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STORAGE STABILITY OF DDT WATER-DISPERSIBLE POWDERS *

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SYNOPSIS

The authors describe a study of the change in suspensibility of two series of 75% DDT water-dispersible powders during storage in simulated commercial packages at 27°C, 50°C and 65°C and as "shelf" samples at ambient temperatures. All the products tested in both series were made under the conditions of regular commercial production, and all those in the second series were prepared in 1954 under contract to the International Cooperation Administration (ICA) and according to ICA specifications.

The results showed that: (*a*) a high initial suspensibility is no criterion of "shelf" life; (*b*) the suspensibility after the so-called "tropical storage pre-treatment" is no criterion of stability, except perhaps in the case of very poorly formulated products; (*c*) periodic observations, made over a period of a month or more, of the suspensibility of powders stored in commercial or simulated commercial packages at 50°C and 65°C will provide an indication as to the probable long-term stability of the products in question; and (*d*) it seems likely that a reasonable estimate of long-term storage stability at ambient temperatures could be obtained by subjecting the samples to high temperatures for a very short period.

In the preceding paper, Pearce, Gooden & Johnson[1] presented some background information on the use of DDT water-dispersible powders in malaria control programmes sponsored by the International Cooperation Administration (ICA) and the World Health Organization (WHO). The present report concerns studies of the effect of high-temperature storage on the suspensibility of the DDT powders generally available in the USA in 1954-55, including a series manufactured under ICA contract. As indicated by Pearce et al.[1] and Johnson[2] the ability to stay in suspension

* Paper submitted to WHO Expert Committee on Specifications and Chemistry of Pesticides, November 1958

[1] See article on page 913.

[2] Johnson, D. R. (1953) *Agric. Chem.*, 8, No. 3, p. 97

for a substantial period of time is a prime requisite of powders to be used in malaria control programmes. This property is commonly referred to as suspensibility and will be used as the principal measure of the stability of the powders under study in this report.

Significance of Suspensibility Values

Before presenting the data on storage, it seems desirable to establish some guides for interpreting suspensibility values. These values are determined by a highly empirical procedure in which the amount of DDT remaining in suspension in an aliquot taken at the mid-point of a graduated cylinder is determined after allowing a thoroughly mixed suspension containing 2.5 g of DDT per 100 ml (i.e., 2.5% weight/volume) of water of a specified hardness and temperature to stand for 30 minutes. Both the WHO Expert Committee on Insecticides [1] and the ICA [2] require that 1.5% DDT or 60% of the original concentration of a 2.5% suspension be present in the aliquot. [3] In addition, the ICA requires a suspensibility of 1.2, or 48%, after " tropical storage pre-treatment " and WHO [4] a suspensibility of 1.25%, or 50%, after similar treatment. Although these minimum requirements are arbitrary, experience indicates that an initial suspensibility of 1.5 for a product is sufficiently high for practical purposes. The WHO Expert Committee on Insecticides [5] had originally suggested a value of 2.0, which is hard to meet without adding substantially to the cost. Moreover, it has been generally observed that products with a very high suspensibility are usually very difficult to re-suspend once they have settled. Assuming then that 1.5 is a satisfactory practical standard so far as manufacture and use are concerned, the question arises as to what is the lower limit that can be tolerated in the field. In this connexion it is noteworthy that practically all of the products used in overseas malaria control programmes that have been referred to the Technical Development Laboratories (TDL), Savannah, Ga., USA, because of poor suspensibility have given values of less than 1.0 in suspensibility tests. This suggests that a suspensibility of 1.0 may be taken as the limit below which difficulties will begin to be experienced in use. A product which does not deteriorate in suspensibility during long storage in tropical areas below 1.0 could be expected to

[1] World Health Organization, Expert Committee on Insecticides (1952) *Wld Hlth Org. techn. Rep. Ser.*, 54, 18

[2] International Cooperation Administration (1956) *DDT, 75 percent, water-dispersible powder*, Washington, D C (ICA Specification No. 81056, 10 August 1956)

[3] In this paper " suspensibility " will be expressed as a number representing the weight to volume percentage of DDT in suspension. " Percentage suspensibility ", which is coming into use, represents the percentage of the original amount of DDT remaining in suspension—for example, $\frac{1.5}{2.5} \times 100 = 60\%$ suspensibility.

[4] World Health Organization (1953) *Specifications for pesticides*, Geneva, p. 106

[5] World Health Organization, Expert Committee on Insecticides (1951) *Wld Hlth Org. techn. Rep. Ser.*, 34, 21

give little trouble. However, it should be pointed out that difficulties can and have been experienced with powders which give higher values than 1.0. The major difficulties encountered with such products appear to be excessive caking with failure of the caked material to disintegrate readily on exposure to water and/or the presence of some coarse aggregates which tend to clog the spray nozzles. The latter difficulties do not appear to have been experienced with products made to ICA specifications.

Storage Experiments

The tropical storage stability of DDT water-dispersible powders has been considered extensively by the WHO Expert Committee on Insecticides. The general opinion of the Committee seemed to indicate that storage of these powders under the high temperatures frequently encountered in tropical and semi-tropical areas was likely to produce serious deleterious effects, especially on the suspensibility characteristics. In view of the fact that little or no direct experimental or field observational data were available to evaluate this factor, it was recommended that TDL undertake some storage studies on representative commercial products. Accordingly, two series of samples were tested for storage characteristics. The first series was a group obtained in 1954 mainly through the co-operation of the National Agricultural Chemicals Association and its membership.[1] In the request to formulators for products to be used in the study, it was indicated that both experimental and production-line materials would be acceptable; but it was specified that all products be made in regular production equipment. Although 75% DDT powders were preferred, it was also indicated that some 50% and 67% DDT products would be of interest. Since only a very small number of the latter were received, the present report will be limited to 75% products.

The second series of samples was obtained in 1955 from production-line materials supplied under ICA contracts and meeting the ICA specification.[2] Thus, this series represents material actually purchased under the specification and sent overseas.

Experimental method

All products were received packaged in 100- or 150-pound (45 or 70 kg) fibre drums with sealed plastic liners. In Series I, the amount of each sample received varied from about 300 to 1000 pounds (135-450 kg) and in Series II single 100-pound drums of each sample were supplied. One-

[1] The following companies contributed materials for Series I: E. I. duPont deNemours & Co.; General Chemical Division; Michigan Chemical Co ; Niagara Chemical Division; Stauffer Chemical Co.; Geigy Agricultural Chemicals.

[2] The following companies supplied products for Series II: Diamond Alkali Co.; Geigy Agricultural Chemicals; Michigan Chemical Co ; Monsanto Chemical Co.; Niagara Chemical Division; Pesticide Export Corporation; Stauffer Chemical Co.

to two-pound (about 0.5-1 kg) laboratory reference samples were taken from each product on receipt. All products were repackaged and tested for storage qualities by the following procedure:

Samples of each product weighing approximately 8-10 pounds (3.5-4.5 kg) were placed in mailing-tubes—outside diameter, 4 inches (10 cm); length, 36 inches (90 cm)—with a plywood end and a polyethylene bag liner. The powder was tamped to a weight-to-height ratio equivalent to that in the original drum container when received, the bag liner was sealed and the samples thus repackaged were placed in storage cabinets held at three different temperatures: ambient (approximately 27°C), 50°C, and 65°C. At intervals of one week or longer, a tube was removed, opened, and examined for visible changes; and samples were taken from the top and bottom and tested for suspensibility by the ICA method.[1]

Experimental results

Summaries of the suspensibility data on the cylinder-stored samples and on shelf-stored samples for Series I and Series II are presented in Tables 1 and 2, respectively. The values for the cylinder-stored samples are averages of the values obtained on samples taken from the top and bottom of each cylinder when opened. The values for " As received ", " After tropical storage pre-treatment " and " After shelf storage " were all determined on the shelf samples taken at the time of repackaging into the cylinders. In the case of Series II (Table 2), insufficient amounts of the original samples were available for sets of cylinder-packaged samples to be stored at ambient temperature. However, enough of Samples 228, 229, 230 and 231 was available to place one cylinder of each in storage at ambient temperature, and these were held for one year before examination.

Casual inspection of the data in Tables 1 and 2 indicates that deterioration in suspensibility occurred with all products and at all temperatures, the degree of deterioration increasing as the storage temperature increased. In general, the largest drop in suspensibility tended to take place in the first week or month. It is noteworthy that some products withstood the 50°C and 65°C storage temperatures remarkably well. See, for example, Samples 52, 105, and 227 (Table 1) and Samples 228, 231 and 234 (Table 2). There seems to be no relation between initial suspensibility and storage stability. Compare 51 and 227 (Table 1) and 228 and 232 (Table 2), for example. Also, the suspensibility after tropical storage pre-treatment is not indicative of storage stability. Apart from the foregoing observations it is difficult to draw any general conclusions from the data in Tables 1 and 2 without further analysis. If one plots suspensibility against storage time for each sample at a given temperature the trend is towards the production of a

[1] International Cooperation Administration (1956) *DDT, 75 percent, water-dispersible powder*, Washington, D.C. (ICA Specification No. 81056, 10 August 1956)

TABLE 1. EFFECT OF STORAGE TEMPERATURE ON SUSPENSIBILITY OF 75% DDT WATER-DISPERSIBLE POWDERS: SERIES I

Sample No.	Suspensibility									
	as received	after TS pre-treatment *	after cylinder storage							after 4 years' shelf storage **
			°C	1 month	3 months	4½ months	6 months	9 months	12 months	
51	1.90	1.85	27	1.65	1.65	1.55	1.65	1.50	1.50	0.30
			50	1.10	1.15	1.10	1.05	1.30	1.05	
			65	0.80	0.60	0.50	0.40	0.40	0.20	
52	1.95	1.70	27	1.75	1.70	1.80	1.60	1.70	1.65	1.60
			50	1.20	1.35	1.20	1.45	1.55	1.35	
			65	0.75	0.65	0.50	0.50	0.45	0.45	
80	1.70	1.80	27	1.65	1.55	1.70	1.65	1.45	1.45	0.65
			50	1.55	1.00	0.75	0.35	0.35	0.35	
			65	0.25	0.15	0.10	0.10	0.05	0.05	
104	1.90	1.85	27	1.70	1.65	1.70	1.60	1.55	1.45	0.80
			50	1.30	1.20	1.45	1.05	0.95	0.90	
			65	0.45	0.25	0.30	0.35	0.20	0.20	
105	2.00	1.95	27	1.65	1.70	1.65	1.70	1.60	1.70	1.60
			50	1.35	1.00	1.00	0.85	0.65	0.80	
			65	1.40	0.50	0.40	0.40	0.30	0.35	
106	1.60	1.40	27	1.65	1.55	1.40	1.55	1.50	1.40	0.90
			50	0.90	0.55	0.70	0.80	0.80	0.65	
			65	0.70	0.40	0.35	0.30	0.30	0.30	
171	1.30	1.40	27	1.35	1.30	1.40	1.25	1.35	1.30	0.55
			50	1.15	1.25	1.25	1.30	1.25	1.30	
			65	1.10	0.95	0.70	0.60	0.80	0.55	
227	1.50	1.25	27	1.50	1.45	1.40	1.40	1.40	1.35	1.35
			50	1.10	1.25	1.30	1.25	1.20	1.30	
			65	0.95	0.95	0.95	0.90	0.90	0.95	

* TS pre-treatment = tropical storage treatment, which consists in heating a sample with a specified weight on it for 20 hours at 55°C.

** One- to two-pound samples taken from the original drums as received were placed in common storage, during which the temperature varied approximately from 15°C to 40°C, probably averaging about 30°C.

TABLE 2. EFFECT OF STORAGE TEMPERATURE ON SUSPENSIBILITY OF 75% DDT WATER-DISPERSIBLE POWDERS: SERIES II

Sample No.	Suspensibility								
	as received	after TS pre-treatment *	after cylinder storage						after 3 ½ years' shelf storage **
			°C	1 week	1 month	4 months	6 months	1 year	
228	2.00	2.00	27	—	—	—	—	1.85	1.30
			50	1.70	1.75	1.35	1.20	—	
			65	1.40	1.10	0.70	0.55	—	
229	1.50	1.55	27	—	—	—	—	0.95	0.70
			50	1.40	1.35	1.25	1.15	—	
			65	0.85	0.95	0.40	0.35	—	
230	1.50	1.40	27	—	—	—	—	1.10	1.00
			50	1.45	1.10	0.80	0.80	—	
			65	1.10	0.85	0.65	0.65	—	
231	2.20	2.15	27	—	—	—	—	1.05	1.10
			50	1.80	1.75	1.75	1.60	—	
			65	1.65	1.95	1.75	1.65	—	
232	2.35	2.00	50	1.45	1.35	1.00 ***	0.95	—	0.30
			65	1.00	0.90	0.40 ***	0.30	—	
233	1.70	1.70	50	1.65	1.70	1.60 ***	1.40	—	0.60
			65	1.00	0.65	0.65 ***	0.55	—	
234	1.80	1.50	50	1.20	1.30	1.10 ***	0.95	—	0.50
			65	1.05	1.10	1.10 ***	0.75	—	

* TS pre-treatment = tropical storage treatment, which consists in heating a sample with a specified weight on it for 20 hours at 55°C.

** One- to two-pound samples taken from the original drums as received were placed in common storage, during which the temperature varied approximately from 15°C to 40°C, probably averaging about 30°C.

*** 3-months' storage.

parabolic curve. However, because of variations in the history of the samples, sampling errors and errors in suspensibility measurement, considerable deviation from a parabolic function is encountered for some samples. In an effort to arrive at some general observations, several methods of treating the data in Tables 1 and 2 have been attempted. The one that seemed most informative and at the same time valid consisted in averaging

the suspensibility values for all samples in a series, as received, after tropical storage pre-treatment and at each temperature and each storage period. Subsequently, the samples in each series were grouped according to suspensibility after shelf storage. Group 1 in each series included all shelf samples with suspensibility values of 1.0 or above and Group 2, all samples with suspensibility values below 1.0. The results of treatment of the data in Tables 1 and 2 in this manner are presented in Tables 3 and 4, respectively. The average suspensibilities at each temperature and at each storage time have also been averaged and included in the tables.

To illustrate the effect of storage temperature on suspensibility, the averages of all suspensibilities at each temperature and storage time have been plotted, using the average suspensibility of all samples as received as the initial or zero time. A graph of Series I data (Table 3) is presented in Fig. 1. The Series II data (Table 4) are similarly presented in Fig. 2. These figures illustrate rather clearly that deterioration in suspensibility during

TABLE 3. AVERAGE SUSPENSIBILITIES: DATA FROM SERIES I (TABLE I)

As received	After TS pre-treatment*	After 4 years' shelf storage	After cylinder storage						
			°C	1 month	3 months	4½ months	6 months	9 months	12 months
All samples									
1.73	1.65	0.97	27°	1.61	1.57	1.58	1.55	1.51	1.48
			50°	1.21	1.09	1.09	1.01	1.01	0.96
			65°	0.80	0.56	0.48	0.44	0.42	0.38
			Average	1.21	1.07	1.05	1.00	0.98	0.94
Samples grouped according to shelf suspensibilities									
Group 1 — Nos. 52, 105, 227 — shelf suspensibility, 1.00 or above									
1.82	1.63	1.52	27°	1.63	1.62	1.62	1.57	1.57	1.57
			50°	1.22	1.20	1.17	1.18	1.13	1.13
			65°	1.03	0.70	0.62	0.60	0.55	0.58
			Average	1.29	1.17	1.14	1.12	1.08	1.09
Group 2 — Nos. 51, 80, 104, 106, 171 — shelf suspensibility, below 1.00									
1.68	1.66	0.65	27°	1.60	1.54	1.55	1.54	1.47	1.42
			50°	1.20	1.03	1.05	0.91	0.93	0.85
			65°	0.66	0.47	0.39	0.35	0.35	0.26
			Average	1.15	1.01	1.00	0.93	0.92	0.84

* TS pre-treatment = tropical storage treatment, which consists in heating a sample with a specified weight on it for 20 hours at 55°C.

FIG. 1. CHANGE IN AVERAGE SUSPENSIBILITY WITH STORAGE TIME FOR SERIES I POWDERS STORED AT 27°C, 50°C AND 65°C

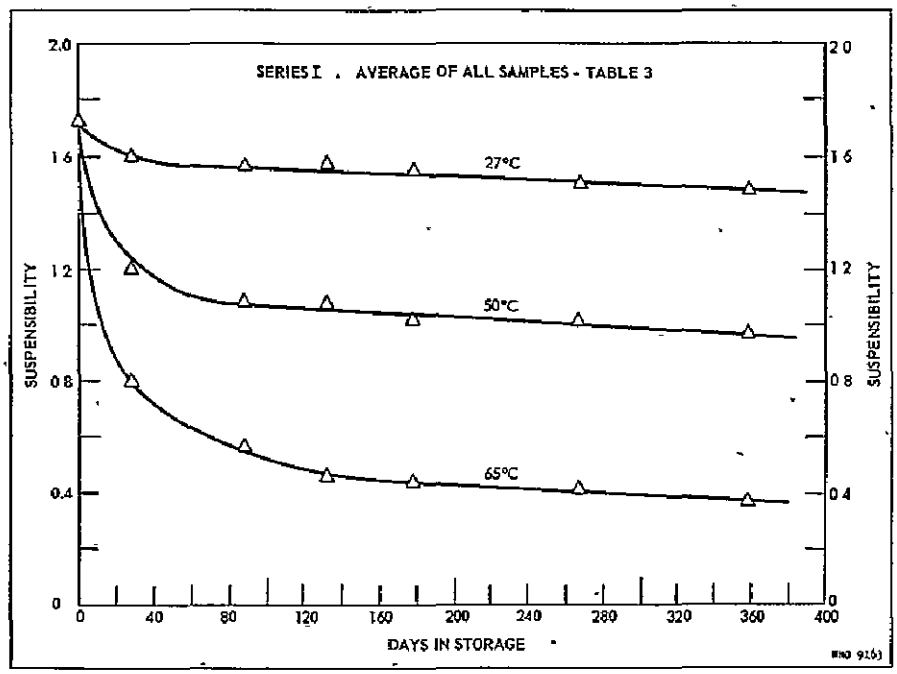


FIG. 2. CHANGE IN AVERAGE SUSPENSIBILITY WITH STORAGE TIME FOR SERIES II POWDERS STORED AT 50°C AND 65°C

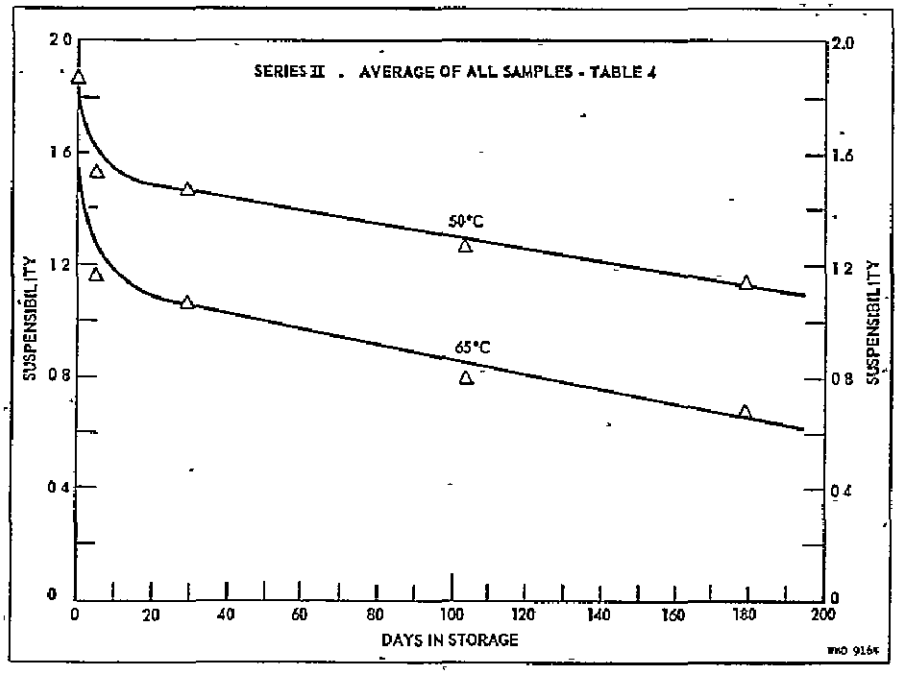


TABLE 4. AVERAGE SUSPENSIBILITIES: DATA FROM SERIES II (TABLE 2)

As received	After TS pre-treatment *	After 3½ years' shelf storage	After cylinder storage				
			°C	1 week	1 month	3-4 months	6 months
All samples							
1.86	1.76	0.79	50°	1.52	1.47	1.26	1.15
			65°	1.15	1.07	0.81	0.69
			Average	1.34	1.27	1.04	0.92
Samples grouped according to shelf suspensibilities Group 1 — Nos 228, 230, 231 — shelf suspensibility, 1.00 or above							
1.90	1.85	1.13	50°	1.65	1.53	1.30	1.20
			65°	1.38	1.30	1.03	0.95
			Average	1.52	1.42	1.17	1.08
Group 2 — Nos. 229, 232, 233, 234 — shelf suspensibility, below 1.00							
1.84	1.69	0.52	50°	1.43	1.43	1.24	1.11
			65°	0.98	0.90	0.64	0.49
			Average	1.21	1.17	0.94	0.80

* TS pre-treatment = tropical storage treatment, which consists in heating a sample with a specified weight on it for 20 hours at 55°C.

storage follows the same pattern regardless of temperature—i.e., there is a relatively rapid loss in suspensibility within the first month of storage and thereafter the loss is very gradual. In other storage tests it has been observed that it takes about 3 to 7 days' cylinder storage at 50°C and 65°C for a substantial loss in suspensibility to occur. As indicated by the data presented here, the higher temperature produces the more rapid deterioration. These data also offer an explanation as to why the suspensibility after tropical storage pre-treatment does not provide a good indication of the probable storage stability of a product—namely, that the heating period is too short (20-24 hours at 55°C). Thus, the average value after one year's storage at 27°C is about 1.50 (Fig. 1) and this value is reached after about one week's storage at 50°C. One can also see from these curves that storage experiments at ambient temperatures should be started very soon after formulation or one is liable to be starting the tests with a product which is already at or near the flat portion of the deterioration curve.

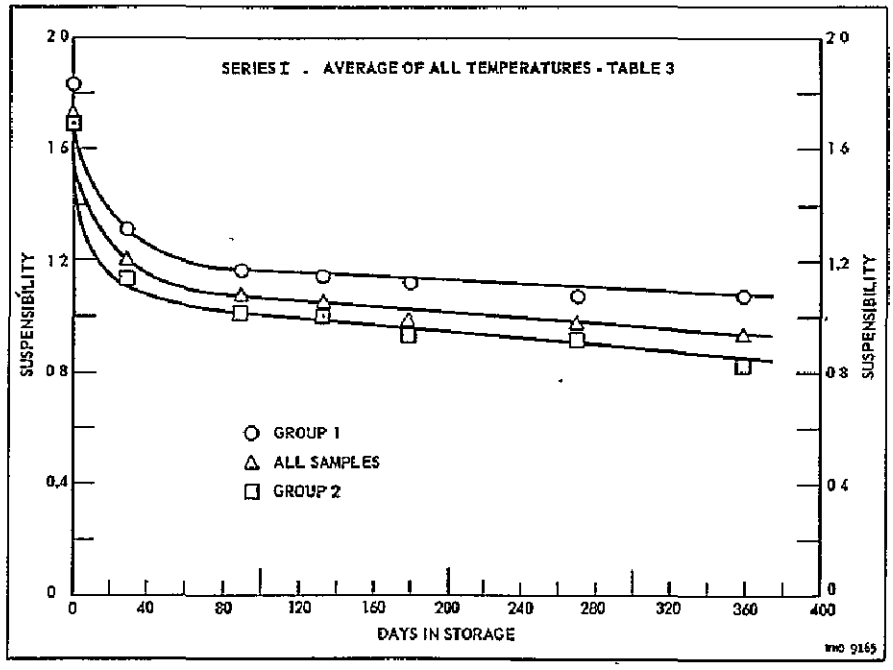
The graphs in Fig. 1 and 2 also indicate that one should be able to devise a high-temperature short-term exposure treatment which would be indicative of long-term storage stability at ambient temperatures. Experi-

ments aimed at this have been started. If one plots the data employed in preparing Fig. 1 and 2 on semi-log paper, i.e., suspensibility against log of time, the curves obtained approximate to straight lines. The authors have attempted to work out equations for each temperature and then by extrapolation and interpolation estimate how many hours at a given high temperature would be required to produce the same suspensibility loss at ambient temperature after one year's storage. It became evident that such a procedure would introduce great errors and the only reliable way of arriving at a rapid laboratory high-temperature test was by actual experimentation. One of the major difficulties in using the present data as a basis for a very short high-temperature storage test is that the deviation from a straight-line semi-log function increases as the temperature increases.

One other point should be mentioned with regard to Fig. 1 and 2. The curves represent the average behaviour of a number of different formulations and, thus, cannot be applied directly to a given formulation. However, similar-type curves can be predicted for most formulations, although the precise storage properties for each should be determined experimentally.

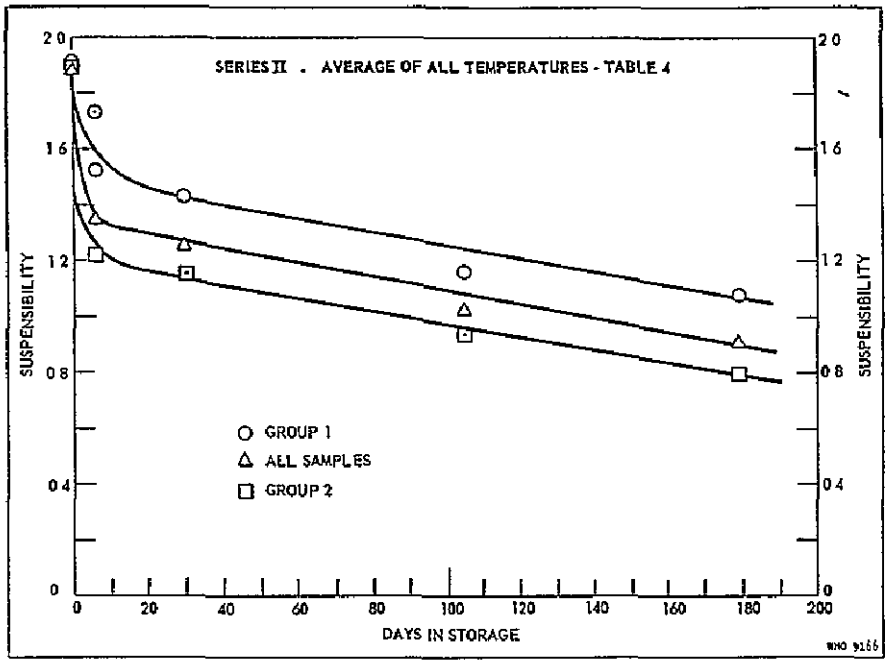
Fig. 3 and 4 have been prepared to illustrate that storage tests of the kind undertaken in this study will provide a good indication of the long-term storage stability of the water-dispersible powders in actual use. The

FIG. 3. CHANGE IN AVERAGE SUSPENSIBILITY* WITH STORAGE TIME AT ALL TEMPERATURES: SERIES I



* Values for all samples and for samples grouped according to shelf suspensibilities are shown.

FIG. 4. CHANGE IN AVERAGE SUSPENSIBILITY* WITH STORAGE TIME AT ALL TEMPERATURES: SERIES II



* Values for all samples and for samples grouped according to shelf suspensibilities are shown.

average suspensibilities at all temperatures and after each storage period were plotted against storage time, using the average suspensibility as received for each group as zero time. The data are taken from Tables 3 and 4 for Fig. 3 and 4, respectively. As pointed out earlier, there is reason for believing that products which show suspensibilities below 1.0 are likely to give trouble in the field. The products were therefore grouped according to whether their suspensibility after shelf storage was 1.0 or above, or below 1.0. The three curves shown in Fig. 3 and in Fig. 4 represent the average of all samples in the respective series (middle curve), the average of all samples with a shelf suspensibility of 1.0 or above (top curve), and the average of all samples with a shelf suspensibility below 1.0 (bottom curve). This grouping of the samples clearly illustrates that relatively short-term storage behaviour at high temperatures will indicate long-term stability in use. The difference in the vertical spread within each set of curves is a reflection of the fact that Series II did not include samples held at ambient temperature in the cylinder-storage tests.

The cylinder-storage method adopted in this study has been used for several years by TDL for evaluating the stability of water-dispersible formulations of both DDT and dieldrin. In practice it has been found that storage in cylinders at 50°C and 65°C for two to four weeks with analysis

of samples at 3, 7, 14 and 28 days will provide a very good indication of the storage stability in the field. One of the most important observations made in connexion with storage tests is that individual batches of a given formulation can show wide variation in storage stability. This has been observed in the case of one formulation where data on both high-temperature storage at TDL and storage overseas were available. Why this should be is not known. There is some indication that it is caused by variation in the composition of the diluent employed. In any case, the use of a very fine and highly uniform diluent seems to correct this variation between batches if careful mixing and milling conditions are also maintained during manufacture. Improvement in the latter factors does not correct deficiencies due to poor quality diluents; however, it has been observed that careful mixing and ample milling are essential to the production of a uniform product. Sample or batch variation has been encountered in the present study. Note, for example, the storage data on Samples 51 and 52 (Table 1) held at 50°C: after one month's storage these samples were lower in suspensibility than they were after nine months' storage. As indicated by Pearce et al.,[1] the present ICA specifications are designed to include inspection of batches at the time of manufacture, in order to ensure maximum uniformity in the performance of all batches.

Physical Condition of Stored Samples

It was anticipated that various degrees of caking or gross agglomeration of the products placed in storage, especially at 50°C and 65°C, were likely to occur. Observations of the condition of the samples on removal from storage showed that caking became more evident as the storage time increased. In determinations of the suspensibility of caked products, the cake was disintegrated by crushing with a spoon or hammer when necessary, care being taken to avoid any grinding action. Several degrees of hardness in the caking could be distinguished. The condition of the samples, which were taken from both the top and the bottom of the cylinders, was graded thus:

A. Powder with or without soft agglomerates, usable as is for preparing suspensions.

B. Soft cake or mass, easily disintegrated by mild crushing action with spoon.

C. Firm cake, disintegrated by strong crushing action with spoon.

D. Hard cake, disintegration with hammer necessary.

Products exhibiting conditions A or B would not cause difficulty in handling in the field. With some extra attention a product in condition C

[1] See article on page 913

TABLE 5. SUSPENSIBILITY AND PHYSICAL CONDITION OF 75% DDT WATER-DISPERSIBLE POWDERS AFTER TWELVE MONTHS' STORAGE: SERIES I

Sample No.		Storage at 27°C		Storage at 50°C		Storage at 65°C	
		suspensi-bility	condition	suspensi-bility	condition	suspensi-bility	condition
51	Top	1.6	A	1.1	A	0.2	D
	Bottom	1 4	B	1.0	B	0.2	D
52	Top	1.7	A	1.4	A	0.2	C
	Bottom	1.6	B	1 3	B	0.7	C
80	Top	1.5	B	0.4	B	0.06	D
	Bottom	1.4	B	0.3	C	0.05	D
104	Top	1.5	A	1.1	A	0.1	A
	Bottom	1.4	B	0.7	B	0.3	C
105	Top	1.7	A	0 6	A	0.4	B
	Bottom	1.7	B	1.0	B	0.3	C
106	Top	1.5	A	0.7	B	0 3	C
	Bottom	1.3	B	0 6	C	0 3	D
171	Top	1 2	A	1.3	A	0.4	B
	Bottom	1.4	B	1.3	B	0.7	B
227	Top	1 5	A	1.45	A	0.95	B
	Bottom	1.2	B	1.15	B	0.9	B

is believed usable. Products exhibiting condition D would be usable in the field only with great difficulty.

A summary of the data collected on the samples at the end of the storage period, showing the condition grading along with the suspensibility value, is presented in Tables 5 and 6 for Series I and II, respectively.

As might be expected, the data indicate a tendency for more or harder caking to occur at the bottom of the cylinders. However, there is no consistent correlation between caking condition and suspensibility, except possibly at the extremes. It is noteworthy that after storage at 50°C and 65°C, most samples were found to be in condition A or B and that only six samples were graded as " D ". The variation in suspensibility between top and bottom exhibited here is more or less typical of all such data. An analysis of all top and bottom suspensibility results justified using averages of the two results for the data presented in Tables 1-4.

TABLE 6. SUSPENSIBILITY AND PHYSICAL CONDITION OF 75% DDT WATER-DISPERSIBLE POWDERS AFTER SIX MONTHS' STORAGE: SERIES II

Sample No.		Storage at 50°C		Storage at 65°C	
		suspensibility	condition	suspensibility	condition
228	Top	1.2	B	0.5	B
	Bottom	1 2	B	0.6	C
229	Top	1.2	B	0.3	C
	Bottom	1 1	B	0.4	C
230	Top	0.7	B	0.6	C
	Bottom	0 9	B	0 7	B
231	Top	1 7	A	1.7	A
	Bottom	1.6	B	1.6	B
232	Top	0 9	B	0 3	C
	Bottom	1.0	C	0.3	D
233	Top	1.5	A	0.5	A
	Bottom	1.3	B	0.6	B
234	Top	1.0	B	0.7	C
	Bottom	0.9	C	0.8	C

The observations with respect to top and bottom suspensibilities have a bearing on the tropical storage pre-treatment method. This procedure consists in heating a sample with a weight on it calculated to be equivalent to the pressure at the bottom of a 100-pound drum. The fact that the suspensibility values of samples taken from the bottom of the cylinders were not consistently lower than those of samples taken from the top indicates that the use of the weight in the tropical storage pre-treatment procedure has no significance. However, in work now in progress it has been observed that removal of the weight in that procedure usually results in a decrease in suspensibility over that of the weighted sample. This phenomenon is being investigated.

Conclusions

The following comments are based largely on the data presented in this paper, but they are influenced to some extent by subsequent experiments

and general observations made on the performance of both DDT and dieldrin water-dispersible powders.

Periodic observations, made over a period of a month or more, of the suspensibility of powders stored in commercial or simulated commercial packages at 50°C and 65°C will provide an index as to the probable long-term stability of DDT or dieldrin water-dispersible powders in the field, so far as suspensibility is concerned. In conducting storage tests, the cylinders described on page 924 may be used in lieu of the actual drum containers with sealed liners. In the case of products in short supply, data obtained on samples taken from a single package opened and resealed at each sampling may be satisfactory. The main advantage of using individual packages for each storage period is that the results will indicate the uniformity of the product. At present no standards of performance for stability in storage at 50°C and 65°C have been established. However, it is believed that products which show less than 20% reduction in suspensibility after one month at 50°C and less than 40% at 65°C will probably possess good stability at ambient temperatures for long periods of time. Commercial products have been tested which show remarkable ability to withstand these high temperatures for long periods.

The study presented here clearly indicates that deterioration of the suspensibility of water-dispersible powders during storage tends to follow a parabolic function, but that there is a large difference in the rate of change of different formulations and often a marked variation in samples of the same formulation. The loss in suspensibility during storage is due primarily to change in particle size and this change can be accelerated by increasing the storage temperature. A rate phenomenon is indicated which suggests that a very short period of storage at high temperatures could form the basis of a rapid laboratory test for determining storage stability at ambient temperatures. This possibility is being investigated.

A high initial suspensibility is not a criterion of shelf life. The suspensibility after tropical storage pre-treatment is not a criterion of stability, except possibly in the case of very poorly formulated products. Thus, products which show a severe drop in suspensibility after such pre-treatment should be looked upon with suspicion.

RÉSUMÉ

Les auteurs ont étudié les changements de l'aptitude à se mettre en suspension de deux séries de poudres de DDT à 75% dispersables dans l'eau, soumises, en emballage commercial, à des températures de 27°, 50° et 65°C.

Les observations périodiques, pendant un mois ou plus, de la dispersabilité des poudres soumises à ces températures, donnent une indication sur le maintien de cette qualité après une longue conservation à la température ambiante, dans les conditions de la pratique. Jusqu'à présent, on n'a pas établi de normes de stabilité après conservation à 50° ou 65°C. Toutefois, on estime que les produits qui présentent moins de 20% de réduction de

leur dispersabilité après un mois à 50°C et moins de 40% à 65°C possèdent une bonne stabilité à la température ambiante, durant de longues périodes. Certains produits commerciaux se sont révélés excellents à cet égard.

D'après les études présentées dans cet article, la diminution au cours de la conservation de l'aptitude des poudres à se mettre en suspension, est une fonction parabolique, mais la vitesse à laquelle les modifications se produisent est variable. Il existe aussi de grandes différences entre les échantillons. Le changement dans la dimension des particules est la principale modification. Il s'accélère à mesure que la température s'élève. L'expérience a montré que les résultats obtenus à la suite d'une conservation de courte durée à haute température peuvent servir de fondement à un test rapide de laboratoire, permettant d'évaluer les qualités de conservation d'une poudre à la température ambiante.

Un degré élevé de dispersabilité de la poudre fraîchement préparée n'est nullement garant de sa stabilité à la suite d'une longue conservation en stock. Les critères actuellement en vigueur pour le traitement avant emmagasinage dans les conditions tropicales sont éventuellement à reviser.

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