How to Get Valid Pesticide Residue Data

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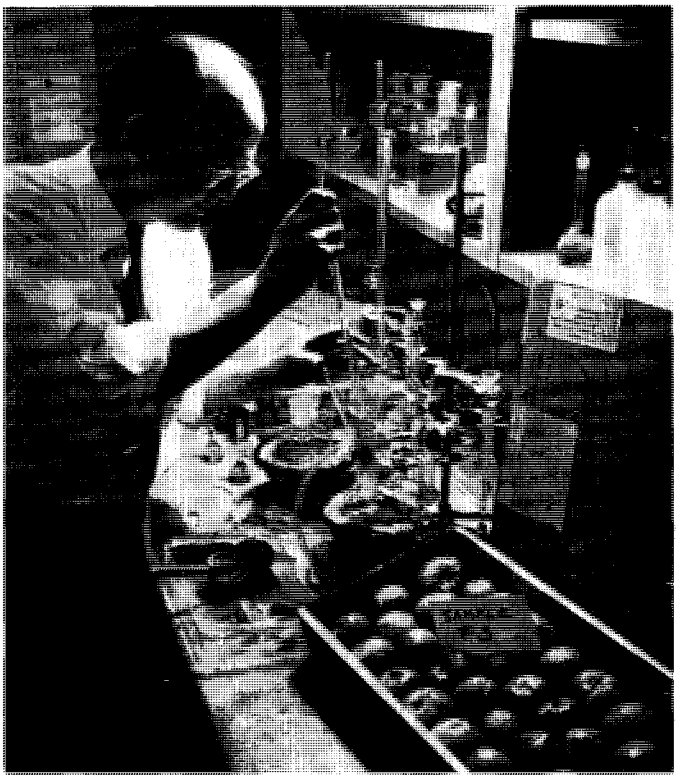
For most efficiency, chemist, pathologist, and entomologist should cooperate closely, with the chemist being informed at least six months before actual field experiment is harvested for residue testing



Du Pont chemist checks fungicide residue on tomatoes

ANALYTICAL CHEMISTRY plays an increasingly important role in research leading to the development of pesticides. A prime requirement for the successful launching of a modern pesticide is the availability of suitable methods of analysis. Analytical procedures for pesticide residues, although inadequate in some instances, are generally quite satisfactory. But analysts still want to know, even with all the modern tools of chemistry and engineering at their disposal, how they can make certain that they will have valid, useful residue data once they have completed their involved and expensive determinations.

This is a basic problem that faces all residue chemists in state, federal, and industrial laboratories. How, they ask, can we organize our field experiments so that we can realize the greatest useful residue information with the least expenditure in time and money?

Without doubt there has been appalling waste and lack of planning in all phases of this and other types of research. Because of the relative recency of this field of investigation, these mistakes can perhaps be "chalked off to experience." But they should certainly be remembered. And in the future, it would seem that planned and standardized procedures should be followed by all, if pesticide residue data are to be comparable and useful.

How can increased efficiency be assured? First and most important there should be very close cooperation between the entomologist, plant pathologist, or other crop scientist, and the chemist who is actually going to run the analyses. This cooperation should begin in the very early stages. Once the entomologist who is to cooperate in the proposed residue study has determined that a compound shows promise insecticidally, he should mention to the chemist the possible need for residue studies. This should be done at least six months before the actual field experiment will be harvested for residues. Before he agrees to a residue experiment in the field, the chemist must then investigate several aspects of this compound:

- What are its physical and chemical properties? How fast will it break down, theoretically, under field conditions? Is it persistent enough to constitute a possible residue hazard?
- How toxic is this chemical to mammals? What is its official or temporary tolerance, if any?
- Will this pesticide and the crop on which it will be tested be of economic importance to the state(s)? Will the compound be used on crops that may present a particular residue hazard? Will dilution of the pesticide by growth be a significant factor with the particular crop to be investigated? (When dealing with conventional insecticides, primary effort should be placed on those crops that have the greatest surface area per unit of fresh weight. There is no point in spending considerable time and effort analyzing for a conventional insecticide on such crops as tomatoes or watermelons at the expense of leafy vegetables. Systemic insecticides present a slightly different problem but in general require a longer breakdown period on the leafy crops as contrasted to crops having a relatively smooth surface.)
- Is there a specific, reliable, and economically feasible analytical method available? Will the cost of specialized equipment required for analysis be prohibitive? (Some specific methods are too expensive or require too much specialized equipment, chemicals, and glassware to be handled by smaller residue laboratories having limited budgets. Therefore, a less expensive nonspecific method may have to be utilized. Whenever possible, how-

ever, the analytical procedure employed should be one approved by the Food and Drug Administration.)

Once the chemist and the entomologist (pathologist, other scientist) have agreed to cooperate on a particular pesticide residue experiment, then for maximum efficiency the following main points should be coordinated between them:

How Many and What Dosage Levels and Formulations Should Be Tested? Here the chemist must be conservative, and avoid being overloaded by too large a field experiment. If the treatments are carefully chosen, maximum information can be obtained from a rather modest field experiment. Experience at the University of Florida's Agricultural Experiment Station indicates that the best procedure is to concentrate residue studies on one station-recommended formulation, either emulsifiable concentrate, wettable powder, or dust. Past tests have revealed no consistent significant differences in residues among these three types of formulation when the amount of active ingredient applied was comparable. It has proved beneficial to ignore small, insignificant formulation differences, and to concentrate instead on two or three widely different dosage levels of one common formulation.

Standardization by the entomologist of dosages applied, in terms of active ingredient per application, will often avert confusion. Use of two dosage levels has proved satisfactory; one should be the station-recommended dosage, and the second could be considerably higher. The number of applications used in the residue test will be as high as, or higher than, that recommended by the station. Residues obtained by following this plan will be reasonably comparable to those encountered on field crops by most growers—including those who tend to be over enthusiastic in their control programs. Residue data obtained from the higher dosage treatments will influence recommendations to growers for proper time intervals between their last application and harvest. This is merely another safety factor.

Replications. Some replication is mandatory, but the extent should depend on economics and the degree of precision desired in residue analysis. Three field replications are sufficient in most instances. The additional time and effort required to gain slight increases in precision by added replications are seldom justified.

Sampling. A complete topic in itself, sampling will not be discussed in detail here. But the importance of careful, unbiased, and representative sampling by trained personnel cannot be over emphasized. It should always be remembered that residue analyses are expensive, whether one uses specific or nonspecific chemical methods, or even bioassay. In addition, even though a well-trained residue chemist may be employing the most recent methods and equipment available, the final accuracy of the residue determination is largely dependent on the original sample sent to him. If the sample has been haphazardly taken, then all the chemist's time and the institution's money have been wasted; an erroneous result is worse than none at all. The chemist's residue data may be precisely determined but woefully inaccurate because of inadequate field sampling.

To improve sampling, variations in location can often be substituted very efficiently for numerous replications at one location. Thus samples taken from an experiment replicated twice at three locations would almost certainly result in more valid and useful residue data on a given pesticide and crop than if the same experiment were replicated six times at one location.

However, to get maximum information out of such standardized experiments at various locations under a general residue program, one person must serve as coordinator. This coordinator should be given the authority to standardize all variables—pesticide, crop, dosage, formulation, number of replications, sampling procedure, solvent used, extraction time, and temperature of sample storage. He should also have the authority to limit the total number of samples, and the time of their arrival at the laboratory. Without some regulation, a residue laboratory can quickly become swamped with samples, many of which may be absolutely useless to analyze.

Climatological data should be furnished with all residue data, since the paramount effect of temperature and rainfall on the degradation and disappearance of most pesticide compounds is well established. Despite the importance of climatological data they are all too rarely appended to residue data.

Harvest Dates. This phase is more important, especially since the passage of the Miller Bill, than is ordinarily realized. Many residue measurements, although taken from samples that were carefully collected and analyzed, are relatively useless because realistic harvest dates were not established well in advance of actual sampling. The need again is for close cooperation and consultation between the chemist and the entomologist. The most practical harvest sampling dates should be determined by serious consideration of (a) physical and chemical characteristics of the pesticide; (b) physical characteristics of the crop; (c) official or temporary tolerance established; (d) economics (entomologist's and chemist's time and laboratory capacity); (e) limitations of size of field plot; (f) adequacy of storage facilities for samples.

Sample Extraction. Extraction procedures, although usually only superficially mentioned, can be of considerable importance in obtaining a valid sample extract. After the representative (1000-gram) sample has been randomly chosen but accurately weighed from the finely-chopped and well-mixed field sample, it should be combined with the appropriate solvent at a standard ratio. The time of extraction depends on the nature of the crop under study and the solubility of the pesticide residue in the solvent. However the extraction time should be standardized accurately within a given residue experiment. After extraction, care should be taken to standardize on the volume of extractant recovered and saved for eventual analysis.

Breadth. Last, but not least in importance, a residue experiment should be "broad" in design. If possible the experiment should not only answer local needs but should also be comprehensive enough to permit statewide and even national inferences to be projected from the data. Every residue experiment should be carefully designed and carried out with the thought that the results if pertinent could eventually be published. Useful, valid residue data are almost never obtained from haphazardly chosen samples taken from experiments primarily designed for other reasons.

In summary, residue data that can be intelligently interpreted by anyone should include harvest and application dates, dosage and formulations (active ingredient per application), number of applications, field replications and sampling and subsampling details, solvent used, extraction time, sample storage temperature, and duration of storage prior to analysis. In addition, temperature and rainfall data encompassing the period from the first application through the last harvest should accompany all residue data.

If each residue laboratory began to work toward standardization of some of these points, at least within classes of similar crops, better and more efficient results would be made available to all. Moreover, all would save time and money, and interpretations of residue data by the federal regulatory agencies would be considerably simplified.