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INSECTICIDE ANALYSIS

Determination of Ronnel, *O,O*-Dimethyl *O*-(2,4,5-Trichlorophenyl) Phosphorothioate, in Sheep and Cattle Dip Solutions

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A method for the determination of ronnel, O,O-dimethyl O-(2,4,5-trichlorophenyl) phosphorothioate, in sheep and cattle dip solution, is described. This method involves a single extraction of ronnel from solution with acetone and hexane. The compound is then hydrolyzed to liberate 2,4,5-trichlorophenol which is determined by a modified 4-aminoantipyrine method. By using a phosphate buffer containing 20% ethyl alcohol a stable dye solution is obtained. The use of alcohol in the buffer increases sensitivity of the method and increases the maximum amount of phenol that can be coupled with the 4-aminoantipyrine without obtaining hazy solutions or the formation of a precipitate.

RONNEL, *O,O*-dimethyl *O*-(2,4,5-trichlorophenyl) phosphorothioate, trademark of The Dow Chemical Co. abroad, has found wide application for the control of parasites of sheep and cattle (*8*, *9*). Recently, ronnel has been used as a sheep and goat dip for the control of external parasites (*7*, *14*, *15*) and is now being tested experimentally as a cattle dip.

The concentration of the insecticide in the dip may be affected by a number of factors, such as selective carry-out, decomposition, settling, or evaporation of water. To maintain a constant level of the compound in the vat, it is necessary to have a method to determine the concentration of insecticide.

Two methods have been suggested for the analysis of ronnel dips. In the first (*5*), ronnel was hydrolyzed to liberate 2,4,5-trichlorophenol which was then isolated by steam distillation. The phenol was then determined by a modification of the antipyrine method (*13*). In the second (*12*), a multiple extraction of the dip with cyclohexane was employed to isolate the ronnel. Cyclohexane was removed by evaporation and the ronnel transferred to acetone and determined by titrating the solution with water until it became turbid. The amount of water employed was inversely proportional to the amount of ronnel present in the acetone solution.

A review of the problems encountered in using these two methods in the field suggested that the most satisfactory approach to the problem would be to use an extraction procedure to isolate ronnel from the dip and a modification of the antipyrine method to determine the 2,4,5-trichlorophenol liberated by the hydrolysis of ronnel.

Materials and Methods

Reagents. 4-Aminoantipyrine, 1%. Dissolve 1.00 gram of 4-aminoantipyrine (Winthrop Laboratories, New York, N. Y.) in 100 ml. of distilled water and store in a brown bottle. Make up fresh weekly.

n-Hexane, technical, 95 mole % (Phillips Chemical Co.).

Ronnel, purified. *O,O*-Dimethyl *O*-(2,4,5-trichlorophenyl) phosphorothioate, 99% or better (The Dow Chemical Co.).

Phosphate buffer. Mix 70 ml. of 0.7*M* dibasic potassium phosphate solution (K_2HPO_4) with 30 ml. of 0.7*M* potassium dihydrogen phosphate (KH_2PO_4) in a 1-liter volumetric flask. Add 500 ml. of water, 200 ml. of absolute ethyl alcohol, and dilute to 1000 ml. with water.

Potassium ferricyanide, 1.4%. Dissolve 1.40 grams of $K_3Fe(CN)_6$ in 100 ml. of distilled water and store in a brown bottle. Make up fresh weekly.

Sodium hydroxide, 4*N*. Dissolve 40 grams of NaOH in about 150 ml. of water, and dilute to 250 ml.

Sodium methylate, 1*N*. Dissolve 54 grams of sodium methylate in methyl alcohol and dilute to 1000 ml. (Olin Mathieson Chemical Corp.).

Stock solution of ronnel. Dissolve 100 mg. of purified ronnel in 100 ml. of acetone.

Procedure. A 10-ml. aliquot of the dip solution is mixed with 10 ml. of acetone and 0.5 ml. of 4*N* sodium hydroxide in a 250-ml., glass-stoppered Erlenmeyer flask. To this solution is added 150 ml. of hexane, and the sample is vigorously shaken for 1 minute. After the two layers are completely separated, a 1-ml. aliquot of the hexane layer is transferred to an 18 × 150 mm. test tube. Two drops of 1*N* sodium methylate solution and a boiling chip are added, and the sample is heated in a boiling water bath until the hexane is distilled off. One milliliter of 50% ethyl alcohol is then added and the sample heated for an additional 5 minutes. After heating, the tube is cooled and 10 ml. of dilute phosphate

Table I. Recovery of Ronnel from Standard Solutions

Ronnel Added, P.P.M.	Absorbance[a] at 500 mμ	Ronnel Recovered, P.P.M.	Recovery, %
2000	0.795	2002	100.1
	0.805	2025	101.2
	0.800	2010	100.5
	0.790	1994	99.7
	0.795	2002	100.1
	0.795	2002	100.1
1000	0.400	1005	100.5
	0.400	1005	100.5
	0.393	989	98.9
	0.385	973	97.3
	0.390	981	98.1
	0.390	981	98.1
500	0.205	510	102.0
	0.202	502	100.4
	0.200	502	100.4
	0.198	495	99.0
	0.195	487	97.4
	0.195	487	97.4
250	0.100	251	100.4
	0.099	251	100.4
	0.100	251	100.4
	0.100	251	100.4
	0.100	251	100.4
	0.102	251	100.4
		Av. recovery	99.8 ± 1.2%

[a] Corrected for a reagent blank of 0.025.

Table II. Recovery of Ronnel from Solutions Prepared with Nankor 44E

Dilution of Nankor 44 E	Absorbance[a] at 500 mμ	Ronnel Calculated p.p.m. added	Ronnel Recovered, p.p.m.	Ronnel Recovered, %
		Water Solutions		
1–200	1.070	2400	2700	112.5
	1.035	2400	2622	109.3
1–300	0.715	1600	1806	112.9
	0.720	1600	1813	113.3
1–400	0.510	1200	1280	106.7
	0.510	1200	1280	106.7
1–600	0.335	800	840	105.0
	0.338	800	848	106.0
1–800	0.255	600	636	106.0
	0.258	600	644	107.3
1–1200	0.164	400	416	104.0
	0.168	400	424	106.0
			Av. recovery	108.0 ± 3.3
		Sheep Dips		
1–200	1.030	2400	2606	108.6
	1.025	2400	2598	108.2
1–400	0.535	1200	1350	112.5
	0.530	1200	1335	111.2
1–800	0.263	600	667	111.2
	0.265	600	675	112.5
1–1600	0.133	300	330	110.0
	0.135	300	338	112.6
				110.8 ± 1.8

[a] Corrected for a reagent blank of 0.025.

buffer added. To develop color, 0.25 ml. of the antipyrine solution is added, followed by 0.25 ml. of the potassium ferricyanide solution. The sample must be mixed thoroughly after the addition of each reagent. The sample is allowed to stand for 20 minutes after the addition of the last reagent and the absorbance then measured at 500 mμ in a 1-cm. cell with water in the reference cell. The dye produced by this reaction is sensitive to ultraviolet light; hence, direct sunlight should be avoided in carrying out this reaction. A reagent blank is run by using 10 ml. of water in place of the 10 ml. of dip solution. Any spectrophotometer or filter photometer with a filter in the range of 500 mμ can be used in this procedure.

The amount of ronnel in 1 ml. of hexane solution is determined from the absorbance reading by reference to a standard curve. The amount of ronnel in 1 ml. of the original dip solution can be calculated from the following equation:

$$\mu g.\text{ of ronnel per ml. of dip soln.} = \frac{\mu g.\text{ of ronnel in 1 ml. of hexane soln.} \times 157}{10}$$

In this procedure, the volume of the organic phase (hexane-acetone) is 157 ml. and the water phase 13 ml.

Standard Curve. Aliquots of the stock solution (1 to 15 ml.) are added to 100-ml. volumetric flasks and made to volume with hexane. A 1-ml. aliquot of each solution is analyzed by the procedure beginning with the addition of sodium methylate. In addition, 1 ml. of hexane is carried through the procedure to obtain the reagent blank. The reagent blank is subtracted from the absorbance readings obtained with the ronnel solutions. The corrected absorbance readings are plotted against micrograms of ronnel to obtain the standard curve. The system adheres to Beer's Law between 5 and 150 μg. of ronnel. The slope of the line determined in a Beckman Model DU spectrophotometer is approximately 0.623 absorbance unit per 100 μg. per 1-cm. light path.

Experimental Results

Recovery of Ronnel from Acetone-Water Solutions. The recovery of ronnel from solutions containing equal parts of acetone and water was first determined. A stock solution containing 2000 p.p.m. of ronnel in acetone was prepared and aliquots diluted with acetone to obtain solutions containing 1000, 500, and 250 p.p.m. Ten milliliters of each solution were mixed with 10 ml. of water and the samples analyzed for ronnel. The results are given in Table I.

The recovery was calculated by determining the amount of ronnel in 1 ml. of hexane solution from the standard curve and then using the following equation:

$$\%\text{ ronnel recovered} = \frac{A \times B(157) \times 100}{C(10) \times D} = \frac{1570A}{D}$$

A = μg. of ronnel per ml. (p.p.m.) of hexane-acetone solution (determined from standard curve)
B = volume of hexane-acetone solution (157 ml.)
C = volume of dip original extracted (10 ml.)
D = μg. of ronnel per ml. (p.p.m.) added

Recovery of Ronnel from Solutions of Nankor 44E. A similar experiment was conducted in which Nankor 44E insecticide, an emulsifiable formulation of ronnel, was employed. The Nankor 44E was diluted with water as indicated and 10 ml. of each diluted solution mixed with 10 ml. of acetone and analyzed for ronnel. The results are given in Table II. A recovery of greater than 100% is to be expected since Nankor is made from a technical grade of ronnel with minimum specifications of at least 85% active ingredient. The active composition of this technical grade of ronnel may vary from 85 to 92% active ingredient. Minimum specifications for Nankor are for at least 44.1% active compound. To ensure that the minimum specifications are met, an excess amount of ronnel is added to this formulation. In addition, some of the impurities present in the technical grade of ronnel will give a positive test with the antipyrine reaction. All of these factors contribute to give a recovery greater than 100% when the results are calculated from a standard curve prepared from an analytical grade of ronnel, and the concentration of ronnel in the Nankor

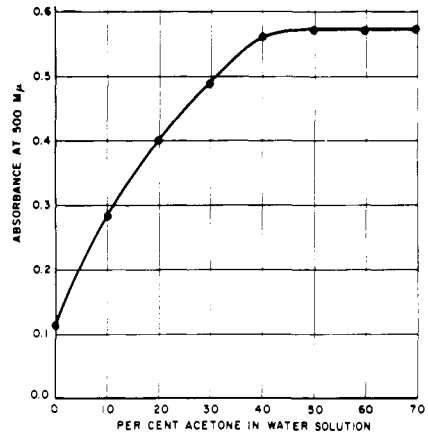

Figure 1. Effect of various concentrations of acetone on extraction of ronnel from water solutions

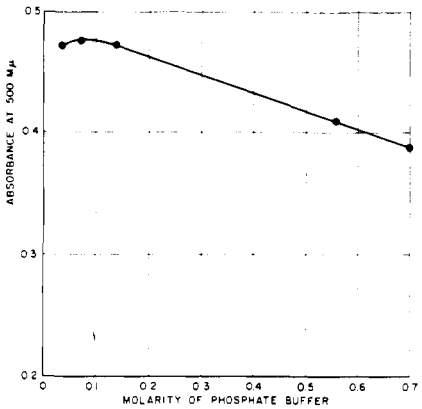

Figure 2. Effect of molarity of phosphate buffer on color development

formulation is calculated on the basis of the minimum specifications for this product.

Recovery of Ronnel from Sheep Dips. Dip samples to be analyzed would normally be taken from tanks which have been in operation for a period of time. These solutions are usually contaminated with dirt, fecal material, feed, etc., carried in by animals. A satisfactory method must be usable on these contaminated samples.

To check the method with contaminated dips, a sample of dip was obtained from a commercial dip tank through which 750 head of sheep had been run. This sample was analyzed to be sure that it did not contain ronnel or any other compounds which would interfere with the test. Aliquots of the dip were then mixed with various amounts of Nankor 44E and the samples analyzed. Results are shown in Table II.

Discussion

In developing the procedure described above for the determination of ronnel in cattle and sheep dips, each step in the procedure was checked in detail to determine the optimum conditions.

Extraction of Ronnel from Dips. When either hexane or cyclohexane is used to extract ronnel from dip solutions, several extractions are necessary to obtain quantitative separation of the compound. However, the addition of acetone increased the efficiency of the hexane extraction and an experiment was designed to determine the amount necessary for optimum extraction. A series of dip solutions were prepared in which the amount of acetone was increased stepwise up to 70%. Twenty-milliliter aliquots of each solution were shaken vigorously with 150 ml. of the solvent, either cyclohexane or hexane. After separation of the layers, 1 ml. of organic solvent layer was analyzed for ronnel. The results obtained with hexane are shown in Figure 1. Hexane gave the best extraction of the ronnel. The absorbance readings show that the amount of ronnel extracted by hexane increased as the per cent of acetone increased up to 50%. Increasing the amount of acetone over 50% did not increase the absorbance readings. Thus, in the procedure, 10 ml. of the dip sample is mixed with 10 ml. of acetone to give a solution containing 50% acetone which is extracted with 150 ml. of hexane.

Table III. Absorbance Readings Obtained by Using 20% of Various Solvents in Dilute Phosphate Buffer

Solvent	Absorbance at 500 mμ after 20 Minutes
Acetone	0.940
Acrylonitrile	1.020
tert-Butyl alcohol	1.113
Dimethylformamide	0.645
Dioxane	0.855
Ethyl alcohol	1.032
Ethylene glycol	0.850
Isopropyl alcohol	1.132
Methyl alcohol	0.655
n-Propyl alcohol	0.968

Hydrolysis of Ronnel. The amount of sodium methylate and the heating time are important for the hydrolysis of the compound. The data obtained from a study in which the normality of the sodium methylate was varied while keeping the volume constant indicated that maximum conversion occurs in the normality range of 1.0 to 3.5*N*. Above 3.5*N* there was a decrease in color production which was associated not with the hydrolysis of the ronnel, but is related to the effect of the sodium methylate on the condensation of the phenol with the 4-aminoantipyrine. Maximum color formation occurs in the pH range 7.4 to 7.8. If too much sodium methylate is added, the pH of the solution will increase beyond the optimum range. In practice, two drops of 1*N* sodium methylate should be used, and the amount used should always be constant.

The length of time the solution is heated after the addition of the 50% ethyl alcohol is not critical. A study of the hydrolysis reaction indicated that the hydrolysis is essentially complete after the hexane had been removed. The additional 5-minute heating period is recommended to ensure complete hydrolysis under all circumstances.

Table IV. Extraction of Ronnel and Related Products from a 50% Acetone Solution

Compound	% Recovery NaOH added	% Recovery HCl added
2,4,5-Trichlorophenyl dihydrogenphosphate hydrate	0	<1
O-Methyl *O*-(2,4,5-trichlorophenyl) hydrogen phosphate	0	18
O-Methyl *O*-(2,4,5-trichlorophenyl) hydrogen phosphorothioate methylamine salt	0	100.0
2,4,5-Trichlorophenol	0	97.8
Ronnel	99.8	99.5

Determination of 2,4,5-Trichlorophenol. The condensation of 2,4,5-trichlorophenol with 4-aminoantipyrine has been employed as an analytical procedure for the determination of 2,4,5-trichlorophenol in biological materials (*4*, *13*). This reaction appears to be influenced by many factors, such as the strength of the phosphate buffer and the ratio of the amount of 4-aminoantipyrine used to the amount of potassium ferricyanide employed.

In an earlier method (*13*) for determination of ronnel, 0.7*M* phosphate buffer pH 8.0 was employed. Concentrations of 0.1 to 4 μg. of phenol per ml. of solution are determined by this method. This corresponds to an upper limit of about 7 μg. of ronnel per ml. Above this concentration, the solution tends to become hazy and precipitation occurs.

The effect of changing the strength of the phosphate buffer was studied, and the results indicated that as the concentration of the buffer was decreased, there was an increase in the absorbance readings (Figure 2). Phosphate buffers of 0.035, 0.07, and 0.14*M* gave about the same response. In general, the 0.07*M* buffer gave slightly higher readings, and this buffer was employed for all subsequent analyses.

These observations agree with those made by Schenk on the determination of Dowicide 2S (2,4,6-trichlorophenol) in hay (*10*, *11*). He found that a dilute phosphate buffer (0.055*M*) gave better results than were obtained using sodium carbonate solution, previously recommended by Gottlieb (*6*).

Difficulties were encountered in main-

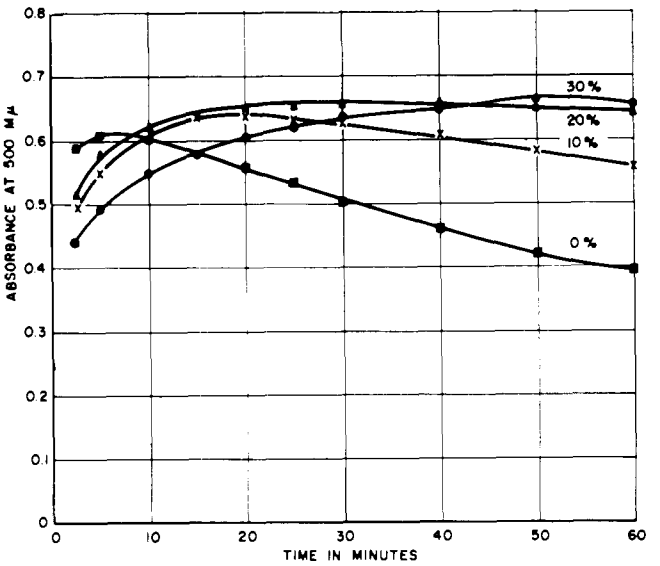

Figure 3. Effects of various concentrations of ethyl alcohol in the buffer on color development

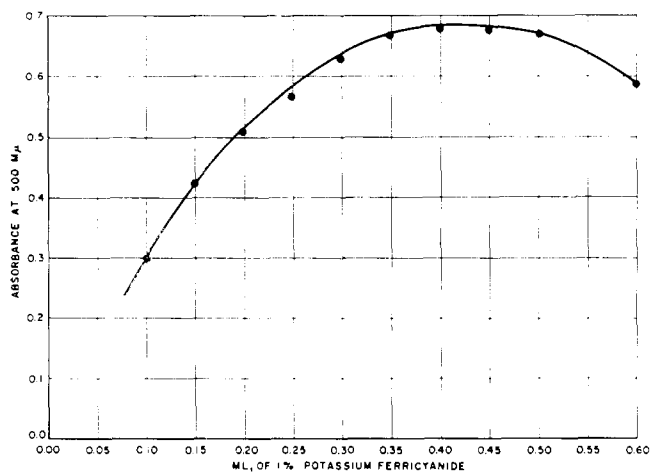

Figure 5. Effect of ratios of 4-aminoantipyrine to potassium ferricyanide on color development

taining stable solutions containing higher concentrations on the phenol when a dilute phosphate buffer was used.

The addition of alcohol to the buffer increased the stability of the color and appeared to increase the limits of the concentration of phenol which could be detected by this method. A series of 0.07*M* phosphate buffers containing various concentrations of ethyl alchol were then compared. Results, shown in Figure 3, indicated that as the concentration of alcohol was increased, the length of time necessary to obtain maximum color was increased and the stability of the solution was increased. With no alcohol, maximum color was obtained in 7 minutes, with the color being stable for only a few minutes. With 20% alcohol, maximum color was obtained in 20 minutes, and the color was relatively stable for an additional 30 minutes. With 30% alcohol, maximum color was obtained in about 40 minutes, and the color was relatively stable.

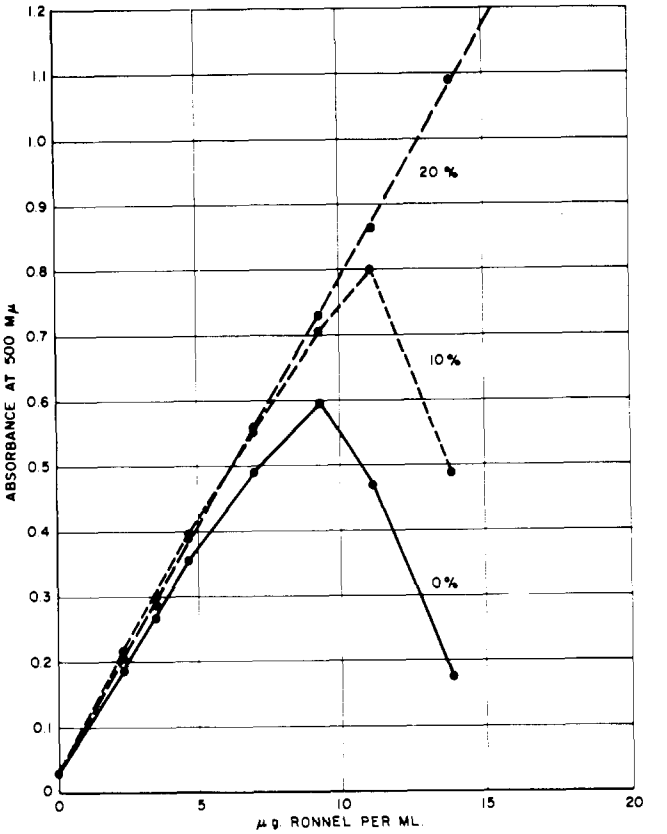

Figure 4. Effect of ethyl alcohol on limits of determination of ronnel

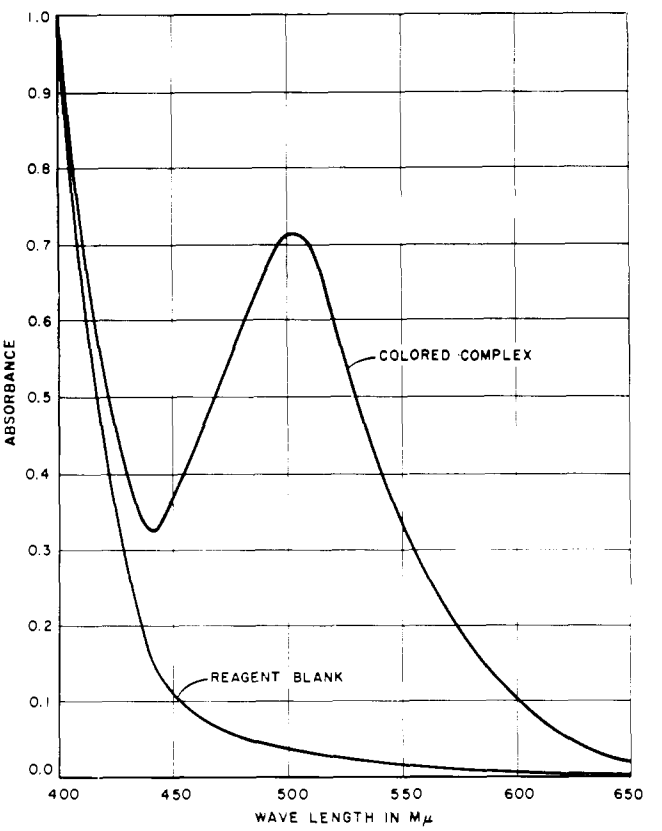

Figure 6. Spectral curve of 2,4,5-trichlorophenol 4-aminoantipyrine complex and reagent blank

The buffer containing 20% alcohol was chosen because of the shorter time period necessary for maximum color development.

When stronger buffers than 0.07*M* were employed, there was a tendency to obtain turbid solutions when 20% alcohol was added to the buffer.

Several other water-miscible solvents were compared with alcohol. The solvents and the results are shown in Table III. Isopropyl and *tert*-butyl alcohol both gave slightly higher absorbance readings than did ethyl alcohol with the same amount of ronnel. Thus, either isopropyl or *tert*-butyl alcohol could be used in the buffer in place of ethyl alcohol.

The effect of alcohol concentration on the maximum amount of ronnel which could be determined with the 0.07*M* phosphate buffer without precipitation was studied. The results are shown in Figure 4. All absorbance readings were made after 20 minutes. With no alcohol in the buffer, the maximum concentration of ronnel which could be determined without precipitation was 7 μg. per ml. With 10% alcohol in the buffer, the limit was increased to about 9.5 μg.; while with the 20% alcohol, the limit was increased to 15 μg. of ronnel per ml. The use of the 0.07*M* phosphate buffer containing 20% alcohol thus doubled the concentration of ronnel which could be measured and also increased the stability of the color complex. As pointed out by Emerson (*1–3*), the ratio of 4-aminoantipyrine to potassium ferricyanide is important in obtaining maximum color. The optimum ratio was determined by selecting a concentration of aminoantipyrine which would be in excess of the maximum amount necessary to couple with the phenol present and then varying the amount of potassium ferricyanide added. This was accomplished by using 0.25 ml. of 1% 4-aminoantipyrine and varying the amount of 1% potassium ferricyanide solution used. The results shown in Figure 5 indicate that 0.35 ml. of the 1% potassium ferricyanide solution gave maximum color formation. In this procedure, the yellow color of the potassium ferricyanide contributes to the final color. Under these conditions, a minimum amount of potassium ferricyanide should be employed to minimize the reagent blank (Figure 6).

In the procedure described above, it is more convenient to use the same volume of both the aminoantipyrine solution and the potassium ferricyanide solution. For this reason, 0.25 ml. of 1.4% potassium ferricyanide solution is employed which will add the same amount of compound as would the addition of 0.35 ml. of a 1% solution.

Specificity. In using an organic phosphate insecticide, there is always the possibility that the compound may be decomposed by chemical or biological reactions with the formation of a series of hydrolyzed products. If an analytical procedure is to be specific for the determination of ronnel, the ronnel must be separated from its possible degradation products, such as the phenol and those phosphates or thiophosphates still containing the phenol radical. All of these compounds will form sodium salts and should distribute in favor of the aqueous phase in the extraction procedure described above. To check this possibility, dilute acetone solutions of 2,4,5-trichlorophenol and various degradation products of ronnel were prepared and extracted by the procedure described above. The results of these studies are shown in Table IV.

Data indicate that all the ronnel can be extracted from either an acid or an alkaline solution while none of the trichlorophenol or other degradation products are extracted from an alkaline solution. From an acid solution, extraction of trichlorophenol was complete while various amounts of the other degradation products were obtained depending on the chemical nature of the compound.

By employing an alkaline extraction procedure, this method is then specific for the ronnel, and free phenols and other degradation products of ronnel do not interfere with the test.

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GRAIN FUMIGANTS

Inhibitors of Carbon Disulfide Decomposition during Gas Chromatography of Fumigant Vapors

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FUMIGATION RESEARCH has been facilitated by recent developments in gas chromatography, a useful method for qualitative and quantitative analyses of volatile chemicals.

In studies of differential sorption and distribution of the vapors of various fumigants when applied to stored grain and other commodities, gaseous samples are withdrawn from the interstices of fumigated material and analyzed (*4, 5, 8*). Compositions of liquid formulations used in such experiments are also verified by gas chromatography.

Carbon disulfide (CS_2) is one of the most common components of liquid fumigant formulations. To reduce fire hazards in commercial formulations, CS_2 is mixed with a large percentage of a nonflammable fumigant such as carbon tetrachloride (*2*) or chloroform (*7*). Fire hazards associated with CS_2 are often further minimized by addition of a small percentage of a fire inhibitor such as *n*-pentane, petroleum ether, or sulfur dioxide (*1, 3*).

Many common fumigants used in liquid formulations have low boiling points and therefore can be analyzed by gas chromatography at moderate or low column temperatures (e.g., 40° C.) and without a heated sample inlet. However, a few, such as ethylene dibro-