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A New High-Temperature Test for Predicting the Storage Stability of DDT Water-Dispersible Powders *

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The importance of providing DDT water-dispersible powders having good initial suspensibility and good storage life for use in malaria control or eradication programmes has been clearly indicated in the report of Pearce, Gooden & Johnson.^a

Pearce, Goette & Sedlak^b observed that neither the initial suspensibility nor the suspensibility after the so-called "tropical storage pre-treatment" gives an indication of the probable storage life of these powders. However, their study did suggest that if the tropical storage test is modified by a substantial increase in temperature of treatment, then the effect on suspensibility would correlate with probable change in suspensibility at ambient temperatures. Miles et al.^c carried out preliminary studies in this direction and demonstrated that treatment of the powders at 70°C or 75°C for 1-20 hours produced a lowering of suspensibility similar to that observed by Pearce et al.^b at 50°C and 65°C for much longer periods. Thus, in general the change in suspensibility with time tends to approach a parabolic function and the effect of temperature is to increase the relative slope.

In order to make use of these observations in establishing a high temperature treatment that would be indicative of the storage stability at normal temperatures, it was necessary to establish an acceptable limit for the lowering of suspensibility at the temperature selected. The present paper provides data suitable for this purpose and describes an empirical heat-treatment procedure that gives reproducible results.

* Paper presented at the Division of Agricultural and Food Chemistry, American Chemical Society, 139th Meeting, St. Louis, Mo., USA, in March 1961. The studies described here were accomplished as part of a contractual agreement between the Communicable Disease Center and the International Cooperation Administration (now Agency for International Development).

^a Pearce, G. W., Gooden, E. L. & Johnson, D. R. (1959) *Bull. Wld Hlth Org.*, 20, 913.

^b Pearce, G. W., Goette, M. B. & Sedlak, V. A. (1959) *Bull. Wld Hlth Org.*, 20, 921.

^c Miles, J. W., Goette, M. B., Grimm, L. & Pearce, G. W. (1959) *Bull. Wld Hlth Org.*, 20, 937.

Methods

Early in 1960, all formulators who were manufacturing DDT powder for the International Cooperation Administration (ICA) were requested to furnish the Technical Development Laboratories of the Communicable Disease Center with 10 fresh 1-pound samples of DDT 75% water-dispersible powder taken from their regular production over a period of several days. Upon receipt, these samples were tested for suspensibility according to ICA Specification 72059.^d At the same time, another portion of each sample was heated for two hours in an oil-bath according to the procedure outlined below and the suspensibility determined. The samples were then sealed in glass jars and stored on a shelf in a non-air-conditioned laboratory. Samples were withdrawn from the jars at 2, 8, 24, 36 and 52 weeks and tested for suspensibility.

The procedure for the 70°C oil-bath treatment was as follows. Five grams of DDT 75% water-dispersible powder were placed in a 25 mm × 200 mm test-tube, and the tube was clamped in an oil-bath maintained at 70°C ± 0.1°C to a depth of 3½ inches (9 cm). After two hours, the tube was removed and allowed to come to room temperature. The powder in the tube was mixed gently with a spatula to break up lumps, and 3.33 g were weighed out for the suspensibility test. The suspensibility (expressed as a number representing the weight to volume percentage of DDT in suspension) was then determined by the method described in ICA Specification 72059.^d

Results

A summary of the suspensibility data is presented in Table 1. The data for 2, 8, 24 and 36 weeks have been omitted to conserve space. Several observations of significance can be made from the data presented in this table. Note that the ranges in

^d International Cooperation Administration (1959) *DDT, 75 percent, water-dispersible powder*, Washington, D.C. (ICA Specification 72059, 20 July 1959).

TABLE 1
SUMMARY OF SUSPENSIBILITY DATA ON PRODUCTION SAMPLES

Manufacturer ^a	No. of samples	Suspensibilities					
		As received		After 2 hours at 70°C		After 52 weeks' ambient storage	
		Range	Average	Range	Average	Range	Average
A	10	2.06-2.21	2.14	1.83-1.99	1.92	1.74-1.88	1.82
B	10	1.76-1.89	1.81	1.66-1.89	1.77	1.51-1.69	1.57
E	10	1.87-2.18	2.02	1.53-2.09	1.85	1.42-1.93	1.72
G	10	1.57-2.22	1.82	0.98-2.12	1.48	0.78-1.88	1.17
H1 ^a	10	1.70-1.94	1.80	1.78-1.93	1.87	1.43-1.84	1.70
H2 ^a	10	1.68-1.82	1.77	1.67-1.80	1.72	1.39-1.68	1.71
I	10	1.44-2.02	1.76	1.08-1.63	1.44	1.39-1.68	1.51
M	10	2.12-2.31	2.20	1.28-1.66	1.45	1.36-1.70	1.53
N	10	1.89-2.15	2.03	0.82-1.48	1.18	0.51-1.66	0.88

^a Manufacturer H supplied two sets of samples.

suspensibilities "as received" for the samples submitted by manufacturers A, B, H, M and N are relatively small, whereas those for G, I and E are substantially larger. Thus, three of the eight manufacturers submitted samples which indicate considerable variation in initial performance. The ranges in suspensibility after two hours at 70°C are quite wide for all manufacturers except A, B and H; and only A and B supplied samples which exhibited a narrow range after 52 weeks at ambient storage. A correlation between suspensibilities after two hours at 70°C and 52 weeks' storage is suggested by

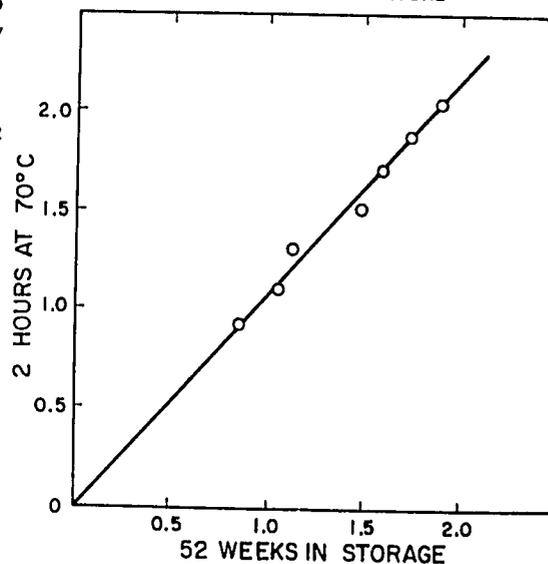
the data as presented in Table 1, but this correlation is much better illustrated by the presentation in Table 2.

The data have been arranged in Table 2 without regard to the manufacturer. The samples the suspensibilities of which after two hours at 72°C fell

TABLE 2
SUSPENSIBILITIES AFTER 2 HOURS AT 70°C AND AFTER 52 WEEKS' STORAGE

No. of samples	Average as received	Range after 2 hours at 70 C	Average after 52 weeks
4	1.89	0.80-0.99	0.74
5	1.91	1.00-1.19	1.05
10	1.91	1.20-1.39	1.12
14	1.96	1.40-1.59	1.48
25	1.84	1.60-1.79	1.60
25	1.98	1.80-1.99	1.74
5	2.14	2.00-2.19	1.89

CORRELATION BETWEEN SUSPENSIBILITIES AFTER 2 HOURS AT 70 C AND AFTER 52 WEEKS OF STORAGE AT AMBIENT TEMPERATURE



within ranges of 0.20 suspensibility unit were grouped together, and the number in each range is indicated, as well as their average suspensibilities both as received and after 52 weeks' storage. It is seen that with this arrangement of the data an excellent correlation is evident between suspensibility after two hours at 70°C and after 52 weeks' storage. A plot of these data in the accompanying figure shows a direct linear relationship. This finding is particularly advantageous since one can predict the suspensibility of a newly prepared product after a

year of storage by determining the suspensibility after two hours' treatment at 70°C. Actually, the 70°C test will be indicative of the suspensibility after a few months in storage, since the largest change in suspensibility occurs in the first few weeks in storage.

The correlation presented is believed particularly significant because the samples employed are from actual production lines and are representative of all products supplied to ICA in 1960. The 70°C test described herein has been incorporated in ICA Specification 2373-1 dated 1 June 1961.