Amendment to the Enforcement Ordinance of the Food Sanitation Law and the Standards and Specifications for Foods and Food Additives

The government of Japan will designate Propiconazole as an authorized food additive.

Summary

Under Article 10 of the Food Sanitation Law (hereinafter referred to as the "Law"), food additives shall not be used or marketed without authorization by the Minister of Health, Labour and Welfare (hereinafter referred to as "the Minister"). In addition, when specifications or standards are established for food additives based on Article 11 of the Law and stipulated in the Ministry of Health, Labour and Welfare Notification (Ministry of Health and Welfare Notification No. 370, 1959), 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 Propiconazole as a food additive. The conclusion of the committee is outlined below.

Outline of conclusion

The Minister, based on Article 10 of the Law, should designate Propiconazole, as a food additive unlikely to harm human health, and establish standards for use and compositional specifications, based on Article 11 of the Law (see Attachment).

Attachment

Propiconazole
プロピコナゾール

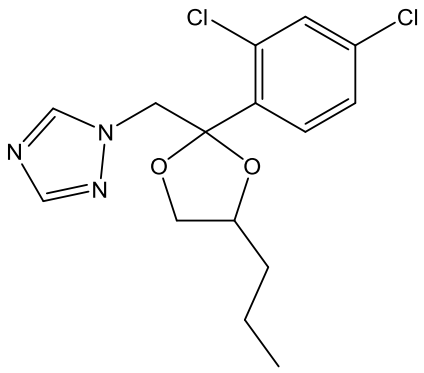
Standards for use (draft)

Propiconazole is permitted for use on apricot, cherry, citrus fruits (excluding *unshū* oranges), nectarine, peach, and Japanese plum. It shall not remain more than 0.008 g/kg in citrus fruits (excluding *unshū* oranges), 0.004 g/kg in apricot, cherry, nectarine, and peach(for apricot, nectarine, and peach, the limit is applied to one kilogram of each fruit from which the stone is removed and for cherry, the limit is applied to one kilogram of the fruit from which the stone and peduncle are removed), and 0.0006 g/kg in Japanese plum(the limit is applied to one kilogram of the fruit from which the stone is removed).

Compositional specifications (draft)

Substance name Propiconazole

Structural formula



Molecular formula $C_{15}H_{17}Cl_2N_3O_2$

Molecular weight 342.22

Chemical name [CAS number]
(2*RS*, 4*RS*; 2*RS*, 4*SR*)-1-[2-(2,4-dichlorophenyl)-4-propyl-1,3-dioxolan-2-ylmethyl]-1*H*-1,2,4-triazole［60207-90-1］

Content Propiconazole contains not less than 95.0% of propiconazole ($C_{15}H_{17}Cl_2N_3O_2$).

Description Propiconazole is a colorless to dark yellow-red viscous liquid. It is odorless.

Identification Determine the infrared absorption spectrum of Propiconazole, as directed in the Liquid Film Paste Method under Infrared Spectrophotometry, and compare it with the Reference Spectrum. Both spectra exhibit absorptions having about the same intensity at the same wavenumbers. Use optical plates made from sodium chloride.

Specific Gravity d_{20}^{20}: 1.288–1.290.

Purity

Lead Not more than 2 μg/g as Pb (2.0 g, Method 1, Control Solution: Lead Standard Solution 4.0 mL, Flame Method). After the sample is charred, ignite it at 450°C in an electric furnace.

Assay

Test Solution and Standard Solution Weigh accurately about 50 mg each of Propiconazole and propiconazole for assay, add 20 mL of the internal standard solution to each, add acetone to dissolve the mixture, and then make each of the solutions up to exactly 100 mL with acetone.

Internal Standard Solution Dissolve 75 mg of fludioxonil for assay in acetone to make exactly 50 mL.

Procedure Analyze 1-μL portions of the test solution and the standard solution by gas chromatography using the operating conditions given below. Determine the peak area ratios of propiconazole to fludioxonil for the test solution and the standard solution, and express as Q_T and Q_S, respectively. Calculate the amount of propiconazole by the formula:

$$\text{Content (\%) } of\ propiconazole\ (C_{15}H_{17}Cl_2N_3O_2)$$

$$= \frac{Weight\ (mg)\ of\ propiconazole\ for\ assay}{Weight\ (mg)\ of\ the\ sample} \times \frac{Q_T}{Q_S} \times 100$$

Operating Conditions

Detector: Hydrogen flame-ionization detector.

Column: A fused silica tube (0.25 mm internal diameter and 30 m length) coated with a 0.25-μm thick layer of dimethylpolysiloxan for gas chromatography.

Column temperature: Inject at 200°C, then raise the temperature at a rate of 5°C/minute to 280°C.

Injection port temperature: A constant temperature of about 250°C.

Detector temperature: A constant temperature of about 300°C.

Carrier gas: Helium.

Flow rate: Adjust the retention time of propiconazole to 10–15 minutes.

Injection method: Split.

Split ratio: 1:10.

Reagents and Test Solutions (TS)

Propiconazole for assay $C_{15}H_{17}Cl_2N_3O_2$ [60207-90-1] A colorless to yellow semi-gelatinous substance or a transparent viscous liquid.

Content Not less than 97.0% of propiconazole ($C_{15}H_{17}Cl_2N_3O_2$).

Identification Measure the absorption spectrum of propiconazole for assay as directed in the Liquid Film Method under Infrared Spectrophotometry. It exhibits absorptions at about 2960 cm^{-1}, 2870 cm^{-1}, 1587 cm^{-1}, 1506 cm^{-1}, and 1466 cm^{-1}, 1273 cm^{-1}, 1138 cm^{-1}, and 1028 cm^{-1}. Use optical plates made from sodium chloride.

Specific gravity d_{20}^{20}: 1.288–1.290.

Assay Weigh accurately about 40 mg of propiconazole for assay and about 4 mg of 1,4-BTMSB-d_4, and dissolve them together in 4 mL of deuterated acetone. Transfer the resulting solution to an NMR tube of 5 mm in external diameter, stopper tightly, and measure ^{1}HNMR spectra using an NMR spectrometer at a proton resonance frequency of 400 MHz or more. Assuming the signal of 1,4-BTMSB-d_4 as δ0.00 ppm, determine the signal area intensity (A) (corresponding to 1 hydrogen) at around δ 7.05–7.13 ppm. Assuming the signal area intensity of 1,4-BTMSB-d_4 as 18.00, when the conversion value of A and the purity of 1,4-BTMSB-d_4 are designated as I and P(%), respectively, determine the content of propiconazole by the following formula. Confirm that the signal from propiconazole around δ 7.05–7.13 ppm is not overlapped with that from a contaminant.

$$\text{Content (\%) of propiconazole } (C_{15}H_{17}Cl_2N_3O_2) = \frac{\text{Weight (mg) of 1,4 BTMSB}-d_4 \times \text{I} \times \text{P}}{\text{Weight (mg) of the sample}} \times 1.511$$

Operating conditions

Digital resolution: Not more than 0.25.

Spinning: Off.

^{13}C decoupling: Present.

Acquisition time: Not less than 4 seconds.

Spectral range: Not less than 20 ppm including between −5 ppm and 15 ppm.

Flip angle: 90°.

Delay time: 60 seconds.
Dummy scans: 2 or more.
Number of accumulation: Not less than 8.
Temperature at measurement: 20–30°C.

Deuterated acetone CD_3COCD_3 [666-52-4] Use a product exclusively produced for NMR spectrum measurement.

Reference spectrum

Propiconazole