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Fluoride and Sulfate Residues in Foods Fumigated with Sulfuryl Fluoride

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Fluoride (F^-) and sulfate (SO_4^{2-}) residues in eight foodstuffs fumigated with 36 and 360 mg/L of sulfuryl fluoride (SF) for 20 h were quantified by high-performance ion chromatography. Fluoride residues were independently confirmed by F^- electrode analysis. At both SF exposures, levels of F^- and SO_4^{2-} increased concurrently within most commodity types, although in disproportionate ratios between commodities. Aeration period prior to analysis had no effect on residue levels in all commodities tested. Maximum F^- residues were observed in dried beef and maximum SO_4^{2-} residues in dry milk. Vegetable oil was virtually free of anionic residues. Neither F^- nor SO_4^{2-} residues were proportional to fumigant exposure concentrations nor to anion ratios expected from complete hydrolysis of SF.

Sulfuryl fluoride (SF) is a fumigant registered for the control of structural and household pests such as termites, wood-boring beetles, and cockroaches. For safety and convenience, the SF label specifies that food items may remain in structures during fumigation if the food is placed in air-tight containers such as 4-mil-thick (102-μm) polyethylene bags (Dow Chemical, 1982). Negligible SF residues in the ppb range have been reported from foodstuffs that were protected by polyethylene film during fumigation at 10 times the accumulated dosage (720 mg·h/L) of SF normally used for drywood termite control (Osbrink et al., 1988). Transient volatile residues of SF in unprotected foodstuffs were found to be <0.3 ppm (with the exception of vegetable oil) within 8 h after fumigation (Osbrink et al., 1988). Also, a portion of the parent compound (SO_2F_2) may be converted into fixed alteration products in certain food matrices (Meikle and Stewart, 1962). Meikle (1964) showed that graham flour fumigated for 92 h with 32 mg/L of ^{35}S-labeled SF contained nonvolatile radiolabeled alteration products. His qualitative distribution study indicated that the ^{35}S moiety was incorporated into the amino acid and protein of the flour and also resided as free sulfate (SO_4^{2-}). The fluorine constituent of degraded SF was speculated to be free fluoride (F^-), the companion product derived, in part, by phosphate-catalyzed hydrolysis of SF within the matrix. However, Meikle (1964) lacked a means for quantification and selective detection of F^-, the degradation product from SF exposure that poses a potential health risk (Dunning, 1965).

High-performance ion chromatography (HPIC) was used by Bouyoucos et al. (1983) to evaluate time-weighted exposures of humans to SF. SF collected in charcoal traps was hydrolyzed by alkaline solution, and the resultant F^- and SO_4^{2-} anions were quantified by HPIC. Concentrations of F^- in aqueous media have been successfully analyzed with electrode probes specific for detecting this anion (Liu et al., 1987; Ekstrand, 1977). In our present study, both analytical methods were adopted for verifying and quantifying the soluble anionic SF degradation products in aqueous extracts of a broad variety of fumigated food commodities.

MATERIALS AND METHODS

Note: Sulfuryl fluoride is a toxic, colorless, and odorless gas that must be handled with extreme caution by certified personnel. The TLV For SF is 5 ppm, and STEL is 10 ppm.

The following eight food items were fumigated with SF in a 4.2-m^3 chamber following the procedure of Scheffrahn et al. (1987): unbleached enriched wheat flour (Pillsbury), Kibbles 'n Bits dog food (Ken-L Ration), nonfat 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 beef, dog food, and acetaminophen were finely ground in a coffee mill before fumigation to ensure homogeneity of samples.

The subdivided food samples were exposed to SF at 36 and 360 mg/L for 20 h (ca. 10 and 100 times the drywood termite rate) in open disposable cups filled with 5-g portions. The apples and snack cakes were exposed whole. After SF exposure, the samples were stored in the cups at ca. 25 °C in an air-conditioned laboratory. At 1, 8, and 15 days postfumigation, two fumigated samples and two samples of unfumigated commodity (also 5 g) from identical food lots were individually placed for 1 h on a mechanical shaker at room temperature in 50 mL of deionized water (SO_2F_2 solubility in water is 750 ppm). 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. Of the supernatant formed by the centrifugation, 5 mL was passed through a C_{18} Sep-Pak column (Waters Associates), a 0.45-μm pore sized 25-mm disposable syringe filter (Cameo

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II, Fisher Scientific), and stored at −20 °C in polyethylene vials until HPIC analysis. Crude supernatants (5 mL) were frozen and stored as above for F^- electrode analysis. Two samples of each commodity were prepared and analyzed for each SF exposure rate and aeration interval. Fumigations at each exposure rate were replicated twice for a total of 192 treated and 192 control samples.

HPIC was performed on a Dionex 2000i ion chromatograph fitted with a 50-μL sample loop, an AG4A guard column, an HPIC-AS4A Ionpac analytical column, a Dionex anion micromembrane suppressor, and a conductivity detector. The eluent, 2 mM Na_2CO_3 and 4 mM NaOH in deionized water, was pumped through the system at a rate of 2 mL/min. Before analysis, filtered extract supernatants were thawed at room temperature until liquid and then diluted to an extent previously determined with eluent as follows: (1) Oil, acetaminophen, and apple were diluted 5-fold except for apple extracts exposed to 36 mg/L (1- and 8-day samples only), which were not diluted. (2) Flour, cake, and dog food were diluted 10-fold. (3) Dry milk and beef were diluted 20-fold. These dilutions allowed maintenance of a constant detector output range of either 30 or 100 microsiemens during F^- and SO_4^{2-} quantitation and resulted in longer usable life of the guard and analytical columns. Chromatographic traces were recorded on a Spectra-Physics 4290 computing integrator. Standards, KF and Na_2SO_4 (J. T. Baker Chemical Co.), were diluted in eluent and chromatographed before and after every analysis session to monitor changes in retention times of the anionic analytes. Retention times (R_t) were ca. 1 min for F^- and 5 min for SO_4^{2-}. Recovery experiments were carried out as above except that the deionized 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, because the 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 coeluting unknown compounds.

Each foodstuff extract was also analyzed for F^- content by F^--selective electrode determination. Crude extract supernatants (5 mL) were thawed and diluted with 5 mL of water plus 10 mL of FAD (F^- analysis diluent, Corning). 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 taking readings of fumigated and control samples. The probe provided a linear response above 0.4 ppm (w/w, F^- equiv in food). Recovery experiments were carried out in duplicate at 100 ppm (w/w, F^- equiv) with KF fortifications.

RESULTS AND DISCUSSION

Residual amounts of F^- and SO_4^{2-} in foodstuffs resulting from direct exposure to SF are listed in Tables I (36 mg/L) and II (360 mg/L). At both SF exposure rates, increased concentrations of F^- and SO_4^{2-} were detected in fumigated foods compared to unfumigated controls, suggesting that these anions were degradation products of SF within commodities. Residue levels among samples from three aeration intervals showed no trends toward increase or decrease over the 2-week aeration span, indicating that the anionic analytes were permanent and had formed during initial SF fumigation. Oil had no appreciable anionic residues even though Osbrink et al. (1988) found that this commodity contained the highest levels of residual SF.

Table I. Fluoride and Sulfate Residues in Eight Food Commodities from Exposure to Sulfuryl Fluoride at 36 mg/L for 20 h and Postfumigation Aeration of 1, 8, or 15 days [Values (ppm w/w ± SD, n = 4) Corrected for Background and Percent Recovery]

food	days aerated	ion chromatography F^-	ion chromatography SO_4^{2-}	ion electrode: F^-
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
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
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
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 milk	1	97.1 ± 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

Table II. Fluoride and Sulfate Residues in Eight Food Commodities from Exposure to Sulfuryl Fluoride at 360 mg/L for 20 h and Postfumigation Aeration of 1, 8, or 15 days [Values (ppm w/w ± SD, n = 4) Corrected for Background and Percent Recovery]

food	days aerated	ion chromatography F^-	ion chromatography SO_4^{2-}	ion electrode: F^-
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
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	489.6 ± 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
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
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 milk	1	859.1 ± 147.5	2330.1 ± 702.5	870.8 ± 252.5
	8	811.4 ± 77.1	2237.2 ± 429.4	849.1 ± 183.0
	15	819.3 ± 88.2	2379.7 ± 522.1	804.7 ± 152.6

This indicated that the extraction procedure did not hydrolyze any transient SF still residing in the commodities. Meikle (1964) also observed that SF does not hydrolyze in water alone but requires a catalyst.

As predicted by the work of Meikle and Stewart (1962) and Meikle (1964), the commodities incorporating both proteins and fats in this study 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, probe) and 1245 ppm (1551 ppm, probe) for the 36 and 360 mg/L exposures, respectively. At the corresponding low and high exposure, residues were as follows: dry milk, 109 (92) and 819 (805); flour, 62 (70)

Table III. Background Concentrations (ppm w/w) and Percent Recoveries of Fluoride and Sulfate from Samples of Unfumigated Foodstuffs Analyzed by HPIC and F^- Electrode

matrix	HPIC std addn,[b] ppm	HPIC background level[a] F^-	HPIC background level[a] SO_4^{2-}	HPIC mean % rec F^-	HPIC mean % rec SO_4^{2-}	ELEC: mean % rec, F^-
apple	250	47.0	13.3	80.4	107.7	99.6
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
flour	500	49.2	188.8	85.4	90.8	95.9
acetaminophen	250	12.9	137.1	88.6	106.8	107.2
oil	250	11.1	44.8	86.8	95.9	103.2
powdered 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 determinations were below 0.2 ppm. [b] ppm equivalent fortifications of F^- and SO_4^{2-} to 5 g of matrix (i.e. 10-fold the concentration in extraction water) for HPIC analysis only. F^- electrode fortification was 100 ppm for all matrices.

and 315 (344); dog food, 38 (21) and 429 (521). Background levels of F^-, as determined by electrode, were below 0.2 ppm in unfumigated foodstuffs. Low F^- concentrations (ca. 0.1–1 ppm) are normal for a wide variety of common foods as reported by San Filippo and Battistone (1971). High background levels at R_t of F^- observed by HPIC analysis (Table III) were possibly due to organic acids or anions in the extracts coeluting with F^-. Thus, peaks corresponding to F^- in the HPIC traces of unfumigated food extracts were not F^- as verified by electrode analysis and further diagnosed from slight variations in R_t from F^- standards when chromatographed in highly dilute eluting solvent. Several additional unknown anions with R_t values different from those of F^- and SO_4^{2-} were detected by HPIC in some of the fumigated foodstuffs. Our findings suggest that if only F^- concentrations are sought, the electrode technique is simpler, faster, less expensive, and generally as or more accurate than HPIC analysis.

SO_4^{2-} residues (Tables I and II; 36 and 360 mg/L exposures, respectively) were greatest (15-day samples) in dry milk (144 and 2380 ppm), beef (189 and 669), dog food (70 and 197), and flour (59 and 185). Although the vegetable oil contained the greatest SF residues after hours of aeration in a related study (ca. 6 and 71 ppm at 36 and 360 mg/L exposures; Osbrink et al., 1988), little or no F^- or SO_4^{2-} residue was detected for this commodity, further suggesting highly matrix-specific degradation routes for SF in the present study. Mean percent recoveries of anions in spiked samples varied with commodity from 62 to 91% for F^- (59 and 107% by electrode) and 77 to 111% for SO_4^{2-} (Table III). 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 IV) of the anionic residues listed in Tables I and II indicates that their amounts are not proportional to SF exposure rates as was previously observed for the parent compound (Osbrink et al., 1988). The 360 to 36 mg/L residue ratios between commodities ranged from 1.72 and 11.3 for F^- and 2.16 and 16.45 for SO_4^{2-} (15-day samples). Additionally, the ratios of F^-/SO_4^{2-} at both SF exposure rates (Table IV) were highly variable between commodities, suggesting the substrate-specific nature of SF degradation. Assuming that SF 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 IV) are generally

Table IV. Ratios[a] of Fluoride and Sulfate Residue Amounts (w/w) in Foodstuffs Fumigated with Sulfuryl Fluoride at 36 and 360 mg/L and Aerated for 15 Days

food	360/36 ratio F^-	360/36 ratio SO_4^{2-}	F^-/SO_4^{2-} ratio 36 mg/L	F^-/SO_4^{2-} ratio 360 mg/L
apple	1.72	2.16	1.00	0.83
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
flour	5.12	3.12	1.04	1.79
acetaminophen	8.36	2.78	0.83	2.51
oil	–	–	–	–
dry milk	7.53	16.45	0.75	0.34

[a] Calculated from corrected HPIC values (Tables I and II). [b] Negative value, not calculated.

higher than the theoretical ratios, which support the findings of Meikle (1964) that a greater proportion of SO_4^{2-} is bound in nonionic form (i.e. protein adjuncts).

Fluoride residue levels in food resulting from a structural fumigation, practiced in accordance with SF label instructions, can be estimated from the above findings and those of Osbrink et al. (1988). A typical termite fumigation would result in an SF exposure of ca. 72 mg·h/L, while the maximum label rate for control of wood-boring beetle eggs would be ca. 720 mg·h/L. On the basis of a residue reduction ratio of 5.8 for a 10-fold decrease in exposure concentration (Table IV), F^- residues in unprotected dried beef would equal 52 and 300 ppm, respectively. If the beef were protected in two 2-mil polyethylene bags in compliance with the SF label, the resulting protection factor of ≥98% (Osbrink et al., 1988) would, predictably, reduce F^- residues in beef in the above examples to ca. 1 and 6 ppm, respectively. F^- residues in most commodities would be well below 1 ppm. Preliminary findings conducted by us suggest that the above predicted values for F^- residues are valid using conventional "twist tie" sealing methods for double 2-mil bags and that Ziploc closures would reduce F^- levels by up to an additional 95% of the twist tie levels at a 7200 mg·h/L SF exposure. Even at 100 times the drywood termite rate (7200 mg·h/L), we have found that F^- residues in beef are ca. 3 and <1 ppm for the remaining commodities when protected by double 2-mil Ziploc-sealed bags. Normal human dietary intake of water containing up to 8 ppm F^- is considered safe, even at chronic exposures (Dunning, 1965). F^- supplements of 1 ppm in municipal water supplies are recommended and implemented for reduction in dental caries (San Filippo and Battistone, 1971).

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Zingiberene and Resistance to Colorado Potato Beetle in *Lycopersicon hirsutum* f. *hirsutum*

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Zingiberene was detected in foliage extracts of *Lycopersicon hirsutum* f. *hirsutum*. 2-Tridecanone was not present in *L. hirsutum* f. *hirsutum* but did occur in *L. hirsutum* f. *glabratum*. F_2's of *L. hirsutum* f. *glabratum* × *L. hirsutum* f. *hirsutum* segregated for the presence of zingiberene and 2-tridecanone. The occurrence of zingiberene in *L. hirsutum* f. *hirsutum* coincided with the sporadic appearance of resistance to Colorado potato beetle (*Leptinotarsa decemlineata*) in this subspecies. Resistance to Colorado potato beetle in the F_2's was correlated with zingiberene content and with 2-tridecanone content. Extracts of *L. hirsutum* f. *hirsutum* foliage were toxic to Colorado potato beetle larvae at zingiberene contents estimated at 12-25 μg/larva. Zingiberene content of *L. hirsutum* f. *hirsutum* leaflets was estimated to be from 160 to 250 μg/2-cm^2 leaflet.

Resistance to Colorado potato beetle (CPB), *Leptinotarsa decemlineata* Say, has been identified in *Lycopersicon hirsutum* f. *glabratum* C. H. Mull (*gla*) (Schalk and Stoner, 1976) and attributed to the presence in glandular trichomes of certain toxic compounds (Kennedy and Sorenson, 1985), specifically, the methyl ketone 2-tridecanone (Kennedy et al., 1985). 2-Tridecanone also provides resistance in *gla* to the tobacco hornworm (*Manduca sexta* L.) (Fery and Kennedy, 1987; Williams et al., 1980) and contributes to resistance to the tomato fruitworm (*Heliothis zea* Boddie) (Dimock and Kennedy, 1983). *Gla* trichomes also contain 2-undecanone, which is toxic to *Pieris brassicae* L. (Lundgren et al., 1985).

Lycopersicon hirsutum f. *hirsutum* Humb. and Bonpl. (*hir*) is similar to *gla* in that the two subspecies share comparable densities of type IV and type VI trichomes (Snyder and Hyatt, 1984), the predominant types of glandular trichomes in *Lycopersicon* (Luckwill, 1943). The response of trichome density to day length is also similar in *gla* and *hir*. Type IV densities increase and type VI densities decrease under short days in both (Snyder and Hyatt, 1984). However, *hir* differs from *gla* in several respects. *Hir* has very little 2-tridecanone (Fery and Kennedy, 1987; Soost et al., 1968; Lin et al., 1987; Lundgren et al., 1985), has less 2-undecanone (Lin et al., 1987; Lundgren et al., 1985; Soost et al. 1968) and more 2-dodecanone (Soost et al., 1968) than *gla*, and contains predominantly sesquiterpenoids, including two C_{15} compounds that are absent in *gla* (Snyder et al., 1987; Snyder and Hyatt, 1984). Lin et al. (1987) discerned differences between *gla* and *hir* for three types of sesquiterpenes. Sesquiterpene A occurred in *gla* but not in *hir* and sesquiterpenes B and C in *hir* but not *gla*. Trichome extracts containing sesquiterpene B were toxic to larvae of *Keiferia lycopersicella* and *Spodoptera exigua* (Lin et al., 1987). Lundgren et al. (1985) reported that their accession of *gla* contained the sesquiterpene zingiberene, which was not detected in the *hir* accession they examined, but was the predominating component of the volatile fraction of another *hir* accession (Andersson et al., 1980). Sesquiterpenes have been implicated as insect antifeedants (Frazier, 1986; Mabry and Gill, 1979), but also as ovipositional stimulants (Juvik et al., 1988).

Hir, though resistant to a number of arthropod pests (Carter and Snyder, 1985; Juvik et al., 1982), is reportedly susceptible to CPB (Fery and Kennedy, 1987). However, an *hir* accession with which we have worked has exhibited sporadic resistance to CPB (Carter, 1987). Our objectives were to compare CPB resistance of this *hir* accession to that of *gla*, to analyze the inheritance of resistance in their segregating progeny, and to identify the factors conferring resistance to CPB in *hir*.

MATERIALS AND METHODS

Plant Material. Seeds of *L. hirsutum* f. *glabratum* C. H. Mull PI 134417 (*gla*) and *L. hirsutum* f. *hirsutum* Humb. and Bonpl. PI 126445 (*hir*) were obtained from the North Central Regional Plant Introduction Station at Ames, IA. *Gla* and *hir* were grown in the greenhouse at 16-30 °C and 14 h light/10 h dark. Crosses were made of *gla* as female parent and *hir* as the male. The *gla* × *hir* F_2 seeds were germinated in the greenhouse in spring 1986, and *gla* and *hir* were propagated by cuttings. The parent clones and F_2 seedlings were transplanted to the field in a completely randomized design in May 1986. The individual parent and F_2 plants were propagated by cuttings, grown in the greenhouse, and assayed again in May 1987.

Chemical Extraction and Identification. Foliage extracts were obtained in September 1986. Ten leaflets (ca. 2 cm^2 each) from upper leaves of each plant were extracted in 10 mL of distilled hexane for 2 h. Each sample

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