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Japan proposes the revision of MRLs for 11 agricultural chemicals

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Approved By:
Jess K. Paulson
Prepared By:
Jess K. Paulson

Report Highlights:
On Friday, June 17, 2016, the Ministry of Health, Labor and Welfare (MHLW) of the Government of Japan (GOJ) announced changes to the Maximum Residue Levels (MRLs) for the pesticides Bicyclopyrone, 1,3-Dichloropropene, Ethofumesate, Etofenprox, Fluazifop-butyl, and Isopyrazam, and for the veterinary drugs Erythromycin, Flumethrin, Metoclopramide, Piperazine, and Vedaprofen. MHLW also established analytical methods for agricultural and veterinary chemicals in food for Ipronidazole, Kimetridazole, Metronidazole, Ronidazole, Chloramphenicol, and for Captafol in animal and fishery products.

The Embassy comment period for these proposals is open until Friday, July 1, 2016. MHLW will also notify these MRLs to the WTO, which will allow another opportunity for interested parties to comment on these changes. Japan has previously notified proposed revisions to Etofenprox (JPN/SPS/435) and Fluazifop-butyl (JPN/SPS/458).

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 (MHLW) 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 System[1] for agricultural and veterinary chemicals in food. All foods distributed in the Japanese marketplace are subject to regulation of the system.

The 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:	Bicyclopyrone, 1,3-Dichloropropene, Ethofumesate, Etofenprox, Fluazifop-butyl, Isopyrazam
Veterinary drug:	Erythromycin, Flumethrin, Metoclopramide, Piperazine, Vedaprofen

[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

Bicyclopyrone (pesticide: herbicide): 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.

1,3-Dichloropropene (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 MAFF. This action will not strengthen the current regulation for any commodities.

Ethofumesate (pesticide: herbicide): Not permitted for use in Japan.
The MHLW is going to establish MRLs in some commodities in some commodities in response to a request for setting MRLs by the MAFF. The MHLW is also going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System.

Etofenprox (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 a WTO member country through WTO/SPS notification (G/SPS/N/JPN/435). This action will not strengthen the current regulation for any commodities.

Fluazifop-butyl (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.

Isopyrazam (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 MRLs by the MAFF and 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).

Erythromycin (veterinary drug: antibiotic): 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.

Flumethrin (veterinary drug: ectoparasiticide): Permitted for use in Japan.
The Minister of Agriculture, Forestry and Fisheries asked the minister of Health, Labour and Welfare for comments about application for manufacture and sales approval as a veterinary drug, and modification of the application of this substance, under the Act on Securing Quality, Efficacy and Safety of Pharmaceuticals, Medical Devices, Regenerative and Cellular Therapy Products, Gene Therapy Products, and Cosmetics. In response to this, the MHLW is going to modify MRLs in some commodities that were provisionally set at the introduction of the Positive List System.

Metoclopramide (veterinary drug: gastrointestinal medicine): 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.

Piperazine (veterinary drug: ectoparasiticide): 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.

Vedaprofen (veterinary drug: anti-inflammatory agent): 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. This action will not strengthen the current regulation for any commodities.

Bicyclopyrone

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Corn (maize, including pop corn and sweet corn)	○ 0.03		IT		0.03: USA

Note: The residue definition is sum of Bicyclopyrone, metabolite B [2-(2-methoxy-ethoxymethyl)-6-trifluoromethyl-nicotinic acid] (including metabolites that are hydrolized to metabolite B) and metabolite K [2-(2-hydroxy-ethoxymethyl)-6-trifluoromethyl-nicotinic acid] (including metabolites that are hydrolized to metabolite K), expressed as Bicyclopyrone.

* 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.

○: Commodities for which MRLs are to be maintained or increased
IT : Import tolerance

1,3-Dichloropropene

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Soybeans, dry	○ 0.01	0.01	§		
Peanuts, dry	○ 0.01	0.01	§		
Potato	○ 0.01	0.01	§		
Taro	○ 0.01	0.01	§		
Sweet potato	○ 0.01	0.01	§		
Yam	○ 0.01	0.01	§		
Konjac	○ 0.01	0.01	§		
Sugar beet	○ 0.01	0.01	§		
Japanese radish, roots (including radish)	○ 0.01	0.01	§		
Japanese radish, leaves (including radish)	○ 0.01	0.01	§		
Turnip, roots (including rutabaga)	○ 0.01	0.01	§		
Turnip, leaves (including rutabaga)	○ 0.01	0.01	§		
Chinese cabbage	○ 0.01	0.01	§		
Cabbage	○ 0.01	0.01	§		
Kale	○ 0.01		Request		
Komatsuna(Japanese mustard spinach)	○ 0.01	0.01	§		
Kyona	○ 0.01	0.01	§		
Qing-geng-cai	○ 0.01	0.01	§		
Other cruciferous vegetables	○ 0.01		Request		
Burdock	○ 0.01	0.01	§		
Lettuce (including cos lettuce and leaf lettuce)	○ 0.01	0.01	§		
Other composite vegetables	○ 0.01	0.01	§		
Onion	○ 0.01	0.01	§		
Welsh (including leek)	○ 0.01	0.01	§		
Garlic	○ 0.01	0.01	§		
Nira	○ 0.01	0.01	§		
Multiplying onion (including shallot)	○ 0.01	0.01	§		
Other liliaceous vegetables	○ 0.01	0.01	§		
Carrot	○ 0.01	0.01	§		
Parsley	○ 0.01	0.01	§		
Celery	○ 0.01	0.01	§		
Mitsuba	○ 0.01	0.01	§		
Tomato	○ 0.01	0.01	§		
Pimiento (sweet pepper)	○ 0.01	0.01	§		
Egg plant	○ 0.01	0.01	§		
Other solanaceous vegetables	○ 0.01	0.01	§		
Cucumber (including gherkin)	○ 0.01	0.01	§		
Pumpkin (including squash)	○ 0.01	0.01	§		
Oriental pickling melon (vegetable)	○ 0.01	0.01	§		
Water melon	○ 0.01	0.01	§		
Melons	○ 0.01	0.01	§		
Makuwauri melon	○ 0.01	0.01	§		
Other cucurbitaceous vegetables	○ 0.01	0.01	§		
Spinach	○ 0.01	0.01	§		
Okra	○ 0.01	0.01	§		
Ginger	○ 0.01	0.01	§		
Peas, immature (with pods)	○ 0.01		Request		
Kidney beans, immature (with pods)	○ 0.01	0.01	§		
Green soybeans	○ 0.01	0.01	§		
Other vegetables	○ 0.01	0.01	§		
Strawberry	○ 0.01	0.01	§		

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Other herbs	○ 0.01	0.01	§		⋮
Mineral water	○ 0.02	0.02		0.02※	⋮

Note: The residue definition is sum of (*E*)-1,3-Dichloropropene and (*Z*)-1,3-Dichloropropene.

* 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 or increased

§: Permitted for use in Japan.

Request : The MRL was modified in response to MAFF request

※MRL for mineral water is based on the WHO Guideline Value for drinking-water. (A guideline value normally represents the concentration of a constituent that does not result in any significant risk to health over a lifetime of consumption.)

Ethofumesate

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
					Codex ppm	National ppm	
Sugar beet	○	0.3	0.1	Request		0.3	USA
Onion	○	0.3	0.1			0.25	USA
Welsh (including leek)	●		0.1				
Garlic	○	0.3	0.1			0.25	USA
Multiplying onion (including shallot)	●		0.1				
Other liliaceous vegetables	●		0.1				
Other umbelliferous vegetables	●		0.1				
Other vegetables	○	5	1			5.0	USA
Other oil seeds	●		0.02				
Other spices	●		1				
Other herbs	●		1				
Cattle, muscle	○	0.5	0.05			0.5	Australia
Pig, muscle	○	0.5	0.05			0.5	Australia
Other terrestrial mammals, muscle	○	0.5	0.05			0.5	Australia
Cattle, fat	○	0.5	0.3			0.5	Australia
Pig, fat	○	0.5	0.3			0.5	Australia
Other terrestrial mammals, fat	○	0.5	0.3			0.5	Australia
Cattle, liver	○	0.5	0.3			0.5	Australia
Pig, liver	○	0.5	0.3			0.5	Australia
Other terrestrial mammals, liver	○	0.5	0.3			0.5	Australia
Cattle, kidney	○	3	0.3				
Pig, kidney	○	3	0.3				
Other terrestrial mammals, kidney	○	3	0.3				
Cattle, edible offal	○	3	0.3				
Pig, edible offal	○	3	0.3				
Other terrestrial mammals, edible offal	○	3	0.3				
Milk	○	0.2	0.2			0.2	Australia

Note: The residue definition will be changed to sum of Ethofumesate, metabolite M2 [2,3-dihydro-3,3-dimethyl-2-oxobenzofuran-5-yl methanesulfonate], metabolites that are converted to metabolite M2 by heat treatment under acidic condition (including metabolite M3 [2-(2-hydroxy-5-methanesulfoxy-phenyl)-2-methylpropanoic acid] and its conjugates), expressed as Ethofumesate.
The current residue definition is Ethofumesate 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

○: Commodities for which MRLs are to be maintained or increased (* It should be noted that the residue definition will be changed.)

Request：The MRL was modified in response to MAFF request

Etofenprox

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
					Codex ppm	National ppm
Rice (brown rice)	○	0.5	0.5	§		
Wheat	○	0.5	0.5	§		
Barley	○	0.5	0.5			
Rye	○	0.5	0.5			
Corn (maize, including pop corn and sweet corn)	●	0.3	0.5	§	0.05	
Other cereal grains	○	3		Request		
Soybeans, dry	○	0.2	0.2	§	0.05	
Beans, dry	○	0.2	0.2	§	0.05	
Peas	○	0.05	0.05	§		
Broad beans	○	0.05	0.05	§	0.05	
Peanuts, dry	○	0.05	0.05	§		
Other pulses	○	0.05	0.05	§	0.05	
Potato	●	0.05	0.1	§		
Taro	○	0.1	0.1	§		
Sweet potato	●	0.03	0.1	§		
Yam	○	0.1	0.1	§		
Sugar beet	●	0.3	0.5	§		
Sugarcane	●	0.03	0.1	§		
Japanese radish, roots (including radish)	○	2	2	§		
Japanese radish, leaves (including radish)	○	10	10	§		
Turnip, roots (including rutabaga)	○	2	2			
Turnip, leaves (including rutabaga)	○	10	10			
Chinese cabbage	○	5	5	§		
Cabbage	○	2	2	§		
Brussels sprouts	○	2	2			
Broccoli	○	10		Request		
Other cruciferous vegetables	○	1	1	§		
Lettuce (including cos lettuce and leaf lettuce)	○	2	2	§		
Other composite vegetables	○	2	2	§		
Welsh (including leek)	○	2	2	§		
Multiplying onion (including shallot)	○	2	2			
Mitsuba	○	5	5	§		
Other umbelliferous vegetables	○	2	2	§		
Tomato	○	2	2	§		
Pimiento (sweet pepper)	○	5	5	§		
Egg plant	○	2	2	§		
Other solanaceous vegetables	○	2	2			
Cucumber (including gherkin)	●	1	2	§		
Pumpkin (including squash)	○	1	1	§		
Water melon	○	2	2	§		
Melons	○	2	2	§		
Makuwauri melon	○	2	2			
Other cucurbitaceous vegetables	○	1	1	§		
Okra	○	3	3	§		
Ginger	○	2	2	§		
Peas, immature (with pods)	○	2	2	§		
Kidney beans, immature (with pods)	○	2	2	§		
Green soybeans	●	3	5	§		
Other vegetables	○	10	5	§ ・ Request		
Unshu orange, pulp	○	2	2	§		
Citrus natsudaidai, whole	○	3	3	§		

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
				Codex ppm	National ppm	
Lemon	○ 5	5	§			
Orange (including navel orange)	○ 5	5	§			
Grapefruit	○ 5	5	§			
Lime	○ 5	5	§			
Other citrus fruits	○ 5	5	§			
Apple	○ 2	2	§	0.6		
Japanese pear	○ 2	2	§	0.6		
Pear	○ 2	2	§	0.6		
Peach	○ 2	2	§			
Nectarine	○ 0.6	0.6		0.6		
Grape	○ 4	4		4		
Japanese persimmon	○ 2	2	§			
Mango	○ 5	5	§			
Rapeseeds	○ 0.01	0.01		0.01		
Chestnut	○ 2	2	§			
Tea	○ 10	10	§			
Other spices	○ 20	20	§			
Other herbs	○ 0.7	0.7	§			
Cattle, muscle	○ 0.5	0.5				
Pig, muscle	○ 0.5	0.5				
Other terrestrial mammals, muscle	○ 0.5	0.5				
Cattle, fat	○ 7	7		0.5		
Pig, fat	○ 7	7		0.5		
Other terrestrial mammals, fat	○ 7	7		0.5		
Cattle, liver	○ 0.5	0.5		0.05		
Pig, liver	○ 0.5	0.5		0.05		
Other terrestrial mammals, liver	○ 0.5	0.5		0.05		
Cattle, kidney	○ 0.5	0.5		0.05		
Pig, kidney	○ 0.5	0.5		0.05		
Other terrestrial mammals, kidney	○ 0.5	0.5		0.05		
Cattle, edible offal	○ 0.5	0.5		0.05		
Pig, edible offal	○ 0.5	0.5		0.05		
Other terrestrial mammals, edible offal	○ 0.5	0.5		0.05		
Milk	○ 0.5	0.5		0.02		
Chicken, muscle	○ 0.01	0.01		0.01	0.01	USA
Other poultry animals, muscle	○ 0.01	0.01		0.01	0.01	USA
Chicken, fat	○ 1	0.5			1.0	USA
Other poultry animals, fat	○ 1	0.5			1.0	USA
Chicken, liver	○ 0.06	0.02		0.01		
Other poultry animals, liver	○ 0.06	0.02		0.01		
Chicken, kidney	○ 0.06	0.02		0.01		
Other poultry animals, kidney	○ 0.06	0.02		0.01		
Chicken, edible offal	○ 0.06	0.02		0.01		
Other poultry animals, edible offal	○ 0.06	0.02		0.01		
Chicken, eggs	○ 0.4	0.1		0.01	0.40	USA
Other poultry, eggs	○ 0.4	0.1		0.01	0.40	USA
Fish	○ 0.8	0.8				
Raisin	○ 8	8		8		

Note: The residue definition is Etofenprox 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
○: Commodities for which MRLs are to be maintained or increased
§：Permitted for use in Japan.
Request：The MRL was modified in response to MAFF request

Fluazifop-butyl

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Soybeans, dry	○ 3	1	§ · IT		2.5 USA
Beans, dry	○ 5	5	§		
Peas	● 0.2	5			0.15 Canada
Broad beans	● 0.2	5			0.15 Canada
Peanuts, dry	● 2	5	§		1.5 USA
Other pulses	○ 0.1	0.1			
Potato	○ 0.1	0.1	§		
Taro	●	0.1			
Sweet potato	● 0.05	0.5	§		
Yam	● 0.05	0.1	§		
Konjac	●	0.1			
Other potatoes	●	0.1			
Sugar beet	○ 0.2	0.2	§		
Sugarcane	●	0.1			
Japanese radish, roots (including radish)	● 0.2	0.5	§		
Japanese radish, leaves (including radish)	○ 0.2	0.2	§		
Turnip, roots (including rutabaga)	●	0.1			
Turnip, leaves (including rutabaga)	●	0.1			
Horseradish	●	0.1			
Watercress	●	0.1			
Chinese cabbage	●	0.1			
Cabbage	○ 2	2	§		
Brussels sprouts	●	2			
Kale	●	0.1			
Komatsuna(Japanese mustard spinach)	●	0.1			
Kyona	●	0.1			
Qing-geng-cai	●	1			
Cauliflower	●	1			
Broccoli	○ 1	1	§		
Other cruciferous vegetables	●	1			
Burdock	●	0.1			
Salsify	●	0.5			
Artichoke	●	0.2			
Chicory	●	0.2			
Endive	●	6			
Shungiku	●	0.2			
Lettuce (including cos lettuce and leaf lettuce)	●	0.1			
Other composite vegetables	●	0.2			
Onion	● 0.3	0.5	§		
Welsh (including leek)	●	0.1			
Garlic	● 0.3	0.5	§		
Nira	●	0.1			
Asparagus	○ 3	3	§		3.0 USA
Multiplying onion (including shallot)	●	0.1			
Other liliaceous vegetables	●	0.1			
Carrot	● 1	2	§		
Parsnip	●	0.5			
Parsley	●	0.2			
Celery	●	0.02			
Mitsuba	●	0.2			
Other umbelliferous vegetables	●	0.5			

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Tomato	• 0.05	0.1	§		
Pimiento (sweet pepper)	•	0.02			
Egg plant	•	0.1			
Other solanaceous vegetables	•	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	•	0.2			
Bamboo shoots	•	0.5			
Ginger	•	0.5			
Peas, immature (with pods)	•	0.1			
Kidney beans, immature (with pods)	•	0.1			
Green soybeans	○ 0.1	0.1	§		
Other vegetables	•	0.5			
Unshu orange, pulp	• 0.05	0.1	§		
Citrus natsudaidai, whole	• 0.05	0.1	§		
Lemon	• 0.05	0.1	§		
Orange (including navel orange)	• 0.05	0.1	§		
Grapefruit	• 0.05	0.1	§		
Lime	• 0.05	0.1	§		
Other citrus fruits	• 0.05	0.1	§		
Apple	•	0.1	§		
Japanese pear	• 0.05	0.1	§		
Pear	• 0.05	0.1	§		
Quince	○	0.01			
Loquat	○	0.01			
Peach	•	0.05			
Nectarine	•	0.05			
Apricot	•	0.05			
Japanese plum (including prune)	•	0.05			
Mume plum	•	0.05			
Cherry	•	0.05			
Strawberry	•	0.2			
Raspberry	•	0.2			
Blackberry	•	0.2			
Blueberry	•	0.2			
Cranberry	•	0.2			
Huckleberry	•	0.2			
Other berries	•	0.2			
Grape	•	0.2			
Japanese persimmon	•	0.1			
Banana	○ 0.1	0.1			
Kiwifruit	•	0.05			
Papaya	•	0.05			
Avocado	•	0.02			
Pineapple	○ 0.05	0.05			
Guava	•	0.05			

Commodity	MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
				Codex ppm	National ppm
Mango	•	0.05			
Passion fruit	•	0.05			
Other fruits	•	0.1			
Sunflower seeds	•	0.5			
Sesame seeds	•	0.5			
Safflower seeds	•	0.5			
Cotton seeds	•	0.5			
Rapeseeds	•	0.5			
Other oil seeds	•	0.5			
Pecan	•	0.05			
Other nuts	•	0.1			
Coffee beans	○ 0.1	0.1			
Hop	•	0.05			
Other spices	• 0.3	0.5	§		0.3 Canada
Other herbs	•	1			
Cattle, muscle	• 0.03	0.05			
Pig, muscle	• 0.03	0.05			
Other terrestrial mammals, muscle	• 0.03	0.05			
Cattle, fat	• 0.03	0.05			
Pig, fat	• 0.03	0.05			
Other terrestrial mammals, fat	• 0.03	0.05			
Cattle, liver	• 0.03	0.05			
Pig, liver	• 0.03	0.05			
Other terrestrial mammals, liver	• 0.03	0.05			
Cattle, kidney	• 0.03	0.05			
Pig, kidney	• 0.03	0.05			
Other terrestrial mammals, kidney	• 0.03	0.05			
Cattle, edible offal	• 0.03	0.05			
Pig, edible offal	• 0.03	0.05			
Other terrestrial mammals, edible offal	• 0.03	0.05			
Milk	• 0.03	0.05			
Chicken, muscle	• 0.02	0.05			
Other poultry animals, muscle	• 0.02	0.05			
Chicken, fat	• 0.02	0.05			
Other poultry animals, fat	• 0.02	0.05			
Chicken, liver	• 0.04	0.05			
Other poultry animals, liver	• 0.04	0.05			
Chicken, kidney	• 0.04	0.05			
Other poultry animals, kidney	• 0.04	0.05			
Chicken, edible offal	• 0.04	0.05			
Other poultry animals, edible offal	• 0.04	0.05			
Chicken, eggs	○ 0.05	0.05			
Other poultry, eggs	○ 0.05	0.05			

Note: The residue definition will be changed to sum of Fluazifop-butyl and Fluazifop acid (including metabolites that are hydrolized to Fluazifop acid), expressed as Fluazifop-butyl. Fluazifop-butyl includes Fluazifop-P-butyl, and Fluazifop acid includes Fluazifop-P acid.
The current residue definition is sum of Fluazifop-butyl, Fluazifop acid, Fluazifop-P acid and Fluazifop-P-butyl, expressed as Fluazifop-butyl.

* 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
○: Commodities for which MRLs are to be maintained or increased (* It should be noted that the residue definition will be changed.)
§ : Permitted for use in Japan.
IT : Import tolerance

Isopyrazam

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
					Codex ppm	National ppm	
Wheat	o	0.2	0.2		0.03	0.2	EU
Barley	o	0.6	0.6		0.07	0.6	EU
Rye	o	0.2	0.2		0.03	0.2	EU
Other cereal grains	o	0.2	0.2		0.03	0.2	EU
Chinese cabbage	o	5		Request			
Cabbage	o	3		Request			
Lettuce (including cos lettuce and leaf lettuce)	o	10		Request			
Tomato	o	3		Request			
Egg plant	o	2		Request			
Cucumber (including gherkin)	o	1		Request			
Pumpkin (including squash)	o	0.05		IT		0.05	New Zealand
Melons	o	0.05		Request			
Apple	o	5		Request			
Japanese pear	o	3		Request			
Pear	o	3		Request			
Peach	o	0.2		Request			
Apricot	o	5		Request			
Japanese plum (including prune)	o	2		Request			
Mume plum	o	5		Request			
Strawberry	o	5		Request			
Grape	o	10		Request			
Japanese persimmon	o	2		Request			
Banana	o	0.06	0.06		0.06		
Cattle, muscle	o	0.01	0.01		0.01		
Pig, muscle	o	0.01	0.01		0.01		
Other terrestrial mammals, muscle	o	0.01	0.01		0.01		
Cattle, fat	o	0.01	0.01		0.01		
Pig, fat	o	0.01	0.01		0.01		
Other terrestrial mammals, fat	o	0.01	0.01		0.01		
Cattle, liver	o	0.02	0.02		0.02		
Pig, liver	o	0.02	0.02		0.02		
Other terrestrial mammals, liver	o	0.02	0.02		0.02		
Cattle, kidney	o	0.02	0.02		0.02		
Pig, kidney	o	0.02	0.02		0.02		
Other terrestrial mammals, kidney	o	0.02	0.02		0.02		
Cattle, edible offal	o	0.02	0.02		0.02		
Pig, edible offal	o	0.02	0.02		0.02		
Other terrestrial mammals, edible offal	o	0.02	0.02		0.02		
Milk	o	0.01	0.01		0.01		
Chicken, muscle	o	0.01	0.01		0.01		
Other poultry animals, muscle	o	0.01	0.01		0.01		
Chicken, fat	o	0.01	0.01		0.01		
Other poultry animals, fat	o	0.01	0.01		0.01		
Chicken, liver	o	0.01	0.01		0.01		
Other poultry animals, liver	o	0.01	0.01		0.01		
Chicken, kidney	o	0.01	0.01		0.01		
Other poultry animals, kidney	o	0.01	0.01		0.01		
Chicken, edible offal	o	0.01	0.01		0.01		
Other poultry animals, edible offal	o	0.01	0.01		0.01		
Chicken, eggs	o	0.01	0.01		0.01		
Other poultry, eggs	o	0.01	0.01		0.01		

Note: The residue definition is sum of anti and syn isomers of Isopyrazam.
* 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 or increased
Request：The MRL was modified in response to MAFF request
IT：Import tolerance

Erythromycin

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
					Codex ppm	National ppm	
Cattle, muscle	○	0.2	0.05	§		0.2	EU
Pig, muscle	○	0.2	0.05	§		0.2	EU
Other terrestrial mammals, muscle	●	0.2	0.3	§		0.2	EU
Cattle, fat	○	0.2	0.05	§		0.2	EU
Pig, fat	○	0.2	0.05	§		0.2	EU
Other terrestrial mammals, fat	○	0.2	0.2	§		0.2	EU
Cattle, liver	○	0.2	0.05	§		0.2	EU
Pig, liver	○	0.2	0.05	§		0.2	EU
Other terrestrial mammals, liver	●	0.2	0.3	§		0.2	EU
Cattle, kidney	○	0.2	0.05	§		0.2	EU
Pig, kidney	○	0.2	0.05	§		0.2	EU
Other terrestrial mammals, kidney	●	0.2	0.3	§		0.2	EU
Cattle, edible offal	○	0.2	0.05	§			
Pig, edible offal	○	0.2	0.05	§			
Other terrestrial mammals, edible offal	●	0.2	0.3	§			
Milk	○	0.04	0.04	§		0.04	Australia
Chicken, muscle	○	0.1	0.05	§	0.1		
Other poultry, muscle	●	0.1	0.2		0.1		
Chicken, fat	○	0.1	0.05	§	0.1		
Other poultry, fat	●	0.1	0.2		0.1		
Chicken, liver	○	0.1	0.05	§	0.1		
Other poultry, liver	●	0.1	0.2		0.1		
Chicken, kidney	○	0.1	0.05	§	0.1		
Other poultry, kidney	●	0.1	0.2		0.1		
Chicken, edible offal	○	0.1	0.05	§			
Other poultry, edible offal	●	0.1	0.2				
Chicken eggs	●	0.05	0.09		0.05		
Other poultry, eggs	●	0.05	0.09				
Salmoniformes (such as salmon and trout)	●		0.2				
Anguilliformes (such as eel)	●		0.2				
Perciformes (such as bonito, horse mackerel, mackerel, sea bass, sea bream and tuna)	○	0.06	0.06	§			
Other fish	●		0.2				
Shelled molluscs	●		0.2				
Crustaceans	●		0.2				
Other aquatic animals	●		0.2				

Note: The residue definition will be changed to Erythromycin A. The current residue definition is Erythromycin.

* This compound shall not be included in any commodity for which MRL is not given in the above table and in any commodity 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

○: Commodities for which MRLs are to be maintained or increased (* It should be noted that the residue definition will be changed.)

§ : Permitted for use in Japan.

Flumethrin

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL		
					Codex ppm	National ppm	
Cattle, muscle	○	0.2	0.01	§	0.2		
Pig, muscle	○		0.005				
Other terrestrial mammals, muscle	●	0.01	0.06			0.01	EU
Cattle, fat	○	0.2	0.2	§	0.2		
Pig, fat	○		0.005				
Other terrestrial mammals, fat	○	0.2	0.2			0.15	EU
Cattle, liver	○	0.05	0.04	§		0.05	Australia
Pig, liver	○		0.005				
Other terrestrial mammals, liver	●	0.02	0.06			0.02	EU
Cattle, kidney	○	0.05	0.03	§		0.05	Australia
Pig, kidney	○		0.005				
Other terrestrial mammals, kidney	●	0.01	0.06			0.01	EU
Cattle, edible offal	○	0.05	0.03	§		0.05	Australia
Pig, edible offal	○		0.005				
Other terrestrial mammals, edible offal	●	0.02	0.1				
Milk	○	0.05	0.05		0.05		
Chicken, muscle	●	0.01	0.03	§ · Request			
Other poultry, muscle	○		0.005				
Chicken, fat	○	0.6	0.03	§ · Request			
Other poultry, fat	○		0.005				
Chicken, liver	●	0.01	0.03	§ · Request			
Other poultry, liver	○		0.005				
Chicken, kidney	●	0.01	0.03	§ · Request			
Other poultry, kidney	○		0.005				
Chicken, edible offal	●	0.01	0.03	§ · Request			
Other poultry, edible offal	○		0.005				
Chicken eggs	○	0.03	0.03				
Other poultry, eggs	●		0.03				
Salmoniformes (such as salmon and trout)	○		0.005				
Anguilliformes (such as eel)	○		0.005				
Perciformes (such as bonito, horse mackerel, mackerel, sea bass, sea bream and tuna)	○		0.005				
Other fish	○		0.005				
Shelled molluscs	○		0.005				
Crustaceans	○		0.005				
Other aquatic animals	○		0.005				
Honey (including royal-jelly)	○	0.005	0.005			0.005	EU

Note: The residue definition is sum of isomers of Flumethrin.

* 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
○: Commodities for which MRLs are to be maintained or increased
§ : Permitted for use in Japan.
Request : The MRL was modified in response to MAFF request

Metoclopramide

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
					Codex ppm	National ppm
Cattle, muscle	○	0.03	0.03	§		
Pig, muscle	○	0.03	0.03	§		
Other terrestrial mammals, muscle	○		0.005			
Cattle, fat	○	0.03	0.03	§		
Pig, fat	○	0.03	0.03	§		
Other terrestrial mammals, fat	○		0.005			
Cattle, liver	○	0.03	0.03	§		
Pig, liver	○	0.03	0.03	§		
Other terrestrial mammals, liver	○		0.005			
Cattle, kidney	○	0.03	0.03	§		
Pig, kidney	○	0.03	0.03	§		
Other terrestrial mammals, kidney	○		0.005			
Cattle, edible offal	○	0.03	0.03	§		
Pig, edible offal	○	0.03	0.03	§		
Other terrestrial mammals, edible offal	○		0.005			
Milk	○	0.005	0.005	§		
Chicken, muscle	○		0.005			
Other poultry, muscle	○		0.005			
Chicken, fat	○		0.005			
Other poultry, fat	○		0.005			
Chicken, liver	○		0.005			
Other poultry, liver	○		0.005			
Chicken, kidney	○		0.005			
Other poultry, kidney	○		0.005			
Chicken, edible offal	○		0.005			
Other poultry, edible offal	○		0.005			
Chicken eggs	○		0.005			
Other poultry, eggs	○		0.005			
Salmoniformes (such as salmon and trout)	○		0.005			
Anguilliformes (such as eel)	○		0.005			
Perciformes (such as bonito, horse mackerel, mackerel, sea bass, sea bream and tuna)	○		0.005			
Other fish	○		0.005			
Shelled molluscs	○		0.005			
Crustaceans	○		0.005			
Other aquatic animals	○		0.005			
Honey (including royal-jelly)	○		0.005			

Note: The residue definition will be changed to Metoclopramide (including its metabolites that are hydrolized to Metoclopramide by hydrochloric acid). The current residue definition is Metoclopramide 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 maintained or increased (* It should be noted that the residue definition will be changed.)

§ : Permitted for use in Japan.

Piperazine

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
					Codex ppm	National ppm
Cattle, muscle	●		0.05			
Pig, muscle	●	0.03	0.3	§		
Other terrestrial mammals, muscle	●	0.01	0.05	§		
Cattle, fat	●		0.05			
Pig, fat	○	0.5	0.5	§		
Other terrestrial mammals, fat	○	0.09	0.05	§		
Cattle, liver	●		0.05			
Pig, liver	●	0.2	1	§		
Other terrestrial mammals, liver	○	2	0.05	§		
Cattle, kidney	●		0.05			
Pig, kidney	●	0.3	0.6	§		
Other terrestrial mammals, kidney	○	2	0.05	§		
Cattle, edible offal	●		0.05			
Pig, edible offal	○	0.5	0.1	§		
Other terrestrial mammals, edible offal	○	3	0.05	§		
Milk	●		0.05			
Chicken, muscle	●	0.03	0.1	§		
Other poultry, muscle	●		0.1			
Chicken, fat	○	0.1	0.1	§		
Other poultry, fat	●		0.1			
Chicken, liver	●	0.08	0.1	§		
Other poultry, liver	●		0.1			
Chicken, kidney	○	0.6	0.1	§		
Other poultry, kidney	●		0.1			
Chicken, edible offal	○	0.6	0.1	§		
Other poultry, edible offal	●		0.1			
Chicken eggs	●		2			
Other poultry, eggs	●		2			
Salmoniformes (such as salmon and trout)	●		0.05			
Anguilliformes (such as eel)	●		0.05			
Perciformes (such as bonito, horse mackerel, mackerel, sea bass, sea bream and tuna)	●		0.05			
Other fish	●		0.05			
Shelled molluscs	●		0.05			
Crustaceans	●		0.05			
Other aquatic animals	●		0.05			
Honey (including royal-jelly)	●		0.05			

Note: The residue definition is Piperazine 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
○: Commodities for which MRLs are to be maintained or increased
§ : Permitted for use in Japan.

Vedaprofen

Commodity		MRL (draft) ppm	MRL (current) ppm	Registration	Reference MRL	
					Codex ppm	National ppm
Other terrestrial mammals, muscle	○	0.05	0.05			0.05 EU
Other terrestrial mammals, fat	○	0.02	0.02			0.02 EU
Other terrestrial mammals, liver	○	0.1	0.1			0.1 EU
Other terrestrial mammals, kidney	○	1	1			1 EU
Other terrestrial mammals, edible offal	○	1	0.1			

Note: The residue definition is Vedaprofen 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 maintained or increased

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, pimiento (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 Ipronidazole, Dimetridazole, Metronidazole and Ronidazole
- Analytical method for Chloramphenicol
- Analytical method for Captafol (animal and fishery products)

Notification(draft)
Analytical Method for Ipronidazole, Dimetridazole, Metronidazole, Ronidazole (Targeted to animal and fishery products)

Target compounds to be determined

Ipronidazole: Ipronidazole, 1-Methyl-2-(2′-hydroxyisopropyl)-5-nitroimidazole (Ipronidazole metabolite B)
Dimetridazole: Dimetridazole, 2-Hydroxymethyl-1-methyl-5-nitroimidazole (HMMNI)
Metronidazole: Metronidazole, 1-(2-Hydroxyethyl)-2-hydroxymethyl-5-nitroimidazole (Metronidazole metabolite A)
Ronidazole: Ronidazole, HMMNI

1. Instrument
   Liquid Chromatograph/Tandem Mass Spectrometer (LC-MS/MS)

2. Reagents and test solutions
   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 compositional substances.

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

Formic Acid: Use a reagent not containing any substance that may interfere with analysis of the target compositional substances.

Strongly Acidic Cation Exchange Resin Cartridge Column (500 mg): A polyethylene column of 12–13 mm in inner diameter packed with 500 mg of strongly acidic cation exchange resin or a column equivalent to the specified one in separation capability.

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

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 compositional substances, wash with an appropriate 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 compositional substances.

Methanol: Use a reagent not containing any substance that may interfere with analysis of the target compositional substances.

3. Reference standard
   Reference standard of Ipronidazole: Contains not less than 98% of Ipronidazole.
   Reference standard of Ipronidazole Metabolite B: Contains not less than 98% of

Ipronidazole Metabolite B.

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

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

Reference standard of Metronidazole Metabolite A: Contains not less than 98% of Metronidazole Metabolite A.

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

4. Procedure

a. Extraction

Homogenize 10.0 g of the test sample (for honey, 10.0 g of the test sample dissolved in 10 mL of water) with 1 mL of acetic acid and 50 mL of acetone. Centrifuge the mixture at 3,000 rpm for 5 minutes and collect the supernatant. Homogenize the residue (for honey, the residue dissolved in 5 mL of water) again with 30 mL of acetone, centrifuge under the same conditions, and collect the supernatant. Combine the supernatants and add acetone to make exactly 100 mL.

Transfer exactly 10 mL of the resulting solution and evaporate to about 2 mL at a temperature not exceeding 40°C. Shake the concentrate with 10 mL of acetonitrile saturated by n-hexane and 10 mL of n-hexane and centrifuge at 3,000 rpm for 5 minutes. Evaporate the acetonitrile layer at a temperature not exceeding 40°C to remove the solvent. Dissolve the residue by adding 5 mL of 2% (vol) formic acid.

b. Clean-up

Pour 5 mL of acetonitrile and 5 mL of 2% (vol) formic acid into a strongly acidic cation exchange resin cartridge column (500 mg) and discard the effluent. Pour the solution obtained in section 4-a, then pour 5 mL of 2% (vol) formic acid, 5 mL of methanol, and 5 mL of 0.5% ammonia solution sequentially, and discard the effluent. Pour 10 mL of a 1:3 mixture of acetonitrile and water and collect the eluate.

Dissolve 2 g of ammonium sulfate in the resulting eluate and extract twice by shaking with 10 mL of ethyl acetate each time. Combine the ethyl acetate extracts, add anhydrous sodium sulfate to dehydrate, and remove the anhydrous sodium sulfate by filtration. Evaporate the filtrate at a temperature not exceeding 40°C to remove the solvent. Dissolve the residue by adding 0.1% (vol) formic acid to make exactly 1 mL. Use this solution as the test solution.

5. Measurement

a. Calibration curve

Prepare several solutions of each Reference Standard in 0.1% (vol) formic acid with different concentrations. Inject them into LC-MS/MS to prepare a calibration curve for each Reference Standard using the peak-height or peak-area method. When the test solution is prepared as directed in this method, the concentration of each target compound in the test solution that is equivalent to 0.0001 mg/kg (0.0002 mg/kg) in the test sample is 0.0001 mg/L (0.0002 mg/L).

b. Quantification

Inject the test solution in LC-MS/MS and determine the content of each compound from the corresponding calibration curve prepared in section 5-a.

c. Confirmation tests

Conduct confirmation tests using LC-MS/MS.

d. Measurement conditions

Column: Octadecylsilanized silica gel (3 mm in inner diameter, 150 mm in length, 3 μm in

particle size)
Column temperature: 40°C
Mobile phase: Run 0.1% (vol) formic acid (solution A) and 0.1% formic acid solution in acetonitrile (solution B) at the following concentration gradients:

Time (minute)	Solution A (%)	Solution B (%)
0	98	2
5	90	10
15	5	95
20	5	95

Ionization mode: Electrospray ionization (positive ion mode)
Main ions (m/z)
Ipronidazole: Precursor ion 170; Product ions 124, 109
Ipronidazole Metabolite B: Precursor ion 186; Product ions 168, 122
Dimetridazole: Precursor ion 142; Product ions 96, 95
Metronidazole: Precursor ion 172; Product ions 128, 82
Metronidazole Metabolite A: Precursor ion 188; Product ions 126, 123
Ronidazole: Precursor ion 201; Product ions 140, 55
HMMNI: Precursor ion 158; Product ions 94, 55
Injection volume: 5 µL
Retention time
Ipronidazole: about 15 minutes
Ipronidazole Metabolite B: about 13 minutes
Dimetridazole: about 12 minutes
Metronidazole: about 11 minutes
Metronidazole Metabolite A: about 11 minutes
Ronidazole: about 12 minutes
HMMNI: about 11 minutes

6. Limit of Quantification
Ipronidazole: 0.0001mg/kg
Ipronidazole Metabolite B: 0.0001mg/kg
Dimetridazole: 0.0002mg/kg
Metronidazole: 0.0001mg/kg
Metronidazole Metabolite A: 0.0001mg/kg
Ronidazole: 0.0002mg/kg
HMMNI: 0.0002mg/kg

Analytical Method for Chloramphenicol
(Targeted to animal and fishery products)

The target compound to be determined is Chloramphenicol and Chloramphenicol Glucuronic Acid Conjugate.

1. Instrument
   Liquid Chromatograph/Tandem Mass Spectrometer (LC-MS/MS)

2. Reagents and test solutions
   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 compositional substances.

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

Divinylbenzene-N-vinylpyrrolidone Co-polymer Cartridge Column (500 mg): A polyethylene column of 12–13 mm in inner diameter packed with 500 mg of divinylbenzene-N-vinylpyrrolidone co-polymer or a column equivalent to the specified one in separation capability.

Methanol: Use a reagent not containing any substance that may interfere with analysis of the target compositional substances.

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 compositional substances, wash with an appropriate solvent, such as n-hexane, before use.

β-Glucuronidase (type IX-A): Use β-glucuronidase type IX-A derived from Escherichia coli. One unit of this substance is the amount of enzyme that produces 1.0 μg of phenolphthalein at pH 6.8 at 37°C in one hour when phenolphthalein β-D-glucuronido is used as the substrate.

β-Glucuronidase Solution: Dissolve an appropriate amount of β-glucuronidase (type IX-A) in 0.1 mol/L phosphate buffer (pH 6.8) to make a solution of 1500 units/mL. Prepare before use.

0.1 mol/L Phosphate Buffer (pH 6.8): Dissolve 1.36 g of potassium dihydrogen phosphate in water to make 100 mL (Solution 1). Dissolve 1.42 g of disodium hydrogenphosphate in water to make 100 mL (Solution 2). Mix the two solutions and adjust the pH to 6.8.

3. Reference standard
   Reference standard of Chloramphenicol : Contains not less than 98% of Chloramphenicol

4. Procedure

a. Extraction

In the case of muscle, fat, liver, kidney, and fish/shellfish, weigh 10.0 g of the test sample, previously chopped and homogenized. In the case of milk, egg, honey, and royal jelly, weigh 10.0g of the test sample, previously mixed well uniformly. In the case of dried royal jelly, weigh 5.00 g of the test sample, previously mixed well uniformly, add 10 mL of water, and allow to stand for 30 minutes.

To the weighed sample, add 50 mL of methanol, homogenize the mixture, and centrifuge at 3,000 rpm for 5 minutes to collect the supernatant. To the residue, add 30 mL of methanol, homogenize the mixture, and centrifuge under the same conditions to collect the supernatant. Combine the two supernatants to make exactly 100 mL with methanol. Transfer exactly 4 mL and remove the solvent at a temperature not exceeding 40ºC. To the residue, add 9 mL of 0.1 mol/L phosphate buffer (pH 6.8) and mix well using an ultrasonic treatment.

b. Hydrolysis

To the solution prepared in section 4-a, add 1 mL of β-glucuronidase solution and warm for 60 minutes at 37ºC to hydrolyze. Add 10 mL of ethyl acetate and extract by shaking. Centrifuge the mixture at 3000 rpm for 5 minute and collect the ethyl acetate layer. To the water layer, add 10 mL of ethyl acetate, extract by shaking, and centrifuge under the same conditions to collect the ethyl acetate layer. Combine the ethyl acetate layers, evaporate at a temperature not exceeding 40ºC to remove the solvent. To the residue, add 5 mL of a 1:1 mixture of water and methanol and mix well by an ultrasonic treatment.

c. Clean-up

Pour 5 mL of methanol and 5 mL of a 1:1 mixture of water and methanol sequentially into a divinylbenzene-N-vinylpyrrolidone co-polymer cartridge column (500 mg) and discard the effluent. Charge the column with 5 mL of the solution obtained in section 4-b, pour 5 mL of a 1:1 mixture of water and methanol, and discard the effluent. Then pour 10 mL of a 1:4 mixture of water and methanol, and evaporate the eluate at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue in a 3:7 mixture of acetonitrile and water to make exactly 2 mL. For dried royal jelly, make exactly 1 mL.

5. Measurement

a. Calibration curve

Prepare several solutions of Chloramphenicol Reference Standard in a 3:7 mixture of acetonitrile and water with different concentrations. Inject them into LC-MS/MS to prepare a calibration curve using the peak-height or peak-area method. When the test solution is prepared as directed in this method, the concentration of chloramphenicol in the test solution that is equivalent to 0.0005 mg/kg in the test sample is 0.0001 mg/L.

b. Quantification

Inject the test solution in LC-MS/MS and determine the content of chloramphenicol from the calibration curve prepared in section 5-a.

c. Confirmation tests

Conduct confirmation tests using LC-MS/MS.

d. Measurement conditions

Column: Octadecylsilanized silica gel (2.1 mm in inner diameter, 150 mm in length, 3 µm in particle size)

Column temperature: 40°C
Mobile phase: A 3:7 mixture of acetonitrile and 10 mmol/L ammonium acetate
Ionization mode: ESI (–)
Main ions (m/z): Precursor ion 321; product ions 152
Precursor ion 323; product ions 152
Injection volume: 5 µL
Retention time: About 4 minutes

6. Limit of Quantification
0.0005mg/kg (royal jelly:0.005mg/kg)

The target compound to be determined is captafol.

1. Instrument
   Gas chromatograph equipped with electron capture detector (GC-ECD)

2. Reagents and test solutions
   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 compositional substances.

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

Ether: Use a reagent not containing any substance that may interfere with analysis of the target compositional substances.

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

Graphite Carbon Cartridge Column (250 mg): A polyethylene column of 8–9 mm in inner diameter packed with 250 mg of graphite carbon or a column equivalent to the specified one in separation capability.

Synthetic Magnesium Silicate Cartridge Column (910 mg): A polyethylene column of 8–9 mm in inner diameter packed with 910 mg of synthetic magnesium silicate or a column equivalent to the specified one in separation capability.

Ethyl Acetate: Use a reagent not containing any substance that may interfere with analysis of the target compositional substances.

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

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 compositional substances, wash with an appropriate 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 compositional substances.

3. Reference standard
Reference standard of captafol : Contains not less than 97% of captafol.

4. Preparation of test solutions
   a. Extraction
(i) Muscle, fat, liver, kidney, and fish/shellfish

For fat, weigh 5.00 g of the test sample, previously chopped and homogenized. For other products, weigh 10.0 g of the test sample, previously chopped and homogenized. For products that are very small in size, such as shijimi shellfish (bivalve), weigh the test sample accurately, grind and homogenize it with 10% (vol) phosphoric acid solution (a half amount of the sample by weight), then weigh an amount equivalent to 10.0 g of the ground sample.

Grind the weighed sample with 20 mL of 3% (vol) phosphoric acid solution (10 mL of water for shijimi shelfish) and 100 mL of acetone, filter it by suction, and collect the filtrate. Grind the residue on the filter paper with 50 mL of acetone and filter in the same manner. Combine the filtrates and evaporate to about 20 mL at a temperature not exceeding 40ºC. Add 100 mL of 10% (w/v) sodium chloride solution and extract twice by shaking with 100 mL of n-hexane and then with 50 mL of n-hexane. Combine the n-hexane extracts, dehydrate with anhydrous sodium sulfate, and remove the anhydrous sodium sulfate by filtration. Evaporate the filtrate at a temperature not exceeding 40ºC to remove the solvent. To the residue, add 30 mL of n-hexane and extract twice by shaking with 30 mL of acetonitrile saturated by n-hexane each time.

Combine the acetonitrile extracts, and evaporate at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue in a 1:1 mixture of acetone and n-hexane and make exactly 20 mL. Transfer exactly 2 mL of the resulting solution (exactly 4 mL for fat samples) and evaporate it at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue by adding 5 mL of a 1:4 mixture of ether and n-hexane.

(ii) Milk, egg, and honey

Weigh 10.0 g of the test sample, previously homogenized, grind it with 20 mL of 3% (vol) phosphoric acid solution and 100 mL of acetone, centrifuge at 3,000 rpm for 5 minutes, and collect the supernatant. Grind again the residue with 50 mL of acetone (for honey, 20 mL of water and 50 mL of acetone) and centrifuge under the same conditions. Combine the supernatants and evaporate to about 20 mL (to about 50 mL for honey) at a temperature not exceeding 40ºC. To the concentrate, add 100 mL of 10% sodium chloride solution and extract twice by shaking with 100 mL of n-hexane and then with 50 mL of n-hexane. Combine the n-hexane extracts, dehydrate with anhydrous sodium sulfate, remove the anhydrous sodium sulfate by filtration, and evaporate the filtrate at a temperature not exceeding 40ºC to remove the solvent. To the residue, add 30 mL of n-hexane and extract twice by shaking with 30 mL of acetonitrile saturated by n-hexane each time. Combine the acetonitrile extracts and evaporate at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue by adding a 1:1 mixture of acetone and n-hexane and make exactly 20 mL. Transfer exactly 2 mL of the resulting solution and evaporate it at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue by adding 5 mL of a 1:4 mixture of ether and n-hexane.

b. Clean-up

(i) Synthetic magnesium silicate column chromatography

Pour 5 mL of n-hexane into a synthetic magnesium silicate column (910 mg) and discard the effluent. Pour the solution obtained in section 4-a, pour 5 mL of a 1:4 mixture of ether and n-hexane and discard the effluent. Then pour 30 mL of a 1:9 mixture of ethyl acetate and n-hexane and evaporate the eluate at a temperature not exceeding 40ºC to remove the solvent. Dissolve the residue adding 5 mL of acetonitrile.

(ii) Graphite carbon column chromatography

Pour 5 mL of acetonitrile into a graphite carbon column (250 mg) and discard the effluent. Pour the acetonitrile solution obtained in section b-i, then pour 15 mL of acetonitrile, and evaporate the eluate at

a temperature not exceeding 40°C to remove the solvent. Dissolve the residue in n-hexane and make exactly 2 mL. Use this solution as the test solution.

5. Measurement

a. Calibration curve

Prepare captafol solutions (500 mg/L) by dissolving Captafol Reference Standard in acetone. Dilute 1 mL of the standard stock solution with acetone to exactly 25 mL (20 mg/L). Prepare several standard solutions with different concentrations by diluting the resulting solution with n-hexane. Inject them into a GC-ECD to prepare a calibration curve using the peak-height or peak-area method. When the test solution is prepared as directed in this method, the concentrate of captafol in the test solution that is equivalent to 0.01 ppm in the test sample is 0.005 mg/L.

b. Quantification

Inject the test solution in the GC-ECD and determine the content of captafol from the calibration curve prepared in section 5-a.

c. Confirmation tests

Conduct confirmation tests using the GC-ECD.

d. Measurement conditions

Column: 5% phenyl-methyl silicone (0.25 mm in inner diameter, 30 m in length, coated with a 0.25 µm thick layer)

Column temperature: Hold at 50°C for 1 minute, raise to 125°C at 15°C/min, then raise to 300°C at 10°C/min , and hold at 300°C for 5 minutes.

Injection port temperature: 230°C

Detector temperature: 300°C

Carrier gas: Helium

Injection volume: 1 µL

Retention time: About 18 minutes

6. Limit of Quantification

0.01mg/kg