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The Chief of the Malaria Section  
has the honour to communicate hereunder the  
following note:

THE DELIVERY AND ESTIMATION OF  
DEPOSITS OF WATER-DISPERSIBLE INSECTICIDE  
FORMULATIONS IN MALARIA CONTROL

by

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The object of this paper is to describe some of the problems which have been met in estimating insecticide deposits in the field by chemical methods. The problems described are those which have been posed in Western Sokoto, Northern Nigeria, in the course of the Malaria Control Pilot Project which is being carried out there.

Three insecticides are in use in the project area, DDT, BHC, and dieldrin. The chemical estimation of the amounts of these insecticides sprayed on to walls and roofs, and the amount persisting as time passes, is of obvious importance.

DDT and BHC formulations are used at such a sufficiently high concentration or with such a high amount of the inert "filler" that their deposits are plainly visible on all types of surface. This is in contradistinction to the recently introduced insecticides, aldrin and dieldrin. These are used at lower working concentrations than either DDT or BHC and the deposit is not visible, especially on the mud walls of buildings in the tropics. These newer insecticides are more costly weight for weight than DDT or BHC, and their accurate spraying is an economic necessity.

## I. SAMPLING METHODS

The collection of samples can be made either during the actual spraying of a building or immediately after the sprayman has left. For reasons to be discussed later the second method is more accurate than the first for assessing the dosage delivered.

Two methods have been used for sampling the sprayed deposit during the application, (1) the direct weighing of the deposit sprayed on to a known area of weighed aluminium foil, and (2) exposing sheets of absorbent paper to the spray followed by extraction and chemical analysis of the toxicant. The former method has the advantage of rapidity and convenience since it is capable of use at the spraying site. However it is subject to a number of errors which can give rise to inaccurate results. It has been found that the second method, sampling on absorbent paper, provides a more accurate means of assessing the deposit sprayed on to a surface.

### 1.1 Sampling with metal foil

A number of previously dried and weighed pieces of corrugated aluminium foil, 6 inches square, are securely fixed at various points on the surface to be sprayed. After spraying the foils are dried and are reweighed. The increase in weight of the foils gives the dosage of formulation sprayed.

Although the method is extremely simple it is subject to serious error which makes its value in the field very doubtful. Appreciable loss of formulation occurs due to splashing and draining of the spray from the foil's surface. Unless the foils are weighed immediately after drying, loss of powder can occur due to "flaking" and, if the foils have to be transported any distance before weighing, by mechanical vibration. The foils are also liable to pick up particles of the wall surface to which they have been attached. Since the amount of deposit on 6 sq. in. of foil will be of the order of 100 mg for DDT and BHC spraying, and 25 mg for dieldrin, a large error can be introduced in the weighing, especially under field conditions.

## 1.2. Sampling with absorbent paper

This method eliminates all the errors liable to be introduced when aluminium foil is used to sample the deposit. Splashing and drainage from absorbent paper is negligible and the papers can be packed and transported to the laboratory without any loss occurring. It has been found more convenient to employ foolscap size absorbent paper rather than filter papers, since this enables a larger area of the spray pattern to be sampled.

The sampling procedure is applicable to all water-dispersible powders. The absorbent paper used is foolscap size, 13 in. x 8 in. (32.5 cm x 20.5 cm) duplicating paper. In a circular African hut with mud walls 5-6 ft high, 12-15 ft in diameter and with a conical roof 9-12 ft high, four sheets of paper have been found to give a representative result. The four sheets are fastened securely with drawing pins to the walls to be sprayed equidistant round the hut and at heights of 1, 3 and 5 ft from the floor, and one midway between the top of the wall and the apex of the roof. After spraying the papers are immediately taken down whilst still moist in order to avoid any mechanical loss. The papers are allowed to dry in the air before extraction and chemical analysis.

These two procedures, for assessing the dosage of an insecticide formulation sprayed on a surface during the actual spraying operation, are subject to considerable human error. The actual placing of visible sampling sheets in a building to be sprayed produces a psychological tendency for the sprayman to give the sheets preferential treatment. Analysis will indicate, erroneously, heavy application.<sup>1</sup> It is not uncommon for an unsupervised labourer to spray the same sheet two or three times if not watched the whole time. When supervised, higher dosages will always be obtained due to the normal effect of the sprayman taking extreme care when under observation. Since the absolute dosage is not usually required in the field, this increase is negligible compared with the other factors involved.

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<sup>1</sup> Davidson, G. (1955) J. trop. Med. Hyg. 58, 49

In order to eliminate the human tendency to overspray when under observation, a more accurate assessment of the deposit can be made by sampling the sprayed surface immediately after the application. On smooth non-porous surfaces such as painted walls, metal work, glass etc., a sample can be obtained by wiping with moistened cotton wool. On surfaces which are porous, such as the sun-dried mud of buildings in the tropics, a greater problem arises. It is not practicable to wipe or brush the surface without losing much of the sample through airborne dispersion. To overcome this it has been the practice to scrape the surface attempting to remove only the outer superficial deposit. On walls of coarse texture the amount of surface removed will not necessarily be the superficial deposit and an alternative method has been proposed.<sup>2</sup> This method involves the pressing of strips of adhesive tape firmly to the sprayed surface. On removal from the wall the tape retains the superficial insecticide deposit. A proportion of loose mud granules also adhere but this is negligible when compared with that obtained by scraping. In either of these two methods the toxicant is extracted and determined chemically. It is also necessary to determine if any deposit remains from a previous spraying before the new dosage is applied.

### 1.3 Sampling by scraping the surface

Many different types of scraping appliances have been tried for scraping mud surfaces. They include scalpels, razors, chisels etc., but they all tend to abrade the surface causing loss of a certain amount of the sample as airborne material. It has been found in practice that a sharpened triangular paint scraper, with sides 6 cm long, is the most efficient means of scraping friable surfaces.<sup>3</sup> This is used in conjunction with a metal stencil incorporating a suitably sized trough to collect the scrapings and giving a scrapable surface of 3 in. x 3 in. Since most of the wall surfaces are uneven it is necessary to collect the scrapings which fall down behind the stencil on a sheet of paper spread beneath the portion of wall being sampled. In a circular hut four samples, giving a sampled area of 36 sq. in., taken

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<sup>2</sup> Barlow, F. (1953) Working document WHO/Insecticides/22

<sup>3</sup> Bruce-Chwatt, L. J. Private communication

equidistant round the hut and at graduating distances up the wall, have been found to give a representative sample for the whole hut.

The chemical determination of insecticide residues on mud walls has often been made during field trials using this procedure. The insecticide found is intended to give an indication of the deposit available for pick-up by alighting insects at intervals after spraying. As pointed out by Barlow (loc. cit.) it is uncertain how much of the recovered insecticide was actually lying on the outer surface of the wall. The thinner the layer removed the more likely is it that all the insecticide found was directly available for pick-up off the surface. Under field conditions where the walls are of coarse texture and often containing a considerable proportion of fortuitously admixed straw, it is impossible to remove less than 0.3 to 0.5 mm of the surface. A large proportion of the toxicant recovered from this surface may not be available to insects alighting on it for two reasons, (1) if the wall is of coarse texture the toxicant may have penetrated into cracks inaccessible to the insect, and (2) on a typical tropical mud wall in which sorption occurs, the greater proportion of the insecticide found in the 0.3 to 0.5 mm layer may be held by adsorptive forces and again not available for pick-up.

#### 1.4 Sampling with adhesive tape

To obtain a more exact knowledge of the external deposit of toxicant "entomologically" available, Barlow has put forward the sampling procedure using adhesive tape (loc. cit.).

Six 2 in. x 2 in. pieces of transparent adhesive tape are cut from a 2 in. wide roll of "Sellotape". In the field it is more accurate to cut off a 12 in. strip first and cut this into six approximately equal lengths. These pieces are pressed firmly at regular graduating distances round the hut. Each piece is rubbed with a rounded piece of wood to ensure adequate contact with the surface: it was found that the handle of the paint scraper already described gave the best results. The six pieces from one hut are peeled off and placed together in a 150-ml flask. Transference into the flask is facilitated by rolling each strip into a cylinder.

The insecticide is extracted from the tape by shaking with four successive quantities of 20 ml of acetone. The acetone extract is filtered into a 150-ml flat-bottomed flask and the insecticide estimated chemically. A blank determination is carried out on the "Sellotape".

This procedure gives accurate results on most types of mud surfaces except extremely coarse or rough walls. Even on these surfaces the method gives a more reliable indication of surface deposits although the efficiency of recovery may be of the order of 80%. The tape method also partially eliminates the interfering colour obtained on extraction of wall scrapings.

#### 1.5 Mensuration of the area sprayed

The most direct and quickest method of assessing the deposit is by mensuration of the total area sprayed by a known volume of diluted formulation. Assuming that the sprayman maintains a steady rate of spraying, then by observing the number of buildings sprayed, the total area sprayed can be calculated. In the case of the poorly educated labour used in control schemes this would enable the controller of the spraying operations to obtain a quick check on any particular sprayman. In this way the traverse rate for the whole team could be standardized, which if accomplished, would lead to greater accuracy and economy than is at present the practice.

In circular huts with conical roofs the total surface area which is sprayed is given by:

$$\text{Area} = 1.6 D (L + 2h)$$

Where D is the diameter, L the slant height of the roof and h is the height of the wall. The distance L is usually easier to measure than the height of the roof.

This formula can be simplified further since the majority of huts have an average constant roof pitch of 50°:

$$\text{Area} = 3.2 D (0.4 D + h)$$

with the symbols having the same significance as before.

## 2. PREPARATION OF SAMPLES FOR ANALYSIS

Both sample papers and wall scrapings are extracted in a suitable extraction apparatus with a solvent. In the field it is convenient to use an extraction apparatus consisting of a 500-ml conical flask and an internal coil condenser. The sample, in a Whatman extraction thimble or the sample paper itself, is attached to the lowest whorl of the condenser by means of a piece of wire. The flask is heated on a sand bath ensuring that the reflux passes through the sample. This assembly is more robust than the usual Soxhlet extractor, a factor which is advantageous when the supply position is precarious.

Acetone has been found convenient for the extraction of DDT, BHC and dieldrin samples, but it is better to use n-hexane for BHC and benzene for dieldrin. Highly coloured extracts are generally obtained from wall scrapings when acetone is the solvent. This colour is particularly troublesome in the determination of insecticides as chloride by Volhard titration, since the extraneous colour is the same as the end-point of the titration. The chloride can be estimated potentiometrically when the colour of the solution is of no consequence. The coloured extracts are due to the presence of ground nut oil, which is used for cooking, on the walls of most of the inhabitable huts. The colouring was at first thought to be due to the presence of soot on the walls but this has now been found not to be the case. Cattle dung is also used to decorate the interior of huts in some localities.

The interfering colour can be partially avoided by the use of non-polar solvents such as petroleum ether, hexane or benzene. However even with these solvents some colour is obtained. This can be removed by passing the extract down a column containing fuming sulfuric acid absorbed on an inert clay. It is necessary to use a solvent for the insecticide which does not react with the sulfuric acid. Carbon tetrachloride is the usual solvent.

The solvent is evaporated from the extract and the residue is dissolved in 20 ml of carbon tetrachloride. Three g of a high-grade kieselguhr (Johns-Manville's Hyflo supercel is suitable) is intimately mixed with 20% oleum (2 ml) in a mortar and enough

carbon tetrachloride to form a stiff slurry. Five ml of carbon tetrachloride is poured into a glass cylinder, 10 cm long, closed at the lower end with glass wool, and the mixture added with light tapping. A pyrex test tube, 19 mm x 150 mm with a hole blown in the bottom, was found to be satisfactory. When excess of the carbon tetrachloride has almost ceased to drain, the extract is poured into the column and filtered into a 150-ml flask. The column is washed out with four successive portions of 3-4 ml of carbon tetrachloride. The carbon tetrachloride is evaporated off and the residue taken to dryness by blowing in a stream of air from a blowing ball to avoid loss of insecticide by volatilization.

A simpler method for eliminating the colour is by passing the coloured extract through a column containing 3 g of hyflo supercel intimately mixed with 1 g of decolourizing charcoal. The test tube column already described is used. The supercel charcoal mixture is mixed to a smooth paste with the extraction solvent and added to the column. Five ml of the solvent is poured in and when this just ceases to drain, the extract, evaporated to approximately 25 ml, is added. The column is washed out with four successive 5-ml portions of the solvent. Benzene and petroleum ether are the best solvents to use for this method.

### 3. CHEMICAL ANALYSIS OF THE EXTRACTS

#### 3.1 DDT

An acetone extract, containing not more than 400 mg of DDT, is evaporated to approximately 25 ml and cooled to 25-30°C; 10 ml of 1 N ethanolic potassium hydroxide (methylated spirit is suitable) is added to the acetone solution. The flask is kept at 25-30°C for 15 minutes with occasional shaking and then 50 ml of distilled water is added. Ten ml of 2 N nitric acid and exactly 10.0 ml of 0.1 N silver nitrate, or a suitable excess, are then added. The precipitated silver chloride is coagulated by digesting on a water bath for 15-30 minutes with frequent shaking. The silver chloride is filtered off and washed thoroughly with small amounts of distilled water. 2.0 ml of 40% ferric ammonium sulfate indicator containing 2.5% nitric acid is added and the excess silver nitrate titrated with N/20 potassium

thiocyanate. The end-point is the first appearance of the red ferric thiocyanate colour in the bulk of the solution. A blank determination is carried out by repeating the above procedure but omitting the DDT. The difference between the two titres gives the volume of potassium thiocyanate equivalent to the chloride in the original DDT.

It is convenient to standardize the reagents by making a further determination using DDT of accurately known purity. In this way errors resulting from impurities in the reagents, errors in the method itself, the handling of the method in a given laboratory and errors resulting from climatic variations, are eliminated.

### 3.2 BHC

BHC may conveniently be estimated either by the labile chloride method or by a determination of the total chlorine.

For the labile chloride method an acetone extract is evaporated down to a volume of about 2 ml and 10 ml of a 1 N solution of ethanolic potassium hydroxide is added. The mixture is refluxed for half an hour and 20 ml of water is added. The solution is acidified with 2 N nitric acid; 2 ml excess is added. The chloride is determined as described for DDT, a slight excess of 0.1 N silver nitrate being added to precipitate all the chloride ion as silver chloride. One ml of 0.1 N silver nitrate is equivalent to 0.00970 g of BHC. Alternatively the reagents may be standardized by making a further determination using BHC of accurately known purity.

The second method requires the extraction of the sample with n-hexane. The extract is evaporated to approximately 50 ml and when relatively cool 1.5 g of sodium, cut into small pieces, and 5 ml of isopropyl alcohol are added. After refluxing vigorously for 30 minutes, excess sodium metal is removed by adding 20 ml of distilled water cautiously down the condenser. The refluxing is continued until no sodium is visible. After cooling to room temperature a further 20 ml of water is added. The flask is removed and the solution neutralized by adding 50% nitric acid and then 2.0 ml in excess. The contents are transferred to a separatory funnel and the flask washed out with two successive portions of 20 ml of distilled water.

After shaking vigorously the layers are allowed to separate and the aqueous portion run off into a conical flask. The hexane layer is washed twice with 20 ml portions of water and these washings added to the contents of the conical flask.

The chloride in the solution is determined as previously described. In this case 1 ml of 0.1 N silver nitrate is equivalent to 0.00485 g of BHC.

This method can be used for semi-micro quantities of BHC. In this case the chloride is precipitated by adding a slight excess of N/20 silver nitrate and the excess titrated with N/40 potassium thiocyanate. In tropical climates difficulty is often experienced in titrating silver solutions more dilute than this.

### 3.3 Dieldrin

Dieldrin cannot be determined by labile chlorine methods and a total chloride determination has to be used. A revised Stepanow determination is described, this can also be used for BHC and DDT.<sup>4</sup> Under conditions of excessive humidity the reaction with sodium in isopropyl alcohol does not appear to proceed to completion and a technique similar to that described for BHC has been successfully used. This gives accurate results down to 5 mg of dieldrin in the sample, below this amount the chloride content of the reagents decreases the accuracy.

The procedure is identical with that for BHC except that the extraction is performed with AnalaR benzene. If other grades of benzene are used interference in the determination is caused by the presence of thiophene which results in the formation of sulfides and hydrogen sulfide on acidification. A precipitate of silver sulfide is formed when silver nitrate is added. Petroleum ether can also be used and has advantages over the use of benzene.

The benzene extract is evaporated to approximately 50 ml and 1.5 g of sodium is added. 5.0 ml of isopropyl alcohol is added and the mixture refluxed vigorously for two hours. The extraction and chloride estimation are carried out as described for BHC. One ml of 0.1 N silver nitrate is equivalent to 0.00633 g of dieldrin.

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<sup>4</sup> World Health Organization, Expert Committee on Insecticides (1952) Fourth Report, 6.1.1.

### 3.4 Purification of sodium for sodio-reduction determinations

In determinations involving the use of sodium it is advantageous to purify the sodium, since all grades of sodium are liable to contain varying amounts of chloride unevenly distributed through the sample.

This can be performed adequately by carefully melting a quantity of sodium under liquid paraffin in a pyrex test tube. When all the sodium has melted it is gently stirred to bring the dross to the surface and to agglomerate the globules. The clean sodium is withdrawn through a heated pipette and released under some more liquid paraffin in a separate test tube. When required the sodium is washed with AnalaR benzene or n-hexane and cut into small pieces.

## 4. CONCLUSIONS PRESENTED BY THE RESULTS OBTAINED

There are several contributory causes why the theoretical dosage may not be obtained on a particular surface. In the Western Sokoto scheme a series of analyses showed that incorrect or uneven dosages were obtained due to five main factors. These were (i) the measuring out of incorrect amounts of formulation, (ii) the influence of the wall height of the sprayed building, (iii) the influence of the suspensibility of the formulation, (iv) the personal factor in the sprayers and (v) wear in the sprayers.

### 4.1 Incorrect measure

In imagocidal schemes it is the practice to issue the formulation to the sprayers in containers cut to hold the required weight of charge. For convenience these containers are usually cut at the headquarters where the stock of insecticide is held. After several months' storage all water-dispersible powders settle so that the volume of the powder is as much as 30%-40% less than it was on arrival. This is particularly noticeable with 50% dieldrin powders. When drums of formulation are transported over unmade roads to the site of the spraying programme, the powder is violently agitated and assumes its original bulk. The measure cut before the transfer of the powder will now hold a decreased weight of charge. In one case a

nominal 9 oz measure of a 50% dieldrin water-dispersible powder weighed before transportation was found to contain only 6-1/2 oz at the spraying site. It is therefore necessary to check the actual weight of the formulation issued to the spraymen at regular intervals.

#### 4.2 The influence of the wall height of the buildings

A series of results are shown in Table 1 for the spraying of a 75% DDT water-dispersible powder. The deposit was sampled with absorbent paper placed at fixed distances up the walls. After spraying, the papers were removed and the DDT determined as already described. The scheduled dosage was 270 mg/ft<sup>2</sup>.

Table 1. Dosage of the DDT deposit in relation to the height on the circular wall in mud huts in Western Sokoto

| Team number | Dosage of technical DDT in mg/ft <sup>2</sup> |      |      |      |
|-------------|---|------|------|------|
|             | 1 ft  | 3 ft | 6 ft | roof |
| Team 1      | 218   | 192  | 179  | 196  |
| Team 2      | 321   | 306  | 278  | 273  |
| Team 3      | 194   | 183  | 173  | 142  |
| Team 4      | 262   | 288  | 271  | 321  |

Note: The distances were measured from the floor.

The results show that the deposit is not uniform, there being a tendency for the deposit to decrease with the height of the wall. This is caused by the spraymen failing to keep the spray nozzle at a fixed distance from the wall surface. At the top of the wall, and on the roof, the nozzle is further away from the surface and a decreased deposit results. Decreased deposits will also be caused by the extra effort necessary to hold the spray lance above the sprayman's head causing

him to move more quickly over the higher parts of buildings. The results for Team 4, Table 1, were for a tall sprayman who was able to reach the top of the wall and the roof better than his shorter co-workers.

This non-uniform spraying must be taken into account when samples of the surface are taken for evidence of residual activity of insecticides. The samples should be taken at varying heights along the hut wall to give a truly representative result.

#### 4.3 The influence of the suspensibility of the formulation

The importance of the suspensibility of a water-dispersible powder has been often over-emphasized. Provided that the powder disperses readily on addition of water, even a suspensibility as low as half that required by WHO specifications will enable the powder to be sprayed without leaving any residue. This is true especially with compression pumps since the dispersion is constantly being agitated by the action of the spraymen carrying the pumps from house to house. The most important property of a water-dispersible powder in the field is that it should be readily wetted on addition of water. If this does not occur the powder will not disperse but agglomerate into a plastic, unsprayable, mass. Powders which are wetted with difficulty can be efficiently sprayed by thoroughly mixing the formulation in a separate container before adding the diluted dispersion to the sprayer. It is found convenient to mix all DDT water-dispersible powders in this way as they are liable to have low suspensibilities and do not readily become wetted with water.

When a water-dispersible powder of low suspensibility is sprayed, a variable deposit rate is obtained. In trials with a 50% dieldrin water-dispersible powder in a compression pump, samples of the spray were taken at varying intervals. The results are shown in Table 2.

However with the constant agitation involved in the use of compression pumps, the dispersion will be continuously remixed during the spraying and the suspensibility greatly enhanced.

Table 2. Deposit rates of a dieldrin formulation in relation to the time after the preparation of the suspension

| Time after mixing<br>(minutes) | Dieldrin in mg/ft <sup>2</sup> |      |
|--------------------------------|--------------------------------|------|
|                                | (A)                            | (B)  |
| 0                              | 56.2                           | 48.2 |
| 5                              | 42.1                           | 38.6 |
| 10                             | 36.8                           | 33.5 |
| 15                             | 48.8                           | 49.0 |

A water-dispersible powder may show low suspensibility and fail to disperse readily with water after prolonged storage in the tropics. The chief cause of this deterioration is due to the powder becoming damp in the high humidity. The particle size is increased and the suspensibility is lowered. The danger of this occurring is increased by the widespread practice of packing water-dispersible powders in unlined fibre drums. These are not completely impervious to water and the powder readily absorbs moisture. This cause of deterioration in storage can be eliminated by the use of air-tight steel drums.

The disadvantage of using fibre drums was shown when a large consignment of a 50% dieldrin water-dispersible powder packed in this way was found to have a lower suspensibility than required by specification. After prolonged storage the suspensibility was only 21% and the powder failed to be wetted at all by water. In this case it is most probable that insufficient suspending agent had been added to the formulation. It was found possible to spray this powder by the addition of a surface active agent to increase the suspensibility. When "Teepol", a highly soluble branched chain C<sub>10</sub> - C<sub>18</sub> sodium alkyl sulfate, was added, the suspensibility was increased to a maximum value of 60%. The concentration of the surface active agent in the solution to give this maximum value for the suspensibility approximated to the critical micelle concentration of the "Teepol". Since "Teepol" is marketed

as a 20% solution no difficulty was experienced in dispensing the correct quantity. The powder was "pre-mixed" and was sprayed as efficiently as a powder conforming to WHO specification.

#### 4.4 The personal factor in the spraymen

The chief cause for incorrect dosages is the use of faulty techniques by the spraymen. As emphasized by Davidson (loc. cit.) this is due to the widespread practice of employing the least intelligent of the available labour for the actual spraying operation. This class of labour is incapable of learning from its mistakes and wide variations in dosages can always be expected (Table 1). The discrepancy between the deposits delivered by various spraymen is shown in Table 3 which gives the dosages obtained during the spraying of a 50% dieldrin powder.

Table 3. Individual variations of the deposit of a dieldrin formulation by various spraymen

|          |   | Dieldrin deposit in mg/ft <sup>2</sup> |      |
|----------|---|--|------|
|          |   | A                                      | B    |
| Sprayman | 1 | 79.2                                   | 84.4 |
| "        | 2 | 70.4                                   | 64.9 |
| "        | 3 | 41.8                                   | 83.4 |
| "        | 4 | 49.0                                   | 48.8 |
| "        | 5 | -                                      | 48.8 |
| "        | 6 | -                                      | 62.2 |

The theoretical dosage was 50 mg/ft<sup>2</sup>. The variations cannot be attributed to wear on the sprayers since the sprayers were in good condition and had new stainless steel nozzles.

#### 4.5 Wear in the sprayers

After considerable usage it is found that pump nozzles become eroded and deliver an increased volume of spray. It is particularly noticeable when brass nozzles are used. This wear caused by normal usage is in addition to wear on the pumps caused by gross mishandling.

During the spraying of a 75% DDT water-dispersible powder, three successive average determinations of the sprayed deposit after approximately two-week intervals showed increased deposits. The values increased initially from  $240 \pm 50 \text{ mg/ft}^2$  to  $320 \pm 40 \text{ mg/ft}^2$  and at the conclusion of the spraying cycle a deposit of  $345 \pm 50 \text{ mg/ft}^2$  was recorded. The nozzles after this last determination were showing visible signs of wear. The use of stainless steel nozzles would minimize this erosion.

#### SUMMARY

Procedures for sampling insecticide deposits in the field, both for evidence of the dosage delivered during spraying and for residual activity, are discussed. The estimation of sub-macro amounts of DDT, BHC and dieldrin in samples, and problems connected with interference in the usual analytical techniques are described. Results on the spraying of water-dispersible powders are reviewed in the light of field experience.