FACTORS AFFECTING THE RELEASE OF DDT FROM GRANULES [1]

I. SOLVENTS, EMULSIFIERS, CARRIERS, PARTICLE SIZE

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INTRODUCTION. Although granular formulations continue to be used successfully in many areas of insect control, little is known of the various factors which govern their efficacy under practical conditions. During application, advantages of less drifting, greater penetration of foliage, or concentration of the insecticide in a confined area of insect activity do not always insure that granulars will attain maximum effectiveness in insect control. To be effective the insecticide must leave the granule and either (1) be available at the surface of the granule to come into contact with the insect, or (2) pass from the granule to the area of insect activity. These processes may occur shortly prior to or coincident with the time of insect activity, but if one or the other does not take place the insecticide is, in effect, partly or completely wasted.

An extensive bibliography concerning granular insecticides (Sferra, 1962) indicates the interest and possible future of such formulations in mosquito control. Factors which govern the release of toxicants from granules for mosquito larval control have been investigated by several workers. Toxicants have included dieldrin (Labrecque *et al.*, 1956); chlorthion, EPN (Weidhaas, 1957); parathion (Weidhaas, 1957; Mulla, 1960a, 1960b; Mulla and Axelrod, 1960a, 1960b); malathion, Bayer 29493 (Mulla and Axelrod, 1960a); and paris green (Rogers and Rathburn, 1960; Sutherland and Bast, 1961).

Although DDT continues to be used on a wide scale for larval control, littl is known of the factors which affect th release of this insecticide from granule into water. In New Jersey, since 194 DDT granules have been used on an ex perimental scale to a varying degree. I initial studies carriers such as sawdust an paper strips were examined, and graduall studies evolved to other available carrier such as tobacco stems and coffee grounds Results were variable, ranging from com plete control (Vannote, 1951) of mosquit larvae to no control. Similar results hav been obtained by mosquito control per sonnel in New Jersey and other parts o the country with commercially prepare DDT granules. In laboratory studies Taf (1952) found DDT to be rapidly release from bentonite and released to the great est concentration from tobacco. Sferr (1957) investigated the release of DDT from granules with agitation under labora tory conditions. Carriers such as tobacco attapulgite, bentonite, and talc were in vestigated.

The objective of current research is tc study the various factors such as carriers solvents, and additives which affect the release of DDT and other insecticides from granules. It is hoped that informa tion gained from these studies will lead to the development of granular formulations for specific situations in mosquito larval control, including the following: (1) a formulation which will release the toxicant as completely as possible within a short interval of time after application, (2) a formulation which can be applied during the winter months and release the toxicant later coincidentally with larval activity, and (3) a formulation which on repeated wetting will release a proportionate amount of the toxicant.

CRITERION OF EVALUATION. Before DDT

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granular formulations could be evaluated, it was necessary to know exactly how such formulations cause toxicity to mosquito larvae. It is generally accepted that granules, regardless of insecticide or carrier, must release the toxicant into the water and not maintain it concealed within the granular particle. Generally, this release is into surrounding water, and it might be assumed that an evaluation of the release could be based on determination of the amount of DDT in the water. When, however, granules are prepared with a relatively non-volatile solvent such as heavy aromatic naphtha, the situation and release evaluation becomes more complex. It is conceivable that the DDT and solvent, being lighter than water, might concentrate at the surface to cause larval mortality, a situation analogous to oil and DDT being sprayed on the surface of the water, whereby the release evaluation would have to include the DDT in the surface oil deposits.

To determine if DDT and solvent at the surface, or DDT and solvent dispersed in the water, is responsible for toxicity of solvent-DDT-impregnated granules, an apparatus (Fig. 1) was constructed and

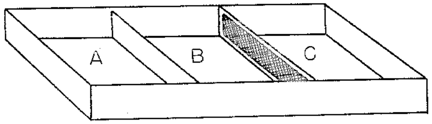

FIG. 1.—Apparatus for determining criterion of evaluation of granular insecticides for mosquito larval control.

used. A large stainless steel pan, measuring 5 feet by 2 feet, was divided into three sections with two stainless steel partitions, a solid partition and a screen partition. The height of these partitions was such that when the entire pan was filled with water to a depth of 4 inches, the water level was approximately 1/16 inch above the top edge of the solid partition and in the middle of the top of the frame of the screen partition. The entire system of three compartments consisted of the following: two compartments (A and B) which had the same surface of water, and two compartments (B and C) whose subsurface portions of water were in continuity through the screen but whose water surfaces were separated. With such an arrangement, 500 or more mosquito larvae could be placed in each compartment with no exchange of larvae between adjoining compartments.

When DDT dissolved in heavy aromatic naphtha was applied to the center portion (B) at 1 lb. actual DDT per acre, the following results were obtained. In the center section 91 percent mortality occurred. In compartment A, which shared a common surface with B, 52 percent mortality occurred, indicating some but uneven spreading of DDT and solvent to this compartment. In compartment C, however, only 5 percent mortality occurred, indicating that only very slight dispersal of DDT and solvent (possibly due to larval movement) occurred through the water from the surface of the center compartment. In later tests, when the same amount of solvent and DDT formulated on attapulgite granules was applied to the center compartment, the following average mortalities were recorded: A, 74 percent; B, 100 percent; and C, 90 percent.

Such results indicate that mortality from relatively non-volatile solvent-DDT-impregnated granules is due not only to the occurrence of solvent and DDT at the surface of the water but in addition, and to even a greater extent, to a dispersion of DDT throughout the water. Therefore, in current studies on DDT granules "the ability to release and disperse DDT in water" has been adopted as a major criterion in the evaluation of granular formulations for larval control.

MATERIALS AND METHODS. PREPARATION OF GRANULES. Solvent-impregnated granules were prepared with apparatus and methods already described (Mulla and Axelrod, 1960b). Modifications included the following. A one-quart plastic refrigerator cup with an aluminum disc was coupled to the drive shaft of the motor. Beakers (600 ml.) containing the granules were placed firmly into the cup. The mouths of beakers were covered with cellophane, in the center of which was cut a hole (1-inch diameter) to allow entrance for the atomizer and a glass rod for mixing.

To standardize the impregnation of granules of different bulk densities, approximately 160 cubic centimeters of granules were prepared at a time. Solutions of either 33 percent technical DDT in heavy aromatic naphtha or in acetone were used for impregnation. Heavy aromatic naphtha (HAN, Esso) contained 87 percent aromatics by volume and had a flash point of 155° F. closed cup. Depending on the solvent used, granules were prepared according to the following ratio of ingredients (X=weight in grams of 160 cubic centimeters of granules): HAN-impregnated granules, (X) grams granules+0.25 (X) ml. DDT solution+0.05 (X) ml. rinse; acetone impregnated granules, (X) grams granules+0.20 (X) ml. DDT solution+0.05 (X) ml. rinse. Granules prepared with HAN were sealed in their containers; those prepared with acetone were spread thinly on a flat surface and the acetone allowed to evaporate. Fused DDT granular formulations were made by mixing technical DDT (50+ mesh) with the carrier and heating for a short time at 100° F. All formulations were allowed to cure for 2 to 3 days prior to testing. Percent DDT in the formulations, which was determined by the organic chlorine method, generally ranged between 6 and 6.7.

RELEASE AND DISPERSAL TESTS. To evaluate the release and dispersal of DDT from granules the following method was adopted. To 2-quart mason jars containing 1750 ml. distilled water, 2 grams of the granular formulation were added with no agitation. Each jar was capped and constituted a system. At a specific time thereafter, a system was opened and an aliquot removed by means of an aliquot tube (Fig. 2). Tube end A was dipped under the water surface as a small amount of air was passed through to prevent oil or any surface-floating granules from entering the tube. The aliquot tube was then lowered and allowed to rest on the lip of the mason jar, tube end A being at a depth halfway in the column of water. Fifteen minutes later, a 225 ml. aliquot was siphoned off by applying air pressure to the mouth of the jar; the aliquot was subsequently diluted for bioassay with 3-day-old *Aedes aegypti* larvae. Mortalities were compared to standard dosage mortality curves of DDT with the appropriate solvents. All values in subsequent graphs and tables for the percent formulated DDT in water are the average of bioassays performed on aliquots from duplicate systems.

TOXICITY OF DDT FORMULATIONS TO MOSQUITO LARVAE. Laboratory studies to investigate the release and dispersal of DDT from granules provide information from which only the efficacy of various granular DDT formulations in killing mosquito larvae under field conditions can be predicted. Under field conditions, however, it is often difficult to evaluate such formulations, especially when differences are only slight. In addition, it is difficult to find suitable areas for replication and to apply exact dosages uniformly. Because of such factors that were limiting in field evaluation, granular formulations were tested in the laboratory as follows. Formulations were applied to white enameled pans (16 inches x 10 inches) containing 6 liters of water and over 100 3-day-old *Aedes aegypti* larvae. Rates of application were 0.5 and 0.25 lb. actual DDT/acre. Mortalities were determined at 5, 16, and 24 hours after application.

RESULTS AND DISCUSSION. RELEASE AND DISPERSAL TESTS. One important factor affecting the release and dispersal of DDT

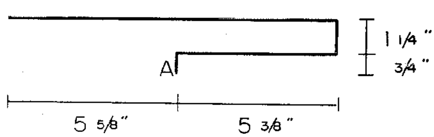

FIG. 2.—Aliquot tube and its dimensions.

is the granular carrier itself. Shown in Figure 3 are the DDT dispersal curves

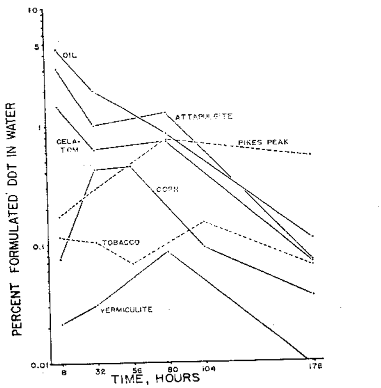

FIG. 3.—Dispersal of DDT from various granuar carriers impregnated with HAN.

of various carriers including attapulgite 24–48 AA RVM (Mineral and Chemicals Corporation of America), Celatom MP-78 (Eagle-Picher Company), Pikes Peak 20–60 (9H66) calcined (General Reduction), corncob 14–20 (Anderson Cob Mills), tobacco stems, and vermiculite No. 4 24–48 (Zonolite). The tobacco stems had previously been subjected to unknown extraction processes. All formulations contained approximately 20 percent HAN and between 6.0 and 6.7 percent technical DDT. Percentages of formulated DDT occurring in the water at specific times after application are graphed on a logarithmic scale and denote that portion of DDT, originally formulated on the granules, which has been released from the main portion of the granules and is dispersed at the time of testing in the water. This has not been expressed as "percent release of toxicant" from granules, since the amounts of DDT adsorbed on glass surfaces and occurring in solvents films at the surface of the water have not been determined.

Included in Figure 3 is a dispersal curve labeled "oil" which represents the dispersal of DDT and HAN when applied to the surface of water and immediately shaken for 15 seconds; the amounts of DDT and HAN used were comparable to those in the granular formulations. As is shown, over a period of a week, decreasing amounts of DDT and HAN remain in the water as the DDT and HAN rise to the surface of the water. This dispersal curve for DDT and HAN not formulated on granules provides a reference for comparison of granular formulations in Figure 3 and subsequent figures. Generally, those granular formulations which have a dispersal curve similar to that for DDT and HAN alone have released and dispersed more DDT than others.

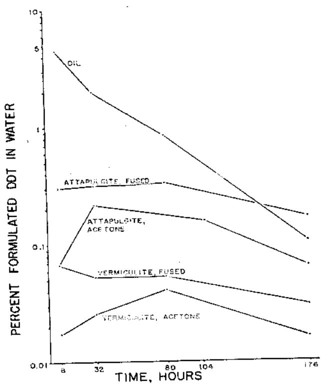

FIG. 4.—Dispersal of DDT from granules prepared by volatile solvent impregnation and melting of toxicant.

At the end of 8 hours, the various types of granules dispersed DDT to varying degrees. Attapulgite dispersed DDT to the greatest degree, almost 100 times the amount dispersed by vermiculite. Both attapulgite and Celatom released and dispersed DDT in a similar manner, the slope of their dispersal curves between 8 and 32 hours indicating that higher amounts of DDT probably were dispersed in the water prior to 8 hours. Pikes Peak

Clay dispersed DDT in increasing amounts up to the third day, after which dispersal declined. Plant materials, such as corncob and tobacco, did not disperse DDT as well as most mineral carriers.

In addition to employing a relatively non-volatile solvent for impregnation, it is also possible to prepare DDT granules by (1) using a volatile solvent such as acetone for impregnation, the solvent being allowed to evaporate, or (2) melting the DDT onto the granules. The DDT dispersal curves for four such formulations on vermiculite No. 4 and attapulgite AA RVM 24–48 are given in Figure 4. Both melted formulations respectively dispersed more DDT than those prepared with acetone. Probably the acetone carried the DDT so far into the granule particles, where DDT solidified when the acetone evaporated, that the insecticide was concealed and prevented from being dispersed. In the melted preparations, DDT penetration into the granule probably occurred to a lesser extent, thereby allowing for more DDT to be dispersed from the surface of the granules. In no case, however, did the amount of dispersed DDT between 8 and 80 hours exceed 32 percent of that dispersed by the HAN-impregnated attapulgite in Figure 3.

Granular formulations may also contain emulsifiers and other additives which may affect the dispersal of DDT. It is of particular interest in the investigations reported herein as to what materials might be added to such formulations to improve or delay release and dispersion of DDT. To determine the effect of emulsifiers on granules, HAN-impregnated attapulgite AA RVM 24-48 and vermiculite No. 4 were prepared containing the following emulsifiers at approximately 2 percent final concentration: Emcol H 500X, blend of non-ionic and anionic (Witco): Triton X-100, non-ionic (Rohm and Haas); and Triton X-151, blend of non-ionic and anionic. Dispersal curves for these formulations, as well as those containing no emulsifier, are shown in Figures 5 and 6. On attapulgite Emcol H 500 X and Triton X-151 increased the amount of dispersed DDT in the water over the entire week. Triton X-100 accomplished the same thing but to a lesser extent.

Results of apparent higher dispersal with emulsifiers on attapulgite do not necessarily mean that emulsifiers were responsible for releasing more DDT from the granules, but more likely that the emulsi-

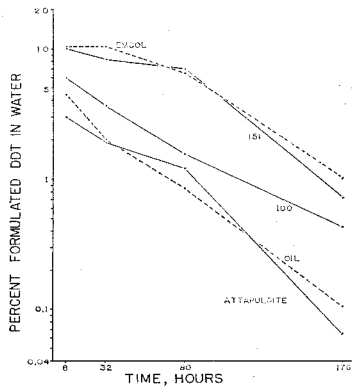

Fig. 5.—Dispersal of DDT from attapulgite granules impregnated with HAN and emulsifiers (2%).

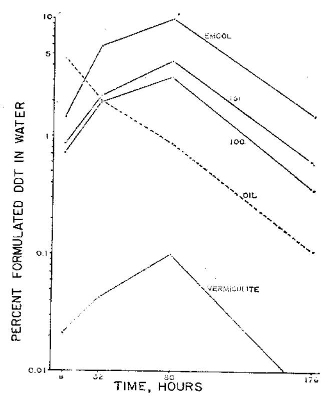

Fig. 6.—Dispersal of DDT from vermiculite impregnated with HAN and emulsifiers (2%).

fiers maintained the DDT and HAN in suspension for a longer time and retarded their rising to the surface of the water. Inclusion of an emulsifier caused vermiculite granules to wet more readily and sink sooner. Emulsifiers were responsible for releasing and dispersing greater amounts of DDT from vermiculite into water, as is evidenced by (1) similarity between the shape of dispersal curves of vermiculite with and without emulsifier, and (2) the increase of dispersed DDT up to the third day in all vermiculite formulations.

As has been discussed previously, when attapulgite AA RVM 24–48 granules were impregnated with DDT and HAN, during the first 4 days they released and dispersed greater amounts of DDT than other carriers. Attapulgite is available in several grades, including A LVM and AA RVM, A signifying non-extruded granules and AA extruded granules. Extrusion is reported to increase sorptive capacities of attapulgite. LVM granules are processed as super-hardened granules which resist disintegration in water. RVM granules, on the other hand, tend to disintegrate in water. Most grades are available in various mesh sizes. To determine if grade or particle size affects the dispersal of DDT, formulations of DDT and HAN on attapulgite LVM and RVM mesh sizes 30–60, 20–35, and 8–15 (Attaclay) were prepared and tested (Table 1).

At 8 and 32 hours, LVM granules generally dispersed slightly greater amounts of DDT than did RVM granules of equal mesh size. As a result of disintegration in water, however, RVM granules may have released and dispersed greater amounts prior to 8 hours. Particle size of attapulgite apparently affects the release and dispersal of DDT, the percent dispersal being inversely proportional to particle size.

The attapulgite samples used in this portion of the investigations were fresh samples of Attaclay (Minerals and Chemicals Corp.) and at 8 hours dispersed more DDT than did an earlier attapulgite sample (Fig. 3). Possibly the fresh samples of attapulgite contained less free and bonded moisture and, therefore, possibly released and dispersed DDT at a slower rate in the time interval prior to 8 hours.

Similar results were obtained with attapulgite granules obtained from another source, Floridin Company. The dispersal curves for Florex AA LVM and Florex AA RVM 30–60 are shown in Figure 7.

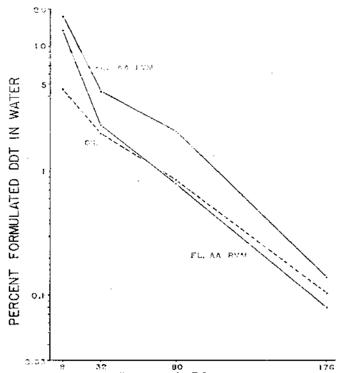

FIG. 7.—Dispersal of DDT from HAN-impregnated attapulgite (Florex) granules.

TABLE 1.—Dispersal of DDT from various HAN-impregnated grades and sizes of attapulgite granules (Attaclay).

	Percent formulated DDT in water					
Time, hours	30–60 LVM	30–60 RVM	20–35 LVM	20–35 RVM	8–15 LVM	8–15 RVM
8	13.6	12.1	12.9	11.4	5.6	4.1
32	2.8	3.0	2.9	2.5	1.5	1.1
80	4.6	4.1	4.3	3.8	2.3	2.8
176	0.2	0.1	0.3	0.2	0.2	0.2

Both were extruded granules obtained from freshly opened containers. As with Attaclay granules, the amount of dispersed DDT from LVM granules at 8 hours is greater than that from RVM, although the RVM granules may have dispersed DDT to a greater extent prior to the eighth hour.

Various grades and sizes of Pikes Peak clay were evaluated for release and dispersion of DDT (Table 2). Relatively small percentages of DDT were dispersed from HAN-impregnated Pikes Peak granules, the amounts of DDT dispersed never exceeding 0.82 percent during the periods tested. On the basis of total amounts of DDT in the formulation there were only slight differences between the amounts of DDT dispersed by various grades and sizes.

TOXICITY OF DDT FORMULATIONS TO MOSQUITO LARVAE. The effects of various freshly prepared DDT formulations on mosquito larvae under laboratory conditions are shown in Tables 3, 4, and 5. Initially, formulations were tested at 0.5 lb. DDT actual per acre. Since no differences were observed between attapulgite formulations impregnated by HAN, e.g. Attaclay 30–60 AA RVM, 30–60 A LVM, 20–35 AA RVM, Florex AA 30–60 RVM, AA 30–60 LVM, the formulations were subsequently tested at 0.25 lb. DDT actual per acre. Generally, the order of relative toxicity of various formulations followed the order of their ability to release and disperse DDT. The various formulations can be grouped in decreasing order of their toxicity to mosquito larvae as follows:

Group 1. HAN-impregnated formulations on attapulgite including Attaclay 30–60 AA RVM and A LVM, Attaclay 20–35 AA RVM, Florex 30–60 AA LVM, 30–60 AA RVM were the most toxic to mosquito

TABLE 2.—Dispersal of DDT from various HAN-impregnated grades and sizes of Pikes Peak Clay.

	Percent formulated DDT in water					
Time, hours	20–60 Dried 9G66	20–60 Calcined 9H66	18–40 Dried 9J66	18–40 Calcined 9K66	15–30 Dried 9L66	15–30 Calcined 9M66
8	0.07	0.17	0.11	0.14	0.17	0.09
32	0.27	0.29	0.30	0.42	0.36	0.55
80	0.26	0.76	0.28	0.82	0.41	0.23
176	0.12	0.53	0.10	0.53	0.23	0.45

TABLE 3.—Toxicity of DDT granular formulations to mosquito larvae, ½ lb. actual DDT per acre.

	Percent mortality[a] ± standard deviation		
Formulation	5 hours	16 hours	24 hours
Attaclay 30–60 AA RVM	99±1	100	..
Attaclay 30–60 A LVM	96±1	100	..
Attaclay 20–35 AA RVM	98±1	100	..
Florex 30–60 AA RVM	99±1	100	..
Florex 30–60 AA LVM	97±0	100	..
Pikes Peak 9H66 calcined	90±1	100	..
Celatom MP-78	94±1	100	..
Tobacco	20±5	95±5	99±2
Corncob 14–20	26±5	85±8	92±2
Vermiculite No. 4	1±1	13±2	25±2
Attaclay 30–60 AA RVM, acetone[b]	62±7	97±1	97±1
Attaclay 30–60 AA RVM, melted[b]	49±6	92±1	100

[a] Average of three determinations.
[b] Applied at 0.46 lb. DDT actual/acre.

larvae, the effect being evident 1 to 2 hours after application. At 0.25 lb. DDT actual per acre no differences were observed between these various attapulgite formulations. Such formulations apparently are so efficient in releasing and dispersing DDT that evidence of their possible differences must await testing at lower dosages.

Group 2. HAN-impregnated formulations of Celatom and Pikes Peak Clay 9H 66 were slower acting and less toxic than *Group 1.*

Group 3. Formulations of Attaclay AA RVM 30–60 prepared by acetone impregnation and by melting DDT were slightly more toxic initially than formulations of *Group 4.*

Group 4. HAN-impregnated corncob and tobacco were slower acting but approximately equal in 24-hour toxicity to *Group 3.*

Group 5. HAN-impregnated vermiculite, unlike corncob and tobacco, did not release visible quantities of solvent while floating on the surface of the water, and was relatively non-toxic at 0.25 lb. DDT actual per acre. The inclusion of 2 percent emulsifier in vermiculite formulations (Table 5) increased toxicity to at least that of *Group 4.*

The toxicity of the above formulations may also be due to the form of the dispersed DDT. Since DDT is virtually insoluble in water, the type of solvent and type of granular carrier probably determine the form of the dispersed DDT. In HAN-impregnated granules, the major portion of the DDT is dissolved in the HAN, which in turn is dispersed as small droplets in the water. If the carrier disintegrates in water or if the carrier sample contains a fraction of very small "fines," these small particles may carry additional amounts of DDT and HAN and, when in suspension, may be ingested by mosquito larvae. With the exception of attapulgite RVM, however, most granular carriers tend not to disintegrate in water, and, therefore, this latter form of dispersed DDT probably does not contribute greatly to the toxic efficiency of HAN-impregnated granules.

TABLE 4.—Toxicity of DDT granular formulations to mosquito larvae, 1/4 lb. actual DDT per acre.

Formulation	Percent mortality[a] ± standard deviation		
	5 hours	16 hours	24 hours
Attaclay 30–60 AA RVM	98±1	100	..
Attaclay 30–60 A LVM	98±1	100	..
Attaclay 20–35 AA RVM	99±1	100	..
Florex 30–60 AA RVM	99±1	100	..
Florex 39–60 AA LVM	99±1	100	..
Pikes Peak 9H66 calcined	72±0	96±2	98±2
Celatom MP-78	61±8	94±4	95±4
Tobacco	6±2	94±3	98±2
Corncob 14–20	12±6	72±12	82±12
Vermiculite No. 4	0	2±3	3±3
Attaclay 30–60 AA RVM, acetone[b]	35±3	85±7	89±6
Attaclay 30–60 AA RVM, melted[b]	18±1	76±9	82±9

[a] Average of three determinations.
[b] Applied at 0.23 lb. DDT actual/acre.

TABLE 5.—Toxicity of DDT-vermiculite granules with various percentages of emulsifier, 1/2 lb. actual DDT per acre.

Percent emulsifier, Emcol H 500X	Percent mortality ± standard deviation, 24 hours
0	22±10
0.4	42±4
1	69±10
2	94±3
Control[b]	0

[a] Average of three determinations.
[b] Contained Emcol at a concentration equal to formulation containing 2 percent.

In DDT granules prepared by acetone impregnation or DDT melting, where no solvent is present to aid the release of DDT from the granule, the importance of DDT on suspended minute carrier particles when present would be increased. In addition, the formulation of colloidal DDT particles over an extended period of time probably would assume more importance.

CONCLUSIONS. Laboratory tests to study various granular formulations for (1) their dispersal of DDT and (2) their toxicity to mosquito larvae have shown that these two properties are affected greatly by the type of carrier, method of preparation, and additives such as emulsifiers. Other factors, such as grades and particle size of carriers, sometimes play a lesser role. The formulation that was found to release and disperse DDT in greatest amounts and to be one of the most toxic to mosquito larvae was attapulgite LVM or RVM prepared by DDT impregnation with heavy aromatic naphtha.

Many other factors such as concentration of solvent and DDT, non-volatile solvents other than HAN, temperature, type of water (e.g., salt, brackish, fresh), and additives to retard toxicant release, need to be studied. The latter is of particular interest in the development of formulations for (1) pre-season treatment and (2) treatment of areas which dry up and reflood.

Although these investigations have dealt only with DDT, many of the findings may be applicable to other toxicants used for mosquito larval control. Dieldrin and other insecticides, which are solids and have water solubilities similar to DDT, may be affected in granular formulation by the same factors and to a similar degree as DDT.

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