Specifications and standards for phenoxyacetic acid herbicides

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SUMMARY

Phenoxyacetic acid herbicides offered for sale in Victoria must comply with the Australian Standard Specification for Herbicides of the Phenoxyacetic Acid Type, AS 1175-1976. In addition, specifications drawn up by the Department of Crown Lands and Survey for herbicides supplied to the Department under contract also require that these herbicides comply with this Standard.

Testing of samples submitted with tenders, of materials held in store, and of materials which have proved to be unsatisfactory in the field revealed that a considerable number of batches of ester formulations of 2,4-D and 2,4,5-T did not meet the required specifications. One area of concern is that a batch which initially meets the specifications may prove to be unstable after storage for a period. The failure of many batches to meet the specifications raises the question as to whether the specifications are adequate or whether there are problems associated with quality control during formulation.

As problems have been experienced with materials supplied to the Department, it is possible that chemicals offered for sale on the open market may not always comply with the prescribed standard. This suggests that a closer look at the situation is required.

INTRODUCTION

All pesticides offered for sale in Victoria must be registered under the Pesticides Act 1958. In this Act there is provision for standards to be prescribed for any pesticide, and where a standard has been prescribed it is an offence to sell any of that pesticide which is not in accordance with the standard. Under this provision of the Act standards have been proclaimed prescribing that any herbicide containing 2,4-D, 2,4,5-T or MCPA shall comply with the Australian Standard Specification for Herbicides of the Phenoxyacetic Acid Type, As 1175-1976 (Vic. Govt. Gaz. 1977).

Each year large quantities of herbicides are used by the Victorian Dpeartment of Crown Lands and Survey for the control of noxious weeds. Approximately 80,000 ℓ of the phenoxyacetic acid herbicides, MCPA, 2,4-D and 2,4,5-T, have been purchased annually. However, with the introduction of the Weedicide Supply Scheme from July 1977, whereby herbicides are sold directly to landholders by the Department for noxious weed control, the amount of these chemicals purchased each year will increase considerably. Herbicides are purchased from various suppliers under contracts let by the State Tender Board. Each contract runs for a two year period.

In addition to the requirement under the Pesticides Act, specifications prepared by the Department also require that phenoxyacetic acid herbicides supplied under contract must comply with the specifications of the Australian Standard. For the current contract

period (1977/79) these herbicides must comply with the specifications of Australian Standard 1175-1976 (Standards Assoc. of Aust. 1976). This standard is a revision of Australian Standard N50-1965 (Standards Assoc. of Aust. 1965) with which these herbicides were required to comply during previous contract periods.

In general, the specifications require that the herbicides must meet certain standards with respect to stability in hard water, stability at low temperature and the percentage of insoluble material. In addition, tolerances are specified for variability in the stated percentage of active ingredient. These specifications are designed to ensure that the quality of the material remains constant from batch to batch and that the material is, and will remain for a reasonable period, in a form suitable for use in the field under a wide range of conditions. For example, a material which meets the requirements for hard water stability should mix readily with water with a hardness equivalent up to 1000 ppm without separation in the case of emulsions, or without forming a precipitate in the case of water soluble materials. Water with this degree of hardness may be encountered on occasions in the field and could be used by Departmental spray gangs for the mixing of herbicides. Also, in many country depots quantities of herbicides are frequently held for extended periods, particularly over the winter months when little spraying is carried out. Materials which meet the requirements for low temperature stability should be capable of being stored under such conditions without crystallizing, or if freezing does occur they should readily go back into solution as the temperature rises.

The specifications for the various herbicides make no reference to the biological effectiveness of the formulation, as it is extremely difficult to define what would be required in such a specification. The requirement that a formulation must meet certain specifications with regard to physical stability does not mean that it necessarily will be any more effective biologically than one which does not comply. However, formulations which are unstable physically, even though they may be effective biologically, would present problems in application if crystallization or separation of material had occurred.

At the beginning of each two year contract period samples submitted with tenders for the supply of herbicides to the Department of Crown Lands and Survey are tested to see that they comply with the specifications. During the 1975/77 contract period tests also were carried out periodically on material held in the Departmental store. Testing of samples submitted following complaints from Departmental field staff of defective herbicide is also carried out. This paper presents the results of testing during the period July 1975 to December 1977.

METHODS AND RESULTS

In all cases samples were tested according to the methods specified in the particular Australian Standard with which herbicides were required to comply for a given contract period.

(a) Testing of samples submitted with tenders

The results of testing of samples of the phenoxyacetic acid herbicides submitted at the beginning of the two contract periods, 1975/77 and 1977/79, for hard water and low temperature stability are summarized in Table 1. At both times several samples of ester

formulations of 2,4-D and 2,4,5-T did not meet the specifications. In all cases the unsatisfactory samples failed the hard water stability test while several samples also failed the low temperature stability test.

In addition, the samples were analysed by the Division of Agricultural Chemistry of the Department of Agriculture to determine their percentage acid equivalent. All samples complied with the specifications in this regard.

Table 1. Number of samples of various phenoxyacetic acid herbicides tested for hard water and low temperature stability, and the number which failed to meet the specifications, at the beginning of the 1975/77 and 1977/79 contract periods

Herbicide	1975/77		1977/79	
	No. tested	No. failed	No. tested	No. failed
MCPA	2	0	3	0
2,4-D amine	2	0	3	0
2,4-D ester (80%)	3	1	4	3
2,4-D ester (40%)	1	0	1	0
2,4,5-T ester (80%)	3	1	3	2
2,4,5-T low volatile ester (40%)	2	1	2	1
Total	13	3	16	6
Percentage failure		23%		38%

(b) Testing of material held in the Departmental store

At approximately three-monthly intervals throughout the contract period 1975/77 samples were taken from each batch of phenoxyacetic acid herbicide being held at that time in the Departmental bulk store in Melbourne. One drum was selected at random from each batch and after thorough shaking a 250 ml sample was withdrawn, care being taken to obtain liquid from various levels in the drum. Tests were then carried out on each sample to determine its stability in hard water and at low temperature.

The results of this testing are summarized in Table 2. Of the 42 different batches of phenoxyacetic acid herbicides tested, 11 batches were below specification. The unsatisfactory batches were all ester formulations and all of them failed the hard water stability test, while six batches of 2,4-D ester also failed the low temperature stability test.

Table 2. Number of batches tested over the contract period 1975/77 of various phenoxyacetic acid herbicides held in store and the number and percentage which failed to meet specifications

Herbicide	No. of batches tested	Failed to meet specification		Tests failed	
		No.	%	Hard water No.	Low temp. No.
MCPA	4	0	0	0	0
2,4-D amine	7	0	0	0	0
2,4-D ester (80%)	13	6	46	6	4
2,4-D ester (40%)	2	2	100	2	2
2,4,5-T ester (80%)	3	0	0	0	0
2,4,5-T low volatile ester (40%)	13	3	23	3	0
Total	42	11	26	11	6

(c) Complaints received from field staff of unsatisfactory herbicide

Periodically complaints are received from Departmental field staff that herbicides supplied are unsatisfactory in some way. The most common complaints are that the formulation has crystallized or that there has been separation of materials in the formulation resulting in a precipitate in the bottom of the drum. Occasionally complaints are received of poor emulsification when the herbicide is mixed with water, flaky impurities in the drum, ineffective control of weeds, and with one batch gas pressure inside had caused expansion of the drums.

A summary of complaints regarding the phenoxyactic acid herbicides during the period 1 July 1975 to 2 December 1977 is given in Table 3. All complaints concerned ester formulations of 2,4-D and 2,4,5-T. A total of 37 complaints, involving 20 batches, and in excess of 4000 litres of material, was received.

Samples were taken of these unsatisfactory materials and subjected to the hard water and low temperature stability tests to see if they complied with the specifications. All but one of the 20 batches involved failed these tests and thus did not meet the specifications. The one batch which passed the tests was claimed to form a poor emulsion when used in the field, but proved to be satisfactory in the laboratory.

Of the 20 batches, six had been tested in the periodical sampling of chemicals held in the Departmental store. At that time five of the batches had been satisfactory while one failed to meet specifications. However, much of that batch had already been distributed to country depots at the time of testing.

Table 3. Number of complaints received, and the number of batches involved, of unsatisfactory phenoxyacetic acid herbicides during the period 1/7/75 to 2/12/77

Herbicide	No. of complaints	No. of batches involved	Nature of complaints
MCPA	0	0	-
2,4-D amine	0	0	-
2,4-D ester (80%)	20	6	Separation of material, crystallization, expansion of drums, ineffective weed control.
2,4-D ester (40%)	1	1	Crystallization.
2,4,5-T ester (80%)	14	11	Separation of material, crystallization, poor emulsification, flaky impurity.
2,4,5-T low volatile ester (40%)	2	2	Separation of material.
Total	37	20	

DISCUSSION

The results of testing of phenoxyacetic acid herbicides carried out by the Department of Crown Lands and Survey have shown that a considerable number of batches of ester formulations did not meet the required specifications. Does this mean that there is a lack of effective quality control in the formulation of these materials, or that the specifications are unrealistic and difficult to meet? Most of the samples tested were required to meet the specifications of Australian Standard N50-1965, but in the revised Standard (AS 1175-1976), which applies for the 1977/79 contract period, the standards set for hard water stability and low temperature stability have been raised considerably. This has made the specifications even more difficult to meet as indicated by the 38% failure of samples tested at the beginning of the 1977/79 contract period compared with 23% at the beginning of the 1975/77 period.

Another area of considerable concern is that a formulation which initially meets the specifications with regard to stability in hard water and at low temperature may prove to be unstable after storage for a period. This is borne out by the fact that several batches which proved unsatisfactory in the field had been tested previously and found to be satisfactory but after storage for a time problems, such as crystallization or separation, had occurred. This suggests that the requirement that a formulation be stable in hard water and at low temperature bears no relation to its stability on storage and that the present specifications are not adequate to ensure that a formulation remains stable after storage for a period. Perhaps an additional requirement for stability on storage should be included in the specifications and that the shelf life of the formulation be stated on the label.

Initially the Departmental specifications were intended to ensure that materials purchased by the Department for its own use would be in a form suitable for use in the field, but as the Department has now entered the field of selling herbicides directly to landholders for noxious weed control, it is of even greater importance that materials received comply with the specifications. There is an obligation on the Department to see that herbicides it offers for sale are suitable for use in the field. As a consequence of the failure of many batches to meet the specifications and to meet this obligation the Department now requires that each batch of herbicide delivered be accompanied by a certificate from the manufacturer stating that it meets the specifications for that material. Even under this system there have been cases reported of material giving problems in the field and when these formulations were tested it was found that they no longer met the specifications.

As phenoxyacetic acid herbicides supplied to the Department of Crown Lands and Survey do not always meet the required specifications, and problems are experienced in the field, it is also possible that a similar situation may exist with materials offered on the open market. This indicates that a closer look is required to see that the phenoxyacetic acid herbicides offered for sale comply with the standards prescribed under the Pesticides Act.

REFERENCES

Standards Association of Australia (1965).- Australian Standard Specification for Hormone Weed Killers of the Phenoxyacetic Acid Type, AS N50-1965. Published by Standards Association of Australia, Sydney.

Standards Association of Australia (1976).- Australian Standard specification for Herbicides of the Phenoxyacetic Acid Type, AS 1175-1976. Published by Standards Association of Australia, North Sydney.

Victoria Government Gazette (1977).- Pesticides Act 1958, Standards for Pesticides - Proclamation. Victoria Government Gazette, No. 58, p 2212, 6 July 1977.