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Dissipation dynamic of quintrione in rice plants and terminal residue in rice straw, grain and soil

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Abstract: Quintrione (2-(3,7-dichloroquinoline-8-carbonyl) cyclohexane-1,3-dione) is a new postemergence herbicide just approved by Ministry of Agriculture and Rural Affairs in China. However, the potential environmental pollution caused by quintrione has not been investigated to date. A combined high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) and modified quick, easy, cheap, effective, rugged, and safe (QuEChERS) method was developped for the analysis of quintrione residue in rice plant, grain and soil. The dissipation and terminal residues of quintrione in rice plant were determined by using this method. Results showed that there was a good linear correlation between the concentrations and peak areas with R^2 higher than 0.99. The average recoveries of quintrione in rice plant, soil, paddy water, paddy hull and brown rice varied from 78% to 99% with the relative standard deviation (RSD) ranged from 3.9% to 11%. The limits of quantification (LOQ) of quintrione in rice plant, soil, paddy water, paddy hull and brown rice were all 0.002 mg/kg. The investigation of dissipation dynamic data revealed that quintrione degraded quickly in rice plants with half-life $(t_{1/2})$ values between 6.7 to 12.8 days. 96 days after quintrione application at 900 and 1 350 g a.i./ha on rice seedling, no terminal residual was detected in paddy soil, paddy hull, or brown rice samples. The study will contribute to the setting of the maximum residue limits (MRL) of quintrione in rice and the field application strategy.

Keywords: high-performance liquid chromatography-tandem mass spectrometry; dissipation dynamic; quintrione; rice plant; soil; residue

二氯喹啉草酮在水稻植株上的消解动态及其在稻秆、 稻谷和土壤中的最终残留

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摘 要:二氯喹啉草酮是经中国农业农村部批准登记的茎叶处理除草剂,迄今为止对其潜在的环境污染尚未见报道。本研究建立了一种采用 QuEChERS 前处理方法结合高效液相色谱-串联三重四极杆质谱 (HPLC-MS/MS) 联用技术检测二氯喹啉草酮在水稻植株、稻谷和土壤中残留的方法,并采用该方法测定了二氯喹啉草酮在水稻植株中的消解动态和最终残留。结果表明:二氯喹啉草酮的进样质量浓度与其峰面积间呈良好的线性相关, R²>0.99;其在植株、土壤、田

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水、稻壳和糙米中的平均回收率在 78%~99% 之间,相对标准偏差 (RSD) 在 3.9%~11% 之间,在植株、土壤、田水、稻壳和糙米中的最低检测浓度 (LOQ) 均为 0.002 mg/kg。二氯喹啉草酮在水稻植株中的消解半衰期为 6.7~12.8 d。以有效成分 900~1 350 g/hm² 的剂量于水稻苗期施用1次,在施药 96 d 后采集的土壤、稻壳和糙米中均未检测出二氯喹啉草酮。本研究结果可为水稻中二氯喹啉草酮最大残留限量值和田间使用规范的制定提供参考。

关键词: 高效液相色谱-串联三重四极杆质谱; 消解动态; 二氯喹啉草酮; 水稻植株; 土壤; 残留中图分类号: TQ 450.26 文献标志码: A 文章编号: 1008-7303(2020)02-0388-09

Rice (*Oryza sativa* L.) is one of the most important crops globally, especially in Asia. Asia is the largest producer of rice and its exports account for about 23% of the global rice crop production^[1-6]. Rice production is significantly affected by harmful factors such as weeds. Chemical herbicides have traditionally been used to protect the yield of rice, which was the most direct and effective method^[7-8]. However, after long term application of herbicide in rice fields, many weeds have developed resistance^[9-11]. It is therefore necessary to develop novel herbicides.

Quintrione (CAS number, 130901-36-8), 2-(3,7dichloroquinoline-8-carbonyl) cyclohexane-1,3-dione (Scheme 1), is one of these newly developed broadspectrum herbicides (Beijing Fagaiyin Science and Technology Co., Ltd., in 2011). In China, new herbicides have to be approved before being registered (Registration No. PD20184028) (ICAMA)[12-13]. Quintrione, which can be absorbed by the roots, stems, and leaves of weeds, inhibits the synthesis of 4-hydroxyphenylpyruvate dioxygenase (HPPD), which leads to the depletion of chlorophyll in leaves and thus kills the weeds^[14]. Quintrione can be used in paddy fields to control a variety of weeds, including gramineae, broadleaf and cyperacea, e.g., Echinochloa crus-galli, Digitaria sanguinalis, Setaria viridis, Ammannia baccifera, Ludwigia prostrata, Galium aparine, Abutilon theophrasti, Xanthium sibiricum, Amaranthus spinosus, Kochia scoparia and Stellaria media^[15]. Although quintrione has been approved to be registered in China, the potential risk of environmental pollution caused by quintrione has not been investigated. However, its analogue (quinciorac), another quinoline-type commercially available herbicide, used in paddy fields, has been reported to be harmful to susceptible crops, submerged

macrophytes, fish, and have potential toxicity to mammals^[16-20]. Hence, the usage of quintrione as an organic pesticide may also have potential risks for both the environment and human health.

Molecular weight: 336.17; Density: 1.476 g/cm³; Boiling point: 514.3 °C; Log*P*: 0.630; p*K*a: 2.46

Scheme 1 Structural formula and physical and chemical properties of quintrione

The quick, easy, cheap, effective, rugged, and safe (QuEChERS) method has become one of the the most popular methods for pesticide residue analysis, since it is convenient and practical^[21-25]. Acetonitrile was used in this method as an extraction solvent, followed by an extraction/partitioning step after the addition of a salt mixture. Then, dispersive solidphase extraction (d-SPE) purified the sorbents for clean-up procedures that can be further applied to detect a wide range of pesticide residues in different food and environmental samples. Originally, QuEChERS was introduced for the analysis of pesticide residues in fruits and vegetables with high water content. However, more recently, it has gained significant popularity with or without small modifications for the analysis of pesticides and other compounds in the enormous variety of food and other products with different types of matrices^[26-29]. With Regard to the recent analytical and detection techniques for pesticide residues, high-performance liquid chromatography-tandem mass spectrometry (HPLC-MS/MS) is one of the most commonly used and most widely applicable techniques. It offers a variety of advantages such as high-speed, high selectivity, high

sensitivity, and highly stable detection of analytes. It is usually coupled with the QuEChERS method for the quantitative and qualitative identification of a number of pesticides in various food and environmental matrices^[30-35].

At present, there are no reports describing the environmental fate, terminal residues, or intake risk of quintrione in brown rice under good agricultural practices (GAP). To fill this gap, the following studies were conducted in this work: 1) developing and validating a simple and sensitive method for the determination of the quintrione content in paddy water, paddy soil, rice straw, paddy hull, and brown rice samples by the QuEChERS method and HPLC-MS/MS; 2) investigating the dissipation and the residue of quintrione in the rice paddy system in China; 3) assessing the dietary intake risk of quintrione in rice based on the terminal residue and toxicology data. The data provided by this study will not only provide reasonable guidance on the use of this herbicide, but also serve as a preliminary dietary safety assessment for rice consumption.

1 Materials and Methods

1.1 Chemicals and material

Analytical standard quintrione (98.0% purity) was purchased from Beijing Fagaiyin Science and Technology Co., Ltd. (Beijing, China). Acetonitrile and ultrapure water for HPLC-MS/MS were purchased from Merck KGaA (Darmstadt, Germany). Purified water was prepared by a Milli-Q (Millipore, Bedford, MA, USA). Sodium chloride (98.0% purity) and anhydrous magnesium sulfate (97.0% purity) were purchased from Sinopharm Chemical Reagent Co., Ltd. (Shanghai, China). Primary secondary amine (PSA) was purchased from CNW Technologies GMBH (Düsseldorf, Germany).

1.2 Field experiments

Field trials were performed in three different provinces of China from 2015 to 2016. Site 1 was located in South Central China (N 28.18, E 113.03, Hunan Province, subtropical monsoon humid climate). Site 2 was located in Northeast China (N 45.86, E 126.26, Heilongjiang Province, temperate

continental monsoon climate). Site 3 was located in East China (N 33.99, E 116.83, Anhui Province, warm temperate semi-humid monsoon climate). The soil type in Hunan Province was clay, with the pH value of 6.2. The soil type in Heilongjiang Province was moisture fluvo-aquic, with the pH value of 7.2. The soil type in Anhui Province was loam, with the pH value of 5.9. The field trials were conducted according to the "Guideline on Pesticide Residue Trials" (NY/T 788—2004), issued by the Ministry of Agriculture and Rural Affairs, P. R. China. A control and four treatments were conducted in the field experiments. Each treatment with three repetitions used an area of 30 m². A buffer area of 1 m was used to separate the plots from each other.

To investigate the dissipation of quintrione in paddy water, paddy soil, rice straw, paddy hull and brown rice, 20% OD (Oil Dispersion) was sprayed on the rice paddy at the dose of 1 350 g a.i./ha (1.5 times of the recommended dosage). Representative samples were randomly collected from 6-12 points in each plot at 2 h as well as 1, 3, 5, 7, 14, 21, 28 and 45 d after spraying. Rice samples (rice straw, paddy hull and brown rice) were collected randomly from 6-12 rice plants growing normally on the soil surface (rootless plants), and the sampling amounts were not lower than 1 kg. These samples were cut into small segments of less than 1 cm length, mixed, and reduced by quartering. Then, two copies (200 g per copy) for each sample were put into small sample bags. Water samples (about 5 000 mL per sample) for each plot were collected randomly from more than 10 points per plot. Then, these water samples were uniformity mixed and filtrated by gauze. The filtrate (300 mL) for each sample was transferred into a bottle. Soil samples with less than 1 kg per sample were also randomly collected from more than 10 points per plot (the treated ones and the control) and the soil sampling depth was 0-10 cm. Weeds and rocks were removed from these soil samples, and samples were mixed uniformly, passed through a 20 mesh screen, and reduced by quartering. The treated soil samples (200 g per sample) were packed into sealed plastic containers. All samples were labeled, transported to the laboratory within 8 h, and stored at -20 °C for further analysis.

In terminal residue experiments, 20% OD was sprayed at a low dosage of 900 g a.i/ha (the recommended high dosage) and a high dosage of 1 350 g a.i/ha (1.5 times of the recommended dosage). Each dosage level was sprayed once. Representative paddy soil, water, rice straw, paddy hull and brown rice were randomly collected at harvest date after spraying. Paddy soil samples were collected from each plot (treatment plots and control) randomly, and a total of more than 10 points with a soil sampling depth of 0-10 cm were collected. These soil samples were cleaned from weeds and rocks, mixed uniformity, passed through a 20 mesh screen, reduced by quartering. Then, treated soil samples (200 g per sample) were packed into the sealed plastic containers. Rice straw samples were collected randomly from 6-12 rice plants, which were growing normally on the soil surface (rootless plants), sampling less than 1 kg. These rice straw samples were cut into small segments shorter than 1 cm, mixed, and reduced by quartering. Two copies (200 g per copy) for each sample were put into small sample bags. Rice panicles, with sampling amounts of less than 5 kg, were collected randomly from more than 12 points. After sampling, rice panicles were threshed to obtain brown rice and rice husk. The brown rice and rice husk samples were separately mixed and reduced by quartering. Two copies (200 g per copy) for each brown rice and rice husk sample were put into small sample bags. All samples were labeled, transported to the laboratory within 8 h, and stored at -20 °C for analysis.

1.3 Sample extraction

Rice straw, paddy hull, and brown rice were mixed in a grinder to obtain pulverized samples. The samples were then dried and stored at -20 °C.

Rice straw samples: A pulverized rice straw sample (5.0 g) was weighed into a 100 mL centrifuge tube with 5 g NaCl. 10 mL of pure water and 20 mL of acetonitrile were then added. The mixture was vortexed for 1 min and centrifuged for 3 min at 4 000 r/min. Then, a portion (2 mL) of the obtained

supernatant was transferred to a 4 mL plastic centrifuge tube for cleanup.

Paddy hull samples: A pulverized paddy hull sample (5.0 g) was weighed into a 100 mL centrifuge tube with 5 g NaCl. Then, 10 mL pure water and 20 mL acetonitrile were added. The mixture was vortexed for 1 min and then centrifuged for 3 min at 4 000 r/min. A portion (2 mL) of the obtained supernatant was transferred to a 4 mL plastic centrifuge tube for cleanup.

Brown rice samples: A brown rice sample (10.0 g) was weighed into a 100 mL centrifuge tube with 5 g NaCl. Then, 10 mL pure water and 40 mL acetonitrile were added. The mixture was vortexed for 1 min and then centrifuged for 3 min at 4 000 r/min. A portion (2 mL) of the obtained supernatant was transferred to a 4 mL plastic centrifuge tube for cleanup.

Paddy soil samples: A paddy soil sample (10.0 g) was weighed into a 100 mL centrifuge tube with 5 g NaCl. Then, 40 mL of acetonitrile and 10 mL pure water were added. The mixture was vortexed for 1 min and then centrifuged for 3 min at 4 000 r/min. Then, a portion (2 mL) of the obtained supernatant was transferred to a 4 mL plastic centrifuge tube for cleanup.

Paddy water samples: A paddy water sample (10 mL) was transfered into a 100 mL centrifugal tube. After complication, 40 mL acetonitrile and 5 g sodium chloride were added. The mixture was vortexed for 1 min, centrifuged for 3 min at 4 000 r/min, and collected for purification.

1.4 Cleanup

100 mg of PSA and 400 mg of anhydrous magnesium sulfate were added to the sample (2 mL of the sample extract). After vortexing for 3 min, the samples were centrifuged for 5 min at 10 000 r/min. Then, the extracts were filtered through a 0.22 μ m nylon filter and transferred into a glass vial for HPLC-MS/MS analysis.

1.5 HPLC-MS/MS analysis

All analyses were performed on a Thermo ultimate 3000 HPLC and a Thermo TSQ Endura MS/MS. Separation was achieved by a Hypersil GOLD reverse-phase C_{18} HPLC column (2.1 mm \times 100 mm

i.d., 1.9 µm particle size) coupled with ultra-high purity ZPRBAX RX-SIL porous silica and maintained at 35 °C. The mobile phase consisted of methanol and water, containing 0.1% formic acid at a flow rate of 0.3 mL/min. The gradient elution of the mobile phase began with 20% methanol for 0.5 min, was increased to 95% methanol from 0.5 min to 1.5 min, retained at 95% methanol for 2.5 min, decreased to 20% methanol for 0.1 min, and then maintained at 95% methanol for 2.4 min. The injection volume was 5 μL. The Thermo TSQ Endura vector was used for spectral acquisition via the positive electrospray ionization mode. The parameters for analyzing quintrione are presented in Table 1. The nebulizer gas (N₂) had a pressure of 276 kPa, the desolvation gas temperature was 300 °C, the capillary temperature was 320 °C, and the capillary voltage was 3.5 kV.

Table 1 The parameters with MS/MS for quintrione analysis

Retention time/ min	Precursor ion, m/z	Product ion, m/z	RF Lens/	Collion energy/
3.60	336	224/161	91	12/45

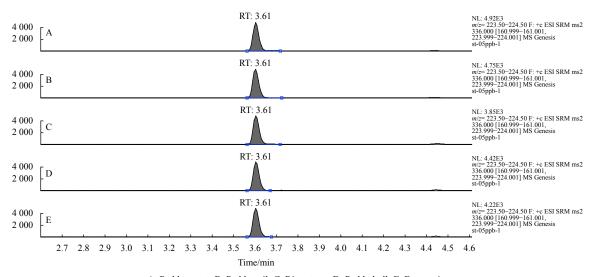
2 Results and Analysis

2.1 Method validation

The HPLC-MS/MS chromatograms in the SRM acquisition mode of the quintrione matrix standards (0.000 5 mg/kg) are described in Fig. 1. The HPLC-MS/MS chromatograms in the SRM acquisition mode

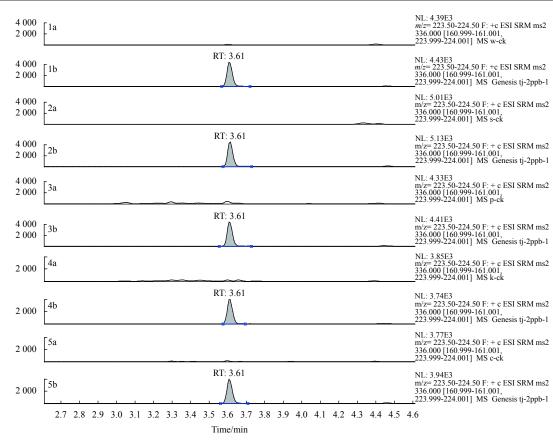
with a spike level (0.002 mg/kg) are depicted in Fig. 2. Calibration curves were generated by plotting the concentration as a function of peak area, and the results of calibration curve for quintrione over a range of 0.5-1 000 µg/L in methanol, paddy soil, rice straw, paddy hull, and brown rice samples are shown in Table 2. The matrix/solvent slope ratios (S_m/S_s) were calculated for these target matrices. Matrix effects were observed with quintrione in paddy water $(S_m/S_s =$ 1.043 0), paddy soil $(S_m/S_s = 0.955 0)$, rice straw $(S_m/S_s =$ 0.830 0), paddy hull $(S_m/S_s = 0.862 3)$, and brown rice $(S_{\rm m}/S_{\rm s}=0.951~8)$. These values were acceptable and were applied to quantify residues of quintrione in paddy water, paddy soil, rice straw, pdaay hull, and brown rice when applying matrix-matched standard solutions.

The LODs for quintrione in paddy water, paddy soil, rice straw, paddy hull, and brown rice were 0.000 6 mg/kg at the 3 × S/N level. The limits of quantification (LOQ) was 0.002 mg/kg at 10 × S/N level. Low LOQs and LODs demonstrated the high sensitivity of this method. The accuracy and precision of the method were estimated using recovery tests at three different spiking levels (0.002, 0.2 and 2 mg/kg), each with five replicates. Of note, an spiking level (16 mg/kg) was added for rice straw. As shown in Table 2, the mean recoveries of quintrione in paddy water, paddy soil, rice straw, paddy hull and brown rice samples were 78%-99% with RSDs of 3.9%-



A. Paddy water; B. Paddy soil; C. Rice straw; D. Paddy hull; E. Brown rice.

Fig. 1 HPLC-MS/MS chromatograms in SRM acquisition mode of quintrione matrix standards (0.000 5 mg/kg)



1a. Blank paddy water;
 1b. Spiked paddy water;
 2a. Blank paddy soil;
 2b. Spiked paddy soil;
 3a. Blank rice straw;
 4a. Blank paddy hull;
 5a. Blank brown rice;
 5b. Spiked brown rice.

Fig. 2 HPLC-MS/MS chromatograms in SRM acquisition mode at 0.002 mg/kg spike level

Table 2 Average recovery, calibration curve, RSD, LODs, and LOQs of quintrione in paddy water, paddy soil, rice straw, paddy hull, and brown rice with five replicates

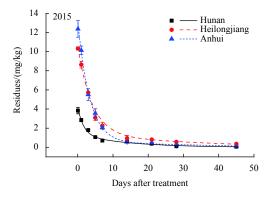
Matrix	Fortification level/(mg/kg)	Average recovery/%	RSD/%	Calibration curve	R^2	LOD/(µg/kg)	LOQ/(µg/kg)	
	0.002	94	7.5			0.6	2	
Paddy water	0.2	88	6.2	y = 20712 x + 143860	0.998 7			
	2	98	5.6					
	0.002	99	8.8		0.999 5	0.6	2	
Paddy soil	0.2	81	9.6	y = 19751 x + 6968.2				
	2	89	5.1					
	0.002	93	11		0.000.2	0.6	2	
D: 4	0.2	86	6.6	15.720 + 01.572				
Rice straw	2	78	7.9	y = 15720 x + 91573	0.999 3			
	16	88	7.3					
	0.002	95	11					
Paddy hull	0.2	88	7.8	y = 16804 x + 127304	0.998 2	0.6	2	
	2	80	9.2					
	0.002	86	9.6			0.6		
Brown rice	0.2	96	6.0	y = 17708 x + 59780	0.998 7		2	
	2	83	3.9					

11%. These parameters satisfy the EU guidelines^[36]. The above results demonstrated that the sensitivity, precision, and reproducibility of the proposed method

were acceptable for the quantitation of quintrione residues in paddy water, paddy soil, rice straw, paddy hull and brown rice samples.

2.2 Dissipation of quintrione in rice plants

The dissipation curves of quintrione during the period from 2015 to 2016 in the paddy field conditions in rice plants are depicted in Fig. 3.



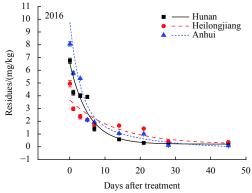


Fig. 3 Dissipation curves of quintrione in rice plants at Hunan, Heilongjiang and Anhui Provinces within two years, described by a first-order kinetic model

The $t_{1/2}$ value and the correlation coefficient of quintrione in rice plants are listed in Table 3. Initial residues of quintrione in rice plants varied among three experimental locations. The data obtained in 2015 showed that the initial residues of quintrione at those three sites were 3.835 mg/kg in site 1, 10.325 mg/kg in site 2, and 12.368 mg/kg in site 3, which had a significant difference to those in 2016 (6.740 mg/kg in Hunan, 4.951 mg/kg in Heilongjiang, and 8.067 mg/kg in Anhui). The initial residues of quintrione in Hunan were less than those in Heilongjiang and Anhui in 2015, whereas initial residues in Heilongjiang were less than those in Hunan and Anhui in 2016. These residue data presented a sustained decrease. More than 50% of the initial residues of quintrione degraded in rice plants by the 7th day after the application and more than 90% of residues dissipated within 28 days after the application. The levels were

0.055 mg/kg (Hunan), 0.354 mg/kg (Heilongjiang), and 0.137 mg/kg (Anhui) at day 45 after quintrione application, which indicated a dissipation of more than 96%. The dissipation dynamic of quintrione in rice plants during 2016 remained similar to that in 2015. 45 days after quintrione was applied, the residual concentrations decreased to 0.245 mg/kg (Hunan), 0.356 mg/kg (Heilongjiang), and 0.083 mg/kg (Anhui), respectively. This indicated a 92% dissipation.

Table 3 Dissipation, $t_{1/2}$ value and correlation coefficient of quintrione in rice plants

Year	Location (Province)	Kinetic equation	R^2	$t_{1/2}/d$
2015	Hunan	$y = 2\ 255.8\ e^{-0.091x}$	0.926 7	7.6
	Heilongjiang	$y = 5 636.9 e^{-0.075x}$	0.837 9	9.2
	Anhui	$y = 6\ 213.7\ e^{-0.101x}$	0.863 7	6.9
2016	Hunan	$y = 3 753.3 e^{-0.080x}$	0.793 7	8.7
	Heilongjiang	$y = 3 \ 191.7 \ e^{-0.054x}$	0.875 1	12.8
	Anhui	$y = 5 \ 407.1 \ e^{-0.103x}$	0.913 3	6.7

To better understand the dissipation regularity of quintrione in rice plants, the data were fitted to firstorder kinetics equations (Table 3). The $t_{1/2}$ values were determined to be 6.9-9.2 days in 2015 and 6.7-12.8 days in 2016, indicating that quintrione dissipated rapidly in rice plants. The $t_{1/2}$ value of quintrione in Anhui was 6.7-6.9 days. This was shorter than that in Hunan, where the $t_{1/2}$ value was 7.6-8.7 days, and also shorter than that in Heilongjiang where the $t_{1/2}$ value was 9.2-12.8 days. The factors that affected the initial residues and $t_{1/2}$ value of the pesticide were quite complex, including the organic matter content, climate, soil type, pH value and light intensity[32, 36-39]. Additionally, microorganisms might also play a major role in the dissipation of this pesticide under field conditions^[37].

2.3 Terminal residuals of quintrione in field trials

The terminal residual levels of quintrione in paddy soil, rice straw, paddy hull and brown rice from Hunan, Heilongjiang and Anhui in 2015 and 2016 are listed in Table 4. They were measured during a pre-harvest interval of 96 days. Quintrione formula was sprayed directly onto the stem leaf of weeds. The terminal residues in rice straw samples were not

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detectable (< LOQ, 0.002 mg/kg) on the 96th day after the application using two dosage levels (900 and 1 350 g a.i/ha). It was noteworthy that residue was detected in rice straw at 0.015-0.021 mg/kg (2015)

and 0.011-0.014 mg/kg (2016) in Anhui. Apart from the results obtained in Anhui, no terminal quintrione residues were detected in paddy soil, paddy hull, or brown rice samples on the 96th day after the application.

Table 4 Terminal residues of quintrione in paddy soil, rice straw, paddy hull and brown rice in field trials at harvest times for 2015 and 2016 (each dosage level was sprayed once)

(mg/kg)

	Dosage	Paddy soil			Rice straw			Paddy hull		Brown rice			
	(g a.i./ha)	Hunan	Heilongjiang	Anhui	Hunan	Heilongjiang	Anhui	Hunan	Heilongjiang	Anhui	Hunan	Heilongjiang	Anhui
2015	900	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	0.015 ± 0.005	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
	1 350	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	0.021 ± 0.003	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
2016	900	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	0.011 ± 0.005	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ
	1 350	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	0.014 ± 0.005	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ	< LOQ

These experimental results showed that when quintrione was applied within the designed field conditions, no quintrione residual could be detected in brown rice. This indicated that quintrione could be safely used on paddy rice. This research could be helpful for governments and other organizations that recommend the maximum residue limit (MRL) for quintrione. These results provide valuable guidance for the use of quintrione in rice fields.

3 Conclusion and Discussion

In summary, a combined HPLC-MS/MS and a modified QuEChERS method has been developped for the determination of the amount of quintrione residue in rice and environmental samples, which was then applied in the study of dissipation and residuals of quintrione in rice paddy fields. The quintrione dissipation dynamic and terminal residuals were studied in paddy filed conditions. The LOD for paddy water, paddy soil, rice straw, paddy hull and brown rice samples was 0.000 6 mg/kg. The average recovery of quintrione ranged from 78% to 99% with an RSDs between 3.9%-11% (n = 5) at levels of 0.002, 0.2 and 2 mg/kg per sample. The dissipation dynamic experiment indicated that quintrione degraded rapidly in rice plants with $t_{1/2}$ value of 6.7-12.8 days. There was a significant difference of $t_{1/2}$ value between the three locations in different years, which required further study in the future. Terminal residuals in the field conditions were not detected in paddy soil, paddy hull, or brown rice

samples in the three sites 96 days after the application of quintrione at 900 and 1 350 g a.i./ha. These results offer valuable guidance for governments and organizations that recommend the MRL of quintrione. This study provides guidance for the proper and safe use of quintrione.

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