PP# 7F0599

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Method Trial for PP #7F0599 (Daconil 2787)

The petitioner's method entitled, "Determination of Daconil 2787 Residues" was evaluated on potatoes and cabbage fortified at the following levels with Daconil 2787:

	Fortification Levels
Potatoes	Zero, 0.1 and 0.2 ppm
Cabbage	Zero, 5.0 and 10.0 ppm

The pesticide was added to the macerated sub-sample prior to extraction with methylene chloride in a Waring Blendor. Aliquots of filtered extract representing 250g of potato or 10g of cabbage were concentrated and cleaned up by column chromatography using Woelm Acidic Alumina (Activity Grade I). Details of the preparation of the column are given in the paper, "Colorimetric Determination of Micro Amounts of Dimethyl Tetrachloroterephthalate," Schuldt, P.H., et. al., Contributions from Boyce Thompson Institute 21, 163 (1961). After transferring the concentrate to the column, possible interfering materials are eluted from the column with 100 mls of methylene chloride and discarded. The column is then eluted with 100 ml of 5% acetone in methylene chloride and the eluate is collected for further analysis.

The petitioner presents two gas chromatographic methods for the determination of Daconil 2787 after cleanup. One method uses a halogen type microcoulometric detector; the other method uses a pulsed mode electron capture detector. We used the standard concentric type electron capture detector in routine use at FDA District Laboratories for our method trial. Sample sizes were selected so that the less sensitive, but more selective, microcoulometric detector could be used if we encountered interferences.

The 5% acetone in methylene chloride eluates were collected in 100 ml volumetric flasks. Before analysis in the gas chromatograph it was necessary to remove the methylene chloride. For the potato samples, 2 ml aliquots (equivalent to 5g potato) were evaporated to dryness and made to 10 ml with benzene. Five ml of the cabbage elutes (equivalent to 0.5 g crop) were evaporated to dryness and made to 50 ml with benzene.

We used somewhat different operating parameters for the gas chromatography than the petitioner since a different type detector was used. A 6' x 6 mm glass column packed with 10% DC-200 on 80/100 mesh Gas Chrom Q was installed in our Barber Colman 5000. Temperatures were set as follows: column oven 198°C; injection port 220°C; detector 210°C. With the carrier gas flow rate at 85 ml nitrogen per minute, Daconil 2787 eluted in 5.85 minutes.

The electrometer was operated at 2×10^{-9} AFS and a 5 mv. recorder was used to record the chromatograms. With the detector voltage set to 50 volts, 0.5 nanograms of Daconil 2787 gave a peak approximately 80% of full scale.

A plot of peak heights versus nanograms of Daconil 2787 was linear in the tested range from 0.1 to 0.5 nanograms. Percent recovery from the fortified samples was calculated on the basis of peak heights found in the sample compared to peak heights of standards analyzed concurrently. All injections of samples and standards were kept constant at 5 µl.

The results of the trial are tabulated below:

ppm Daconil 2787 Added	ppm Daconil 2787 Found	% Recovery
Potatoes		
1. None	<0.01	-
2. None	<0.01	-
3. 0.1	0.096	96%
4. 0.1	0.104	104%
5. 0.2	0.186	93%
6. 0.2	0.188	94%
Cabbage		
1. None	<0.025*	-
2. None	<0.025*	-
3. 5.0	4.4	88%
4. 5.0	4.6	92%
5. 10.0	9.6	96%
6. 10.0	9.9	99%

*Note: This sensitivity was reached by concentrating a portion of the final benzene solution tenfold before analysis.

Comments

1. This is a simple, rapid method which may be used to determine residues of Daconil 2787 at the proposed tolerance levels in potatoes and cabbage. The degree of specificity of the method is dependent on the dlution pattern of Daconil 2787 from alumina and on the retention time in the gas chromatograph.

2. Somewhat greater specificity would be obtained by using a microcoulometric detector instead of an electron capture detector since the latter responds to many compounds which do not contain halogens. Recovery of Daconil 2787 would be expected to be the same with either detector.

3. The retention time for Aldrin was measured under the same conditions used for the gas chromatography of Daconil 2787. Compared to unity for Aldrin, Daconil 2787 had a retention time of 0.52 on the DC-200 column. The response for equal amount of Aldrin and Daconil 2787 was approximately equal. Petitioner reports that for a 6' column packed with DC-11 on 45/60 mesh Chromosorb W the retention time compared to Aldrin is 0.56.

4. In order to determine if Daconil 2787 might be determined by the Mills procedure used by FDA for screening samples for pesticide residues, we attempted to elute Daconil 2787 through Florisil. The compound did not elute in either the 6% ethyl ether in pet. ether or the 15% ethyl ether in pet. ether fractions.

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