

US EPA ARCHIVE DOCUMENT

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
OPP- Pesticide Chemical & Biological Investigations

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SUBJECT: PP# 9F2205 - Results of method trial BAS 352F in or on strawberries

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We were requested by the Chemistry Branch, Hazard Evaluation Division, to conduct a method trial on fungicide BAS 352F in or on strawberries. BAS 352F ([3-(3,5-Dichlorophenyl)]-5-Ethenyl 5-methyl-1,3,oxazolidine-2,4-dione) is a product of BASF Wyandotte Corporation. The BASF analytical method No. 25 dated Nov. 21, 1977, was to be followed. The fortification levels to be run were 0.00, 10.0 and 20.0 ppm, in duplicate.

Method summary:

The petitioner's residue method is based on the determination of 3,5-dichloroaniline. Upon alkaline hydrolysis, the parent and the metabolites degrade to form free 3,5-dichloroaniline which is quantitatively isolated from the hydrolysis mixture by steam distillation and is collected in 1 N sulfuric acid solution. The 3,5-dichloroaniline is extracted from the aqueous distillate by partitioning with chloroform at 2 controlled pH's (first acidic, second basic). The chloroform extracts are dried over sodium sulfate and the 3,5-dichloroaniline is derivatised by using a 4% solution of chloroacetyl chloride in anhydrous chloroform. After derivatisation, the mixture is concentrated and all traces of chloroform and excess chloroacetyl chloride are removed by repetitive additions of hexane and subsequent concentration. The derivative is dissolved in ethyl acetate and measured by GLC equipped with ⁶³Ni electron capture detector. The total residue is expressed in terms of BAS 352F equivalents.

We used a Mikrotek MT 220 GLC equipped with electron capture ⁶³Ni detector and connected to 6' x 4 mm id glass column packed with 3% SP2250 on Supelcoport 80/100 mesh. The GLC conditions were as follows:

Temperatures: Inlet 235°C, Column 200°C, Detector 325°C
Nitrogen Flows: Column 50 ml/min, Purge 50 ml/min

At these parameters the peak for DCAD appeared at the retention time of approximately 5 minutes.

The results:

(sample size 50 g strawberries)

Sample N	BAS 352F Added, ppm	BAS 352F Found, ppm	% Recovery (Uncorrected)
1	none	< .05	---
4	none	< .05	---
2	10 ppm	9.43	94.3%
5	10 ppm	9.96	99.6%
3	20 ppm	14.8	74.0% *
6	20 ppm	18.88	94.4%

* Some foam distilled over in the distillate.

No difficulty was experienced in running the method, except that 200 ml distillate took approximately 2½ hours instead of 90 minutes specified in the method. A set of 3 samples takes approximately 1½ days to analyze.

The method is suitable for regulatory purposes.

Copies to:

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 Petition file *
 Storherr
 MT file (M. Bradley)*
 FDA (Eiduson)
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