

MULTIFACTOR EXPLORATION OF THE INSECTICIDAL EFFICACY AND DEGRADATION OF SULFURYL FLUORIDE IN STORED WALNUTS

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Fumigants are often used to protect specialty crop commodities, such as fruits, tree nuts, and vegetables, from postharvest insect pests. In addition to meeting physicochemical and biocidal requirements, a fumigant must have limited nontarget toxicity, economic cost, and environmental repercussion. Multivariate experimental designs, which facilitate the analyses and interpretation of data, can be used to delineate the contribution of various factors that influence the overall effectiveness of a fumigant. Using this statistics-based approach, existing or novel fumigants can be rapidly and thoroughly screened for optimal dose-duration responses, applicability toward a particular commodity, and physicochemical behavior within a commodity, the target organism(s), and the environment.

Sulfuryl fluoride (SF), a fumigant that been used to target other postharvest insect pests,¹⁻³ has been proposed for treating dried walnut commodity infested with navel orangeworm (*Amyelois transitella*) eggs and diapausing codling moth (*Cydia pomonella*) larvae. Here we detail SF treatment schedules for these species under both atmospheric pressure (NAP) and reduced pressure (100 mmHg) environments. In addition, we report the relative influence of dose, pressure, temperature, and exposure duration on both insect mortality and levels of known residues (e.g., SO₂, FSO₃¹⁻, F¹⁻, SO₄²⁻).^{4,5}

The experimental design was generated, and the conditions for optimal efficacy were elucidated, using Design Expert 7.0 (Stat-Ease, Inc.). A three-factor central composite design was employed,^{6,7} which contained five levels of the three each factors, x_1 – x_3 , and six replicates of the center-point. Center-point conditions of temperature and duration were chosen to accommodate standard industry practice, at least with respect toward analogous methyl bromide protocols. Center-point dose values (1 QT) were those that yielded 99.99% mortality required for quarantine treatment of a particular species at NAP or RP, respectively.

<i>Factor (original units)</i>	<i>Factor levels</i>				
x_1 : dose(mg/L)	1/10 QT	1/2 QT	1 QT	5 QT	10 QT
x_2 : temp (°C)	5	10	15	20	25
x_{3-NAP} : duration (h)	6	12	24	36	48
x_{3-RP} : duration (h)	1	2	4	6	8

QT = quarantine treatment, >99.99% insect mortality

The design involved a total of 34 experiments, which were run in a randomized order in two different time blocks, for each insect species and pressure scenario. The modeled response(s) (y) was insect mortality or residue levels. The full second-order model with all possible two-factor interactions contained 15 parameters:

$$y = \beta_0 + \beta_1x_1 + \beta_{11}x_1^2 + \beta_2x_2 + \beta_{22}x_2^2 + \beta_3x_3 + \beta_{33}x_3^2 + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \beta_{23}x_2x_3 \quad (1)$$

The parameters of this full second-order model include: β_0 , a constant or offset term; β_1 , β_2 , β_3 , estimate the linear effects of the factors; β_{11} , β_{22} , β_{33} estimate the quadratic (curvature) effects of the factors; and β_{12} , β_{13} , β_{23} estimate the interaction effects between every pair of two factors.

The results of this analysis are presented from several perspectives. In light of environmental health concerns surrounding sub-ppm chronic exposures in diet and drinking waters, fluoride residue levels generated from SF hydrolysis are provided. In more practical context, the efficacy of SF relative to methyl bromide for treating stored walnuts infested with these pests is discussed. In conclusion, multivariate experimental techniques have a marked potential for streamlining the development of physicochemical-based approaches that reduce insect damage in perishable and durable commodities.

References.

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