

This report contains the collective views of an international group of experts and does not necessarily represent the decisions or the stated policy of the World Health Organization

Chemistry and specifications of pesticides

Eighth report of the WHO
Expert Committee on Vector
Biology and Control

World Health Organization
Technical Report Series
699



World Health Organization 1984

ISBN 92 4 120699 3

© World Health Organization 1984

Publications of the World Health Organization enjoy copyright protection in accordance with the provisions of Protocol 2 of the Universal Copyright Convention. For rights of reproduction or translation of WHO publications, in part or *in toto*, application should be made to the Office of Publications, World Health Organization, Geneva, Switzerland. The World Health Organization welcomes such applications.

The designations employed and the presentation of the material in this publication do not imply the expression of any opinion whatsoever on the part of the Secretariat of the World Health Organization concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries.

The mention of specific companies or of certain manufacturers' products does not imply that they are endorsed or recommended by the World Health Organization in preference to others of a similar nature that are not mentioned. Errors and omissions excepted, the names of proprietary products are distinguished by initial capital letters.

PRINTED IN SWITZERLAND

83/5985 – Schöler SA – 6 200

CONTENTS

| | Page |
|--|------|
| 1. Introduction..... | 5 |
| 2. New trends in the use of pesticides and formulations in public health..... | 6 |
| 3. Analytical methods and quality control in developing countries..... | 9 |
| 3.1 Development of new analytical methods..... | 9 |
| 3.2 Laboratory equipment..... | 10 |
| 3.3 Applicability of new analytical methods in developing countries..... | 10 |
| 3.4 Need for regional laboratories for quality control of pesticides..... | 11 |
| 4. Standardization of GLC and HPLC equipment..... | 11 |
| 4.1 Gas-liquid chromatography..... | 11 |
| 4.2 High-performance liquid chromatography..... | 12 |
| 5. Specifications and test methodology..... | 13 |
| 5.1 Tolerance limits..... | 13 |
| 5.2 Changes based on FAO specifications..... | 14 |
| 5.3 Interim specifications..... | 15 |
| 5.4 Suspensibility tests..... | 17 |
| 5.5 Heat-stability tests for water-dispersible powders..... | 17 |
| 5.6 Molluscicides..... | 18 |
| 5.7 Repellents..... | 18 |
| 5.8 Biological control agents..... | 19 |
| 5.9 Promising new formulations..... | 20 |
| 6. Supply of pesticides..... | 20 |
| 6.1 Procurement..... | 20 |
| 6.2 Packaging..... | 20 |
| 6.3 Labelling..... | 21 |
| 6.4 Disposal of pesticides and containers..... | 21 |
| 7. Collaboration with other organizations..... | 22 |
| 7.1 Food and Agriculture Organization of the United Nations..... | 22 |
| 7.2 Collaborative International Pesticide Analytical Council..... | 22 |
| 8. Recommendations..... | 23 |
| Acknowledgements..... | 23 |
| Annex 1 Recommended general changes in existing specifications and methods..... | 24 |
| Annex 2 Recommended changes in analytical methods used in existing specifications..... | 28 |
| Annex 3 Recommended specifications for new pesticides and formulations.... | 32 |
| Annex 4 Recommended cautionary notices for dustable powders..... | 43 |

WHO EXPERT COMMITTEE ON VECTOR BIOLOGY AND CONTROL

Geneva, 8-14 November 1983

*Members**

- Dr Atta-ur-Rahman, Professor and Co-Director, HEJ Research Institute of Chemistry, University of Karachi, Karachi, Pakistan (*Vice-Chairman*)
Mr J. Henriët, Chief, Chemistry Section, Department of Agriculture, National Phytopharmaceutical Research Establishment, Gembloux, Belgium (*Chairman*)
Dr J.W. Miles, Chief, Control Technology Branch, Division of Parasitic Diseases, Center for Infectious Diseases, Centers for Disease Control, Atlanta, GA, USA (*Rapporteur*)
Mr S.H. Tan, Senior Agricultural Officer, Department of Agriculture, Crop Protection Branch, Kuala Lumpur, Malaysia
Mrs Y. Yingchol, Chief, Pesticide Analyses Branch, Division of Agricultural Toxic Substances, Department of Agriculture, Bangkok, Thailand

Representatives of other organizations

- Food and Agriculture Organization of the United Nations*
Dr A.V. Adam, Plant Protection Service, FAO, Rome, Italy

Secretariat

- Dr W.R. Bontoyan, Head, Analytical Chemistry Section, Environmental Protection Agency, Agricultural Research Center, Beltsville, MD, USA (*Temporary Adviser*)
Dr J.F. Copplestone, Chief, Pesticides Development and Safe Use, Division of Vector Biology and Control, WHO, Geneva, Switzerland
Dr N.G. Gratz, Director, Division of Vector Biology and Control, WHO, Geneva, Switzerland
Dr P.J. Madati, Chief Government Chemist, Government Chemical Laboratory, Ministry of Health, Dar es Salaam, United Republic of Tanzania, (*Temporary Adviser*)
Dr G. Quélenec, Scientist, Pesticides Development and Safe Use, Division of Vector Biology and Control, WHO, Geneva, Switzerland (*Secretary*)
Dr A.M.S. Silva Fernandes, Directorate-general of Agricultural Production Protection, Directorate of Toxicology Services, Quinta da Marques, Oeiras, Portugal (*Temporary Adviser*)

* Unable to attend: Dr Y.P. Volkov, Chief, Department of Chemical Sciences, All-Union Scientific Research Institute for Disinfection and Sterilization, Moscow, USSR.

CHEMISTRY AND SPECIFICATIONS OF PESTICIDES

Eighth Report of the WHO Expert Committee on Vector Biology and Control

1. INTRODUCTION

The WHO Expert Committee on Vector Biology and Control met in Geneva from 8 to 14 November 1983 to study recent developments in the chemistry and specifications of pesticides used in public health, to establish new or revised specifications for the WHO manual *Specifications for pesticides used in public health*,¹ to provide guidance for improving the specifications of new types of formulations and active ingredients, and to make suggestions for the preparation of new interim specifications.

Dr N.G. Gratz, Director, Division of Vector Biology and Control, opened the meeting on behalf of the Director-General and remarked that specifications for pesticides are of major importance for the Member States of WHO. They facilitate and encourage the purchase of pesticides of good quality that are both effective against susceptible vectors of diseases or intermediate hosts and acceptable from the point of view of safety. In addition, the analytical methods contained in the specifications are useful in the quality control of pesticides after storage under adverse climatic conditions. Specifications are also widely used by Member States and by WHO in connection with the purchase of very large quantities of pesticides for use in public health programmes. As an example, WHO has received 480 requests, most of them from developing countries, for the new series of interim specifications for pesticides. This reflects the importance of the decisions taken and the recommendations made by the Expert Committee concerning the chemistry and specifications of pesticides.

The efficacy of any product used in public health depends to a large extent on the physical and chemical properties of the formula-

¹*Specifications for pesticides used in public health*, 5th edition, Geneva, World Health Organization, 1979.

tion, which should be related to the recommended method of application. These properties must therefore be defined very accurately so that the same pesticide manufactured by several different companies has the same efficacy in the field. This aspect of specifications has become very much more important since a large number of pesticides are now no longer protected by patents and have become ordinary commodities. Strict attention must also be paid to impurities that may be present in the active ingredient or be formed by reaction with other constituents in the formulation and potentiate the toxicity of the pesticide. For some pesticides, this may necessitate the use of analytical methods that allow the detection of very small amounts of impurities in both the technical material and in formulations.

Such refined techniques may require the use of expensive equipment that may not be easily obtained and operated in developing countries as a consequence of limited funds and difficulties in obtaining spare parts, purified gases, etc. It was therefore strongly suggested that consideration should be given to the possibility of sharing equipment and resources, within a particular country, between ministries needing to use them. The development of collaborating centres capable of operating at regional level was also recommended.

New trends in vector control methodology are emerging and these will lead to the use of more selective pesticides for controlling a greater number of vectors or nuisance insects. Vector control operations, both in villages and in towns, will now increasingly involve community participation. As a result, greater attention will have to be paid to the storage conditions of pesticides and their transportation, the labelling of containers, and the quality of packages and their disposal. The Committee was requested to pay special attention to these problems in so far as they have a bearing on the establishment of specifications.

2. NEW TRENDS IN THE USE OF PESTICIDES AND FORMULATIONS IN PUBLIC HEALTH

Pesticides continue to play an important global role in the control of vectorborne diseases. In the recent past a number of disease control programmes have tended to be based solely on pesticides and in others pesticides have been used on a large scale, a state of affairs that will continue for many years to come. However, a gradual shift

in approach is evident, primarily as a consequence of human, socioeconomic, biological, political, and environmental factors and of a gradual move in disease control programmes away from a vertical administration and towards their inclusion in primary health care programmes. Some of these changes have occurred as a result of the shift in basic approach from eradication to the control of diseases such as malaria and the inherent flexibility of the latter approach. The emphasis on primary health care arose from the recognition that previous health care strategies often resulted both in major inequalities and in inappropriate and costly services. Furthermore, these strategies all too often ignored the social and economic origins of ill health. Primary health care emphasizes individual and community self-reliance, whenever possible.

Although chemical control of disease vectors has produced spectacular results, the developments described above have made it necessary to reduce reliance on chemicals and to use the alternative methods of biological control and environmental management, as called for by the concept of integrated vector control. This has been the subject of a comprehensive review by the Expert Committee on Vector Biology and Control in its seventh report.¹

The methods used in integrated vector control vary greatly depending on local conditions, the ecology of the vector species, vectorial status, and resistance to pesticides. Where outdoor feeding and resting behaviour render residual spraying ineffective, a combination of methods may be more appropriate. Thus antilarval measures, which may include the use of larvicides, biological control agents and/or environmental management practices, are employed in vectorborne disease control in situations where adult vector control is impracticable for economic or operational reasons. As an example, in mosquito control in urban and semi-arid or arid areas, where vector breeding habitats tend to be restricted and well defined, such anti-larval measures may provide the most practical approach. For small-container breeders, e.g., *Aedes aegypti*, in south-east Asia and the Americas, however, a combination of environmental sanitation methods, larviciding and space spraying is often needed for effective control.

The wide application of chemical control agents has led to the development of new types of formulations for use in public health

¹ WHO Technical Report Series, No. 688, 1983.

—slow-release formulations, flowable concentrates, monomolecular surface-active compounds, etc. These may need to be considered for inclusion in future specifications.

During the past decade, the three major classes of pesticides—the organochlorines, organophosphorus compounds, and carbamates—have continued to be the main compounds used for the control of arthropods. Owing to the prohibitively high costs of development, few new products designed specifically for public health use have been produced by the chemical industry. Developments include the use of synthetic pyrethroid pesticides on an increasing scale. There has so far, however, been only limited use of insect growth regulators, such as the juvenile hormone analogue, methoprene, and the moulting inhibitor, diflubenzuron. In addition, there has been a gradual shift to the more economical use of pesticides and this has led to modifications in methods of application and in formulation in order to achieve target dosage efficiency.

As part of the integrated approach, biological control agents, particularly microbial toxins, are coming into use and greater attention is being paid to the use of larvivorous fish. With some types of agents, the efficacy and persistence can be enhanced by suitable formulations. The place of biological control in vector control was reviewed by the Expert Committee on Vector Biology and Control in its sixth report.¹

Toxicological studies of the use of chemical pesticides may affect future specifications and were therefore of concern to the Committee. In its third report² the Expert Committee on Vector Biology and Control came to some general conclusions as to the limits of acceptable acute toxicity for conventional indoor application of organophosphorus compounds and carbamates. Since then, field studies have clearly shown that bendiocarb, a carbamate of relatively high mammalian toxicity, may be applied safely by modifying the conventional methods of presentation and application. The supply of an 80% water-dispersible powder in preweighed sachets each containing the required amount of pesticide for one pump-charge made the mixing of the material acceptable from the safety point of view, since the task of breaking down bulk supplies into smaller packages for the field (bagging) was eliminated altogether.

¹ WHO Technical Report Series, No. 679, 1982.

² WHO Technical Report Series, No. 634, 1979.

In 1982 the scheme for the evaluation and testing of new pesticides for public health, which had been in operation since 1960, was reconsidered by a meeting of directors of collaborating centres.¹ As the format of the revised scheme differed from that of the previous one, the name was changed to WHO Pesticide Evaluation Scheme (WHOPES). It consists of a number of phases, of which phases 1, 2 and 3 deal with laboratory evaluation, small- and large-scale field trials, respectively, while phase 4 is new and is concerned with the development of specifications for the active ingredient and for appropriate formulations of pesticides for the types of application found to be effective. It requires information on physical and chemical properties, and collaborative studies of analytical methods. It is conducted by WHO collaborating centres and by WHO in consultation with industry. The analytical procedures are standardized in collaboration with the relevant international organizations.

One of the main differences between WHOPES and the previous scheme is that compounds may be tested against several vectors by various methods of application requiring different types of formulations. Decisions on passing from one phase to the next depend on compound, vector, and use. The observation of serious toxicity can lead to the testing of a compound being stopped at any time.

The Committee noted the modern trends in vector control and the effects that these might have on specifications for pesticides in the future, one such effect being the more frequent issue of interim specifications.

3. ANALYTICAL METHODS AND QUALITY CONTROL IN DEVELOPING COUNTRIES

3.1 Development of new analytical methods

The Committee considered that it is important to keep analytical methods for pesticides under constant review, so as to take advantage of improvements in technology and thereby achieve greater accuracy, sensitivity, specificity, reproducibility, ease of manipulation and, where possible, economy in the analytical methods used.

¹ Unpublished WHO document WHO/VBC/82.846 Rev. 1.

3.2 Laboratory equipment

The equipment required for the new instrumental methods of analysis and quality control of pesticides includes thin-layer chromatographic apparatus, chromatographs for gas-liquid chromatography (GLC), with flame-ionization detectors, and for high-performance liquid chromatography (HPLC), with ultraviolet detectors, as well as a range of packed GLC and HPLC columns, compressed gases and HPLC-grade solvents. Specific recommendations for GLC and HPLC equipment are given in section 4, below.

3.3 Applicability of new analytical methods in developing countries

The Committee realized the difficulties faced by developing countries in the acquisition and maintenance of modern equipment, such as gas chromatographs and high-performance liquid chromatographs. The alternative of using wet chemical or colorimetric methods was considered, but the Committee was of the opinion that the reduced specificity of these procedures, as well as their unreliability from the point of view of detecting degradation products and impurities, made it imperative that the more modern procedures should be adopted. This was all the more necessary because FAO, the Collaborative International Pesticide Analytical Council (CIPAC) and the Association of Official Analytical Chemists (AOAC) have already adopted the newer analytical procedures. It was stressed that the cost of high-performance liquid chromatographs and gas-liquid chromatographs was not exorbitant and, in view of the efficiency of these systems and the accuracy and reproducibility of the results obtained, as well as the risk that sub-standard products may come on to the market if less accurate chemical procedures are used, it was highly desirable that the modern chromatographic procedures should be generally adopted.

In view of the acute shortage of suitably trained technical manpower, adequate modern analytical equipment, and the necessary spare parts in developing countries, the Committee recommended that steps should be taken to:

- (i) Promote the standardization of equipment in order to facilitate servicing and reduce its cost.
- (ii) Encourage the suppliers, from whom the bulk of the pesticides used are obtained, to contribute towards the acquisition of

- analytical equipment and spare parts and provide for the servicing of this equipment.
- (iii) Make an inventory of, and pool, analytical resources so that optimum use may be made of the facilities available in each country.
 - (iv) Train technical personnel to operate and maintain analytical instruments, either by means of regional workshops and courses or by sending them to suitable centres abroad, when necessary.

3.4 Need for regional laboratories for quality control of pesticides

The Committee agreed that, since the necessary laboratory facilities for the analysis and quality control of pesticides do not exist in many developing countries, it was important to designate certain centres where the requisite expertise and laboratory facilities do exist as WHO regional centres (or where necessary, subregional centres) for pesticide analysis. These centres should be further strengthened in order to enable them to carry out pesticide analyses for countries at the regional level. They could also serve as focal points for research and training activities aimed at the development of new and safe pesticides.

4. STANDARDIZATION OF GLC AND HPLC EQUIPMENT

The Committee recommended that WHO specifications should be based on analytical methods accepted by international scientific bodies such as the CIPAC and AOAC; it therefore, also recommended the minimum standard basic equipment required by these methods. The recommended AOAC/CIPAC methods for the most part require gas-liquid chromatographs and high-performance liquid chromatographs, which give more specific, reliable, and accurate analytical results than those obtained by wet analysis.

The cost of GLC and HPLC equipment, when compared to that of the general laboratory glassware used for wet analysis, is high, but it can be kept to a minimum if laboratory managers follow the Committee's recommendations, which are given below.

4.1 Gas-liquid chromatography

The basic GLC instrument requirements are as follows:

- A high-sensitivity flame-ionization (FI) detector having a wide dynamic range.
- An electrometer with a sensitivity of 10 μ A.
- A recorder with a full-scale deflection of 1 mv.
- All-glass on-column injection with an independent variable temperature control.
- A two-column capacity oven with variable temperature control.
- A borosilicate glass column, 1–2 m long, 2 mm in internal diameter, and 6 mm in external diameter.

The Committee also recognized that, in addition to recommending these basic GLC instrument requirements, there is a need for stationary phases accepted by AOAC and CIPAC for pesticide formulation analysis (see Table 1). The solid supports (80–120 mesh) should be as inert as possible.

The Committee also recommended the use of standardized procedures for the preparation of GLC columns (see Annex 1).

Table 1. Recommended stationary phases for GLC column packing

| Stationary phase | Temperature range (°C) | Designation |
|--|------------------------|-------------------------------|
| Methyl silicone | 20–350 | OV-101, SP-2100, SE-30, E-301 |
| Methyl silicone (50%) + phenyl silicone (50%) | 20–350 | OV-17, SP-2250 |
| Trifluoropropyl silicone (50%) + methyl silicone (50%) | 20–280 | OV-210, SP-2401 |
| Methyl silicone (50%) + phenyl silicone (25%) + cyanopropyl silicone (25%) | 20–280 | OV-225 |
| Ethylene glycol | 50–225 | Carbowax 20M |
| Phenyl silicone (50%) + cyanopropyl silicone (50%) | 50–275 | Silar 5 CP, SP-2300 |
| Cyanopropyl silicone | 50–225 | Silar 10C, SP-2340 |

4.2 High-performance liquid chromatography

The basic HPLC instrument requirements are as follows:

- A pumping system having a constant-volume displacement pump capable of generating a pressure of 17×10^6 Pa, giving flow rates in the range 0.5–10 ml/min, and not making any significant contribution to the detector baseline.

- A detector with a fixed wavelength of 254 nm, a signal-to-noise ratio of 5 and a linear dynamic range of 3–4 orders of magnitude.
- Stainless steel columns 10–30 cm long and 4 mm in internal diameter.
- With regard to the column material, the normal phase should be silica (5–14 μm) and the reverse phase C-18 bonded silica (5–14 μm).

5. SPECIFICATIONS AND TEST METHODOLOGY

The Committee reviewed the specifications and methods contained in the fifth edition of the WHO manual *Specifications for pesticides used in public health*¹ and recommended a number of changes, additions, and deletions (see Annexes 1 and 2). It also reviewed a number of interim specifications that have been issued subsequent to the publication of the fifth edition. In the light of WHO policy to include in the manual only those specifications that have been shown to be of practical use in public health and for which analytical methods have been collaboratively tested, recommendations were made for the inclusion of certain interim specifications in a new edition of the manual (see Annex 3).

5.1 Tolerance limits

Concern has been expressed regarding the current WHO tolerance for pesticides in formulations with a label declaration of more than 500 g/kg of the active ingredient.

Tolerances should reflect the three causes of apparent variation in the content of active ingredient in pesticide products, namely the manufacturing process, sampling, and the chemical method of analysis. The expression of the tolerance in the specifications in the form $\pm 5\%$ (1000 minus nominal content) takes into account only the variation that is a direct result of the manufacturing process. The Committee recommended that the tolerance should also be based on

¹ *Specifications for pesticides used in public health*, 5th edition, Geneva, World Health Organization, 1979.

the reproducibility (precision) of the method of analysis and sampling technique, and should be expressed as:

—up to 500 g/kg: $\pm x$ % of the nominal content

—above 500 g/kg: $\pm y$ g/kg.

The Committee drew attention to the declared minimum content of the active ingredient in technical materials, which takes into consideration the precision of the analytical method.

The Committee also recommended that a higher plus tolerance should be established to permit higher than nominal active ingredient content for those products that degrade during storage.

5.2 Changes based on FAO specifications

Certain pesticides are of mutual interest to WHO and FAO. For some of them both organizations have published specifications, established mainly for purchase and quality control purposes at the international level. FAO specifications may also be used for registration purposes. The Committee considered that it would be most useful if harmonization could be achieved whenever possible between WHO and FAO specifications.

The Committee reviewed the clauses where differences exist between the specifications of the two organizations and recommended the general changes given in Annex 1.

The Committee felt that the time prescribed for the cold tests is too short and recommended that a study should be carried out in order to determine the optimum duration for such tests.

In the case of water-dispersible powders and dustable powders, the Committee recommended that the words “accelerated storage treatment” should be replaced by “heat stability”. Furthermore, recognizing that different temperatures and times are used for various products, the Committee recommended that research should be conducted to establish relevant temperatures and times for those compounds that have not been investigated. It is suggested that 54 ± 1 °C for 3 days should be used as a starting condition. The Committee recommended that the test should be performed in sealed bottles for all formulations except DDT water-dispersible powders for overseas shipment, and that the pressure conditions should be deleted.

The Committee recommended that studies should be conducted on new formulations of water-dispersible powders in order to determine the effect of heat on the stability of the active ingredient. Where decomposition occurs, a value for the limit of decomposition after a heat stability test should be inserted in the appropriate specifications.

It was also recommended that, for emulsion concentrates, the cold stability test following heat stability treatment should be deleted.

The Committee further recommended that the limits for the nominal content of formulated products as well as the tolerances should be harmonized with those adopted by FAO.

With regard to analytical methods for active ingredient contents, the Committee recommended that AOAC and/or CIPAC methods should be used.

The Committee agreed that requirements for limits of impurities should be included only in those cases where the impurities concerned would affect odour, colour, toxicity or stability. The Committee suggested that the limits of impurities should be harmonized with those specified by FAO whenever possible.

The Committee recommended that the standard waters used by both organizations should be the same; in order to determine the nature of the standard waters to be used, however, additional research is necessary.

The Committee suggested that FAO should consider the use of the following WHO procedures:

- wet and dry sieve tests;
- determination of dustability of dustable powders;
- use of sealed containers for determination of heat stability of water-dispersible powders;
- determination of cold stability of liquid formulations;
- emulsion stability test.

5.3 Interim specifications

The Committee was informed that, subsequent to the previous meeting of the Expert Committee on Vector Biology and Control on the chemistry and specifications of pesticides in 1977,¹ studies had

¹ WHO Technical Report Series, No. 620, 1978.

been carried out on a number of interim specifications for pesticides¹ with a view to their inclusion in the next edition of the WHO manual *Specifications for pesticides used in public health*.

After reviewing the existing interim specifications and reports on analytical methods, as well as a report on chemical methods used to control arthropod vectors and pests of public health importance,² the Committee recommended the following compounds and formulations for inclusion in the WHO manual:

- (i) dichlorvos, emulsifiable concentrate;
- (ii) fenthion, water-dispersible powder;
- (iii) trichlorfon, water-soluble powder;
- (iv) bendiocarb, technical, and water-dispersible powder;
- (v) deltamethrin, technical, water-dispersible powder, emulsifiable concentrate, and dustable powder;
- (vi) temephos, sand granules.

The specifications and summaries of analytical methods for bendiocarb and deltamethrin and their formulations are presented in Annex 3.

The Committee also noted a continuing need for the compounds and formulations given below; insufficient data were available, however, to permit incorporation in the WHO manual, and it was therefore recommended that they should be retained in the form of interim specifications³ for the reasons stated:

- (i) permethrin, technical, and emulsifiable concentrate: the cis/trans ratios varied over a wide range in commercial products, and the Committee therefore recommended that studies should be conducted to determine the relationship between cis/trans ratios and biological efficacy;
- (ii) deltamethrin, ultra-low volume (ULV) liquid: data on the physical characteristics of the product are lacking;
- (iii) pirimiphos-methyl, technical, water-dispersible powder and emulsifiable concentrate: a method of analysis for the active ingredient has not been adopted by AOAC or CIPAC.

¹ Unpublished WHO documents VBC/IS/82.02, 82.03, 82.05, 83.06, 83.07 and 83.08.

² Unpublished WHO document VBC/82.841 Rev. 1.

³ Unpublished WHO documents VBC/IS/82.04, 83.08 and 83.09.

It was further recommended that specifications for the following compounds and formulations should be revised and issued in the form of interim specifications:

- (i) chlorpyrifos-methyl, technical, and emulsifiable concentrate;
- (ii) chlorphoxim, technical, water-dispersible powder and emulsifiable concentrate for *Simulium* control.

5.4 Suspensibility tests

The two methods used by WHO for determining the suspensibility were reviewed. The Committee agreed that the methods used for DDT water-dispersible powder for overseas shipment and for malathion water-dispersible powder should be retained, except that, for malathion, the six water washings called for should be replaced by four water washings only.

Since it has been shown in laboratory experiments¹ that no relationship exists between the malathion retained on the carrier and the biological effectiveness, the Committee suggested that studies should be conducted in the field to confirm this finding.

The Committee recommended that the suspensibility test in standard soft water without pretreatment should be deleted for those compounds where the required suspensibility values before and after heat stability treatment are equal.

5.5 Heat-stability tests for water-dispersible powders

The Committee reviewed data presented on the effect of leakage of moisture from sealed bottles during the accelerated storage (heat stability) test and recommended a change in the wording of the test description so as to prevent such leakage. The recommended revised wording for heat stability treatment for malathion powders is given in Annex 1, and it was recommended that similar wording should be used in the description of such treatment for all water-dispersible powders, with the exception of DDT water-dispersible powder for overseas shipment.

¹ Unpublished WHO document WHO/VBC/82.853.

5.6 Molluscicides

The role of molluscicides in the control of schistosomiasis was reviewed and it was noted that there is a need for new molluscicides, in spite of the fact that safe and effective drugs are now available for use in treating the disease. Niclosamide continues to be the most widely used pesticide for snail control. Pentachlorophenol and copper sulfate have been shown to be less effective in large-scale field trials and are unlikely to be used in future programmes, and it was therefore recommended that specifications for them should be deleted from the manual. It was noted that the compound sodium 2,5-dichloro-4-bromophenol has been successfully tested against a local snail species in Japan; field trials of this product against other snail hosts of schistosomiasis are needed. Other molluscicides under development for schistosomiasis control at the present time include the organotins, nicotinilides, certain amides, including fluoracetamide and metaldehyde, as well as several products of plant origin.¹

It was recommended that specifications for niclosamide, technical, water-dispersible powder, and emulsifiable concentrate, and for trifenmorph should be retained in the next edition of the WHO manual. It was further recommended that the specification for niclosamide emulsion concentrate should be revised, taking into consideration the fact that the product is manufactured from the free base and not from the ethanolamine salt. The preparation of interim specifications for new molluscicides was recommended as data become available.

5.7 Repellents

Repellents are chemicals used for the protection of human beings and other animals from the attacks of ticks and blood-sucking insects; they are used primarily in situations where other chemical control methods are not feasible and where individual protection is essential. They are formulated as liquids, lotions, creams, foams, solid waxes, and aerosols. At present, the most effective compounds are the following: benzyl benzoate, butyl ethyl propanediol, deet (*N,N*-diethyl-*m*-toluamide), dibutyl phthalate, dimethyl carbamate, dimethyl phthalate, ethyl hexanediol, indalone, and *o*-chloro

¹ Unpublished WHO document WHO/VBC/83.879.

diethylbenzamide. In recent years, a number of alicyclic carboximides of heterocyclic amines have been reported as being very effective. Permethrin, when applied to clothing, can also be used as an effective repellent.

The effectiveness of a repellent treatment depends on the compound, the dosage of application, the species of insect and the climatic conditions. The Committee recommended the retention of technical deet in the WHO manual. The development of interim specifications is recommended for promising new repellents only when efficacy and toxicological data become available.

5.8 Biological control agents

The challenge of finding a fundamentally new class of vector control agent has been taken up by WHO through the UNDP/World Bank/WHO Special Programme for Research and Training in Tropical Diseases in collaboration with the Division of Vector Biology and Control. Microbial insecticides are under development and have recently reached the stage of inclusion in operational programmes. The greatest progress has been made with the biological control agent *Bacillus thuringiensis* serotype H-14, which has proved highly effective in the field against the larvae of a number of species of blackflies and mosquitos and is commercially available. It has also been shown in laboratory and field trials that *B. thuringiensis* H-14 applied at the rates required to control blackflies and mosquitos causes minimal harm to the vast majority of aquatic non-target organisms.

From the viewpoint of its formulations, *B. thuringiensis* H-14, together with its endotoxin, may be likened to a chemical larvicide, and acts only on the gut wall after ingestion. Water-dispersible powders and suspension concentrates, special types of granules and slow-release formulations have now been developed by industry, and it would therefore be desirable to establish specifications for such products. The Committee discussed this subject at length but concluded that, because of the difficulty of assaying these products, it would not be possible at this stage to establish specifications. The Committee recommended, however, that further work should be carried out so that specifications for such products may be established as soon as possible.

5.9 Promising new formulations

A number of new formulations of pesticides were reviewed, including ULV formulations, granules, flowable concentrates and slow-release formulations. In the case of a ULV formulation of deltamethrin, the Committee recommended that the specification for this product should remain as an interim specification as some of the relevant physical properties are not yet defined. No specific need for granules other than for temephos was reported, and no new specifications for granules were recommended. The Committee took note of the development of flowable concentrates and slow-release formulations, and recommended that progress with these products should be monitored and that interim specifications for them should be developed as the need arises.

6. SUPPLY OF PESTICIDES

6.1 Procurement

In addition to purchasing pesticides for WHO projects from its funds, the Organization has operated a supply service to Member States since 1950. This service is available to agencies under the jurisdiction of the health administration or comparable authority of Member States.

The Committee felt that this service provided by the Organization is highly beneficial, especially to developing countries, and recommended that it should be made known to all Member States.

6.2 Packaging

Since most of the developing countries purchase their pesticides from foreign sources, specifications for pesticide containers are important. The Committee was informed that European suppliers have difficulties in complying with the WHO interim specifications for corrugated boxes for shipping DDT and malathion water-dispersible powders.¹ The Committee was informed that WHO had entered into a contract with a laboratory to study the ability of boxes from different sources to pass the tests described in the above specifications.

¹ Unpublished WHO document WHO/VBC/78.698.

6.3 Labelling

The Committee recommended that, for all compounds, the specifications should require the clear marking of each package with the month and year of manufacture or formulation.

The Committee noted that the Second Government Consultation on International Harmonization of Pesticide Registration Requirements, organized by FAO in Rome in October 1982, recommended that all pesticide labels should include the expiry date, if one exists, or the date of manufacture or formulation.¹ The Committee strongly emphasized the desirability of all manufacturers following this FAO recommendation.

In line with a previous Expert Committee's recommendations,² the cautionary notices for different dustable powders for use against vectors and pests of public health importance are recommended for inclusion in the next edition of the WHO manual *Specifications for pesticides used in public health* under the section "Packing and marking of packages". It was pointed out that these represent *minimum* requirements, and further instructions for safe handling may be added by the manufacturer or required by the purchasing or registration authorities.

The Committee noted that different types of dustable powders may be used for different applications and recommended that a clear distinction should be made between them in the cautionary notices, e.g., by means of wording such as:

- for application to skin, clothing or bedding;
- not for application to skin, clothing or bedding.

When appropriate, this distinction should also appear in the title of a particular specification.

The recommended wording of the cautionary notices is shown in Annex 4.

6.4 Disposal of pesticides and containers

The Committee was briefed on the report on methods of disposal of surplus pesticides and pesticide containers in developing

¹ *Report of the Second Government Consultation on International Harmonization of Pesticide Registration Requirements*, Rome, Food and Agriculture Organization of the United Nations, 1982.

² WHO Technical Report Series, No. 356, 1967.

countries.¹ The Committee was also informed that WHO proposes to sponsor research into the possibility of decontaminating containers made of semipermanent materials. The Committee recommended that, in the meantime, pesticide containers should not be used for the storage of food, animal feed, or water. However, they strongly supported the suggestion that studies should be carried out on the possibility of decontaminating empty metal drums.

7. COLLABORATION WITH OTHER ORGANIZATIONS

7.1 Food and Agriculture Organization of the United Nations

The Committee noted with satisfaction the continuing collaboration between FAO and WHO and the great importance given by FAO to maintaining close working relations with WHO in the field of pesticides in general and specifications for pesticides in particular. Jointly developed technical standards, jointly prepared guidelines, data sheets and publications, such as *Rodenticides: analyses, specifications, formulations*,² are examples of the effective cooperative arrangements existing between the two organizations. Participation of FAO and WHO representatives in meetings on specifications organized by one or other of the organizations also underlines the importance attached to cooperation and coordination in this field. Special attention was paid by the Expert Committee to the harmonization of FAO and WHO specifications, taking into account the difference in their respective objectives. FAO has proposed that, in certain cases, the joint issue of specifications might be considered.

The FAO Panel of Experts, at its next meeting tentatively planned for November 1984, might consider the suggestions for the harmonization of specifications proposed by the WHO Expert Committee.

7.2 Collaborative International Pesticide Analytical Council

The Committee was informed of the continued cooperation between CIPAC and WHO in the development of analytical methods

¹ Unpublished WHO document WHO/VBC/83.884.

² *Rodenticides: analyses, specifications, formulations*, Rome, Food and Agriculture Organization of the United Nations, 1979 (FAO Plant Production and Protection Paper No. 16).

for pesticides. The Committee recommended the continued participation of WHO in CIPAC meetings at which other organizations, such as the AOAC and the International Group of National Associations of Pesticide Manufacturers (GIFAP), are represented. This would avoid the description of different analytical methods for the same compound, and CIPAC's coordinating role in the development of analytical methods is therefore highly useful to WHO.

8. RECOMMENDATIONS

Apart from recommending the revision of existing specifications for pesticides (Annexes 1 and 2) and the establishment of specifications for new pesticides (Annex 3) the Committee also made certain recommendations and suggestions of a more general nature, as follows:

1. The development of specification requirements for new types of formulations should be carried out in parallel with field tests on efficacy and safety.

2. During the period between meetings of the Expert Committee, if the need arises to strengthen an existing specification, the secretariat should issue interim specifications that would supersede the existing specifications and recommend the use of those interim specifications in connection with the purchase of pesticides and their formulations.

3. Close collaboration should be maintained with FAO, especially with the objective of harmonizing FAO and WHO specifications.

4. If resources are available, the established specifications and methods should be published in a sixth edition of *Specifications for pesticides used in public health*.

ACKNOWLEDGEMENTS

The Committee acknowledged the valuable contribution to its work made by the following WHO staff members: Dr A. Dubitskij, Pesticides Development and Safe Use; Dr F. McCullough, Ecology and Control of Vectors; Mr G. Nickitas, Chief, Supply Services; Dr C. Pant, Chief, Ecology and Control of Vectors; Dr N. Rishikesh, Ecology and Control of Vectors, and Dr M. Vandekar, Pesticides Development and Safe Use.

Annex 1

RECOMMENDED GENERAL CHANGES IN EXISTING SPECIFICATIONS AND METHODS¹

Specifications

The Committee recommended the following general changes in certain sections of the existing specifications.

Technical products

(i) *Material* (section 1.1)

The description of the material should be replaced by the following: "The material shall consist of ... [ISO common name] together with related manufacturing compounds and shall be in the form of ..."

Water-dispersible powders

(i) *Description and ingredients* (section 1.1)

The description of the material should be replaced by the following: "The material shall consist of a homogeneous mixture of technical ... [ISO common name] together with filler(s) and other necessary formulants and shall be in the form of ..."

(ii) *Active ingredient content* (section 1.2.1)

The tolerance of " $\pm 5\%$ of (1000 minus nominal content)" for a nominal content above 500 g/kg should be replaced by " $\pm x$ g/kg", the value of x depending upon the manufacturing process, sampling, and the analytical method used for determining the active ingredient content.

¹ *Specifications for pesticides used in public health*, 5th edition, Geneva, World Health Organization, 1979.

(iii) *Sieving after accelerated storage* (section 1.2.2)¹

“Accelerated storage” should be replaced by “heat stability treatment”.

(iv) *Suspensibility* (section 1.2.3)²

The clause “In standard soft water without pretreatment” should be deleted in cases where the value of the suspensibility in standard soft water without pretreatment is the same as the value of the suspensibility in standard hard water after accelerated storage.

(v) *Heat stability treatment*

The following procedure is recommended for the determination of the heat stability of malathion water-dispersible powders and it is also recommended that similar wording should be used in the description of such treatment for all water-dispersible powders, with the exception of DDT water-dispersible powder for overseas shipment: “Fill a 50-ml³ wide-mouth glass bottle to within 1 cm of the top with the sample. Seal the bottle with a phenolic plastic cap having a soft liner which will ensure a good seal. Turn the cap firmly to ensure a tight seal and place in a forced-draught oven maintained at ... °C⁴ for ... hours (days).⁵ At the end of the heating period, remove from the oven and allow to come to room temperature before removing the cap.”

Emulsion concentrate

(i) *Title*

Replace “emulsion concentrate” by “emulsifiable concentrate” in order to follow the GIFAP International Coding System.

¹ Section 1.2.3 in the case of malathion.

² Section 1.2.4 in the case of malathion.

³ 100-ml if a larger quantity of the sample is required for the tests.

⁴ Temperature required by the specification.

⁵ Time required by the specification.

(ii) *Description and ingredients* (section 1.1)

Replace the description of the material by the following wording: "The material shall consist of technical ... [ISO common name] dissolved in suitable solvents with other necessary formulants added. It shall be in the form of a stable liquid free from suspended matter and sediment."

(iii) *Active ingredient content* (section 1.2.1)

The tolerance of " $\pm 5\%$ of (1000 minus nominal content)" for a nominal content above 500 g/kg should be replaced by " $\pm x$ g/kg", the value of x depending upon the manufacturing process, sampling, and the analytical method used for determining the active ingredient content.

(iv) *Heat stability treatment* (section 1.2.5)

Delete the requirement for compliance with section 1.2.2 on the cold test.

Dusting powder

(i) *Title*

Replace "dusting powder" by "dustable powder" in order to follow the GIFAP International Coding System.

(ii) *Description and ingredients* (section 1.1)

Replace the description of the material by the following: "The material shall consist of a homogeneous mixture of technical ... [ISO common name] together with carriers and any other necessary formulants. It shall be a fine, free-flowing powder, free from hard lumps."

General Methods

The Committee also reviewed Part IV, Methods, of the 5th edition of the WHO manual and recommended the deletion of the methods that are obsolete or no longer relevant to the specifications that will appear in the next edition of the manual. The methods recommended for deletion are:

WHO/M/9. Determination of material insoluble in dichlorodifluoromethane.

WHO/M/15. Parr peroxide-bomb (total chlorine) method.

WHO/M/16.R1. Revised Stepanow (total organic chlorine) method.

WHO/M/19. Separation and determination of gamma-isomer content of HCH.

The Committee also recommended the addition to Part IV of the following general method, which is relevant to more than one specification.

Preparation and conditioning of GLC columns

Pass dry nitrogen through the column to remove moisture. Fill the column with a solution of 50 ml/l dimethyldichlorosilane in toluene and allow to stand for 5 minutes. Empty the column. Rinse it once with toluene and then several times with methanol until the rinsings are neutral to litmus. Attach an 8-cm funnel to the exit end of the column. While tapping the tube with a small wooden rod, add the prepared packing material in small quantities until the exit end of the tube is filled to within about 1.5 cm of that end. Move the funnel to the entrance of the column. Insert a small wad of silane-treated glass wool in the exit end of the column and attach a vacuum pump of moderate power to that end. Continue to add packing material slowly while tapping until the tube is filled to within about 2 cm of the entrance end. Insert a small wad of glass wool in the entrance end, compressing the glass wool only enough to hold the packing in place.

Heat the column overnight (at least 15 h) at approximately 20 °C below the maximum temperature recommended for the liquid phase. The heating should be conducted with the exit end of the column unconnected to the detector but with the carrier gas flowing at the recommended rate. Connect the exit end of the column to the detector, set the controls to provide the conditions required by the method and allow the instrument to come to equilibrium. Inject 3- μ l aliquots of standard solution into the chromatograph until constant response is obtained. This criterion is met when at least three consecutive injections give response ratios that agree to within 2%.

Annex 2

RECOMMENDED CHANGES IN ANALYTICAL METHODS USED IN EXISTING SPECIFICATIONS

The Committee recommended that the existing methods should be deleted and replaced by new ones in the following specifications:

HCH

For technical and refined HCH (specification WHO/SIT/2.R5), HCH water-dispersible powder (specification WHO/SIF/2.R5), HCH emulsion concentrate (specification WHO/SIF/5.R5) and HCH dust-able powder (specification WHO/SIF/17.R4), the existing method should be replaced by the GLC method.

Summary of method

The method employs GLC with an OV-210 column for the separation of gamma-isomer from other isomers, and di-n-propyl phthalate as internal standard.¹

Pyrethrum

For pyrethrum (specification WHO/SIT/7.R1), the existing method should be replaced by the GLC method.

Summary of method

The method involves determination of pyrethrins and piperonyl butoxide by GLC using a column packed with 5% OV-101 or 5% OV-1 on 80-100 mesh Chromosorb W(HP) and dicyclohexyl phthalate as internal standard.²

Diazinon

For technical diazinon (specification WHO/SIT/9.R4), diazinon water-dispersible powder (specification WHO/SIF/9.R4) and diazinon

¹ Unpublished WHO document VBC/PDS/EC/83.19.

² *Journal of the Association of Official Analytical Chemists*, 65(2): 455-456 (1982).

emulsion concentrate (specification WHO/SIF/13.R4), the existing method should be replaced by the GLC method.

Summary of method

The method employs GLC with a flame-ionization detector and a glass column, 4 mm in internal diameter, packed with 10% silicone DC 200 on 80–100 mesh Gas-Chrom Q, using aldrin as an internal standard.¹

Dichlorvos

For technical dichlorvos (specification WHO/SIT/16.R1), both of the existing methods should be replaced by the GLC method.

Summary of method

The dichlorvos content is determined by GLC, using a column packed with 3% OV-25 on Gas-Chrom Q or Chromosorb W(HP) and diethyl pimelate as internal standard.²

Fenitrothion

For technical fenitrothion (specification WHO/SIT/17.R1), fenitrothion water-dispersible powder (specification WHO/SIF/29.R1) and fenitrothion emulsion concentrate (specification WHO/SIF/37), the existing method should be replaced by the GLC method.

Summary of method

Samples of technical fenitrothion and formulations are dissolved in chloroform with fluoranthene added as internal standard. The fenitrothion content is determined by GLC using a flame-ionization detector.³

¹ *Official methods of analysis of the AOAC*, 13th edition, Arlington, VA, Association of Official Analytical Chemists, 1980, pp. 121–122.

² Unpublished WHO document VBC IS 82.03.

³ Unpublished WHO document VBC PDS/EC/83.21.

Propoxur

For technical propoxur (specification WHO/SIT/18.R1) and propoxur water-dispersible powder (specification WHO/SIF/30.R1), the existing method should be replaced by the HPLC method.

Summary of method

Samples of technical propoxur or formulated products are extracted with acetonitrile and an internal standard, *n*-butyrophenone, is added. The propoxur content is determined by HPLC using a reverse-phase 10- μ m, C-18 silica-bonded column eluted with a 60 : 40 mixture of acetonitrile and water.¹

Temephos

For technical temephos (specification WHO/SIT/19.R1), temephos emulsion concentrate (specification WHO/SIF/31.R1), temephos emulsion concentrate for *Simulium* control (specification WHO/SIF/34) and temephos granules, the existing method should be replaced by the HPLC method.

Summary of method

The sample is dissolved in ethyl acetate, *p*-nitrophenyl *p*-nitrobenzoate is added as internal standard and, after dilution with *n*-hexane, the sample is injected into the liquid chromatographic column. The response ratio of pesticide to internal standard is compared with that of the standard to give the content of temephos in the sample.²

In the case of temephos granules, a footnote should be added to the effect that the product, when added to drinking-water at the recommended dosage, will not impart an objectionable taste.

Chlorpyrifos

For technical chlorpyrifos (specification WHO/SIT/21) and chlorpyrifos emulsion concentrate (specification WHO/SIF/36), the existing method should be replaced by the HPLC method.

¹ Adopted as an official method at the 97th meeting of the International Association of Official Analytical Chemists, Washington, DC, October 1983.

² Unpublished WHO document VBC/IS/82.05.

Summary of method

The method involves the determination of chlorpyrifos by reverse-phase HPLC using a Zorbax ODS column, acetonitrile-water-acetic acid as eluent, and 1,4-dibromonaphthalene as an internal standard.¹

Malathion

For technical malathion (specification WHO/SIT/10.R4), malathion water-dispersible powder (specification WHO/SIF/10.R4), malathion emulsion concentrate (specification WHO/SIF/14.R4) and malathion dusting powder (specification WHO/SIF/22.R3), the existing methods of analysis for malathion content should be modified as follows:²

Preparation of standard solutions (section 2.1.4)³

Internal standard solution. Prepare a 30 g/l solution of the internal standard in chloroform.

Malathion standard solutions. Weigh accurately quantities of the malathion standard of about 425, 500 and 575 mg directly into separate preweighed 50-ml volumetric flasks.

Malathion, technical and emulsifiable concentrate (section 2.1.8)³

Sample preparation and analysis. Weigh accurately a quantity of the sample containing about 500 mg of malathion directly into a preweighed 50-ml volumetric flask.

Malathion, water-dispersible powder and dustable powder (section 2.1.8)³

Sample preparation and analysis. Weigh accurately about 1.0 g of sample, transfer to a 200-ml screw-capped bottle and add 5 ml of internal standard solution by pipette. Add 50 ml chloroform and shake for approximately 0.5 min. Filter a few ml of the supernatant

¹ *Journal of the Association of Official Analytical Chemists*, **64**(2): 503–504 (1981).

² Unpublished WHO document VBC/PDS/EC/83.20.

³ *Specifications for pesticides used in public health*, 5th edition, Geneva, World Health Organization, 1979.

solution and hold for GLC analysis. The filtration step is best accomplished by drawing 4–5 ml of the supernatant solution into a 10-ml Varipet syringe (Manostat Co., 519 8th Avenue, New York, NY 10018), fitting the syringe with a 13-mm Swinnex filter holder (Millipore SXOO 01300) provided with a glass-fibre filter (Gelman Type A-E, 13 mm), and forcing the solution through the filter into a small screw-capped vial.

Malathion, water-dispersible powder (section 2.2)¹

Determination of isomalathion content. Operating conditions for the gas-liquid chromatograph (section 2.2.6)¹ are as follows:

| | |
|------------------------|--------|
| <i>Temperature</i> | |
| Oven | 180 °C |
| <i>Retention times</i> | |
| Isomalathion peak | 26 min |
| Internal standard peak | 7 min |

Annex 3

RECOMMENDED SPECIFICATIONS FOR NEW PESTICIDES AND FORMULATIONS

The Committee recommended that specifications for the new pesticides bendiocarb and deltamethrin should be included in the next edition of the WHO manual.

The analytical methods and specifications for these compounds and their formulations are summarized below.

BENDIOCARB

Summary of analytical method

The bendiocarb content is determined by the HPLC method, using propiophenone in acetonitrile (10 ml/litre) as internal stan-

¹ *Specifications for pesticides used in public health*, 5th edition, Geneva, World Health Organization, 1979.

standard. Columns slurry-packed with Partisil 10 ODS 2 are used. The eluting solvent consists of acetonitrile mixed with water. The sample is injected into a liquid chromatographic column. The response ratio of pesticide to internal standard is compared with the response ratio of the standard to give the bendiocarb content of the sample.

TECHNICAL BENDIOCARB

1. SPECIFICATION

1.1 Material

The material shall consist of bendiocarb together with related manufacturing compounds and shall be in the form of a white or off-white solid, practically odourless, and free from extraneous impurities or added modifying agents.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and with the following requirements:

| | <i>Minimum</i> | <i>Maximum</i> |
|--------------------------------|----------------|----------------|
| bendiocarb content | 960 g/kg | |
| melting point (method WHO/M/5) | 125 °C | |
| water content (method WHO/M/7) | | 5 g/kg |

1.3 Packing and marking of packages

The technical bendiocarb shall be packed in suitable clean containers, as specified in the order.

All packages shall bear, durably and legibly marked on the container, the following:

- Manufacturer's name
- Technical bendiocarb to specification WHO/SIT/...
- Batch or reference number, and date of test
- Net weight of contents
- Date of manufacture

and the following minimum cautionary notice:

- Technical bendiocarb is a carbamate compound that inhibits cholinesterase. It is poisonous if swallowed or inhaled.
- Keep the material out of the reach of children and well away from foodstuffs, animal feed, and their containers.
- If poisoning occurs call a physician. Atropine is a specific antidote and artificial respiration may be needed.

BENDIOCARB WATER-DISPERSIBLE POWDER

1. SPECIFICATION

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical bendiocarb together with filler and other necessary formulants and shall be in the form of a fine, free-flowing powder that wets readily on stirring into water. The technical bendiocarb used in the manufacture of the water-dispersible powder shall comply with the requirements of specification ...

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and with the following requirements.

1.2.1 Bendiocarb content (g/kg basis)

The content of bendiocarb shall not differ from the nominal content by more than the following amounts:

| <i>Nominal content</i> | <i>Tolerance permitted</i> |
|------------------------|-----------------------------|
| Up to 500 g/kg | ± 5% of the nominal content |
| Above 500 g/kg | ± 25 g/kg |

The average content of all samples taken shall not be lower than the nominal content.

1.2.2 Sieving after heat stability treatment

Not less than 98% of the powder (dry weight) after heat stability treatment shall pass through a 74- μ m sieve when tested by the method described in WHO/M/4.

1.2.3 Suspensibility

In standard hard water after heat stability treatment. A minimum of 50% of the bendiocarb (5.0 g/l) shall be in suspension 30 minutes after agitating a suspension containing 10 g/l of bendiocarb, prepared in standard hard water from the powder subjected to the heat stability treatment.

1.3 Packing and marking of packages

The bendiocarb water-dispersible powder with nominal content up to and including 200 g/kg shall be packed in suitable clean drums, kegs or boxes, as specified in the order. The packages shall contain a lining or bag of polyethylene or equivalent, with a nominal thickness of 0.1 mm. The lining or bag shall be hermetically sealed after filling.

The bendiocarb water-dispersible powder with nominal content higher than 200 g/kg shall be prepacked in individual sealed laminated foil/plastic sachets in the amount corresponding to one pump-charge.

The design of the sachet should be such that it can be opened and easily emptied without spillage. The sachets shall be packed in suitable clean drums, kegs or boxes, as specified in the order. The packages shall contain a lining or bag of polyethylene or equivalent, with a nominal thickness of 0.1 mm.

All packages shall bear, durably and legibly marked on the container, the following:

- Manufacturer's name
- Bendiocarb water-dispersible powder to specification ...
- Bendiocarb ... g/kg
- Batch or reference number, and date of test
- Net weight of contents
- Date of formulation

and the following minimum cautionary notice:

- Bendiocarb is a carbamate compound that inhibits cholinesterase. It is poisonous if swallowed or inhaled.
- Keep the material out of the reach of children and well away from foodstuffs, animal feed, and their containers.
- If poisoning occurs, call a physician. Atropine is a specific antidote and artificial respiration may be needed.

DELTAMETHRIN

Summary of analytical method

After dilution of the samples, the deltamethrin content is determined by comparing the response of the sample with that of a deltamethrin standard of known purity by high-performance liquid chromatography on a column packed with silica.

TECHNICAL DELTAMETHRIN

1. SPECIFICATION

1.1 Material

The material shall consist of deltamethrin together with related manufacturing compounds and shall be in the form of a white to cream-coloured crystalline powder free from extraneous impurities or added modifying agents.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and with the following requirements:

| | <i>Minimum</i> | <i>Maximum</i> |
|--|--|----------------|
| deltamethrin content | 980 g/kg | |
| isomer R | | 10 g/kg |
| melting point (method WHO/M/5) | 98 °C | |
| acid chloride corresponding to deltamethrin | | 2 g/kg |
| acid + anhydride corresponding to deltamethrin | | 10 g/kg |
| optical rotation | $[\alpha]_D^{20} = 57^\circ \pm 1.5^\circ$ | |

1.3 Packing and marking of packages

The technical deltamethrin shall be packed in suitable clean containers, as specified in the order.

All packages shall bear, durably and legibly marked on the container, the following:

- Manufacturer's name
- Technical deltamethrin to specification ...
- Batch or reference number, and date of test
- Net weight of contents
- Date of manufacture

and the following minimum cautionary notice:

- Deltamethrin is a pyrethroid that acts predominantly on the central nervous system, high dosages leading to tonic seizures in experimental animals. A high concentration in air may be an irritant and contact with concentrated product may induce a temporary tingling sensation, particularly on the face. It may be hazardous if swallowed. Do not inhale spray mist. Avoid skin contact; wear protective gloves, clean protective clothing, and a face mask (surgical type) when handling the material. Wash hands and exposed skin thoroughly after using.
- Keep containers out of the reach of children and well away from foodstuffs and animal feed and their containers.
- Deltamethrin is toxic to aquatic wildlife. Avoid accidental contamination of water.
- If poisoning occurs, call a physician. Treatment is symptomatic.

DELTAMETHRIN WATER-DISPERSIBLE POWDER

1. SPECIFICATION

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical deltamethrin together with filler and other necessary formulants and shall be in the form of a fine, free-flowing whitish powder that wets readily on stirring into water. The technical deltamethrin used in the manufacture of the water-dispersible powder shall comply with the requirements of specification ...

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and with the following requirements.

1.2.1 Deltamethrin content (g/kg basis)

The content of deltamethrin shall not differ from the nominal content by more than the following amount:

| <i>Nominal content</i> | <i>Tolerance permitted</i> |
|------------------------|-----------------------------|
| Up to 500 g/kg | ± 5% of the nominal content |

Higher nominal contents are not currently available.

The average content of all samples taken shall not be lower than the nominal content.

1.2.2 Sieving after heat stability treatment

Not less than 98% of the powder (dry weight) after heat stability treatment shall pass through a 74- μ m sieve when tested by the method described in WHO/M/4.

1.2.3 Suspensibility

In standard hard water after heat stability treatment. A minimum of 50% of the deltamethrin (0.025 g/l) shall be in suspension 30 minutes after agitating a suspension containing 0.05 g/l of deltamethrin prepared in standard hard water from the powder subjected to the heat stability treatment.

1.2.4 Acidity or alkalinity

The acidity or alkalinity of the powder, determined by the method described in WHO/M/3, shall not be greater than 5 g/kg calculated as H₂SO₄, or 5 g/kg calculated as NaOH.

1.3 Packing and marking of packages

The deltamethrin water-dispersible powder shall be packed in suitable clean drums, as specified in the order. The drums shall

contain a lining or bag of polyethylene or equivalent, with a nominal thickness of 0.1 mm. The lining or bag shall be hermetically sealed after filling.

All packages shall bear, durably and legibly marked on the container, the following:

- Manufacturer's name
- Deltamethrin water-dispersible powder to specification ...
- Deltamethrin ... g/kg
- Batch or reference number, and date of test
- Net weight of contents
- Date of formulation

and the following minimum cautionary notice:

- Deltamethrin is a pyrethroid that acts predominantly on the central nervous system, high dosages leading to tonic seizures in experimental animals. A high concentration in air may be an irritant and contact with concentrated product may induce a temporary tingling sensation, particularly on the face. It may be hazardous if swallowed. Do not inhale spray mist. Avoid skin contact; wear protective gloves, clean protective clothing, and a face mask (surgical type) when handling the product. Wash hands and exposed skin thoroughly after using.
- Keep containers out of the reach of children and well away from foodstuffs and animal feed and their containers.
- Deltamethrin is toxic to aquatic wildlife. Avoid accidental contamination of water.
- If poisoning occurs, call a physician. Treatment is symptomatic.

DELTAMETHRIN EMULSIFIABLE CONCENTRATE

1. SPECIFICATION

1.1 Description and ingredients

The material shall consist of technical deltamethrin dissolved in suitable solvents with other necessary formulants added. It shall be in the form of a stable liquid free from suspended matter and sediments.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and the following requirements.

1.2.1 Deltamethrin content

The content of deltamethrin shall not differ from the nominal content by more than the following amounts:

| <i>Nominal content</i> | <i>Tolerance permitted</i> |
|------------------------|-----------------------------|
| Up to 500 g/kg | ± 5% of the nominal content |

Higher nominal contents are not currently available

The average content of all samples taken shall not be lower than the nominal content.

1.2.2 Cold test

No separation of solid or oily material shall occur.

1.2.3 Flash-point

The flash-point of the product shall comply with all national and/or international transport regulations.

1.2.4 Stability of emulsion

In standard soft water. Any separation, including creaming/oiling at the top and creaming/oiling sedimentation at the bottom, of 100 ml of emulsion prepared in standard soft water with 5 ml of concentrate shall not exceed 2 ml when tested as described in method WHO/M/13.R1.

In standard hard water. Any separation, including creaming/oiling at the top and creaming/oiling sedimentation at the bottom, of 100 ml of emulsion prepared in standard hard water with 5 ml of concentrate shall not exceed 2 ml when tested as described in method WHO/M/13.R1.

1.2.5 *Heat stability treatment*

The concentrate, after heat stability treatment, shall comply with the requirements of sections 1.2.1, 1.2.4 and 1.2.6 of this specification.

1.2.6 *Acidity or alkalinity*

The acidity or alkalinity of the concentrate, determined by the method described in WHO/M/3, shall not be greater than 0.5 g/kg calculated as H₂SO₄ or 0.5 g/kg calculated as NaOH.

1.3 **Packing and marking of packages**

The deltamethrin emulsifiable concentrate shall be packed in suitable, clean containers, as specified in the order.

All packages shall bear, durably and legibly marked on the containers, the following:

- Manufacturer's name
- Deltamethrin emulsifiable concentrate to specification ...
- Deltamethrin ... g/kg
- Batch or reference number, and date of test
- Net weight of contents
- Date of formulation
- Instructions for dilution

and the following minimum cautionary notice:

- Deltamethrin is a pyrethroid that acts predominantly on the central nervous system, high dosages leading to tonic seizures in experimental animals. A high concentration in air may be an irritant and contact with concentrated product may induce a temporary tingling sensation particularly on the face. It may be hazardous if swallowed. Do not inhale spray mist. Avoid skin contact; wear protective gloves, clean protective clothing, and a face mask (surgical type) when handling this concentrate. Wash hands and exposed skin thoroughly after using.
- Keep containers out of the reach of children and well away from foodstuffs and animal feed and their containers.
- Deltamethrin is toxic to aquatic wildlife. Avoid accidental contamination of water.
- If poisoning occurs, call a physician. Treatment is symptomatic.

DELTAMETHRIN DUSTABLE POWDER
(not for application to skin, clothing or bedding)

1. SPECIFICATION

1.1 Description and ingredients

The material shall consist of a homogeneous mixture of technical deltamethrin together with carriers and any other necessary formulants. It shall be in the form of a fine, free-flowing powder, free from hard lumps.

1.2 Chemical and physical requirements

The material, sampled from any part of the consignment, shall comply with the requirements of section 1.1 and with the following requirements.

1.2.1 Deltamethrin content (g/kg basis)

The content of deltamethrin shall not differ from the nominal deltamethrin content by more than $\pm 10\%$. The average content of all samples taken shall not be lower than the nominal content.

1.2.2 Sieving after heat stability treatment

Not less than 98% of the powder (dry weight) after heat stability treatment shall pass through a 150- μm sieve when tested by the method described in WHO/M/4.

1.2.3 Dustability after heat stability treatment

After heat stability treatment, the powder shall issue freely without clogging or bridging, when tested in a hand dusting apparatus conforming to specification WHO/EQP/4.R2.

1.2.4 Acidity or alkalinity

The acidity or alkalinity of the powder, determined by the method described in WHO/M/2, shall not be greater than 1 g/kg calculated as H_2SO_4 or 2 g/kg calculated as NaOH.

1.3 Packing and marking of packages

The deltamethrin dustable powder shall be packed in suitable clean drums, as specified in the order.

All packages shall bear, durably and legibly marked on the container, the following:

- Manufacturer's name
- Deltamethrin dustable powder to specification ...
- Deltamethrin ... g/kg
- Batch or reference number, and date of test
- Net weight of contents
- Date of formulation

and the following minimum cautionary notice:

- Not for application to skin, clothing or bedding. Deltamethrin is a pyrethroid that acts predominantly on the central nervous system, high dosages leading to tonic seizures in experimental animals. A high concentration in air may be an irritant and contact with concentrated product may induce a temporary tingling sensation in the skin. It may be hazardous if swallowed. Do not inhale cloud of dust. Avoid skin contact; wear protective gloves and clean protective clothing when handling the material. Wash hands and exposed skin thoroughly after using.
- Keep container out of the reach of children and well away from foodstuffs and animal feed and their containers.
- Deltamethrin is toxic to aquatic wildlife. Avoid accidental contamination of water.
- If poisoning occurs, call a physician. Treatment is symptomatic.

Annex 4

RECOMMENDED CAUTIONARY NOTICES FOR DUSTABLE POWDERS

Bendiocarb dustable powder

The dustable powder containing 10 g/kg of bendiocarb has been in commercial use for about 5 years against household pests (cockroaches, fleas, bedbugs, etc.) but has never been recommended for application to skin or clothing. When specifications for bendiocarb

dustable powders are developed they should carry the title: Bendiocarb dustable powder (not for application to skin, clothing or bedding, and the following minimum cautionary notice is recommended:

Not for application to skin, clothing or bedding. Bendiocarb is a carbamate compound that inhibits cholinesterase. It is poisonous if swallowed or inhaled. Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers. If poisoning occurs call a physician. Atropine is a specific antidote.

Deltamethrin dustable powder

Owing to the irritative properties of deltamethrin it is highly unlikely that deltamethrin dustable powders will be recommended for application to skin or clothing. The title of the corresponding specification should be: Deltamethrin dustable powder (not for application to skin, clothing or bedding).

The recommended minimum cautionary notice should read as follows:

- Not for application to skin, clothing or bedding. Deltamethrin is a pyrethroid that acts predominantly on the central nervous system, high dosages leading to tonic seizures in experimental animals. A high concentration in air may be an irritant and contact with concentrated product may induce a temporary tingling sensation in the skin. It may be hazardous if swallowed. Do not inhale cloud of dust. Avoid skin contact; wear protective gloves and clean protective clothing when handling the material. Wash hands and exposed skin thoroughly after using.
- Keep container out of the reach of children and well away from foodstuffs and animal feed and their containers.
- Deltamethrin is toxic to aquatic wildlife. Avoid accidental contamination of water.
- If poisoning occurs, call a physician. Treatment is symptomatic.

DDT dustable powder

In view of the low mammalian dermal toxicity and excellent safety record of DDT during the last 40 years, it is considered that there is no need to distinguish between its various uses. The following

wording for the dustable powders, formulated both for application to skin or clothing or for other purposes, is recommended:

- Keep well away from foodstuffs and animal feed and their containers.

HCH dustable powder

It is recommended that HCH dustable powder should be employed for uses *other* than application to skin or clothing and carry the following minimum cautionary notice:

- Not for application to skin, clothing or bedding. Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers.

Its gamma-isomer, lindane, may be recommended both for application to skin or clothing and for other uses. Although the content of active ingredient will probably differ, the minimum cautionary notice may read:

- Keep well away from foodstuffs and animal feed and their containers.

Malathion dustable powder

Malathion dustable powder, when formulated for application to skin, clothing or bedding, will normally have a 10 g/kg nominal content of active ingredient. The following minimum cautionary notice is then recommended:

- The powder may be hazardous if swallowed. Keep away from foodstuffs and animal food and their containers. Store in a cool place and use as soon as possible after formulation to avoid decomposition, which may occur in conditions of prolonged storage in warm conditions.

The following minimum cautionary notice for malathion dustable powder with a higher nominal content than 10 g/kg, for other uses than for application to skin, clothing or bedding, is recommended:

- Not for application to skin, clothing or bedding. Malathion is an organophosphorus compound that inhibits cholinesterase. It is poisonous if swallowed. Keep the material out of the reach of children and well away from foodstuffs and animal feed and their

containers. If poisoning occurs, call a physician. Atropine and pralidoxime are specific antidotes, and artificial respiration may be needed. Store in a cool place and use as soon as possible after manufacture to avoid decomposition, which may occur under conditions of prolonged storage in warm conditions.

Permethrin dustable powder

Permethrin dustable powder may be recommended either for application to skin or clothing or for other uses, provided that the nominal content of the active ingredient is appropriate to the particular use. When interim specifications for permethrin dustable powder for application to skin, clothing or bedding are developed, the following minimum cautionary notice is recommended:

—The powder may be hazardous if swallowed. Keep away from foodstuffs and animal feed and their containers. If poisoning occurs, call a physician. Treatment is symptomatic.

For dustable powders for other uses, the following minimum cautionary notice is proposed:

—Not for application to skin, clothing or bedding. Permethrin is a pyrethroid that acts predominantly on the nervous system. It may be hazardous if swallowed. Do not inhale cloud of dust. Avoid skin contact. Wash hands and exposed skin thoroughly after use. Keep the material out of the reach of children and well away from foodstuffs and animal feed and their containers. If poisoning occurs call a physician. Treatment is symptomatic.

**WORLD HEALTH ORGANIZATION
TECHNICAL REPORT SERIES**

Recent reports:

| No. | | Sw. fr. |
|-----|--|---------|
| 655 | (1980) Resistance of vectors of disease to pesticides Fifth report of the WHO Expert Committee on Vector Biology and Control (82 pages)..... | 6.— |
| 656 | (1981) Assessment of public health and social problems associated with the use of psychotropic drugs Report of the WHO Expert Committee on Implementation of the Convention on Psychotropic Substances, 1971 (54 pages)..... | 4.— |
| 657 | (1981) The effect of female sex hormones on fetal development and infant health Report of a WHO Scientific Group (76 pages)..... | 5.— |
| 658 | (1981) WHO Expert Committee on Biological Standardization Thirty-first report (324 pages)..... | 21.— |
| 659 | (1981) Wholesomeness of irradiated food Report of a Joint FAO/IAEA/WHO Expert Committee (34 pages)... | 3.— |
| 660 | (1981) Nongonococcal urethritis and other selected sexually transmitted diseases of public health importance Report of a WHO Scientific Group (142 pages)..... | 9.— |
| 661 | (1981) Rapid laboratory techniques for the diagnosis of viral infections Report of a WHO Scientific Group (60 pages)..... | 4.— |
| 662 | (1981) Health effects of combined exposures in the work environment Report of a WHO Expert Committee (76 pages)..... | 5.— |
| 663 | (1981) Education and training in occupational health, safety and ergonomics Eighth report of the Joint ILO/WHO Committee on Occupational Health (48 pages)..... | 3.— |
| 664 | (1981) Recommended health-based limits in occupational exposure to selected organic solvents Report of a WHO Study Group (84 pages)..... | 6.— |
| 665 | (1981) Neuronal aging and its implications in human neuronal pathology Report of a WHO Study Group (88 pages)..... | 6.— |
| 666 | (1981) Intestinal protozoan and helminthic infections Report of a WHO Scientific Group (152 pages)..... | 10.— |
| 667 | (1981) The role of the health sector in food and nutrition Report of a WHO Expert Committee (92 pages)..... | 6.— |
| 668 | (1981) Disability prevention and rehabilitation Report of a WHO Expert Committee (40 pages)..... | 3.— |
| 669 | (1981) Evaluation of certain food additives Twenty-fifth report of the Joint FAO/WHO Expert Committee on Food Additives (48 pages)..... | 3.— |
| 670 | (1981) Research on the menopause Report of a WHO Scientific Group (120 pages)..... | 8.— |
| 671 | (1982) Tuberculosis control Report of a Joint IUAT/WHO Study Group (26 pages)..... | 3.— |

| | | |
|-----|--|------|
| 672 | (1982) Control of vitamin A deficiency and xerophthalmia Report of a Joint WHO/UNICEF/USAID/Helen Keller International/ IVACG Meeting (70 pages)..... | 7.— |
| 673 | (1982) WHO Expert Committee on Biological Standardization Thirty-second report (180 pages)..... | 13.— |
| 674 | (1982) Treponemal infections Report of a WHO Scientific Group (75 pages)..... | 6.— |
| 675 | (1982) Chemotherapy of leprosy for control programmes Report of a WHO Study Group (33 pages)..... | 4.— |
| 676 | (1982) Interferon therapy Report of a WHO Scientific Group (28 pages)..... | 3.— |
| 677 | (1982) Recommended health-based limits in occupational exposure to pesticides Report of a WHO Study Group (110 pages)..... | 8.— |
| 678 | (1982) Prevention of coronary heart disease Report of a WHO Expert Committee (53 pages)..... | 5.— |
| 679 | (1982) Biological control of vectors of disease Sixth report of the WHO Expert Committee on Vector Biology and Control (39 pages)..... | 4.— |
| 680 | (1982) Malaria control and national health goals Report of the Seventh Asian Malaria Conference (68 pages)..... | 6.— |
| 681 | (1982) WHO Expert Committee on Specifications for Pharmaceutical Preparations Twenty-eighth report (33 pages)..... | 4.— |
| 682 | (1982) Bacterial and viral zoonoses Report of a WHO Expert Committee with the participation of FAO (146 pages)..... | 11.— |
| 683 | (1982) Evaluation of certain food additives and contaminants Twenty-sixth report of the Joint FAO/WHO Expert Committee on Food Additives (51 pages)..... | 5.— |
| 684 | (1983) Recommended health-based occupational exposure limits for selected vegetable dusts Report of a WHO Study Group (78 pages)..... | 6.— |
| 685 | (1983) The use of essential drugs Report of a WHO Expert Committee (46 pages)..... | 4.— |
| 686 | (1983) Primary prevention of essential hypertension Report of a WHO Scientific Group (40 pages)..... | 4.— |
| 687 | (1983) WHO Expert Committee on Biological Standardization Thirty-third report (184 pages)..... | 13.— |
| 688 | (1983) Integrated vector control Seventh report of the WHO Expert Committee on Vector Biology and Control (72 pages)..... | 6.— |
| 689 | (1983) A rational approach to radiodiagnostic investigations Report of a WHO Scientific Group on the Indications for and Limita- tions of Major X-Ray Diagnostic Investigations (49 pages)..... | 5.— |
| 690 | (1983) New approaches to health education in primary health care Report of a WHO Expert Committee (44 pages)..... | 4.— |
| 691 | (1983) Prevention of liver cancer Report of a WHO Meeting (30 pages)..... | 4.— |