SULFURYL FLUORIDE (218)

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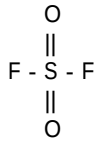
EXPLANATION

At the 36th session of the CCPR, sulfuryl fluoride was listed in the Evaluation for New Compounds for residue review by the 2005 JMPR. The manufacturer submitted data for sulfuryl fluoride in cereals, tree nuts and dried fruits, and submitted information on the physical and chemical properties, metabolism (plant and animal), environmental fate, analytical methods, stability in stored analytical samples, use pattern and good agricultural practices (GAP), supervised field trials, fate of residues in storage and in processing and national residue limits.

IDENTITY

ISO Common name:		Sulfuryl fluoride
Chemical name:		
	IUPAC	Sulfuryl fluoride
	CAS	Sulfuryl fluoride
CAS No:		002699-79-8
CIPAC Number:		757
Synonyms		SF
Structural formula:		
Molecular formula:		SO_2F_2
Molecular weight:		102.1

Physical and chemical properties

Pure active ingredient

Table 1. Physical and Chemical Properties

Properties	Measurement	Reference
Appearance	Colorless Gas	Comb, A.L. 2001.
Vapor pressure @ 25, 35 and 45°C	1.61 x 10^6 Pa @ 20 ºC	Krieger, M.S. 2001a
Volatility, Henry's Law Constant @ 20°C	Not Conducted - Gas	Krieger, M.S. 2001b
Melting point	-136.7 °C	McDonald, Hildenbrand. 1957
Partition coefficient		
K_{ow} @ 24.7 ± 1.4°C (pH 7), not dependent on pH.	K_{ow} = 0.14	Comb, A.L. 2001.
Water Solubility @ 20 °C ± 0.5 °C	1040 mg/L	Comb, A.L. 2001.
Solvent Solubility @ 24.7 ± 0.3 °C		Comb, A.L. 2001.
ethyl acetate, g/kg solvent	59	
acetone, g/kg solvent	71	
xylene, g/kg solvent	25	

methanol, g/kg solvent	33	
n-heptane, g/kg solvent	22	
1,2-dichloroethane, g/kg solvent	25	
Relative density @ 25 °C ± 0.5 °C	Not Determined: Gas	Not Applicable
Hydrolysis @ 24.9 ± 1.6 °C		Krieger, M.S. 2001d, Cady 1974
$DT_{50,}$ pH 4	281 days	
$DT_{50,}$ pH 7	6.7 hrs	
$DT_{50,}$ pH 9	4.0 minutes	
Photolysis (aqueous) @ 25 °C	Not determined (in water) due to the high vapor pressure and low water solubility of SF	Not Applicable
Dissociation constant	Not applicable – SF does not reversibly ionize	Not Applicable
Thermal stability	No decomposition or sublimation occurs at the melting or boiling point	Not Applicable

Technical material

Minimum purity: ≥ 99.8%
Appearance: Not applicable
Density: Not applicable
Melting point: -136.7 °C (McDonald, 1957)
Stability: Half-life in air ≤ 3.2 years (Kreiger, 2002)

Formulations

Tradename: ProFume®, Vikane®. ProFume and Vikane are both equivalent to the technical product. There are no formulations of SF that differ from the technical product.

METABOLISM AND ENVIRONMENTAL FATE

Animal metabolism

The Meeting received information on fluoride levels and distribution in eggs of laying hens that were orally dosed through drinking water (Report No. NAP03), and metabolism of fluoride in lactating and dry cows that were orally dosed through diet (Report No. H02). No study was provided on the direct administration of sulfuryl fluoride.

Fluoride residues were measured in laying hens of 40-weeks of age given increased fluoride intake in the drinking water/L for 4 weeks. Twenty-two laying hens were randomly divided into two groups of 11 each and they were all given the same diets. The first group was given water with sodium fluoride added (23.8 mmol/L) as a treated group, and the second group was given tap water (0.2 mmol/L F^-) as a control group. A total of 60 eggs were collected from the treated group and 42 from the control group and among those 24 and 22 eggs were set for hatching, respectively. Fluoride levels in eggs and bones of hens and bones of chicks were analyzed using an ion-selective electrode. Fluoride content in shells, yolks and albumens were compared in test and control groups of hens (Table 2).

Table 2. Mean of fluoride content in egg shell, yolks and albumen of hens (Based on Report N. NAP03).

Group	Parameter	Egg shell	Egg yolk	Egg albumen
Treated	Mean	22.84	14.12	10.67
	SD	2.56	2.42	2.36
Control	Mean	13.68	14.85	10.36
	SD	2.42	3.34	1.02

Mean = arithmetic mean
SD = standard deviation

Fluoride was measured in kidney and milk in three 500 kg lactating and dry cows, dosed orally with fluoride ion each day (between 37.7 and 60.3 mg/kg/day depending on stages of lactation) (Report No. H02). No study was provided on the direct administration of radiolabeled or unlabelled sulfuryl fluoride to these animals.

Table 3 shows the elimination of fluoride by the gastrointestinal tract, kidneys and mammary gland. The digestive tract excreted 78 to 93% of the absorbed fluoride ion. Excretion from the kidney and in milk ranged from 4.7 to 21% and 2.3 to 4.4%, respectively. This data indicates dietary fluoride ion is absorbed by and effectively excreted from cattle with only a small fraction of the total, 2 to 4%, being identified in milk.

Table 3. Quantity of fluoride eliminated during dry and lactation periods (%) (Based on Report No. H02).

Routes of elimination	Dry period	Stages of lactation		
		1	2	3
Mammary gland	–	4.4	2.6	2.3
Kidneys	21.3	12.1	6.6	4.7
Gastrointestinal tract	78.7	88.5	90.9	93.0
Total	100.0	100.0	100.0	100.0

Plant metabolism

The Meeting received information on the fate of sulfuryl fluoride after fumigation treatment on wheat flour (Meikle, 1964, Report J. Agric. Food Chem., 12:464-467), unbleached enriched wheat flour (Pillsbury), Kibbles ‘n Bits dog food (Ken-L Ration), non-fat dry milk (Carnation), vegetable cooking oil (Crisco), dried beef, acetaminophen (Extra-strength Tylenol, McNeilab), Red Delicious Washington apples and Twinkies snack cakes (Hostess, individually wrapped in cellophane) (Scheffrahn, 1989, Report J. Agric. Food Chem., 1989, 37.203-206).

Graham flour containing both bran and the germ was fumigated with radiolabeled sulfuryl-^{35}S fluoride, equivalent to 32 mg/L (2 pounds/1000 cubic feet) in a fumigation chamber under vacuum and reduced pressure for 92 hours at room temperature. Flour was then removed and aerated in a hood to a constant radioactivity count rate. The fumigated flour was extracted for 1 hour with 80% ethanol and the resulting flour residue was extracted a second time for 7 hours with 80% ethanol before being air-dried. The insoluble residue following the alcohol extraction was extracted with a solution of 5 % trichloroacetic acid (TCA). All radioactivity determinations were counted in dish planchets using a thin end window Geiger-Muller (GM) tube. Paper chromatograms were counted in increments using a special aluminium slide made to fit a Tricerlab SC9D shielding unit. Aqueous ethanol extract was treated with Dowex 50. The eluate and aqueous column washings were combined, and the combined aqueous solution from the Dowex 50 was treated with Dowex 3. The Dowex 3 resin was eluted with 2N ammonia, and was then eluted with 1N sodium hydroxide or 1N hydrochloric acid in an attempt to elute the residual radioactivity. The eluted Dowex 3 resin was analyzed for residual radioactivity with paper chromatography. The radioactive residues eluted with 2N Ammonia from the Dowex 3 resin

was analyzed via descending paper chromatography. Radiolabeled sulfate^{35}S fortified flour was analyzed through the separation scheme used for fumigated flour. Sulfuryl fluoride was degraded in phosphate buffered water.

As a result of successive fractionation steps applied to graham flour after fumigation with sulfuryl- ^{35}S fluoride, the radioactivity was found to be distributed as shown in Figure 2. The extractable radioactive residue (76% of the total radioactive residue, TRR) was quantitatively eluted from the Dowex 50 cation exchange resin and then subsequently completely retained on the Dowex 3 anion exchange resin. These results indicate that the residues are anionic in character, i.e., carboxylic acids, sulfate, etc. indicating that the predominant reaction may involve free amino groups of amino acids and protein. If there had been reactions with hydroxyl it would be expected to find some radioactivity in the neutral fraction. This was not the case. Upon elution of the Dowex 3 with 2N NH_3 15% of the TRR remained bound to the resin with 61% of the TRR eluting. A proposed explanation for the relative intractability of this tightly bound activity is the formation of N-(amino acid or protein)-N'-(resin) sulfamides via an *in situ* reaction between the sulfonyl derivatives of the amino acid (protein) and the free amino group of the resin. Acid hydrolysis liberated the bound radioactive residue from the Dowex 3 resin. Subsequent paper chromatography with ninhydrin development of the resulting spots, of the hydrolysates of Dowex 3 resin containing the radioactive residue and control hydrolysate from a separate Dowex 3 resin revealed the presents of amino acids only on the resin with the radioactive residue. This indicated that the bound radioactivity was associated with these amino acids as described earlier.

Paper chromatography analysis of the radioactive residue eluted from the Dowex 3 resin demonstrated the presence of three regions of radioactivity. The retention characteristic of one of the three regions was consistent with that of the sulfate-^{35}S standard. The remaining two regions of radioactivity were eluted from the paper and subjected to acid hydrolysis, clean-up, and subsequent paper chromatography with four different solvent systems. Development of the spots with ninhydrin revealed the presence of at least eight different amino acids, suggesting that the elutable radioactive residue was either free amino acids or soluble polypeptides. The correlation of radioactive residue and amino acids with the affinity of the radioactive residue for the Dowex 3 anion exchange resin supports the proposal that the sulfuryl fluoride reacts with free amino groups of the free amino acids, polypeptides and proteins.

Figure 1 shows that insoluble flour residues, after 80% ethanol extraction, retained 24% of the radioactivity. In addition, experiments with radioactive sulfate showed that the same extraction pattern resulted in the fumigated flour. Subsequent treatment of the flour-sulfate-^{35}S residue with 5% aqueous TCA solution completely dissolved the sulfate-^{35}S as the protein precipitated. However, in the case of the fumigated extracted flour residue, the TCA treatment dissolved very little radioactivity - this was retained in the precipitated protein fraction. Therefore the activity remaining in the insoluble residue fraction was not sulfate, but was in some way fixed to the protein fraction of the residue. Figure 2 shows the proposed scheme for the breakdown of sulfuryl fluoride in graham flour.

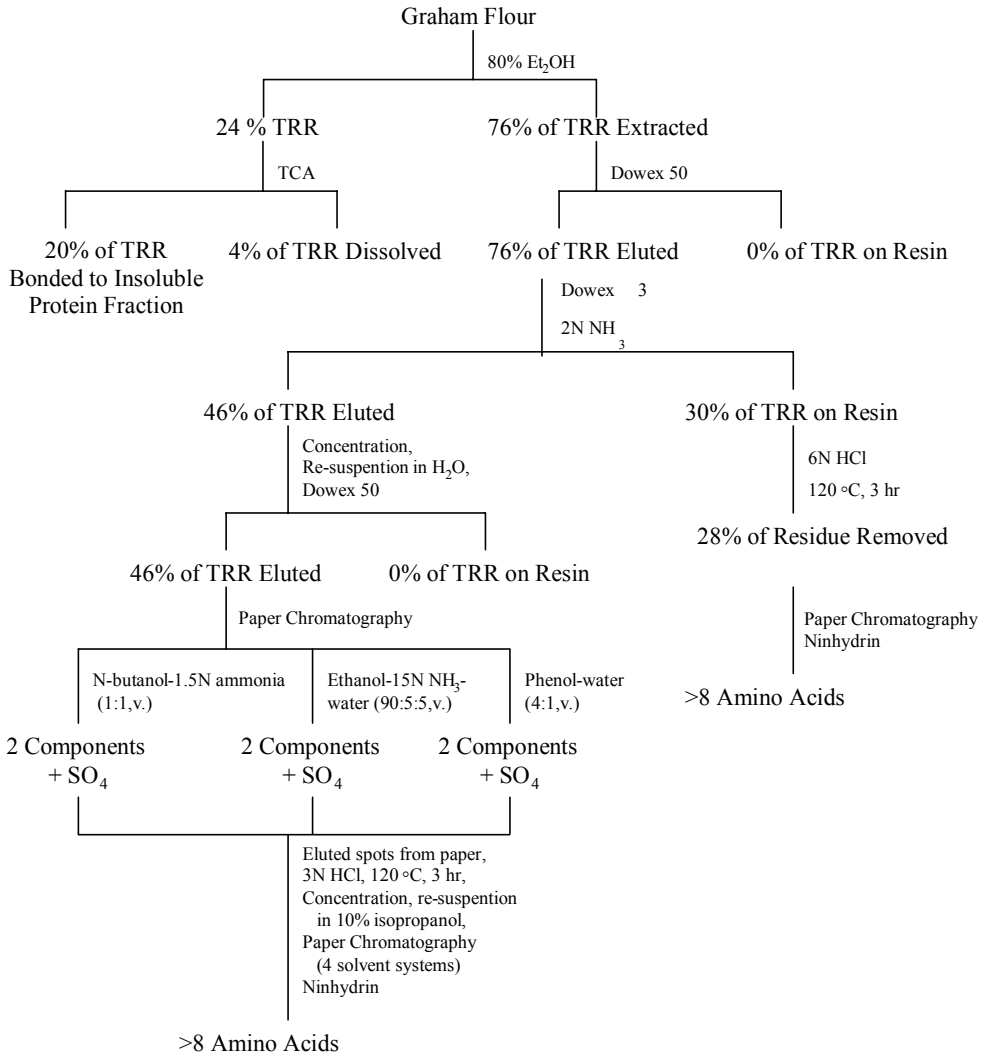


Figure 1. Fractionation of graham flour fumigated with sulfuryl S35 fluoride

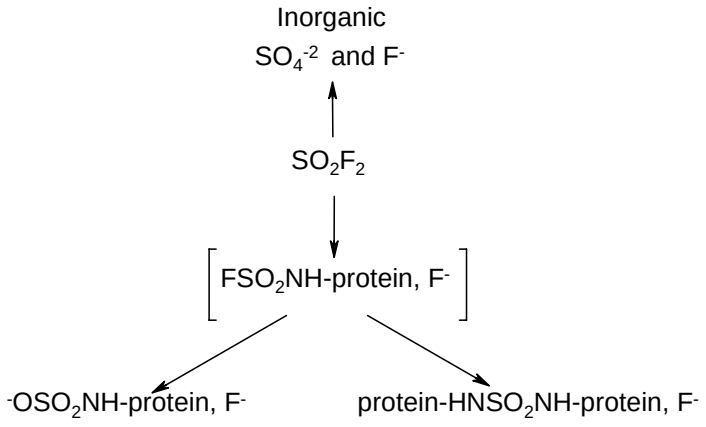


Figure 2. Proposed scheme for the breakdown of sulfuryl fluoride in graham flour

Sulfate is formed as a result of conventional hydrolysis of sulfuryl fluoride. This reaction proceeds stepwise, first to fluorosulfonic acid, then sulfate and it has been shown to be catalysed by phosphate. An additional product of the proposed scheme for the breakdown of sulfuryl fluoride is inorganic fluoride. The total fluoride residue in graham flour under normal fumigation procedures is about 15 ppm.

These results indicate that the fate of sulfuryl fluoride in graham flour is driven predominantly by nucleophilic reactions involving the free amino groups of amino acids and proteins. The products of this reaction would be the N-fluorosulfonyl derivatives, N,N'-disubstituted sulfamides, and N-substituted sulfamate. This strongly suggests that the reactivity of the intermediate, N-fluorosulfonyl functional group is sufficient to cause these derivatives to be ultimately converted in vivo to N,N'-disubstituted sulfamides and N-substituted sulfamic acids. The nucleophilic substitution of the fluoro group of the N-fluorosulfonyl intermediate must be reasonably facile. Sulfate is formed as a result of conventional hydrolysis of sulfuryl fluoride. This reaction proceeds stepwise, first to fluorosulfonic acid, then sulfate. An additional product of the proposed scheme for the breakdown of sulfuryl fluoride is inorganic fluoride.

Fluoride and sulfate residues in eight foods items fumigated with sulfuryl fluoride were studied in a 4.2 m^3 chamber (Scheffrahn, 1989, J. Agric. Food Chem. 37:203 206). The food items include unbleached enriched wheat flour (Pillsbury), Kibbles 'n Bits dog food (Ken-L Ration), non-fat dry milk (Carnation), vegetable cooking oil (Crisco), dried beef, acetaminophen (Extra-strength Tylenol, McNeilab), Red Delicious Washington apples, and Twinkies snack cakes (Hostess, individually wrapped in cellophane). The dried beef, dog food and acetaminophen were finely ground in a coffee mill before fumigation to ensure homogeneity of samples.

Subdivided food samples (5 g) were exposed to sulfuryl fluoride at 36 and 360 mg/L for 20 h in open disposable cups. The apples and snack cakes were exposed whole. After sulfuryl fluoride exposure, the samples were stored in the cups at 25 ºC in an air conditioned laboratory. At 1, 8, and 15 days post fumigation, two fumigated samples (5 g) and two control samples (5 g) from identical food lots were individually placed on a mechanical shaker at room temperature in 50 mL of deionised water for 1 h. Prior to residue extraction, the apples and snack cake were finely chopped. Twenty milliliters of the resultant suspensions was centrifuged at 2000 rpm for 30 min, and 5 mL the supernatant was passed through a C18 Sep-Pak column and a disposable syringe filer and stored at – 20 ºC in polyethylene vials until HPLC analysis. Crude supernatants (5 mL) were frozen and also stored for F^- electrode analysis. Two samples of each commodity were prepared and analyzed for each sulfuryl fluoride exposure rate and aeration interval. Fumigation at each exposure rate was replicated twice for a total of 192 treated and 192 control samples.

HPLC analysis was performed on a Dionex 2000i ion chromatograph for fluoride anion and residues in treated commodities. Recovery experiments were carried out as above except that the deionised water was fortified with both standards at 25 ppm for apple, acetaminophen, and oil; 50 ppm for flour, dog food, and cake; and 100 ppm for dry milk and beef. These represented residue levels of 250, 500, and 1000 ppm, respectively, as 5-g food samples were extracted in 50 ml of water. Recovery samples were carried through procedures in duplicate for each commodity. Unfumigated, unfortified samples were extracted and chromatographed as described above to determine background levels of the analyte anions and co-eluting unknown compounds.

Fluoride selective electrode analysis was performed for fluoride anion residues in treated commodities. A fluoride-selective combination electrode (F^- and reference electrode in one probe) connected to a digital pH meter (Orion 701) was calibrated with KF standard solutions prior to sample analysis. The probe provided a linear response above 0.4 ppm (w/w. F^- equivalent in food). Recovery experiments were carried out in duplicate at 100 ppm (w/w, F^- equivalent) with KF fortification.

Residues of F^- and SO_4^{2-} in commodities resulting from direct exposure to sulfuryl fluoride are listed in Tables 4 and 5. At both sulfuryl fluoride exposure rates increased concentration of F^- and

SO_4^{2-} were detected in fumigated commodities compared to unfumigated controls, suggesting that these anions were degradation products of sulfuryl fluoride within commodities. Residue levels among samples from three aeration intervals did not change over the 2-week aeration span, indicating that the anionic analytes were permanent and had formed during initial sulfuryl fluoride fumigation. Oil had no appreciable anionic residues even though this commodity was reported containing the highest sulfuryl fluoride residues. This indicated that the extraction procedure did not hydrolyze any transient sulfuryl fluoride still residing in the commodities.

Table 4. Fluoride and Sulfate Residues in Eight Commodities from Exposure to Sulfuryl Fluoride at 36 mg/L (Scheffrahn, 1989, Report J. Agric. Food Chem., 1989, 37.203-206)

Commodity		Ion Chromatography				SI Probe	
Fumigated	Days	F^-		SO_4^{2-}		F^-	
with SO_2F_2	Aerated	ppm	±SD	ppm	±SD	ppm	±SD
Apple	1	12.8	16.6	15.0	13.5	4.9	1.2
	8	24.5	21.6	10.4	10.2	6.9	3.9
	15	24.5	18.1	23.5	11.5	9.1	3.3
Dried Beef	1	171.2	160.8	106.1	29.6	270.7	28.0
	8	214.6	178.2	158.2	26.7	272.9	18.4
	15	216.3	91.4	189.0	35.7	281.3	22.5
Cake	1	24.2	43.7	17.0	18.6	4.5	7.3
	8	-12.9	49.0	7.4	4.7	9.0	10.0
	15	-5.6	48.0	2.8	12.4	4.5	3.2
Dog food	1	36.5	39.7	43.7	83.1	23.6	1.3
	8	41.9	23.6	61.7	28.5	20.8	0.7
	15	38.0	17.8	70.2	40.2	20.7	1.4
Wheat flour	1	70.0	4.8	38.6	7.1	70.2	4.4
	8	59.6	2.5	45.7	6.5	67.4	1.8
	15	61.6	6.5	59.3	3.8	70.2	4.4
Acetaminophen	1	18.7	2.2	7.6	6.5	15.3	1.2
	8	13.5	1.3	7.0	6.0	14.3	0.7
	15	11.5	2.5	13.8	3.4	14.0	0.8
Vegetable oil	1	0.6	0.4	0.5	0.5	-0.4	0.5
	8	-1.4	2.3	-0.4	2.0	-0.3	0.3
	15	-0.1	0.5	0.5	1.2	-0.3	0.3
Dry non-fat milk	1	97.7	27.5	104.8	73.7	81.6	7.7
	8	101.8	18.7	77.7	26.8	78.5	3.5
	15	108.8	23.3	144.3	9.5	91.6	11.3

Values (ppm w/w, ± standard deviation, n = 4) Corrected for background and percent recovered.

Table 5. Fluoride and Sulfate Residues in Eight Commodities from Exposure to Sulfuryl Fluoride at 360 mg/L (Scheffrahn, 1989, Report J. Agric. Food Chem., 1989, 37.203-206)

Commodity		Ion Chromatography				SI Probe	
Fumigated	Days	F^-		SO_4^{2-}		F^-	
with SO_2F_2	Aerated	ppm	±SD	ppm	±SD	ppm	±SD
Apple	1	30.8	14.2	21.7	11.7	19.6	3.1
	8	27.0	18.7	38.7	12.3	24.6	2.4
	15	42.2	9.8	50.8	8.9	30.3	5.8

Dried Beef	1	1342.3	260.9	502.6	15.0	1691.8	377.6
	8	1185.1	257.9	608.9	28.0	1551.1	361.9
	15	1245.2	170.9	668.7	41.8	1551.1	361.9
Cake	1	43.6	42.9	8.0	7.1	41.0	33.8
	8	99.1	126.3	24.5	37.9	88.0	94.5
	15	28.4	100.3	26.0	28.1	40.4	57.0
Dog food	1	498.6	29.9	136.6	22.8	596.0	24.8
	8	431.2	39.4	184.9	23.4	514.2	20.8
	15	429.4	14.6	196.9	27.6	520.5	35.9
Wheat flour	1	455.1	10.9	150.7	12.0	475.4	11.2
	8	346.8	8.0	171.2	13.0	372.4	12.7
	15	315.4	10.3	185.3	3.4	344.3	11.6
Acetaminophen	1	119.7	28.9	23.1	14.4	113.3	22.5
	8	100.7	9.5	33.8	8.8	98.6	6.4
	15	96.1	7.4	38.3	9.8	97.6	5.9
Vegetable oil	1	0.4	1.1	2.8	0.3	-0.2	0.2
	8	-0.3	1.1	1.2	1.0	-0.2	0.2
	15	-0.2	1.5	1.1	0.8	-0.2	0.2
Dry non-fat milk	1	859.9	147.5	2330.1	702.5	870.8	252.5
	8	811.4	77.1	2237.2	429.4	849.1	188.0
	15	819.3	88.2	2379.7	522.1	804.7	152.6

Values (ppm w/w, ± standard deviation, n = 4) Corrected for background and percent recovered.

The F^- selective electrode (SI probe) and HPLC analytical techniques were compared. As expected the commodities incorporating both proteins and fats yielded the highest permanent residues at both exposure rates. Concentrations of F^- in the dried beef 15 days after fumigation averaged 216 ppm (281 ppm, SI probe) and 1245 ppm (1551 ppm, SI probe) for the 36 and 360 mg/L exposures, respectively. At the corresponding low and high exposure, residues (ppm) were: dry milk 109 (92) and 819 (805); flour milk 62 (70) and 315 (344); dog food 38 (21) and 429 (521). Background levels of F^-, as determined by SI probe, were below 0.2 ppm in unfumigated commodities. Low F^- concentrations (0.1 – 1 ppm) are normal for a wide variety of common foods. High background levels at R_t of F^- observed by HPLC analysis (Table 6) were possibly due to organic acids of anions in the extracts co-eluting with F^-. Thus, peaks corresponding to F^- were verified by SI probe analysis and further diagnosed from slight variations in R_t from F^- standards when chromatographed in highly diluted eluting solvent. Several additional unknown anions with R_t values different from those of F^- and SO_4^{2-} were detected by HPLC in some of the fumigated commodities. Findings from this comparison suggest that if F^- concentrations are sought, the SI probe technique is simpler, faster, less expensive, and generally as or more accurate than the HPLC analysis.

The SO_4^{2-} residues (Tables 4 and 5) were greatest (15 day samples) in dry milk (144 and 2380 ppm, at 36 and 360 mg/L exposures, respectively), beef (189 and 669), dog food (70 and 197), and flour (59 and 185). Although the vegetable oil contained the greatest sulfuryl fluoride residue after hours of aeration in a related study (6 and 71 ppm at 36 and 360 mg/L exposures, respectively, Osbrink *et al*., 1988), little or no F^- and SO_4^{2-} residue was detected for this commodity, further suggesting highly matrix specific degradation routes for sulfuryl fluoride in commodities. Mean percent recoveries of anions in spiked samples varied with commodity from 62 to 91% for F^- (59 and 107% by SI probe) and 77 to 111% for SO_4^{2-} (Table 6). The overall low, but variable, residues in the cake might have been associated with the inconsistent protection afforded by the manufacturer's wrapper. A comparison (Table 7) of the anionic residue list in Table 4 and Table 5 indicates that their amounts are not proportional to sulfuryl fluoride exposure rates. The 360 to 36 mg/L residue ratio between commodities ranged from 1.72 and 11.3 for F^- and 2.16 and 16.4 for SO_4^{2-} (15-day samples). Additionally, the ratios of F^-/SO_4^{2-} at both sulfuryl fluoride exposure rates (Table 7) were highly

variable between commodities, suggesting the substrate-specific nature of sulfuryl fluoride degradation. Assuming the sulfuryl fluoride is completely degraded to free F^- and SO_4^{2-}, the F^-/SO_4^{2-} ratio would equal 0.396. The F^-/SO_4^{2-} ratios (Table 7) are generally higher than the theoretical ratios indicating that a greater proportion of SO_4^{2-} is bound in non-ionic form (i.e. protein adjuncts).

Table 6. Background Concentrations (ppm w/w) and Percent Recoveries of Fluoride and Sulfate from Samples of Unfumigated Commodities (Scheffrahn, 1989, Report J. Agric. Food Chem., 1989, 37.203-206).

	HPLC					SI Probe
	Std Addn [b]	Background Level [a]		Mean % Recovery		Mean % Recovery
Matrix	ppm	F^-	SO_4^{2-}	F^-	SO_4^{2-}	F^-
Apple	250	47.0	13.3	80.4	107.7	99.6
Dried Beef	1000	3127.2	378.5	86.4	76.6	99.3
Cake	500	235.6	217.9	90.8	102.2	99.2
Dog Food	500	191.1	641.7	61.7	101.4	58.6
Wheat Flour	500	49.2	188.8	85.4	90.8	95.9
Acetaminophen	250	12.9	137.1	88.6	106.8	107.2
Vegetable oil	250	11.1	44.8	86.8	95.9	103.2
Dry non-fat milk	1000	145.0	988.6	67.9	110.8	92.0

[a] ppm equivalents of peaks corresponding to integration at retention times of F^- and SO_4^{2-}. Some matrices contain additional unknown anions with R_t equivalent to analytes sought. All values are means of two sample determinations. Background levels for F^- electrode (SI Probe) determinations were below 0.2 ppm.

[b] ppm equivalents fortifications of F^- and SO_4^{2-} to 5 g of matrix (i.e. 10-fold the concentration in extraction water) for HPLC analysis only. F^- SI probe analysis fortification was 100 ppm for all matrices.

Table 7. Ratios of Fluoride and Sulfate Residue Amounts (w/w) in Commodities Fumigated with Sulfuryl Fluoride (Scheffrahn, 1989, Report J. Agric. Food Chem., 1989, 37.203-206).

	360/36 Ratio		F^- / SO_4^{2-} Ratio	
Matrix	F^-	SO_4^{2-}	36 mg/L	360 mg/L
Apple	1.72	2.16	1.00	0.83
Dried Beef	5.76	3.53	1.14	1.86
Cake	--[b]	9.29	--	1.06
Dog Food	11.30	2.80	0.40	2.18
Wheat Flour	5.12	3.12	1.04	1.79
Acetaminophen	8.36	2.78	0.83	2.51
Vegetable oil	--	--	--	--
Dry non-fat milk	7.53	16.45	0.75	0.34

The residue values are consistent with the findings from the fate of sulfuryl fluoride in wheat graham flour. The breakdown pathway appears to be driven by a nucleophilic substitution of the fluoride and does not appear to follow any metabolic process common to living tissues. The residue results for matrices such as flour, dried beef, acetaminophen, cake and dog food which have no metabolic capability, clearly demonstrate that sulfuryl fluoride is degraded via non-metabolic pathway. High residues of fluoride resulting from fumigation have occurred in the proteinaceous, materials containing a solvent system, such as the fat in meat. On the contrary where high fat but low or no protein content is present (e.g. vegetable oil) the low residues occur.

The model system used to investigate the fate of sulfuryl fluoride, graham flour demonstrates the universal fate of sulfuryl fluoride in contact with matrices that contain biochemicals. Dried fruit and tree nuts are not unique to nature, containing the same types of biochemicals as all other matrices investigated. Sulfuryl fluoride will degrade via the same universal non-metabolic nucleophilic substitution route. Therefore the residue definition of inorganic fluoride and sulfuryl fluoride observed in all other commodities will be the same in treated dried fruit and tree nuts.

Sulfuryl fluoride rapidly penetrates all matrices reacting primarily with the amino groups of proteins. Any unreacted sulfuryl fluoride present in the matrix is rapidly lost from leaving only fluoride and sulfate as the terminal residues. The combination of the sulfuryl fluoride fate experiment in graham flour and the residue studies provide a clear understanding of the breakdown pathway of sulfuryl fluoride in a wide range of matrices. That the breakdown pathway is universal to all matrices, both crop/food and non-food, and proceeds via a non-metabolic route. These data demonstrate that the residue formation occurred during the fumigation and that sulfuryl fluoride is degraded through a highly matrix-specific mechanism, i.e. nucleophilic substitution reactions involving the free amino groups of amino acids and sulfuryl fluoride or conventional hydrolysis of sulfuryl fluoride. The extent of fluoride residue formation could be mediated by a solvent, such as fat, which assists in the retention of the sulfuryl fluoride gas and thus keeping the sulfuryl fluoride in contact with the matrix long enough for the degradation reactions to occur. Simultaneously, sulfuryl fluoride is rapidly lost from the matrix through desorption. Residue levels among samples collected at different time intervals during 2-weeks of aeration showed no trends toward increase or decrease over the total aeration period. It is concluded that the fluoride anionic analytes were permanent and had formed during initial sulfuryl fluoride fumigation.

Plant fate studies demonstrated that sulfuryl fluoride rapidly penetrates all matrices reacting primarily with the amino groups of proteins. Any unreacted sulfuryl fluoride present in the matrix was rapidly lost from the matrix leaving only fluoride and sulfate as the terminal residues. Sulfate is an ion that abundant in nature and is essential to all living things. Sulfate residues resulting from the degradation of sulfuryl fluoride are insignificant in comparison to naturally occurring levels. Sulfate is not viewed as a residue of concern nor is it part of the residue definition. Fluoride anion is the other residue resulting from the breakdown of sulfuryl fluoride. Fluoride ion is the only non- transient part of the residue definition and its deposition has been extensively study and reported in the literature. Deposition of fluoride ion is known to occur in hard tissues such as bone and teeth when consumed as part of the diet. In lactating cattle fluoride ion is eliminated primarily through excretion with only about 2 to 4% of the absorbed dose being eliminated in the milk. Based on the findings of plant and animal degradation and residue deposition studies, the residue definition should be sulfuryl fluoride and fluoride ion in plants and fluoride ion in animals.

Environmental fate in soil and in water-sediment systems

Soil and water methods were not developed for determination of residues of SF. SF is a structural fumigant, which is not used on soil or water nor will it reach these compartments. In addition, its physiochemical characteristics dictate that it will not partition into soil or water since > 99.99% of the fumigant will remain as a gas in air.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Analytical residue methods for the determination of fluoride residues in dried fruits and tree nuts, for the determination of sulfuryl fluoride residues in dried fruit and tree nuts, for the determination of fluoride anion residues in corn, wheat, corn oil and flour, and for the determination of sulfuryl fluoride residues in corn, wheat and rice, were presented to the Meeting.

A Fluoride-Selective Electrode with a Double Known Addition Calibration Technique (GRM 01.11) was described for the determination of fluoride residues in dried fruits and tree nuts (Creasy, 2001, Report GRM 01.11).

Residual fluoride from the fumigation of dried fruits and tree nuts with sulfuryl fluoride was extracted by shaking with Total Ionic Strength Adjustment Buffer (TISAB)/ water solution (1:1 v/v). The centrifuged and filtered aliquot of the extract sample was then quantified using a Fluoride-Selective Electrode with the Double Known Addition Technique. This method is specific for fluoride, and no further confirmation is necessary, and since this method utilizes a fluoride specific electrode using the double known standard addition technique, there is no calibration curve to calculate the sample results. The instrument was calibrated by analyzing standards of a known concentration and recording the output mV reading. A control sample and reagent blank were also analyzed. The fluoride found from the control sample was subtracted from the analytical batch fortification samples to get an accurate recovery value. The treated samples subtract any fluoride found from the reagent blank sample. Since fluoride is prevalent in nature, the fluoride interference could not be eliminated. Method GRA 01.11 had a validated limit of quantitation of 2.4 mg/kg for dried fruit and tree nuts (Table 8).

Validation of the method GRM 01.11 on walnuts and raisin was carried out as part of the method (Davis, 2002, Report GH-C 5434). The validation on raisin was considered unsuccessful. The validation for two of the walnuts samples had recoveries outside the acceptance range (66% and 133%); however, the average recovery was within the acceptable range. The RSD value for the 24.0 mg/kg recovery was outside the acceptable RSD range, but the overall RSD was within the acceptable RSD range. Problems were encountered in the ability of the fluoride probes to get acceptable slope values. To eliminate this problem the probes were placed in Milli-Q water for at least one minute between each sample. This allowed the mV readings to re-equilibrate and to help eliminate the possibility of carryover between samples. The third validation on walnut showed that the overall average recovery for the method was 86%, with a relative standard deviation (RSD) of 19%.

Table 8. Method GRM 01.11 – Validation Data (Creasy, 2001, Report GRM 01.11)

Reference	Matrix	Fortification Level (µg/g)	Recovery Rate (%)		RSD (%)	n
			Mean	range		
GRM 01.11	Raisins	2.4	109	83-123	14	7
		23.7	88	67-100	17	4
		43.1	112	92-131	NA	2
		2.4-43.1	103	67-131	18	13
	Prunes	2.6	100	79-109	12	7
		14.2	92	85-98	NA	2
		28.4	96	90-102	NA	2
		2.6-28.4	98	79-109	11	11
	Figs	2.5	93	68-116	17	7
		10.2	88	85-90	NA	2
		35.6	98	88-108	NA	2
		2.5-35.6	93	68-116	15	11
	Dates	2.5	109	78-127	18	7
		4.9	103	91-115	NA	2
		8.6	94	86-102	NA	2
		47.4	78	75-81	NA	2
		47.7	114	97-131	NA	2
		2.5-47.7	103	75-131	19	15
	Almonds	2.4	109	95-123	10	7
		6.4	77	73-81	NA	2
		23.7	86	76-95	NA	2
		2.4-23.7	99	73-123	17	11

Reference	Matrix	Fortification Level (µg/g)	Recovery Rate (%) Mean	range	RSD (%)	n
	Pecans	2.4	96	86-109	8	7
		4.8	92	82-101	NA	2
		7.2	97	95-98	NA	2
		2.4-7.2	95	82-109	8	11
	Pistachios	2.5	108	100-118	8	7
		7.4	111	103-118	NA	2
		2.5-7.4	108	100-118	8	9
	Walnut	2.4	115	101-139	18	3
		2.5	106	94-121	13	3
		4.8	114	113-114	NA	2
		36.0	93	91-95	NA	2
		2.4-36.0	108	94-139	14	10
	Walnuts	2.4	85	77-93	8	5
		4.8	77	66-89	15	3
		24.0	93	78-133	25	5
		2.4-24.0	86	66-133	19	13

Gas Chromatography with Electron Capture Detection (GRM 01.12) was described for the determination of sulfuryl fluoride residues in dried fruits and tree nuts (Creasy, 2001, Report GRM 01.12). Residues of sulfuryl fluoride were extracted from dried fruit or tree nuts sample after blended with water and analyzed using gas chromatograph with electron capture detection using a J&W Scientific GS-Q Megabore 30 m × 0.53 mm i.d. capillary column. A calibration curve for four standard concentrations ranging from 1.7–21 ng/g for the dried fruit and 1.7–105 ng/g for the tree nuts is specified in the method. A calibration curve for a tree nut analysis is included in the method, but no calibration curve is included in the method for the dried fruits. Linearity is indicated by the correlation coefficients from power regression analysis, which is reported to be greater than 0.96 for all of the calibration curve determinations during the method validation. The walnut chromatograms demonstrated that no background interference occurred and the corresponding result for dried fruit and tree nuts show all control sample results to be 0.0 ng/g. Method GRM 01.12 (Table 9) has a validated limit of quantitation of 4.2 µg/kg.

Table 9. Method GRM 01.12 – Validation Data (Creasy, 2001, Report GRM 01.12)

Reference	Matrix	Fortification Level (ng/g)	Recovery Rate (%) Mean	range	RSD (%)	n
GRM 01.12	Raisins	4.2	79	67-96	13	10
		18.3	66	65-67	NA	2
		21.1	79	74-83	NA	2
		4.2-21.1	77	65-96	13	14
	Prunes	4.2	79	75-112	42	7
		14.5	98	97-98	NA	2
		63.1	93	93	NA	2
		4.2-63.1	77	65-96	13	11
	Figs	4.2	100	78-114	13	7
		15.2	96	92-100	NA	2
		63.1	94	93-95	NA	2
		4.2-63.1	98	78-114	11	11
	Dates	4.2	114	110-119	4	7
		20.0	103	90-115	NA	2
		4.2-20.0	98	90-119	11	9
	Almonds	4.2	83	71-97	10	7

Reference	Matrix	Fortification Level (ng/g)	Recovery Rate (%) Mean	range	RSD (%)	n
	Pecans	4.2	85	69-98	11	7
	Pistachios	4.2	91	83-100	8	7
		31.9	91	89-93	NA	2
		105.0	96	93-98	NA	2
		4.2-105.0	92	83-100	6	11
	Walnuts	4.2	105	99-112	5	7
		24.0	118	118	NA	2
		103.6	96	94-98	NA	2
		4.2-103.6	106	94-118	8	11

Validation of the method GRM 01.12 on dried fruit and nuts were carried out as part of the method (Davis, 2002, ReportGH-C 5422) (Table 10). During the validation exercise minor modifications to the original procedure were occasionally required. Problems were encountered with the use of non-side port needles. It was determined that it was very important to use side port needles, as the non-side port needles can plug very quickly and cause erratic recovery results. It was also determined that the opening of the valve assembly after spiking the air-tight blender jar was not necessary, since this can cause leakage of the spiked gas and erratic recoveries. The first method attempt was successful with recoveries averaging 99% (RSD 4%) for the raisins and 93% (RSD 8%) for the walnuts.

Table 10. Independent Laboratory Validation of Method GRM 01.12 (Davis, 2002, Report GH-C 5422)

Reference	Matrix	Fortification Level (ng/g)	Recovery (%) Mean	range	RSD (%)	n
GRM 01.12	Raisins	4.3	97	95-100	2	5
		8.5	103	100-107	4	3
		42.0	99	95-106	4	5
		4.3-42.0	99	95-107	4	13
	Walnuts	4.3	88	80-97	7	5
		8.5	93	92-93	1	3
		42.0	97	96-105	6	5
		4.3-42.0	93	80-105	8	13

Fluoride Selective Electrode (GRM 01.17) was described for the determination of fluoride anion in corn, wheat, maize oil and flour (Rick, 2002, Report GRM 01.17). Residual fluoride from fumigation with sulfuryl fluoride was extracted using Total Ionic Strength Adjustment Buffer (TISAB)/water solution (1:1 v/v). The samples were macerated using a PolyTron homogeniser and shaken on a flatbed shaker except for corn oil, and then quantified using Fluoride-Selective Electrode with the Double Known Addition Technique. The methods GRM 01.17 and 011057 Appendix A are the same method. This method utilizes a fluoride specific electrode using the double known standard addition technique (see above for details).

Validation of the method GRM 01.17 on corn, wheat, corn oil and flour were carried out as part of the method (Lala, 2002, Report 020013/1404) (Table 11 and 12). The LOQ was 0.5 mg/kg for maize, wheat, maize oil and flour. GRM 01.17 was independently validated for corn oil and raisins and the method was slightly modified to accommodate the analysis of raisins. In previous work it was found that the 0.5 mg/kg limit of quantitation (LOQ) as stated in the method was difficult to achieve, so it was decided to raise the LOQ to 2.0 mg/kg. The change in LOQ was not an issue since the proposed

tolerances for these crop varieties were higher than 2.0 mg/kg. The average recoveries were found at 99.5% (RSD 4.4%) for the corn oil and 88.8% (RSD 6.4%) for the raisins.

Table 11. Independent Laboratory Validation of Method GRM 01.17 (Lala, 2002, Report 020013/1404).

Reference	Matrix	Fortification Level (µg/g)	Recovery Rate (%)		RSD (%)	n
			Mean	range		
GRM 01.17	Wheat Grain	0.5	79	66-93	15	7
010057		2.0	97	93-99	3	3
Appendix A		5.0	90	85-94	5	3
		50.0	76	70-85	4	3
		0.5-50.0	84	66-99	16	16
	Maize Grain	0.5	102	94-121	9	7
		2.0	102	99-105	3	3
		5.0	102	98-106	4	3
		50.0	98	93-101	4	3
		0.5-50.0	101	93-121	16	16
	Wheat Flour	0.5	99	91-106	6	7
		2.0	101	96-106	5	3
		5.0	99	97-102	2	3
		50.0	96	90-103	7	3
		0.5-50.0	99	90-106	16	16
	Maize Oil	0.5	104	85-118	10	7
		2.0	112	102-123	11	6
		5.0	105	95-119	9	5
		50.0	119	114-127	3	3
		0.5-50.0	109	85-127	10	21

Table 12. Independent Laboratory Validation of Method GRM 01.17 (Lala, 2002, Report 020013/1404)

Reference	Matrix	Fortification Level (µg/g)	Recovery (%)		RSD (%)	n
			Mean	range		
GRM 01.17	Maize Oil	2.0	99	90-104	6	5
		20.0	101	98-106	4	5
		2.0-20.0	100	98-106	4	10
GRM 01.17	Raisins	2.0	91	81-102	8	5
		20.0	87	83-90	6	5
		2.0-20.0	89	81-102	6	10

Fluoride Selective Electrode (011057 – Appendix A) was described for the determination of fluoride anion in maize, wheat, corn oil and flour (Rick, 2001, Report 011057). Residual fluoride from fumigation with sulfuryl fluoride is extracted using Total Ionic Strength Adjustment Buffer (TISAB)/ water solution (1:1 v/v). Samples of maize, wheat and flour were then macerated using a PolyTron homogeniser. An aliquot of the extract is centrifuged and then quantified using Fluoride-Selective Electrode with the Double Known Addition Technique. The methods 011057 Appendix A and GRM 01.17 are the same method. The Appendix A method was reformatted and renamed GRM 01.17. Method 011057 Appendix A has a validated limit of quantitation of 0.5 µg/g for corn, wheat, corn oil and flour, which is adequate to support pre-registration data requirements.

Validation of the method 011057 – Appendix A on corn, wheat, corn oil and flour were carried out as part of the method (Davis, 2002, Report 010115) (Table 13). Since the contract lab was

unable to attain the 0.5 ug/g limit of quantitation (LOQ) as stated in the method, the LOQ was raised to 2.0 µg/g. The average recoveries found were 102% (RSD 8%) for the wheat grain and 96% (RSD 6%) for the wheat flour.

Table 13. Independent Laboratory Validation of Method 011057 (Davis, 2002, Report 010115)

Reference	Matrix	Fortification Level (µg/g)	Recovery (%)		RSD (%)	n
			Mean	range		
011057-Appendix A GRM 01.17	Wheat Grain	2.0	103	92-113	8	5
		10.0	96	84-105	11	3
		20.0	103	95-107	5	5
		2.0-20.0	102	84-113	8	13
	Wheat Flour	2.0	95	87-105	7	5
		10.0	95	92-97	3	3
		20.0	99	92-108	6	5
		2.0-20.0	96	87-108	6	13

Gas Chromatography with Electron Capture Detection (011057 – Appendix B) was described for the determination of sulfuryl fluoride residues in maize, wheat and rice commodities (Rick, 2001, Report 011057) (Table 14). Residues of sulfuryl fluoride are extracted by blending samples with water in an air-tight blender jar. A calibration curve for three standard concentrations, injected in duplicate, ranging from 4.0-80.0 ng/ g was specified in the method. A calibration curve was included in the method demonstrating linearity by the correlation coefficient (r^2) of 0.994. The wheat grain chromatogram included in the method demonstrated that no background interference occurred, and the corresponding result tables for wheat commodities, maize commodities, and rice commodities, showed all control sample results to be 0.0 ng/g. Method 011057 – Appendix B has a validated limit of quantitation of 8.0 µg/kg.

Table 14. Independent Laboratory Validation of Method 011057 Appendix B (Rick, 2001, Report 011057).

Reference	Matrix	Fortification Level (ng/g)	Recovery Rate (%)		RSD (%)	n
			Mean	range		
011057 Appendix B	Whole Wheat Grain	8.0	94	58-153	21	28
		80.0	93	80-114	12	16
		8.0-80.0	93	58-153	18	44
	Wheat Flour	8.0	89	57-116	16	30
		80.0	85	71-94	8	16
		8.0-80.0	88	57-116	14	46
	Wheat Germ	8.0	71	55-80	14	6
		20.0	86	65-126	17	22
		80.0	89	84-96	5	6
		200.0	80	67-92	10	10
		8.0-200.0	83	55-126	15	44
	Wheat Bran	8.0	100	95-110	6	6
		80.0	83	75-91	7	6
		8.0-80.0	92	75-110	12	12
	Red Dog	8.0	88	83-96	6	6
		80.0	93	88-100	5	6
		8.0-80.0	90	83-100	6	12
	Wheat Shorts	8.0	84	72-93	10	6
		80.0	84	81-86	2	6
		8.0-80.0	84	72-93	7	12

Maize Grain	8.0	113	98-128	9	12
	80.0	94	62-118	18	18
	8.0-80.0	101	62-128	17	30
Maize Flour	8.0	103	89-121	8	11
	80.0	82	61-112	22	17
	8.0-80.0	91	61-121	20	29
Maize Starch	8.0	88	81-98	3	6
	80.0	85	81-89	3	6
	8.0-80.0	86	81-98	6	12
Maize Grits	8.0	98	84-129	13	12
	80.0	85	78-92	6	12
	8.0-80.0	91	78-129	13	24
Polished Rice	8.0	81	52-105	20	13
	80.0	89	74-99	9	12
	8.0-80.0	85	52-105	16	25
Rice Bran	8.0	82	63-100	11	18
	80.0	79	67-97	9	20
	8.0-80.0	80	63-100	10	38

Note: The RSD for the Whole Wheat Grain 8.0 ng/g recovery values is slightly outside the acceptable limits, but the overall RSD is within the acceptable range. The RSD for the maize flour had one outlier of 58% at the 8.0 ng/g and one at 80.0 ng/g of 123% tossed out. The RSD for the recoveries at 80.0 ng/g is still above the acceptable limits, but the overall RSD is within the acceptable limits.

Validation of the method 011057 – Appendix B for the determination of sulfuryl fluoride residues in corn, wheat and rice commodities by Gas Chromatography with Electron Capture Detection was carried out as part of the method (Davis, 2002, Report 010114) (Table 15). The average recoveries were found at 104% (RSD 10%) for the wheat grain and 94% (RSD 11%) for the wheat flour.

Table 15. Independent Laboratory Validation of Method 011057 – Appendix B (Davis, 2002, Report 010114)

Reference	**Matrix**	**Fortification Level (ng/g)**	**Recovery (%)**		**RSD (%)**	**n**
			Mean	**range**		
011057- Appendix B	Wheat Flour	8.3	93	86-97	5	5
		78.9	95	74-108	14	5
		8.3-78.9	94	74-108	11	10
011057- Appendix B	Wheat Grain	8.3	112	108-114	2	5
		78.9	95	85-102	7	5
		8.3-78.9	104	85-114	10	10

No analytical methods were developed for animal tissue matrices since there are no uses of sulfuryl fluoride on fodder commodities.

Sulfuryl fluoride has not been subjected to multi-residue methods testing, because the DFG S19 multi-residue methods of Germany are inapplicable for sulfuryl fluoride. Sulfuryl fluoride is a gas and the extraction procedures used in DFG S19 would not be compatible. Fluoride has not been subjected to multi-residue methods testing because the DFG S19 multi-residue methods are inapplicable for the fluoride ion. Fluoride is not detectable by GC so the procedures used in DFG S19 would not be applicable.

Table 16. Summary of Residue Method Testing for Sulfuryl fluoride

Matrix	Method	Analyte	LOQ	Reference
Dates Raisins Figs Prunes Almonds Pecans Pistachio Walnuts	Fluoride Specific Electrode	Fluoride	2.4 mg/kg	GRM 01.11
Dates Raisins Figs Prunes Almonds Pecans Pistachio Walnuts	GC/ECD	Sulfuryl Fluoride	4.2 µg/kg	GRM 01.12
Wheat Grain Wheat Flour Maize Grain Maize Oil	Fluoride Specific Electrode	Fluoride	0.5 mg/kg	GRM 01.17
Wheat Grain Wheat Flour Maize Grain Maize Oil	Fluoride Specific Electrode	Fluoride	0.5 mg/kg	Method Described in Report 011057 -Appendix A,

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of fluoride residues in wheat grain, wheat flour, raisin, walnut, maize grain, and maize flour under storage conditions (Foster, 2001, Report, 010017). Samples were fortified at 20 mg/kg with fluoride and stored under ambient conditions for 30 days, and then placed in a freezer at approximately –20°C after the 30 day sampling time-point. The samples were analyzed using the draft analytical method "Residue Method Validation for the Determination of Fluoride Anion in Corn, Wheat, Corn Oil, and Flour" as noted in Dow AgroSciences Laboratory Notebook C1169. This method had a validated limit of quantitation of 0.6 mg/kg for wheat grain, 0.5 mg/kg for maize grain, and 0.3 mg/kg for wheat flour. Results are summarized in Table 17. Recoveries of fluoride in wheat grain and flour, dried fruit (raisins), nuts walnuts), maize grain and meal at the last sampling ranged from 80 to 100% based on the fortified amount of 20 mg/kg.

Table 17. Storage stability of Fluoride Ion (F^-) in several substrates fortified at 20 mg/kg (Foster, 2001, Report, 010017)

Matrix	% Fluoride Remaining				
	0 Day	34-36 Day	74-77 Day	102-105 Day	138-141 Day
Wheat Grain	92	88	97	85	92
Wheat Flour	100	87	78	72	55
Raisin	98	100	93	94	98
Walnut	97	106	113	116	110
Maize Grain	119	93	93	93	91
Maize Meal	101	110	102	97	98

USE PATTERN

Sulfuryl fluoride is registered in USA, Caribbean Islands, Japan and Sweden, and is authorized for use in Germany as a fumigant for control of wood destroying and stored product insects in buildings including residential homes, churches, museums, historical landmarks, rare book collections, scientific and medical research laboratories. The product is also used to fumigate shipping containers and also all vehicles except aircraft and subsurface water vessels. The information available to the Meeting on commodities that may be fumigated with sulfuryl fluoride is presented in the following paragraphs:

Dried Fruits: Date; Fig; Plum; Prune, dried; Grape, raisin; Other dried fruit (e.g., apricots).

Tree Nuts including Almond; Pecan; Walnut; Beechnut; Butternut; Cashew; Chestnut; Chinquapin; Filbert nut, Brazil nut; hickory nut, macadamia.

Other: Pistachio

Cereals and Small Grains: Barley; Maize; Maize, pop; Oat; Rice; Wheat; Millet; Rice, wild; Sorghum; Triticale .

Cereal and Small Grain Processed Products: Maize flour; Maize grits; Maize meal; Rice, brown; Rice, polished; Rice bran; Rice hulls; Wheat bran; Wheat flour; Wheat germ; Wheat milled byproducts; Wheat shorts.

Dosage

Sulfuryl fluoride dosages are properly calculated as the product of fumigant concentration (C) x Exposure Time (T) = CTP commonly given in gram-hours per cubic meter ($g \cdot hr/m^3$) units. In the past, pest control fumigators, when using fumigants such as methyl bromide and phosphine simplified target dosages to a rate g/m^3 methyl bromide and/or phosphine. This approach has the significant flaw of not accounting for variable gas loss rates. The result is overdosing (gas loss rate slower than planned) or under dosing (gas loss rate was faster than planned) in many situations. The maximum dosage (or maximum "label rate") for sulfuryl fluoride use is 1,500 $g \cdot hr \cdot /m^3$ –equivalent to 1,500 mg·hr/L-(or "CT" or "CTP") with a maximum fumigation concentration of 128 g/m^{-3}. (For two trials on each of the tree nuts below, GAP included the use of 200 CTP when treated under vacuum fumigation conditions). The total target CTP for a single fumigation is calculated based on several parameters, the specific environmental conditions in the area to be fumigated, target pests, planned fumigant holding period and estimated fumigant loss rate (half-loss time) from the fumigated area. As temperature within the fumigated area increases the amount of total CTP needed decreases. The length of the fumigant holding period has an inverse relationship to the initial concentration of fumigant needed to achieve the target CTP, as well as, the fumigant half-loss time. Application of sulfuryl fluoride to a fumigated area is accomplished by releasing the appropriate mass of fumigant through shooting lines (tubes that connect the cylinders containing the fumigant outside the area to the release point within the fumigated area) into the air space within the fumigated area, i.e. sealed mills, chambers or trapped areas. After achieving the target CTP, the fumigated area is aerated to reduce the fumigant concentration to levels below the acceptable SF concentration for worker reentry. The aeration of structures usually take only several hours, while commodities such as dried fruit and tree nuts are allowed to aerate (desorption of sulfuryl fluoride from the commodity) for 24 hours prior to offering to consumers.

Mode of Entry

Sulfuryl fluoride occupies the air spaces within the fumigated site. These air spaces may be visible (cracks and crevices within structures) or unseen (pores within wooden members or flour deposits).

Table 18. Good Agricultural Practices (GAP) for Post-harvest Uses in USA.

Crop and/or situation	Product Name	Pests or Group of pests controlled	Formulation		Application				Remarks
			Type	Conc. of a.s.	Method	Number	Interval between apps. (min)	Rate	
Stored product pests in emptied cereal mills, empty grain storage areas	ProFume	Stored product insect pests	Fumigant Gas. Packaged as a liquid under pressure. As liquid is released it immediately converts to a gas state and disperses in the confined space of the fumigation.	99.8%	Fumigation of building or storage container by releasing fumigant from cylinder, confining for 2-72+ hrs and then aerating fumigant until ambient air concentration is ≤ 5 ppm.	1/year	N/A A particular plant will be fumigated on a schedule which ranges from 3 times per year to once every few years.	Units are different for a fumigation product. CTP= Concentration X Time Product. Max. CTP = 1500. Max Conc. is 128 g/m^3	The dosage (concentration x exposure time) used is dependent on the temperature, species and life stage. Representative fumigation conducted with a 24 h fumigation at about 25^{0}C

Table 18. Good Agricultural Practices (Gap) for Pesticide Use in USA (cont.)

Post harvest commodities	Product Name	Pests controlled	Formulation		Application				Remarks
			Type	Conc. of a.s.	Method	Number min max	Interval between apps. (min)	Rate	
Dried Fruits (e.g. raisins, prunes, apricots, etc.) and Tree Nuts (e.g. walnuts, almonds & pistachios). Fumigation of stored commodity in permanent or temporary chambers	ProFume	Stored Product Insect Pests. Examples include beetles and Lepidoptera pests	Fumigant Gas. Packaged as a liquid under pressure. As liquid is released it immediately converts to a gas state and disperses in the confined space of the fumigation.	99.8%	Fumigation. Release fumigant from cylinder into confined air space; confine gas for 2–72+ hours, then exhaust. Vacuum fumigation follows same procedure, with vacuum to increase insecticidal activity.	1–4 x per commodity Most commonly only fumigated 1-2 times. Depends on length of commodity storage	Generally several months, if refumigation occurs at all. Refumigate when commodity becomes reinfested during storage.	Short term fumigation of 2 to 4 h exposure using max 128 g/m^3 (CTP 1500). Standard exposure of 24 to 48 h. Dosage decreases as temperature increases.	The dosage (concentration x exposure time) used is dependent on the temperature. Max Concentration x Time dosage range for stored product insects expected to be 1500 g-h/m^3. Representative fumigation conducted with 24 h fumigation at about 25^{0}C.

Products based on this active ingredient are not authorised for use on agricultural crops in the Netherlands.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on sulfuryl fluoride supervised trials for cereals, dried fruits and tree nuts. All reported trials were for post-harvest use. The conditions for maximum GAP for residue levels in commodities for supported uses can be summarized as follows:

Table 19. Supervised Trial Residue Trials – Conditions of Product Use

Crop	Method of Application	Application rate[1] (mg·hr/L)	No. of applications	Growth stage at application	Post-fumigation interval
Cereals	Fumigation	200–1500	1	Post-Harvest	1–7 days
Dried Fruit	Fumigation	200–1500	5	Post-Harvest	1–15 days
Tree Nuts	Fumigation	200–1500	3	Post-Harvest	1–17 days

[1]The lower rate (200 mg h/L) is considered equivalent GAP to the 1500 when performed under reduced pressure, i.e. vacuum fumigation; this pertains especially to two trials on each of the tree nuts.

Residue results and details of the supervised residue trials in cereal grains, dried fruits and tree nuts are presented as in Table 19 though Table 43. The GAPs in these trials were varied depending on

the product use. In many cases, a worst case scenario has been employed, i.e. high application rate (maximum concentration x time product).

Table 20. Residues of Sulfuryl Fluoride in Barley - Grain after post-harvest application by fumigation

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	200	1	< 0.008	1.01	Rick, 2001
USA	2001	1	200	1	< 0.008	1.03	Rick, 2001
USA	2001	1	200	1	< 0.008	1.00	Rick, 2001
USA	2001	1	200	1	< 0.008	0.85	Rick, 2001
USA	2001	1	1,670	1	< 0.008	2.79	Rick, 2001
USA	2001	1	1,640	1	< 0.008	3.05	Rick, 2001
USA	2001	1	1,620	1	< 0.008	2.76	Rick, 2001
USA	2001	1	1,660	1	< 0.008	2.92	Rick, 2001
USA	2001	1	970	1	N/A	4.47	Rick, 2001
USA	2001	1	1,140	1	N/A	7.09	Rick, 2001
USA	2001	1	980	1	N/A	9.25	Rick, 2001
USA	2001	1	1,800	1	N/A	6.49	Rick, 2001
USA	2001	1	1,790	1	N/A	7.96	Rick, 2001
USA	2001	1	940	1	N/A	4.27	Rick, 2001
USA	2001	1	270	1	N/A	3.48	Rick, 2001
USA	2001	1	290	1	N/A	2.58	Rick, 2001
Germany	2000	1	2016	1	N/A	23.3	Perkins, 2002
England	2000	1	1585	1	N/A	21.0	Perkins, 2002
Italy	2001	1	1380	1	N/A	11.75	Perkins, 2002
England	2002	1	1636	1	N/A	18.4	Bostock, 2002
Germany	2002	1	1484	1	N/A	10.0	Blaschke, 2002
USA	2002	1	1761	1	N/A	18.4	Barnekow, 2002

Table 21. Residues of Sulfuryl Fluoride in Maize – Whole Kernel after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,640	1	0.026	N/A	Rick, 2000
				4	< 0.008	2.25	
				7	< 0.008	2.07	
USA	2000	1	1,730	1	0.020	N/A	Rick, 2000
				4	< 0.008	2.26	
				7	< 0.008	2.13	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	250	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	270	1	N/A	2.07	Rick, 2001
USA	2001	1	940	1	N/A	1.11	Rick, 2001
USA	2001	1	1,790	1	N/A	1.86	Rick, 2001
USA	2001	1	1,800	1	N/A	1.71	Rick, 2001
USA	2001	1	980	1	N/A	5.31	Rick, 2001
USA	2001	1	1,140	1	N/A	2.25	Rick, 2001
USA	2001	1	970	1	N/A	1.03	Rick, 2001
USA	2001	1	1,380	1	< 0.008	1.64	Rick, 2001
USA	2001	1	1,500	1 2	0.018 < 0.008	N/A 1.31	Rick, 2001
USA	2001	1	1,260	1	< 0.008	1.36	Rick, 2001
USA	2001	1	1,600	1	< 0.008	1.42	Rick, 2001
USA	2001	1	210	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	230	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	190	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	240	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	290	1	N/A	<0.50	Rick, 2001
USA	2001	1	270	1	N/A	<0.50	Rick, 2001
USA	2001	1	940	1	N/A	1.00	Rick, 2001
USA	2001	1	1,790	1	N/A	1.19	Rick, 2001
USA	2001	1	1,800	1	N/A	1.52	Rick, 2001
USA	2001	1	980	1	N/A	2.05	Rick, 2001
USA	2001	1	1,140	1	N/A	1.42	Rick, 2001
USA	2001	1	970	1	N/A	1.09	Rick, 2001
USA	2001	1	1,460	1	< 0.008	0.879	Rick, 2001
USA	2001	1	1,550	1	< 0.008	0.966	Rick, 2001
USA	2001	1	1,560	1	< 0.008	1.41	Rick, 2001
USA	2001	1	1,450	1	< 0.008	0.764	Rick, 2001
USA	2001	1	220	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	250	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	230	1	< 0.008	<0.50	Rick, 2001
USA	2001	1	290	1	< 0.008	<0.50	Rick, 2001

Table 22. Residues of Sulfuryl Fluoride in Maize - Flour after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,620	1	<u>≤ 0.008</u>	N/A	Rick, 2000
				4	< 0.008	14.9	
				7	< 0.008	18.9	
USA	2000	1	1,660	1	<u>≤ 0.008</u>	N/A	Rick, 2000
				4	< 0.008	15.4	
				7	< 0.008	19.2	
Germany	2000	1	2,016	1	N/A	55.0	Perkins, 2002
England	2000	1	1585	1	N/A	55.5	Perkins, 2002
Italy	2001	1	1380	1	N/A	24.27	Perkins, 2002
England	2002	1	1636	1	N/A	70.1	Bostock, 2002
Germany	2002	1	1484	1	N/A	14.1	Blaschke, 2002
USA	2002	1	1761	1	N/A	37.4	Barnekow, 2002

Table 23. Residues of Sulfuryl Fluoride in Maize - Starch after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,620	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	3.82	
				7	N/A	3.91	
USA	2000	1	1,660	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	5.35	
				7	N/A	5.30	

Table 24. Residues of Sulfuryl Fluoride in Maize - Meal after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,930	1	<u>≤ 0.008</u>	N/A	Rick, 2000
				4	< 0.008	5.60	
				7	N/A	5.24	
USA	2000	1	2,200	1	<u>≤ 0.008</u>	N/A	Rick, 2000
				4	< 0.008	6.30	
				7	N/A	6.14	

Table 25. Residues of Sulfuryl Fluoride in Maize - Grits after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,840	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	9.09	
				7	N/A	7.74	
USA	2000	1	2,250	1	0.014	N/A	Rick, 2000
				4	< 0.008	9.17	
				7	N/A	8.26	

Table 26. Residues of Sulfuryl Fluoride in Maize - Oil after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,840	1	7.840	N/A	Rick, 2000
				4	2.664	< 0.60	
				7	4.384	< 0.60	
USA	2000	1	2,250	1	5.848	N/A	Rick, 2000
				4	2.511	< 0.60	
				7	3.128	< 0.60	

Table 27. Residues of Sulfuryl Fluoride in Oats after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	200	1	< 0.008	1.87	Rick, 2001
USA	2001	1	270	1	N/A	7.78	Rick, 2001
USA	2001	1	940	1	N/A	6.82	Rick, 2001
USA	2001	1	1,790	1	N/A	14.0	Rick, 2001
USA	2001	1	1,800	1	N/A	12.5	Rick, 2001
USA	2001	1	980	1	N/A	15.2	Rick, 2001
USA	2001	1	1,140	1	N/A	9.15	Rick, 2001
USA	2001	1	970	1	N/A	7.88	Rick, 2001
USA	2001	1	1,530	1	< 0.008	8.27	Rick, 2001
USA	2001	1	1,530	1	< 0.008	7.50	Rick, 2001
USA	2001	1	1,540	1	< 0.008	7.00	Rick, 2001
USA	2001	1	1,630	1	< 0.008	7.39	Rick, 2001
USA	2001	1	170	1	< 0.008	1.85	Rick, 2001
USA	2001	1	180	1	< 0.008	1.81	Rick, 2001
USA	2001	1	160	1	< 0.008	1.76	Rick, 2001
USA	2001	1	290	1	N/A	7.46	Rick, 2001

Table 28. Residues of Sulfuryl Fluoride in Rice after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	210	1	< 0.008	0.561	Rick, 2001
USA	2001	1	270	1	N/A	3.84	Rick, 2001
USA	2001	1	940	1	N/A	4.54	Rick, 2001
USA	2001	1	1,790	1	N/A	7.29	Rick, 2001
USA	2001	1	1,800	1	N/A	6.24	Rick, 2001
USA	2001	1	980	1	N/A	11.8	Rick, 2001
USA	2001	1	1,140	1	N/A	6.95	Rick, 2001
USA	2001	1	970	1	N/A	3.84	Rick, 2001
USA	2001	1	1,590	1	< 0.008	2.38	Rick, 2001
USA	2001	1	1,730	1	< 0.008	2.16	Rick, 2001
USA	2001	1	1,570	1	< 0.008	2.17	Rick, 2001
USA	2001	1	1,510	1	< 0.008	2.23	Rick, 2001
USA	2001	1	210	1	< 0.008	0.563	Rick, 2001
USA	2001	1	220	1	< 0.008	0.576	Rick, 2001
USA	2001	1	210	1	< 0.008	0.611	Rick, 2001
USA	2001	1	290	1	N/A	2.35	Rick, 2001
USA	2001	1	220	1	< 0.008	< 0.50	Rick, 2001
USA	2001	1	180	1	< 0.008	< 0.50	Rick, 2001
USA	2001	1	210	1	< 0.008	< 0.50	Rick, 2001
USA	2001	1	200	1	< 0.008	<0 .50	Rick, 2001
USA	2001	1	1,710	1	< 0.008	2.01	Rick, 2001
USA	2001	1	1,460	1	< 0.008	2.03	Rick, 2001
USA	2001	1	1,520	1	< 0.008	1.96	Rick, 2001
USA	2001	1	1,500	1	< 0.008	1.83	Rick, 2001
USA	2001	1	970	1	N/A	4.99	Rick, 2001
USA	2001	1	1,140	1	N/A	5.47	Rick, 2001
USA	2001	1	980	1	N/A	16.1	Rick, 2001
USA	2001	1	1,800	1	N/A	7.64	Rick, 2001
USA	2001	1	1,790	1	N/A	10.7	Rick, 2001
USA	2001	1	940	1	N/A	3.21	Rick, 2001
USA	2001	1	270	1	N/A	2.23	Rick, 2001
USA	2001	1	290	1	N/A	2.42	Rick, 2001
USA	2000	1	2,190	1 4 7	0.025 < 0.008 N/A	N/A 5.49 5.97	Rick, 2000
USA	2000	1	2,240	1 4 7	0.016 < 0.008 N/A	N/A 7.80 8.46	Rick, 2000
Germany	2000	1	2,016	1	N/A	6.75	Perkins, 2002
England	2000	1	1585	1	N/A	14.6	Perkins, 2002

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
Italy	2001	1	1380	1	N/A	3.56	Perkins, 2002
England	2002	1	1636	1	N/A	8.35	Bostock, 2002
Germany	2002	1	1484	1	N/A	2.80	Blaschke, 2002
USA	2002	1	1761	1	N/A	7.93	Barnekow, 2002

Table 29. Residues of Sulfuryl Fluoride in Rice-Bran after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,100	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	28.5	
				7	N/A	24.6	
USA	2000	1	2,300	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	24.2	
				7	N/A	26.3	

Table 30. Residues of Sulfuryl Fluoride in Rice-Polished after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,210	1	≤ 0.008	N/A	Rick, 2000
				4	N/A	1.47	
				7	N/A	1.30	
USA	2000	1	1,980	1	≤ 0.008	N/A	Rick, 2000
				4	N/A	1.60	
				7	N/A	1.40	

Table 31. Residues of Sulfuryl Fluoride in Rice-Hulls after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,060	1	0.057	N/A	Rick, 2000
				4	< 0.008	32.8	
				7	< 0.008	30.3	
USA	2000	1	1,540	1	0.056	N/A	Rick, 2000
				4	< 0.008	23.6	
				7	< 0.008	23.3	

Table 32. Residues of Sulfuryl Fluoride in Wheat – Whole Grain after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	250	1	< 0.008	0.850	Rick, 2001
USA	2001	1	240	1	< 0.008	0.859	Rick, 2001
USA	2001	1	250	1	< 0.008	0.850	Rick, 2001
USA	2001	1	240	1	< 0.008	0.859	Rick, 2001
USA	2001	1	230	1	< 0.008	0.905	Rick, 2001
USA	2001	1	240	1	< 0.008	0.925	Rick, 2001
USA	2001	1	1,770	1	< 0.008	2.05	Rick, 2001
USA	2001	1	1,960	1	< 0.008	2.24	Rick, 2001
USA	2001	1	1,718	1	< 0.008	2.11	Rick, 2001
USA	2001	1	1,620	1	< 0.008	1.93	Rick, 2001
USA	2001	1	970	1	N/A	3.61	Rick, 2001
USA	2001	1	1,140	1	N/A	5.00	Rick, 2001
USA	2001	1	980	1	N/A	23.6	Rick, 2001
USA	2001	1	1,800	1	N/A	5.80	Rick, 2001
USA	2001	1	1,790	1	N/A	4.79	Rick, 2001
USA	2001	1	940	1	N/A	4.56	Rick, 2001
USA	2001	1	270	1	N/A	2.76	Rick, 2001
USA	2001	1	290	1	N/A	2.55	Rick, 2001
USA	2000	1	1,610	1	N/A	1.98	Rick, 2000
USA	2000	1	1,410	1	N/A	2.00	Rick, 2000
USA	2000	1	1,800	1	N/A	1.78	Rick, 2000
USA	2000	1	1,510	1	N/A	1.85	Rick, 2000
USA	2000	1	1,770	1	N/A	1.47	Rick, 2000
USA	2000	1	1,630	1	N/A	1.99	Rick, 2000
USA	2000	1	270	1 4 7	< 0.008 < 0.008 N/A	N/A N/A 0.676	Rick, 2000
USA	2000	1	260	1 4 7	< 0.008 < 0.008 N/A	N/A N/A 0.651	Rick, 2000
USA	2000	1	1,420	1 4 7	0.008 < 0.008 N/A	N/A N/A 1.74	Rick, 2000
USA	2000	1	1,300	1 4 7	< 0.008 < 0.008 N/A	N/A N/A 1.52	Rick, 2000
USA	2000	1	3,120	1 4 7	0.036 < 0.008 N/A	N/A N/A 2.03	Rick, 2000

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,460	1	0.021	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	1.85	
USA	2000	1	1,600	1	0.033	N/A	Rick, 2000
				4	0.044	N/A	
				7	0.021	0.790	
USA	2000	1	1,640	1	0.032	N/A	Rick, 2000
				4	0.042	N/A	
				7	0.019	0.909	
USA	2000	1	1,890	1	0.009	N/A	Rick, 2000
				4	< 0.008	1.94	
				7	N/A	1.84	
USA	2000	1	1,880	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	1.92	
				7	N/A	1.93	
USA	2000	1	1,870	1	0.014	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	2.79	
USA	2000	1	1,820	1	0.013	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	2.96	
USA	2000	1	1,790	7	N/A	3.93	Rick, 2000
USA	2000	1	1,730	7	N/A	2.91	Rick, 2000
USA	2000	1	1,790	7	N/A	2.22	Rick, 2000
USA	2000	1	1,780	7	N/A	2.36	Rick, 2000
USA	2000	1	2,190	7	N/A	3.20	Rick, 2000
USA	2000	1	2,190	7	N/A	4.08	Rick, 2000
USA	2000	1	1,840	1	0.011	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	3.10	
USA	2000	2	3,670	1	0.010	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	5.02	
USA	2000	3	5,710	1	0.029	N/A	Rick, 2000
				4	0.009	N/A	
				7	ND	6.15	
USA	2000	4	7,930	1	0.021	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	7.95	
USA	2000	1	1,990	1	0.009	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	3.94	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	2	3,700	1	0.009	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	5.02	
USA	2000	3	5,740	1	0.022	N/A	Rick, 2000
				4	0.009	N/A	
				7	ND	6.31	
USA	2000	4	8,000	1	0.023	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	ND	8.00	
USA	2001	1	240	1	< 0.008	0.803	Rick, 2001
USA	2001	1	270	1	< 0.008	0.877	Rick, 2001
USA	2001	1	210	1	< 0.008	0.886	Rick, 2001
USA	2001	1	280	1	< 0.008	0.857	Rick, 2001
USA	2001	1	1,620	1	≤ 0.008	2.64	Rick, 2001
USA	2001	1	2,120	1	< 0.008	3.50	Rick, 2001
USA	2001	1	1,780	1	≤ 0.008	3.30	Rick, 2001
USA	2001	1	1,550	1	≤ 0.008	2.66	Rick, 2001
USA	2001	1	970	1	N/A	2.12	Rick, 2001
USA	2001	1	1,140	1	N/A	3.89	Rick, 2001
USA	2001	1	980	1	N/A	8.36	Rick, 2001
USA	2001	1	1,800	1	N/A	4.84	Rick, 2001
USA	2001	1	1,790	1	N/A	5.65	Rick, 2001
USA	2001	1	940	1	N/A	3.74	Rick, 2001
USA	2001	1	270	1	N/A	2.06	Rick, 2001
USA	2001	1	290	1	N/A	2.13	Rick, 2001
USA	2001	1	250	1	< 0.008	0.721	Rick, 2001
USA	2001	1	250	1	< 0.008	0.518	Rick, 2001
USA	2001	1	230	1	< 0.008	0.898	Rick, 2001
USA	2001	1	240	1	< 0.008	0.753	Rick, 2001
USA	2001	1	1,820	1	≤ 0.008	4.84	Rick, 2001
USA	2001	1	1,640	1	≤ 0.008	3.38	Rick, 2001
USA	2001	1	1,850	1	≤ 0.008	3.80	Rick, 2001
USA	2001	1	1,550	1	≤ 0.008	4.21	Rick, 2001
USA	2001	1	970	1	N/A	3.59	Rick, 2001
USA	2001	1	1,140	1	N/A	4.86	Rick, 2001
USA	2001	1	980	1	N/A	6.54	Rick, 2001
USA	2001	1	1,800	1	N/A	4.73	Rick, 2001
USA	2001	1	1,790	1	N/A	6.06	Rick, 2001
USA	2001	1	940	1	N/A	2.98	Rick, 2001
USA	2001	1	270	1	N/A	2.50	Rick, 2001
USA	2001	1	290	1	N/A	2.59	Rick, 2001
Germany	2000	1	2,016	1	N/A	12.8	Perkins, 2002

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
England	2000	1	1585	1	N/A	12.3	Perkins, 2002
England	2000	1	1585	1	N/A	14.3	Perkins, 2002
Italy	2001	1	1380	1	N/A	6.18	Perkins, 2002
Italy	2001	1	1380	1	N/A	4.50	Perkins, 2002
England	2002	1	1636	1	N/A	9.21	Bostock, 2002
England	2002	1	1636	1	N/A	14.3	Bostock, 2002
Germany	2002	1	1484	1	N/A	2.92	Blaschke, 2002
Germany	2002	1	1484	1	N/A	5.90	Blaschke, 2002
USA	2002	1	1761	1	N/A	9.16	Barnekow, 2002

Table 33. Residues of Sulfuryl Fluoride in Wheat - Bran after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,720	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	36.7	
				7	N/A	34.0	
USA	2000	1	1,760	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	35.5	
				7	N/A	34.8	
USA	2000	1	1,720	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	37.1	
				7	N/A	34.8	
USA	2000	1	1,730	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	31.9	
				7	N/A	34.5	

Table 34. Residues of Sulfuryl Fluoride in Wheat-Flour after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,360	7	N/A	33.5	Rick, 2000
USA	2000	1	1,480	7	N/A	37.8	Rick, 2000
USA	2000	1	1,140	7	N/A	21.5	Rick, 2000
USA	2000	1	1,110	7	N/A	26.4	Rick, 2000

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,560	7	N/A	25.9	Rick, 2000
USA	2000	1	1,360	7	N/A	25.7	Rick, 2000
USA	2000	1	1,400	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	34.2	
USA	2000	2	3,350	1	0.009	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	62.6	
USA	2000	3	5,040	1	0.008	N/A	Rick, 2000
				4	0.009	N/A	
				7	N/A	93.7	
USA	2000	4	6,950	1	0.015	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	93.2	
USA	2000	1	1,830	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	44.7	
USA	2000	2	3,390	1	0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	62.6	
USA	2000	3	5,240	1	0.009	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	101	
USA	2000	4	7,300	1	0.013	N/A	Rick, 2000
				4	0.008	N/A	
				7	< 0.008	97.1	
USA	2000	1	1,764	7	N/A	31.6	Rick, 2000
USA	2000	1	1,790	7	N/A	33.3	Rick, 2000
USA	2000	1	1,620	7	N/A	35.3	Rick, 2000
USA	2000	1	1,240	7	N/A	28.2	Rick, 2000
USA	2000	1	1,680	7	N/A	39.9	Rick, 2000
USA	2000	1	1,660	7	N/A	43.4	Rick, 2000
USA	2000	1	1,380	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	33.4	
USA	2000	1	1,590	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	37.8	
USA	2000	1	1,720	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	37.1	
				7	N/A	34.8	
USA	2000	1	1,720	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	36.7	
				7	N/A	34.0	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,560	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	14.6	
USA	2000	1	1,360	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	15.7	
USA	2000	1	2,680	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	1.85	
USA	2000	1	2,550	1	0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	2.03	
USA	2000	1	1,180	1	≤ 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	21.1	
USA	2000	1	1,260	1	0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	19.3	
USA	2000	1	240	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	7.34	
USA	2000	1	250	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	6.88	
Germany	2000	1	2,016	1	N/A	55.0	Perkins, 2002
England	2000	1	1585	1	N/A	55.0	Perkins, 2002
Italy	2001	1	1380	1	N/A	40.82	Perkins, 2002
England	2002	1	1636	1	N/A	51.1	Bostock, 2002
Germany	2002	1	1484	1	N/A	28.7	Blaschke, 2002
USA	2002	1	1761	1	N/A	43.0	Barnekow, 2002

Table 35. Residues of Sulfuryl Fluoride in Wheat-Germ after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,410	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	94.2	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	2,440	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	95.8	
USA	2000	1	1,150	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	55.3	
USA	2000	1	1,220	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	58.6	
USA	2000	1	230	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	17.1	
USA	2000	1	240	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	18.1	
USA	2000	1	1,460	7	N/A	73.2	Rick, 2000
USA	2000	1	1,580	7	N/A	66.3	Rick, 2000
USA	2000	1	1,400	7	N/A	83.9	Rick, 2000
USA	2000	1	1,400	7	N/A	84.1	Rick, 2000
USA	2000	1	1,370	7	N/A	41.6	Rick, 2000
USA	2000	1	1,430	7	N/A	43.5	Rick, 2000
USA	2000	1	1,470	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	17.4	
USA	2000	1	1,710	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	18.6	
USA	2000	1	1,480	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	56.8	
				7	N/A	59.0	
USA	2000	1	1,430	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	54.2	
				7	N/A	52.0	
USA	2000	1	1,550	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	72.3	
USA	2000	1	1,540	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	82.6	
USA	2000	1	1,630	7	N/A	104	Rick, 2000
USA	2000	1	1,620	7	N/A	90.3	Rick, 2000
USA	2000	1	1,180	7	N/A	58.8	Rick, 2000
USA	2000	1	1,080	7	N/A	60.1	Rick, 2000
USA	2000	1	2,020	7	N/A	53.7	Rick, 2000

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Dosage (CT) (mg·hr/L)	PFI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2000	1	1,870	7	N/A	54.3	Rick, 2000
USA	2000	1	1,340	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	88.5	
USA	2000	2	3,390	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	158	
USA	2000	3	5,090	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	218	
USA	2000	4	7,010	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	235	
USA	2000	1	1,290	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	81.9	
USA	2000	2	3,520	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	121	
USA	2000	3	5,420	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	201	
USA	2000	4	7,360	1	< 0.008	N/A	Rick, 2000
				4	< 0.008	N/A	
				7	N/A	222	

Table 36. Residues of Sulfuryl Fluoride in Dates after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	210	1	ND	ND	Byrne, 2001
USA	2001	1	210	1	ND	ND	Byrne, 2001

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	1,470	1	0.0072	N/A	Byrne, 2001
				4	N/A	ND	
		2	2,970	1	ND	N/A	
				5	N/A	ND	
		3	4,510	1	0.0065	N/A	
				5	N/A	< 2.40	
		4	6,000	1	0.0140	N/A	
				4	0.0100	< 2.40	
		5	7,500	1	0.0140	N/A	
				2	0.0092	N/A	
				5	0.0120	N/A	
				8	< 0.0055	N/A	
				15	ND	2.74	
USA	2001	1	1,490	1	0.0074	N/A	Byrne, 2001
				4	N/A	ND	
		2	3,000	1	< 0.0021	N/A	
				5	N/A	< 2.40	
		3	4,440	1	0.0050	N/A	
				5	N/A	< 2.40	
		4	5,960	1	0.0100	N/A	
				4	0.0084	< 2.40	
		5	7,440	1	0.0200	N/A	
				2	0.0190	N/A	
				5	0.0058	N/A	
				8	< 0.0042	N/A	
				15	ND	< 2.40	

Table 37. Residues of Sulfuryl Fluoride in Figs after post-harvest application by fumigation (cont.)

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	*F^-*	
USA	2001	1	200	1	0.0046	ND	Byrne, 2001
USA	2001	1	200	1	0.0047	ND	Byrne, 2001
				7	0.0072	ND	
USA	2001	1	1,480	1	0.0400	N/A	Byrne, 2001
				6	N/A	< 2.40	
		2	2,960	1	0.0120	N/A	
				2	0.0090	N/A	
				5	0.0050	< 2.40	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	*F*⁻	
USA	2001	1	1,440	1	0.0340	N/A	Byrne, 2001
				6	N/A	< 2.40	
		2	2,950	1	0.0140	N/A	
				2	< 0.0042	N/A	
				5	N/A	< 2.40	

Table 38. Residues of Sulfuryl Fluoride in Dried Plums after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	*F*⁻	
USA	2001	1	220	N/A	ND	ND	Byrne, 2001
USA	2001	1	220	N/A	ND	ND	Byrne, 2001
USA	2001	1	1,540	1	ND	N/A	Byrne, 2001
				2	N/A	ND	
		2	3,030	1	ND	N/A	
				2	N/A	< 2.40	
		3	4,540	1	< 0.0042	N/A	
				4	N/A	< 2.40	
		4	6,060	1	ND	N/A	
				5	N/A	< 2.77	
USA	2001	1	1,610	1	ND	N/A	Byrne, 2001
				2	N/A	ND	
		2	3,130	1	ND	N/A	
				2	N/A	< 2.40	
		3	4,650	1	< 0.0042	N/A	
				4	N/A	< 2.48	
		4	6,170	1	ND	N/A	
				5	N/A	< 2.40	

Table 39. Residues of Sulfuryl Fluoride in Raisins after post-harvest application by fumigation (cont.)

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*SF*	*F*⁻	
USA	2001	1	220	1	ND	N/A	Byrne, 2001
				5	N/A	ND	
USA	2001	1	220	1	ND	N/A	Byrne, 2001
				5	N/A	ND	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*SF*	*F^-*	
USA	2001	1	2,500	1	< 0.0042	N/A	Hartsell, 2000
				4	ND	N/A	
				7	ND	ND	
		2	5,060	N/A	N/A	N/A	
		3	7,580	1	< 0.0042	N/A	
				4	ND	N/A	
				7	ND	< 2.20	
		4	10,080	N/A	N/A	N/A	
		5	12,600	1	ND	N/A	
				4	ND	N/A	
				7	ND	< 2.20	
USA	2001	1	2,500	1	ND	N/A	Hartsell, 2000
				4	ND	N/A	
				7	ND	ND	
		2	5,040	N/A	N/A	N/A	
		3	7,560	1	ND	N/A	
				4	ND	N/A	
				7	ND	< 2.20	
		4	10,060	N/A	N/A	N/A	
		5	12,580	1	ND	N/A	
				4	ND	N/A	
				7	ND	< 2.20	
USA	2001	1	2,510	4	< 0.0042	< 2.20	Hartsell, 2000
				7	N/A	<2.20	
USA	2001	1	2,530	4	ND	ND	Hartsell, 2000
				7	ND	ND	
USA	2001	1	2,460	4	< 0.0042	< 2.20	Hartsell, 2000
				7	N/A	<2.20	
USA	2001	1	2,530	4	< 0.0042	< 2.20	Hartsell, 2000
				7	ND	ND	
USA	2001	1	2,540	4	ND	< 2.20	Hartsell, 2000
				7	ND	ND	
USA	2001	1	2,530	4	ND	< 2.20	Hartsell, 2000
				7	ND	ND	

Table 40. Residues of Sulfuryl Fluoride in Almonds after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	200	1	0.0116	N/A	Byrne, 2001
				2	< 0.0042	N/A	
				3	N/A	3.22	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*Sulfuryl fluoride (SF)*	*Fluoride ion (F^-)*	
USA	2001	1	200	1	0.0104	N/A	Byrne, 2001
				2	ND	N/A	
				3	N/A	3.60	
USA	2001	1	1,520	1	0.0360	4.28	Byrne, 2001
		2	3,060	1	0.0680	N/A	
				2	0.0440	N/A	
				5	0.0076	7.44	
		3	4,550	1	0.116	N/A	
				5	0.0160	N/A	
				8	< 0.0042	N/A	
				15	ND	9.80	
USA	2001	1	1,560	1	0.0300	5.04	Byrne, 2001
		2	3,100	1	0.0470	N/A	
				2	0.0220	N/A	
				5	N/A	7.31	
		3	4,590	1	0.126	N/A	
				5	0.0150	N/A	
				8	< 0.0042	N/A	
				15	ND	9.22	
USA	2001	1	200	1	0.0190	N/A	Byrne, 2001
				2	0.0075	N/A	
				5	ND	N/A	
				7	N/A	N/D	
USA	2001	1	240	1	0.0120	N/A	Byrne, 2001
				2	< 0.0042	N/A	
				5	ND	N/A	
				7	N/A	N/D	

Table 41. Residues of Sulfuryl Fluoride in Pecans after post-harvest application by fumigation (cont.)

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	*F^-*	
USA	2001	1	200	1	0.0394	N/A	Byrne, 2001
				2	0.0243	N/A	
				5	0.0061	N/A	
				8	ND	ND	
USA	2001	1	200	1	0.0570	N/A	Byrne, 2001
				2	0.0195	N/A	
				5	0.0050	N/A	
				8	ND	< 2.40	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	F^-	
USA	2001	1	1,530	1	2.274	N/A	Byrne, 2001
				6	0.1020	N/A	
				10	0.0160	N/A	
				13	N/A	8.00	
		2	2,990	1	5.040	N/A	
				12	0.0120	N/A	
				13	N/A	16.2	
		3	4,500	1	4.472	N/A	
				2	3.536	N/A	
				5	0.2500	N/A	
				8	0.0680	N/A	
				15	ND	N/A	
				16	N/A	21.0	
USA	2001	1	1,540	1	2.542	N/A	Byrne, 2001
				5	0.1040	N/A	
				10	0.0180	N/A	
				13	N/A	9.14	
		2	2,980	1	4.839	N/A	
				12	0.0150	N/A	
				13	N/A	14.7	
		3	4,490	1	5.158	N/A	
				2	1.5920	N/A	
				5	0.2060	N/A	
				8	0.0570	N/A	
				15	ND	N/A	
				16	N/A	20.7	
USA	2001	1	200	1	1.142	N/A	Byrne, 2001
				2	0.380	N/A	
				5	0.0450	N/A	
				8	0.0058	N/A	
				15	ND	< 2.40	
USA	2001	1	210	1	1.222	N/A	Byrne, 2001
				2	0.459	N/A	
				5	0.0510	N/A	
				8	0.0054	N/A	
				15	ND	< 2.40	

Table 42. Residues of Sulfuryl Fluoride in Pistachios after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PFI[a] (days)	*SF*	*F*⁻	
USA	2001	1	220	1	ND	N/A	Byrne, 2001
				5	N/A	< 2.40	
USA	2001	1	220	1	ND	N/A	Byrne, 2001
				5	N/A	< 2.66	
USA	2001	1	1,520	1	0.270	N/A	Byrne, 2001
				5	0.0240	N/A	
				7	N/A	4.12	
		2	3,020	1	0.0570	N/A	
				5	ND	N/A	
				6	N/A	11.6	
		3	4,540	1	0.0360	N/A	
				2	0.0090	N/A	
				5	ND	15.8	
USA	2001	1	1,610	1	0.285	N/A	Byrne, 2001
				5	0.0230	N/A	
				7	N/A	4.07	
		2	3,130	1	0.0700	N/A	
				5	ND	N/A	
				6	N/A	10.4	
		3	4,650	1	0.0540	N/A	
				2	0.0160	N/A	
				5	ND	15.6	
USA	2001	1	210	1	0.0220	N/A	Byrne, 2001
				2	ND	N/A	
				3	N/A	ND	
USA	2001	1	200	1	0.0140	N/A	Byrne, 2001
				2	ND	N/A	
				3	N/A	< 2.40	

Table 43. Residues of Sulfuryl Fluoride in Walnuts after post-harvest application by fumigation.

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*SF*	*F*⁻	
USA	2001	1	220	1	0.0710	N/A	Byrne, 2001
				5	< 0.0042	N/A	
				7	ND	N/A	
				8	N/A	ND	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*SF*	F^-	
USA	2001	1	210	1	0.0730	N/A	Byrne, 2001
				5	< 0.0042	N/A	
				7	ND	N/A	
				8	N/A	ND	
USA	2001	1	180	1	0.580	N/A	Byrne, 2001
				2	0.320	N/A	
				5	0.0910	N/A	
				8	0.0240	N/A	
				15	ND	< 2.40	
USA	2001	1	180	1	0.625	N/A	Byrne, 2001
				2	0.404	N/A	
				5	0.0980	N/A	
				8	0.0290	N/A	
				15	ND	ND	
USA	2001	1	2,500	1	1.492	N/A	Hartsell, 2000
				4	0.145	N/A	
				7	0.0070	< 2.20	
		2	4,990	N/A	N/A	N/A	
		3	7,520	1	4.764	N/A	
				4	0.863	N/A	
				7	0.211	12.6	
		4	10,040	N/A	N/A	N/A	
		5	12,530	1	5.079	N/A	
				4	1.974	N/A	
				7	0.6110	N/A	
				11	0.1810	22.8	
USA	2001	1	2,500	1	1.577	N/A	Hartsell, 2000
				4	0.102	N/A	
				7	< 0.0042	< 2.20	
		2	4,990	N/A	N/A	N/A	
		3	7,520	1	2.412	N/A	
				4	0.795	N/A	
				7	0.210	15.6	
		4	10,020	N/A	N/A	N/A	
		5	12,530	1	4.410	N/A	
				4	2.164	N/A	
				7	0.720	N/A	
				11	0.246	28.6	

Country	Year	Application Details			Residue (mg/kg)		Dossier ref.
		No.	Cumulative Dosage (CT) (mg·hr/L)	PPI[a] (days)	*SF*	F^-	
USA	2001	1	2,500	1	1.492	N/A	Hartsell, 2000
				4	0.145	N/A	
				7	< 0.007	< 2.20	
		2	5,030	N/A	N/A	N/A	
		3	7,510	1	2.896	N/A	
				4	0.676	N/A	
				7	0.090	12.6	
USA	2001	1	2,500	1	1.577	N/A	Hartsell, 2000
				4	0.102	N/A	
				7	< 0.0042	< 2.20	
		2	5,030	N/A	N/A	N/A	
		3	7,510	1	3.268	N/A	
				4	0.718	N/A	
				7	0.098	15.6	
USA	2001	1	2,490	4	0.245	2.88	Hartsell, 2000
				7	< 0.0042	N/A	
USA	2001	1	2,550	4	0.044	7.00	Hartsell, 2000
				7	0.006	5.72	
USA	2001	1	2,490	4	0.208	7.86	Hartsell, 2000
				7	0.068	9.80	
USA	2001	1	2,530	4	0.252	3.09	Hartsell, 2000
				7	< 0.0042	N/A	
USA	2001	1	2,540	4	0.045	7.20	Hartsell, 2000
				7	0.005	5.82	
USA	2001	1	2,490	4	0.252	3.09	Hartsell, 2000
				7	< 0.0042	N/A	
USA	2001	1	2,550	4	0.883	N/A	Hartsell, 2000
				7	0.419	N/A	
				11	N/A	4.48	
USA	2001	1	2,570	4	1.615	N/A	Hartsell, 2000
				7	0.609	N/A	
				11	N/A	5.98	
USA	2001	1	2,490	4	1.642	N/A	Hartsell, 2000
				7	0.470	N/A	
				11	N/A	5.54	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

At present, sulfuryl fluoride is only used on stored (i.e. post-harvest) food commodities.

Fluoride anion does not degrade further under conditions that would be encountered in normal agricultural and physical processing. However, fluoride ion would be subject to extraction by a polar solvent such as water, resulting in the removal of the residue from the matrix when the water was removed. This removal of fluoride ion is the basis of the analytical methods used for quantifying the amount of fluoride ion in fumigated commodities.

In processing

The Meeting received information on the fate of incurred residues of sulfuryl fluoride during the processing of wheat/maize (Rick, 2000, Report 001103). A whole grain wheat and kernel maize residue trial was conducted in Michigan, USA to generate whole grain wheat and kernel maize for a processing study. ProFume® was applied post-harvest as a neat gas fumigant at the dose 1787 mg·hr/L and, 1565 mg·hr/L, to the harvested grains, wheat and maize, respectively. Application was made by introduction of the gas fumigation into the sealed structure. The fumigated maize and wheat grain samples were shipped immediately after collection by overnight delivery to the processing facility at Texas A&M University. Sample processing was initiated upon receipt using Texas A&M. The processing scheme is outlined in Figures 3, 4 and 5.

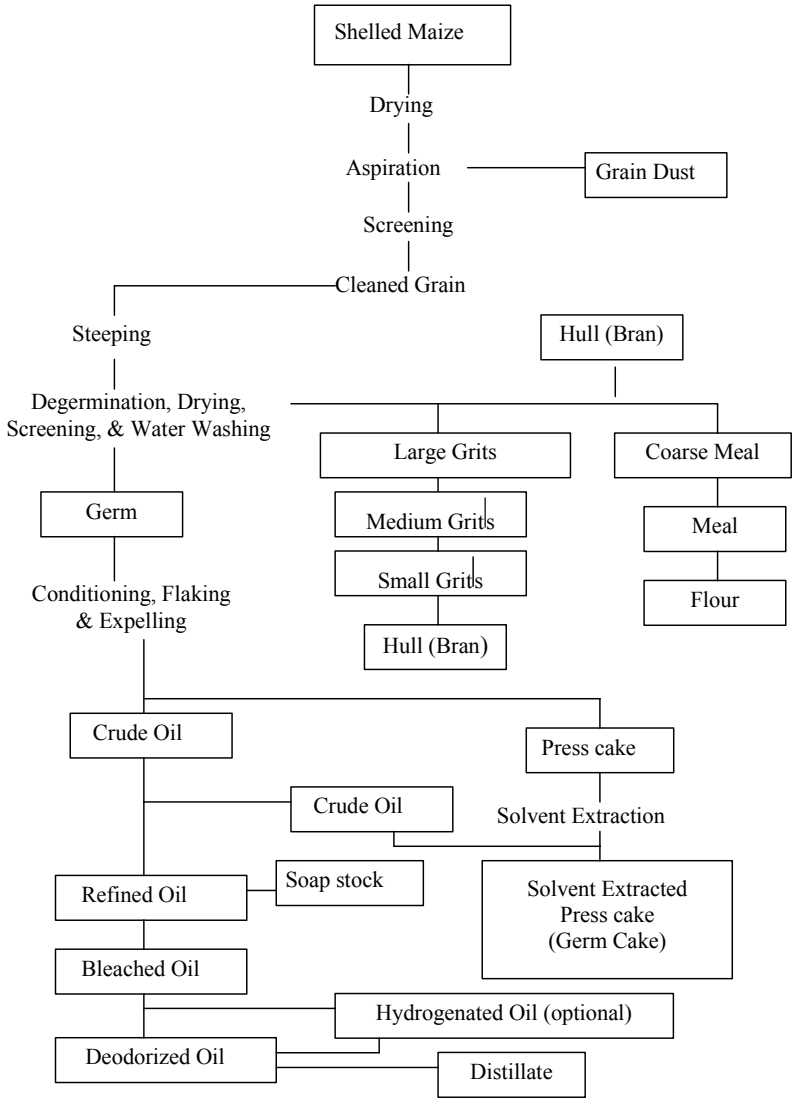


Figure 3. Schematic diagram of the processing of maize (dry milling) (Rick, 2000, Report 001103)

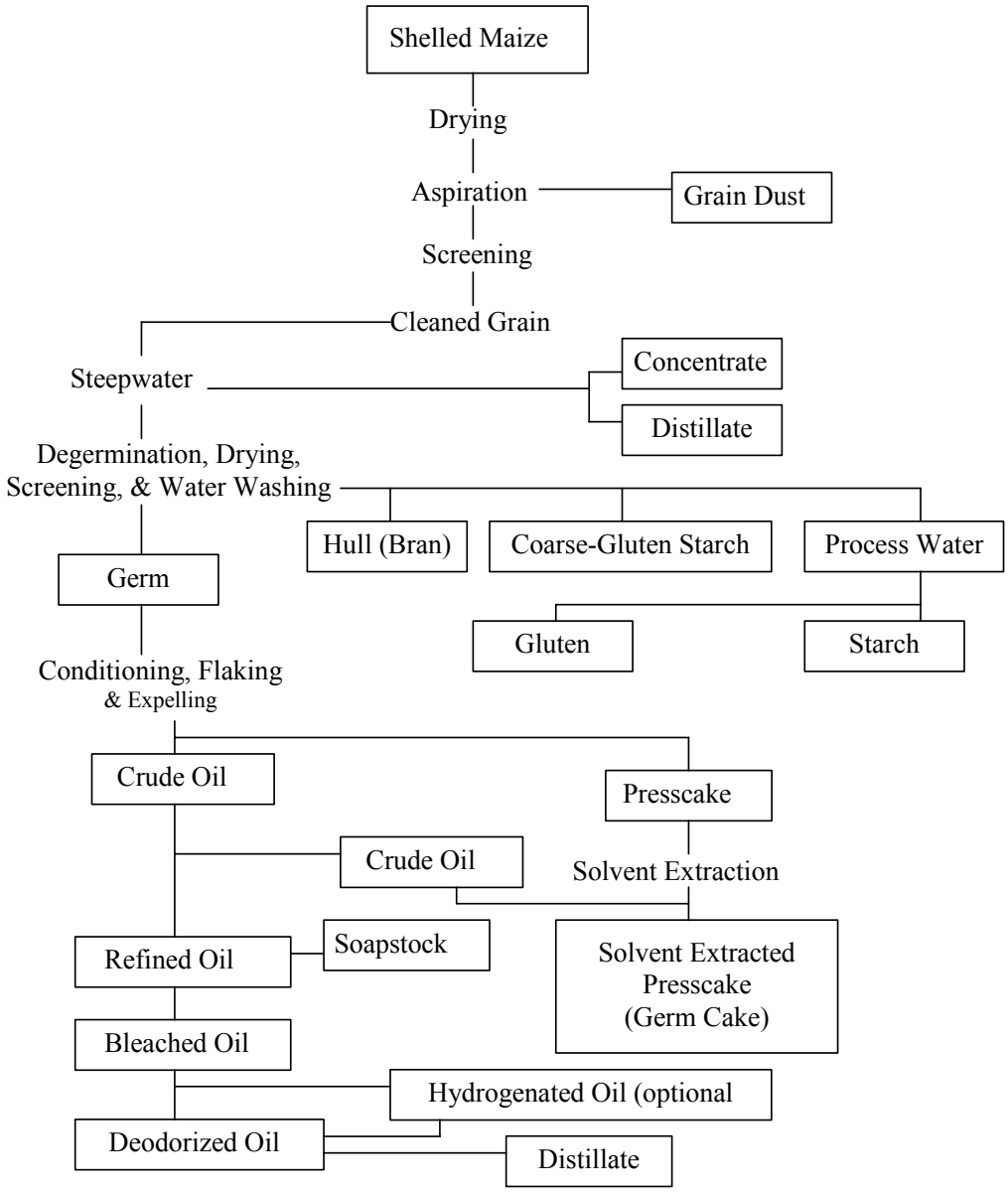


Figure 4. Schematic diagram of the processing of maize (wet milling) (Rick, 2000, Report 001103)

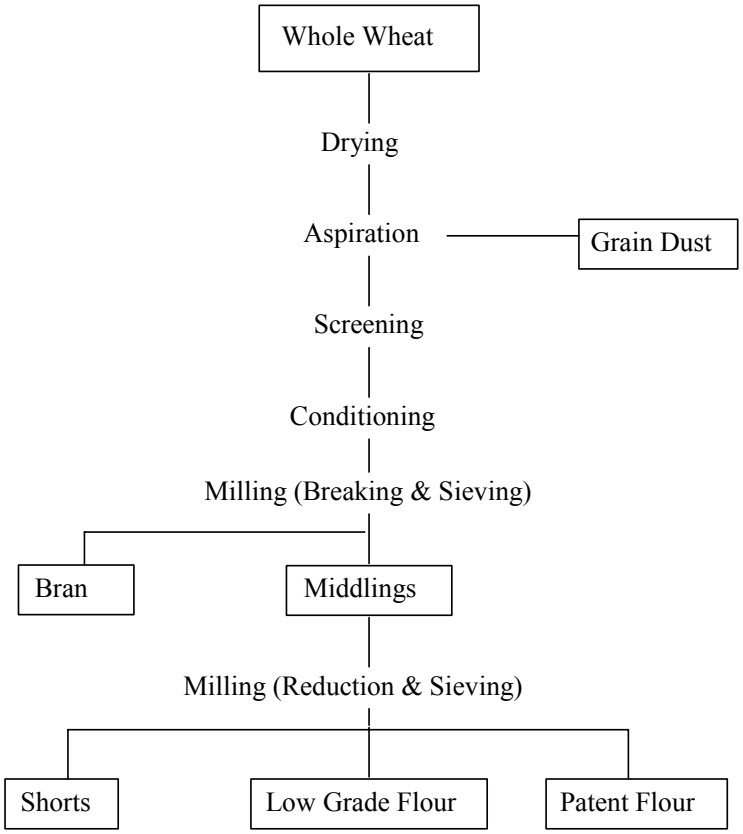


Figure 5: Schematic diagram of the processing of wheat (Rick, 2000, Report 001103)

Processed fractions were frozen as they were generated and all processed samples were shipped by overnight delivery for analysis. Control and treated samples of wheat flour, shorts, bran, middlings, impurities and germ, and maize flour, meal, grits, oil impurities, oil wet and starch (wet) were analyzed. The limit of quantitation levels were 0.6 mg/kg for whole wheat grain and maize grain, 0.3 mg/kg for wheat flour, shorts, middlings and impurities, and maize meal, grits and oil, and 0.8 mg/kg for wheat germ and bran, and maize impurities, and 0.4 mg/kg for maize starch and 0.5 mg/kg for maize flour. The fluoride concentrations measured in whole grains and processed products from bulk exposure and subsequent commodity processing are summarized in Tables 44 and 45 for wheat and maize, respectively. The fluoride concentration determined in the whole grain wheat prior to shipment to Texas A&M University for processing was 1.19 mg/kg. Following processing, the processed wheat products were reanalysed for fluoride to evaluate the effect of processing on residue levels. The concentrations ranged from 0.446 mg/kg for wheat flour (37.5% of initial value) to 5.74 mg/kg for wheat germ (482% of initial value). The only wheat commodity besides germ that showed a significant increase in fluoride levels was the bran that had fluoride levels of 3.05 mg/kg (256% of initial value). The elevated values in bran and germ are an indication that the fluoride anion selectively accumulates in those wheat fractions (possibly because the sulfuryl fluoride accumulates to a greater degree in those relatively oily fractions during fumigation where it then becomes converted to the immobile fluoride anion).

The fluoride concentration determined in whole maize prior to processing was 1.76 mg/kg (Table 45). After processing, quantifiable concentrations of fluoride in maize fractions ranged from 0.826 mg/kg for maize grits (46.9% of initial value) to 9.67 mg/kg for impurities (549% of initial value). Impurities are considered to be similar to the aspirated grain fraction. Fluoride levels were below the limit of quantitation (0.3 mg/kg) for maize oil and wet-milled cornstarch.

The processing of sulfuryl fluoride-fumigated whole grain wheat containing fluoride ion at a concentration of 1.19 mg/kg yielded flour, shorts, bran, middlings, impurities, and germ containing fluoride at concentrations of 0.446, 1.50, 3.05, 0.718, 1.07, and 5.74 mg/kg, respectively. The elevated fluoride levels in wheat germ and wheat bran indicates that fluoride selectively accumulates in those grain fractions. The processing of fumigated whole grain maize containing fluoride ion at a concentration of 1.76 mg/kg produced flour, meal, grits, impurities, containing fluoride ion at concentrations of 1.29, 1.37, 0.826, and 9.67 mg/kg. Thus, the impurities were the only corn fraction containing elevated levels of fluoride. Fluoride was below the limit of quantitation (0.3 mg/kg) in maize oil (dry- and wet-milled) and wet-milled starch.

Table 44. Comparison of Fluoride Residue Levels among Various Grain Products Processed from Fumigated Whole Grain Wheat (Rick, 2000, Report 001103)

Wheat Commodity	Fluoride Concentration [a] Control Commodity (ug/g)	Fluoride Concentration [a] Exposed Commodity [b] (ug/g)	% of Initial Fluoride Conc. (%)
** **Whole Grain**	<LOQ (0.6) [c]	**1.19**	--
Whole Grain	<LOQ (0.6)	1.30	109
Flour	<LOQ (0.3)	0.446	37.5
Shorts	0.339	1.50	126
Bran	<LOQ (0.8)	3.05	256
Middlings	<LOQ (0.3)	0.718	60.3
Impurities	0.306	1.07	89.9
Germ	<LOQ (0.8)	5.74	482

** Fluoride Concentration measured in grain sampled prior to shipment to Texas A&M.

[a] Each value represents the average of two measurements per commodity.

[b] Fluoride concentrations for the exposed samples represent net values; if the fluoride level in the control sample of a commodity was greater than the LOQ, the control fluoride concentration was subtracted from the total fluoride concentration determined for the exposed commodity.

[c] <LOQ = Less than Limit of Quantiation; Limit of Quatitation for each commodity shown in parentheses.

Table 45. Comparison of Fluoride Residue Levels among Various Grain Products Processed from Fumigated Whole Maize (Rick, 2000, Report 001103)

	Corn Commodity	Fluoride Concentration [a] Control Commodity (ug/g)	Fluoride Concentration [a] Exposed Commodity [b] (ug/g)	% of Initial Fluoride Conc. (%)
**	**Whole Grain**	<LOQ (0.6) [c]	**1.76**	--
	Whole Grain	<LOQ (0.6)	1.89	107
	Flour	0.493	1.29	73.3
	Meal	<LOQ (0.3)	1.37	77.8
	Grits	<LOQ (0.3)	0.826	46.9
	Oil	<LOQ (0.3)	<LOQ (0.3)	NA
	Impurities	<LOQ (0.8)	9.67	549
	Oil (wet)	<LOQ (0.3)	<LOQ (0.3)	NA
	Starch (wet)	0.403	<LOQ (0.3)	NA

** Fluoride Concentration measured in grain sampled prior to shipment to Texas A&M.

[a] Each value represents the average of two measurements per commodity.

[b] Fluoride concentrations for the exposed samples represent net values; if the fluoride level in the control sample of a commodity was greater than the LOQ, the control fluoride concentration was subtracted from the total fluoride concentration determined for the exposed commodity.

[c] <LOQ = Less than Limit of Quantiation; Limit of Quatitation for each commodity shown in parentheses.

RESIDUES IN ANIMAL COMMODITIES

No data or information was supplied.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data or information was supplied.

NATIONAL MAXIMUM RESIDUE LIMITS

National tolerances have been established by USA for the parent residue (sulfuryl fluoride) and degradate (fluoride). Tables 46 and 47 provide information on existing national MRLs. The information was supplied by the manufacturer.

Table 46. List of National MRLs: Fluoride

Country	Commodity	Approved MRL (mg/kg)
United States[1]	Barley bran, post-harvest	45.0
	Barley flour, post-harvest	45.0
	Barley grain, post-harvest	15.0
	Barley pearled, post-harvest	45.0

	Maize aspirated grain fractions, post-harvest	55.0
	Maize flour, post-harvest	35.0
	Maize grain, post-harvest	10.0
	Maize grits, post-harvest	10.0
	Maize meal, post-harvest	30.0
	Maize pop grain, post-harvest	10.0
	Fruit dried, post-harvest (other than raisin)	3.0
	Grape raisin, post-harvest	7.0
	Millet grain, post-harvest	40.0
	Nut tree, post-harvest	10.0
	Oat flour, post-harvest	75.0
	Oat grain, post-harvest	25.0
	Oat rolled, post-harvest	75.0
	Pistachio, post-harvest	10.0
	Rice bran, post-harvest	31.0
	Rice grain, post-harvest	12.0
	Rice hulls, post-harvest	35.0
	Rice polished, post-harvest	25.0
	Rice wild grain, post-harvest	25.0
	Sorghum grain, post-harvest	40.0
	Triticale grain, post-harvest	40.0
	Wheat bran, post-harvest	40.0
	Wheat flour, post-harvest	125.0
	Wheat germ, post-harvest	130.0
	Wheat grain, post-harvest	40.04
	Wheat milled byproducts, post-harvest	130.0
	Wheat shorts, post-harvest	40.0

[1] U.S Code of Federal Regulations Sec. 180.145 Fluorine compounds; tolerances for residues.

Table 47. List of National MRLs: Sulfuryl Fluoride

Country	Commodity	Approved MRL (mg/kg)
United States[2]	Barley bran, post-harvest	0.05
	Barley flour, post-harvest	0.05
	Barley grain, post-harvest	0.1
	Barley pearled, post-harvest	0.05
	Maize aspirated grain fractions, post-harvest	0.05
	Maize flour, post-harvest	0.01
	Maize grain, post-harvest	0.05
	Maize grits, post-harvest	15.0
	Maize meal, post-harvest	0.01
	Maize pop grain, post-harvest	0.05
	Fruit dried, post-harvest	0.05
	Millet grain, post-harvest	0.1
	Nut tree, post-harvest	3.0
	Oat flour, post-harvest	0.05
	Oat grain, post-harvest	0.1
	Oat rolled, post-harvest	0.1
	Pistachio, post-harvest	3.0
	Rice bran, post-harvest	0.01
	Rice grain, post-harvest	0.04
	Rice hulls, post-harvest	0.1
	Rice polished, post-harvest	0.01
	Rice wild grain, post-harvest	0.05
	Sorghum grain, post-harvest	0.1
	Triticale grain, post-harvest	0.1
	Wheat bran, post-harvest	0.05
	Wheat flour, post-harvest	0.05
	Wheat germ, post-harvest	0.02
	Wheat grain, post-harvest	0.1
	Wheat milled byproducts, post-harvest	0.05
	Wheat shorts, post-harvest	0.05

[2] U.S Code of Federal Regulations. Sec. 180.575 Sulfuryl fluoride compounds; tolerances for residues.

APPRAISAL

Sulfuryl fluoride is a post-harvest and structural fumigant for controlling a wide range of insect pests. Sulfuryl fluoride penetrates the insect's body through inhalation in actively respiring life stages or diffusion into the egg. It is a non-specific target poison acting by disrupting the glycolysis and citric acid cycles, thereby depriving the insect of the necessary energy for survival. Upon sulfuryl fluoride entering a target organism it is broken down to the insecticidally active fluoride anion which then inhibits the insect's metabolism. It is being evaluated for the first time by the 2005 Joint Meeting on Pesticide Residues.

Animal Metabolism

No adequate animal metabolism study for sulfuryl fluoride was available.

Degradation in stored products

The metabolism/degradation of ^{35}S- sulfuryl fluoride was studied after fumigation of a variety of food items.

Wheat flour was fumigated with ^{35}S- sulfuryl fluoride at 32 mg/L in a fumigation chamber under reduced pressure for 92 h at room temperature. The insoluble flour residue remaining after 80% ethanol extraction retained 24% of the radioactivity. Radiolabeled residues were characterized as anionic, and some of the radiolabeled residue was characterized as amino acids or soluble polypeptides. Sulfate is formed as a result of conventional hydrolysis of sulfuryl fluoride. This reaction proceeds stepwise, first to fluorosulfonic acid and then to the sulfate anion. An additional product of the breakdown of sulfuryl fluoride is inorganic fluoride.

Seven food items contained in open cups were fumigated with sulfuryl fluoride at 36 and 360 mg/L for 20 h in a chamber of 4.2 m^3 volume. The food items included unbleached enriched wheat flour, dry dog food, non-fat dry milk, vegetable cooking oil, dried beef, Red Delicious Washington apples and snack cakes. Fluoride and sulfate residue levels were analysed at 1, 8, and 15 days after the treatment for both fumigation concentrations.

After the exposure to sulfuryl fluoride at 36 mg/L, fluoride residues found on the seven commodities ranged from approximately nil (for vegetable oil) to 170 mg/kg (for dried beef) at day one; 215 mg/kg (for dried beef) at day eight; and 216 (for dried beef) at day fifteen. Sulfate residues found on the seven commodities were up to 106 mg/kg at day one, 160 mg/kg at day eight and 189 mg/kg at day fifteen.

After exposure to sulfuryl fluoride at 360 mg/L, fluoride residues found on the seven commodities were up to 1300 mg/kg in dried beef at day one, 1200 mg/kg at day eight and 1200 mg/kg at day fifteen.

Any unreacted sulfuryl fluoride present in the matrix degrades to fluoride and sulfate as the terminal residues.

Environmental fate

Sulfuryl fluoride is a structural fumigant used only for post harvest treatment. Since there are no uses on agriculture crops, an environmental fate study is not applicable.

Methods of analysis

The Meeting received separate methods for the analysis of sulfuryl fluoride and fluoride anion. It was concluded that adequate analytical methods exist both for the monitoring/enforcement of MRLs and for data gathering in fumigation facilities.

Gas chromatography with electron capture detection is suitable for the determination of sulfuryl fluoride residues in dried fruits, tree nuts, maize, wheat and rice commodities. A limit of quantification (LOQ) of 0.008 mg/kg was typically achieved.

The method for the analysis of fluoride anion uses aqueous extraction followed by use of a fluoride selective electrode. This method is suitable for the determination of fluoride in cereal grains, dried fruits and tree nuts. An LOQ of 0.2–2.4 mg/kg was typically achieved for fluoride ion.

No analytical methods were developed for animal tissue matrices.

Stability of pesticide residues in stored analytical samples

Residues of fluoride in maize, wheat grain, raisin, walnut, and maize meal are considered to be stable when stored at room temperature for at least 35 days, and when stored frozen at approximately –20°C for at least 138 days. The exception is for wheat flour, which is stable for at least 104 days. No data on storage stability for sulfuryl fluoride was provided.

Definition of the residue

The degradation of sulfuryl fluoride results in the formation of sulfate and inorganic fluoride. Sulfate residues resulting from the degradation of sulfuryl fluoride are insignificant in comparison to naturally occurring levels.

Residue data revealed that sulfuryl fluoride could be present in a commodity following the 24 h aeration period. The measured levels of sulfuryl fluoride in small grains, grain process fractions, and in dried fruit were extremely low, except for maize oil. The sulfuryl fluoride retained on tree nuts was higher, but declined rapidly with time. With the possible presence of sulfuryl fluoride on a commodity following the 24 h aeration, sulfuryl fluoride was considered as suitable for monitoring purposes. Fluoride is ubiquitous in the environment and is not suitable as a residue for enforcement purposes.

Adequate analytical methods exist for the determination of fluoride and sulfuryl fluoride.

The Meeting concluded that the residue definition for monitoring/enforcement is “sulfuryl fluoride”, and for dietary intake considerations “sulfuryl fluoride and fluoride ion” measured separately.

Results of the supervised trials on crops

Fumigation treatments in the supervised trials for cereals, dried fruits and tree nuts summarized in the following paragraphs represent a wide range of treatment rates, calculated as the product of fumigant Concentration (C) x Exposure Time (T) or CTP with either single or multiple applications, and residues determined at different PFIs (Post-Fumigation Intervals). Based on the maximum cumulative CTP of 1500 (or 1,500 gram-hours per cubic meter given as g·h/m^3 or mg·h/L), and the consideration of allowing a $\pm$ 25% GAP variation, residues generated from a single application at $\pm$ 25 GAP (1125–1875 CTP, or 1,125-1,875 mg·h/L or g·h/ m^3) will be used for MRL estimation and dietary risk assessment. Since sulfuryl fluoride (SF) residues are rapidly degraded to F^- (fluoride ion) after 24 h and the latter is stable in treated commodities, the Meeting decided that SF residues collected from 1 day PFI, and F^- residues collected at all PFIs would be used for MRL, STMR and HR estimations.

Cereals

The U.S. GAP specifies that for stored product pests a particular plant will be fumigated on a schedule from 3 times per year to once every few years at the maximum cumulative CTP of 1500 g h/m^3, and the maximum cumulative CTP for vacuum fumigation of 200 g h/m^3. In practice, stored cereal grains are likely to receive only one fumigation treatment. Sulfuryl fluoride residues reported in the following paragraphs were all from 1-day PFI, and were analysed immediately; fluoride residues were the highest residues from each sample irrespective of the PFI.

Barley

In trials matching US GAP conducted in the USA, England, Germany, and Italy the sulfuryl fluoride (SF) residues were < 0.008 (4) mg/kg. Fluoride ion (F^-) residues in ranked order were: 2.8, 2.8, 3.1, 6.5, 7.1, 8.0, 10, 12, 18, 18, and 21 mg/kg.

Maize

In trials matching US GAP conducted in the USA the SF residues were: < 0.008 (7), 0.02(2), and 0.03 mg/kg. F^- residues in ranked order were: 0.8, 0.9, 1.0, 1.2, 1.3, 1.4(4), 1.5, 1.6, 1.7, 1.9 and 2.3(3) mg/kg.

Oat

In seven trials matching US GAP conducted in the USA, SF residues were < 0.008 (4) mg/kg. F^- residues in ranked order were: 7.0, 7.4, 7.5, 8.3, 9.2, 12, and 14 mg/kg.

Rice

In 19 trials matching US GAP conducted in the USA, England, Italy, and Germany SF residues were < 0.008 (8) mg/kg. F^- residues were: 1.8, 2.0, 2.0, 2.0, 2.2, 2.2, 2.2, 2.4, 2.8, 3.6, 5.5, 6.2, 7.0, 7.3, 7.6, 7.9, 8.4, 11, and 15 mg/kg.

Wheat

In 52 trials matching US GAP conducted in the USA, England, Italy, and Germany, SF residues in ranked order, were: < 0.008 (13), 0.01(3) and 0.03(2) mg/kg. F^- residues in ranked order were: 1.5, 1.8, 1.9(2), 2.0(3), 2.1(2), 2.2(2), 2.4, 2.6, 2.7, 2.9(2), 3.3, 3.4, 3.8, 3.9(2), 4.2, 4.5, 4.7, 4.8(3), 4.9, 5.0, 5.7, 5.8, 5.9, 6.1, 6.2, 9.2(2), 12 and 14(2) mg/kg.

The Meeting noted that maize, rice, and wheat along with barley and oats represent major commercial cereal grain commodities. Since residues of sulfuryl fluoride and fluoride ion among the five cereal commodities are comparable (< 0.008 – 0.03 mg/kg for sulfuryl fluoride, and 0.8 – 21 mg/kg for fluoride ion), a group MRL and STMR may be estimated for cereal grains. Overall, a total of 44 SF residues in ranked order, were: < 0.008 (36), 0.01(3), 0.02(2) and 0.03(3) mg/kg. A total of 92 F^- residues in ranked order were: 0.8, 0.9, 1.0, 1.2, 1.3, 1.4(4), 1.5(2), 1.6, 1.7, 1.8(2), 1.9(3), 2.0(6), 2.1(2), 2.2(5), 2.3(3), 2.4(2), 2.6, 2.7, 2.8(3), 2.9(2), 3.1, 3.3, 3.4, 3.6, 3.8, 3.9(2), 4.2, 4.5, 4.7, 4.8(2), 4.8, 4.9, 5.0, 5.5, 5.7, 5.8, 5.9, 6.1, 6.2(2), 6.5, 7.0(2), 7.1, 7.3, 7.4, 7.5, 7.6, 7.9, 8.0, 8.3, 8.4, 9.2(3), 10, 11, 12(3), 14(3), 15, 18(2) and 21 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an HR of 0.03 mg/kg, and an STMR of 0.008 mg/kg for sulfuryl fluoride; and estimated an HR of 21 mg/kg and an STMR of 3.5 mg/kg for fluoride ion for cereal grains.

Cereal grain milling fractions and milled cereal products

Maize flour

In trials matching US GAP conducted in the USA, England, Germany, and Italy the SF residues were < 0.008 (2) mg/kg. F^- residues in ranked order were: 14, 19(2), 24, 37, 56 and 70 mg/kg.

Maize meal

Two trials were conducted in the USA at higher than US GAP rate. SF residues were < 0.008 mg/kg; F^- residues were 5.6 and 6.3 mg/kg.

Rice bran

Two trials were conducted in the USA at higher than US GAP rate. SF residues were < 0.008 mg/kg; F^- residues were 24.2 and 28.5 mg/kg.

Rice, polished

Two trials were conducted in the USA at higher than US GAP rate. SF residues were < 0.008 mg/kg; F^- residues were 1.5 and 1.6 mg/kg.

Wheat bran

In four trials matching US GAP conducted in the USA the SF residues were < 0.008 mg/kg (4). F^- residues in ranked order were 34, 36 and 37(2) mg/kg.

Wheat flour

In 32 trials matching US GAP conducted in the USA, England, Germany, and Italy, SF residues in ranked order were all < 0.008 (10) mg/kg. F^- residues in ranked order were: 15, 16, 19, 21, 22, 26, 26, 28, 29, 33, 34(2), 35, 37(2), 38(2), 40, 41, 43(2), 45, 51 and 55 mg/kg.

Wheat germ

In 20 trials matching US GAP conducted in the USA, SF residues were all < 0.008 (10) mg/kg. F^- residues were: 17, 19, 42, 44, 54, 55, 59(3), 66, 72, 73, 82, 83, 84 (2) 88, 90 and 104 mg/kg.

Residue data was insufficient for estimating maximum residue levels and STMRs for rice bran, rice polished, maize starch, maize meal, maize grits, and rice bran individually. However, group MRLs and STMRs may be estimated for the members of the Codex commodity group's cereal grain milling fractions and milled cereal products, utilizing the data from rice bran and rice polished trials for milled cereal products; and maize flour, maize meal, wheat bran, wheat flour and wheat germ trials for cereal grain milling fractions.

Overall, a total of 30 SF residues in ranked order, were: < 0.008 (29) and 0.06 mg/kg. A total of 58 F^- residues in ranked order were: 3.9, 5.4, 5.6, 14, 15, 16, 17, 19(4), 21, 22, 24(2), 26(2), 28, 29, 33, 34(3), 35, 36, 37(5), 38(2), 40, 41, 42, 43(2), 44, 45, 51, 54, 55(2), 56, 59(3), 66, 70, 72, 73, 82, 83, 84(2), 88, 90.3 and 104 mg/kg. The Meeting estimated maximum residue levels of 0.1 mg/kg, HRs of 0.06 mg/kg, and STMRs of 0.008 mg/kg for sulfuryl fluoride in cereal grain milling fractions and milled cereal products; and estimated an HR of 104 mg/kg and a STMR of 37 mg/kg for fluoride in both cereal grain milling fractions and milled cereal products.

Dried Fruits

For fumigation of stored dried fruits and tree nuts commodities the U.S. EPA specifies that the maximum cumulative CTP is 1500 g h/m^3 and the maximum cumulative CTP for vacuum fumigation is 200 g h/m^3. In practice, stored dried fruits can be treated as many as four times with fumigation at a maximum cumulative CTP of 1500 g h/m^3. Sulfuryl fluoride residues reported in the following paragraphs were all from a 1 day PFI, analysed immediately after sample collection; F^- residues were the highest residues from each sample.

Dates

In two trials matching US GAP conducted in the USA, SF residues were 0.007(2) mg/kg. F^- residues were not detected (< 2.4 mg/kg).

Figs

In two trials matching US GAP conducted in the USA, SF residues were 0.03 and 0.04 mg/kg. F^- residues were < 2.4(2) mg/kg.

Dried plum

In two trials matching US GAP conducted in the USA, SF residues were not detected (< 0.0042 mg/kg). F^- residues were also not detected (< 2.4 mg/kg LOQ).

Raisin

Eight post-harvest fumigation trials conducted in the USA were all at above GAP rates. SF residues were not detected (< 0.0042 mg/kg) (4) mg/kg. F^- residues were below the LOQ: < 2.2 mg/kg (2) and < 2.4(2) mg/kg.

Data was insufficient to estimate maximum residue levels or STMRs on each commodity individually; however, since residues of sulfuryl fluoride and fluoride ion on each commodity from limited fumigation trials are comparable and consistent (< 0.0042 – 0.04 mg/kg for sulfuryl fluoride, and < 2.2 – < 2.4 mg/kg for fluoride ion), a crop group MRL and STMR may be estimated. SF residues from the four dried fruits were < 0.004 (6), 0.007(2), 0.03 and 0.04 mg/kg. F^- residues were: < 2.2(2) and < 2.4 (8) mg/kg. The Meeting estimated an MRL of 0.06 mg/kg, an HR of 0.04 mg/kg, and an STMR of 0.004 mg/kg for sulfuryl fluoride; and estimated an HR of 2.4 mg/kg and an STMR of 2.4 mg/kg for fluoride ion in dried fruits.

Tree Nuts

For fumigation of stored dried fruits and tree nuts commodities the U.S. EPA specifies that the maximum cumulative CTP is 1500 g h/m^3 and the maximum cumulative CTP for vacuum fumigation is 200 g h/m^3. In practical terms, stored tree nuts can receive as many as four fumigation treatments at a maximum cumulative CTP of 1500 g h/m^3. Sulfuryl fluoride residues reported in the following paragraphs were all from a 1 day PFI, analysed immediately after sample collection; fluoride ion residues were the highest residues from each sample.

Almonds

In four trials matching US GAP conducted in the USA, SF residues were 0.01, 0.02, 0.03, 0.04 mg/kg. F^- residues were: < 2.4(2), 4.3 and 5.0 mg/kg.

Pecans

In four trials matching US GAP conducted in the USA, SF residues were: 1.1, 1.2, 2.3 and 2.5 mg/kg. F^- residues were < 2.4(2), 8.0 and 9.1 mg/kg.

Pistachios

In four trials matching US GAP conducted in the USA, SF residues were: 0.01, 0.02, 0.27, 0.29 mg/kg. F^- residues were < 2.4(2) and 4.1(2) mg/kg.

Walnuts

In two trials at above US GAP conducted in the USA, SF residues were 0.58 and 0.63 mg/kg. F^- residues were < 2.4(2) mg/kg.

Since residues of sulfuryl fluoride and fluoride ion on the four commodities tested are comparable (0.01 – 2.5 mg/kg for sulfuryl fluoride, and < 2.4 – 9.1 mg/kg for fluoride ion), a crop group MRL and STMR may be estimated. Overall, SF residues from the four tree nuts commodities, in ranked order, were 0.01(2), 0.02(2), 0.03, 0.04, 0.27, 0.29, 0.58, 0.63, 1.1, 1.2, 2.3 and 2.5 mg/kg. F^- residues from the four tree nuts commodities, in ranked order, were: < 2.4 (8), 4.1)2), 4.3, 5.0, 8.0 and 9.1 mg/kg. The Meeting estimated a maximum residue level of 3.0 mg/kg, an HR of 2.5 mg/kg and an STMR of 0.28 mg/kg for sulfuryl fluoride; and estimated an HR of 9.1 mg/kg and an STMR of 2.4 mg/kg for fluoride for tree nuts except coconuts.

Fate of residues in storage and processing

In storage

Sulfuryl fluoride rapidly degrades to fluoride under typical GAP conditions. No significant decline in the residue of fluoride was observed for the maize grain and wheat grain for 138 days, raisin and walnut for 141 days, and maize meal for 140 days of storage after treatment with sulfuryl fluoride. At present, sulfuryl fluoride is only registered for use on stored (i.e. post-harvest) food commodities. Sulfuryl fluoride is unstable and readily desorbs from the commodity or degrades under storage conditions, yielding fluoride and sulfate as the terminal residues.

In processing

Post-harvest fumigation on whole grain wheat and kernel maize was conducted to determine the fate of incurred residues of sulfuryl fluoride during the processing of the grain. Whole grain wheat and kernel maize were fumigated at 1787 mg·h/L and 1565 mg·h/L, respectively. The fumigated grain samples were then processed, and the control and treated processed samples, wheat flour, shorts, bran, middlings, impurities and germ, and maize flour, meal, grits, oil impurities, oil wet and starch (wet) were analysed. The LOQs were 0.6 mg/kg (fluoride) for whole wheat grain and maize grain; 0.3 mg/kg for wheat flour, shorts, middlings and impurities, maize meal, grits and oil; and 0.8 mg/kg for wheat germ and bran, and maize impurities; 0.4 mg/kg for maize starch; and 0.5 mg/kg for maize flour.

The processing of sulfuryl fluoride-fumigated whole grain wheat, containing fluoride ion at a concentration of 1.19 mg/kg, yielded flour, shorts, bran, middlings, impurities and germ containing fluoride at concentrations of 0.45, 1.50, 3.05, 0.72, 1.07, and 5.74 mg/kg, respectively. The elevated fluoride ion levels in wheat germ and wheat bran indicate that fluoride ion selectively accumulates in those grain fractions. The processing of fumigated whole grain maize, containing fluoride ion at a concentration of 1.76 mg/kg, produced flour, meal, grits and impurities containing fluoride ion at concentrations of 1.29, 1.37, 0.83, and 9.67 mg/kg, respectively. Thus, the maize impurities were the only fraction where it appears that fluoride ion concentrates.

Supervised fumigation trials conducted in food storage facilities on processed cereal grain commodities resulted in higher fluoride residues than those from processing studies, where the whole grains were fumigated and then processed. As a consequence, the higher residues values (HR) are derived from direct treatment rather than from the processing of the raw agricultural products, viz grains.

Farm animal feeding studies

No animal feeding studies were submitted.

Farm animal dietary burden

The Meeting considered the dietary burden for fluoride resulting from feeding treated commodities to dairy cattle and poultry. No animal dietary burden for sulfuryl fluoride could be estimated since no data was submitted.

Table 48. Estimated maximum dietary burden of farm animals

Commodity	Group	HR (mg/kg)	Basis of residue	% Dry matter	Residue dw mg/kg	*Diet content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Barley-grain	GC	21	HR	88	23.9	*50*	*40*	*75*	12.0	9.6	17.9
Maize – whole kernel	GC	2.3	HR	88	2.6						
Oats	GC	14	HR	89	15.7						
Rice	GC	14.6	HR	88	16.6						
Wheat – whole grain	GC	14.3	HR	89	16.1						
Wheat-bran	CF	37.1	HR	88	42.2	*40*	*50*	*25*	16.9	21.1	10.6
TOTAL						*90*	*90*	*100*	28.9	30.7	28.5

Table 49. Estimated median dietary burden of farm animals

Commodity	Group	Residue (mg/kg)	Basis of residue	% Dry matter	Residue, dw mg/kg	*Diet content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cows*	*Poultry*	Beef cattle	Dairy cows	Poultry
Barley-grain	GC	8.0	STMR	88	9.1						
Maize – whole kernel	GC	1.4	STMR	88	1.6						
Oats	GC	8.3	STMR	89	9.3	*50*	*40*	*80*	4.7	3.7	7.4
Rice	GC	3.6	STMR	88	4.1						
Wheat – whole grain	GC	3.9	STMR	89	4.4						
Wheat-bran	CF	36.1	STMR	88	41.0	*40*	*50*	*20*	16.4	20.5	8.2
TOTAL						*90*	*90*	*100*	21.1	24.2	15.6

The calculated dietary burden for estimation of maximum residue level was 28.9 ppm for beef cattle, 30.7 ppm for dairy cattle and 28.5 ppm for poultry. The calculated dietary burden of fluoride for estimation of STMR level was 21.1 ppm for beef cattle, 24.2 ppm for dairy cattle and 15.6 ppm for poultry. No recommendation for maximum residues level in animals could be made since adequate feeding studies were not submitted.

RECOMMENDATIONS

On the basis of the data from supervised trials and processing studies, the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for compliance with MRLs in plant commodities: *sulfuryl fluoride*; and for dietary intake considerations in plant commodities: *sulfuryl fluoride and fluoride ion*, measured separately.

Table 50. Summary of recommendations - Sulfuryl fluoride:

Commodity		MRL, mg/kg		HR mg/kg	STMR or STMR-P mg/kg
CCN	Name	New	Previous		
GC 0080	Cereal grains	0.05 Po		0.03	0.008
CF 0081	Cereal brans, processed	0.1 Po		0.06	0.008
CF 1255	Maize flour	0.1 Po		0.06	0.008
CF 0645	Maize meal	0.1 Po		0.06	0.008
CF 1250	Rye flour	0.1 Po		0.06	0.008
CF 1251	Rye wholemeal	0.1 Po		0.06	0.008
CF 1210	Wheat germ	0.1 Po		0.06	0.008
CF 1211	Wheat flour	0.1 Po		0.06	0.008
CF 1212	Wheat wholemeal	0.1 Po		0.06	0.008
CM 0081	Bran, unprocessed of cereal grain (except buckwheat, cañihua and quinoa)	0.1 Po		0.06	0.008
CM 0649	Rice, husked	0.1 Po		0.06	0.008
CM 1205	Rice, polished	0.1 Po		0.06	0.008
DF 0167	Dried fruits	0.06 Po		0.04	0.004
TN 0085	Tree nuts	3 Po		2.5	0.28

Table 51. Summary of recommendations - Fluoride:

Commodity		HR, mg/kg	STMR or STMR-P mg/kg
CCN	Name		
GC 0080	Cereal grains	21	3.5
CF 0081	Cereal brans, processed		
CF 1255	Maize flour	70	24
CF 0645	Maize meal	104	37
CF 1250	Rye flour	104	37
CF 1251	Rye wholemeal	104	37
CF 1210	Wheat germ /	104	66
CF 1211	Wheat flour	55	35
CF 1212	Wheat wholemeal	104	37
CM 0081	Bran, unprocessed of cereal grain (except buckwheat, cañihua and quinoa)	104	37
CM 0649	Rice, husked	104	37
CM 1205	Rice, polished	104	37
DF 0167	Dried fruits	2.4	2.4
TN 0085	Tree nuts	9.1	0.28

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of sulfuryl fluoride resulted in recommendations for MRLs and STMR values for raw and processed commodities. Data on consumption were available for 18 food commodities and were used to calculate dietary intake. The results are shown in Annex 3 of the 2005 JMPR Report.

The IEDIs in the five GEMS/Food regional diets, based on estimated STMRs were 1% of the maximum ADI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues of sulfuryl fluoride from uses that have been considered by the JMPR is unlikely to present a public health concern.

The Meeting concluded that the dietary intake of fluoride associated with the use of sulfuryl fluoride as a fumigant (range of 7–15 mg/person/day across the five GEMS/Food regional diets) should be included in an overall assessment of fluoride from all sources. Upper levels for fluoride intakes have been proposed by a number of organizations. The dietary risk assessment for fluoride from fumigant use needs to be considered in light of the overall exposure to fluoride from other sources and FAO and WHO are requested to further investigate how this issue can be addressed at an international level.

Short-term intake

The IESTI of sulfuryl fluoride calculated on the basis of the recommendations made by the JMPR represented 0–3% of the ARfD (0.3 mg/kg bw) for children and 0–5% for the general population. The Meeting concluded that the short-term intake of residues of sulfuryl fluoride on commodities that have been considered by the JMPR is unlikely to present a public health concern. The results are shown in Annex 4 of the 2005 JMPR Report.

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