

SPECIFICATIONS OF THE INTERNATIONAL COOPERATION ADMINISTRATION FOR DDT WATER-DISPERSIBLE POWDER FOR USE IN MALARIA CONTROL PROGRAMMES

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SYNOPSIS

Background information is presented on the development of specifications for 75% DDT water-dispersible powder for use in malaria control programmes supported by the International Cooperation Administration (ICA) of the United States Government. Early difficulties with DDT powders used in these programmes were investigated and it was found that the most critical requirements involved packaging, suspensibility and storage stability. ICA specifications were evolved to meet these requirements. The suspensibility test developed is described, and the importance of inspection of the material procured is discussed.

By the end of 1955 the United States Government, through the International Cooperation Administration (ICA), was supporting malaria control activities in 20 countries of the world, and it is estimated that 179 million people were receiving protection against malaria as a result of residual-spraying programmes. Since that date there has been a considerable expansion in the ICA-supported malaria control or eradication programmes. Many of these are in operation in countries in which similar programmes supported by the World Health Organization are also in effect; in these cases there is joint planning of the over-all campaign, although the responsibilities of the ICA and WHO staffs are distinct. In view of the community of interest of these two bodies, it has been thought advisable to outline the

development of the ICA specifications for one of the primary weapons in the fight against malaria — 75% DDT water-dispersible powder.

Development of Specifications

Water-dispersible powders containing 75% DDT have proved to be the most practical formulations for use in malaria control or eradication programmes. Other formulations, such as solutions and emulsions of DDT, are less desirable in view of the cost, the difficulty of transporting liquids in the field, and the porous nature of most wall surfaces involved; in houses of a certain standard, however, it is often essential to use these in preference to the water-dispersible formulations. Hand-spraying equipment without agitators is used almost exclusively for the application of insecticides. Therefore, water-dispersible powders must be formulated to remain in suspension for substantial periods of time. Some of the original DDT powder purchased for use in malaria programmes proved to have poor suspension properties and was packaged unsatisfactorily (Johnson 1953a, 1953b). The powder would settle out rapidly, tending to clog the sprayers and to produce very inadequate coverage of the walls. In addition, the material, which was packed in paper bags, frequently caked and the bags were often broken. As a result of these factors, the success of malaria control programmes was jeopardized. It was apparent that stringent specifications were needed to prevent the shipment overseas of poorly formulated and unsatisfactorily packaged wettable powders.

In developing these specifications several major points required attention. First among these was packaging. Since trouble had been experienced with the first shipments procured because of packaging in paper bags, the best solution appeared to be to require packaging in fibre drums with hermetically sealed plastic liners. Accordingly, such packaging was incorporated in the specifications. It is believed that this requirement has helped to prevent gross deterioration of products during shipment and storage under severely adverse conditions.

The packaging requirement also includes a cubage limit. Shipping rates are partly based on cubage occupied, and the question arose whether it would be desirable to compact the products in order to reduce the shipping costs. It was felt, however, that excessive compacting might increase the risk of caking or other deterioration. Until adequate data are available showing that compacting to reduce cubage has no deleterious effect, the minimum cubage has been set at 5.25 cubic feet (about 150 litres) for a 100-pound (45-kg) drum.¹

The most important aspect of the specifications is the suspensibility test. At the time of preparation of the original draft, the only information

¹ In more recent ICA specifications 100 pounds (45 kg) packed in drums of 26 US gallons (3.7 cubic feet or 105 litres) is specified.

concerning suspensibility requirements in powders used in international malaria control was that provided in the earlier reports of the Expert Committee on Insecticides of the World Health Organization.

In the past it had been assumed that the poor quality of much of the material received in the field was due largely to deterioration during shipment and storage under tropical conditions. The tropical storage test or pre-treatment had been incorporated in the WHO specifications by the Expert Committee on Insecticides in order to indicate whether a powder would change upon exposure to a relatively high temperature (55°C) with simultaneous application of pressure equivalent to that on the bottom of a typical 100-pound drum (25 g per cm²). It was indicated in the early WHO specifications that after such laboratory treatment, the powder was to be tested by means of a standard screen to determine whether any appreciable amount of agglomeration or change in particle size had occurred.

In developing the ICA specification, it was believed that it would be more desirable to measure the effect of the tropical storage treatment on suspensibility rather than on agglomeration as originally prescribed by WHO. For this reason a suspensibility test before and after tropical storage treatment was introduced. The following is a description of this test, abridged to include only the elements of immediate interest here:

Preparation of suspension

290 ml of standard hard water (as defined in *Specifications for Pesticides*, World Health Organization, 1956, p. 109) at approximately 30°C are added to 20 g of the powder previously weighed into a jar. The jar shall be a screw-cap glass jar of one United States liquid quart (0.95 litre) capacity, clear and transparent, substantially cylindrical, approximately 16 cm in inside height (vertical clearance), and without abrupt shoulders or features of other shape that would interfere with the efficient transfer of the suspension from the jar to another vessel. The material charged to the jar shall be rotated end over end at the rate of 60 revolutions per minute for two minutes; the axis of rotation shall be midway between the top and bottom of the jar and perpendicular to its vertical axis.

Suspensibility test

An aliquot of the suspension prepared as above is used for this test. Immediately after the rotation for two minutes is completed the jar is opened and stirred with a glass stirring-rod for one minute. A 50-ml aliquot is measured out immediately with a graduate and transferred to a 100-ml graduate, 18 cm in internal depth from the bottom to the 100-ml mark. A portion of standard hard water at about 30°C and not more than 40 ml in volume is used to rinse the transfer vessel. All rinsings are added to the 100-ml graduate. The graduate is placed in a constant temperature bath or cabinet held at 30° ± 1°C. After the contents have come to this temperature, enough standard hard water at 30° ± 1°C is added to bring the volume to the 100-ml mark. The graduate is then stoppered and smoothly inverted¹ 30 times by hand and then replaced immediately in the constant temperature bath. After standing 30 minutes, a 25-ml aliquot is removed in the manner described in the report of the WHO Expert Committee on Insecticides (1952), p. 67, paragraph 8.5, and examined for technical DDT content by any convenient method.

¹ Any sediment caked on the bottom of the cylinder must be resuspended with a minimum of agitation before starting the 30 inversions.

The above suspensibility test is to be performed on the material as received and also after the tropical storage test given in the first paragraph of section 2.2 on p. 109 of *Specifications for Pesticides* (World Health Organization, 1956). Before the tropical storage test, the 25-ml aliquot must contain at least 0.375 g of technical DDT, i.e., 1.5% weight to volume. After the tropical storage test the 25-ml aliquot must contain at least 0.300 g of technical DDT, i.e., 1.2% weight to volume.

In view of the fact that it was necessary to depend on commercial laboratories for compliance tests of products made under contract, it was felt desirable to submit a group of samples to several laboratories to obtain information not only on the reproducibility of the suspensibility test but also on the variability between laboratories. Such a collaborative study was arranged and six samples of 75% DDT water-dispersible powders were sent to five commercial laboratories specializing in analytical services. The samples were also tested by the Technical Development Laboratories (TDL), Communicable Disease Center, Public Health Service, Savannah, Ga., where study of the specification requirements had been initiated in 1953. The results of this testing are shown in the table.

The participating laboratories were requested to perform the suspensibility test before and after the tropical storage pre-treatment as prescribed in the ICA specification. Casual inspection of the data in the table indicates reasonably good agreement among the laboratories, with the exception of the data obtained on sample 106, which gave quite variable results, even in the same laboratory. This seems to be characteristic of occasional samples which TDL has encountered. Sample 106 was included in the test group since it was one known to give quite variable results. It has been the experience of TDL that both the earlier WHO and the ICA procedures for suspensibility are quite reproducible, but that extensive practice and close attention to details on the part of the analyst are required for good results. This observation seems to be substantiated by the data in the table. Laboratory No. 3 and TDL had had the most experience with the suspensibility test, and their results agree remarkably well. The procedure was completely new to Laboratory No. 1, and this laboratory showed the widest deviation from the average of all laboratories. Since this initial series of tests were made, Laboratory No. 1 improved its technique to a point where it agreed very well with TDL values on an additional group of samples tested.

The standard deviation and the percentage variation from the mean have been calculated for the data in the table. In respect of the results of Laboratory No. 3 and TDL, an average value for each laboratory was taken for the calculation in order not to give more weight to their values than to those of the other laboratories. The average standard deviation was found to be about ± 0.2 . The coefficient of variation is 11.5 for all test results, i.e., results before and after tropical storage pre-treatment. The coefficient of variation before tropical storage pre-treatment is only 5.5, but after pre-treatment it is 17.5. This indicates that the pre-treatment

**RESULTS OF COLLABORATIVE STUDY ON ICA SUSPENSIBILITY
TESTS OF 75% DDT WATER-DISPERSIBLE POWDERS**

Sample and test *	Percentage of DDT in suspension after 30 minutes						
	Lab. 1	Lab. 2	Lab. 3		Lab. 4	Lab. 5	TDL
			analysis 1	analysis 2			
Sample 51							
A	1.53	1.75	1.67 1.67	1.72 1.66	1.78	1.56	1.79
B	1.46	1.90	1.95 1.90	1.91 1.93	1.63	20.9	1.94 1.92 1.77
Sample 52							
A	1.61	2.04	1.79 1.85	1.78 1.82	1.80	1.93	1.84
B	1.48	2.14	2.02 2.02	1.99 1.99	1.62	2.12	1.95 1.89 1.93
Sample 80							
A	1.59	1.47	1.61 1.68	1.67 1.61	1.70	1.48	1.70
B	1.00	1.69	1.88 1.81	1.81 1.85	1.30	1.89	1.85 1.84 1.80
Sample 104							
A	1.61	1.71	1.66 1.64	1.66 1.62	1.67	1.65	1.80
B	1.19	1.72	1.93 1.88	1.90 1.88	1.60	1.96	1.93 1.93 1.90
Sample 105							
A	1.65	1.79	1.68 1.69	1.72 1.67	1.70	1.72	1.82
B	1.48	1.90	2.02 1.98	2.03 2.02	1.68	2.06	2.03 2.09 1.88
Sample 106							
A**	1.57	1.69	1.65 1.60	1.62 1.58	1.58	1.53	1.60
B	0.97	1.41	1.62 1.69	1.66 1.65	1.25	1.94	0.89 0.94 1.46

*A = Suspensibility before tropical storage pre-treatment.

B = Suspensibility after tropical storage pre-treatment.

** In addition to values tabulated, Laboratory No. 3 reported results of 1.2, 1.6, 1.7 and 1.8.

introduces a factor tending to produce more variation. Considering the fact that the group of samples included one which was known to give variable results and also that certain of the laboratories had limited experience with this type of test, the reproducibility of results between laboratories is good. There is reason to believe that all the laboratories represented in the table agree better in practice than the results presented here might indicate, mainly as a result of subsequent experience with the test. The authors have available additional data indicating that both the WHO and the ICA suspensibility tests are reproducible between laboratories within 0.2 unit. A single experienced analyst can reproduce his results much closer, within 0.1 unit or better. Accordingly, it has been recommended that agreement within 0.2 unit of ICA suspensibility test results by two laboratories for the same sample should be adopted as a standard. This allows a variation of 13.3% on a sample nominally testing 1.5; e.g., values of 1.4 and 1.6 would be considered within the acceptable limits of the method.

Inspection of Products Purchased by ICA

The ICA specification calls for a composite sample of each 50 tons to be taken for inspection purposes. Replicate samples are taken; one is sent to TDL for future reference, and one is submitted to an approved testing laboratory. Copies of the inspection data obtained by the inspecting laboratory are supplied to TDL. Analytical reports were received during the first part of 1955 representing composite samples of about 5800 tons, or more than 11 000 000 pounds, of 75% DDT water-dispersible powders.

In setting up this sampling schedule, it was recognized that it was weak, in that the sampling was too limited. However, the costs of inspection and analysis are so great that it was not considered feasible to increase the number of samples or to sample individual batches. In any case, the inspection data received are of interest. The average for all samples is 1.75 before and 1.57 after tropical storage pre-treatment. (The ICA specification requires a minimum of 1.5 before and 1.2 after tropical storage treatment.) Only one sample out of the entire group could be considered as not meeting the suspensibility requirements and this material was rejected.

On the basis of the results on these samples, it would seem logical to assume that all the material purchased under the specification was of high quality. However, reports received from the field during the study period indicated that some individual lots or batches of material were seriously deficient in suspensibility characteristics. Fortunately, it was possible to obtain samples of this deficient material along with a sample of satisfactory material from the same shipment. Suspensibility tests were conducted on these samples, and it became apparent that in spite of the ICA specifications batches of material were being received for use in the field which had zero suspensibility and were therefore unusable without further treatment. It is

of particular significance that all the defective samples were from the same shipment and known to be of the same formulation; altogether about 15% of the shipment was reported defective. Physical examination of defective samples showed that there was no difference in particle size from samples of satisfactory material, and agglomeration during storage or shipment could therefore be ruled out. In this connexion, data are presented in another paper,¹ which indicate that water-dispersible DDT powders made to ICA specifications can withstand long periods of storage at 50°C and higher without suffering such extreme loss in suspensibility as was here encountered. It might be assumed that the unsatisfactory batches in this shipment were of faulty manufacture, especially since the great majority of batches in that shipment were satisfactory. However, this may not be so as changes other than in particle size can occur with aging and can significantly affect suspensibility.

The importance of inspecting individual batches was indicated by the above experience. It was found that if satisfactory and unsatisfactory batches were mixed in equal quantity, the suspensibility of the mixture was that of the satisfactory material. It was even observed that mixtures of bad to good material in a ratio of 5:1 or higher still showed good suspensibility. These observations would seem to explain why the method of inspection called for in the early ICA specifications failed to detect unsatisfactory batches in composite samples taken from the shipment in question. Later revisions² of the specifications have incorporated provisions to remedy this situation.

RÉSUMÉ

Les poudres à 75 % de DDT, dispersables dans l'eau, sont considérées comme les préparations les plus pratiques dans les campagnes visant à l'éradication du paludisme. La difficulté de leur usage réside dans leur aptitude plus ou moins grande à se mettre en suspension, qualité qui peut être grandement altérée par un stockage de longue durée. Le succès de la lutte antipaludique exige que des normes soient établies, garantissant la dispersabilité des poudres, condition essentielle de leur efficacité.

La première de ces normes concerne l'emballage. On exige maintenant des récipients de fibre doublé de plastique hermétiquement clos, occupant un volume donné, suffisant pour que soit évité un empilement préjudiciable à la conservation de la poudre. La seconde a trait à la faculté de la poudre à se mettre en suspension. Les spécifications de l'International Cooperation Administration (ICA), qui diffèrent en certains points de celles du Comité OMS d'experts des Insecticides, prévoient l'étude de l'effet de la conservation dans les conditions tropicales en fonction de la dispersabilité, et non plus de l'agglomération des particules. Les auteurs décrivent le test prescrit à cet effet.

¹ See page 921 of this issue.

² International Cooperation Administration (1955, 1956, 1958, 1959) *DDT, 75%, water-dispersible powder*, Washington, D.C. (ICA Specification No. 101055, 10 October 1955; revised 10 August 1956 and issued as ICA Specification No. 81056; revised 4 September 1958 and issued as ICA Specification No. 9458; revised 9 January 1959 and issued as ICA Specification No. 1859)

Une évaluation comparative du test, effectuée dans plusieurs laboratoires sur les mêmes échantillons de poudres, a montré que les méthodes recommandées tant par le Comité OMS d'experts que par l'ICA sont reproductibles, à condition qu'elles soient exécutées par des analystes très expérimentés.

Les auteurs exposent les conditions d'inspection des produits achetés par l'ICA pour les campagnes antipaludiques. Les normes exigent qu'un échantillon mixte soit prélevé pour chaque lot de 50 tonnes d'insecticide, et fixent le coefficient de dispersabilité à 1,5 avant et à 1,2 après le traitement dans les conditions tropicales. Les chiffres obtenus sont en général supérieurs aux normes. Il arrive cependant que des lots soient défectueux, alors même que la grosseur des particules est conforme aux normes. Certains d'entre eux pourtant ont passé inaperçus car le mélange avec des lots satisfaisants masque leurs imperfections. On a constaté en effet que des chiffres de dispersabilité de tels mélanges à parties égales de non conformes et de conformes — ou même en proportions de 5:1 — sont à peu près ceux des lots satisfaisants. Des révisions ultérieures des spécifications ont remédié à ces lacunes dans le dépistage des lots de qualité insuffisante.

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