(8)

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.

DP Number:	309015	Case No.:	294172
PC Code:	078003	Submission:	S597868
40 CFR 180.	575	MRID Nos.:	45396301, 45510302, 45510302, 45510303, 45510304, 45603901, 45632902

From: Michael Doherty, Chemist
Registration Action Branch 2
Health Effects Division (7509C)

Through: Richard Loranger, Branch Senior Scientist
Registration Action Branch 2
Health Effects Division (7509C)

To: Dan Kenny/Meredith Laws
Insectice/Rodenticide Branch
Registration Division (7505C)

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

NOV 1 2004

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

Tolerances are currently est: olished under an experimental use permit for residues of sulfuryl fluoride in/on walnuts and : aisins (40 CFR 180.575) and for residues of inorganic fluoride resulting from the use of ei her sulfuryl fluoride or cryolite (40 CFR 180.145). Sulfuryl fluoride is highly reactive and break s down to form sulfate and fluoride anions. Parent sulfuryl fluoride and the fluoride anion are t e residues of concern for both the tolerance expression and risk assessment purposes.

Storage stability data were n ot 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 ve y short, storage stability data are not needed for sulfuryl fluoride, per se. Storage stability da a 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 da . It is unclear whether this reflects a true dissipation of fluoride from the samples or an inci ase in "bound" residues. Background residues of fluoride in the control samples for all commod ies 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 cont ol samples relates to residues in treated commodities or to the regulation of fluoride anion is u clear 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 meth od that uses headspace analysis. Based on validation data, the limit of detection (LOD) is 0.00 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-s lective electrode with the double-known-addition technique for quantifying residues. The petiti ner initially reported that the LOD and LOQ for the fluoride method are 0.2 and 0.5 ppm, respec ively. Following the independent laboratory validation, the LOQ for the fluoride method was in creased to 2 ppm. The petitioner has <u>not</u> demonstrated that either method is capable of extracti g 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 oth methods and (2) OPP accept the analytical methods without a laboratory validation base l 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 i nvestigating the effects of various fumigation parameters on sulfuryl fluoride and/or fluoride ani n 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 whe those parameters appear to have little effect on residue levels.

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

(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 se rate is marginally adequate. Residue data for both sulfuryl fluoride and luoride anion should be submitted. The data should be from samples in at least th ee different grain mills that were treated according to the proposed maximum se. The matrices to be analyzed should include raw and processed commodit es of wheat, rice, sorghum, and corn, including corn oil.

- The number of dried ut and tree fruit magnitude of the residue studies conducted at the maximum pro osed use rate is marginally adequate. Residue data for both sulfuryl fluoride and luoride anion should be submitted for representative commodities for thes two groups. Samples should be treated according to the proposed maximum se.
- Both HED and the A alytical Chemistry Branch are concerned about the ability of the analytical metho to extract incurred residues. Data showing the ability of the sulfuryl fluoride a nd fluoride anion methods to extract and accurately quantify incurred residues in r w and processed cereal grain matrices should be submitted. Furthermore, HED is requesting that the sulfuryl fluoride method be validated for corn oil. If the curre t method proves to be acceptable for corn oil, then the requested label restri tion (see below) can be lifted and HED will recommend that a tolerance be establi hed for corn oil.
- The sulfuryl fluoride nethod has not been shown to be specific to sulfuryl fluoride. An interfer nce study for sulfuryl fluoride should be submitted.
- Cereal grain commo ities, including aspirated grain fractions, are significant livestock feed items. Feeding studies were not submitted to determine the extent of secondary residue that may occur in livestock commodities. HED is requesting data show ng the transfer of fluoride from feedstuffs into livestock commodities. A feed ng study is not being requested for sulfuryl fluoride.
- A revised Section F (roposed Tolerances) is required.

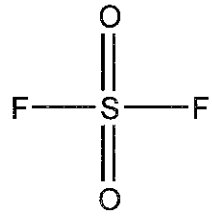
HED notes that data are sufficient to set sulfuryl fluoride and fluoride anion tolerances provided that the following modifications are nade to the label:

- The total fumigation ate shall not exceed 1500 mg·hr/L at ambient pressure or 200 mg·hr/L under re luced pressure.
- Active aeration of at east 24 hours at not less than 1 chamber volume/min shall occur for all commo ities prior to their entering commerce. This requirement does not apply to mill fum gations since practices to ensure worker safety will result in adequate aeration.
- Corn oil shall be rem ved from the premises prior to fumigation.

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

Background

Dow AgroSciences has requ sted permanent tolerances for residues of sulfuryl fluoride and fluoride anion esulting from the post-harvest fumigation of stored cereal grains, cereal grain rocessed 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 studi s have not been submitted for sulfuryl fluoride. Following application, sulfuryl fluoride breal s down to form sulfate and fluoride anion. Sulfate is not of toxicological concern. Fluoride ar ion is of toxicological concern due to potential skeletal fluorosis. The residues of concerr for both tolerance enforcement and risk assessment purposes are sulfuryl fluoride and fluoride a ıion. The lack of nature of the residue studies is not considered to be a deficiency and ı o 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 flu ıride would be expected to hydrolyze to form sulfate and fluoride anions. The residues of c ncern in livestock are sulfuryl fluoride and the fluoride anion; however, due to the nature of the s ılfuryl fluoride molecule, HED believes it is unlikely that secondary residues of sulfuryl fluo ide will occur in livestock commodities. The lack of nature of the residue studies is not consid ıred to be a deficiency and no further data are required to fulfill this guideline.

860.1340 Residue Analytical Me hods

DER: M. Doherty, 1/13/04, MRID 45(32902 (Sulfuryl Fluoride; 45632902.der.wpd)
M. Doherty, 1/13/04, MRID 45(03901 (Fluoride anion; 45603901.der.wpd)

Sulfuryl Fluoride. The pro)osed method utilizes headspace analysis to quantitate residues of sulfuryl fluoride. Resi ues of sulfuryl fluoride are extracted with water in an air-tight blender. The samples are agitated n the blender for five minutes to transfer residues of the highly volatile sulfuryl fluoride fr n the grain commodity to the headspace above the sample. The headspace is then analyzed fo residues of sulfuryl fluoride by injecting an aliquot into a gas chromatograph with electron-capt re 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. 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 rang d from 0.00099 to 0.00129 ppm, resulting in calculated limits of detection and quantification of a pproximately 0.003 and 0.010 ppm, respectively. There were no apparent residues of sulfuryl flu oride in the control samples. Due to the gaseous nature of sulfuryl fluoride, fortifications wei e made to the headspace above the commodities and not to the commodities themselves. The me iod has not been radiovalidated and it is unclear if the method will successfully extract incurred r sidues. The method has not been shown to be specific to sulfuryl fluoride. It is likely that th e 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 455103 2 (45510302.der.wpd)

Sulfuryl Fluoride. Storage s ability data for sulfuryl fluoride were not submitted. Sulfuryl fluoride analysis of field tri l samples was completed very shortly following the fumigation/aeration of the grain con modities. 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 homogen zed and fortified with an aqueous fluoride solution to a concentration of 20 ppm fluoride. T ne 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 sampl , a reagent blank, and two daily concurrent recovery samples, were analyzed for fluoride nion. Fluoride residues were extracted using an aqueous solution and analyzed using a fluori e ion selective electrode. The reported LOQ for the method is 0.6 ppm for wheat grain, 0.3 ppm or wheat flour, and 0.5 ppm for corn grain. LOQs for other matrices were not given.

Residues of fluoride anion w re 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 throug out the study, with only 55% remaining at 140 days. Regression of the residue data indic tes a loss of approximately 0.3% per day. The study design does not permit determination of wh ther or not the loss reflects dissipation of the fluoride residues or an increase in "bound" r sidues. This loss should be taken into account when interpreting the results of residue stu lies in wheat flour. HED will not assume that fluoride anion is stable in all commodities. F urthermore, HED notes that the residues in the control samples for all matrices indicate a l ss of fluoride with time and that this loss appears to be of the same order of magnitude as that obs rved in wheat flour. The significance of this trend as it relates to storage stability and field t ial results is unclear at this time.

Conclusions. The storage st bility data support the storage intervals and conditions for samples from the field trial and proc ssing studies.

860.1400 Water, Fish, and Irrigat d Crops

DER: None

There are no proposed uses f r 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 l esidues in Cereal Grains. Residue data have been pooled across loading factor and aeration time. The proposed ıse pattern is a maximum of 2 fumigations, with a total rate not to exceed 1500 mg·hr/L, and a 24-hour aeration per od. Data from rates less than ~1000 or greater than ~1800 mg·hr/L or from more than 2 fumigations have not b en 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 < OQ in all matrices except corn oil (2.6 ppm maximum) and wheat grain (0.04 ppm maximum; 1) °C only). Quantifiable residue of sulfuryl fluoride persisted in these two commodities even afte 7 days of aeration (wheat grain treated at 10 °C only).

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

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

MRID 45510303. Controlle l laboratory and operational grain mill fumigations using sulfuryl fluoride were conducted wi h whole cereal grains (hard red winter wheat, soft red winter wheat, durum wheat, medium grain ɔrown rice, medium grain white rice, white field corn, popcorn, barley, and oats) and repre entative processed commodities (wheat germ, wheat flour, and corn meal; grain mill fumigatio s only) in order to determine the magnitude of sulfuryl fluoride and fluoride anion residues hat occur following treatment with sulfuryl fluoride. In the laboratory, commodities were fumi ated for 24 hours at either 200 mg·hr/L or 1500 mg·hr/L and then aerated for 24 hours prior to an ılysis. 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 sulfu yl fluoride were determined using GC/ECD headspace analysis (laboratory studies only). S amples of the laboratory- and the grain mill-treated commodities were analyzed for fluc ide anion using aqueous extraction and fluoride selective-ion electrode. Sulfuryl fluoride ana ysis was completed immediately after the 24-hour aeration period.

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

Residues of fluoride anion v ere dependent upon the sulfuryl fluoride treatment rate. Residues in whole grains from the l boratory fumigations ranged from <0.5 ppm to 2 ppm at the 200-mg·hr/L rate and from 1 ppm t 8 ppm at the 1500-mg·hr/L rate. Residues in/on grains from the mill fumigations ranged from 1 ɔpm 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 gra ns and ranged from 7 ppm to 50 ppm at the lower rate and from 11 ppm to 90 ppm at the highe · 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 Da a from Crop Fumigation Trials with Sulfuryl Fluoride.

Treatment ID - Crop	Fumigation Number	Fum gation Rate, ng·hr/L (cun ulative rate)	Aeration Time, days	Residue Levels*				
				n	Min.	Max.	Mean	Std. Dev.
B - Dates	5	1491	1	4	13.00	23.00	16.75	4.35
B - Dates	5	(7475)	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	1	4	11.00	15.00	13.00	1.63
B - Figs	2	(2960)	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	1	3	4146.00	5532.00	4906.00	702.65
B - Pecans	2	(2985)	15	4	12.00	16.00	13.75	1.71
B - Pecans	3	1510	1	4	4276.00	6030.00	4950.50	842.09
B - Pecans	3	(4495)	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	1	4	51.00	70.00	63.25	8.73
B - Pistachios	2	(3024)	5	4	1.05	1.05	1.05	0.00
B - Pistachios	3	1506	1	4	35.00	56.00	44.75	10.81
B - Pistachios	3	(4530)	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 r ½ LOQ values were used as follows: sulfuryl fluoride ½ LOD = 1.05 ppb, ½ LOQ = 2.1 ppb; fluoride anion ½ L)D = 0.7 ppm, ½ LOQ = 1.2 ppm.

Supervised post harvest fum gation trials were conducted with walnuts, pistachios, pecans, almonds, dates, figs, dried p ums, and raisins. Single fumigations were carried out at normal atmospheric pressure or und r 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 combinatio ., 2 replicate fumigations were made and 2-3 samples were collected from each replicate. The t ials monitored residues of sulfuryl fluoride and fluoride anion, as well as the time required f r sulfuryl fluoride residues to dissipate to < LOQ (4.2 ng/g). Sulfuryl fluoride residues were anal zed using a head space method; fluoride anion was analyzed using a fluoride selective-ion electr de and a double-known-addition technique. Residue levels of sulfuryl fluoride varied based on he commodity and the treatment regime. For most commodities, residues had dissipate l 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 conten (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 vac um fumigation versus treatment at ambient pressure. In oily commodities, multiple fumigations esulted in higher residues of sulfuryl fluoride at a given aeration time. Pooled across all of t e variables addressed in this study, sulfuryl fluoride residues ranged from <2.1 ppb to 6030 ppb (.03 ppm). Residue levels of fluoride were measured only after residues of sulfuryl fluoride ha l dissipated to below detectable levels; therefore, the effect of aeration time on fluoride levels c nnot be assessed from these data. Generally, fluoride residues appear to be more depende t on the number of fumigations than on the treatment rate, treatment pressure, or commodity. ( )verall, fluoride residue levels ranged from <1.4 ppm to 21.8 ppm.

Conclusions. Significant da a gaps are associated with these studies. The majority of the maximum fluoride residue levels w e found in samples from the grain mill fumigations. Replication within those studies wa low and there is a fairly high degree of variation in the residue results. Furthermore, labora ory studies show that fluoride levels accumulate with successive fumigations and the grai mill samples were subjected to only one fumigation. HED is requesting that the petitioner conc ct fumigations in at least 3 different grain mills. The studies should include raw and proc ssed 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, t e submitted residue data are considered sufficient for granting a conditional registration f sulfuryl fluoride and for establishing sulfuryl fluoride and fluoride anion tolerances. Provided he label is changed such that (1) the total fumigation rate does not exceed 1500 mg·hr/L unde ambient conditions or 200 mg·hr/L under reduced pressure conditions, and (2) corn oil must be emoved from the premises prior to fumigation, the submitted data are sufficient to set s lfuryl fluoride and fluoride anion tolerances for stored cereal grain commodities, dried frui s, and tree nuts.

To determine appropriate tol rance 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 Accumulatio ı in Rotational Crops

DER: None

The requested uses for sulf ıryl fluoride are for post-harvest fumigation. There are no rotational crop issues associated w th 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 sulf ıryl fluoride are for post-harvest fumigation. There are no rotational crop issues associate wi ı 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 C ɔmmittee has not been consulted in determining the residues of concern for this chemical. Tole ances have been proposed for both sulfuryl fluoride and fluoride anion. These are the resid ıes 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 permi for sulfuryl fluoride fumigation of walnuts and raisins. The petitioner has requested that 40 CF R parts 180.575 and 180.145 be amended as indicated in Table 5. In many cases, HED has ı ecommended higher tolerances than those proposed by the petitioner. HED's tolerance recom nendations are also in Table 5. There are no international harmonization issues associated wi h 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	–

cc: M. Doherty, RAB2 Reading File

Sulfuryl Fluoride/078003
DACO 7.4.1/OPPTS 860.1500/OECD IIA 6.3.1, 6.3.2, 6.3.3 and IIIA 8.3.1, 8.3.2, 8.3.3
Crop Field Trial - Sulfuryl Fluoride and Fluoride in Cereal Grains

Primary Evaluator: Michael A. Doherty, Ph.D.
Chemist
Registration Action Branch 2, HED (7509C)
Date: 10/14/04

Approved by: Richard A. Loranger
Richard A. Loranger, Ph.D.
Branch Senior Scientist
Registration Action Branch 2, HED (7509C)
Date: 10/14/04

NOTE: This DER corrects the residues reported in the original DER for corn grits. The study volume reported residues of sulfuryl fluoride in units of parts per billion (ppb). The original DER reported the maximum residue in corn grits as 14.4 ppm rather than 14.4 ppb (i.e., 0.0144 ppm).

STUDY REPORTS:

45396301. Rick, D., Marty, G., Krieger, S. McGuirk, R. (2000) Evaluation of Sulfuryl Fluoride Fumigation Variables on Residue Levels in Crop Commodities: Laboratory Identification Number: 001103. Unpublished study prepared by Dow AgroSciences LLC 108 p.

EXECUTIVE SUMMARY:

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) 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 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, fumigatio temperature, and the number of fumigations. Quantifiable residues of fluoride anion were fou d in all matrices except corn oil (<0.5 ppm), further demonstrating the stability of paren sulfuryl fluoride in that matrix.

STUDY/WAIVER ACCEPTABI ITY/DEFICIENCIES/CLARIFICATIONS:

Under the conditions and pa ameters used in the study, the field trial residue data are classified as scientifically acceptabl . A large amount of data was submitted to characterize the effects of the fumigation parameter on residue levels. However, within a given set of parameters (i.e., a use pattern), ther is very little replication. Pooling data across parameters that do not significantly affect residue le vels can be done to increase the number of replicates.

The acceptability of this stu y for regulatory purposes is addressed in the forthcoming U.S. EPA Residue Chemistry Sumn ary Document (DP Barcode D283007).

COMPLIANCE:

Signed and dated GLP, qual y assurance, and data confidentiality information were provided. There is no claim of conf dentiality and all phases of the study followed Good Laboratory Practice standards.

A. BACKGROUND INFORM ATION

TABLE A.1. Test Compound Nom nclature

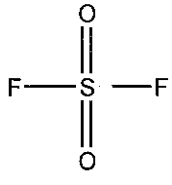
Chemical Structure		F^-
Common name	Sulfuryl f oride	Fluoride
Company experimental name	Sulfuryl f oride	-none-
IUPAC name		Fluoride
CAS name		Fluoride
CAS #	002699-7 -8	16984-48-8
End-use product/EP	Profume; Vikane	Profume

TABLE A.2. Physicochemical Prop rties of the Technical Grade Test Compound. Properties for fluoride were not available.

Parameter	Value	Reference
Melting point/range	-136°C	Vikane MSDS
pH	Not Provided	

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound. Properties for fluoride were not available.

Parameter	Value	Reference
Density	Not Provided	
Water solubility	1.67 g/L	Vikane MSDS
Solvent solubility	Not Provided	
Vapor pressure (10°C)	9150 mm Hg	Vikane MSDS
Dissociation constant (pK_a)	Not Provided	
Octanol/water partition coefficient Log(K_{OW})	Not Provided	
UV/visible absorption spectrum	Not Provided	

B. EXPERIMENTAL DESIGN

B.1. Study Site Information

The fumigations of the grain commodities with sulfuryl fluoride were conducted under laboratory conditions in a series of eight experiments:

Experiment 1- Evaluate the influence of fumigation, sample collection location, and time on fluoride residue levels in the tested commodities. Dosage = 1500 mg•hr/L. Analytes = sulfuryl fluoride and fluoride anion.

Experiment 2- Evaluate the influence of chamber loading on fluoride resiude levels in the tested commodities. Dosage = 1500 mg•hr/L. Chambers loaded to 1, 50, and 80% of chamber capacity. Analyte = fluoride anion.

Experiment 3- Evaluate the influence of dose on commodity residue levels. Dosages = 250 mg•hr/L, 1000 mg•hr/L, and 2500 mg•hr/L. Analytes = sulfuryl fluoride and fluoride anion.

Experiment 4- Evaluate the influence of temperature on commodity residue levels. Dosage = 1500 mg•hr/L. Fumigation temperatures = 10 or 30°C. Analytes = sulfuryl fluoride and fluoride anion.

Experiment 5- Evaluate the influence of repeat fumigations on commodity residue levels. Dosage = 1500 mg•hr/L. 4 fumigations, each at a 7-day interval. Analytes = sulfuryl fluoride and fluoride anion.

Experiment 6- Evaluate the influence of vacuum fumigation on commodity residue levels. Dosage = 1500 mg•hr/L. Each commodity was vacuum-fumigated. Analyte = fluoride anion.

Experiment 7- Evaluate the influence of sulfuryl fluoride concentration and exposure time on commodity fluoride residue levels. Dosage = 1500 mg•hr/L. Analyte = fluoride anion.

Experiment 8- Evaluate the influence of processing on processed commodity residue fluoride levels. Dosage = 1500 mg•hr/L. Analyte = fluoride anion. Data from Experiment 8 are addressed in a separate DER for processed commodities (45396301.de1.wpd).

Table B.1.1. Crop, Fumigation, and Sampling Inf rmation

Crop	Matrices	Fu igation[a]				Sampling Procedures			
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L)[b]	Loading Factor (%)	Interval (days)[c]	Location		Number and wt. of samples
							SF	Fluoride	
Experiment 1									
Corn	grain, flour, cornstarch, grits, meal, and oil	22 ± 2.5	1	1500	10	1	Middle	NA	Two, 10-25 g
						4	Middle	Top, Mid, Bottom	
						7[d]	Middle	Middle	
Wheat	grain, bran, flour, shorts, red dog, and germ	22 ± 2.5	1	1500	10	1	Middle	NA	Two, 10-25 g
						4	Middle	Top, Mid, Bottom	
						7[d]	Middle	Middle	
Rice	grain, hulls, polished	22 ± 2.5	1	1500	10	1	Middle	NA	Two, 10-25 g
						4	Middle	Top, Mid, Bottom	
						7[d]	Middle	Middle	
Experiment 2									
Wheat	grain, flour, and germ	22 ± 2.5	1	1500	1	7	NA	Middle	Two, 10-25 g
					50				
					80				
Experiment 3									
Wheat	grain, flour, and germ	22 ± 2.5	1	250	10	1, 4, 7	Middle	Middle[e]	Two, 10-25 g
Wheat	grain, flour, and germ	22 ± 2.5	1	1000	10	1, 4, 7	Middle	Middle[e]	Two, 10-25 g
				2500					
Experiment 4									
Wheat	grain, flour, and germ	10	1	1500	10	1, 4, 7	Middle	Middle[e]	Two, 10-25 g
		30							
Experiment 5									
Wheat	grain, flour, and germ	22 ± 2.5	4[f]	1500	10	1, 4, 7[d]	Middle	Middle[e]	Two, 10-25 g
Experiment 6[g]									
Wheat	grain, flour, and germ	22 ± 2.5	1	1500	10	7	NA	Middle	Two, 10-25 g
Experiment 7									
Wheat	grain, flour, and germ	22 ± 2.5	1	1500	10	7	NA	Middle	Two, 10-25 g

[a] No tank mixes or adjuvants were used.
[b] 1 mg-hr/L = 1 oz-h/1000 cu ft.
[c] After fumigation, all samples were aerated fo 24 hours, which is designated Day 1.
[d] Commodities analyzed for sulfuryl fluoride (F) on day 7 only if SF concentrations in day 4 samples were >LOQ (>8ug/kg).
[e] Fluoride sampled at 7 day interval only.
[f] 7 day interval between fumigations.
[g] Fumigation in experiment 6 was conducted a near-vacuum conditions (~100 mm Hg). Fumigation in all other experiments was conducted at normal atmospheric pressure.

TABLE B.1.2. Trial Numbers and Geographical Locations

NAFTA Growing Region	Crop 1			Crop 2			Crop 3		
	Submitted	Requested		Submitted	Requested		Submitted	Requested	
		Canada	US		Canada	US		Canada	US
Not applicable to a post-harvest use pattern.									

B.2. Sample Handling and Preparation

Following the final fumigation for each of the above-described experiments, the fumigation chambers were aerated with ambient air at approximately 5-10 L/min for 24 hours. The end of the aeration period is designated as Day 1. As rapidly as possible following the aeration period, commodities were analyzed for sulfuryl fluoride for Experiments 1, 3, 4, and 5. Samples were stored at fumigation temperatures and subsequently analyzed for residues of sulfuryl fluoride on Day 4 and Day 7 (if quantifiable residues were found on Day 4). Samples were analyzed for residues of fluoride anion on Day 7 (plus Day 4 for Experiment 1).

B.3. Analytical Methodology

Sulfuryl Fluoride. Residues of sulfuryl fluoride in crop matrices are extracted with water by blending in sealed jars. An aliquot of headspace is removed and immediately injected into a GC equipped with an electron capture detector (ECD). The GC/ECD response is calibrated using an external standard. The reported LOQs for residues of sulfuryl fluoride are 0.008 ppm in/on various grain matrices and 0.02 ppm in/on rice hulls and wheat germ. This method has undergone successful review by the Agency's Analytical Chemistry Branch/BEAD, which has recommended that the method be radiovalidated and that OPP accept the method for tolerance-enforcement purposes.

Fluoride Anion. Residues of fluoride anion in crop matrices are extracted with equal volumes of water and Total Ionic Strength Adjusting Buffer (TISAB) by blending in a homogenizer. A sample of the aqueous layer is removed and centrifuged. A fluoride electrode and a single junction reference electrode are placed in the solution, which is stirred during measurement. During the course of measurement, aliquots of a fluoride standard are added to the solution in two separate steps to complete the double known addition. The reported LOQs for residues of fluoride anion are 0.6 ppm in/on various whole grain matrices, 0.3 ppm in/on corn and wheat flour, cornstarch, corn meal and grits, and rice bran, 0.8 ppm in/on rice hulls and wheat bran, germ, "red dog", and shorts, and 0.5 ppm in/on corn oil. This method has undergone successful review by the Agency's Analytical Chemistry Branch/BEAD, which has recommended that the method be radiovalidated and that OPP accept the method for tolerance-enforcement purposes.

C. RESULTS AND DISCUSSION

There are no recommendations in the OPPTS 860.1500 Guideline pertaining to the

number of trials that should be sub[illegible]nitted to support post-harvest uses of pesticides. The results from these studies reflect investiga[illegible]ions into a number of use-pattern variables and, when taken separately, represent very little rep[illegible]cation for a specific use pattern. Pooling data across treatment regimes is appropriate fo[illegible] calculating summary statistics (Tables C.4.1 and C.4.2).

Based on the submitted cor[illegible]current recovery data, the analytical method for sulfuryl fluoride is adequate for data collec[illegible]ion purposes. The analytical method for fluoride anion regularly produces concurrent reco[illegible]eries that are highly variable and often well in excess of 100%. Fluoride anion concentratio[illegible]ns in untreated samples were below the LOQ for all commodities; therefore, backgrour[illegible]l levels of fluoride do not account for the excessive recoveries of fluoride anion. Despite the high[illegible] recoveries, the fluoride anion analytical method is adequate for data collection purposes due to[illegible]he inherent variability of ion-specific electrodes.

Sulfuryl Fluoride. Residue[illegible] of sulfuryl fluoride in cereal grains appear to be independent of the fumigation dose and chambe[illegible] loading factor for the doses and factors included in the residue studies. The independence[illegible] of residues on fumigation dose is likely due to the prevalence of non-quantifiable levels of sulfur[illegible]l fluoride in the cereal grains. The independence on loading factor indicates that during the fum[illegible]gation process, the fumigant was evenly disbursed throughout the fumigation chambe[illegible]. After one day of aeration, a number of corn, rice, and wheat commodities had quantifiable leve[illegible]s of sulfuryl fluoride (Table 2.3.1). The highest residues were found in corn grits (14.4 ppm) and[illegible]corn oil (7.8 ppm). Maximum residues in other commodities after one day of aeration were <0.1[illegible] ppm. After four days of aeration, residues of sulfuryl fluoride were below the LOQ (0.0[illegible]3 or 0.02 ppm, depending on the commodity) in all tested commodities with the exceptions o[illegible]corn oil and wheat grain (10 °C only). Quantifiable residue of sulfuryl fluoride persisted in the[illegible]e commodities even after 7 days of aeration (wheat grain from the 10 °C study only).

Fluoride Anion. As with th[illegible]e sulfuryl fluoride residues, residues of fluoride in the cereal grain commodities were independe[illegible]nt of fumigation chamber loading. However, fluoride residues are dependent on fumigation dosin[illegible] level and appear to be positively correlated to fumigation temperature. Residues of fluoride[illegible]re independent of the aeration time, presumably due to the rapid conversion of labile sulfuryl[illegible]uoride residues into stable fluoride anion residues. Fluoride residues are highly dependent on th[illegible]e commodity. The highest residues occurred in wheat flour and germ (Table 2.3.2). The lowes[illegible] residues were found in corn oil (<0.5 ppm), which further emphasises the stability of parent s[illegible]lfuryl fluoride in that matrix.

Table C.1. Summary of Concurrent Analytic[illegible]l Method Validation.

Crop Matrix	Fortification Level (ppm)		Recoveries (%)	Mean Recovery ± SD	LOD (ppm)	LOQ (ppm)
Experiment 1 – Sulfuryl Fluoride						
Corn Grain	8.0-80.0	82, 117	[illegible]0, 73, 114, 105, 116, 111, 110, 104, 102, 124, 107, 86, 101	101 ± 16	NR	0.008
Corn Flour	8.0-80.0	66, 104	[illegible]5, 60, 100, 104, 122, 107, 104, 102, 94, 105, 92, 79, 66	91 ± 19	NR	0.008

Crop Matrix	Fortification Level (ppm)	Recoveries (%)	Mean Recovery ± SD	LOD (ppm)	LOQ (ppm)
Cornstarch	8.0-80.0	91, 90, 82, 82, 85, 88	86 ± 4	NR	0.008
Corn Grits	8.0-80.0	115, 104, 105, 91, 81, 87, 90, 85, 89, 86, 78, 84	91 ± 11	NR	0.008
Corn Oil	8.0-80.0	73, 76, 84, 83, 88, 96, 98, 92, 100, 75, 93, 96	88 ± 9	NR	0.008
Wheat Grain	8.0-80.0	80, 67, 87, 81, 95, 88	83 ± 10	NR	0.008
Wheat Flour	8.0-80.0	97, 90, 95, 87, 84, 86	90 ± 5	NR	0.008
Wheat Bran	8.0-80.0	108, 98, 95, 88, 79, 82	92 ± 11	NR	0.008
Wheat Germ	8.0-80.0	78, 61, 74, 86, 90, 90	80 ± 11	NR	0.02
Wheat Red Dog	8.0-80.0	85, 84, 94, 98, 91, 90	90 ± 5	NR	0.008
Wheat Shorts	8.0-80.0	86, 91, 74, 85, 83, 84	84 ± 6	NR	0.008
Polished Rice	8.0-80.0	100, 104, 89, 93, 97, 90, 84, 66, 61, 72, 76, 89, 91	86 ± 13	NR	0.008
Rice Bran	8.0-80.0	79, 88, 85, 79, 77, 76, 66, 82, 76, 90, 73, 92, 90, 77, 89, 76, 72, 74, 83	80 ± 7	NR	0.008
Experiments 3-5 – Sulfuryl Fluoride					
Wheat Grain	8.0-80.0	146, 111, 96, 96, 82, 115, 94, 84, 91, 96, 91, 89, 84, 109, 102, 88, 82	97 ± 16	NR	0.008
Wheat Flour	8.0-80.0	63, 73, 109, 94, 106, 84, 88, 78, 79, 87, 97, 106, 82, 72, 93, 92, 85, 99	88 ± 13	NR	0.008
Wheat Germ	20.0-200	122, 83, 93, 81, 75, 89, 81, 70, 76, 79, 80, 76, 94, 88, 75, 83	84 ± 12	NR	0.02
Experiment 1 – Fluoride Anion					
Corn Grain	0.5-50	179, 124, 116, 90, 98	121 ± 35	NR	0.6
Corn Flour	0.5-50	183, 139, 103, 76, 114	123 ± 41	NR	0.3
Cornstarch	0.5-50	111, 116, 103, 109	110 ± 5	NR	0.3
Corn Meal	0.5-50	139, 112, 101, 90	110 ± 21	NR	0.3
Corn Grits	0.5-50	136, 106, 91, 99	108 ± 20	NR	0.3
Corn Oil	0.5-50	122, 121, 111, 101, 97	110 ± 11	NR	0.5
Wheat Grain	0.5-50	109, 99, 112, 125	111 ± 11	NR	0.6
Wheat Bran	0.5-50	109, 95, 96	100 ± 8	NR	0.8
Wheat Flour	0.5-50	97, 107, 125	110 ± 13	NR	0.3
Wheat Germ	0.5-50	110, 101, 96	102 ± 7	NR	0.8
Wheat Red Dog	0.5-50	130, 141	136 ± 8	NR	0.8
Wheat Shorts	0.5-50	129, 126, 180	145 ± 30	NR	0.8
Wheat Middlings	0.5-50	129	129	NR	0.8
Wheat Impurities	0.5-50	100	100	NR	0.8
Polished Rice	0.5-50	163, 110	137 ± 37	NR	0.3
Paddy Rice	0.5-50	93, 106	100 ± 9	NR	0.3
Rice Bran	0.5-50	118, 113	116 ± 4	NR	0.3
Rice Hulls	0.5-50	101, 103	102 ± 1	NR	0.8
Experiments 2-7 – Fluoride Anion					

Crop Matrix	Fortification Level (ppm)	Recoveries (%)	Mean Recovery ± SD	LOD (ppm)	LOQ (ppm)
Wheat Grain	2	86, [illegible]29, 115, 127, 135, 106, 114, 121, 115, 12[illegible] 134, 127, 118, 115, 126, 115	119 ± 12	NR	0.6
Wheat Flour	20	11[illegible] 123, 95, 88, 78, 90, 115, 124, 89, 111, 12[illegible] 100, 98, 123, 100, 109	105 ± 15	NR	0.3
Wheat Germ	50	12[illegible] 124, 90, 129, 78, 75, 101, 104, 86, 124, 11[illegible] 109, 108, 98, 106, 101	104 ± 16	NR	0.8

Table C.2. Summary of Storage Conditions			
Matrix	RAC or Extract	Storage Temperature (°C)	Duration (days)
Cereal Grains	RAC	[illegible]; 10, 22, and 30 for Experiment 4	8 - 21

Table C.3. Residues of Sulfuryl Fluorid[illegible] and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluori[illegible]e.

Crop	Matrix	Fumigation[a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# [illegible]f ap[illegible]l.	Dose: CT Rate (mg-hr/L)[b]	Loading Factor (%)			
Experiment 1								
Corn	grain	22 ± 2.5	[illegible]	1500	10	1	0.0257, 0.020	NA
						4	<0.008, <0.008	2.25, 2.26
						7[c]	<0.008, <0.008	2.07, 2.13
	flour	22 ± 2.5	[illegible]	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	14.9, 15.4
						7[c]	<0.008, <0.008	18.9, 19.2
	cornstarch	22 ± 2.5	[illegible]	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	3.82, 5.35
						7[c]	NA	3.91, 5.30
	meal	22 ± 2.5	[illegible]	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	5.60, 6.30
						7[c]	NA	5.24, 6.14

Table C.3. Residues of Sulfuryl Fluoride and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluoride.

Crop	Matrix	Fumigation [a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L) [b]	Loading Factor (%)			
	grits	22 ± 2.5	1	1500	10	1	<0.008, 0.0144	NA
						4	<0.008, <0.008	9.09, 9.17
						7 [c]	NA	7.74, 8.26
	oil	22 ± 2.5	1	1500	10	1	7.840, 5.848	NA
						4	2.664, 2.511	<0.5, <0.5
						7 [c]	4.384, 3.128	<0.5, <0.5
Wheat	grain	22 ± 2.5	1	1500	10	1	0.009, <0.008	NA
						4	<0.008, <0.008	1.94, 1.92
						7 [c]	NA	1.84, 1.93
	bran	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	36.7, 37.1
						7 [c]	NA	34.0, 36.0
	flour	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	29.9, 29.2
						7 [c]	NA	32.2, 33.5
	shorts	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	35.5, 31.9
						7 [c]	NA	34.8, 34.5
	red dog	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	32.7, 31.7
						7 [c]	NA	33.3, 32.3
	germ	22 ± 2.5	1	1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	56.8, 54.2

Table C.3. Residues of Sulfuryl Fluoride nd Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluorid .

Crop	Matrix	umigation [a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# o app	Dose: CT Rate (mg-hr/L) [b]	Loading Factor (%)			
						7 [c]	NA	59.0, 52.0
Rice	Paddy rice grain	22 ± 2.5	1	1500	10	1	0.0251, 0.0159	NA
						4	<0.008, <0.008	5.49, 7.80
						7 [c]	NA	5.97, 8.46
	bran	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	28.5, 24.2
						7 [c]	NA	24.6, 26.3
	polished	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	1.47, 1.60
						7 [c]	NA	1.30, 1.40
	hulls	22 ± 2.5	1	1500	10	1	0.0573, 0.0563	NA
						4	<0.008, <0.008	32.8, 23.6
						7 [c]	<0.008, <0.008	30.3, 23.4
Experiment 2								
Wheat	grain	22 ± 2.5	1	1500	1	7	NA	1.95, 2.00
					50			1.78, 1.85
					80			1.47, 1.99
	flour	22 ± 2.5	1	1500	1	7		33.5, 37.8
					50			21.5, 26.4
					80			25.9, 25.7
	germ	22 ± 2.5	1	1500	1	7		43.5, 41.6
					50			84.1, 83.9
					80			66.3, 73.2
Experiment 3								

Table C.3. Residues of Sulfuryl Fluoride and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluoride.

Crop	Matrix	Fumigation[a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L)[b]	Loading Factor (%)			
Wheat	grain	22 ± 2.5	1	250	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	0.68, 0.65
				1000	10	1	0.00849, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	1.74, 1.52
				2500	10	1	0.0355, 0.0208	NA
						4	<0.008, <0.008	NA
						7	NA	2.03, 1.85
Wheat	flour	22 ± 2.5	1	250	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	6.88, 7.34
				1000	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	19.3, 21.1
				2500	10	1	0.00825, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	39.9, 42.8
Wheat	germ	22 ± 2.5	1	250	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7	NA	18.1, 17.1
				1000	10	1	<0.020, <0.020	NA

Table C.3. Residues of Sulfuryl Fluori e and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluo de.

Crop	Matrix	Fumigation [a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L) [b]	Loading Factor (%)			
						4	<0.020, <0.020	NA
						7	NA	58.6, 55.3
				2500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7	NA	95.8, 94.2
Experiment 4								
Wheat	grain	10		1500	10	1	0.0328, 0.0323	NA
						4	0.0435, 0.0423	NA
						7	0.0210, 0.0192	0.79, 0.91
		30		1500	10	1	0.0142, 0.0127	NA
						4	<0.008, <0.008	NA
						7	NA	2.79, 2.96
	flour	10		1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	15.7, 14.6
		30		1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7	NA	37.8, 33.4
	germ	10		1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7	NA	17.4, 18.6
		30		1500	10	1	<0.020, <0.020	NA

Table C.3. Residues of Sulfuryl Fluoride and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluoride.

Crop	Matrix	Fumigation[a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L)[b]	Loading Factor (%)			
						4	<0.020, <0.020	NA
						7	NA	72.3, 82.6
Experiment 5								
Wheat	grain	22 ± 2.5	1	1500	10	1	0.0108, 0.0902	NA
						4	<0.008, <0.008	NA
						7[c]	NA	3.10, 3.94
			2	1500	10	1	0.0953, 0.0860	NA
						4	<0.008, <0.008	NA
						7[c]	NA	5.02, 5.02
			3	1500	10	1	0.0288, 0.0225	NA
						4	<0.008, <0.008	NA
						7[c]	NA	6.15, 6.31
			4	1500	10	1	0.0214, 0.0232	NA
						4	<0.008, <0.008	NA
						7[c]	NA	7.95, 8.00
	flour	22 ± 2.5	1	1500	10	1	<0.008, <0.008	NA
						4	<0.008, <0.008	NA
						7[c]	NA	44.7, 34.2
			2	1500	10	1	0.00844, 0.00936	NA
						4	<0.008, <0.008	NA
						7[c]	NA	62.5, 62.6
			3	1500	10	1	0.00949, 0.008.52	NA

Table C.3. Residues of Sulfuryl Fluoride and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluoric .

Crop	Matrix	Fumigation [a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# c app.	Dose: CT Rate (mg-hr/L) [b]	Loading Factor (%)			
						4	<0.008, 0.00899	NA
						7 [c]	NA	101, 93.7
			4	1500	10	1	0.0129, 0.015.4	NA
						4	0.00837, <0.008	NA
						7 [c]	NA	97.1, 93.2
	germ	22 ± 2.5	1	1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7 [c]	NA	88.5, 81.9
			2	1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7 [c]	NA	158, 121
			3	1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7 [c]	NA	218, 201
			4	1500	10	1	<0.020, <0.020	NA
						4	<0.020, <0.020	NA
						7 [c]	NA	235, 222
Experiment 6 [Fumigation in experiment was conducted at near-vacuum conditions (~100 mm HG).]								
Wheat	grain	22 ± 2.5	1	1500	10	7	NA	3.20, 4.08
	flour						NA	33.3, 31.6
	germ						NA	53.7, 54.3
Experiment 7								
Wheat	grain	22 ± 2.5	1	1500	10	7	NA	3.93, 2.91
							NA	2.22, 2.36

Table C.3. Residues of Sulfuryl Fluoride and Fluoride Anion in Cereal Grain Commodities following Post-Harvest Fumigation with Sulfuryl Fluoride.

Crop	Matrix	Fumigation[a]				Sampling Interval (days)	Sulfuryl fluoride Residues (ppm)	Fluoride anion Residues (ppm)
		Temp. (C)	# of appl.	Dose: CT Rate (mg-hr/L)[b]	Loading Factor (%)			
	flour	22 ± 2.5	1	1500	10	7	NA	43.4, 39.9
							NA	28.2, 35.3
	germ	22 ± 2.5	1	1500	10	7	NA	104, 90.3
							NA	58.8, 60.1

[a] No tank mixes or adjuvants were used.

[b] 1 mg-hr/L = 1 oz-h/1000 cu ft.

[c] Commodities analyzed for sulfuryl fluoride on Day 7 only if sulfuryl fluoride concentration in Day 4 samples were > LOQ (>0.008 ppm).

Table C.4.1. Summary of Sulfuryl Fluoride Residues in Cereal Grains. Residue data have been pooled across dosage level and loading factor.

Crop	Commodity	Temp. (°C)	Fumigation. No.	Aeration (days)	n	Sulfuryl Fluoride Residues (ppm)			
						Min	Max	Mean	Std Dev
Corn	cornstarch	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	flour	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	grain	22	1	1	2	<0.020	0.026	0.023	0.004
Corn	grits	22	1	1	2	<0.008	0.014	0.011	0.005
Corn	meal	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	oil	22	1	1	2	5.848	7.840	6.844	1.409
Rice	bran	22	1	1	2	<0.008	<0.008	<0.008	0.000
Rice	hulls	22	1	1	2	0.056	0.057	0.057	0.001
Rice	Paddy rice grain	22	1	1	2	0.016	0.025	0.021	0.007
Rice	polished	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	bran	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	10	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	30	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	22	1	1	10	<0.008	<0.008	<0.008	0.000
Wheat	flour	22	2	1	2	<0.008	0.009	0.009	0.001
Wheat	flour	22	3	1	2	0.009	0.009	0.009	0.001
Wheat	flour	22	4	1	2	0.013	0.015	0.014	0.002
Wheat	germ	10	1	1	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	30	1	1	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	1	1	10	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	2	1	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	3	1	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	4	1	2	<0.020	<0.020	<0.020	0.000
Wheat	grain	10	1	1	2	0.032	0.033	0.033	0.000
Wheat	grain	30	1	1	2	0.013	0.014	0.013	0.001
Wheat	grain	22	1	1	10	<0.008	0.090	0.021	0.026
Wheat	grain	22	2	1	2	0.086	0.095	0.091	0.007
Wheat	grain	22	3	1	2	0.023	0.029	0.026	0.004
Wheat	grain	22	4	1	2	0.021	0.023	0.022	0.001

Crop	Commodity	Temp. (°C)	Fumigation. No.	Aeration (days)	n	Sulfuryl Fluoride Residues (ppm)			
						Min	Max	Mean	Std Dev
Wheat	red dog	22	1	1	2	<0.008	<0.008	<0.008	0.000
Wheat	shorts	22	1	1	2	<0.008	<0.008	<0.008	0.000
Corn	cornstarch	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	flour	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	grain	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	grits	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	meal	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	oil	22	1	4	2	2.511	2.664	2.588	0.108
Rice	bran	22	1	4	2	<0.008	<0.008	<0.008	0.000
Rice	hulls	22	1	4	2	<0.008	<0.008	<0.008	0.000
Rice	Paddy rice grain	22	1	4	2	<0.008	<0.008	<0.008	0.000
Rice	polished	22	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	bran	22	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	10	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	30	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	22	1	4	10	<0.008	<0.008	<0.008	0.000
Wheat	flour	22	2	4	2	<0.008	<0.008	<0.008	0.000
Wheat	flour	22	3	4	2	<0.008	0.009	<0.008	0.001
Wheat	flour	22	4	4	2	<0.008	<0.008	<0.008	0.000
Wheat	germ	10	1	4	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	30	1	4	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	1	4	10	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	2	4	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	3	4	2	<0.020	<0.020	<0.020	0.000
Wheat	germ	22	4	4	2	<0.020	<0.020	<0.020	0.000
Wheat	grain	10	1	4	2	0.042	0.044	0.043	0.001
Wheat	grain	30	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	grain	22	1	4	10	<0.008	<0.008	<0.008	0.000
Wheat	grain	22	2	4	2	<0.008	<0.008	<0.008	0.000
Wheat	grain	22	3	4	2	<0.008	<0.008	<0.008	0.000
Wheat	grain	22	4	4	2	<0.008	<0.008	<0.008	0.000
Wheat	red dog	22	1	4	2	<0.008	<0.008	<0.008	0.000
Wheat	shorts	22	1	4	2	<0.008	<0.008	<0.008	0.000
Corn	cornstarch	22	1	7	2	NA[A]	NA	NA	NA
Corn	flour	22	1	7	2	<0.008	<0.008	<0.008	0.000
Corn	grain	22	1	7	2	<0.008	<0.008	<0.008	0.000
Corn	grits	22	1	7	2	NA	NA	NA	NA
Corn	meal	22	1	7	2	NA	NA	NA	NA
Corn	oil	22	1	7	2	3.128	4.384	3.756	0.888
Rice	bran	22	1	7	2	NA	NA	NA	NA
Rice	hulls	22	1	7	2	<0.008	<0.008	<0.008	0.000
Rice	Paddy rice grain	22	1	7	2	NA	NA	NA	NA
Rice	polished	22	1	7	2	NA	NA	NA	NA
Wheat	bran	22	1	7	2	NA	NA	NA	NA
Wheat	flour	10	1	7	2	NA	NA	NA	NA
Wheat	flour	30	1	7	2	NA	NA	NA	NA
Wheat	flour	22	1	7	20	NA	NA	NA	NA
Wheat	flour	22	2	7	2	NA	NA	NA	NA
Wheat	flour	22	3	7	2	NA	NA	NA	NA
Wheat	flour	22	4	7	2	NA	NA	NA	NA
Wheat	germ	10	1	7	2	NA	NA	NA	NA

Crop	Commodity	Temp. (°C)	Fumigation. No.	Aeration (days)	n	Sulfuryl Fluoride Residues (ppm)			
						Min	Max	Mean	Std Dev
Wheat	germ	30	1	7	2	NA	NA	NA	NA
Wheat	germ	22	1	7	20	NA	NA	NA	NA
Wheat	germ	22	2	7	2	NA	NA	NA	NA
Wheat	germ	22	3	7	2	NA	NA	NA	NA
Wheat	germ	22	4	7	2	NA	NA	NA	NA
Wheat	grain	10	1	7	2	0.019	0.021	0.02	0.001
Wheat	grain	30	1	7	2	NA	NA	NA	NA
Wheat	grain	22	1	7	26	NA	NA	NA	NA
Wheat	grain	22	2	7	2	NA	NA	NA	NA
Wheat	grain	22	3	7	2	NA	NA	NA	NA
Wheat	grain	22	4	7	2	NA	NA	NA	NA
Wheat	red dog	22	1	7	2	NA	NA	NA	NA
Wheat	shorts	22	1	7	2	NA	NA	NA	NA

[A] NA = Not Analyzed.

Table C.4.2. Summary of Fluoride Anion Residues in Cereal Grains. Residue data have been pooled across loading factor and aeration time.

Crop	Commodity	Temp. (°C)	Fumigation No.	Dosage (mg-hr/L)	n	Fluoride Anion Residues (ppm)			
						Min	Max	Mean	SD
Corn	cornstarch	22	1	1500	4	3.82	5.35	4.60	0.84
Corn	flour	22	1	1500	4	14.90	19.20	17.10	2.26
Corn	grain	22	1	1500	4	2.07	2.26	2.18	0.09
Corn	grits	22	1	1500	4	7.74	9.17	8.57	0.69
Corn	meal	22	1	1500	4	5.24	6.30	5.82	0.49
Corn	oil	22	1	1500	4	<0.50	<0.50	<0.50	0.00
Rice	bran	22	1	1500	4	24.20	28.50	25.90	1.96
Rice	hulls	22	1	1500	4	23.40	32.80	27.53	4.76
Rice	Paddy rice grain	22	1	1500	4	5.49	8.46	6.93	1.43
Rice	polished	22	1	1500	4	1.30	1.60	1.44	0.13
Wheat	bran	22	1	1500	4	34.00	37.10	35.95	1.38
Wheat	flour	10	1	1500	2	14.60	15.70	15.15	0.78
Wheat	flour	30	1	1500	2	33.40	37.80	35.60	3.11
Wheat	flour	22	1	250	2	6.88	7.34	7.11	0.33
Wheat	flour	22	1	1000	2	19.30	21.10	20.20	1.27
Wheat	flour	22	1	1500	18	21.50	44.70	32.57	6.18
Wheat	flour	22	1	2500	2	39.90	42.80	41.35	2.05
Wheat	flour	22	2	1500	2	62.50	62.60	62.55	0.07
Wheat	flour	22	3	1500	2	93.70	101.00	97.35	5.16
Wheat	flour	22	4	1500	2	93.20	97.10	95.15	2.76
Wheat	germ	10	1	1500	2	17.40	18.60	18.00	0.85
Wheat	germ	30	1	1500	2	72.30	82.60	77.45	7.28
Wheat	germ	22	1	250	2	17.10	18.10	17.60	0.71
Wheat	germ	22	1	1000	2	55.30	58.60	56.95	2.33
Wheat	germ	22	1	1500	18	41.60	104.00	67.01	17.86
Wheat	germ	22	1	2500	2	94.20	95.80	95.00	1.13
Wheat	germ	22	2	1500	2	121.00	158.00	139.50	26.16
Wheat	germ	22	3	1500	2	201.00	218.00	209.50	12.02
Wheat	germ	22	4	1500	2	222.00	235.00	228.50	9.19
Wheat	grain	10	1	1500	2	0.79	0.91	0.85	0.08
Wheat	grain	30	1	1500	2	2.79	2.96	2.88	0.12

Crop	Commodity	Temp. (°C)	umigation No.	Dosage (mg-hr/L)	n	Fluoride Anion Residues (ppm)			
						Min	Max	Mean	SD
Wheat	grain	22	1	250	2	0.65	0.68	0.67	0.02
Wheat	grain	22	1	1000	2	1.52	1.74	1.63	0.16
Wheat	grain	22	1	1500	18	1.47	4.08	2.47	0.84
Wheat	grain	22	1	2500	2	1.85	2.03	1.94	0.13
Wheat	grain	22	2	1500	2	5.02	5.02	5.02	0.00
Wheat	grain	22	3	1500	2	6.15	6.31	6.23	0.11
Wheat	grain	22	4	1500	2	7.95	8.00	7.98	0.04
Wheat	red dog	22	1	1500	4	31.70	33.30	32.50	0.67
Wheat	shorts	22	1	1500	4	31.90	35.50	34.18	1.57

D. CONCLUSION

Data from these studies inc icate that residues of sulfuryl fluoride are not highly dependent upon the treatment rate or loading factor. This is likely due to the combined volatility and high reactivity of the parent c mpound. Residues of fluoride appear to be independent of the loading factor and the aeration tim , indicating that once the sulfuryl fluoride decomposes to form fluoride, that fluoride is rath recalcitrant. Following that line of reasoning, it is not surprising that fluoride content is highly dependent on the treatment rate and number of fumigations.

HED notes that MRID 455 0303 also addresses residue levels of fluoride in cereal grain commodities.

E. REFERENCES

Analytical Methods: DER 45603 01
Storage Stability: DER 45510302

F. DOCUMENT TRACKIN G

RDI: MADoherty (10/16/03); WD ew (10/28/03); RALoranger (10/27/03)
Petition Number(s): PP1F06312
PC Code: 078003

Template Version September 2003

13544

R103444

Chemical:	**Sulfuryl fluoride**
PC Code:	**078003**
HED File Code	**11000 Chemistry Reviews**
Memo Date:	**10/12/2004**
File ID:	**DPD309015**
Accession Number:	**412-05-0036**

HED Records Reference Center
11/02/2004