Table I. Stop Mold B Concentration in Wash Water

| Field Test, % | | Titrimetric Method, % |
Packing house estimate	Oregon State Univ. estimate	
...	<0.5	0.49
...	0.7	0.78
...	>0.8	1.04
...	0.8	0.84
...	0.6	0.61
...	>0.8	0.85
...	0.8	0.98
...	0.8	0.78
>0.6	>0.6	0.72
0.5	0.5	0.61
>0.6	0.7	0.74
...	0.8	0.97
0.5	0.5	0.53
0.6	0.6	0.68
0.5	0.5	0.61
0.5	0.5	0.55
0.6	0.6	0.69
>0.6	0.7	0.71
>0.6	0.7	0.77

Concentrations over 0.8% will appear dark blue. If a closer estimate of the high concentrations is desired, these samples can be diluted 1 to 1 with water and then tested.

Results and Discussion

To test the method, samples of wash water were taken from the packing houses and analyzed by the field test and the titrimetric method. In additional tests, the Stop Mold B concentration was estimated by the packing house operators and this laboratory by using the field test and were checked by the titrimetric method in this laboratory. The results listed in Table I show relatively good agreement. The water in washing tanks becomes dirty after some usage and this makes the test more difficult to interpret because of interference of the background color. With some experience, the operator learns to discard this background color and to base his estimate on the color due to Stop Mold B only. The personnel in packing houses were not experienced in chemical testing, but were nevertheless able to learn the test in a short time and to perform it satisfactorily.

A variation greater than ±0.2 ml. in measuring the volume of either Stop Mold B solution or Folin-Ciocalteau reagent will invalidate the results. However, with care, it is possible to achieve satisfactory results with the syringe.

The reaction between sodium o-phenylphenate tetrahydrate and the Folin-Ciocalteau reagent is dependent on pH. The reaction product is yellow at acid pH and blue at alkaline pH. The pH of the final colored solution was measured and the following values were found: 0.4% solution, pH 2.9; 0.6% solution, pH 7.1; and 0.8% solution, pH 9.8. The pH of the final solution is regulated by the amount of acid present in the reagent and the amount of o-phenylphenol sodium salt present in the wash solution. Therefore, it is possible to change the range of this test by changing the amount of acid in the test reagent. Increasing the amount of acid will make it possible to test for higher concentrations of Stop Mold B; lowering the amount of acid will have the opposite effect. The exact amount of acid necessary for any desired concentration range of Stop Mold B has to be determined empirically.

There are several formulations of the fungicidal chemical, sodium o-phenylphenate, on the market and the method described in this paper has been tested only with the formulation Stop Mold B. It is possible that the concentration of sodium o-phenylphenate is not the only factor affecting pH and that therefore this method as described may not work for other formulations. The use of buffers and other additives in the wash tanks may also invalidate the test. Modifications in the concentrations of some of the reagents used may be necessary before this method can be employed under other circumstances.

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STRUCTURAL FUMIGANTS

The Residue Potential of Sulfuryl Fluoride, Methyl Bromide, and Methanesulfonyl Fluoride in Structural Fumigations

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SULFURYL FLUORIDE has several advantages over the widely used methyl bromide as a structural fumigant, one of the most important being the absence of odor problems in household materials (12). The majority of structural fumigations are performed on fully furnished houses which contain numerous and highly diverse items such as foodstuffs, wearing apparel, bed clothes, and other effects. Since residues of a fumigant might constitute a health hazard, the relative sorption and desorption rate of a fumigant is important from the standpoint of safety as well as performance. Various aspects of the residue forming tendency of sulfuryl fluoride are reported here. Range-finding residue determinations for methyl bromide and methanesulfonyl fluoride, an experimental fumigant, are included for comparative purposes.

Materials and Methods

Synthesis of Sulfuryl-S^{35} Chloride. Sulfur-S^{35} dioxide (7.63 mmoles, specific activity 1.31 mc. per mmole) and dry chlorine (7.63 mmoles) were transferred in vacuo to a reaction tube containing a few granules of activated charcoal. Activation of the charcoal was accomplished as follows: The charcoal was heated for 2 hours on a steam bath in 10% nitric acid, thoroughly washed with distilled water, and dried. It was then heated at 400° C. in a slow stream of chlorine and sulfur dioxide diluted with nitrogen for 30 minutes. This catalyst was still active after atmospheric exposure at room temperature for 3 months. The reactants were allowed to stand at −40° C. for 30 minutes after first venting the system with dry air. The fluid contents of the reaction tube were transferred in vacuo to a side arm. Then the temperature of the side arm was adjusted to −60° C.

A range-finding residue study of a new structural fumigant, sulfuryl fluoride, showed that no hazardous residues were found except in the case of foods with a high protein and lipide content. Refrigeration of these foods during fumigation mitigates this residue problem.

and the reaction tube to −195° C.; in this way the unreacted sulfur dioxide and chlorine were slowly distilled back into the reaction tube leaving the sulfuryl-S^{35} chloride in the side arm. This procedure was carried out several times until no sulfur dioxide and chlorine appeared to remain. The product was used immediately. The yield was 824 mg. (80%).

Synthesis of Sulfuryl-S^{35} Fluoride. The reactor tube for the synthesis of sulfuryl-S^{35} fluoride was a 1.5 × 25 cm. borosilicate glass tube mounted vertically and carrying a 28/12 ⚓ outer at the top and an 18/7 ⚓ inner at the bottom. The tube was wound with 3.8 feet of Chromel wire (GS. 30A), 6.66 ohms per foot, giving a spacing of 1 cm. between turns. The winding was wrapped with sheet asbestos, and this in turn was wrapped with aluminum foil. The tube was packed with aluminum oxide in the form of Hengar Granules (Hengar Co., Philadelphia, Pa.) upon which was deposited potassium fluoride (AR grade). This packing had a capacity for the formation of 15 to 20 mmoles of sulfuryl fluoride.

The sample injection tube, a 1.5 × 7 cm. borosilicate glass tube, was capped with a rubber serum-bottle stopper and carried a side arm as a nitrogen inlet; the whole apparatus was mounted on the top of the reactor tube. Nitrogen, the carrier for the reaction products, was dried by passing it through a column of Dehydrite; the flow rate was measured by a flowmeter.

The purification train consisted of a series of traps and drying tubes. The apparatus was assembled in the following manner: A U-tube trap, 7-mm. I.D. borosilicate glass tubing, was connected to the bottom of the reactor tube. The trap was held at −40° C. and served to remove unreacted sulfuryl chloride, sulfuryl chlorofluoride, and some sulfur dioxide. A vapor trap fitted with a gas dispersion inlet tube followed; in operation, the trap was charged with 20 ml. of 0.1M sodium bisulfite solution at room temperature, and this served to trap chlorine. Next was another U-tube identical to the first; this was maintained at −50° C. and served to condense water and sulfur dioxide. A borosilicate glass drying tube, 1.2 × 10 cm., followed this trap. The drying tube, filled with a 6-cm. column of Dehydrite, served to dry the stream of sulfuryl fluoride gas. The last trap was a U-tube, 9-mm. I.D. borosilicate glass tubing, maintained at −195° C., and this served to collect the product.

The reactor tube was preheated to 400° C. with dry nitrogen passing through to dry the potassium fluoride. The nitrogen flow rate was then adjusted to 14 ml. per minute. The sulfuryl-S^{35} chloride was injected dropwise, in 0.05-ml. increments, directly onto the packing in the reactor tube by a 1-ml. syringe having a stainless steel needle 15 cm. long. There was a time lapse of 3 minutes between increments.

After the sulfuryl chloride had been added, the system was flushed with dry nitrogen at 14 ml. per minute for 15 minutes. The sulfuryl-S^{35} fluoride was then transferred to a storage vessel on the vacuum manifold. Quantitized manometrically, the yield was 514 mg., 66% from sulfur-S^{35} dioxide.

When the sulfuryl fluoride was condensed to zero vapor pressure by liquid nitrogen and then warmed rapidly to −78° C., the same vapor pressure was attained promptly as when the compound was cooled rapidly from room temperature to −78° C. This reproducibility of a vapor pressure measurement was indicative of a high degree of purity with respect to volatility.

The product from a preliminary synthesis was converted to sulfamide. m.p. 90° C. [92° C. (14)].

Synthesis of Methane-C^{14}-Sulfonyl Chloride. Methyl-C^{14} iodide (635 mg., 4.47 mmoles, specific activity 0.224 mc. per mmole) was transferred in vacuo to a low pressure, water-jacketed borosilicate bomb tube (11) containing thiourea (340 mg., 4.47 mmoles) dissolved in 4 ml. of water, and the mixture was heated on a steam bath for 1 hour with occasional shaking. The bomb contents, along with water washings, were then poured into a solution of silver nitrate (760 mg., 4.47 mmoles in 2 ml. of water). This mixture was shaken for 20 minutes at room temperature, centrifuged, and the precipitate of silver iodide was thoroughly washed with water.

The water solution of S-methyl-C^{14}-isothiouronium nitrate was filtered through paper and concentrated to 4 ml. under reduced pressure. Chlorine was passed through this solution at a rapid rate for 30 minutes, while the temperature was held at 0° to 6° C. A 5% solution of sodium sulfite was then added, and the aqueous phase extracted with three portions of alcohol-free ether. The ether solution was washed with a small portion of water and dried over anhydrous magnesium sulfate. The ether was removed leaving 333 mg. of lachrymatory prod-

uct, 66% yield. The amide had a m.p. of 88° C. when recrystallized from water [88° C. (2)].

Synthesis of Methane-C^{14}-Sulfonyl Fluoride. To methane-C^{14}-sulfonyl chloride (333 mg., 2.91 mmoles) was added 1 ml. of a potassium fluoride solution (6.8 grams of potassium fluoride in 10 ml. of water), and the mixture was shaken at room temperature for 1 hour. Water was then added to dissolve the potassium chloride formed. The mixture was extracted with two portions of alcohol-free ether, and the combined ether phases were dried over anhydrous magnesium sulfate. The ether was removed leaving a colorless liquid: 214 mg., 75% yield; $n^{19}{}_D$ 1.3608 [$n^{20}{}_D$ 1.3596 (5)]; b.p. 123° C. [124° C. (5)]; amide, m.p. 88° C.

Synthesis of Methyl-C^{14} Bromide. Methyl-C^{14} bromide was prepared in 65% yield (182 mg.) from barium carbonate-C^{14}, specific activity 0.655 mc. per mmole (6, 10).

Preparation of Materials. The following materials were considered representative of those normally associated with a household.

Dried beef	Paper toweling
Corn oil	Polyethylene
Parmesan cheese	Dried peas
Cotton	Viscose rayon
Feathers	Black sponge rubber
White flour	Sandy loam soil
Gluten	Corn starch
Leather	Sucrose
Powdered, nonfat milk	White pine wood
Nylon	Wool

Most of these were processed for fumigation by grinding in a Wiley mill to pass a 60-mesh screen. Such items as white flour, powdered milk, gluten, and corn oil were not processed. Three replicates of each material, equal in weight, were placed in 1-inch steel dishes, and these served as containers during fumigation and counting.

Fumigation. The fumigation chambers were 1100-ml. Erlenmeyer flasks especially adapted to facilitate introduction, purging, and trapping of the radioactive fumigants. Gaseous agitation within the chambers was achieved by a propeller activated by a magnetic stirrer. Methyl bromide and sulfuryl fluoride were transferred to the fumigation chambers directly from a vacuum manifold. Methanesulfonyl fluoride was introduced as a 1% solution in methyl isobutyl ketone. The solution was measured into a capillary tube and dispensed by wicking it onto a piece
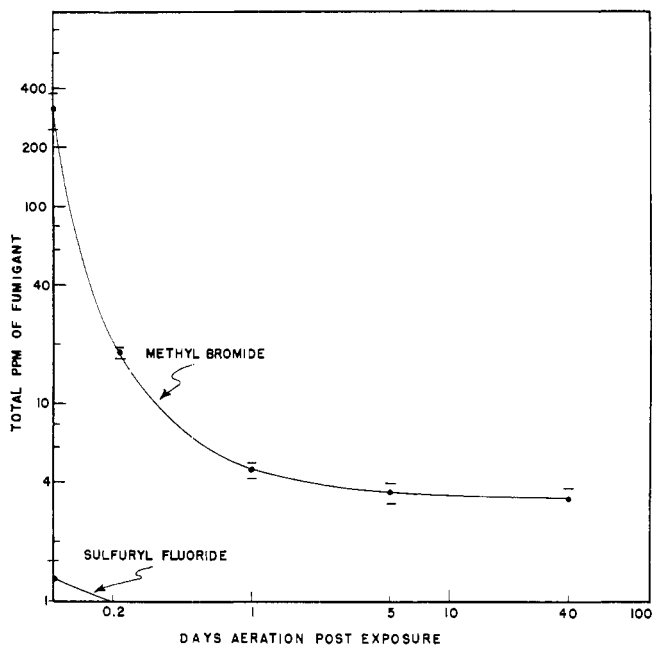
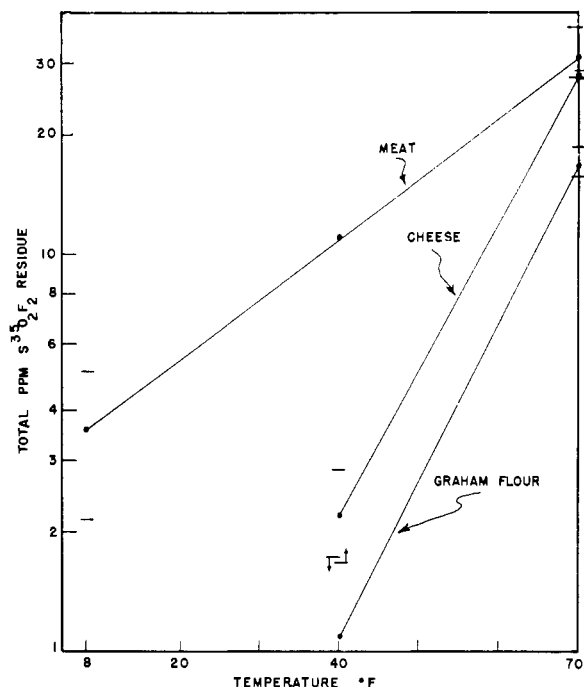Figure 1. Desorption of methyl bromide and sulfuryl fluoride from wood

Figure 2. Effect of temperature on sulfuryl fluoride residues

of cleansing paper in the bottom of the flask at the time of closure.

The fumigations were carried out at 70° to 80° F. for a 16-hour exposure period. The concentration of each fumigant was approximately 10 times that required to give 100% control of the drywood termite, *Kalotermes minor* Hagen (*12*). Methyl bromide at this concentration approximates the dosage

used in commercial structural fumigations to control this pest. The extremely high toxicity of methanesulfonyl fluoride to termites is indicated in Table I. This material is also very toxic to mammals (*11*).

At the end of the exposure period, the fumigants were removed from the flasks by purging with air at the rate of 5 liters per minute for 2 minutes. The exhaust gasses were trapped with liquid nitrogen.

Counting Procedures. All counting was done at infinite thickness with thin, end-window Geiger tubes. To prevent the escape of volatile radioactive materials during counting, the planchets were covered with a single thickness of Saran Wrap secured in place with a rubber band. This cover was removed during subsequent aeration periods.

The initial count was started 2 to 10 minutes after purging. The samples were aerated for 5 hours, 1 day, 5 days, and 20 to 40 days, and were counted at the end of each period.

When the count rate reached a constant value, the sample was combusted, and the barium carbonate-C^{14} and barium sulfate-S^{35} were counted. A comparison of the carbonate and sulfate count rates with calibration curves made it possible to estimate the final parts per million residue and to interpret the previous count rates on the fumigated materials in these terms.

Dry combustion, described by Niederl and Niederl (*9*), was used to convert the material fumigated with methane-C^{14}-sulfonyl fluoride and methyl-C^{14} bromide to barium carbonate-C^{14}.

The digestion procedure used to con

Table I. Concentration of Chemical Used in Fumigation

Compound	Concentration, Mg./L.
Sulfuryl fluoride	28
Methyl bromide	30
Methanesulfonyl fluoride	0.072

Table II. Rate of Desorption of Fumigants by Materials Showing Transient Residues

Material Fumigated	Average Residues Expressed as P.P.M. of Fumigant[a]								
	Methyl Bromide			Sulfuryl Fluoride[b]			Methanesulfonyl Fluoride		
	Initial	5 Hours	24 Hours	Initial	5 Hours	24 Hours	Initial	5 Hours	24 Hours
Corn oil	4584(425)	572(113)	0	24	5.3	0	17(4.1)	0	0
Rayon	1518(114)	12(0.6)	10(1.2)	37(17)	25(0.4)	2.6(0.2)	44(0.2)	12(1.4)	0
Wood	294(62)	18(0.9)	4.7(0.5)				39(2.2)	12(0.6)	1.5(1.2)
Paper toweling	326(38)	30(2.9)	12(1.3)				31(0.2)	0	0
Gluten	521(19)	241(4.3)	236(11)				19(0.2)	3.6(1.9)	3.0(0.9)
Feathers	533(0)	223(16)	248(4.6)				45(3.7)	18(2.3)	14(0.6)
White flour	263(39)	79(10)	66(1.5)				2.3(0.5)	0	0
Nylon	308(6.5)	161(6.9)	175(6.9)				19(3.8)	5.5(0.9)	0
Wool	311(8.8)	221(20)	235(9.4)				19(2.6)	8.3(0.4)	6.1(0.4)
Sponge rubber	193(12)	161(6.5)	178(4.6)				16(1.7)	8.4(3 0)	10(3.1)

[a] The standard deviation is given in parentheses and was calculated from the range (*7*).
[b] The missing values for sulfuryl fluoride are given in Table III as terminal residues. The initial, 5-, and 24-hour values were not significantly different from the terminal, or final residue.

Table III. Average Terminal Residues Expressed as Halide Equivalent 40 Days Post Fumigation[a]

Material Fumigated	CH₃Br	SO₂F₂	CH₃SO₂F
Dried beef	...	55(15)	...
Parmesan cheese	...	12(0.4)	...
Corn oil	<0.8	<0.2	<0.4
Cotton	0.9(0.3)	<0.2	0.5(0.2)
Feathers	176(2.8)	6.6(0.1)	2.8(0.2)
White flour	54(0.5)	2.7(0.1)	0.5(0.04)
Gluten	182(7.1)	2.8(0.1)	0.7(0.2)
Leather	30(1.8)	8.4(0.6)	2.9(0.1)
Dry nonfat milk	...	<0.2	...
Nylon	114(4.8)	0.8(0.1)	0.7(0.2)
Paper toweling	7.4(0.4)	0.4(0.04)	<0.4
Peas	...	2.8(0.3)	...
Polyethylene	<0.8	<0.2	<0.4
Rayon	8.9(0.8)	1.0(0.07)	<0.4
Black sponge rubber	119(6.7)	1.5(0.2)	2.0(0.2)
Soil	...	2.3(0.2)	...
Starch	...	3.8(0.2)	...
Sucrose	<0.8	<0.2	...
Wood	3.0(0.5)	0.2(0.03)	<0.4
Wool	159(15)	2.5(0.2)	1.5(0.2)

[a] The standard deviation is given in parentheses and is calculated from the range (7). The minimum detectable parts per million as halide equivalent, are: 0.8 for CH₂Br, 0.2 for SO₂F₂, and 0.4 for CH₃SO₂F.

Table IV. Residues in White Flour Expressed as Halide Equivalent

Fumigant	Residue P.P.M.	Relative[a] Residue	Specific[b] Residue × 10⁵	Relative[b] Specific Residue
SO₂F₂	2.7	1	9.6	1
CH₃Br	54	20	180	19
CH₃SO₂F	0.5	0.2	695	72

[a] Relative residue was calculated by assigning the value of unity to the residue of SO₂F₂.

[b] Specific residue = $\dfrac{\text{residue, p.p.m.}}{\mu g./\text{liter, fumigant}}$; the relative specific residue was calculated by assigning a value of unity to the specific residue of SO₂F₂.

vert the materials fumigated with sulfuryl-S³⁵ fluoride to barium sulfate-S³⁵ is described by Comber, Watson, and Grucci (4).

Results and Discussion

An understanding of the nature and degree of sorption and desorption of a structural fumigant is important largely from the standpoint of safety to the occupants. In discussing these results, the fumigated materials are divided into two groups: those having transient residues in which the fumigant is partially or totally desorbed, and those having permanent residues.

Transient Residues. In practice, it is generally desirable for a structural fumigant to dissipate from the atmosphere to an acceptable level within a few hours after the aeration period begins. If many household items should be slow to release a fumigant, a potentially dangerous situation could arise in which a persisting hazardous level of fumigant could be present in the atmosphere; the degree of hazard would, of course, be dependent upon both the toxicity and the concentration of released fumigant. Table II lists materials characterized by transient residues. The presence of an item in this table does not imply that there is an absence of ter-

minal residues; it simply indicates that there was a dissipation of the fumigant.

With most materials there was either insignificant sorption of sulfuryl fluoride, or else its desorption occurred so rapidly that the rate could not be measured under the experimental conditions, except for corn oil and rayon. Even here, the rate was sufficiently rapid so as not to present a hazard.

Methyl bromide was generally sorbed extensively. In every case, except that of corn oil, however, its desorption rate was sufficiently rapid so as to avoid any hazard of atmospheric contamination. The contribution of corn oil to this hazard would be insignificant under practical fumigation conditions, since edible oils would ordinarily represent only an infinitesimal portion of the household items exposed. The high affinity of corn oil for methyl bromide is due to its solvent power for the fumigant. Rayon shows a similar tendency, but because of the relatively rapid desorption rate and because of the aeration procedures commonly used in commercial practice, the transient residues constitute little or no problem.

Table II shows that methanesulfonyl fluoride is desorbed more slowly than the other two fumigants. Although the atmospheric concentration would be

small, this compound presents a distinct hazard because of its high toxicity to warm blooded animals (11). Its lethal concentration to rats is 3 mg. per kg. according to Myers and Kemp (8).

Since wood constitutes a large portion of most structures fumigated for termites, it is important to understand the role wood plays in releasing fumigant to the atmosphere after fumigation. Figure 1 shows the sorption and desorption of sulfuryl fluoride and methyl bromide from white pine wood. Sulfuryl fluoride is only sparingly sorbed and rapidly desorbed from this structural material in comparison to methyl bromide. This behavior can be important in commercial fumigation practice for reasons already stated.

Permanent Residues. The permanent residues found, as represented by radioactivity, are almost certainly not the original fumigant. Studies of the metabolic fate of methyl bromide in food products indicated that it is completely converted to inorganic bromide and methylated products (13, 15, 16). Work in progress allows us to conclude that the degradation of sulfuryl fluoride is also complete. To the authors' knowledge, nothing is known about the metabolic fate of methanesulfonyl fluoride, but it seems a reasonable supposition that it too will be destroyed, at least in part. As a result, the data for terminal residues are presented as halide equivalents of the fumigants in Table III.

Table III shows the potential hazard from residues of these compounds. With sulfuryl fluoride, neither odor nor corrosion problems are existent in practical fumigation work, so only permanent residues in food items are important. Not all proteinaceous materials are prone to acquire high residues of this material; this characteristic is indicated by the relatively low level of residue in gluten and nonfat powdered milk. Where high residues of this fumigant have occurred in proteinaceous materials, a solvent system, such as the fat in meat or cheese, is present.

During a practical fumigation it is undesirable to remove food from a structure because of the labor involved and the problems of preservation. An experiment was conducted to determine if a reduction in temperature during the exposure period would reduce the sulfuryl fluoride reaction with meat, graham flour, and Parmesan cheese, and give a consequent reduction of terminal residues. Figure 2 shows that when three replicates of each food were exposed for 24 hours to sulfuryl-S³⁵ fluoride at the concentration shown in Table I and at temperatures of 70°, 40°, and 8° F., the residues were reduced to a very low level. These temperatures are similar to those in a house, a refrigerator, and a deep freeze, respectively.

The residue-forming tendency of the

three fumigants in Table III can be brought out in the relationship of residue to fumigant concentration. Table IV shows the halide equivalent residue of the three compounds in white flour. If equal concentration of fumigants had been used, these residues would have assumed different proportions relative to one another. We define these values as relative specific residues.

Methanesulfonyl fluoride is more reactive than the other two compounds relative to those reactions leading to permanent residues. The relative specific residue, then, would appear to be a good indicator of chemical reactivity in these residue-forming reactions. This relationship appears to be well supported by the data from Kenaga (7) in which confused flour beetles were placed under columns of white flour at varying depths and then fumigated for 16 hours with each of the three com-

pounds at the rate of 0.5 pound per 1000 cubic feet. The penetrating properties were measured; sulfuryl fluoride gave 100% control through 9 inches of flour, methyl bromide through 5 inches, and methanesulfonyl fluoride only in the first inch.

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PESTICIDE ANALYSIS METHODOLOGY

An Automatic Sampling Device for Improved Reproducibility in Testing Granular Materials

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Physical testing of granulated insecticide formulations has been hampered by inadequate reproducibility, particularly in determination of dust content. The variation is due in large measure to the difficulty of maintaining a thorough dispersal of the dust in the stock sample. The turntable sampler described here is a practical means of effecting a pronounced improvement of reproducibility in analytical testing of granular materials for dust content and related qualities. The apparatus is easily constructed.

A DRY-SIEVE TEST of an essentially macroscopic granular material is likely to be regarded as a simple operation. In granulometric analysis, however, even as in chemical analysis, each type of formulation presents its own peculiar problems.

Since the essential advantages of the granulated form for pesticides depend on its free-flowing quality in the dry state without the hazards and inconvenience of dustiness, it follows that to ensure good physical quality one must first be able to specify a reasonable maximum tolerance for dust content, and then be able to test accurately for compliance with the stated requirement. The dust particles, constituting a minor component of the total composition, and being of a lower order of size than the granules, are free to shift around in the intergranular spaces in the most haphazard manner, making it virtually impossible for the analyst to maintain a uniform distribution of the dust while withdrawing a sample for test. For some years, it was

considered sufficient merely to tumble the stock sample thoroughly just before withdrawing the test sample, but this has proved to be not enough.

In a series of collaborative tests sponsored by the Association of Official Agricultural Chemists (2), in which the material passing the 250-micron sieve by dry shaking was considered to be the dust fraction, the interlaboratory mean deviations were 1 to 2% of the sample weight. This amount represents an embarrassing uncertainty of determination in relation to the frequently specified tolerance of 5% for the dust fraction. The mean deviation among replicate runs by a single observer is usually smaller, but still serious. In replicate observations made by the author on each of four samples (at least three and usually four runs per sample) according to the standardized procedure of Interim Federal Specifications for granulated insecticides (3), the average results for the respective samples in terms of per cent dust, or per cent under 250 microns,

were 0.8, 1.2, 2.5, and 4.5. The mean deviations were 0.1, 0.5, 0.6, and 0.7, respectively; that is, from one eighth to nearly half of the dust fraction.

Accumulated experiences such as the foregoing show that one cannot assume the attainment of a representative test sample by taking a few grabs at random, even when the stock sample has been tumbled. For statistical safety, the ideal plan would be to traverse the whole stock sample in a narrow swath, taking little bites of uniform size at frequent regular intervals from beginning to end. In effect, that is just what is accomplished by the new apparatus described here.

The principle is familiar and well established, being used in gross sampling of coal during loading or unloading of freight cars (7), and in sampling of truck loads of bulk-handled peanuts (4). The new device, called a turntable sampler, is a convenient means of applying the same principle to the small-scale operation of drawing 20 grams or so from the few ounces of material usually available