Table VIII. Results of Large Scale Aerial Test to Determine the Effectiveness of Two Applications Each of Two Different Formulations of Chlordan against Imported Fire Ants at Lyman, Miss.[a]

Insecticide Formulation, %	Chlordan Application Rate, Lb./Acre	No. of Subplots	Av. Pretreatment Count of Active Mounds per Subplot	Reduction, %, in Active Mounds after Following Months				
				1	3	5	8	12
Chlordan, 2.5 Montmorillonite ARVM, 80.5 Deactivator "H," 7 Heavy aromatic naphtha, 10	0.25	6	32	29	28	81	86	95
Chlordan, 2.5 Attapulgite AARVM, 83.1 Deactivator "H," 4.4 Heavy aromatic naphtha, 10	0.25	16	31	38	44	85	89	87
Check ...	..	7	38	0	14	42	48	

[a] First application made in February 1962, and second in June 1962. Bulk application rate 10 lb./acre.

Table IX. Insecticide Soil Residues Following Granular Application of 1/4 Pound per Acre of Chlordan by Airplane

Type of Granule	Date of First Treatment	Chlordan Found,[a] P.P.M.	Date of Second Treatment	Chlordan Found, P.P.M.
Attapulgite AARVM	2-2-62	0.17 (Sampled 3-19-62)	6-5, 6-62	0.20 (Sampled 10-30-62)
Montmorillonite ARVM	2-12-62	0.08 (Sampled 3-28-62)	6-7, 8-62	0.20[b]

[a] 1/4 pound per acre gives approximately 0.75 p.p.m. in the top inch of soil at time of application.
[b] Also found 0.03 p.p.m. heptachlor epoxide on these plots.

lations of chlordan in test 5 is recorded in Table VIII, and the chemical analyses of insecticide residues from them in Table IX.

The soil from plots treated with chlordan in this and other tests was analyzed, in addition, for heptachlor epoxide resulting from heptachlor impurity in technical chlordan.

Acknowledgment

The authors wish to thank Zonolite Corp., Travelers Rest, S. C., for the vermiculite; American Colloid Corp., Skokie, Ill., for the bentonite; General Reduction Co., Chicago, Ill., for the montmorillonite; and Minerals and Chemical Corp., Menlo Park, N. J., and Floridin Corp., Tallahassee, Fla., for the attapulgite used in this study.

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Received for review November 12, 1963. Accepted January 27, 1964.

BIRD REPELLENCY

A Laboratory Method for Evaluating Chemicals as Bird Repellents

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THE SEARCH for bird repellents to protect seeds and agricultural crops, as reported by Neff and Meanley (*4*), dates back approximately 150 years; these early studies, however, were usually intermittent and directed at solving specific local problems. A review of previous work in this field (*2-7*) shows that within recent years a more concentrated effort has been made to evaluate the effectiveness of various chemicals as bird repellents; but there is clearly indicated a need for a quantitative method for reliably comparing one chemical against another. Such a method would be of special importance in correlating repellent activity and structure of a large volume of chemicals.

A statistical method for evaluating the effectiveness of candidate bird repellents involving concentration-effect measurements was recently adapted from the method of Litchfield and Wilcoxon (*1*). The adapted method involves the calculation of an R_{50} value, analogous to the LD_{50} used in toxicological studies. A similar procedure was reported by Tigner and Besser (*8*) for appraising chemicals as rodent repellents.

This paper describes the methodology involved in the evaluation of chemicals as bird repellents, including reproducibility data under the chosen laboratory conditions.

Procedure

Choice of Bird Species and Test Seed. The red-winged blackbird (*Agelaius phoeniceus*) was chosen as the test species because it is one of the primary birds

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A statistical method involving concentration-effect measurements has recently been adapted for evaluating chemicals as red-winged blackbird repellents. The technique involves the calculation of an R_{50} value (confidence limits at $\alpha = 0.05$) expressed as the concentration of chemical required to repel 50% of the test birds under given test conditions, and yields an estimating equation from which R_1 to R_{99} can be determined. Although the most reliable and reproducible data are obtained using only new blackbirds, for general comparative purposes satisfactory results are obtained using previously tested birds.

involved in depredations to agricultural crops in the United States. Milo maize and hulled rice seeds were utilized as test materials for the repellent evaluations. Both grains were readily accepted by the birds, were uniform, and were of sufficient size so that an adequate coating of chemical could be deposited upon the surface.

Training of Test Birds. The wild birds were held in laboratory holding cages (20 × 15 × 22 inches) in groups of 10, and offered only rice or milo and water for a 2- or 3-day period. Under these conditions, red-winged blackbirds consumed approximately 10 grams (400 to 500 seeds) per bird daily. The birds were then placed in individual cages ($9^3/_4$ × 7 × 8 inches) in a 60-cage bioassay rack, and offered 25 seeds and water for a 16- to 18-hour period. The cages were covered with small ($^3/_8$ inch) mesh bronze wire to avoid loss of any seeds that might be spilled. Those birds that consumed the 25 seeds, usually more than 90% of the candidates, were retained for the repellent tests.

Application of Chemicals to Seeds. The chemicals soluble in acetone were dissolved in the solvent, and 6- to 12-ml. aliquants of the solutions, depending upon the concentrations desired, were added to 100-gram quantities of rice or milo contained in 1-pint glass jars. The seeds were coated with the chemicals by shaking the jars; the acetone was evaporated by periodic venting of the containers. Those chemicals insoluble in acetone were added directly to the rice, and 6- to 12-ml. volumes of acetone were added to form a slurry; the seeds were then coated as above.

Concentration-Effect Measurements. Twenty-five treated seeds were placed in each of 10 aluminum cups ($3^1/_4$-inch diameter × $1^3/_4$ inches high) and offered to 10 individually caged male and female redwings for a 16- to 18-hour (3 P.M. to 8 A.M.) test period; water was available during this time. A bird was considered repelled if 13 or more seeds were uneaten at the end of the test.

Depending upon the initial results, subsequent concentrations were increased or reduced in geometric progressions. The concentration, expressed in per cent of chemical (w./w.) contained on 100 grams of seed, and percentage of birds repelled were plotted on two- or three-cycle logarithm-probability paper. Three to five concentrations were normally sufficient to establish a regression line. The regression line was plotted from which the R_{50} value (that concentration of chemical expected to repel 50% of a given blackbird population) and confidence limits ($\alpha = 0.05$) were calculated. R_1 to R_{99} could be estimated from the line.

Results and Discussion

The reproducibility of the method, as given in Table I, was appraised by replicating each of 10 chemicals two or three times. The redwings used in each test had previously been tested one to

Table I. Reproducibility of Concentration-Repellent Effect Determinations for Chemicals Applied to Milo and Rice Seed and Tested with Male and Female Redwings (Previously Tested One to Five Times)

Code or Chemical Name	Replication	R_{50}, %	Confidence Limits $\alpha = 0.05$, %	Slope Function
DRC-1324	1[a]	0.002	0.001–0.004	3.64
	2[a]	0.005	0.002–0.010	3.78
DRC-1318	1[a]	0.02	0.01–0.06	4.30
	2[a]	0.05	0.01–0.16	12.80
DRC-1330	1[a]	0.12	0.06–0.24	6.84
	2[a]	0.21	0.05–0.99	11.30
1-Hydroxy-2-pyridine thione disulfide	1[b]	0.30	0.17–0.49	2.36
	2[a]	0.18	0.04–0.77	9.57
	3[a]	0.12	0.03–0.54	9.58
1,1'Iminodianthraquinone	1[b]	0.20	0.10–0.30	2.40
	2[a]	0.23	0.11–0.48	3.13
Tetramethylthiuram disulfide	1[b]	0.37	0.20–0.68	1.95
	2[b]	0.32	0.20–0.51	3.74
	3[a]	0.25	0.12–0.52	4.22
9,10-Anthraquinone	1[b]	0.13	0.07–0.23	2.57
	2[a]	0.26	0.12–0.57	4.77
	3[a]	0.49	0.25–0.96	2.92
Benzanthrone	1[b]	0.29	0.08–0.99	7.54
	2[a]	0.56	0.22–1.46	4.39
n-Dodecylguanidine acetate	1[b]	0.40	0.17–0.96	6.97
	2[a]	0.50	0.31–0.80	2.09
	3[a]	0.62	0.39–0.99	2.08
1,3,5-Trinitrobenzene aniline complex	1[b]	0.90	0.71–1.13	1.46
	2[b]	1.07	0.95–1.21	1.21

[a] Rice. [b] Milo.

Table II. Comparison of Concentration-Repellent Effect Determinations Using New Birds and Birds Previously Tested 1 to 5 Times

Chemical Code	Replication	R_{50}, %	Confidence Limits $\alpha = 0.05$, %	Slope Function
DRC-1324	1[a]	0.007	0.003–0.015	3.63
	2[a]	0.007	0.003–0.015	3.84
	3[b]	0.002	0.001–0.004	3.64
	4[b]	0.005	0.002–0.01	3.78
DRC-1325	1[a]	0.02	0.009–0.03	3.44
	2[b]	0.004	0.002–0.01	8.00
DRC-1343	1[a]	0.11	0.05 –0.23	3.26
	2[b]	0.06	0.03 –0.12	2.24
	3[b]	0.02	0.008–0.05	5.37
DRC-958	1[a]	2.3	0.89 –5.98	2.84
	2[b]	0.82	0.65 –1.04	1.30

[a] New birds. [b] Birds previously tested 1 to 5 times.

five times on other chemicals. With the exception of borderline cases (1,3,5-trinitrobenzene aniline complex and DRC-1324), the R_{50} of each replicate falls within the permissible statistical limits of the other. Also, the R_{50} values of those chemicals applied to rice fall within the limits of those applied to milo, with the exception of 1-hydroxy-2-pyridine thione disulfide and 9,10-anthraquinone.

In another series of tests, four chemicals were applied to hulled rice, and each treatment was offered to two groups of redwings: group 1 consisted of birds not previously tested or new birds, and group 2 contained birds previously tested one to five times. Both groups included males and females. The results indicated that previously tested birds were more easily repelled than new birds (Table II).

The results of this study show that the concentration-repellent effect method can be used to evaluate the comparative effectiveness of red-winged blackbird repellents. Although the most reliable and reproducible data are obtained using only new blackbirds, in cases where the supply of test birds is limited, for general comparative purposes satisfactory results are obtained using previously tested birds.

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Received for review October 23, 1963. Accepted January 24, 1964.

PHOSPHATE ROCK CLASSIFICATION

Phosphate Rock Solubilization by Repeated Extractions with Citrate Solutions

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The potential value of phosphate rock as a supplier of phosphorus can be estimated by any one of three solvents: neutral ammonium citrate, 2% citric acid, or alkaline ammonium citrate. Values for phosphorus solubility of 24 calcium phosphate materials were obtained in single and four consecutive extractions by each of the three solvents. In calcium phosphates of the apatite type, the solubility increased as the acidity of the citrate extractant increased, but the reverse occurred in phosphals and pseudowavellites. The ranking of the material was about the same whether based on a single extractant value or on the total value from the repetitive extractions, but the latter procedure grouped the rocks according to their solubility more effectively, especially with the citric acid and neutral citrate. Among the newer rock discoveries, Peruvian, North Carolina, and sea-bottom rocks compared very favorably with highly soluble rocks from Curacao and North Africa.

World consumption of phosphate rock amounted to about 46 million long tons in 1962. During the past few years, the agricultural usage of phosphate rock, either for direct application to the soil or for the manufacture of commercial fertilizer, has been rising about 8% per year. This demand points to the urgent need for the discovery of new phosphate rock fields and the appraisal of deposits heretofore considered uneconomical for mining. Active exploration calls for practical and reliable procedures for determining the suitability of new deposits for fertilizer purposes.

Earlier investigations have been made with the view of establishing reliable indices of reactivity and agronomic merit of phosphate rocks. They have related to surface area (*7*), solubility in fertilizer solvents, readily exchangeable phosphorus, particle weight and density, composition of the apatite component (*4*), and pore structure (*3*). Inasmuch as the citrate solubility (*4*), determined from single extractions with 2% citric acid and neutral ammonium citrate, gave very good correlation with crop yields in greenhouse cultures, further investigation of this chemical property was pursued. Included in the study were solubility measurements based on single extractions with 2% citric acid, neutral and alkaline ammonium citrate, and the cumulative solubility obtained from four consecutive extractions with these citrate solutions.

Test Materials

Fourteen mineral phosphates, representative of well known varieties or sources of phosphate rock, were used in the study (Table I). Ten of the materials had been well characterized in the earlier studies (*3*, *4*, *7*), while the other materials were added to provide fuller