

## ANALYSIS OF FORMULATIONS AND RESIDUES - SOME CURRENT CONSIDERATIONS

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I will give a very brief outline of recent Australian work on analysis of formulations and residues with emphasis on determination of amounts of chemicals as a tool for improving usage of chemicals. I feel sure that my comments on Australia will apply to other countries and I will be brief because I think the discussion will prove more useful than my talk.

**FORMULATIONS:** The following faults with formulations have been observed.

- 1) Some formulations were supplied by manufacturer at less than stated strength.
- 2) Some formulations broke down in containers - i.e. lost chemical strength.
- 3) Some formulations congealed in containers - i.e. lost physical properties.
- 4) Some formulations were delivered with added noxious compounds (solvents).
- 5) Some formulations, when diluted with water, became heterogeneous (i.e. settling occurred).
- 6) Some formulations, when diluted with hard but not soft water, became heterogeneous.

There is therefore a need to determine chemical and physical properties of formulations under conditions of use. If a new chemical is to be used, such determinations should be carried out, as a precaution, in extensive trials before full introduction. In all cases, sampling is required to determine concentrations. Therefore there is a need for rapid and cheap methods of analysis.

A cheap analysis usually means analysis by color, or a titrimetric analysis. Such methods of analyses are declining in highly developed countries. This is a pity, for two reasons:

- 1) Such methods are cheaper than electronic methods and therefore more universally applicable.
- 2) Such methods usually have constant machine response and primary analytical standards, and are therefore less prone to the gross errors that can occur in electronic analyses.

**RESIDUES ON GRAINS:** From analyses of residues on grains we have found, in Australia:

- 1) The average residue is predictable.
- 2) A small area of grain, e.g. 100 tonnes, in a large well-treated storage can contain no insecticide at

- all because, for example, the insecticide pump was turned off for an hour during receipt of grain.
- 3) Experimental treatments can be mislabelled.
  - 4) Some storages can receive erratic application of insecticides because of the position of the spray.
  - 5) Marked improvements in effectiveness of protectants can be achieved by even small changes in temperature (a few degrees is important) or by partial turning of bins to even out residues.

The value of determination of residues, in this context, is that they point to areas where improvements can be made. They result in better control of insects, at little extra cost and sometimes at reduced cost. However, the value of such work is usually only of local benefit - e.g. this spray is wrongly placed, that water is too hard, etc. Therefore each country and each district must determine its own residues.

Therefore we need cheap, rapid methods to analyze residues.

In my experience, small differences in residues between storages usually lead to no practical improvement. For example, if residues in two storages are 4 and 5 g/t respectively, one cannot draw conclusions about application in either storage. If, however, residues are 0.5 and 5 g/t, one can draw conclusions and often the source of error can be detected, and corrected. For such purposes, there is need for many analyses, but they do not have to be precise. In Australia, we have found semiquantitative methods of analysis to be very useful, for example analysis of bioresmethrin or carbaryl by a color reaction on a plate for Thin-Layer Chromatography. Such methods are very rapid, inexpensive and sufficiently precise ( $\pm 20\%$ ) for most purposes.

**COLLABORATIVE ANALYTICAL PROGRAMS:** Such programs, where several analysts analyze a given sample, are excellent checks on reliability of procedures and help to detect, and therefore correct, errors made by a particular analyst.

**SUGGESTIONS FOR INTERNATIONAL COOPERATION ON METHODS OF ANALYSIS SUITABLE FOR LABORATORIES WITH LIMITED FINANCES:** There are two major objections to the use of determination of chemical residues as a tool for improving the benefits of chemical usage. These objections are the cost of chemical laboratories, and the reliability of results from one laboratory. I put forward for your consideration some proposals to reduce cost and increase reliability.

- 1) A list should be collected of regional and international laboratories prepared to check suitable methods of analysis. Such collaborative programs are the best test of reliability. For most purposes regional programs are adequate.

- 2) Residue chemists could develop methods of analysis suitable for a given type of equipment if laboratories with limited resources could standardize on a given type of equipment.
- 3) Emphasis should be placed on volumetric and color methods of analysis, where possible (e.g. malathion, pyrethrins, fenitrothion, carbaryl).
- 4) Regional organizations should ensure that suitable methods of analysis are evaluated before the introduction of new insecticides. Such organizations could request developmental work from laboratories outside the region.
- 5) Analytical procedures suitable for developing country would be evaluated more frequently in developed countries if they were more aware of the analytical requirements and analytical equipment of developing countries. Would it be worthwhile to exchange such information within regions, and between regions?
- 6) Might it be useful, at the next Conference, and informally at this Conference, to have one period devoted to sub-programs, for example, a program on analysis of residues?