File: PP# 7F0599

Petitions Control Branch and
Division of Toxicological Evaluation

October 24, 1967

Pesticides Branch, Division of Food
Standards and Additives

AF 25-202
(Diamond Alkali Co.)

PP #7F0599: Method tryout for Daconil 2787.

The petitioner's method entitled, "Determination of Daconil 2787 Residues" was subjected to a tryout on cabbage and potatoes in our Laboratory Investigations Section, PB/FSA, and in the Buffalo District Laboratory. Crops were fortified with Daconil only (i.e., not with its metabolites).

The method calls for the use of either a microcoulometric detector (specific for halogens), or a pulsed mode electron capture detector. For purposes of this tryout the standard concentric type electron capture detector, which is employed routinely at FDA District Laboratories, was used.

In place of the column packing specified by the petitioner (20% DC-11 on 45/60 mesh Chromosorb W), a packing consisting of 10% DC-200 on 80/100 mesh Gas Chrom Q was used. The retention times of Daconil, relative to aldrin, on both columns are approximately the same.

The results of duplicate analysis of cabbage and potatoes are shown below at fortification levels appropriate to the proposed tolerances.

Fortification Level	Percent Recovery	
Potatoes	PB/FSA	BD/FDA
0.1	96	107.3
0.1	104	109.4
0.2	93	117.3
0.2	94	119.6
Cabbage		
5.0	88	107.4
5.0	92	107.6
10.0	96	108.3
10.0	99	108.3

Data submitted by our field laboratory indicates recoveries as being over 100%. The highest recovery values are somewhat suspect since they represent analyses made two weeks after sample extraction (the aged

standard showing an appreciable decrease in response in this time.) Mechanical manipulation such as injecting different volumes of sample and standard, and the use of peaks responding to more than 60% of full scale could also be factors in these high recoveries. Nonetheless, we find the overall recovery range satisfactory.

Interference was not encountered and apparent residues of Daconil were not detected (less than 0.01 ppm) in the controls. Based on the control values and using the electron capture detector, the estimated level of sensitivity is 0.01 ppm. Use of the microcoulometric detector would result in a slightly lower sensitivity of approximately 0.02 ppm. Either sensitivity is adequate. On the basis of the tryout, the method appears to be adequate for the determination of Daconil per se. However, questions regarding the adequacy of this method were raised in the detailed review (B. Malone, DFC, on 8/28/67) of this petition. These are discussed again below in the light of the results of this tryout.

In the absence of data we are unable to form an opinion as to whether or not those compounds identified as metabolites (hydroxytrichloroisophthalonitrile, trichlorodicyanoaniline and 2,4,5-trichloroisophthalonitrile) would have been detected (if present) by the method.

Even though fortification studies validate the extraction procedure for Daconil, the use of dichloromethane (which is immiscible with water) to extract weathered residues of Daconil and metabolites is questionable. The recommended method of extraction for watery crops utilizes a dual solvent system in which one solvent is miscible with water. In addition, the extraction of chlorinated pesticides with dichloromethane is generally not recommended and has been shown to be somewhat inefficient (see Burke and Porter, JAOAC, 49, 1157; 1966).

There is no confirmatory method. If it can be demonstrated, however, that the elution of Daconil (and metabolites) from the alumina column is selective, the combined use of the electron capture and microcoulometric detectors would render the method reasonably specific.

Because of the aforementioned unresolved questions with respect to the extraction of weathered residues of Daconil and metabolites, the detection of the metabolites (if present) by the method and the question of specificity we cannot conclude that the method is adequate for enforcement purposes.

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