UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OFFICE OF
PREVENTION, PESTICIDES
AND TOXIC SUBSTANCES

MEMORANDUM

Date: October 12, 2004

Subject: PP#1F06312 – Sulfuryl Fluoride. Section 3 Registration for the Post-harvest Fumigation of Stored Cereal Grains, Dried Fruits, and Tree Nuts, and Fumigation of Grain Milling Establishments. Corrected Summary of Analytical Chemistry and Residue Data.

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NOTE: This Residue Chemistry Summary Document corrects errors in the original summary document addressing petition 1F06312 (D283007, M. Doherty, 1/13/04). The original summary document is based in part on DER 45396301 which incorrectly reported residues of sulfuryl fluoride in corn grits as 14.4 ppm rather than 14.4 ppb. The DER has been corrected. This document reflects the data in the corrected DER.

Executive Summary

Sulfuryl fluoride is being proposed as a methyl bromide replacement to control post-harvest insect and rodent pests in stored grain, dried fruit, and tree nut commodities and in grain milling establishments. Sulfuryl fluoride is a fumigant and, in the form of ProFume™, is formulated as 99+% active ingredient. The fumigation rate for sulfuryl fluoride is the product of the fumigant concentration and exposure time. The maximum target rate is 1500 mg·hr/L for normal atmospheric fumigations and 200 mg·hr/L for vacuum fumigations. Double fumigations are recommended for insect infestations where eggs may be present, with the second fumigation timed to control newly hatched, immature stages. The proposed label specifies that all food

commodities be aerated for a minimum of 24 hours prior to the foods entering commerce.

Tolerances are currently established under an experimental use permit for residues of sulfuryl fluoride in/on walnuts and raisins (40 CFR 180.575) and for residues of inorganic fluoride resulting from the use of either sulfuryl fluoride or cryolite (40 CFR 180.145). Sulfuryl fluoride is highly reactive and breaks down to form sulfate and fluoride anions. Parent sulfuryl fluoride and the fluoride anion are the residues of concern for both the tolerance expression and risk assessment purposes.

Storage stability data were not submitted for sulfuryl fluoride. Samples were analyzed for sulfuryl fluoride residues as rapidly as possible following the post-fumigation aeration period. Because the storage interval was very short, storage stability data are not needed for sulfuryl fluoride, per se. Storage stability data for fluoride anion indicate that fluoride is stable in wheat grain, corn grain, corn meal, raisins, and walnuts for up to 140 days. Fluoride residues decline in wheat flour at a rate of 0.3% per day. It is unclear whether this reflects a true dissipation of fluoride from the samples or an increase in "bound" residues. Background residues of fluoride in the control samples for all commodities in the storage stability study decrease with time and the rate appears to be of the same order of magnitude as that observed for wheat flour. How this decline of fluoride anion in the control samples relates to residues in treated commodities or to the regulation of fluoride anion is unclear at this time.

The petitioner has proposed separate methods for the analysis of sulfuryl fluoride and fluoride anion. Residues of sulfuryl fluoride are extracted with water, allowed to volatilize, and then determined by a GC/ECD method that uses headspace analysis. Based on validation data, the limit of detection (LOD) is 0.004 ppm and the limit of quantitation (LOQ) is 0.008 ppm (0.02 ppm for corn grain and wheat germ). The method for the analysis of fluoride anion uses aqueous buffered extraction and a fluoride-selective electrode with the double-known-addition technique for quantifying residues. The petitioner initially reported that the LOD and LOQ for the fluoride method are 0.2 and 0.5 ppm, respectively. Following the independent laboratory validation, the LOQ for the fluoride method was increased to 2 ppm. The petitioner has <u>not</u> demonstrated that either method is capable of extracting incurred residues from cereal grain commodities. Both methods have been reviewed by the Agency's Analytical Chemistry Branch, which recommended that (1) the petitioner radiovalidate both methods and (2) OPP accept the analytical methods without a laboratory validation based on the submitted data (Method Review Memorandum, D. Wright, D282408, 8/14/03).

The proposed tolerances are based on minimal data. Although the petitioner submitted a large quantity of data from studies investigating the effects of various fumigation parameters on sulfuryl fluoride and/or fluoride anion residue levels, very few studies were conducted according to the proposed label directions. In examining the residue data, HED has pooled data across various fumigation parameters when those parameters appear to have little effect on residue levels.

Cereal Grains. Generally, residues of sulfuryl fluoride were below the limit of quantitation in the cereal grain commodities following fumigation at ~1500 mg·hr/L and an

active aeration period of 24 hours. Occasionally, quantifiable residues of sulfuryl fluoride were found in/on wheat grain (maximum residue = 0.095 ppm), rice grain (0.025 ppm), rice hulls (0.057 ppm), corn grain (0.026 ppm), corn grits (0.0144 ppm). All samples of fumigated corn oil had quantifiable residues of sulfuryl fluoride, with a maximum residue of 7.84 ppm. Apparent residues of sulfuryl fluoride were less than the LOQ in all control samples. For cereal grains, residues of fluoride anion were greater than the LOQ in all commodities except corn oil. Fluoride residues increase with multiple fumigations and appear to be recalcitrant, not declining following longer aeration intervals. Fluoride residues from mill fumigation studies were generally greater than those that resulted from laboratory fumigation studies of cereal grain commodities. Following a single fumigation at ~1500 mg·hr/L, maximum residues ranged from 5.3 ppm (corn grain) to 104 ppm (wheat germ). Measurable fluoride anion residues occurred in most control samples; the residue levels varied from one commodity to another and ranged from 0.03 to 2.08 ppm.

Processing studies conducted with whole grain corn and whole grain wheat showed that residues of fluoride concentrate in wheat shorts (1.26X), wheat bran (2.56X), wheat germ (4.82X), and corn "impurities" (5.49X). Impurities are described as being similar to aspirated grain fractions. The processed cereal grain commodities were generated using simulated commercial practices. Sulfuryl fluoride analyses were not done for commodities processed from treated grain samples. This is not considered to be a data gaps since sulfuryl fluoride is not expected to survive the milling process.

Dried Fruits and Tree Nuts. In dried fruits and tree nuts, residue levels of sulfuryl fluoride varied based on the commodity and the treatment regime. For most commodities, residues had dissipated to <2.1 ppb within 6 days of active aeration (1.5 chamber volumes/min) following fumigation. Sulfuryl fluoride residues were more persistent in commodities with higher oil content (e.g, walnuts, pecans, almonds), typically requiring closer to 14 days for residues to dissipate to <2.1 ppb. At the same fumigation rate, residues of sulfuryl fluoride were greater following vacuum fumigation versus treatment at ambient pressure. In oily commodities, multiple fumigations resulted in higher residues of sulfuryl fluoride at a given aeration time. Pooled across all of the variables addressed in this study, sulfuryl fluoride residues ranged from <2.1 ppb to 6030 ppb (6.03 ppm). Residue levels of fluoride were measured only after residues of sulfuryl fluoride had dissipated to below detectable levels; therefore, the effect of aeration time on fluoride levels cannot be assessed from these data. Generally, fluoride residues appear to be more dependent on the number of fumigations than on the treatment rate, treatment pressure, or commodity. Overall, fluoride residue levels ranged from <1.4 ppm to 21.8 ppm.

Recommendations

The residue chemistry databases for both sulfuryl fluoride and fluoride anion are considered marginally adequate to set tolerances based on the proposed use pattern. As a condition of registration, HED is recommending that further residue data are collected to ensure that the tolerances being recommended by HED are appropriate.

Residue Chemistry Deficiencies

- The number of cereal grain magnitude of the residue studies conducted at the maximum proposed use rate is marginally adequate. Residue data for both sulfuryl fluoride and fluoride anion should be submitted. The data should be from samples in at least three different grain mills that were treated according to the proposed maximum use. The matrices to be analyzed should include raw and processed commodities of wheat, rice, sorghum, and corn, including corn oil.
- The number of dried nut and tree fruit magnitude of the residue studies conducted at the maximum proposed use rate is marginally adequate. Residue data for both sulfuryl fluoride and fluoride anion should be submitted for representative commodities for these two groups. Samples should be treated according to the proposed maximum use.
- Both HED and the Analytical Chemistry Branch are concerned about the ability of the analytical methods to extract incurred residues. Data showing the ability of the sulfuryl fluoride and fluoride anion methods to extract and accurately quantify incurred residues in raw and processed cereal grain matrices should be submitted. Furthermore, HED is requesting that the sulfuryl fluoride method be validated for corn oil. If the current method proves to be acceptable for corn oil, then the requested label restriction (see below) can be lifted and HED will recommend that a tolerance be established for corn oil.
- The sulfuryl fluoride method has not been shown to be specific to sulfuryl fluoride. An interference study for sulfuryl fluoride should be submitted.
- Cereal grain commodities, including aspirated grain fractions, are significant livestock feed items. Feeding studies were not submitted to determine the extent of secondary residues that may occur in livestock commodities. HED is requesting data showing the transfer of fluoride from feedstuffs into livestock commodities. A feeding study is not being requested for sulfuryl fluoride.
- A revised Section F (Proposed Tolerances) is required.

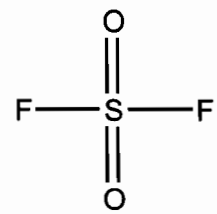
HED notes that data are sufficient to set sulfuryl fluoride and fluoride anion tolerances provided that the following modifications are made to the label:

- The total fumigation rate shall not exceed 1500 mg·hr/L at ambient pressure or 200 mg·hr/L under reduced pressure.
- Active aeration of at least 24 hours at not less than 1 chamber volume/min shall occur for all commodities prior to their entering commerce. This requirement does not apply to mill fumigations since practices to ensure worker safety will result in adequate aeration.
- Corn oil shall be removed from the premises prior to fumigation.

Provided such label changes are made, HED is recommending a conditional registration with the sulfuryl fluoride and fluoride anion tolerances shown in Table 5.

Background

Dow AgroSciences has requested permanent tolerances for residues of sulfuryl fluoride and fluoride anion resulting from the post-harvest fumigation of stored cereal grains, cereal grain processed commodities, dried fruit, and tree



nuts. Sulfuryl fluoride is a potential methyl bromide replacement. Dow AgroSciences was recently given an experimental use permit for the post-harvest fumigation of walnuts and raisins.

Sulfuryl fluoride is a highly volatile compound with a boiling point of -55°C and a vapor pressure of 0.02 Torr. At 20°C, sulfuryl fluoride has a vapor density of 4.3 g/L (heavier than air) and is both colorless and odorless. The log K_{OW} is estimated to be 0.41. Sulfuryl fluoride has a very low solubility in water (0.075 g/100 g). Solubility in other solvents are 0.78 g/100 g in Wesson oil, 1.74 g/100 g in acetone, and 2.12 g/100 g in chloroform.

860.1200 Directions for Use

Table 1. Summary of Directions for the Post-harvest Use of Sulfuryl Fluoride from the Proposed Label.

Applic. Timing, Type, and Equip.	Formulation [EPA Reg. No.]	Applic. Rate (mg·hr/L)	Max. No. Applic. per Season	Max. Seasonal Applic. Rate (mg·hr/L)	Aeration (hours)	Use Directions and Limitations
Post-harvest fumigation of sealed mills, warehouses, chambers, and other storage structures.	ProFume [62719-XXX]	1500 (ambient pressure) 200 (vacuum fumigation)	2	3000 (ambient pressure) 400 (vacuum fumigation)	24	Food commodities must be aerated for 24 hours prior to entering commerce.

The proposed label has sufficient information to allow the Agency to evaluate the residue trials in light of the proposed use patterns. Prior to registration, HED is requesting that the label and use manual be modified to specify a maximum fumigation rate of 1500 mg·hr/L at ambient pressure or 200 mg·hr/L at reduced pressure, that commodities be actively aerated (not less than 1 chamber volume/min) for at least 24 hours prior to their entering commerce, and that corn oil be removed from any premises prior to fumigation.

860.1300 Nature of the Residue - Plants

DER: None
MARC Decision Memo: None

Nature of the residue studies have not been submitted for sulfuryl fluoride. Following application, sulfuryl fluoride breaks down to form sulfate and fluoride anion. Sulfate is not of toxicological concern. Fluoride anion is of toxicological concern due to potential skeletal fluorosis. The residues of concern for both tolerance enforcement and risk assessment purposes are sulfuryl fluoride and fluoride anion. The lack of nature of the residue studies is not considered to be a deficiency and no further data are required to fulfill this guideline.

860.1300 Nature of the Residue - Livestock

DER: None
MARC Decision Memo: None

As with plants, sulfuryl fluoride would be expected to hydrolyze to form sulfate and fluoride anions. The residues of concern in livestock are sulfuryl fluoride and the fluoride anion; however, due to the nature of the sulfuryl fluoride molecule, HED believes it is unlikely that secondary residues of sulfuryl fluoride will occur in livestock commodities. The lack of nature of the residue studies is not considered to be a deficiency and no further data are required to fulfill this guideline.

860.1340 Residue Analytical Methods

DER: M. Doherty, 1/13/04, MRID 45632902 (Sulfuryl Fluoride; 45632902.der.wpd)
M. Doherty, 1/13/04, MRID 45603901 (Fluoride anion; 45603901.der.wpd)

Sulfuryl Fluoride. The proposed method utilizes headspace analysis to quantitate residues of sulfuryl fluoride. Residues of sulfuryl fluoride are extracted with water in an air-tight blender. The samples are agitated in the blender for five minutes to transfer residues of the highly volatile sulfuryl fluoride from the grain commodity to the headspace above the sample. The headspace is then analyzed for residues of sulfuryl fluoride by injecting an aliquot into a gas chromatograph with electron-capture detection (GC/ECD). The method has been validated using fortification/ recovery experiments on wheat, corn, and rice commodities. Fortification levels ranged from 0.008 to 0.08 ppm (0.2 ppm for wheat). Average recoveries and standard deviations were 88 ± 14% for wheat, 94 ± 16% for corn, and 82 ± 11% for rice. Standard deviations at the 0.008-ppm fortification level ranged from 0.00099 to 0.00129 ppm, resulting in calculated limits of detection and quantification of approximately 0.003 and 0.010 ppm, respectively. There were no apparent residues of sulfuryl fluoride in the control samples. Due to the gaseous nature of sulfuryl fluoride, fortifications were made to the headspace above the commodities and not to the commodities themselves. The method has not been radiovalidated and it is unclear if the method will successfully extract incurred residues. The method has not been shown to be specific to sulfuryl fluoride. It is likely that the method would extract and detect other volatile halogenated compounds (e.g., methyl bromide).

The method underwent an independent laboratory validation. After a considerable amount of consultation between the confirming laboratory and Dow AgroSciences, the laboratory was able to successfully validate the method for wheat grain (average recovery = 104 ± 10%) and wheat flour (94 ± 10%). Other cereal grain commodities were not tested.

The method has been reviewed by the Agency's Analytical Chemistry Branch (ACB; D. Wright Jr., D282408, 8/14/2003). Although the ACB concurs with HED regarding the ability of the method to extract incurred residues and recommends that the petitioner radiovalidate the method, they recommend that OPP accept the data and that an Agency method validation is not required at this time (D. Wright, D282408, 8/14/2003). Furthermore, HED notes that although control samples of corn oil were analyzed, the method was not validated for corn oil. Due to the physicochemical properties of sulfuryl fluoride, oily commodities are the most likely to bear residues following fumigation. In addition to radiovalidation of the method, HED requests that the method performance be verified for corn oil.

Fluoride Anion. The method consists of homogenizing and shaking the sample in the presence of water and total ionic strength adjusting buffer, centrifuging the sample, and analyzing an aliquot of the supernatant with a fluoride-selective electrode. Quantitation of fluoride residues is achieved using a double-known-addition technique.

The method was validated by the petitioner using whole grain wheat, whole grain corn, wheat flour, and corn oil. Two independent laboratory validations were performed; the first used whole grain wheat and wheat flour, and the second used corn oil and raisins. Recovery of fluoride was comparable across all tested commodities and between the petitioner validations and the independent laboratory validations. Generally, recovery of fluoride fell within the acceptable range of 70-120% after values were corrected for background levels of fluoride in control samples. Initial laboratory validation experiments showed limits of detection (LOD) and quantification (LOQ) of approximately 0.2 and 0.5 ppm, respectively. During the independent laboratory validation, the LOQ was increased to 2.0 ppm.

As with the sulfuryl fluoride method, the ability of the method to extract incurred residues has not been demonstrated. HED requests that the petitioner characterize the efficacy of the method with respect to incurred residues.

Conclusions. Both the sulfuryl fluoride and the fluoride anion methods have undergone review by the Agency's ACB. The ACB recommends that the methods be radiovalidated and that they be accepted without a full method validation.

860.1360 Multiresidue Methods

DER: None

Multiresidue method studies have not been submitted to the Agency. Based on their physicochemical properties, neither sulfuryl fluoride nor fluoride anion are likely to be suitable for the multiresidue techniques currently in use. The lack of multiresidue studies is not considered to be a deficiency and no further data are required to fulfill this guideline.

860.1380 Storage Stability

DER: M. Doherty, 1/13/04, MRID 45510302 (45510302.der.wpd)

Sulfuryl Fluoride. Storage stability data for sulfuryl fluoride were not submitted. Sulfuryl fluoride analysis of field trial samples was completed very shortly following the fumigation/aeration of the grain commodities. Storage stability data are not required to support the sulfuryl fluoride residue analysis for the field trial samples because of this short storage interval.

Fluoride Anion. Samples of wheat grain, wheat flour, corn grain, corn meal, raisins, and walnuts were individually homogenized and fortified with an aqueous fluoride solution to a concentration of 20 ppm fluoride. The fortified samples were stored at ambient temperatures for 30 days and then placed into frozen (-20°C) storage for the remainder of the study. On Day 0 and approximately Day 35, Day 75, Day 105, and Day 140 after fortification, the storage stability samples, along with a control sample, a reagent blank, and two daily concurrent recovery samples, were analyzed for fluoride anion. Fluoride residues were extracted using an aqueous solution and analyzed using a fluoride ion selective electrode. The reported LOQ for the method is 0.6 ppm for wheat grain, 0.3 ppm for wheat flour, and 0.5 ppm for corn grain. LOQs for other matrices were not given.

Residues of fluoride anion were stable for the duration of the study (~140 days) in wheat grain, corn grain, corn meal, raisins, and walnuts. In wheat flour, however, fluoride residues exhibited a continual decline throughout the study, with only 55% remaining at 140 days. Regression of the residue data indicates a loss of approximately 0.3% per day. The study design does not permit determination of whether or not the loss reflects dissipation of the fluoride residues or an increase in "bound" residues. This loss should be taken into account when interpreting the results of residue studies in wheat flour. HED will not assume that fluoride anion is stable in all commodities. Furthermore, HED notes that the residues in the control samples for all matrices indicate a loss of fluoride with time and that this loss appears to be of the same order of magnitude as that observed in wheat flour. The significance of this trend as it relates to storage stability and field trial results is unclear at this time.

Conclusions. The storage stability data support the storage intervals and conditions for samples from the field trial and processing studies.

860.1400 Water, Fish, and Irrigated Crops

DER: None

There are no proposed uses for sulfuryl fluoride that would result in residues in fish or irrigated crops. A discussion of the 860.1400 guideline is not germane to this petition.

860.1460 Food Handling

DER: None

There are no proposed uses for sulfuryl fluoride in or around food handling areas. A discussion of the 860.1460 guideline is not germane to this petition.

860.1480 Meat, Milk, Poultry, and Eggs

DER: None

Studies describing residues of sulfuryl fluoride and fluoride anion in meat, milk, poultry, and eggs have not been submitted to the Agency. Due to the nature of the sulfuryl fluoride molecule, HED believes it is unlikely that secondary residues of sulfuryl fluoride will occur in livestock commodities. The ability of fluoride anion to be transferred from animal feeds to livestock commodities is unclear. Since cereal grain commodities, including aspirated grain fractions, are a major cattle and poultry feed item, HED is requesting that livestock feeding studies be done for the fluoride anion.

860.1500 Crop Field Trials

DER: M. Doherty, 1/13/04, MRID 45396301 (45396301.der.wpd)
M. Doherty, 1/13/04, MRID 45510303 (45510303.der.wpd)
M. Doherty, 1/13/04, MRID 45510304 (45510304.der.wpd)

Cereal Grains

Table 2. Summary of Sulfuryl Fluoride Residues in Cereal Grains. Residue data have been pooled across treatment rate and loading factor. The proposed use pattern is a maximum of 2 fumigations, with a total rate not to exceed 1500 mg·hr/L, and a 24-hour aeration period. Data from more than 2 fumigations or aeration periods greater than 1 day have not been included in this table.

Crop	Form	Location	Temp. (°C)	Fumigation No.	Aeration (days)	n	Sulfuryl Fluoride Residues (ppm)			
							Min	Max	Mean	Std Dev
Corn	Starch	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	Flour	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	Grain	Lab	22	1	1	2	<0.020	0.026	0.023	0.004
Corn	Grits	Lab	22	1	1	2	<0.008	0.014	0.011	0.005
Corn	Meal	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	Oil	Lab	22	1	1	2	5.848	7.840	6.844	1.409
Rice	Bran	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Rice	Hulls	Lab	22	1	1	2	0.056	0.057	0.057	0.001
Rice	Grain	Lab	22	1	1	2	0.016	0.025	0.021	0.007
Rice	Polished	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	Bran	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	Flour	Lab	30	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	Flour	Lab	10	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	Flour	Lab	22	1	1	10	<0.008	<0.008	<0.008	0.000
Wheat	Flour	Lab	22	2	1	2	<0.008	0.009	0.009	0.001
Wheat	Germ	Lab	22	1	1	10	<0.020	<0.020	<0.020	0.000
Wheat	Germ	Lab	30	1	1	2	<0.020	<0.020	<0.020	0.000

Crop	Form	Location	Temp. (°C)	Fumigation No.	Aeration (days)	n	Sulfuryl Fluoride Residues (ppm)			
							Min	Max	Mean	Std Dev
Wheat	Germ	Lab	10	1	1	2	<0.020	<0.020	<0.020	0.000
Wheat	Germ	Lab	22	2	1	2	<0.020	<0.020	<0.020	0.000
Wheat	Grain	Lab	22	1	1	10	<0.008	0.090	0.021	0.026
Wheat	Grain	Lab	10	1	1	2	0.032	0.033	0.033	0.000
Wheat	Grain	Lab	30	1	1	2	0.013	0.014	0.013	0.001
Wheat	Grain	Lab	22	2	1	2	0.086	0.095	0.091	0.007
Wheat	Red dog	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	Shorts	Lab	22	1	1	2	<0.008	<0.008	<0.008	0.000

Table 3. Summary of Fluoride Anion Residues in Cereal Grains. Residue data have been pooled across loading factor and aeration time. The proposed use pattern is a maximum of 2 fumigations, with a total rate not to exceed 1500 mg·hr/L, and a 24-hour aeration period. Data from rates less than ~1000 or greater than ~1800 mg·hr/L or from more than 2 fumigations have not been included in this table. Bolded rows indicate the maximum residue for each crop/commodity.

Crop	Commodity	Location	Temp. (°C)	Fumig. No.	Rate[A] (mg-hr/L)	n	Fluoride Anion Residues (ppm)			
							Min	Max[B]	Mean	SD
Barley	Grain	Lab	30	1	1646	4	2.76	3.05	2.88	0.13
Barley	**Grain**	**Mill**	**Amb.[C]**	**1**	**1012**	**4**	**4.27**	**9.25**	**6.27**	**2.37**
Barley	Grain	Mill	Amb.	1	1798	2	6.49	7.96	7.23	1.04
Corn	Flour	Lab	22	1	1500	4	14.90	19.20	17.10	2.26
Corn	Flour	Mill	Amb.	1	1012	4	17.20	24.10	19.35	3.19
Corn	**Flour**	**Mill**	**Amb.**	**1**	**1798**	**2**	**22.70**	**24.10**	**23.40**	**0.99**
Corn	Grain	Lab	30	1	1436	4	1.31	1.64	1.43	0.15
Corn	Grain	Lab	22	1	1500	4	2.07	2.26	2.18	0.09
Corn	Grain	Lab	30	1	1504	4	0.76	1.41	1.00	0.28
Corn	Grain	Mill	Amb.	1	1012	4	1.00	2.05	1.39	0.48
Corn	**Grain**	**Mill**	**Amb.**	**1**	**1012**	**4**	**1.03**	**5.31**	**2.43**	**2.00**
Corn	Grain	Mill	Amb.	1	1798	2	1.19	1.52	1.36	0.23
Corn	Grain	Mill	Amb.	1	1798	2	1.71	1.86	1.79	0.11
Corn	**Grits**	**Lab**	**22**	**1**	**1500**	**4**	**7.74**	**9.17**	**8.57**	**0.69**
Corn	Meal	Lab	22	1	1500	4	5.24	6.30	5.82	0.49
Corn	Meal	Mill	Amb.	1	1012	4	10.50	21.00	14.83	4.66
Corn	**Meal**	**Mill**	**Amb.**	**1**	**1798**	**2**	**21.60**	**26.40**	**24.00**	**3.39**
Corn	**Oil**	**Lab**	**22**	**1**	**1500**	**4**	**<0.50**	**<0.50**	**<0.50**	**0.00**
Corn	**Starch**	**Lab**	**22**	**1**	**1500**	**4**	**3.82**	**5.35**	**4.60**	**0.84**
Oats	Grain	Lab	30	1	1560	4	7.00	8.27	7.54	0.53
Oats	**Grain**	**Mill**	**Amb.**	**1**	**1012**	**4**	**6.82**	**15.25**	**9.78**	**3.77**
Oats	Grain	Mill	Amb.	1	1798	2	12.50	14.00	13.25	1.06
Rice	**Bran**	**Lab**	**22**	**1**	**1500**	**4**	**24.20**	**28.50**	**25.90**	**1.96**
Rice	Brown	Lab	30	1	1599	4	2.16	2.38	2.24	0.10
Rice	**Brown**	**Mill**	**Amb.**	**1**	**1012**	**4**	**3.84**	**11.80**	**6.78**	**3.60**
Rice	Brown	Mill	Amb.	1	1798	2	6.24	7.29	6.77	0.74
Rice	**Grain**	**Lab**	**22**	**1**	**1500**	**4**	**5.49**	**8.46**	**6.93**	**1.43**
Rice	**Hulls**	**Lab**	**22**	**1**	**1500**	**4**	**23.40**	**32.80**	**27.53**	**4.76**
Rice	Polished	Lab	22	1	1500	4	1.30	1.60	1.44	0.13
Rice	Polished	Lab	30	1	1547	4	1.83	2.03	1.96	0.09
Rice	**Polished**	**Mill**	**Amb.**	**1**	**1012**	**4**	**3.21**	**16.10**	**7.44**	**5.85**
Rice	Polished	Mill	Amb.	1	1798	2	7.64	10.70	9.17	2.16
Wheat	**Bran**	**Lab**	**22**	**1**	**1500**	**4**	**34.00**	**37.10**	**35.95**	**1.38**
Wheat	Flour	Lab	22	1	1000	2	19.30	21.10	20.20	1.27
Wheat	Flour	Lab	10	1	1500	2	14.60	15.70	15.15	0.78
Wheat	Flour	Lab	22	1	1500	18	21.50	44.70	32.57	6.18
Wheat	Flour	Lab	30	1	1500	2	33.40	37.80	35.60	3.11
Wheat	Flour	Lab	22	2	1500	2	62.50	62.60	62.55	0.07
Wheat	Flour	Mill	Amb.	1	1012	4	21.20	49.70	36.48	12.14
Wheat	Flour	Mill	Amb.	1	1798	2	46.4	49.30	47.85	2.05

Crop	Commodity	Location	Temp. (°C)	Fumig. No.	Rate[A] (mg-hr/L)	n	Fluoride Anion Residues (ppm)			
							Min	Max[B]	Mean	SD
Wheat	Flour, Semolina	Mill	Amb.	1	1012	4	13.10	27.30	21.63	6.46
Wheat	Flour, Semolina	Mill	Amb.	1	1798	2	25.50	28.40	26.95	2.05
Wheat	Flour, Unbleached White	Mill	Amb.	1	1012	4	22.90	51.50	37.50	14.89
Wheat	Flour, Unbleached White	Mill	Amb.	1	1012	4	26.00	57.00	39.33	13.17
Wheat	Flour, Unbleached White	Mill	Amb.	1	1798	2	41.70	45.30	43.50	2.55
Wheat	Flour, Unbleached White	Mill	Amb.	1	1798	2	40.40	49.30	44.85	6.29
Wheat	Flour, Whole	Mill	Amb.	1	1012	4	22.40	32.90	26.83	4.50
Wheat	**Flour, Whole**	**Mill**	**Amb.**	**1**	**1012**	**4**	**28.10**	**82.30**	**44.83**	**25.48**
Wheat	Flour, Whole	Mill	Amb.	1	1798	2	35.60	40.40	38.00	3.39
Wheat	Flour, Whole	Mill	Amb.	1	1798	2	40.50	48.20	44.35	5.44
Wheat	Germ	Lab	22	1	1000	2	55.30	58.60	56.95	2.33
Wheat	Germ	Lab	10	1	1500	2	17.40	18.60	18.00	0.85
Wheat	Germ	Lab	22	1	1500	18	41.60	104.00	67.01	17.86
Wheat	Germ	Lab	30	1	1500	2	72.30	82.60	77.45	7.28
Wheat	**Germ**	**Lab**	**22**	**2**	**1500**	**2**	**121.00**	**158.00**	**139.50**	**26.16**
Wheat	Germ	Mill	Amb.	1	1012	4	33.40	41.10	38.10	3.31
Wheat	Germ	Mill	Amb.	1	1012	4	41.40	89.70	64.88	24.32
Wheat	Germ	Mill	Amb.	1	1798	2	55.90	63.30	59.60	5.23
Wheat	Germ	Mill	Amb.	1	1798	2	78.80	85.90	82.35	5.02
Wheat	Grain	Lab	22	1	1000	2	1.52	1.74	1.63	0.16
Wheat	Grain	Lab	10	1	1500	2	0.79	0.91	0.85	0.08
Wheat	Grain	Lab	22	1	1500	18	1.47	4.08	2.47	0.84
Wheat	Grain	Lab	30	1	1500	2	2.79	2.96	2.88	0.12
Wheat	Grain	Lab	22	2	1500	2	5.02	5.02	5.02	0.00
Wheat	Grain	Lab	30	1	1713	4	3.38	4.84	4.06	0.62
Wheat	Grain	Lab	30	1	1768	4	1.93	2.24	2.08	0.13
Wheat	Grain	Lab	30	1	1769	4	2.64	3.50	3.03	0.44
Wheat	Grain	Mill	Amb.	1	1012	4	2.98	6.54	4.49	1.57
Wheat	Grain	Mill	Amb.	1	1012	4	2.12	8.36	4.53	2.68
Wheat	**Grain**	**Mill**	**Amb.**	**1**	**1012**	**4**	**3.61**	**23.60**	**9.19**	**5.30**
Wheat	Grain	Mill	Amb.	1	1798	2	4.84	5.65	5.25	0.57
Wheat	Grain	Mill	Amb.	1	1798	2	4.79	5.80	5.30	0.71
Wheat	Grain	Mill	Amb.	1	1798	2	4.73	6.06	5.40	0.94
Wheat	**Red dog**	**Lab**	**22**	**1**	**1500**	**4**	**31.70**	**33.30**	**32.50**	**0.67**
Wheat	**Shorts**	**Lab**	**22**	**1**	**1500**	**4**	**31.90**	**35.50**	**34.18**	**1.57**

[A] For the grain mills, application rates were grouped into categories of <300 mg•hr/L, >300 mg•hr/L and <1200 mg•hr/L, and > 1200 mg•hr/L. The statistics listed for application rate and residue levels are for samples within each application rate grouping.
[B] Due to the design of this study, the maximum residue level and the highest average residue levels are identical.
[C] Amb. = Ambient Temperature

MRID 45396301. Studies were conducted to determine the magnitude of sulfuryl fluoride and fluoride anion residues in/on stored grain commodities following fumigation of the commodities with sulfuryl fluoride. The studies were designed to investigate the effects of temperature (10, 22, or 30 °C), number of fumigations (1-4), fumigation rate (250, 1000, 1500, or 2500 mg·hr/L), fumigation chamber loading (1, 10, 50, or 80% of capacity), and aeration time (1, 4, or 7 days; active aeration at approx. 1 chamber volume/min) on sulfuryl fluoride and fluoride anion residues. Residues of sulfuryl fluoride were analyzed using a GC/ECD headspace method. Residues of fluoride anion were analyzed using a fluoride ion selective electrode following the double standard addition technique.

For sulfuryl fluoride, residues were independent of fumigation rate, number of fumigations, temperature, and chamber loading factor. After one day of aeration, residues of sulfuryl fluoride were <LOQ in many commodites and <0.1 ppm in all commodities except corn grits (0.0144 ppm maximum) and corn oil (7.8 ppm maximum). Following four days of aeration,

residues of sulfuryl fluoride were <LOQ in all matrices except corn oil (2.6 ppm maximum) and wheat grain (0.04 ppm maximum; 10 °C only). Quantifiable residue of sulfuryl fluoride persisted in these two commodities even after 7 days of aeration (wheat grain treated at 10 °C only).

For anionic fluoride, residues were independent of chamber loading and aeration time. Residues vary significantly from one commodity to another, with maximum residues observed in wheat germ and wheat flour. The residue values in these commodities are positively correlated with the fumigation rate, fumigation temperature, and the number of fumigations. Quantifiable residues of fluoride anion were found in all matrices except corn oil (<0.5 ppm), further demonstrating the stability of parent sulfuryl fluoride in that matrix.

A large amount of data was submitted to characterize the effects of the fumigation parameters on residue levels. However, within a given set of parameters (i.e., a use pattern), there is very little replication. Pooling data across parameters that do not significantly affect residue levels can be done to increase the number of replicates. Even so, replication is only marginally sufficient to support tolerances for some of the use patterns conducted in this study.

MRID 45510303. Controlled laboratory and operational grain mill fumigations using sulfuryl fluoride were conducted with whole cereal grains (hard red winter wheat, soft red winter wheat, durum wheat, medium grain brown rice, medium grain white rice, white field corn, popcorn, barley, and oats) and representative processed commodities (wheat germ, wheat flour, and corn meal; grain mill fumigations only) in order to determine the magnitude of sulfuryl fluoride and fluoride anion residues that occur following treatment with sulfuryl fluoride. In the laboratory, commodities were fumigated for 24 hours at either 200 mg·hr/L or 1500 mg·hr/L and then aerated for 24 hours prior to analysis. In the grain mills, fumigations were for 24 hours at either ~280 mg·hr/L, ~1000 mg·hr/L, or ~1800 mg·hr/L and were followed by a 24-hour active aeration interval. Residues of sulfuryl fluoride were determined using GC/ECD headspace analysis (laboratory studies only). Samples of the laboratory- and the grain mill-treated commodities were analyzed for fluoride anion using aqueous extraction and fluoride selective-ion electrode. Sulfuryl fluoride analysis was completed immediately after the 24-hour aeration period.

Following the 24-hour aeration interval, residues of sulfuryl fluoride were less than the LOQ (0.008 ppm) in all laboratory-fumigated commodities, with the exception of one white corn subsample fumigated at the 1500-mg·hr/L rate. Residues in that sample were 0.019 ppm and dissipated to < 0.008 ppm by 48 hours after fumigation. Residues of sulfuryl fluoride were not determined in samples from the grain mills.

Residues of fluoride anion were dependent upon the sulfuryl fluoride treatment rate. Residues in whole grains from the laboratory fumigations ranged from <0.5 ppm to 2 ppm at the 200-mg·hr/L rate and from 1 ppm to 8 ppm at the 1500-mg·hr/L rate. Residues in/on grains from the mill fumigations ranged from 1 ppm to 8 ppm at the lower rate (~280 mg·hr/L) and from 1 ppm to 24 ppm at the higher rates (~1000 - 1800 mg·hr/L). Residues in processed commodities were higher than those in whole grains and ranged from 7 ppm to 50 ppm at the lower rate and from 11 ppm to 90 ppm at the higher rate. Although there is a fairly high degree of variability

across treatment replicates, the bulk of the variability in the residue levels appears to be due to properties of the commodities themselves.

There are no specific guidelines regarding the number of trials and replicates required for post-harvest pesticide uses. In the laboratory, two fumigation levels were used (200 and 1500 mg·hr/L) for each commodity and there were four replicates per fumigation level. In the grain mills, three fumigation levels were used (~280, ~1000, and ~1800 mg·hr/L) for each grain and processed commodity and three samples were collected per fumigation. This is equivalent to one trial at each fumigation level for each commodity. Given the residue picture from the entire available database, HED considers this to be a marginally acceptable number of trials for supporting sulfuryl fluoride or fluoride anion tolerances.

Dried Fruit and Tree Nuts

Table 4. Summary of Residue Data from Crop Fumigation Trials with Sulfuryl Fluoride.

Treatment ID - Crop	Fumigation Number	Fumigation Rate, mg·hr/L (cumulative rate)	Aeration Time, days	Residue Levels*				
				n	Min.	Max.	Mean	Std. Dev.
Sulfuryl Fluoride, ppb								
A - Almond	1	203	1	4	8.90	12.40	11.03	1.55
A - Almond	1		2	5	1.05	2.10	1.26	0.47
A - Dates	1	208	1	4	1.05	1.05	1.05	0.00
A - Dried Plums	1	219	1	4	1.05	1.05	1.05	0.00
A - Figs	1	197	1	5	1.05	7.20	4.41	2.19
A - Pecans	1	199	1	5	32.20	60.30	46.40	10.85
A - Pecans	1		2	4	17.30	25.30	21.90	3.40
A - Pecans	1		5	4	4.80	7.40	5.53	1.25
A - Pecans	1		8	4	1.05	1.05	1.05	0.00
A - Pistachios	1	214	1	4	1.05	1.05	1.05	0.00
A - Raisins	1	221	1	4	1.05	1.05	1.05	0.00
A - Walnuts	1	217	1	4	67.80	79.00	72.58	5.05
A - Walnuts	1		5	4	1.05	2.10	1.84	0.53
A - Walnuts	1		8	4	1.05	1.05	1.05	0.00
B - Almond	1	1534	1	4	28.00	40.00	33.50	4.93
B - Almond	2	1538 (3072)	1	4	44.00	75.00	57.50	13.63
B - Almond	2		2	5	22.00	55.00	35.20	13.81
B - Almond	2		5	2	7.00	8.10	7.55	0.78
B - Almond	3	1488 (4560)	1	4	107.00	128.00	121.00	9.49
B - Almond	3		5	4	14.00	18.00	15.50	1.91
B - Almond	3		8	4	1.05	2.10	1.84	0.53
B - Almond	3		15	4	1.05	1.05	1.05	0.00
B - Dates	1	1484	1	4	6.10	8.30	7.28	1.09
B - Dates	2	1504 (2988)	1	4	1.05	2.10	1.58	0.61
B - Dates	3	1493 (4481)	1	4	5.20	7.10	6.10	0.79
B - Dates	4	1503 (5984)	1	4	8.60	15.00	11.65	2.65
B - Dates	4		5	4	7.50	11.00	9.13	1.45

Table 4. Summary of Residue Data from Crop Fumigation Trials with Sulfuryl Fluoride.

Treatment ID - Crop	Fumigation Number	Fumigation Rate, mg·hr/L (cumulative rate)	Aeration Time, days	Residue Levels*				
				n	Min.	Max.	Mean	Std. Dev.
B - Dates	5	1491 (7475)	1	4	13.00	23.00	16.75	4.35
B - Dates	5		2	4	7.50	19.00	13.88	5.54
B - Dates	5		5	4	5.50	16.00	8.85	4.88
B - Dates	5		8	6	1.05	8.00	3.69	2.93
B - Dates	5		15	4	1.05	1.05	1.05	0.00
B - Dried Plums	1	1575	1	4	1.05	1.05	1.05	0.00
B - Dried Plums	2	1504	1	5	1.05	1.05	1.05	0.00
B - Dried Plums	3	1516	1	5	1.05	2.10	1.47	0.58
B - Dried Plums	4	1521	1	4	1.05	1.05	1.05	0.00
B - Figs	1	1462	1	4	33.00	41.00	37.00	4.08
B - Figs	2	1498 (2960)	1	4	11.00	15.00	13.00	1.63
B - Figs	2		2	4	2.10	9.30	5.58	4.02
B - Figs	2		5	2	4.50	5.60	5.05	0.78
B - Pecans	1	1533	1	4	2224.00	2688.00	2407.50	199.71
B - Pecans	1		5	4	99.00	105.00	102.75	2.63
B - Pecans	1		15	4	15.00	20.00	16.50	2.38
B - Pecans	2	1452 (2985)	1	3	4146.00	5532.00	4906.00	702.65
B - Pecans	2		15	4	12.00	16.00	13.75	1.71
B - Pecans	3	1510 (4495)	1	4	4276.00	6030.00	4950.50	842.09
B - Pecans	3		2	4	1304.00	3915.00	2564.00	1187.76
B - Pecans	3		5	4	199.00	261.00	228.50	27.50
B - Pecans	3		8	4	57.00	69.00	62.75	6.65
B - Pecans	3		15	4	1.05	1.05	1.05	0.00
B - Pistachios	1	1517	1	4	252.00	303.00	277.25	22.37
B - Pistachios	1		5	4	18.00	29.00	23.25	4.57
B - Pistachios	2	1507 (3024)	1	4	51.00	70.00	63.25	8.73
B - Pistachios	2		5	4	1.05	1.05	1.05	0.00
B - Pistachios	3	1506 (4530)	1	4	35.00	56.00	44.75	10.81
B - Pistachios	3		2	4	8.10	16.00	12.25	3.85
B - Pistachios	3		5	4	1.05	1.05	1.05	0.00
C - Almond	1	218	1	4	12.00	20.00	15.50	4.12
C - Almond	1		2	4	1.05	8.80	4.56	3.62
C - Almond	1		4	4	1.05	1.05	1.05	0.00
C - Pecans	1	206	1	4	1095.00	1306.00	1182.50	91.03
C - Pecans	1		2	4	369.00	462.00	419.75	46.35
C - Pecans	1		5	4	39.00	55.00	48.00	6.83
C - Pecans	1		8	5	4.50	7.20	5.64	1.04
C - Pecans	1		15	4	1.05	1.05	1.05	0.00
C - Pistachios	1	202	1	5	13.00	26.00	18.20	5.36
C - Pistachios	1		2	4	1.05	1.05	1.05	0.00
C - Walnuts	1	183	1	4	569.00	640.00	602.50	30.09
C - Walnuts	1		2	4	290.00	425.00	362.00	57.17
C - Walnuts	1		5	4	90.00	101.00	94.50	4.80
C - Walnuts	1		8	4	24.00	29.00	26.75	2.63
C - Walnuts	1		15	4	1.05	1.05	1.05	0.00

Fluoride Anion (ppm)

Table 4. Summary of Residue Data from Crop Fumigation Trials with Sulfuryl Fluoride.

Treatment ID - Crop	Fumigation Number	Fumigation Rate, mg·hr/L (cumulative rate)	Aeration Time, days	Residue Levels*				
				n	Min.	Max.	Mean	Std. Dev.
A - Almond	1	203	2	4	3.20	3.77	3.42	0.26
A - Dates	1	208	6	4	0.70	0.70	0.70	0.00
A - Dried Plums	1	219	1	4	0.70	0.70	0.70	0.00
A - Figs	1	197	1	5	0.70	0.70	0.70	0.00
A - Pecans	1	199	8	4	0.70	1.20	0.95	0.29
A - Pistachios	1	214	5	4	1.20	2.91	1.63	0.86
A - Raisins	1	221	5	5	0.70	0.70	0.70	0.00
A - Walnuts	1	217	9	4	0.70	0.70	0.70	0.00
B - Almond	1	1534	1	4	4.07	5.96	4.66	0.89
B - Almond	2	1538 (3072)	5	4	6.65	7.97	7.37	0.68
B - Almond	3	1488 (4560)	15	4	9.07	9.90	9.51	0.37
B - Dates	1	1484	4	4	0.70	0.70	0.70	0.00
B - Dates	2	1504 (2988)	5	4	0.70	1.20	0.95	0.29
B - Dates	3	1493 (4481)	5	4	1.20	1.20	1.20	0.00
B - Dates	4	1503 (5984)	4	4	0.70	2.74	1.46	0.89
B - Dates	5	1491 (7475)	15	4	1.20	3.09	1.67	0.95
B - Dried Plums	1	1575	2	4	0.70	0.70	0.70	0.00
B - Dried Plums	2	1504 (3079)	2	4	0.70	1.20	1.08	0.25
B - Dried Plums	3	1516 (4595)	4	4	0.70	2.56	1.42	0.80
B - Dried Plums	4	1521 (6116)	5	4	1.20	3.14	1.69	0.97
B - Figs	1	1462	6	4	1.20	1.20	1.20	0.00
B - Figs	2	1498 (2960)	5	4	0.70	2.43	1.26	0.82
B - Pecans	1	1533	13	4	7.72	9.59	8.57	0.79
B - Pecans	2	1452 (2985)	13	4	13.80	17.40	15.45	1.50
B - Pecans	3	1510 (4495)	16	4	19.80	21.80	20.83	1.02
B - Pistachios	1	1517	7	4	3.58	4.56	4.10	0.42
B - Pistachios	2	1507 (3024)	6	4	9.98	12.60	11.05	1.11
B - Pistachios	3	1506 (4530)	5	4	13.20	17.90	15.70	2.05
C - Almond	1	218	7	4	0.70	0.70	0.70	0.00
C - Pecans	1	206	16	4	0.70	1.20	1.08	0.25
C - Pistachios	1	202	3	4	0.70	1.20	0.83	0.25
C - Walnuts	1	183	15	4	0.70	1.20	0.83	0.25

*Note that residues are expressed as ppb for sulfuryl fluoride and ppm for fluoride anion. For purposes of

calculating the summary statistics, ½ LOD or ½ LOQ values were used as follows: sulfuryl fluoride ½ LOD = 1.05 ppb, ½ LOQ = 2.1 ppb; fluoride anion ½ LOD = 0.7 ppm, ½ LOQ = 1.2 ppm.

Supervised post harvest fumigation trials were conducted with walnuts, pistachios, pecans, almonds, dates, figs, dried plums, and raisins. Single fumigations were carried out at normal atmospheric pressure or under vacuum conditions at rates of approximately 200 mg•hr/L. Additionally, multiple fumigations (2-5) were conducted, each at approximately 1500 mg•hr/L. For each treatment/crop combination, 2 replicate fumigations were made and 2-3 samples were collected from each replicate. The trials monitored residues of sulfuryl fluoride and fluoride anion, as well as the time required for sulfuryl fluoride residues to dissipate to < LOQ (4.2 ng/g). Sulfuryl fluoride residues were analyzed using a head space method; fluoride anion was analyzed using a fluoride selective-ion electrode and a double-known-addition technique. Residue levels of sulfuryl fluoride varied based on the commodity and the treatment regime. For most commodities, residues had dissipated to <2.1 ppb within 6 days of aeration (1.5 chamber volumes/min) following fumigation. Sulfuryl fluoride residues were more persistent in commodities with higher oil content (e.g, walnuts, pecans, almonds), typically requiring closer to 14 days for residues to dissipate to <2.1 ppb. At the same fumigation rate, residues of sulfuryl fluoride were greater following vacuum fumigation versus treatment at ambient pressure. In oily commodities, multiple fumigations resulted in higher residues of sulfuryl fluoride at a given aeration time. Pooled across all of the variables addressed in this study, sulfuryl fluoride residues ranged from <2.1 ppb to 6030 ppb (6.03 ppm). Residue levels of fluoride were measured only after residues of sulfuryl fluoride had dissipated to below detectable levels; therefore, the effect of aeration time on fluoride levels cannot be assessed from these data. Generally, fluoride residues appear to be more dependent on the number of fumigations than on the treatment rate, treatment pressure, or commodity. Overall, fluoride residue levels ranged from <1.4 ppm to 21.8 ppm.

Conclusions. Significant data gaps are associated with these studies. The majority of the maximum fluoride residue levels were found in samples from the grain mill fumigations. Replication within those studies was low and there is a fairly high degree of variation in the residue results. Furthermore, laboratory studies show that fluoride levels accumulate with successive fumigations and the grain mill samples were subjected to only one fumigation. HED is requesting that the petitioner conduct fumigations in at least 3 different grain mills. The studies should include raw and processed commodities of wheat, rice, sorghum, and corn, including corn oil, and both sulfuryl fluoride and fluoride anion should be included in the analyses.

Despite these deficiencies, the submitted residue data are considered sufficient for granting a conditional registration for sulfuryl fluoride and for establishing sulfuryl fluoride and fluoride anion tolerances. Provided the label is changed such that (1) the total fumigation rate does not exceed 1500 mg·hr/L under ambient conditions or 200 mg·hr/L under reduced pressure conditions, and (2) corn oil must be removed from the premises prior to fumigation, the submitted data are sufficient to set sulfuryl fluoride and fluoride anion tolerances for stored cereal grain commodities, dried fruits, and tree nuts.

To determine appropriate tolerance levels for fluoride in/on wheat and corn commodities,

HED has taken the maximum fluoride residue resulting from a single fumigation of between ~1000 and ~1800 mg·hr/L from the appropriately pooled data. The resulting residue level was then corrected for fumigation rate when the maximum residue occurred at a rate below 1500 mg·hr/L. Based on the available data, HED believes that tolerance levels shown in Table 5 are appropriate.

860.1520 Processed Food and Feed

DER: M. Doherty, 1/13/04, MRID 45396301 (45396301.de1.wpd)

As part of a study examining the effects of various fumigation parameters on the residue levels of sulfuryl fluoride and fluoride anion in cereal grains, the petitioner included a processing study describing levels of fluoride anion in commodities obtained from the processing of treated wheat and corn grain.

Wheat and corn grain were fumigated under controlled conditions at a rate of 1500 mg·hr/L. Following a 24-hour aeration period, the grain samples were analyzed for fluoride anion levels and shipped to Texas A&M University for processing. Wheat bran and wheat germ showed concentration factors of 2.56X and 4.82X, respectively. Fluoride anion residues in other processed wheat commodities were less than or equal to those in the whole grain. Of the corn commodities, only aspirated grain fractions ("impurities") showed a concentration (5.49X) of fluoride residues. The processed cereal grain commodities were produced using simulated commercial practices.

Conclusions. The submitted data indicate that fluoride anion residues concentrate in wheat bran, wheat germ, and corn aspirated grain fractions, and that tolerances separate from those of the unprocessed grains may be appropriate for those commodities. For wheat bran and wheat germ, the tolerances derived from the direct fumigation of those commodities are sufficient to cover residue increases that result from the processing of treated grain. HED is recommending a tolerance for corn, aspirated grain fractions of 55 ppm based on the ~5.5X processing factor and the corn grain tolerance of 10 ppm. HED has used the tolerance value rather than the highest average residue as the basis for the recommended tolerance due to uncertainties in the dataset. Based on the available data, tolerances based on direct-treatment are sufficient to cover residue increases that may result from the processing of treated whole grains.

860.1850 Confined Accumulation in Rotational Crops

DER: None

The requested uses for sulfuryl fluoride are for post-harvest fumigation. There are no rotational crop issues associated with that use pattern and a discussion of the 860.1850 guideline is not germane to this petition.

860.1900 Field Accumulation in Rotational Crops

DER: None

The requested uses for sulfuryl fluoride are for post-harvest fumigation. There are no rotational crop issues associate with that use pattern and a discussion of the 860.1900 guideline is not germane to this petition.

860.1550 Proposed Tolerances

Due to the simplicity of the sulfuryl fluoride molecule and its breakdown products, the Metabolism Assessment Review Committee has not been consulted in determining the residues of concern for this chemical. Tolerances have been proposed for both sulfuryl fluoride and fluoride anion. These are the residues of toxicological concern. Temporary tolerances have been established under 40 CFR 180.575 (sulfuryl fluoride) and 180.145 (fluoride anion) in order to support an experimental use permit for sulfuryl fluoride fumigation of walnuts and raisins. The petitioner has requested that 40 CFR parts 180.575 and 180.145 be amended as indicated in Table 5. In many cases, HED has recommended higher tolerances than those proposed by the petitioner. HED's tolerance recommendations are also in Table 5. There are no international harmonization issues associated with this petition.

Table 5. Tolerance Summary for Sulfuryl Fluoride			
Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Sulfuryl Fluoride			
Barley, bran	None	0.05	Translated from wheat, flour
Barley, flour	None	0.05	Translated from wheat, flour
Barley, grain	0.01	0.10	Translated from wheat, grain
Barley, pearled	None	0.05	Translated from wheat, flour
Corn, aspirated grain fractions	None	0.05	Translated from wheat, flour
Corn, field, flour	0.01	0.01	–
Corn, field, grain	0.04	0.05	–
Corn, field, grits	0.01	0.02	–
Corn, field, meal	0.01	0.01	–
Corn, field, refined oil	9	None	Recommend use restriction on corn, oil.
Corn, pop, grain	0.04	0.05	–
Millet, grain	0.05	0.10	Translated from wheat, grain
Oat, flour	0.08	0.05	Translated from wheat, flour
Oat, grain	0.01	0.10	Translated from wheat, grain
Oat, rolled	0.08	0.10	Translated from wheat, grain
Rice, bran	0.01	0.01	–
Rice, brown	0.01	None	Covered by rice, grain
Rice, grain	0.04	0.05	–
Rice, hulls	0.08	0.10	–
Rice, polished	0.01	0.01	–
Rice, wild, grain	0.05	0.05	–
Sorghum, grain	0.05	0.10	Translated from wheat, grain
Triticale, grain	0.05	0.10	Translated from wheat, grain
Wheat, bran	0.01	0.05	Translated from wheat, flour
Wheat, flour	0.03	0.05	–
Wheat, germ	0.01	0.02	–
Wheat, grain	0.05	0.10	–
Wheat, milled by-products	0.01	0.05	Translated from wheat, flour
Wheat, shorts	0.01	0.05	Translated from wheat, flour
Nut, tree, group 14	6	3.0	–

Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Fruit, dried	—	0.05	A dried fruit group tolerance was not proposed. Tolerances for "fruit, dried" should be proposed and the individual listings omitted.
Dates	0.03	See Fruit, dried	–
Figs	0.05	See Fruit, dried	–
Plums, dried	0.01	See Fruit, dried	–
Raisins	0.01	See Fruit, dried	–
All other dried fruits	0.05	See Fruit, dried	–
Fluoride			
Barley, bran	98	45.0	–
Barley, flour	98	45.0	–
Barley, grain	10	15.0	–
Barley, pearled	98	45.0	–
Corn, aspirated grain fractions	98	55.0	–
Corn, field, flour	26	35.0	Translated from 24 ppm at 1012 mg·hr/L
Corn, field, grain	7	10.0	–
Corn, field, grits	10	10.0	–
Corn, field, meal	28	30.0	Translated from 21 ppm at 1012 mg·hr/L
Corn, field, refined oil	3	None	Recommend use restriction on corn, oil.
Corn, pop, grain	7	10.0	–
Millet, grain	24	40.0	–
Oat, flour	98	75.0	–
Oat, grain	17	25.0	–
Oat, rolled	98	75.0	–
Rice, bran	31	30.0	Translated from 11.8 ppm at 1012 mg·hr/L
Rice, brown	14	20.0	–
Rice, grain	10	12.0	–
Rice, hulls	35	35.0	–
Rice, polished	18	25.0	–
Rice, wild, grain	24	12.0	–
Sorghum, grain	24	40.0	–
Triticale, grain	24	40.0	–
Wheat, bran	40	40.0	–
Wheat, flour	10	125.0	–

Commodity	Proposed Tolerance (ppm)	Recommended Tolerance (ppm)	Comments (correct commodity definition)
Wheat, germ	98	130.0	Translated from 89.7 ppm at 1012 mg·hr/L
Wheat, grain	25	40.0	–
Wheat, milled by-products	98	130.0	Translated from wheat, germ
Wheat, shorts	38	40.0	–
Nut, tree, group 14	30	10.0	–
Fruit, dried, except grape, raisins	—	3.0	A dried fruit group tolerance was not proposed. Tolerances for "fruit, dried" should be proposed and the individual listings omitted.
Dates	5	See Fruit, dried	–
Figs	5	See Fruit, dried	–
Plums, dried	5	See Fruit, dried	–
Raisins	5	7.0	**Grape, raisin** - This tolerance is higher than other dried fruits because of the potential for fluoride residues from cryolite on this commodity.
All other dried fruits	5	See Fruit, dried	–

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