such as 1-isopropyl-4,6-dimethylnaphthalene.^{2,3}

The maximum residue limit (MRL) for 2,6-DIPN was established to be 0.5 mg/kg in potatoes by the Ministry of Food and Drug Safety (MFDS) of the

★ Corresponding author

Phone : +82-(0)43-719-4211 Fax : +82-(0)43-719-4200

E-mail : yss0520@korea.kr

This is an open access article distributed under the terms of the Creative Commons Attribution Non-Commercial License (<http://creativecommons.org/licenses/by-nc/3.0>) which permits unrestricted non-commercial use, distribution, and reproduction in any medium, provided the original work is properly cited.

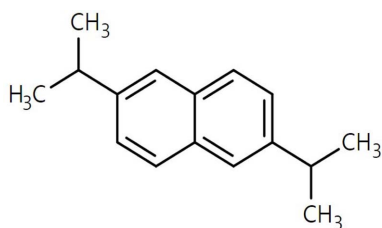


Fig. 1. Molecular structure of 2,6-DIPN.

Republic of Korea, and other foreign regulatory authorities, including the US Environmental Protection Agency (US EPA) and the Japan Food Chemical Research Foundation (JFCRF) have set the MRL for 2,6-DIPN at 0.02 to 6.0 mg/kg (16 items including potatoes and milk) and 0.5 to 1.0 mg/kg (17 items including potatoes and milk), respectively. However, the Codex Alimentarius Commission and the European Commission have not yet established the MRL for 2,6-DIPN in food and feed of plant or animal origin. While the MFDS and JFCRF have defined the risk assessment for the presence of residual 2,6-DIPN based on the parent compound alone, the US EPA defined this measurement to include residual 2,6-DIPN as well as its metabolites and degradants.⁴⁻⁸

The toxicity of 2,6-DIPN was evaluated by the US EPA, which included the investigations of the subchronic toxicity study in rats as the toxicological endpoint. The subchronic toxicity study determined the NOAEL (no observed adverse effect level) to be 104 mg/kg/day. The LOAEL (Lowest observed adverse effect level) is 208 mg/kg/day, and decrease in body weight gain and amount of feed intake and increase in organ weights of adrenal gland and kidney were observed. Consequently, the NOAEL derived from the subchronic toxicity study was assessed as the endpoint for short- and intermediate-term dietary assessments and a reference dose (RfD) was determined to be 1 mg/kg/day by dividing the 104 mg/kg/day into a 100-fold uncertainty factor.³

In the USA and European Union, the presence of residual 2,6-DIPN has been examined in recycled paper and board products used as food packaging materials, to assess the possibilities for the potential migration of this material into food via direct contact

or gas phase transport.⁹ However, the establishment of the MRL of 2,6-DIPN requires an analytical method to analyze the amount of the plant growth regulator applied to the crops. Therefore, the objective of this study was to develop and validate an analytical method for the detection and analysis of the residual 2,6-DIPN in five agricultural products (hulled rice, potato, soybean, mandarin, and green pepper) for enabling safety management in imported and domestic agricultural products.

2. Experimental

2.1. Chemicals and samples

The pesticide analytical standard of 2,6-DIPN was purchased from Sigma Aldrich (Buchs, Switzerland) and HPLC-grade acetonitrile, acetone, and ethyl acetate were sourced from Merck (Darmstadt, Germany). Magnesium sulfate anhydrous (MgSO₄) and sodium chloride (NaCl) were supplied by Junsei (Tokyo, Japan) and Merck (Darmstadt, Germany), respectively. Florisil[®] SPE cartridges (1 g, 6 cc) were procured from Waters (Milford, MA, USA) and the PTFE syringe filter (0.2 μm × 13 mm) was obtained from Teknokroma (Barcelona, Spain). The samples used in the experiment were hulled rice, potato, soybean, mandarin, and green pepper. The impurity limits for all of these agricultural products were proposed by MFDS and were used to verify the analytical method for the residual pesticide content in all of the agricultural products purchased, which were not treated with pesticides. Approximately 1 kg of brown rice and soybean were pulverized and passed through a standard sieve of 420 μm, and approximately 1 kg of potato, mandarin, and green pepper, respectively, were chopped and homogenized. The processed samples were preserved in a plastic bottle and stored at -50 °C until use.

2.2. Preparation of standard solutions

A stock solution of 2,6-DIPN at a concentration of 1,000 μg/mL was prepared in acetone, and intermediate solutions were prepared by further dilution of the stock solution with the same solvent to concentrations

of 100 and 10 µg/mL, respectively. Calibration solutions were prepared by serially diluting the intermediate standard solutions with acetone to yield solutions of concentrations of 0.0125, 0.025, 0.05, 0.125, 0.25, 0.625, and 1.25 µg/mL, respectively. The matrix-matched solutions for the quantification were prepared by dilution of the calibration solutions with the extracts of the untreated samples and included more than 90 % of the matrix. The standard stock solution was stored under refrigeration (-20 °C) in amber glass vials, and the working solutions were freshly prepared before each analysis.

2.3. Analysis conditions for GC-MS/MS

The analysis of 2,6-DIPN was conducted with an Agilent Technologies 7890B gas chromatograph (California, USA) equipped with an Agilent Technologies 7010 GC/MS Triple Quad mass spectrometer (California, USA). The analyte was separated on a HP-5MS column (0.25 mm I.D. × 30 m, 0.25 µm particle size, Agilent Technologies, CA, USA). The column temperature was programmed as follows: the initial temperature was 60 °C, which was held for 1 min. The temperature was increased to 180 °C at 30 °C/min, followed by an increase to 220 °C at 10 °C/min. Finally, the temperature was increased to 300 °C at 40 °C/min and was then held for 2 min. The total run time was 13 min. Helium was used as the carrier gas at a flow rate of 1 mL/min. The injector temperature was maintained at 280 °C, and the injection volume was 1 µL in the splitless mode. In the ion source of the instrument was used in its positive electron ionization mode (EI+) with 70 eV of electron energy for ionizing the analyte and its fragmentation into smaller ions. The ion source and interface temperatures were 230 °C and 280 °C, respectively.

2.4. Sample preparation

The processed sample (5 g) was weighed precisely and transferred to a 50 mL Teflon centrifuge tube. A mixture of acetonitrile/ethyl acetate (20 mL, 50/50, v/v) was added into the tube, which was then shaken for 10 min with a shaker (Tokyo Rikakikai Co. Ltd., Eyela MMV-1000W, Bunkyo, Japan), followed by

which, 4 g of MgSO₄ and 1 g of NaCl were added and the samples were hand-shaken for 1 min, followed by centrifugation for 10 min at 4,000 G. Florisil® cartridges were conditioned with 5 mL of the acetonitrile/ethyl acetate (50/50, v/v) mixture, and were then loaded with 5 mL of the supernatant of the extracts and taken into the 15 mL Teflon centrifuge tube. Finally, 2,6-DIPN was eluted with 5 mL of the acetonitrile/ethyl acetate (50/50, v/v) mixture, and the eluent was mixed and filtered through a syringe filter prior to GC-MS/MS analysis.

2.5. Method validation

To validate the analytical method, its specificity, linearity, limit of detection (LOD) and limit of quantification (LOQ) of the instrument, LOQ of the analytical method (MLOQ), accuracy, and repeatability were considered and assessed by Codex Alimentarius Commission guidelines¹⁰ for pesticide residue analysis and the MFDS guidelines¹¹ on the standard procedures for preparing an analysis method. The specificity of the method was compared through blank samples, and the blank samples spiked with 2,6-DIPN to verify that the analysis results do not contain any interfering peaks at the retention times of the standards. Linearity was evaluated by the coefficient of determination (R^2) of a matrix-matched calibration curve, calculated in the 0.00125-0.125 µg/mL range. The LOD and LOQ of the instrument were defined as the signal-to-noise ratios (S/N) at three and ten times, respectively. The MLOQ was calculated according to the LOQ of the instrument, and the amounts of the sample and extraction solvent, and the dilution factor. The MLOQ was evaluated as follows:

$$\text{MLOQ} = \text{LOQ of the instrument (}\mu\text{g/mL)} \times (\text{amount of the extraction solvent/amount of the sample}) \times \text{dilution factor}^1$$

¹dilution factor = total amount of the eluents through the cartridge/amount of the sample extracts into cartridge

The accuracy was assessed in terms of recovery by fortifying the blank samples at three spiking levels (0.01 mg/kg (LOQ), 0.1 mg/kg (10 × LOQ) and 0.5 mg/kg (50 × LOQ)), and with five replicates of each.

Table 1. Molecular weight (MW), Exact mass, precursor and product ion, collision energy (CE), and retention time (RT) of 2,6-DIPN for GC-MS/MS in positive electron ionization mode (EI⁺)

Compound	MW	Exact mass	Precursor ion (<i>m/z</i>)	Product ion (<i>m/z</i>)	CE (eV)	RT (min)
2,6-DIPN	212.3	212.16	197	155 ^a	12	8.3
			212	155	22	
			212	197	18	

^aQuantification ion

The repeatability was estimated from the coefficient of variation (CV%) of the samples from the inter-laboratory analyses.

3. Results and Discussion

3.1. Instrumental conditions

2,6-DIPN is a neutral and relatively non-polar compound and is highly volatile, with a vapor pressure of 81.3 mPa (25°C).² Therefore, gas chromatography was judged to be the suitable technique for analyzing the target analyte. For compound detection, tandem mass spectrometry (MS/MS) was selected, which has relatively high selectivity and assures analytical sensitivity even at low concentration levels. The separation of the 2,6-DIPN from the extracts was carried out using an HP-5MS column that is commonly used for pesticide analysis. Optimization of the precursor ions, product ions, and the collision energy was conducted via 1 µL injection of the standard solution (0.1 µg/mL) into the GC-MS/MS. First, a Q3 scan was performed under the typical 70 eV electron impact ionization to select the optimal precursor ions for the selected-ion monitoring (SIM) analysis through the total ion chromatogram (TIC) and mass spectrum. The base peak was identified at 197 *m/z* due to CH₃ radical loss. The next largest mass spectrometric peak was observed at 212 *m/z*, which is the exact mass of 2,6-DIPN, and the third-largest peak was obtained at 155 *m/z* due to a loss of 57 mass units corresponding to the C₄H₉ group.¹ Next, the optimal conditions for multiple reaction monitoring (MRM) were confirmed during the MS/MS analysis to maximize the selectivity and detection strength of the analysis. Based on the two ions (197 and 212) obtained

through the Q3 scan, the product ions were examined at the collision energies across 5 to 50 eV. The fragment ions at 197 and 212 *m/z* showed the highest intensities at 141, 155, and 182 *m/z*, and 155, 169, and 197 *m/z*, respectively. The most intense transition was used for the quantification, while the second and third most intense transitions were employed for confirmation as follows: (transition 1) 197→155; (transition 2) 212→197; and (transition 3) 212→155. The optimum collision energy for each transition is presented in Table 1.

3.2. Optimization of sample extraction and partitioning

While considering the extraction of the residues from the samples containing water, most of the pesticide residues are typically present on the surface or on the inside of the sample, which is hydrated. When non-polar organic solvents that do not mix well with water are directly used as the extraction solutions, the permeability of the pesticide residues into the extraction solvents is very low, and their extraction is not sufficient. Therefore, water-soluble organic solvents such as acetonitrile and methanol, are commonly used. In the case of dried samples such as cereals, the extraction efficiency with the water-soluble organic solvent is low at the dried state. Also, low polar pesticides are rather strongly adsorbed by dry samples. Therefore, a certain amount of water was added to the samples before the addition of the extraction solvent and the waiting period for the sample to get sufficiently wet.¹²

2,6-DIPN is relatively non-polar with an octanol-water partition coefficient (log P_{ow}) of 5.45, and therefore, it does not dissolve well in water-soluble

Table 2. Extraction efficiency of 2,6-DIPN according to ethyl acetate ratio in acetonitrile and methanol solvent

Extraction solvent	Recovery \pm SD ^a (%)	Extraction solvent	Recovery \pm SD (%)
ACN ^b /EA ^c = 100/0	75.8 \pm 0.5	MeOH ^d /EA = 100/0	78.6 \pm 0.1
ACN/EA = 90/10	86.4 \pm 6.1	MeOH /EA = 90/10	103.2 \pm 24.3
ACN/EA = 70/30	89.8 \pm 6.5	MeOH /EA = 70/30	103.6 \pm 18.5
ACN/EA = 50/50	96.9 \pm 2.2	MeOH /EA = 50/50	109.3 \pm 17.1

^aStandard deviation^bAcetonitrile^cEthyl acetate^dMethanol

organic solvents. Thus, we mixed the water-soluble organic solvent and the non-polar organic solvent to facilitate penetration into the sample, for assessing the extraction efficiency of 2,6-DIPN, for selecting the optimal conditions according to ethyl acetate ratio in acetonitrile and methanol. When acetonitrile and methanol were used as the solvent individually, the recoveries were the lowest at 75.8 % and 78.6 %, respectively, and when two solvents were mixed with ethyl acetate in 50 % ratios, the recoveries were the highest at 96.9 % and 109.3 %, respectively (Table 2). However, regardless of the ratio of methanol and ethyl acetate, the standard deviation was quite high. Therefore, the extraction of the target compound with a mixture of acetonitrile and ethyl acetate in a 50:50 ratio was considered better.

The efficiency of the partitioning reagent during the extraction step was tested by adding MgSO₄ and NaCl into the hulled rice samples. When the two experiments were carried out with 4 g of MgSO₄ and 1 g NaCl, and 6 g of MgSO₄ and 1.5 g NaCl, respectively, no substantial differences in the recoveries were observed when the reagent used was less (90.8 \pm 2.0 %) or more (93.9 \pm 5.6 %). Therefore, the partitioning step was conducted using 4 g of MgSO₄ and 1 g NaCl to remove water and the interferences from the sample and to separate the analyte from the aqueous layer into the organic layer.

3.3. Optimization of sample purification

The Florisil® SPE cartridges (1 g, 6 cc) were optimized for removing the interfering compound while acquiring with acceptable recovery. Florisil®

is used to adsorb low to moderately polar substances from non-aqueous solutions and commonly used for pesticide analysis in food and environmental samples. To obtain a more purified solution, the extract was concentrated and re-dissolved in organic solvents with a low elution strength such as n-hexane, and the reconstituted sample was loaded into the cartridge. Then, the elution was carried out with the solvent used for re-dissolving the sample or one with higher elution strength. However, 2,6-DIPN was volatilized by 78.2 \pm 2.3 % ($n=10$) during the evaporation. Therefore, the evaporation process was not included in the analytical method, and the elution of 2,6-DIPN was confirmed by applying the original extract to the florisil cartridge. First, 5 mL of the extract was loaded into the cartridge, which was conditioned with 5 mL of the acetonitrile/ethyl acetate (50/50, v/v) solution and the eluent was collected (Fraction 1). Subsequently, 5 mL of the acetonitrile/ethyl acetate (50/50, v/v) solution was added into the cartridge twice, and the respective eluents were collected (Fraction 2 and 3). The recoveries for each fraction were compared, and the sum of the recovery of the eluent obtained from fractions 1 and 2 was 92.7 % (Table 3). Thus, 2,6-

Table 3. Recovery results of 2,6-DIPN from fraction of cartridge

Elution solvent	Fraction	Recovery (%)
Acetonitrile	1 (Loading 5 mL)	68.9
/ethyl acetate	2	23.8
= 50/50	3	- ^a
Total		92.7

^aNot detected

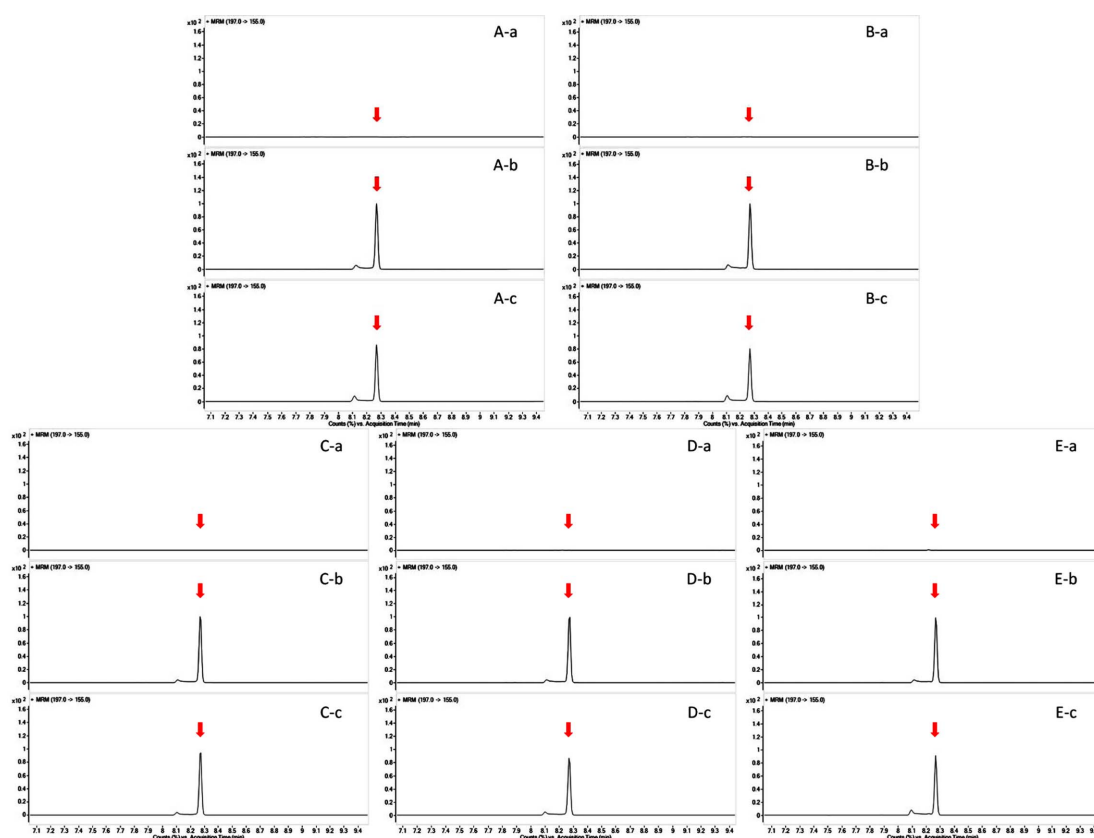


Fig. 2. Representative MRM (quantification ion) chromatograms of 2,6-DIPN in (A) hulled rice, (B) potato, (C) soybean, (D) mandarin, and (E) green pepper in (a) blank sample, (b) matrix-matched standard at 0.0625 $\mu\text{g/mL}$, and (c) recovery sample fortified at 0.5 mg/kg.

DIPN could be effectively purified and collected from various matrix interferences using a florisil cartridge.

3.4. Method validation

The comparison of the blank samples and spiked blank samples was used to evaluate the specificity, and the results revealed that the analytical method has high separability and selectivity since no potential interfering peaks were detected at the standard retention time, as shown in Fig. 2. 2,6-DIPN was quantified with a matrix-matched calibration curve, which was prepared to mitigate the matrix effect in terms of signal suppression or enhancement by the coelution of matrix components.¹³ The linearities of the matrix-matched calibrations in the five agricultural products were excellent and were assessed over seven concen-

tration levels, and the determination coefficients (R^2) were higher than 0.998. The LOD and LOQ of the instrument in the sample analyses were 0.0004 and 0.00125 $\mu\text{g/mL}$, respectively, and the LOQ of the method was calculated as 0.01 mg/kg. These results are sufficient for the determination and quantification of the low residual levels of the analyte in the agricultural samples and can deliver satisfactory analysis with the standards for non-detection of pesticide residues in accordance with the positive list system (PLS). The average recoveries of the five samples were between 72.8–99.3% with $\text{CV} \leq 9.5\%$ (Table 4). The repeatability was assessed by the average coefficient of variation (CV%) of the two inter-laboratory analyses and the results indicated levels of 2,6-DIPN that were less than criteria set for the fortification levels

Table 4. Inter-laboratory validation results of analytical method for the determination of 2,6-DIPN residues in samples

Sample	Fortification (mg/kg)	Recovery \pm CV1 ^a (%)		Ave. ^d (%)	CV2 ^e (%)
		Lab.1 ^b	Lab.2 ^c		
Hulled rice	0.01	81.4 \pm 5.2	86.1 \pm 2.1	83.8	5.2
	0.1	95.2 \pm 2.3	86.3 \pm 0.2	90.8	5.6
	0.5	97.0 \pm 0.4	83.3 \pm 5.1	90.2	9.1
Potato	0.01	73.6 \pm 6.0	73.2 \pm 9.9	73.4	10.0
	0.1	83.2 \pm 4.1	78.9 \pm 1.0	81.1	4.4
	0.5	94.7 \pm 5.5	84.7 \pm 0.9	89.7	7.3
Soybean	0.01	77.7 \pm 9.5	74.2 \pm 3.0	76.0	8.7
	0.1	88.7 \pm 3.5	84.3 \pm 0.8	86.5	3.8
	0.5	98.8 \pm 2.0	83.4 \pm 1.1	91.1	9.4
Mandarin	0.01	72.8 \pm 4.1	77.0 \pm 0.5	74.9	4.6
	0.1	83.1 \pm 2.4	83.5 \pm 2.6	83.3	2.7
	0.5	91.7 \pm 2.3	81.0 \pm 0.3	86.4	7.0
Green pepper	0.01	75.7 \pm 3.3	86.1 \pm 3.0	80.9	7.9
	0.1	84.0 \pm 2.8	91.7 \pm 0.5	87.9	5.2
	0.5	99.3 \pm 1.8	89.2 \pm 0.8	94.3	6.0

^aMean values of 5 times (Lab.1) or 3 times (Lab.2) repetitions with coefficient of variation

^bMinistry of Food and Drug Safety

^cGwangju Regional Korea Food and Drug Administration

^dAverage recovery of inter-laboratory

^eAverage coefficient of variation of inter-laboratory

(1 < conc. \leq 10 μ g/kg: 46 %; 10 < conc. \leq 100 μ g/kg: 34 %; 100 < conc. \leq 1000 μ g/kg: 25 %) (Table 4). All optimized results are reliable and within the satisfactory range specified by Codex Alimentarius Commission and MFDS guidelines.

4. Conclusions

The analytical method using GC-MS/MS was established and validated for the determination of 2,6-DIPN residues in agricultural products. The validation results of the developed method were satisfied in accordance with the CODEX and MFDS guideline. The crop samples used in the experiment were selected as representative crops with high food intake and contain various types of sugars, lipids, and pigments. Finally, the method can be utilized for the routine analysis of 2,6-DIPN in other matrices and effectively applied for enabling safety management in imported and domestic agricultural products.

Acknowledgements

This research was financially supported by a Grant (19161MFDS020) from the Ministry of Food and Drug Safety, and we are grateful this support.

References

1. A. Sturaro, G. Parvoli, R. Rella and L. Doretti, *Journal of Chromatography A*, **688**(1-2), 211-219 (1994).
2. Health Canada Pest Management Regulatory Agency (PMRA), '2,6-Diisopropyl-naphthalene Aceto AmplifyII' (http://publications.gc.ca/collections/collection_2018/sc-hc/h113-9/H113-9-2018-5-eng.pdf), 2018.
3. US Environmental Protection Agency (US EPA), 'Biopesticide registration action document' (https://www3.epa.gov/pesticides/chem_search/reg_actions/registration/decision_PC-055803_1-Oct-03.pdf), 2003.
4. Codex Alimentarius Commission (CAC), Codex pesticides residues in food online database, <http://www.fao.org/fao-who-codexalimentarius/codex-texts/dbs/pestres/>

- pesticides/it/.
5. European Commission (EC), EU pesticide database, <http://ec.europa.eu/food/plant/pesticides/eu-pesticides-database/public/?event=pesticide.residue.selection&language=EN>.
 6. The Japan Food Chemical Research Foundation (JFCRF), Maximum residue limits (MRLs) list of agricultural chemicals in foods, http://db.ffcr.or.jp/front/pesticide_detail?id=25300.
 7. Ministry of Food and Drug Safety (MFDS), Maximum residue limits (MRLs) of pesticide, <http://www.foodsafety-korea.go.kr/residue/search/list.do?currentPageNo=1&searchType=&searchValue=2%2C6-Diisopropylnaphthalene&searchFlag=ALL>.
 8. US Environmental Protection Agency (US EPA), Regulation of pesticide residues on food, https://www.ecfr.gov/cgi-bin/text-idx?SID=e76950edb334efe13b37666626f8f6ed&mc=true&node=se40.26.180_1590&rqn=div8.
 9. K. Zhang, G. O. Noonan and T. H. Begley, *Food Additives and Contaminants*, **25**(11), 1416-1423 (2008).
 10. Codex Alimentarius Commission (CAC), 'Guidelines on good laboratory practice in pesticide residue analysis. CAC/GL 40-1993 (amendment 2010)' (http://www.fao.org/input/download/standards/378/cxg_040e.pdf), 2010.
 11. Ministry of Food and Drug Safety (MFDS), 'Guidelines on standard procedures for preparing analysis method' (https://www.mfds.go.kr/brd/m_210/view.do?seq=12920&srchFr=&srchTo=&srchWord=%EC%8B%9D%ED%92%88%EB%93%B1+%EC%8B%9C%ED%97%98%EB%B2%95+%EB%A7%88%EB%A0%A8&srchTp=0&itm_seq_1=0&itm_seq_2=0&multi_itm_seq=0&company_cd=&company_nm=&page=1), 2016.
 12. Ministry of Food and Drug Safety (MFDS), 'Pesticide analytical residues manual in food code', 4th Edition, Republic of Korea (2013).
 13. S. K. Cho, A. M. Abd El-Aty, K. H. Park, J. H. Park, M. E. Assayed, Y. M. Jeong, Y. S. Park and J. H. Shim, *Food Chemistry*, **136**(3-4), 1414-1420 (2013).

Authors' Position

Han Sol Lee	: Researcher
Ji-Su Park	: Researcher
Su Jung Lee	: Researcher
Hye-Sun Shin	: Examiner
Yun mi Chung	: Scientific officer
Ha na Choi	: Researcher
Sang Soon Yun	: Scientific officer
Yong-hyun Jung	: Senior scientific officer
Jae-Ho Oh	: Director