

5.16 FLUENSULFONE (265)

RESIDUE AND ANALYTICAL ASPECTS

Fluensulfone was evaluated by JMPR for the first time for toxicology in 2013, when an ADI of 0-0.01 mg/kg bw/day and an ARfD of 0.3 mg/kg bw were established. Residues aspects of fluensulfone were evaluated by the 2014 and 2016 JMPR. The evaluation by the 2016 Meeting included a revision to the residue definitions for enforcement and dietary assessment as follows: The definition of the residue for enforcement for plant commodities is the sum of fluensulfone and 3,4,4-trifluorobut-3-ene-1-sulfonic acid (BSA), expressed as fluensulfone; the definition of the residue for dietary risk assessment for plant commodities is fluensulfone; and the definition of the residue for enforcement and dietary risk assessment for animal commodities is fluensulfone. The 2016 Meeting further determined that the residue is fat soluble.

The 48th Session of the CCPR, scheduled fluensulfone for evaluation of additional crop uses by the 2017 JMPR. The 2017 Meeting received residue data reflecting use of fluensulfone on citrus fruits, soya bean, sugarcane, coffee, and black pepper. Additional data were provided relating to analytical methods, storage stability, and processing.

The following residue components are discussed. Structures and chemical names are tabulated below.

Common name/abbreviation	Chemical name	Structure	Molecular weight
Fluensulfone, MCW-2	5-Chloro-2-[(3,4,4-trifluorobut-3-en-1-yl)sulfonyl]thiazole		291.7
Thiazole sulfonic acid, TSA, M-3625	5-Chloro-thiazole-2-sulfonic acid		199.6
Butene sulfonic acid, BSA, M-3627	3,4,4-Trifluorobut-3-ene-1-sulfonic acid		190.1
MeS, M-3626	2-Methylsulfonylthiazole		131.2

Methods of analysis

In the residue and storage stability studies provided to the current Meeting for crops except citrus pulp (dry), orange juice, and orange oil, residues of fluensulfone parent compound and its metabolites TSA and BSA were determined using an LC-MS/MS method (method number 1977W) that was evaluated and found acceptable by the 2014 JMPR. That method uses acetonitrile:H₂O extraction and C18 solid-phase extraction (SPE) for clean-up (BSA and TSA only). Modifications to that method were used for processed citrus and sugar cane commodities.

For orange juice, residues were extracted into methanol. For dried citrus pulp, the pulp was hydrated and then residues were extracted into methanol. For both matrices, the methanol extract was analysed directly for residues of BSA and after C18 SPE clean-up for residues of fluensulfone. For oil, residues were extracted into acetonitrile:H₂O and partitioned against hexane and then against ethyl

acetate. The aqueous phase was analysed for residues of BSA and the organic phase was analysed for residues of fluensulfone; slightly different protocols were used depending on the matrix. For citrus pulp, orange juice, and orange oil, analyses were for fluensulfone and BSA only.

Blackstrap molasses was extracted with acetonitrile:H₂O, centrifuged, filtered, and diluted prior to analysis for both fluensulfone and BSA. Refined sugar was dissolved in water and then methanol was added. For analysis of BSA, the methanol:H₂O extract was analysed without further clean-up; for analysis of fluensulfone, the extract was cleaned-up by C-18 SPE prior to analysis.

For all matrices, analysis was by LC-MS/MS. Validation data generated concurrently with each residue study demonstrated adequate method performance (recoveries 70–100%, maximum 11% RSD).

Stability of residues in stored analytical samples

The stability of fluensulfone residues in various matrices during frozen storage was evaluated by the 2014 and 2016 Meetings. Data submitted to the 2014 Meeting demonstrated stability in raw tomatoes for at least 15 months, processed tomato commodities for at least 6 months, in capsicum, cucumber and melon for at least 16 months. The 2016 Meeting received data for residues of fluensulfone, including the metabolites BSA and TSA, demonstrating stability during frozen storage ($\leq -20^{\circ}\text{C}$) in oranges for at least 18 months, in carrots for at least 17.5 months, in potato tubers for at least 23 months, and in dried potatoes and wet peel for at least 25 months for each of the three analytes.

The 2017 Meeting received storage stability data for peanut matrices including shelled nutmeat, hay, meal, and oil; and sugar cane matrices including billets, tops, and trash. For both crops, storage stability studies were conducted concurrently with supervised residue trials and processing studies.

Residues of both fluensulfone and BSA were stable in peanut matrices for at least 378 days and in sugar cane matrices for at least 55 days. The Meeting noted that although zero-day data from spiked samples were not provided except for peanut hay, the data support the stability determination based on the similarity in residues between the two sample points taken (180 and 378 days for peanut, 20 and 55 days for sugar cane) and on the stability determinations made by the previous Meetings for other matrices.

Results of supervised residue trials on crops

The Meeting received supervised trial data for applications of fluensulfone to citrus, soya beans, sugar cane, coffee, and black pepper. No evidence of registration for use on any of the crops under consideration was provided to the current Meeting; therefore, the Meeting was unable to make estimates of maximum residue levels.

Residues in animal commodities

Of the uses under consideration by the Meeting, citrus, soya bean, and sugar cane have significant livestock feed items. Data were provided for citrus dried pulp, soya bean seed, sugar cane tops, and sugarcane molasses; data were not provided for soya bean feed items (forage, hay, silage, or meal) or sugar cane bagasse.

As there were no GAPs available for the data considered by the Meeting, the Meeting did not re-evaluate the previous dietary burdens and recommendations from the 2016 Meeting, i.e., that '*maximum residue levels for mammalian commodities should be estimated at the LOQ [0.01 mg/kg], with dietary parameters at 0'* and that '*maximum residue levels for eggs, poultry meat and poultry offal should be estimated at the LOQ, with dietary parameters at 0.'*