

THE 195th CONFERENCE FOR PROMOTION OF FOOD IMPORT FACILITATION

(FOOD SAFETY GROUP)

Standards and Evaluation Division
Department of Environmental Health and Food Safety
Pharmaceutical Safety and Environmental Health Bureau
Ministry of Health, Labour and Welfare

Date: Friday, September 23, 2016 (10:00–12:00)

Place: Ministry of Health, Labour and Welfare
Temporary Meeting Room No. 3
1-2-2, Kasumigaseki, Chiyoda-ku, Tokyo

Agenda:

Item 1. Establishment of the Maximum Residue Limits for Agricultural and Veterinary Chemicals in Foods

Pesticide: Hexaconazole, Lepimectin, Prometryn, Prothioconazole, Pyriofenone,
Simeconazole, Spirotetramat, Thifensulfuron methyl, Thifluzamide
Veterinary drug: Albendazole

Item 2. Revision of Analytical Methods for Agricultural and Veterinary drugs in Foods

Veterinary drug: Coumaphos

Item 3. Other matters

Japan's principles of establishing maximum residue limits for agricultural and veterinary chemicals in foods

<The manner of submitting comments>

The Ministry of Health, Labour and Welfare (MHLW) will revise the existing standards and specifications for food as shown in this document. Please provide comments in writing by **Friday, October 7, 2016**.

With regard to the agenda item 1, the SPS notification will be made for the setting or revision of the MRL for the agricultural chemicals. However, the setting or revision of the MRLs for Lepimectin, Prothioconazole and Pyriofenone will NOT be notified to the WTO because their regulations will not be strengthened by this amendment. After the given date, comments should be directed to the enquiry point in accordance with the SPS Agreement.

If you wish to request Japan to adopt the same limits as your country's MRLs, you are requested to submit data supporting your country's MRLs, such as risk assessment and residue data.

<Contact>

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Pesticide/Veterinary drug/Feed additive

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Item 1. Establishment of the Maximum Residue Limits for Agricultural and Veterinary Chemicals in Food

The Food Sanitation Act authorizes the Minister of Health, Labour and Welfare to establish residue standards (maximum residue limits, “MRLs”) for pesticides, feed additives, and veterinary drugs (hereafter referred to as “agricultural and veterinary chemicals”) that may remain in foods. Any food for which standards are established pursuant to the provisions in Article 11, Paragraph 1 of the act is not permitted to be marketed in Japan unless it complies with the established standards.

On May 29, 2006, Japan introduced the Positive List System¹ for agricultural and veterinary chemicals in food. All foods distributed in the Japanese marketplace are subject to regulation of the system.

The Ministry of Health, Labour and Welfare (MHLW) has comprehensively reviewed existing MRLs to modify those that were provisionally set at the introduction of the system. In addition, the MHLW is going to establish MRLs for some commodities. This activity has been targeted at following agricultural chemicals:

Pesticide: Hexaconazole, Lepimectin, Prometryn, Prothioconazole, Pyriofenone,
Simeconazole, Spirotetramat, Thifensulfuron methyl, Thifluzamide
Veterinary drug: Albendazole

¹ The aim of the positive list system is to prohibit the distribution of any foods which contain agricultural chemicals at amounts exceeding a certain level (0.01 ppm) in the Japanese marketplace unless specific maximum residue limits (MRLs) have been set.

Summary

Hexaconazole (pesticide: fungicide): Permitted for use in Japan.

The MHLW is going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System. The MHLW is also going to modify the existing MRLs in some commodities that were set before the introduction of the Positive List System.

Lepimectin (pesticide: insecticide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting MRLs by the Ministry of Agriculture, Forestry and Fisheries (MAFF). This action will not strengthen the current regulation for any commodities.

Prometryn (pesticide: herbicide): Permitted for use in Japan.

The MHLW is going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System.

Prothioconazole (pesticide: fungicide): Not permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting import tolerances based on the Guideline for Application for Establishment and Revision of Maximum Residue Limits for Agricultural Chemicals Used outside Japan (Shokuan No. 0205001, 5 February 2004). This action will not strengthen the current regulation for any commodities.

Pyriofenone (pesticide: fungicide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting MRLs by the Ministry of Agriculture, Forestry and Fisheries (MAFF). This action will not strengthen the current regulation for any commodities.

Simeconazole (pesticide: fungicide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting MRLs by the Ministry of Agriculture, Forestry and Fisheries (MAFF).

Spirotetramat (pesticide: insecticide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting MRLs by the Ministry of Agriculture, Forestry and Fisheries (MAFF). The MHLW is also going to establish MRLs in some commodities in

response to a request for setting import tolerances based on the Guideline for Application for Establishment and Revision of Maximum Residue Limits for Agricultural Chemicals Used outside Japan (Shokuan No. 0205001, 5 February 2004).

Thifensulfuron methyl (pesticide: herbicide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting import tolerances based on the Guideline for Application for Establishment and Revision of Maximum Residue Limits for Agricultural Chemicals Used outside Japan (Shokuan No. 0205001, 5 February 2004). The MHLW is also going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System.

Thifluzamide (pesticide: fungicide): Permitted for use in Japan.

The MHLW is going to establish MRLs in some commodities in response to a request for setting MRLs by the Ministry of Agriculture, Forestry and Fisheries (MAFF).

Albendazole (veterinary drug: parasiticide): Not permitted for use in Japan.

The MHLW is going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System. The MHLW is also going to modify the existing MRLs in some commodities that were set before the introduction of the Positive List System.

Hexaconazole

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Rice (brown rice)	•	0.02			
Wheat	•	0.1			
Barley	•	0.01			
Rye	•	0.01			
Corn (maize, including pop corn and sweet corn)	•	0.02			
Buckwheat	•	0.01			
Other cereal grains	•	0.01			
Soybeans, dry	•	0.05			
Beans, dry	•	0.02			
Peas	•	0.02			
Broad beans	•	0.02			
Peanuts, dry	•	0.05			
Other pulses	•	0.02			
Potato	•	0.02			
Taro	•	0.02			
Sweet potato	•	0.02			
Yam	•	0.02			
Konjac	•	0.02			
Other potatoes	•	0.02			
Sugar beet	•	0.02			
Japanese radish, roots (including radish)	•	0.02			
Japanese radish, leaves (including radish)	•	0.02			
Turnip, roots (including rutabaga)	•	0.02			
Turnip, leaves (including rutabaga)	•	0.02			
Horseradish	•	0.02			
Watercress	•	0.02			
Chinese cabbage	•	0.02			
Cabbage	•	0.1			
Brussels sprouts	•	0.1			
Kale	•	0.02			
Komatsuna(Japanese mustard spinach)	•	0.02			
Kyona	•	0.02			
Qing-geng-cai	•	0.02			
Cauliflower	•	0.02			
Broccoli	•	0.02			
Other cruciferous vegetables	•	0.02			
Burdock	•	0.02			
Salsify	•	0.02			
Artichoke	•	0.02			
Chicory	•	0.02			
Endive	•	0.02			
Shungiku	•	0.02			
Lettuce (including cos lettuce and leaf lettuce)	•	0.02			
Other composite vegetables	•	0.02			
Onion	•	0.02			
Welsh (including leek)	•	0.1			
Garlic	•	0.05			
Nira	•	0.02			
Asparagus	•	0.01			
Multiplying onion (including shallot)	•	0.02			
Other liliaceous vegetables	•	0.02			

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Carrot	•	0.1			
Parsnip	•	0.02			
Parsley	•	0.02			
Celery	•	0.02			
Mitsuba	•	0.02			
Other umbelliferous vegetables	•	0.02			
Tomato	•	0.1			
Pimiento (sweet pepper)	•	0.02			
Egg plant	•	0.02			
Other solanaceous vegetables	•	0.02			
Cucumber (including gherkin)	•	0.05			
Pumpkin (including squash)	•	0.05			
Oriental pickling melon (vegetable)	•	0.02			
Water melon	•	0.5			
Melons	•	0.5			
Makuwauri melon	•	0.5			
Other cucurbitaceous vegetables	•	0.02			
Spinach	•	0.02			
Bamboo shoots	•	0.02			
Okra	•	0.02			
Ginger	•	0.02			
Peas, immature (with pods)	•	0.02			
Kidney beans, immature (with pods)	•	0.02			
Green soybeans	•	0.02			
Button mushroom	•	0.02			
Shiitake mushroom	•	0.02			
Other mushrooms	•	0.02			
Other vegetables	•	0.02			
Unshu orange, pulp	•	0.02			
Citrus natsudaidai, whole	•	0.02			
Lemon	•	0.02			
Orange (including navel orange)	•	0.02			
Grapefruit	•	0.02			
Lime	•	0.02			
Other citrus fruits	•	0.02			
Apple	○ 0.5	0.5	§		
Japanese pear	• 0.3	0.5	§		
Pear	• 0.3	0.5	§		
Quince	•	0.5			
Loquat	•	0.5			
Peach	○ 0.1	0.1	§		
Nectarine	○ 0.5	0.5	§		
Apricot	• 0.2	0.5	§		
Japanese plum (including prune)	• 0.3	0.5	§		
Mume plum	•	0.5			
Cherry	○ 0.5	0.5	§		
Strawberry	•	0.1			
Raspberry	•	0.5			
Blackberry	•	0.5			
Blueberry	•	0.5			
Cranberry	•	0.5			

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Huckleberry	●	0.5			
Other berries	●	0.5			
Grape	●	0.1			
Japanese persimmon	● 0.3	0.5	§		
Banana	●	0.1			
Kiwifruit	●	0.5			
Papaya	●	0.5			
Avocado	●	0.5			
Pineapple	●	0.5			
Guava	●	0.5			
Mango	●	0.5			
Passion fruit	●	0.5			
Date	●	0.5			
Other fruits	● 0.2	0.5	§		
Sunflower seeds	●	0.05			
Sesame seeds	●	0.05			
Safflower seeds	●	0.05			
Cotton seeds	●	0.05			
Rapeseeds	●	0.05			
Other oil seeds	●	0.05			
Ginkgo nut	●	0.05			
Chestnut	●	0.05			
Pecan	●	0.05			
Almond	●	0.1			
Walnut	●	0.05			
Other nuts	●	0.1			
Tea	●	0.05			
Coffee beans	●	0.05			
Hop	●	0.05			
Other spices	●	0.5			
Other herbs	●	0.02			

Note: The residue definition is Hexaconazole only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* Shaded figures indicate provisional MRLs.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Lepimectin

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Corn (maize, including pop corn and sweet corn)	0.05	0.05	§		
Soybeans, dry	0.01	0.01	§		
Taro	0.05	0.05	§		
Sweet potato	0.01	0.01	§		
Japanese radish, roots (including radish)	0.01	0.01	§		
Japanese radish, leaves (including radish)	0.3	0.3	§		
Turnip, roots (including rutabaga)	0.05	0.05	§		
Turnip, leaves (including rutabaga)	0.5	0.5	§		
Chinese cabbage	0.05	0.05	§		
Cabbage	0.05	0.05	§		
Kale	1	1	§		
Komatsuna(Japanese mustard spinach)	1	1	§		
Kyona	0.3	0.3	§		
Qing-geng-cai	1	1	§		
Cauliflower	0.2	0.2	§		
Broccoli	0.05	0.05	§		
Other cruciferous vegetables	1	1	§		
Lettuce (including cos lettuce and leaf lettuce)	1	1	§		
Other composite vegetables	0.7		Request		
Onion	0.05		Request		
Welsh (including leek)	0.01	0.01	§		
Asparagus	0.2		Request		
Tomato	0.3	0.3	§		
Pimiento (sweet pepper)	0.1	0.1	§		
Egg plant	0.2	0.2	§		
Cucumber (including gherkin)	0.1	0.1	§		
Water melon	0.05		Request		
Melons	0.01	0.01	§		
Spinach	2	2	§		
Peas, immature (with pods)	0.2		Request		
Kidney beans, immature (with pods)	0.1		Request		
Green soybeans	0.1	0.1	§		
Other vegetables	0.05		Request		
Unshu orange, pulp	0.01	0.01	§		
Citrus natsudaidai, whole	0.03	0.03	§		
Lemon	0.1	0.1	§		
Orange (including navel orange)	0.1	0.1	§		
Grapefruit	0.1	0.1	§		
Lime	0.1	0.1	§		
Other citrus fruits	0.1	0.1	§		
Apple	0.2	0.2	§		
Japanese pear	0.2	0.2	§		
Pear	0.2	0.2	§		
Peach	0.01	0.01	§		
Cherry	0.2	0.2	§		
Strawberry	0.5	0.5	§		
Grape	0.3	0.3	§		
Tea	0.3	0.3	§		
Other spices	0.3	0.3	§		
Other herbs	1	1	§		
Fish	0.02	0.02			

Note: The residue definition is sum of Lepimectin A3 [(10*E*, 14*E*, 16*E*)-
(1*R*, 4*S*, 5'*S*, 6*R*, 6'*R*, 8*R*, 12*R*, 13*S*, 20*R*, 21*R*, 24*S*)-

21,24-Dihydroxy-5',6',11,13,22-pentamethyl-2-oxo-3,7,19-trioxatetra-cyclo[15.6.1.1^{4,8}.0^{20,24}]pentacosa-
10,14,16,22-tetraene-6-spiro-2'-tetrahydropyran-12-yl(*Z*)-2-methoxyimino-2-phenylacetate] and Lepimectin A4
[(10*E*, 14*E*, 16*E*)-(1*R*, 4*S*, 5'*S*, 6*R*, 6'*R*, 8*R*, 12*R*, 13*S*, 20*R*, 21*R*, 24*S*)-6'-Ethyl-21,24-dihydroxy-5',11,13,22-
tetramethyl-2-oxo-3,7,19-trioxatetra-cyclo[15.6.1.1^{4,8}.0^{20,24}]pentacosa-10,14,16,22-tetraene-6-spiro-2'-
tetrahydropyran-12-yl(*Z*)-2-methoxyimino-2-phenylacetate].

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

Prometryn

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Rice (brown rice)	○ 0.1	0.05	§		
Wheat	○ 0.1	0.1	§		
Barley	● 0.02	0.05	§		
Rye	○ 0.1	0.05	§		
Corn (maize, including pop corn and sweet corn)	● 0.02	0.2	§		
Buckwheat	●	0.05			
Other cereal grains	○ 0.2	0.05	§		
Soybeans, dry	○ 0.1	0.1	§		
Beans, dry	● 0.05	0.1	§		
Peas	●	0.05			
Broad beans	●	0.05			
Peanuts, dry	● 0.02	0.05	§		
Other pulses	●	0.05			
Potato	●	0.05			
Taro	●	0.05			
Sweet potato	●	0.05			
Yam	● 0.02	0.05	§		
Konjac	●	0.05			
Other potatoes	●	0.05			
Sugar beet	●	0.1			
Japanese radish, roots (including radish)	●	0.05			
Japanese radish, leaves (including radish)	●	0.05			
Turnip, roots (including rutabaga)	●	0.05			
Turnip, leaves (including rutabaga)	●	0.05			
Horseradish	●	0.05			
Watercress	●	0.05			
Chinese cabbage	●	0.05			
Cabbage	○ 0.05	0.05	§		
Brussels sprouts	●	0.05			
Kale	●	0.05			
Komatsuna(Japanese mustard spinach)	●	0.05			
Kyona	●	0.05			
Qing-geng-cai	●	0.05			
Cauliflower	●	0.05			
Broccoli	●	0.05			
Other cruciferous vegetables	●	0.05			
Burdock	●	0.05			
Salsify	●	0.05			
Artichoke	●	0.05			
Chicory	●	0.05			
Endive	●	0.05			
Shungiku	●	0.05			
Lettuce (including cos lettuce and leaf lettuce)	●	0.05			
Other composite vegetables	●	0.05			
Onion	● 0.02	0.05	§		
Welsh (including leek)	○ 0.05	0.05	§		
Garlic	●	0.05			
Nira	●	0.05			
Asparagus	●	0.05			
Multiplying onion (including shallot)	●	0.05			
Other liliaceous vegetables	●	0.05			

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Carrot	○ 0.5	0.05	§		0.45: USA
Parsnip	●	0.05			
Parsley	○ 0.6	0.05			0.60: USA
Celery	●	0.05			
Mitsuba	●	0.05			
Other umbelliferous vegetables	○ 0.2	0.05			0.2: USA
Tomato	●	0.1			
Pimiento (sweet pepper)	●	0.1			
Egg plant	●	0.1			
Other solanaceous vegetables	●	0.1			
Cucumber (including gherkin)	●	0.1			
Pumpkin (including squash)	●	0.1			
Oriental pickling melon (vegetable)	●	0.1			
Water melon	●	0.1			
Melons	●	0.1			
Makuwauri melon	●	0.1			
Other cucurbitaceous vegetables	●	0.1			
Spinach	●	0.05			
Bamboo shoots	●	0.05			
Okra	● 0.05	0.1			0.05: USA
Ginger	●	0.05			
Peas, immature (with pods)	●	0.05			
Kidney beans, immature (with pods)	○ 0.05	0.05	§		
Green soybeans	● 0.02	0.05	§		
Button mushroom	●	0.1			
Shiitake mushroom	●	0.1			
Other mushrooms	●	0.1			
Other vegetables	●	0.05			
Other fruits	●	0.1			
Sunflower seeds	●	0.1			
Cotton seeds	●	0.2			
Other spices	●	0.1			
Other herbs	○ 4	0.05			3.5: USA
Cattle, muscle	●	0.05			
Pig, muscle	●	0.05			
Other terrestrial mammals, muscle	●	0.05			
Cattle, fat	●	0.05			
Pig, fat	●	0.05			
Other terrestrial mammals, fat	●	0.05			
Cattle, liver	●	0.05			
Pig, liver	●	0.05			
Other terrestrial mammals, liver	●	0.05			
Cattle, kidney	●	0.05			
Pig, kidney	●	0.05			
Other terrestrial mammals, kidney	●	0.05			
Cattle, edible offal	●	0.05			
Pig, edible offal	●	0.05			
Other terrestrial mammals, edible offal	●	0.05			
Milk	●	0.05			

Note: The residue definition is Prometryn only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* Shaded figures indicate provisional MRLs.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Prothioconazole

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
				Codex ppm	National ppm	
Wheat	○ 0.4	0.4		0.1	0.35	USA
Barley	○ 0.4	0.4		0.2	0.35	USA
Rye	○ 0.4	0.4		0.06	0.35	USA
Corn (maize, including pop corn and sweet corn)	○ 0.4	0.4		0.02	0.35	USA
Buckwheat	○ 0.4	0.4			0.35	USA
Other cereal grains	○ 0.4	0.4		0.06	0.35	USA
Soybeans, dry	○ 0.2	0.2		0.2	0.15	USA
Beans, dry	○ 1	1		1		
Peas	○ 1	1		1		
Broad beans	○ 1	1		1		
Peanuts, dry	○ 0.02	0.02		0.02		
Other pulses	○ 1	1		1		
Potato	○ 0.02	0.02		0.02	0.02	USA
Sugar beet	○ 0.3	0.3		0.3		
Cucumber (including gherkin)	○ 0.3		IT	0.2	0.30	USA
Pumpkin (including squash)	○ 0.3		IT	0.2	0.30	USA
Oriental pickling melon (vegetable)	○ 0.3		IT	0.2	0.30	USA
Other cucurbitaceous vegetables	○ 0.3		IT	0.2	0.30	USA
Blueberry	○ 2		IT	1.7	2.0	USA
Cranberry	○ 0.2		IT	0.17	0.2	USA
Huckleberry	○ 2		IT	1.7	2.0	USA
Other berries	○ 2		IT	1.7	2.0	USA
Rapeseeds	○ 0.2	0.2		0.1	0.15	USA
Cattle, muscle	○ 0.01	0.01		0.01		
Pig, muscle	○ 0.01	0.01		0.01		
Other terrestrial mammals, muscle	○ 0.01	0.01		0.01		
Cattle, fat	○ 0.05	0.05				
Pig, fat	○ 0.05	0.05				
Other terrestrial mammals, fat	○ 0.05	0.05				
Cattle, liver	○ 0.6	0.5		0.6		
Pig, liver	○ 0.6	0.5		0.6		
Other terrestrial mammals, liver	○ 0.6	0.5		0.6		
Cattle, kidney	○ 0.6	0.5		0.6		
Pig, kidney	○ 0.6	0.5		0.6		
Other terrestrial mammals, kidney	○ 0.6	0.5		0.6		
Cattle, edible offal	○ 0.6	0.5		0.6		
Pig, edible offal	○ 0.6	0.5		0.6		
Other terrestrial mammals, edible offal	○ 0.6	0.5		0.6		
Milk	○ 0.004	0.004		0.004		

Note: The residue definition for agricultural products is sum of Prothioconazole and metabolite M17 [2-(1-chlorocyclopropyl)-1-(2-chlorophenyl)-3-(1*H*-1,2,4-triazole-1-yl)-2-propanol], expressed as Prothioconazole. The residue definition for animal products is sum of metabolite M17 and its conjugates, expressed as Prothioconazole.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

○ : Commodities for which MRLs are to be maintained, increased or newly set

IT : Import tolerance

Pyriofenone

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Wheat	○ 1	1	§		
Pimiento (sweet pepper)	○ 1		Request		
Egg plant	○ 1	1	§		
Cucumber (including gherkin)	○ 1	1	§		
Pumpkin (including squash)	○ 0.7		Request		
Water melon	○ 0.05		Request		
Melons	○ 0.2		Request		
Apple	○ 1		Request		
Japanese pear	○ 1		Request		
Strawberry	○ 2	2	§		
Grape	○ 3		Request		

Note: The residue definition is Pyriofenone only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

Simeconazole

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Rice (brown rice)	○ 0.1	0.1	§		
Soybeans, dry	○ 0.2	0.2	§		
Konjac	○ 0.1	0.1	§		
Cabbage	○ 0.05		Request		
Burdock	○ 0.3	0.3	§		
Lettuce (including cos lettuce and leaf lettuce)	○ 0.7		Request		
Welsh (including leek)	○ 0.2	0.2	§		
Garlic	○ 0.1	0.1	§		
Nira	○ 0.1		Request		
Tomato	○ 0.2	0.2	§		
Other solanaceous vegetables	○ 2	2			
Cucumber (including gherkin)	○ 0.3	0.3	§		
Pumpkin (including squash)	○ 0.2	0.2	§		
Water melon	○ 0.1	0.1	§		
Melons	○ 0.1	0.1	§		
Spinach	○ 0.1	0.1	§		
Ginger	○ 0.3		Request		
Unshu orange, pulp	○ 0.1	0.1	§		
Citrus natsudaidai, whole	○ 0.3	0.3	§		
Lemon	○ 0.3	0.3	§		
Orange (including navel orange)	○ 0.3	0.3	§		
Grapefruit	○ 0.3	0.3	§		
Lime	○ 0.3	0.3	§		
Other citrus fruits	○ 0.3	0.3	§		
Apple	○ 0.5	0.5	§		
Japanese pear	○ 0.5	0.5	§		
Pear	○ 0.5	0.5	§		
Peach	○ 0.7	0.7	§		
Nectarine	○ 0.5	0.5	§		
Apricot	○ 1	1	§		
Japanese plum (including prune)	● 0.2	0.3	§		
Mume plum	○ 1	1	§		
Cherry	○ 3	3	§		
Strawberry	○ 3	3	§		
Grape	○ 0.2	0.2	§		
Japanese persimmon	○ 0.2	0.2	§		
Tea	○ 10	10	§		
Other spices	○ 0.3	0.3	§		
Other herbs	○ 30		Request		
Fish	○ 0.02	0.02			

Note: The residue definition is Simeconazole only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

Spirotetramat

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Corn (maize, including pop corn and sweet corn)	2		IT	1.5	1.5: Canada
Soybeans, dry	5	5		4	5.0: USA
Beans, dry	3	3		2	2.5: USA
Peas	3	3		2	2.5: USA
Broad beans	3		IT	2	2.5: USA
Other pulses	3	3		2	2.5: USA
Potato	1	1	§	0.8	
Taro	0.6	0.6			0.60: USA
Sweet potato	0.6	0.6			0.60: USA
Yam	0.6	0.6			0.60: USA
Other potatoes	0.6	0.6			0.60: USA
Japanese radish, leaves (including radish)	7	7		7	
Turnip, leaves (including rutabaga)	7	7		7	
Watercress	7	7		7	
Chinese cabbage	7	7		7	
Cabbage	7	2	IT	2	7: Australia
Brussels sprouts	1	1			
Kale	7	7		7	
Komatsuna(Japanese mustard spinach)	7	7		7	
Kyona	7	7		7	
Qing-geng-cai	7	7		7	
Cauliflower	7	1	IT	1	7: Australia
Broccoli	7	1	IT	1	7: Australia
Other cruciferous vegetables	7	7		7	
Artichoke	1			1	
Chicory	7	7		7	
Endive	7	7		7	
Shungiku	7	7		7	
Lettuce (including cos lettuce and leaf lettuce)	7	7		7	
Other composite vegetables	7	7		7	
Onion	0.8	0.5	IT	0.4	0.8: Canada
Welsh (including leek)	0.8		IT		0.8: Canada
Garlic	0.8		IT		0.8: Canada
Nira	0.8		IT		0.8: Canada
Asparagus	1		Request		
Other liliaceous vegetables	0.8		IT		0.8: Canada
Parsley	5	5			
Celery	5	5		4	
Other umbelliferous vegetables	5	5			
Tomato	3	3	§	1	
Pimiento (sweet pepper)	10	10	§	1	
Egg plant	2	2	§	1	
Other solanaceous vegetables	10	10	§	7	
Cucumber (including gherkin)	2	2	§	0.2	2: Australia
Pumpkin (including squash)	2	2	§	0.5	2: Australia
Oriental pickling melon (vegetable)	0.2	0.2		0.2	
Water melon	0.1	0.1	§		
Melons	0.1	0.1	§		
Makuwauri melon	0.03	0.03			
Other cucurbitaceous vegetables	7	7		7	
Spinach	7	7		7	

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Okra	1	1		1	
Ginger	0.6	0.6			0.60 USA
Peas, immature (with pods)	3	3		1.5	2.5 USA
Kidney beans, immature (with pods)	3	3		1.5	2.5 USA
Green soybeans	3	3		1.5	2.5 USA
Other vegetables	7	7		7	
Unshu orange, pulp※1	0.4		IT		1 Australia
Citrus natsudaidai, whole	1	1		0.5	1 Australia
Lemon	1	1		0.5	1 Australia
Orange (including navel orange)	1	1		0.5	1 Australia
Grapefruit	1	1		0.5	1 Australia
Lime	1	1		0.5	1 Australia
Other citrus fruits	1	1		0.5	1 Australia
Apple	0.7	0.7		0.7	
Japanese pear	0.7	0.7		0.7	
Pear	0.7	0.7		0.7	
Quince	0.7	0.7		0.7	
Loquat	0.7	0.7			
Peach※2	1		IT	3	
Nectarine	3	3		3	
Apricot	3	3		3	
Japanese plum (including prune)	5	5		5	
Mume plum	3	3		3	
Cherry	3	3		3	
Strawberry	10	10	§		
Blueberry	3		IT	1.5	3 Canada
Cranberry	3		IT	0.2	3 Canada
Huckleberry	3		IT		3 Canada
Other berries	3		IT		3 Canada
Grape	2	2		2	
Japanese persimmon	3		IT		2.5 USA
Banana	4		IT		4.0 USA
Papaya	3	3		0.4	2.5 USA
Avocado	0.6	0.6		0.4	0.60 USA
Pineapple	0.3		IT		0.30 USA
Guava	3	3		2	2.5 USA
Mango	0.3	0.3		0.3	
Passion fruit	3	3			2.5 USA
Other fruits	15	13		15	
Cotton seeds	1	1		0.4	1 Australia
Ginkgo nut	0.5	0.5		0.5	
Chestnut	0.5	0.5		0.5	
Pecan	0.5	0.5		0.5	
Almond	0.5	0.5		0.5	
Walnut	0.5	0.5		0.5	
Other nuts	0.5	0.5		0.5	
Coffee beans	0.2		IT		0.20 USA
Hop	15	15		15	
Other spices※3	7		IT	0.5	1 Australia
Other herbs	7	7	§	7	
Cattle, muscle	0.02	0.02		0.05	0.02 USA

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Pig, muscle	○ 0.02	0.02		0.05	
Other terrestrial mammals, muscle	○ 0.02	0.02		0.05	0.02 USA
Cattle, fat	○ 0.02	0.02			0.02 USA
Pig, fat	○ 0.02	0.02			
Other terrestrial mammals, fat	○ 0.02	0.02			0.02 USA
Cattle, liver	○ 0.02	0.02		1	
Pig, liver	○ 0.02	0.02		1	0.02 USA
Other terrestrial mammals, liver	○ 0.02	0.02		1	
Cattle, kidney	○ 0.2	0.02		1	
Pig, kidney	○ 0.02	0.02		1	0.02 USA
Other terrestrial mammals, kidney	○ 0.2	0.02		1	
Cattle, edible offal	○ 0.2	0.02		1	0.20 USA
Pig, edible offal	○ 0.02	0.02		1	0.02 USA
Other terrestrial mammals, edible offal	○ 0.2	0.02		1	0.20 USA
Potato flakes※4	●	1.6			
Pepper, dried※4	●	15		15	
Plum, dried	●	5			
Raisin※4	●	4		4	

Note: The residue definition is sum of Spirotetramat and metabolite M1 [cis-3-(2,5-dimethylphenyl)-4-hydroxy-8-methoxy-1-azaspiro[4.5]dec-3-en-2-one], expressed as Spirotetramat.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

IT : Import tolerance

※1: The draft MRLs for unshu oranges are estimated by using processing factor (residue concentration ratio of pulp to whole: 0.36) based on the Australian MRL for oranges.

※2: The draft MRLs for peaches are estimated by using processing factor (residue concentration ratio of pulp to whole: 0.37) based on the Codex MRL for peaches.

※3: The draft MRLs for other spices are estimated by using processing factor (residue concentration ratio of orange's peel to whole: 7.2) based on the Australian MRL for oranges.

※4: For potato flakes, dried pepper and raisin, the MRLs of their raw commodities (potatoes, other solanaceous vegetables and grapes, respectively) will be applied taking into account each processing factor. JMPR estimates their processing factors as following: 3.5 for potato flakes; 7 for dried pepper and 2.6 for raisin.

Thifensulfuron methyl

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Wheat	● 0.05	0.1	§		0.05 USA
Barley	● 0.05	0.1	§		0.05 USA
Rye	●	0.1			
Corn (maize, including pop corn and sweet corn)	● 0.05	0.1	IT		0.05 USA
Buckwheat	●	0.1			
Other cereal grains	● 0.05	0.1	IT		0.05 USA
Soybeans, dry	○ 0.1	0.1	IT		0.10 USA
Beans, dry	●	0.1			
Peas	●	0.1			
Broad beans	●	0.1			
Peanuts, dry	●	0.1			
Other pulses	●	0.1			
Sugar beet	●	0.05			
Tomato	○ 0.07		IT		0.07 Canada
Safflower seeds	○ 0.05		IT		0.05 USA
Cotton seeds	○ 0.02	0.02	IT		0.02 USA
Rapeseeds	○ 0.02	0.02	IT		0.02 USA
Other oil seeds	○ 0.02	0.02	IT		0.02 USA
Other spices	●	0.1			

Note: The residue definition is Thifensulfuron methyl only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* Shaded figures indicate provisional MRLs.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

IT : Import tolerance

Thifluzamide

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Rice (brown rice)	○ 1	0.5	§ · Request		
Potato	○ 0.05		Request		
Sugar beet	○ 0.05		Request		
Other vegetables	○ 1	1			1 Korea
Fish	● 1	2			

Note: The residue definition is Thifluzamide only.

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

§ : Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

Albendazole

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Cattle, muscle	● 0.02	0.10		0.1	
Pig, muscle	●	0.10		0.1	
Sheep, muscle	/	0.10			
Horse, muscle	/	0.10			
Other terrestrial mammals, muscle (except sheep and horse)	/	0.1			
Other terrestrial mammals, muscle	○ 0.02	/		0.1	
Cattle, fat	● 0.02	0.10		0.1	
Pig, fat	●	0.10		0.1	
Sheep, fat	/	0.10			
Horse, fat	/	0.10			
Other terrestrial mammals, fat (except sheep and horse)	/	0.1			
Other terrestrial mammals, fat	○ 0.02	/		0.1	
Cattle, liver	● 1	5.0		5	
Pig, liver	●	5.0		5	
Sheep, liver	/	5.0			
Horse, liver	/	5.0			
Other terrestrial mammals, liver (except sheep and horse)	/	5			
Other terrestrial mammals, liver	○ 0.8	/		5	
Cattle, kidney	● 1	5.0		5	
Pig, kidney	●	5.0		5	
Sheep, kidney	/	5.0			
Horse, kidney	/	5.0			
Other terrestrial mammals, kidney (except sheep and horse)	/	5			
Other terrestrial mammals, kidney	○ 0.8	/		5	
Cattle, edible offal	○ 1	0.1			
Pig, edible offal	●	5			
Sheep, edible offal	/	3			
Horse, edible offal	/	5			
Other terrestrial mammals, edible offal (except sheep and horse)	/	0.1			
Other terrestrial mammals, edible offal	○ 0.8	/			
Milk	● 0.02	0.10		0.1	
Chicken, muscle	●	0.10		0.1	
Duck, muscle	/	0.10			
Turkey, muscle	/	0.10			
Other poultry, muscle (except duck and turkey)	/	0.1			
Other poultry, muscle	/	/		0.1	
Chicken, fat	●	0.10		0.1	
Duck, fat	/	0.10			
Turkey, fat	/	0.10			
Other poultry, fat (except duck and turkey)	/	0.1			
Other poultry, fat	/	/		0.1	
Chicken, liver	●	5.0		5	
Duck, liver	/	5.0			
Turkey, liver	/	5.0			
Other poultry, liver (except duck and turkey)	/	5			
Other poultry, liver	/	/		5	

Chicken, kidney	●	5.0	5	
Duck, kidney		5.0		
Turkey, kidney		5.0		
Other poultry, kidney (except duck and turkey)		5		
Other poultry, kidney			5	
Chicken, edible offal	●	5		
Other poultry, edible offal	●	5		

Note: The residue definition is metabolite I [5-(Propylsulfonyl)-1*H*-benzimidazole-2-amine].

* The uniform limit 0.01 ppm will be applied to commodities for which draft MRLs are not given in this table and to commodities not listed above.

* Shaded figures indicate provisional MRLs.

* In the Commodity column, for the food categories to which the word other is added, refer to the Notes given in the last two pages of the Attachment.

In the column of draft MRLs, the commodity with a diagonal line indicates the one which is to be deleted and consolidated into the other category as a result of the rearrangement of food categories. For example, "sheep muscle" and "horse muscle" are to be deleted and included in "other terrestrial mammals muscle."

● : Commodities for which MRLs are to be lowered or deleted

○ : Commodities for which MRLs are to be maintained, increased or newly set

Note 1: The name of the substance for which MRLs are set will be changed from "5-(Propylsulfonyl)-1*H*-benzimidazole-2-amine" to "Albendazole."

Note 2: The above draft MRLs expressed as metabolite I are based on the Codex MRLs of Albendazole expressed as parent compound.

Note 3: Codex MRLs for Albendazole are set for all food-producing animals (Codex does not specify the target animal species.).

Notes:

“Other cereal grains” refers to all cereal grains, except rice (brown rice), wheat, barley, rye, corn (maize), and buckwheat.

“Beans, dry” including butter beans, cowbeans (red beans), lentil, lima beans, pegia, sultani, sultapya

“Other legumes/pulses” refers to all legumes/pulses, except soybeans (dry), beans (dry), peas, broad beans, peanuts (dry), and spices.

“Other potatoes” refers to all potatoes, except potato, taro, sweet potato, yam, and konjac.

“Other cruciferous vegetables” refers to all cruciferous vegetables, except Japanese radish roots and leaves (including radish), turnip roots and leaves, horseradish, watercress, Chinese cabbage, cabbage, brussels sprouts, kale, *komatsuna* (Japanese mustard spinach), *kyona*, qing-geng-cai, cauliflower, broccoli, and herbs.

“Other composite vegetables” refers to all composite vegetables, except burdock, salsify, artichoke, chicory, endive, *shungiku*, lettuce (including cos lettuce and leaf lettuce), and herbs.

“Other liliaceous vegetables” refers to all liliaceous vegetables, except onion, welsh (including leek), garlic, *nira*, asparagus, multiplying onion, and herbs.

“Other umbelliferous vegetables” refers to all umbelliferous vegetables, except carrot, parsnip, parsley, celery, *mitsuba*, spices, and herbs.

“Other solanaceous vegetables” refers to all solanaceous vegetables, except tomato, pimienta (sweet pepper), and egg plant.

“Other cucurbitaceous vegetables” refers to all cucurbitaceous vegetables, except cucumber (including gherkin), pumpkin (including squash), oriental pickling melon (vegetable), watermelon, melons, and *makuwauri* melon.

“Other mushrooms” refers to all mushrooms, except button mushroom, and *shiitake* mushroom.

“Other vegetables” refers to all vegetables, except potatoes, sugar beet, sugarcane, cruciferous vegetables, composite vegetables, liliaceous vegetables, umbelliferous vegetables, solanaceous vegetables, cucurbitaceous vegetables, spinach, bamboo shoots, okra, ginger, peas (with pods, immature), kidney beans (with pods, immature), green soybeans, mushrooms, spices, and herbs.

“Other citrus fruits” refers to all citrus fruits, except *unshu* orange (pulp), citrus *natsudaidai* (pulp), citrus *natsudaidai* (peel), citrus *natsudaidai* (whole), lemon, orange (including navel orange), grapefruit, lime, and spices.

“Other berries” refers to all berries, except strawberry, raspberry, blackberry, blueberry, cranberry, and huckleberry.

“Other fruits” refers to all fruits, except citrus fruits, apple, Japanese pear, pear, quince, loquat, peach, nectarine, apricot, Japanese plum (including prune), mume plum, cherry, berries, grape, Japanese persimmon, banana, kiwifruit, papaya, avocado, pineapple, guava, mango, passion fruit, date and spices.

“Other oil seeds” refers to all oil seeds, except sunflower seeds, sesame seeds, safflower seeds, cotton seeds, rapeseeds and spices.

“Other nuts” refers to all nuts, except ginkgo nut, chestnut, pecan, almond and walnut.

“Other spices” refers to all spices, except horseradish, *wasabi* (Japanese horseradish) rhizomes, garlic, peppers chili, paprika, ginger, lemon peels, orange peels (including navel orange), *yuzu* (Chinese citron) peels and sesame seeds.

“Other herbs” refers to all herbs, except watercress, *nira*, parsley stems and leaves, celery stems and leaves.

“Edible offal” refers to all edible parts, except muscle, fat, liver, and kidney

“Other terrestrial mammals” refers to all terrestrial mammals, except cattle and pig.

“Other poultry animals” refers to all poultry, except chicken.

“Other fish” refers to all fish, except salmoniformes, anguilliformes, and perciformes.

“Other aquatic animals” refers to all aquatic animal, except fish, shelled molluscs and crustaceans.

Item 2. Establishment of Analytical Methods for Agricultural and Veterinary Chemicals in Food

The MHLW notifies analytical methods for certain agricultural and veterinary chemicals in the Ministry of Health and Welfare Notification No. 370.

Any ingredients of agricultural chemicals or other chemical substances shall not be detected in these tests.

The MHLW is going to establish the following analytical methods in the Notification No. 370:

- Analytical method for Coumaphos

Notification (draft)
Analytical Method for Coumaphos
(Targeted to agricultural, animal and fishery products)

The target compound to be determined is coumaphos.

1. Instruments

Gas chromatograph-flame thermionic detector (GC-FTD)

Gas chromatograph-flame photometric detector (with interference filter for phosphorus, wavelength: 526 nm) (GC-FPD(P))

Gas chromatograph-nitrogen phosphorus detector (GC-NPD)

Gas chromatograph-mass spectrometer (GC-MS)

2. Reagents

Use the reagent listed in Section C *Reagent/Test Solution, Etc.*, Part II *Food additives*, except the following.

Acetonitrile: Use a reagent not containing any substance that may interfere with the analysis of the target compound.

Acetone: Use a reagent not containing any substance that may interfere with the analysis of the target compound.

Sodium Chloride: Use a reagent not containing any substance that may interfere with analysis of the target compound.

Silica gel for column chromatography (particle size of 63 to 200 μm): Heat silica gel made for column chromatography (particle size of 63 to 200 μm) at 130°C for 12 hours or longer. Cool down to room temperature in a desiccator.

Diatomaceous earth: Use diatomaceous earth for chemical analysis.

Ethyl acetate: Use a reagent not containing any substance that may interfere with analysis of the target compound.

Porous diatomaceous earth cartridge (to hold 20 mL of solution): A polyethylene tube of 20–30 mm in inside diameter packed with granular porous diatomaceous earth prepared for column chromatography (to hold 20 mL of solution), or a cartridge with equivalent to the specified one in separation capability.

Trimethylaminopropylsilanized silica gel/ethylenediamine-*N*-propylsilanized silica gel layered cartridge (500 mg/500 mg): A polyethylene tube of 12–13 mm in inside diameter packed with 500 mg of trimethylaminopropylsilanized silica gel in the upper

layer and 500 mg of ethylenediamine-*N*-propylsilylanized silica gel in the lower layer, or a cartridge equivalent to the specified one in separation capability.

n-Hexane: Use a reagent not containing any substance that may interfere with analysis of the target compound.

Water: Use water suitable for chemical analysis, including distilled water, purified water, or pure water. If it contains any substance that may interfere with analysis of the target compound, wash with a solvent such as *n*-hexane before use.

Anhydrous sodium sulfate: Use a reagent not containing any substance that may interfere with analysis of the target compound.

3. Reference standard

Reference standard of coumaphos: Contains not less than 98% of coumaphos.

4. Procedure

a. Extraction

(i) Grains, legumes, nuts and seeds

Grind sample to pass through a standard sieve (425 μm), weigh 10.0 g of the sample, add 20 mL of water, and let stand for 30 minutes.

Add 100 mL of acetone to the sample, homogenize for 3 minutes, and filter through a filter paper, covered with a 1-cm-thick layer of diatomaceous earth, with suction into a vacuum rotary evaporator flask. Collect the residue on the filter paper, add 50 mL of acetone, homogenize for 3 minutes, treat as described above, combine the filtrate in the vacuum rotary evaporator flask, and remove acetone at below 40°C.

Transfer to a 300 mL separating funnel containing 100 mL of saturated sodium chloride solution. Wash the vacuum rotary evaporator flask with 100 mL of ethyl acetate/*n*-hexane (1:4, v/v), and transfer the washing to the separating funnel. Shake the separating funnel vigorously for 5 minutes with a shaker, let stand, and transfer the ethyl acetate/*n*-hexane layer to a 300 mL conical flask. Add 50 mL of ethyl acetate/*n*-hexane (1:4, v/v) to the aqueous layer, treat as described above, and combine the ethyl acetate/*n*-hexane layers in the conical flask. Add an appropriate quantity of anhydrous sodium sulfate to the ethyl acetate/*n*-hexane layer, let stand for 15 minutes with occasional shaking, and filter into a vacuum rotary evaporator flask. Wash the conical flask with 20 mL of *n*-hexane, and wash the residue on the filter paper with the washing. Repeat this step one more time. Combine the washings in the vacuum rotary evaporator flask, and remove ethyl acetate/*n*-hexane at below

40°C.

Add 30 mL of *n*-hexane to the residue, and transfer to a 100 mL separating funnel. Add 30 mL of acetonitrile saturated with *n*-hexane to the separating funnel, shake vigorously for 5 minutes with a shaker, let stand, and transfer the acetonitrile layer to a vacuum rotary evaporator flask. Add 30 mL of acetonitrile saturated with *n*-hexane to the *n*-hexane layer, treat as described above twice, combine the acetonitrile layers in the vacuum rotary evaporator flask, and remove acetonitrile at below 40°C. Dissolve the residue in 5 mL of acetone/*n*-hexane (1:1, v/v).

(ii) Fruits, vegetables, tea leaves and hops

For fruits and vegetables, weigh about 1 kg of sample accurately, add an appropriate quantity of water (if necessary), homogenize, and then take the sample equivalent to 20.0 g.

For tea leaves and hops, grind the sample, weigh 5.00 g, add 20 mL of water, and let stand for 30 minutes.

Add 100 mL of acetone to the sample, homogenize for 3 minutes, and filter through a filter paper, covered with a 1-cm-thick layer of diatomaceous earth, with suction into a vacuum rotary evaporator flask. Collect the residue on the filter paper, add 50 mL of acetone, homogenize for 3 minutes, treat as described above, combine the filtrate in the vacuum rotary evaporator flask, and remove acetone at below 40°C.

Transfer to a 300 mL separating funnel containing 100 mL of saturated sodium chloride solution. Wash the vacuum rotary evaporator flask with 100 mL of ethyl acetate/*n*-hexane (1:4, v/v), and transfer the washing to the separating funnel. Shake the separating funnel vigorously for 5 minutes with a shaker, let stand, and transfer the ethyl acetate/*n*-hexane layer to a 300 mL conical flask. Add 50 mL of ethyl acetate/*n*-hexane (1:4, v/v) to the aqueous layer, treat as described above, and combine the ethyl acetate/*n*-hexane layers in the conical flask. Add an appropriate quantity of anhydrous sodium sulfate to the ethyl acetate/*n*-hexane layer, let stand for 15 minutes with occasional shaking, and filter into a vacuum rotary evaporator flask. Wash the conical flask with 20 mL of *n*-hexane, and wash the residue on the filter paper with the washing. Repeat this step one more time. Combine the washings in the vacuum rotary evaporator flask, and remove ethyl acetate/*n*-hexane at below 40°C. Dissolve the residue in 5 mL of acetone/*n*-hexane (1:1, v/v).

(iii) Muscle, fat, liver, kidney and fish/shellfish

For muscle, liver, kidney and fish/shellfish, weigh 20.0 g of sample. For fat, weigh 5.00 g of sample.

Add 20 mL of 0.1 mol/L hydrochloric acid to the sample, homogenize, add 100 mL of

acetone/*n*-hexane (1:2, v/v), homogenize again, centrifuge at 3,000 rpm for 5 minutes, and collect the organic layer. Add 50 mL of *n*-hexane to the residue, homogenize, and centrifuge at 3,000 rpm for 5 minutes. Combine the resulting organic layers, dehydrate the extract with anhydrous sodium sulfate, and filter out the anhydrous sodium sulfate. Concentrate the filtrate at below 40°C and remove the solvent. Dissolve the residue in *n*-hexane to make exactly 20 mL.

(iv) Milk, egg and honey

Weigh 10.0 g of sample.

Add 10 mL of 0.1 mol/L hydrochloric acid to the sample, homogenize, add 100 mL of acetone/*n*-hexane (1:2, v/v), homogenize again, centrifuge at 3,000 rpm for 5 minutes, and collect the organic layer. Add 50 mL of *n*-hexane to the residue, homogenize, and centrifuge at 3,000 rpm for 5 minutes. Combine the resulting organic layers, dehydrate the extract with anhydrous sodium sulfate, and filter out the anhydrous sodium sulfate. Concentrate the filtrate at below 40°C and remove the solvent. For milk and egg, dissolve the residue in *n*-hexane to make exactly 10 mL. For honey, dissolve the residue in acetone/*n*-hexane (1:9, v/v) to make exactly 5 mL.

b. Clean-up

(i) Grains, legumes, nuts and seeds, fruits, vegetables, tea leaves and hops

Place 5 g of silica gel for column chromatography (particle size of 63 to 200 μm) suspended in acetone/*n*-hexane (1:1, v/v), and then about 5 g of anhydrous sodium sulfate in a chromatographic tube of 15 mm in inside diameter and 300 mm in length, and let flow out acetone/*n*-hexane (1:1, v/v) to the extent that only a small quantity of acetone/*n*-hexane (1:1, v/v) remains on the top of the column. Transfer the solution obtained in “a. Extraction” to the column, elute with 100 mL of acetone/*n*-hexane (1:1, v/v), collect the eluate in a vacuum rotary evaporator flask, and remove the acetone/*n*-hexane at below 40°C. Dissolve the residue in acetone to make exactly 5 mL, and use this solution as the test solution.

(ii) Muscle, fat, liver, kidney, fish/shellfish, milk and egg

① Porous diatomaceous earth column chromatography

For Muscle, fat, liver, kidney and fish/shellfish, transfer exactly 10 mL of the solution obtained in “a. Extraction (iii)” to a porous diatomaceous earth column (to hold 20 mL of solution). For milk and egg, transfer total solution obtained in “a. Extraction (iv)” to the column. Let stand the column for 10 minutes, and then aspirate the column for 10 minutes and remove most of the solvent. Elute with 90 mL of acetonitrile saturated with *n*-hexane, concentrate the eluate at below 40°C and

remove the solvent. Dissolve the residue in acetone/*n*-hexane (1:9, v/v) to make exactly 5 mL.

② Trimethylaminopropylsilanized silica gel/ethylenediamine-*N*-propylsilanized silica gel layered column chromatography

Add 10 mL of acetone/*n*-hexane (1:9, v/v) to a trimethylaminopropylsilanized silica gel/ethylenediamine-*N*-propylsilanized silica gel layered cartridge (500 mg/500 mg), and discard the effluent. Transfer exactly 2 mL of the solution obtained in “① Porous diatomaceous earth column chromatography” to the cartridge, elute with 10 mL of acetone/*n*-hexane (1:9, v/v), collect the total eluate, concentrate at below 40°C and remove the solvent. Dissolve the residue in acetone/*n*-hexane (1:9, v/v) to make exactly 4 mL (1 mL for fat), and use this solution as the test solution.

(iii) Honey

Add 10 mL of acetone/*n*-hexane (1:9, v/v) to a trimethylaminopropylsilanized silica gel/ethylenediamine-*N*-propylsilanized silica gel layered cartridge (500 mg/500 mg) and discard the effluent. Transfer exactly 2 mL of the solution obtained in “a. Extraction (iv)” to the cartridge, elute with 10 mL of acetone/*n*-hexane (1:9, v/v), collect the total eluate, concentrate at below 40°C and remove the solvent. Dissolve the residue in acetone/*n*-hexane (1:9, v/v) to make exactly 4 mL, and use this solution as the test solution.

5. Measurement

a. Calibration curve

(i) Grains, legumes, nuts and seeds, fruits, vegetables, tea leaves and hops

Prepare coumaphos standard solutions (acetone) of several concentrations. Inject each standard solution to GC-FTD, GC-FPD(P) or GC-NPD, and make a calibration curve by peak-height or peak-area method. When the test solution is prepared following the above procedure, the sample containing 0.01 mg/kg of coumaphos gives the test solution of 0.02 mg/L for grains, legumes, nuts and seeds, 0.04 mg/L for fruits and vegetables, and 0.01 mg/L for tea leaves and hops in concentration.

(ii) Muscle, fat, liver, kidney, fish/shellfish, milk, egg and honey

Prepare coumaphos standard solutions (acetone/*n*-hexane (1:9, v/v)) of several concentrations. Inject each standard solution to GC-FTD, GC-FPD(P) or GC-NPD, and make a calibration curve by peak-height or peak-area method. When the test solution is prepared following the above procedure, the sample containing 0.01 mg/kg of coumaphos gives the test solution of 0.01 mg/L in concentration.

b. Quantification

Inject the test solution to GC-FTD, GC-FPD(P) or GC-NPD, and calculate the concentration of coumaphos from the calibration curve made in “a. Calibration curve”.

c. Confirmation

Confirm using GC-MS.

d. Measurement conditions

(i) For quantification

Condition 1 (GC-FTD, GC-FPD(P) or GC-NPD)

Column: Silicate glass capillary column 0.53 mm in inside diameter, 10–30 m in length coated with methyl silicone for gas chromatography 1.5 μm in film thickness

Column temperature: The column temperature is held at 80°C for 1 minute, followed by an increase of 8°C every minute until reaching 250°C, where it is held for 5 minutes.

Inlet temperature: 230°C

Detector temperature: 280°C

Gas flow rate: Helium is used as the carrier gas. The flow rate should be adjusted to the optimal condition. The flows of air and hydrogen should also be adjusted to the optimal conditions.

Condition 2 (GC-FTD, GC-FPD(P) or GC-NPD)

Column: Silicate glass capillary column 0.32 mm in inside diameter, 10–30 m in length coated with 50% trifluoro propyl methyl silicone for gas chromatography 0.25 μm in film thickness

Column temperature: The column temperature is held at 70°C for 1 minute, followed by an increase of 25°C every minute until reaching 125°C, after which the temperature is increased by 10°C every minute until reaching 235°C, where it is held for 12 minutes.

Inlet temperature: 230°C

Detector temperature: 280°C

Gas flow rate: Helium is used as the carrier gas. The flow rate should be adjusted to the optimal condition. The flows of air and hydrogen should also be adjusted to the optimal conditions.

Condition 3 (GC-FPD(P))

Column: Silicate glass capillary column 0.32 mm in inside diameter, 30 m in length

coated with 35% trifluoro propyl methyl silicone for gas chromatography 0.5 μm in film thickness

Column temperature: The column temperature is held at 60°C for 1 minute, followed by an increase of 25°C every minute until reaching 210°C, after which the temperature is increased by 10°C every minute until reaching 280°C, where it is held for 10 minutes.

Inlet temperature: 250°C

Detector temperature: 280°C

Gas flow rate: Helium is used as the carrier gas. The flow rate should be adjusted to the optimal condition. The flows of air and hydrogen should also be adjusted to the optimal conditions.

Injection volume: 2 μL

Expected retention time: 18 min

(ii) For confirmation

Condition (GC-MS)

Column: Silicate glass capillary column 0.25 mm in inside diameter, 30 m in length coated with 5% phenyl-methyl silicone for gas chromatography 0.25 μm in film thickness

Column temperature: The column temperature is held at 60°C for 1 minute, followed by an increase of 25°C every minute until reaching 210°C, after which the temperature is increased by 10°C every minute until reaching 300°C, where it is held for 8 minutes.

Inlet temperature: 250°C

Carrier gas: Helium is used as the carrier gas. The flow rate should be adjusted to the optimal condition.

Ionization mode (ionization energy): EI (70 eV)

Major monitoring ions (m/z): 364, 362, 226

Injection volume: 2 μL

Expected retention time: 15 min

6. Limit of quantification

0.01 mg/kg