In a low adsorptive soil, with an index tentatively below 30% in this scheme, weed control without crop injury can best be accomplished by adjusting application rates of monuron or diuron downward consistent with the adsorption levels and ease of desorption. In highly adsorptive soils, again tentatively over 70% (K = 9.8 at C = 2 for monuron), these chemicals are not active enough to be used. For these areas, new chemicals are needed which must have one or more of the following properties: greater intrinsic toxicity to be effective at lower concentrations, greater solubility in water but not to the point where the reservoir source life is too limited, and lower soil adsorption. These three factors are considered mutually exclusive and capable of achievement. Other alternatives, such as lowering the adsorptive capacity of some soils by subsoil mixing or other means and increasing the solubility of the herbicides by cosolvents, are being considered.

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FUNGICIDE DETERMINATION

Field Estimation of Stop Mold B Concentrations

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A method for the determination of Stop Mold B concentration in wash tanks depends on its reaction with the Folin-Ciocalteau reagent. Standard and unknown solutions of Stop Mold B are reacted with the reagent and the colors formed compared. This method is simple and rapid and it is most useful in the concentration range of 0.4 to 0.8%.

STOP MOLD B (active ingredient sodium *o*-phenylphenate, 34%) is used for the control of storage rot on apples and pears in the Pacific Northwest (*5, 6*). The fruits are washed in a solution of the fungicide, rinsed with water, packed, and cold-stored. The maintenance of a definite concentration of 0.6% of Stop Mold B in the washing tank during the treatment is important for effective control. Lower concentrations result in poor disease control and higher concentrations are wasteful and could result in excessive residues. A titrimetric method has been used, but is too slow and complicated for packing house use (*7*). A method was desired that would be simple and rapid, but give at least semiquantitative results.

Unsuccessful tests for phenols included the use of Millon's reagent (*3*), ferric chloride (*3*), and 4-aminoantipyrine (*4*). The only successful test for Stop Mold B was with the Folin-Ciocalteau reagent, which is used for phosphatase determination in whole milk (*2*). With this reagent, a simple and reliable test was developed which can be performed by packing house personnel.

Materials and Methods

Preparation of Reagents. The Folin-Ciocalteau reagent is prepared as follows (*1*):

Add 100 grams of sodium tungstate, $Na_2WO_4.2H_2O$, and 25 grams of sodium molybdate, $Na_2MoO_4.2H_2O$, to 700 ml. of water in a 1500-ml. Florence flask. Add 50 ml. of 85% phosphoric acid and 100 ml. of concentrated hydrochloric acid. Connect to a reflux condenser and reflux gently for 8 hours. Then add 150 grams of lithium sulfate, Li_2SO_4, 50 ml. of water, and a few drops of liquid bromine. Boil without a condenser for 15 minutes to remove excess bromine. Cool, dilute to 1000 ml., and filter. The finished reagent should have no greenish tint. The reagent should be well protected against dust.

Prepare the test solution by mixing 900 ml. of methanol, 230 ml. of 0.1N hydrochloric acid, and 18.3 ml. of Folin-Ciocalteau reagent. Test this solution against known amounts of Stop Mold B as described below and if the color range is not satisfactory, adjust the amount of acid. The finished reagent is yellow. A green color indicates the presence of reduced materials, which may mean that the reagent will not act properly. Increasing the acid content will shift the range of the test to higher concentrations of Stop Mold B; lowering the acid content will provide a range for lower concentrations.

Preparation of Standard Solutions. Prepare concentrations of 0.4, 0.6, and 0.8% by diluting a 34% concentrate of the fungicide. Pint bottles with screw-cap lids are convenient for the storage of standards and the volumes can be measured with a syringe or graduated cylinder.

Test Procedure. Using a 5-ml. syringe carefully transfer a 5-ml. portion of each standard, starting with the weakest, to clean test tubes. Rinse the syringe with the unknown wash solution and transfer a 5-ml. portion into the fourth test tube. Then using a second syringe add exactly 3 ml. of the Stop Mold B test solution to each test tube. Shake the tubes and compare the colors. Estimate the concentration of the unknown wash solution by matching its color with one of the standards. Make the comparison against a white background, using the same light source each day.

The colors produced by the standards should be approximately as follows: 0.4% yellow, 0.6% gray, and 0.8% blue.

Table I. Stop Mold B Concentration in Wash Water

Field Test, %		
Packing house estimate	Oregon State Univ. estimate	Titrimetric Method, %
...	<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.