The ovicidal efficacy of sulfuryl fluoride on insect pests of dried fruit and nuts

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Insects and Mortality. Amyelois transitells (navel orangeworm), plodia interpunctella (Hübner)(indianmeal moth), Tribolium confusum (red flour beetle), and Carpophilus hemipterus (dried fruit beetle) were cultured as described on the CPQ insectary website. Ovipositional periods were respectively 36, 48, 48, and 72 hours for dried fruit beetle, red flour beetle, navel orangeworm, and indianmeal moth. The egg treatment container for red flour beetle, navel orangeworm, and indianmeal moth was a 100 x 15 mm clear plastic Petri dish with a small amount of wheat bran diet on the periphery of a weighing dish (1 5/8" x 1 5/8" x 5/16") that accommodated test eggs. The lid of the dish had four 12 mm diameter holes equidistant around the perimeter. Dried fruit beetle eggs were fumigated in a $\frac{1}{2}$ -pint wide mouth canning jar with six egg-laying slides and no lid. The egg-laying slides consisted of two pieces of waxed paper smeared with small amount of an agar diet sandwiched between two standard microscope slides and held together with a rubber band.

Mortality of non-exposed (i.e., control) and fumigant-exposed eggs was assessed following treatment after incubation at 27 °C and 60% RH. Incubation durations were respectively 3, 4, 7, and 7 d for dried fruit beetle, indianmeal moth, navel orangeworm, and red flour beetle. Using a microscope, exposed-egg mortality was diagnosed by the development of white coloration and survivability by vacated egg cases. Control-egg mortality, diagnosed similarly, was respectively ~ 2, 10, 25, and 35% for dried fruit beetle, indianmeal moth, red flour beetle, and navel orangeworm; it was treated numerically using Abbott's method. Dose-mortality regressions were generated using Probit 2007 software; Probit 9 (P9) doses project 99.9968% mortalities. Number of insects specimen treated (n) and regression heterogeneity (H) are noted.

<u>Fumigations.</u> Sulfuryl fluoride exposures occurred in Labonco® 1-cu. ft. chambers modified for fumigation, as were comparative studies with methyl bromide (see CPQ fumigation website). <u>Dose-mortality data was acquired on insect eggs separately in the absence of commodity</u>. Sorption to the commodity load has not been considered.

<u>Chemical Analysis.</u> Gas chromatography retention times and mass spectra were used for chemical verification during fumigation trials. Doses were quantitatively monitored with a Varian 3800 using a gas sampling port with a 10 μ L sample loop, a packed GSQ analytical column (L = 30 m, ID = 4.5 mm) held at 100°C for 10 min, and a PFPD detector at x°C that received only 10% of the column flow. Qualitative mass

spectrometry was with a HP 5890 using 50uL splitless injections (225°C), a megabore GSQ analytical column (L = 60 m, ID = 0.53mm, df = 0.25 μ m) at 100°C for 10 min, and a GCD detector in EI mode.

Summary. For 24 h NAP fumigations, a marked temperature dependence on sulfuryl fluoride ovicidal efficacy is observed. In California, raisins, figs, and dates arriving from the field are disinfested with fumigation upon arrival; due to the seasonality of harvest, temperatures are typically below 60 °F. At and below 60 °F, marginal egg mortality (i.e. LD 95) of dried fruit beetle, red flour beetle, and indianmeal moth is not achieved with sulfuryl fluoride. Prunes, raisins, figs, dates, and walnuts are also subject to "stored-product" fumigations, to combat reinfestation, that can occur during seasons with temperatures between 80-40 °F. However, LD 95 control of dried fruit beetle eggs will not occur \leq 80 °F and LD 95 control of red flour beetle (and dried fruit beetle) eggs will not occur \leq 70F. To achieve adequate control (i.e. LD 99) of dried fruit beetle, red flour beetle, and indianmeal moth eggs at \leq °70 F, sulfuryl fluoride would need to be reregistered, as requisite concentration-time products exceed the 1500 (mg h L⁻¹) maximum specified on the label. Sulfuryl fluoride can not be used for quarantine control of dried fruit beetle, red flour beetle, and indianmeal moth eggs at temperatures \leq 80 °F.

For high-throughput 4 h vacuum fumigations at 100mmHg, sulfuryl fluoride is the only registered fumigant alternative to methyl bromide. Data indicates that dried fruit beetle, red flour beetle, navel orangeworm, and indianmeal moth eggs can not be controlled at quarantine levels (i.e. P9) using sulfuryl fluoride. No better than 60% egg kill would be expected for dried fruit beetle, red flour beetle, and indianmeal moth using sulfuryl fluoride under vacuum at ≤ 60 °F for 4 hours, the standard industry conditions. Even if chamber temperatures were at 80 °F, which drastically reduces walnut quality, doses of sulfuryl fluoride greater than the 128 mg/L label maximum would be required to achieve adequate (i.e. LD 99) dried fruit beetle, red flour beetle, navel orangeworm, and indianmeal moth egg kill.

			55			,						
60 °F			70 °F			80 °F			_	Test Insect		
	95% L.O.C.		95% L.O.C.			95% L.O.C.				.		
upper	lower	LD	upper	lower	LD	upper	lower	LD		(Spp)*	Schedule	
7.3	5.9	6.7	-	-	-(NA)	-	-	-(NA)	LD50	NOW	24 hr NAP	
13.7	11.2	12.2	-	-	-	-	-	-	LD95			
40.3	23.9	28.5	-	-	-	-	-	-	LDP9			
	0.30	6.40		-	-		-	-	Slope (+/-)			
		6.09			-			-	Heterogeneity			
21.8	15.2	18.9	11.7	10.1	10.9	6.1	4.6	5.4	LD50	IMM		
66.3	33.9	41.9	30.0	22.5	25.5	19.1	12.1	14.6	LD95			
449.7	77.8	131.1	125.3	65.7	86.5	117.7	39.9	60.9	LDP9			
	0.22	4.76		0.19	4.44		0.16	3.80	Slope (+/-)			
		18.29			2.92			7.05	Heterogeneity			
40.9	31.1	36.3	36.7	28.5	33.0	30.0	23.1	27.1	LD50	RFB		
106.6	76.0	87.4	96.7	68.7	78.8	54.0	38.8	43.6	LD95			
529.7	217.4	307.6	486.3	192.4	274.8	155.5	65.4	86.3	LDP9			
	0.18	4.31		0.25	4.34		0.61	7.96	Slope (+/-)			
		6.84			3.94			4.97	Heterogeneity			
246.8 596.4 2773.1	184.9 389.6 861.7 0.23	220.6 453.4 1274.5 5.29 12.35	95.4 793.2 25371.0	54.6 181.3 655.1 0.19	75.5 273.9 1737.2 2.70 4.97	60.4 104.1 254.5	51.8 79.9 132.6 0.40	56.5 88.2 167.2 8.49 8.43	LD50 LD95 LDP9 Slope (+/-) Heterogeneity	DFB		
175.2	141.6	160.8		-	-(NA)			-(NA)	LD50	NOW	CT equiv.	
	268.32	292.8	-	-	-	-	-	-	LD95		(1,500 max	
	573.6	684	-	-	-	-	-	-	LDP9	.)	(1,000 maz	
523.2	364.8	453.6	280.8	242.4	261.6	146.4	110.4	129.6	LD50	IMM		
1591.2	813.6	1005.6	720.0	540.0	612.0	458.4	290.4	350.4	LD95			
10792.8	1867.2	3146.4	3007.2	1576.8	2076.0	2824.8	957.6	1461.6	LDP9			
981.6	746.4	871.2	880.8	684.0	792.0	720.0	554.4	650.4	LD50	RFB		
2558.4	1824.0	2097.6	2320.8	1648.8	1891.2	1296.0	931.2	1046.4	LD95			
	5217.6	7382.4	11671.2	4617.6	6595.2	3732.0	1569.6	2071.2	LDP9			
5923.2	4437.6	5294.4	2289.6	1310.4	1812.0	1449.6	1243.2	1356.0	LD50	DFB		
14313.6	9350.4	10881.6	19036.8	4351.2	6573.6	2498.4	1917.6	2116.8	LD95			
66554.4	20680.8	30588.0	608904.0	15722.4	41692.8	6108.0	3182.4	4012.8	LDP9			
	1824.0 5217.6 4437.6 9350.4	2097.6 7382.4 5294.4 10881.6	2320.8 11671.2 2289.6 19036.8	1648.8 4617.6 1310.4 4351.2	1891.2 6595.2 1812.0 6573.6	1296.0 3732.0 1449.6 2498.4	931.2 1569.6 1243.2 1917.6	1046.4 2071.2 1356.0 2116.8	LD95 LDP9 LD50 LD95			

Lethal Doses (mg/L, oz./1000ft³) of Sulfuryl Fluoride to Insect Eggs: 24 hr, NAP treatment

bold red italics = supersedes maximum allowable dose of 128 (mg L⁻¹) on SF label

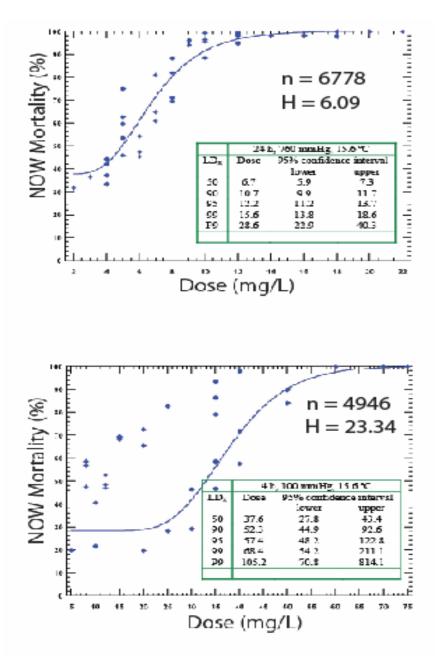
bold italics = supersedes maximum allowable CT product of 1500 (mg h L⁻¹) on SF label NOW = navel orangeworm, Amyelois transitella, IMM = Indian meal moth, Plodia interpunctella RFB = Red flour beetle, Tribolium castaneum; DFB = Dried fruit beetle, Carpophilus hemipterus

	Test Insect		80 °F			70 °F			60 °F		
			95%		L.O.C.		95%	L.O.C.		95% L	.O.C.
Schedule	(Spp)*		LD	lower	upper	LD	lower	upper	LD	lower	upper
24 hr NAP	NOW	LD50	-(NA)	-	-	-(NA)	-	-	6.7	5.9	7.3
		LD95	-	-	-	-	-	-	12.2	11.2	13.7
		LDP9	-	-	-	-	-	-	28.5	23.9	40.3
		Slope (+/-)	-	-		-	-		6.40	0.30	
		Heterogeneity	-			-			6.09		
	IMM	LD50	5.4	4.6	6.1	10.9	10.1	11.7	18.9	15.2	21.8
		LD95	14.6	12.1	19.1	25.5	22.5	30.0	41.9	33.9	66.3
		LDP9	60.9	39.9	117.7	86.5	65.7	125.3	131.1	77.8	449.7
		Slope (+/-)	3.80	0.16		4.44	0.19		4.76	0.22	
		Heterogeneity	7.05			2.92			18.29		
	RFB	LD50	27.1	23.1	30.0	33.0	28.5	36.7	36.3	31.1	40.9
		LD95	43.6	38.8	54.0	78.8	68.7	96.7	87.4	76.0	106.6
		LDP9	86.3	65.4	155.5	274.8	1 92.4	486.3	307.6	217.4	529.7
		Slope (+/-)	7.96	0.61		4.34	0.25		4.31	0.18	
		Heterogeneity	4.97			3.94			6.84		
	DFB	LD50	56.5	51.8	60.4	75.5	54.6	95.4	220.6	184.9	246.8
		LD95	88.2	79.9	104.1	273.9	181.3	793.2	453.4	389.6	596.4
		LDP9	167.2	132.6	254.5	1737.2	655.1	25371.0	1274.5	861.7	2773.1
		Slope (+/-)	8.49	0.40		2.70	0.19		5.29	0.23	
		Heterogeneity	8.43			4.97			12.35		
CT equiv.		LD50	-(NA)	-	-	-(NA)	-	-	160.8	141.6	175.2
(1,500 max	<)	LD95	-	-	-	-	-	-	292.8	268.32	328.8
		LDP9	-	-	-	-	-	-	684	573.6	967.2
	IMM	LD50	129.6	110.4	146.4	261.6	242.4	280.8	453.6	364.8	523.2
		LD95	350.4	290.4	458.4	612.0	540.0	720.0	1005.6	813.6	1591.2
		LDP9	1461.6	957.6	2824.8	2076.0	1576.8	3007.2	3146.4	1867.2	10792.8
	RFB	LD50	650.4	554.4	720.0	792.0	684.0	880.8	871.2	746.4	981.6
		LD95	1046.4	931.2	1296.0	1891.2	1648.8	2320.8	2097.6	1824.0	2558.4
		LDP9	2071.2	1569.6	3732.0	6595.2	4617.6	11671.2	7382.4	5217.6	12712.8
	DFB	LD50	1356.0	1243.2	1449.6	1812.0	1310.4	2289.6	5294.4	4437.6	5923.2
		LD95	2116.8	1917.6	2498.4	6573.6	4351.2	19036.8	10881.6	9350.4	14313.6
		LDP9	4012.8	3182.4	6108.0	41692.8	15722.4	608904.0	30588.0	20680.8	66554.4

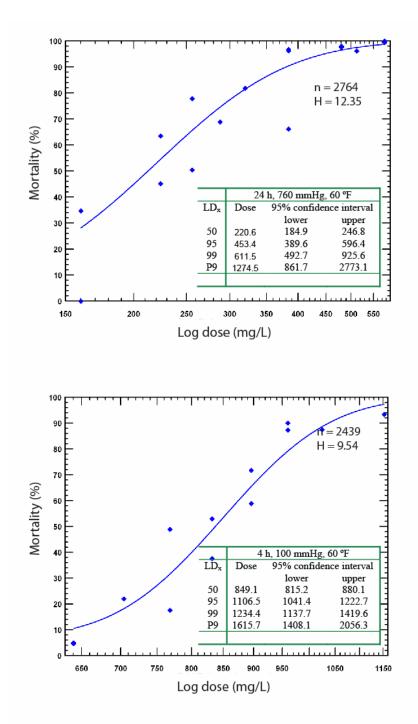
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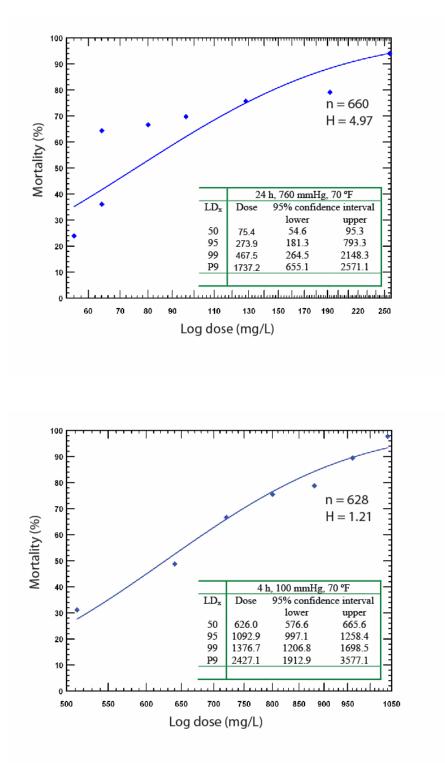
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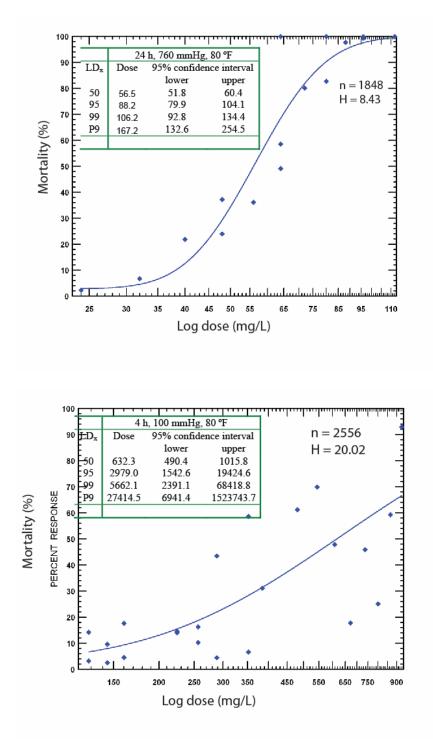
The insecticidal efficacy of sulfuryl fluoride against *Amyelois transitella*, navel orangeworm, in 1 ft³ chambers at 60° F under NAP (top) and *in vacuo* (below).



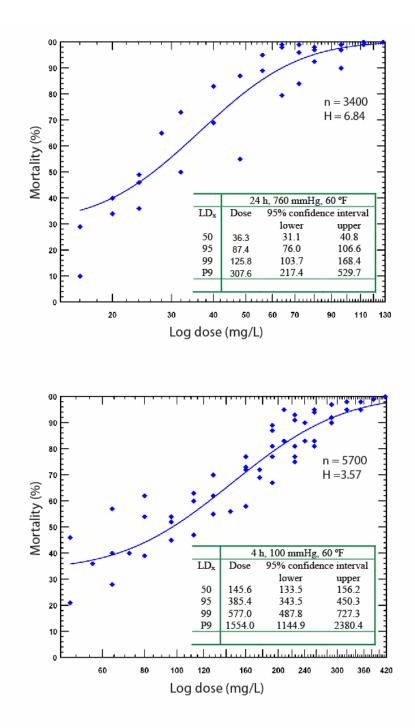
The insecticidal efficacy of sulfuryl fluoride against *Carpophilus hemipterus*, Dried Fruit Beetle, in 1 ft³ chambers at 60°F under NAP (top) and *in vacuo* (below).



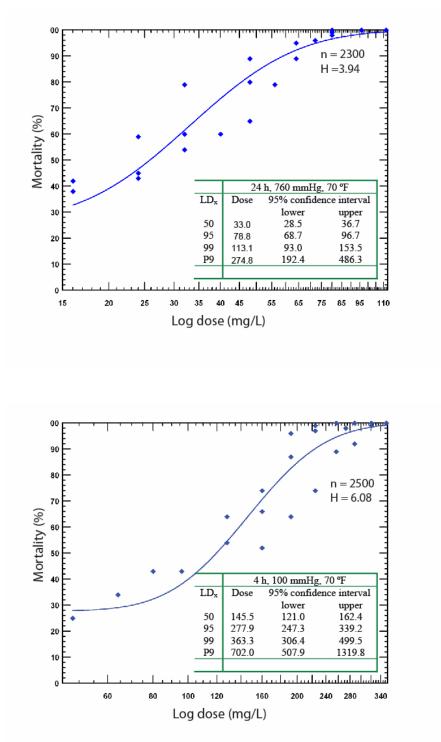
The insecticidal efficacy of sulfuryl fluoride against *Carpophilus hemipterus*, Dried Fruit Beetle, in 1 ft³ chambers at 70°F under NAP (top) and *in vacuo* (below).



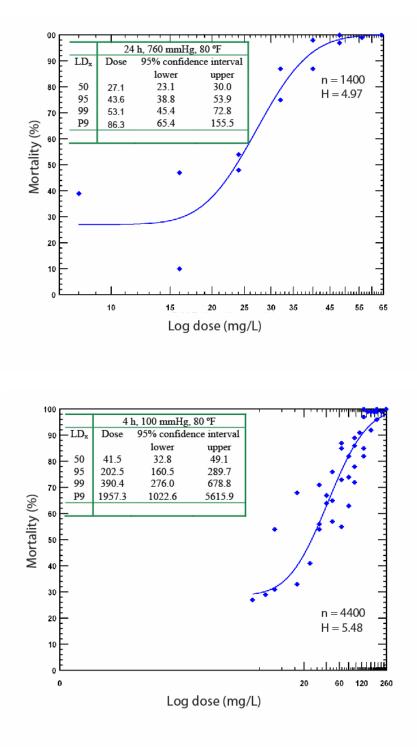
The insecticidal efficacy of sulfuryl fluoride against *Carpophilus hemipterus*, Dried Fruit Beetle, in 1 ft³ chambers at 80°F under NAP (top) and *in vacuo* (below).



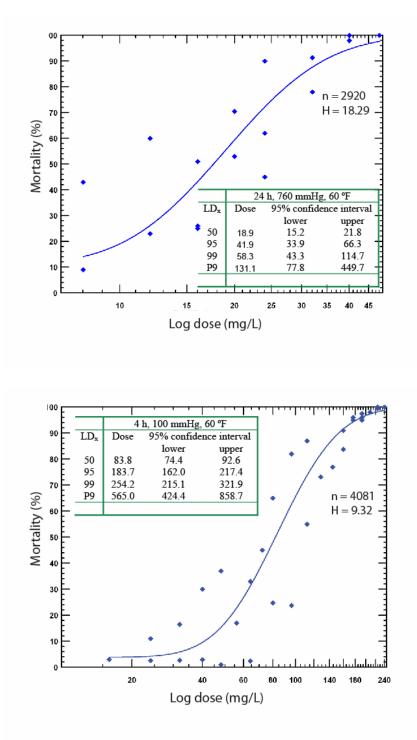
The insecticidal efficacy of sulfuryl fluoride against *Tribolium castaneum*, Red Flour Beetle, in 1 ft^3 chambers at 60°F under NAP (top) and *in vacuo* (below).



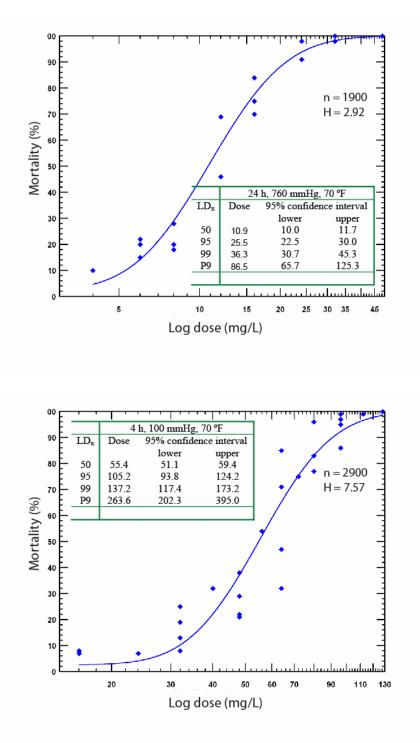
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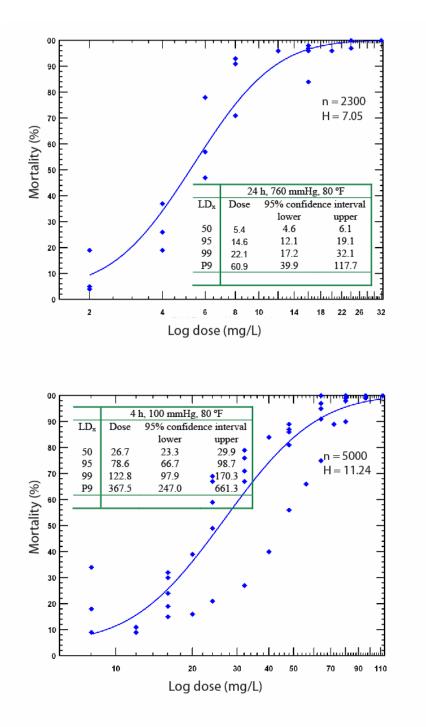
The insecticidal efficacy of sulfuryl fluoride against *Tribolium castaneum*, Red Flour Beetle, in 1 ft^3 chambers at 80°F under NAP (top) and *in vacuo* (below).



The insecticidal efficacy of sulfuryl fluoride against *Plodia interpunctella*, Indianmeal Moth, in 1 ft³ chambers at 60°F under NAP (top) and *in vacuo* (below).



The insecticidal efficacy of sulfuryl fluoride against *Plodia interpunctella*, Indianmeal Moth, in 1 ft³ chambers at 70°F under NAP (top) and *in vacuo* (below).



The insecticidal efficacy of sulfuryl fluoride against *Plodia interpunctella*, Indianmeal Moth, in 1 ft³ chambers at 80°F under NAP (top) and *in vacuo* (below).