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Japan Proposes New MRLs and a Food Additive (pyrimethanil)

Report Categories:
Sanitary/Phytosanitary/Food Safety

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Report Highlights:
On October 16, 2012, the Government of Japan (GOJ) announced proposed changes to the Maximum Residue Levels (MRLs) for three substances; Alachlor, pyrimethanil and spirodiclofen. GOJ also proposed the approval of pyrimethanil as a food additive. The domestic comment period for these changes will close on Tuesday, October 30, 2012. After the domestic comment period closes, the Ministry of Health, Labor, and Welfare (MHLW) will then notify the proposed MRL changes to the WTO/SPS committee, which will then provide another opportunity for interested parties to comments on this subject.

General Information:
On October 16, 2012, GOJ announced proposed changes to MRLs for three substances; Alachlor, pyrimethanil and spirodiclofen. GOJ also proposed the approval of pyrimethanil as post harvest fungicide which is classified into a food additive in Japanese regulation. The comment period for these changes will close on October 30, 2012.

Any parties interested in submitting comments to MHLW should do so as soon as possible. If you are requesting that Japan adopt the same limits as the U.S. maximum residue limits, the request should be accompanied by data supporting U.S. MRLs, such as risk assessment and residue data. The information MHLW requires would include toxicity data for target chemicals, residue trial data that support the MRLs, and target food commodities. Please be advised that Limit of Quantitation (LOQ) and Limit of Dectetion (LOD) data cannot be used as reference data to set up Japanese MRLs. For Japan's MRLs and the details of the information MHLW needs, please visit the following MRLs websites:

Pesticides; http://www.mhlw.go.jp/english/topics/foodsafety/residue/dl/01.pdf
Feed additives; http://www.mhlw.go.jp/english/topics/foodsafety/residue/dl/02.pdf
Veterinary drugs; http://www.mhlw.go.jp/english/topics/foodsafety/residue/dl/03.pdf

MHLW will also notify these proposed changes to the WTO/SPS committee, which will provide an additional chance for interested parties to submit comments on this subject. The actual WTO/SPS notifications can be found at the site below.

http://www.wto.org/english/tratop_e/sps_e/work_and_doc_e.htm

After the WTO comment period closes, a final report will be released based on the conclusions reached by a session of the Pharmaceutical Affairs and Food Sanitation Council scheduled to be held at a later date. This report will constitute as the final decision.

Comments to the GOJ can be made either in Japanese or English and can be sent to the below point of contact:

Pesticide
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Post suggests that the U.S. Embassy - Tokyo also be copied on any comments at agtokyo@usda.gov to allow them to be considered as part of the official U.S. Government comments to the WTO.

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

MHLW is going to develop compositional specifications for food.

Summary

Under the provisions of Article 11, Paragraph 1 of the Food Sanitation Law, MHLW is authorized to establish residue standards (maximum residue limits: MRLs) for pesticides, feed additives, and veterinary drugs (hereafter referred to as just "agricultural chemicals") that may remain in foods. Any food for which standards are established pursuant to the provisions is not permitted to be marketed in Japan unless it complies with the established standards.

On May 29, 2006, the MHLW introduced the positive list system for agricultural chemicals in food.* Basically, all foods distributed in the Japanese marketplace are subject to regulation based on the system.

This time, MHLW has newly established MRLs (draft) for some food commodities as well as has comprehensively reviewed the current MRLs. This activity is targeted to three pesticides (Alachlor, Pyrimethanil, and Spirodiclofen). Details are given below.

Note: The positive list system was established based on the 2003 amendment of the Food Sanitation Law. The system aims to prohibit the distribution of any food in the Japanese marketplace if it contains agricultural chemicals at amounts exceeding a certain level (0.01 ppm) specified under the Law.

Outline of revision

Alachlor (herbicide): Permitted for use in Japan.
MHLW has established a new MRL for fish and shellfish. This action was to respond to a request from the Ministry of Agriculture, Forestry and Fisheries (MAFF). In addition, MHLW has reviewed the MRLs that had been provisionally established at the introduction of the positive list system.

Pyrimethanil (fungicide): Not permitted for use in Japan.
MHLW has newly established MRLs for some crops, in response to an application for 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). Additionally, MHLW has reviewed the MRLs that had been provisionally established at the introduction of the positive list system.

Spirodiclofen (acaricide): Permitted for use in Japan.

MHLW has newly established MRLs for some crops. This action was in response to the MAFF decision to expand the scope of crops to which this compound is applicable and to an application for import tolerances based on the guideline (Shokuan No. 0205001, 5 February 2004). Additionally, MHLW has reviewed the MRLs that had been provisionally established at the introduction of the positive list system.

Alachlor

Commodity	MRL (draft) ppm	MRL (current) ppm
Barley		0.05
Rye		0.05
Corn (maize, including pop corn and sweet corn)	0.02	0.2
Buckwheat		0.05
Other cereal grains	0.05	0.1
Soybeans, dry	0.02	0.2
Beans, dry (including butter beans, cowbeans (red beans), lentil, lima beans, pegia, sultani, sultapya, and white beans)	0.02	0.1
Peas		0.05
Broad beans	0.1	0.1
Peanuts, dry	0.02	0.05
Other legumes/pulses	0.1	0.1
Potato	0.01	0.01
Taro		0.01
Sweet potato	0.02	0.01
Yam		0.01
Konjac		0.01
Other potatoes		0.01
Sugar beet	0.01	0.01
Sugarcane	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
Horseradish		0.01
Watercress		0.01
Chinese cabbage	0.01	0.01
Cabbage	0.01	0.01
Brussels sprouts	0.01	0.01
Kale		0.01
Komatsuna(Japanese mustard spinach)	0.01	0.01
Kyona		0.01
Qing-geng-cai		0.01
Cauliflower		0.01

Broccoli		0.01
Other cruciferous vegetables	0.01	0.01
Burdock		0.01
Salsify		0.01
Artichoke		0.01
Chicory		0.01
Endive		0.01
Shungiku		0.01
Lettuce (including cos lettuce and leaf lettuce)		0.01
Other composite vegetables		0.01
Welsh (including leek)		0.01
Nira		0.01
Asparagus		0.01
Multiplying onion (including shallot)		0.01
Other liliaceous vegetables		0.01
Carrot		0.01
Parsnip		0.01
Parsley		0.01
Celery		0.01
Mitsuba		0.01
Other umbelliferous vegetables		0.01
Spinach	0.01	0.01
Bamboo shoots		0.01
Ginger		0.01
Other vegetables		0.01
Apple		0.01
Japanese pear	0.01	0.01
Pear	0.01	0.01
Quince		0.01
Nectarine		0.01
Apricot		0.01
Japanese plum (including prune)		0.01
Mume plum		0.01
Cherry		0.01
Strawberry	0.01	0.01
Raspberry		0.01
Blackberry		0.01
Blueberry		0.01
Cranberry		0.01
Huckleberry		0.01
Other berries		0.01
Grape	0.01	0.01
Japanese persimmon		0.01
Banana		0.01

Papaya		0.01
Avocado		0.01
Pineapple		0.01
Guava		0.01
Mango		0.01
Passion fruit		0.01
Date		0.01
Other fruits		0.01
Other spices		0.1
Other herbs		0.01
Cattle, muscle	0.02	0.01
Pig, muscle	0.02	0.01
Other terrestrial mammals, muscle	0.02	0.01
Cattle, fat	0.02	0.02
Pig, fat	0.02	0.02
Other terrestrial mammals, fat	0.02	0.02
Cattle, liver	0.02	0.02
Pig, liver	0.02	0.02
Other terrestrial mammals, liver	0.02	0.02
Cattle, kidney	0.02	0.02
Pig, kidney	0.02	0.02
Other terrestrial mammals, kidney	0.02	0.02
Cattle, edible offal (“Edible offal” refers to all edible parts, except muscle, fat, liver, and kidney)	0.02	0.02
Pig, edible offal	0.02	0.02
Other terrestrial mammals, edible offal	0.02	0.02
Milk	0.02	0.01
Chicken, muscle	0.02	0.01
Other poultry animals, muscle	0.02	0.01
Chicken, fat	0.02	0.02
Other poultry animals, fat	0.02	0.02
Chicken, liver	0.02	0.02
Other poultry animals, liver	0.02	0.02
Chicken, kidney	0.02	0.02
Other poultry animals, kidney	0.02	0.02
Chicken, edible offal	0.02	0.02
Other poultry animals, edible offal	0.02	0.02
Chicken, eggs	0.02	0.02
Other poultry, eggs	0.02	0.02
Fish and shellfish	0.06	
Mineral waters (natural mineral waters and bottled/packaged drinking waters)	0.02	0.02

Note: The MRLs are expressed as the sum of alachlor and its metabolites that can be converted to 2,6-diethylaniline (DEA) or 2-ethyl-6-(1-hydroxyethyl)aniline (1-HEEA) upon basic hydrolysis,

calculated as alachlor in the corresponding livestock and poultry products. The MRLs are expressed as alacholore only for other commodities.

* Shaded figures indicate provisional MRLs.
* 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.

Pyrimethanil

Commodity	MRL (draft) ppm	MRL (current) ppm
Beans, dry (including butter beans, cowbeans (red beans), lentil, lima beans, pegia, sultani, sultapya, and white beans)	1	1
Peas	0.5	0.3
Potato	0.05	0.03
Taro	0.05	0.05
Sweet potato	0.05	0.05
Yam	0.05	0.05
Other potatoes	0.05	0.05
Lettuce (including cos lettuce and leaf lettuce)	3	2
Onion	0.2	0.1
Welsh (including leek)	3	2
Carrot	1	
Tomato	2	2
Egg plant	1	1
Other solanaceous vegetables	2	2
Cucumber (including gherkin)	2	2
Other cucurbitaceous vegetables		0.05
Ginger	0.05	0.05
Peas, immature (with pods)	0.3	0.3
Kidney beans, immature (with pods)	3	1
Other vegetables	0.3	0.05
Unshu orange, pulp	0.5	0.5
Citrus *natsudaidai*, whole	10	10
Lemon	10	15
Orange (including navel orange)	10	15
Grapefruit	10	15
Lime	10	15
Other citrus fruits	10	15
Apple	14	5
Japanese pear	1	1

Pear	14	1
Quince	14	0.05
Loquat	0.05	0.05
Peach		3
Nectarine	4	5
Apricot	3	10
Japanese plum (including prune)	2	10
Mume plum		10
Cherry		10
Strawberry	10	10
Raspberry	10	10
Blackberry	10	10
Blueberry	5	10
Cranberry	5	10
Huckleberry	5	10
Other berries	5	10
Grape	10	10
Japanese persimmon		5
Banana	0.1	0.1
Papaya		5
Avocado		5
Pineapple		5
Guava		5
Mango		5
Passion fruit		5
Date		10
Other fruits		10
Almond	0.2	0.2
Other nuts	0.2	0.2
Other spices (roots/rhizomes)	0.05	15
Other herbs		0.05
Cattle, muscle	0.05	0.03
Pig, muscle	0.05	0.05
Other terrestrial mammals, muscle	0.05	0.03
Cattle, fat	0.05	0.01
Pig, fat	0.05	0.05
Other terrestrial mammals, fat	0.05	0.01
Cattle, liver	0.1	0.03
Pig, liver	0.1	0.05
Other terrestrial mammals, liver	0.1	0.03
Cattle, kidney	0.1	0.04
Pig, kidney	0.1	0.05
Other terrestrial mammals, kidney	0.1	0.04
Cattle, edible offal (“Edible offal” refers to all edible parts, except muscle, fat, liver, and kidney)	0.1	0.03

Pig, edible offal	0.1	0.05
Other terrestrial mammals, edible offal	0.1	0.03
Milk	0.01	0.02

Note: MRLs are expressed as pyrimethanil for crops, as the sum of pyrimethanil and its metabolite, 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, calculated as pyrimethanil, for muscle, fat, liver, kidney, and edible offal of livestock and poultry, and as the sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, calculated as pyrimethanil, for milk.

* Shaded figures indicate provisional MRLs.
* 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.
* ”Other spices (roots/rhizomes)” refers to roots and rhizomes of asafetida, turmeric, zedoary, Galangal and licorice.

Spirodiclofen

Commodity	MRL (draft) ppm	MRL (current) ppm
Tomato	0.5	
Pimiento (sweet pepper)	0.2	
Egg plant	2	
Other solanaceous vegetables	5	
Cucumber (including gherkin)	0.1	
Unshu orange, pulp	0.05	0.1
Citrus *natsudaidai*, whole	2	2
Lemon	2	2
Orange (including navel orange)	2	2
Grapefruit	2	2
Lime	2	2
Other citrus fruits	2	2
Apple	2	2
Japanese pear	0.8	2
Pear	0.8	2
Quince	0.8	2
Loquat	0.5	0.8
Peach		1
Nectarine	2	2
Apricot	2	5
Japanese plum (including prune)	2	5
Mume plum	2	5
Cherry	3	5
Strawberry	2	5

Raspberry		5
Blackberry		5
Blueberry		5
Cranberry		5
Huckleberry		5
Other berries	1	5
Grape	2	5
Japanese persimmon		2
Banana		2
Papaya	1	2
Avocado	1	2
Pineapple		2
Guava		2
Mango	1	2
Passion fruit		2
Other fruits	1	5
Ginkgo nut	0.05	
Chestnut	0.1	0.1
Pecan	0.1	0.1
Almond	0.1	0.1
Walnut	0.1	0.1
Other nuts	0.1	0.1
Tea	20	
Coffee beans	0.03	
Hop	40	
Other spices	5	5
Cattle, muscle	0.02	0.02
Pig, muscle	0.02	
Other terrestrial mammals, muscle	0.02	0.02
Cattle, fat	0.02	0.02
Pig, fat	0.02	
Other terrestrial mammals, fat	0.02	0.02
Cattle, liver	0.1	0.1
Pig, liver	0.1	
Other terrestrial mammals, liver	0.1	0.1
Cattle, kidney	0.1	0.1
Pig, kidney	0.1	
Other terrestrial mammals, kidney	0.1	0.1
Cattle, edible offal (“Edible offal” refers to all edible parts, except muscle, fat, liver, and kidney)	0.1	0.1
Pig, edible offal	0.1	
Other terrestrial mammals, edible offal	0.1	0.1
Milk	0.01	0.01

Note: MRLs are expressed as spirodiclofen for crops and as the sum of spirodiclofen and its

metabolite,3-(2,4-dichlorophenyl)-4-hydroxy-1-oxaspiro[4.5]dec-3-en-2-one, calculated as spirodiclofen, for livestock and poultry products.

* Shaded figures indicate provisional MRLs.
* 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.

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

"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.

“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. Designation of a Food Additive

Japan is going to designate Pyrimethanil as an authorized additive.

Under Article 10 of the Food Sanitation Law, food additives shall not be used or marketed without authorization by the Minister of Health, Labour and Welfare. When compositional specifications or standards for use or manufacturing are established for food additives based on Article 11 of the law, those additives shall not be used or marketed unless they meet the standards or specifications.

In response to a request from the Minister, the Committee on Food Additives of the Food Sanitation Council that is established under the Pharmaceutical Affairs and Food Sanitation Council has discussed the adequacy of the designation of Pyrimethanil (CAS No. 53112-28-0) as a food additive. The conclusion of the committee is outlined below.

Pyrimethanil is applied overseas to fruits and vegetables as a fungicide in the pre-harvest period or as antimolding agent in the post-harvest period.

Outline of conclusion

The Minister should, based on Article 10 of the Food Sanitation Law, designate Pyrimethanil as a food additive unlikely to harm human health, and establish compositional specifications and use standards for these substances based on Article 11 of the law. See Attachment 2-1.

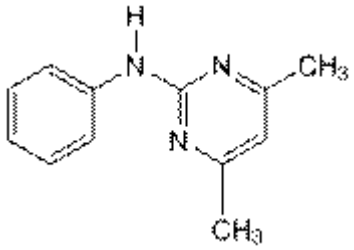
<Additional Information>

Progress in the designation procedure of food additives (54 flavorings and 46 other additives) that have

been proven safe by JECFA (Joint FAO/WHO Expert Committee on Food Additives) and that are widely used in countries other than Japan (Attachment 2-2). As of the end of August, 2012, 9 of the 54 flavorings were not approved yet, and 15 of the 46 non-flavoring additives were unapproved.

Attachment 2-1

Pyrimethanil
ピリメタニル



Standard for use

Pyrimethanil can be used on citrus fruits (excluding *unshū* oranges), apple, pear, and quince. It shall not remain more than 0.010 g/kg in citrus fruits and more that 0.014 g/kg in apple, pear, and quince.

Compositional specifications

Substance name Pyrimethanil

Molecular formula $C_{12}H_{13}N_3$

Mol. Weight 199.25

hemical name [CAS number]

N-(4,6-dimethylpyrimidin-2-yl)aniline［53112-28-0］

Content Pyrimethanil contains 96.0–101.0% of pyrimethanil ($C_{12}H_{13}N_3$).

Description Pyrimethanil occurs as a white to yellowish white, odorless powder.

Identification

Determine the infrared absorption spectrum of Pyrimethanil as directed in the Potassium Bromide Disk Method under Infrared Spectrophotometry, and compare it with the Reference Spectrum. Both spectra exhibit absorptions having about the same intensity at the same wavenumbers.

Purity

(1) Melting point 96–98ºC.

(2) Lead Not more than 2.0 μg/g as Pb (5.0 g, Method 1).

Water Content Not more than 1.0% (2.0 g, Direct Titration).

Assay Weigh accurately about 0.05 g each of Pyrimethanil and pyrimethanil for assay. Dissolve each in methanol to make exactly 50 ml. Take 1 ml each, add a 75:25 mixture of acetonitrile/water to make exactly 20 ml each. Use them as test solution and standard solution respectively. Aanalyze 10 μl portions of the test solution and the standard solution by liquid chromatography using the operating conditions given below. Measure the peak areas (A_T and A_S) of pyrimethanil for the test solution and the standard solution. Calculate the pyrimethanil content by the formula:

Content (%) of pyrimethanil ($C_{12}H_{13}N_3$) =

$$\frac{\text{ight (g) of pyrimethanil for assay}}{\text{Weight (g) of the sample}} \times \frac{A_T}{A_S} \times 100$$

ating conditions

tor: Ultraviolet spectrophotometer (measurement wavelength: 268 nm).
nn: A stainless steel tube of 4.6 mm internal diameter and 25 cm length.
lumn packing material: 5-μm octadecyl silanized silica gel for liquidchromatography.
nn temperature: A constant temperature of 24–40°C.
e phase: To 750 ml of acetonitrile, add 250 ml of water and 2 g of ammonium acetate, and dissolve.
rate: Adjust so that the retention time of pyrimethanil is about 5–6 minutes.

Regents and Solutions

Deuterated methanol CD_3OD Use deuterated methanol produced exclusively for NMR spectral measurement.

Pyrimethanil for Assay $C_{12}H_{13}N_3$ A white crystalline powder.

Content Not less than 99.0% of pyrimetahnil ($C_{12}H_{13}N_3$).

Identification Determine the infrared absorption spectrum of Pyrimethanil as directed in the Potassium Bromide Disk Method under Infrared Spectrophotometry. It exhibits absorption bands at about 3263 cm^{-1}, 1588 cm^{-1}, 1496 cm^{-1}, 1251 cm^{-1}, 757 cm^{-1}, 715 cm^{-1}.

Melting point 96–98°C.

Assay Weigh accurately about 20 mg of Pyrimethanil for Assay and about 4 mg of 1,4-BTMSB-d_4, and add 2 ml of deuterated methanol to dissolve them together. Transfer the resulting solution to an NMR tube of 5 mm in external diameter, stopper tightly, and measure ^{1}HNMR spectra using a spectrometer at a proton resonance frequency of 400 MHz or more. Assuming the signal of 1,4-BTMSB-d_4 as δ 0.23 ppm, when the signal intensities around δ 0.23 ppm, δ 2.32 ppm, and δ 6.56, δ6.80–7.40, and δ 7.66 ppm are designated as A_1 (corresponding to 6 hydrogens), A_2 (corresponding to 1 hydrogen), A_3 (corresponding to 3 hydrogens), and A_4 (corresponding to 2 hydrogens), respectively, confirm that each of (A_1/6)/A_2, (A_1/6)/(A_3/3), (A_1/6)/(A_4/2), A_2/(A_3/3), A_2/(A_4/3), and (A_3/3)/(A_4/2) is 1.0. Then, assuming the signal intensity of 1,4-BTMSB-d_4 as 18.00, when the sum of A_1, A_2, A_3, and A_4 is designated as 1, when the sum of the number of hydrogens and the purity of 1,4-BTMSB-d_4 are designated as N and P(%), respectively, determine the content of pyrimethanil by the following formula. If the signal from Pyrimethanil for Assay is overlapped with the signal from a contaminant, do not use its signal area intensity for the assay.

Content (%) of pyrimethanil ($C_{12}H_{13}N_3$) =

$$\frac{\text{Weight (mg) of 1,4-BTMSB-}d_4 \times I \times P}{\text{Weight (mg) of the sample} \times N} \times 0.8797$$

Operating conditions

Spinning: Off.
^{13}C decoupling: Present.
Acquisition time: 4 seconds.
Spectral range: At least 20 ppm including between −5 ppm and 15 ppm.
Flip angle: 90°.

Delay time: Not less than 60 seconds.

Dummy scans: Not less than 1.

Number of accumulation: Not less than 8.