



*Handwritten notes:*  
1959, 20, 947-953

REPRINT NUMBER

271

*Bull. Org. mond. Santé* } 1959, 20, 947-953  
*Bull. Wld Hlth Org.* }

Furnished by  
Communicable Disease Center  
Technical Development Laboratories  
P. O. Box 769, Savannah, Ga.

## FACTORS AFFECTING THE SUSPENSIBILITY OF DIELDRIN WATER-DISPERSIBLE POWDERS \*

GEORGE W. PEARCE, JAMES W. MILES, MARY B. GOETTE

*Technical Development Laboratories, Communicable Disease Center,  
Bureau of State Services, Public Health Service,  
United States Department of Health, Education, and Welfare,  
Savannah, Ga., USA*

### SYNOPSIS

Various dieldrin water-dispersible powder formulations have been subjected to elevated temperature storage and the effect on their suspensibilities compared. The Shell Chemical Company's 50% dieldrin formulation AC 1630 was recommended for use in the malaria control programmes of the International Cooperation Administration rather than its 50% formulation AC 1631, on the basis of superior performance after storage at 50°C and 65°C. In a similar series of tests on 75% dieldrin water-dispersible powders, Shell formulation AC 1621, which contains Hi-Sil as diluent, was judged better than AC 6621, which contains Micro-Cel as diluent. A series of samples of AC 1621 was subjected to temperatures of 90-120°C for periods of 1-20 hours and the effect on suspensibility determined. Most samples withstood the treatment reasonably well at temperatures up to 110°C for 20 hours.

As an introduction to this report it is believed desirable to present an historical background on dieldrin water-dispersible powders as far as their use in International Cooperation Administration (ICA) programmes is concerned. In 1955 it became apparent to the ICA that its malaria programmes were becoming interested in dieldrin powder as a substitute or alternate for DDT powder and would no doubt request supplies within a short time. Consequently, the Technical Development Laboratories (TDL), Savannah, Ga., USA, were requested to provide a specification for dieldrin powders. The Shell Chemical Company had already developed a formulation and specification for 50% dieldrin water-dispersible powder, designed to meet the standards for suspensibility required for use in malaria control programmes. The Shell formulation and specification was backed by a large amount of research data, which indicated that this formula would provide a good initial suspensibility as well as long storage life. In view of the very serious difficulties encountered initially with DDT

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

powders by the ICA and its predecessors, TDL recommended that the ICA should use the Shell specification, which is both a manufacturing and a performance specification. It is admitted that this is objectionable from a competitive bidding standpoint, but it was felt that every precaution should be taken which would ensure that satisfactory material was purchased for overseas use. The formulation called for in the specification adopted by the ICA<sup>1</sup> was designated AC 1630 by Shell, who supplied drum samples of it to TDL from different suppliers for high-temperature storage tests by the method described by Pearce, Goette & Sedlak<sup>2</sup> for testing DDT powders. In addition to this formulation, Shell also supplied their formulation AC 1631, which was also backed by considerable research data and was believed to be as good as AC 1630. However, the drum sample of AC 1631 did not stand up as well as AC 1630 to TDL's high-temperature tests and as a result AC 1631 was not accepted for use in ICA programmes. Later work has indicated that actually AC 1631 is as good as AC 1630. Shortly after the ICA adopted the Shell manufacturing and performance specification for AC 1630, a large order for this formulation was contracted for by the ICA and shipped to Indonesia. Within a short time of receipt complaints were received by the ICA that some batches or drums of this material were low in suspensibility. Both Shell and TDL undertook to determine the cause of the faulty batches. Although this incident did not seriously affect the control programme in Indonesia, since essentially all of the material was usable, the experience indicated that in spite of the extensive research backing the formula specified it was not fully satisfactory in the field and, as experienced with some DDT powders, wide variation in the performance of batches from the same formulation was encountered. By the time Shell had studied the Indonesian shipment carefully, TDL had obtained additional evidence that the AC 1630 formulation showed a similar behaviour in high-temperature storage tests—i.e., some batches stood up well and others deteriorated rather rapidly.

The explanation of this behaviour has not yet been established beyond question. However, the best explanation so far advanced seems to be that variation in the diluent (Attaclay) used in both AC 1630 and AC 1631 was principally responsible for the variation in storage properties. Shell had also tried Attaclay in 75% dieldrin powders and found it very unsatisfactory. This led to consideration of a change to a 75% dieldrin formulation using Hi-Sil as the diluent. It is economically feasible to use Hi-Sil in 75% dieldrin powders but not in 50%. Hi-Sil is the main diluent used in 75% DDT formulations and its use is believed to be largely responsible for the general good quality of the ICA DDT powders. Shell had already submitted

<sup>1</sup> International Cooperation Administration (1957) *ICA manufacturing specification for 50% dieldrin wettable powder*, Washington, D.C. (ICA No. 1, revised 13 February 1957)

<sup>2</sup> See article on page 921.

to TDL samples of 75% dieldrin containing Micro-Cel as diluent (AC 6621) and took steps to supply drum samples of a Hi-Sil formulation, AC 1621. Although only two drum samples of AC 1621 have been tested so far by TDL, both stood up remarkably well and in addition laboratory exposure to higher temperatures for shorter periods also indicated that this formulation is quite stable. At the same time, the 75% Micro-Cel formulation (AC 6621) has exhibited the same variation between batches or drums as the 50% formulations (AC 1630 and AC 1631). Some of the data on which the above observations are based will be presented in this report. On the basis of these and other data, much of which was supplied by Shell, the ICA has adopted a modified Shell manufacturing and performance specification<sup>1</sup> calling for a 75% dieldrin formulation with Hi-Sil as the diluent. The earlier 50% specification, while still available, has been relegated to the background as far as the ICA is concerned at the present time.

Pearce et al.<sup>2</sup> have made a study of the changes in the suspensibility of DDT water-dispersible powders during storage at elevated temperatures. These changes were found to parallel changes observed in powders held for longer periods under ordinary (shelf) storage conditions. In an extension of this work, Miles et al.,<sup>3</sup> studied the effects of still higher temperatures on DDT water-dispersible powders. An investigation of dieldrin water-dispersible powders along similar lines has been conducted and the results are described here. Both long-term medium-temperature storage and short-term high-temperature treatment were studied.

## Experimental

### *Cylinder storage at 50°C and 65°C*

A number of 50% and 75% dieldrin water-dispersible powders were placed in constant-temperature cabinets held at 50°C and 65°C. The samples were packed in sealed polyethylene-lined cardboard cylinders in the manner described by Pearce et al.,<sup>2</sup> so that the weight-volume ratio of the powder was the same as in the original shipping container. Samples were removed from storage and tested for suspensibility at intervals of one week to four months. Tests were made according to the ICA specifications referred to earlier. The results are presented in Table 1.

The 50% dieldrin formulations, AC 1630 and AC 1631, contain Attaclay as diluent. The 75% dieldrin formulation AC 6621 contains Micro-Cel and the 75% formulation AC 1621 has a Hi-Sil base. All the formulations were based on Shell specifications, but were manufactured by various

<sup>1</sup> International Cooperation Administration (1958) *ICA manufacturing and performance specification for dieldrin, 75 percent, water-dispersible powder*, Washington, D.C. (ICA Specification No. 5958, 9 May 1958)

<sup>2</sup> See article on page 921.

<sup>3</sup> See article on page 937.

**TABLE 1. EFFECT OF STORAGE TEMPERATURE ON SUSPENSIBILITY OF DIELDRIN WATER-DISPERSIBLE POWDERS**

Sample No.	Dieldrin (%)	Shell formulation No.	Suspensibility before storage (%)	Storage temperature (°C)	Suspensibility after storage (%)				Suspensibility after 2 1/2 years' shelf storage (%)
					1 week	4 weeks	12 weeks	16 weeks	
1	50	AC 1630	77	50 65	72 62	74 63	65 60	73 64	38
2	50	AC 1630	68	50 65	71	67	67	69	54
3	50	AC 1631	63	50 65	34 15	29 17	26 15	29 17	18
4	75	AC 6621	84	50 65	39 29	43 32	27 18	41	60
5	75	AC 6621	90	50 65	79 77	75 62	68 37	64	74
6	75	AC 6621	81	50 65	69 49	66 46	61 49		55
7	75	AC 6621	86	50 65	51 32	44 26	35 25	40 25	53
8	75	AC 1621	97	50 65	90 86	82* 84*	86** 78**		80***
9	75	AC 1621	88	50 65	73 64	71* 58*	65** 58**		

\* 2 weeks

\*\* 8 weeks

\*\*\* After 1 year

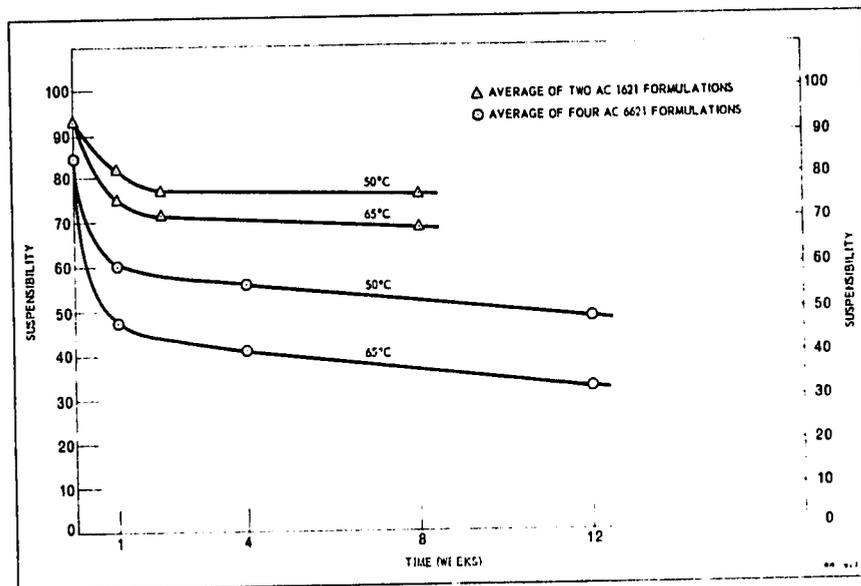
suppliers. A graphical presentation of the change in suspensibility with time at 50°C and 65°C is given in Fig. 1 and 2. It will be observed from Fig. 1 that the average suspensibility values for the Hi-Sil formulations are significantly higher than the corresponding values for the Micro-Cel formulations. In Fig. 2, a comparison of Attaclay formulations AC 1630 and AC 1631 is made. After observing the suspensibility of these powders after storage for 16 weeks at high temperatures, it was recommended that AC 1630 be adopted in preference to AC 1631. Other tests comparing these two 50% dieldrin formulations indicate that there is actually no important difference in their performance. The difference observed here is most likely a difference in batches. On the basis of these limited data, it would seem that the Hi-Sil formulation is superior to the Attaclay and Micro-Cel formulations.

#### *Short-term heat treatment*

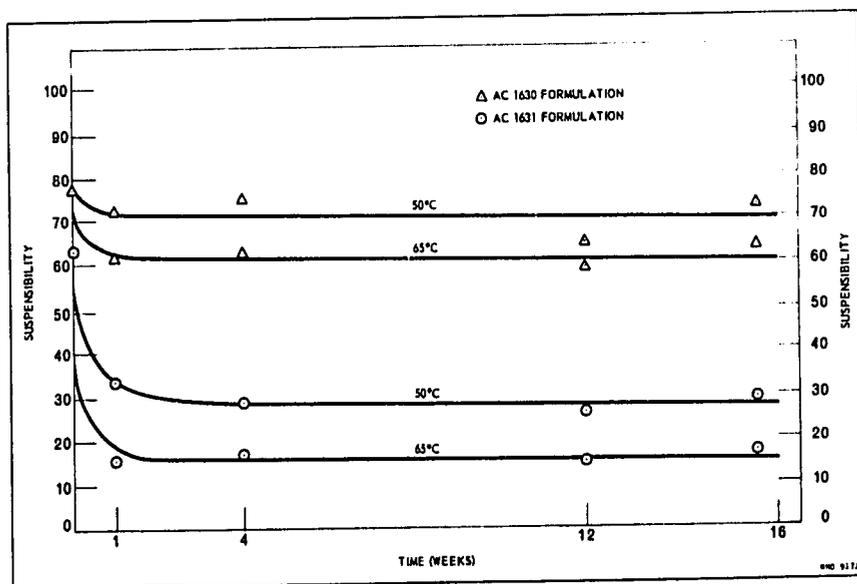
As in the case of DDT (Pearce et al.<sup>1</sup>) the storage data presented in Table 1 indicate that the deterioration in suspensibility of dieldrin powders

<sup>1</sup> See article on page 921.

**FIG. 1. SUSPENSIBILITY OF 75% DIELDRIN WATER-DISPERSIBLE POWDERS STORED AT 50°C AND 65°C**



**FIG. 2. SUSPENSIBILITY OF 50% DIELDRIN WATER-DISPERSIBLE POWDERS STORED AT 50°C AND 65°C**



at relatively high temperatures for short periods of time is a measure of their probable storage stability at ambient tropical temperatures over long periods of time. Miles et al.<sup>1</sup> have determined the effect of several factors on the suspensibility of DDT powders, giving special attention to the effect of temperature. Similar experiments have been initiated with dieldrin powders and the results obtained on a series of samples from the 1958 production of AC 1621 (75% dieldrin, Hi-Sil base) are presented here. Ten-gram samples of the powders were weighed into uncovered 250-ml beakers and heated at various temperatures for the periods of time indicated. The data obtained on the samples of AC 1621 are presented in Table 2. Unfortunately, sufficiently fresh samples of the 50% Attaclay (AC 1630 and AC 1631) and the 75% Micro-Cel (AC 6621) were not available for identical tests. It will be noted that there is considerable variation in the suspensibilities "as received". Also the change in suspensibility after the various heat treatments shows considerable variation among the samples. This seems surprising in view of the fact that the two drum samples of this formulation (AC 1621) stood up remarkably well in the cylinder storage tests. Apparently, the variation between batches is again in evidence. It should be noted that the inspection laboratory reports on duplicates of these samples showed all samples to have a suspensibility of 80% and above. These results, however, represent values obtained within a few days

**TABLE 2. SUSPENSIBILITY OF 75% DIELDRIN WATER-DISPERSIBLE POWDERS (FORMULATION AC 1621) AS RECEIVED AND AFTER HEATING IN OVEN AT TEMPERATURE AND PERIODS INDICATED**

Sample No.	as received	Suspensibility (%)											
		90°C			100°C			110°C			120°C		
		1 hr	4 hrs	20 hrs	1 hr	4 hrs	20 hrs	1 hr	4 hrs	20 hrs	1 hr	4 hrs	20 hrs
B8	78	78	82	77	76	76	72	69	68	61	52	55	46
B9	91	85	84	79	79	75	75	70	70	66	49	55	44
B10	87	76	71	64	62	55	51	37	47	45	27	30	31
B11	81	63	62	61	60	56	55	51	56	49	43	44	31
B12	76	60	53	53	52	49	47	44	47	43	27	26	31
B13	80	72	70	65	68	61	62	59	61	56	43	48	39
B14	88	88	89	93	87	83	83	80	82	75	56	62	53
B15	99	95	92	92	92	90	90	85	79	75	52	62	46
B16	65	67	65	62	62	56	50	48	48	37	34	32	34
B17	66	69	62	60	67	59	57	58	58	52	45	50	43

<sup>1</sup> See article on page 937.

of manufacture, whereas the "as received" values given in Table 2 are values obtained 1-5 months after manufacture. Thus, samples B16 and B17 apparently deteriorated significantly within a few months of manufacture. In general, however, all samples withstood the heat treatment well and it is believed that this formulation is the best so far developed for 75% dieldrin.

It will require considerably more study of heat treatments of the kind described here before a laboratory test for use in specifications can be adopted. The temperatures involved are far above any storage temperatures encountered in tropical areas and it would be undesirable to employ them in a test unless it can be shown that they do not bring about anything more than an acceleration of the changes occurring at ambient temperatures.

### RÉSUMÉ

En 1955, il apparut que la dieldrine serait appelée à remplacer le DDT dans certaines campagnes de désinsectisation entreprises par l'International Cooperation Administration. Les Technical Development Laboratories du US Public Health Service furent chargés d'élaborer des normes en vue d'assurer l'acquisition d'une poudre de dieldrine dispersable dans l'eau, dont l'aptitude à se mettre en suspension après une période de conservation plus ou moins longue, restât satisfaisante. Des deux produits fournis par la Shell Corporation, contenant 50% de dieldrine et de l'Attaclay comme diluant, l'un, le AC-1630, supporta mieux les essais à haute température et fut recommandée de préférence au AC-1631.

Peu de temps après réception de ce produit en Indonésie, on s'aperçut, à l'usage sur le terrain, que la dispersabilité de certains lots de poudres était défectueuse. Le manque d'homogénéité du diluant fut incriminé, et d'autres substances furent essayées à cet effet (Hi-Sil et Micro-Cel), avec des poudres contenant 75% de dieldrine. Après 1-6 semaines de conservation, le premier de ces diluants se montra supérieur au second. Des essais de mise en suspension, avec Hi-Sil (préparation AC-6621), à des températures de 90°-120°C pendant 1-20 heures, indiquèrent que la plupart des lots supportaient assez bien, et jusqu'à 20 heures, des températures allant jusqu'à 110°C. Cette préparation est considérée actuellement comme la meilleure de celles qui ont été mises au point pour des poudres à 75% de dieldrine.