



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

JUN 11 1991

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#OG3892. SAN-582H Herbicide in/on Field Corn.
Revised Analytical Method. Sandoz Crop Protection
Submission Dated 3/22/91.

MRID # 418239-02. DEB # 7824. DP Barcode D162965.

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Background

Deficiencies in Sandoz Crop Protection's analytical method for SAN-582H were stated in Conclusion #3 of our 1/24/91 memo as follows:

The analytical method submitted for SAN-582H and its oxalamide metabolite in corn is inadequate for purposes of this temporary tolerance petition. Recoveries and standard deviations are unacceptably high. Independent laboratory validation produced unsatisfactory recoveries at the 0.01 and 0.05 ppm fortification levels, and chromatogram peaks at these levels were not well defined. The petitioner must develop an analytical method for SAN-582H and the oxalamide metabolite which produces acceptable recoveries. The method must then undergo independent laboratory validation.

Use of diazomethane for methylation of oxalamide is not recommended. If a safer reagent cannot be found, documentation must be provided supporting the need for its use.

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Elsewhere in the same memo (page 13) we concluded that for purposes of the temporary tolerance petition, the residue to be regulated in/on corn was parent only. It was felt that for permanent tolerances there were so many questions concerning the nature of the residue that there was no real point in including the oxalamide metabolite (a soil metabolite) in the temporary tolerance expression. We were not sure whether this metabolite was one of the principal metabolites in the SAN-582H residue.

The present submission includes a different analytical method and independent laboratory confirmation.

Conclusion and Recommendation

Sandoz Crop Protection Corporation has submitted a new analytical method for SAN-582H in corn and soybeans. Acceptable recoveries were obtained at the laboratory in Basel. The method has undergone successful independent laboratory validation in the firm's U.S. laboratories. CBTS now recommends that the method be validated for corn and soybeans at EPA laboratories.

Detailed Considerations

Sandoz has submitted the following report:

"Confirmatory Method Trial of the Residue Method 'A Method for the Determination of Residues of SAN-582H in Corn and Soil Samples'," T.R. Bade, 3/22/91, Project No. 414108, Report No. 14. (MRID # 418239-02)

The report consists of a new analytical method developed by Sandoz Basel as well as validation from an independent laboratory, Sandoz Crop Protection Corporation (SCPC) in Des Plaines, Illinois. According to the petitioner, "SCPC did not consult with Basel during the development of this method or at any time before or during this confirmatory trial on any aspect of this analytical method....[therefore] SCPC fits the criteria for an independent laboratory for this method confirmation trial." CBTS agrees that strictly speaking this is true and the method validation is acceptable in principal, but validation by a completely independent laboratory -- one that had no stake in the outcome -- would have been preferable.

Sandoz Basel's method is given as Appendix III of the report. The date of the Sandoz Basel report is 2/25/91.

Corn samples (whole plant, grain, cobs or straw) are extracted with methanol:water (95:5). The extract is cleaned up using a C-18 column, followed by a silica gel column. The final eluate is evaporated to dryness, the residue is dissolved in 3 mL of toluene and the resulting solution used in GC analysis

(capillary column GC with a thermoionic nitrogen specific detector).

Sandoz Basel's validation involved a series of 26 fortifications in four different sample matrices, including soil. Recoveries are given in the following table.

Table 1

Recoveries Obtained by Sandoz Basel

Matrix	Fortification Level	Average % Recovery
Whole Plant	0.01, 0.02, 0.04, 0.6	101±13 (n=8)
Straw	0.02, 0.04, 0.08, 0.6	97±8 (n=7)
Grain, Cob	0.02, 0.04, 0.06, 0.3	97±2 (n=5)
Soil	0.01, 0.02, 0.27	102±8 (n=6)

Submitted chromatograms show well resolved peaks at the minimum fortification levels.

SCPC's validation included both corn and soybeans. Control samples of corn grain and fodder were fortified with SAN-582H at a concentration equal to the proposed tolerance of 0.01 ppm and at five times that tolerance (0.05 ppm). Control samples of soybean grain and forage were fortified at a concentration of 0.1 ppm and at five times that level. No modifications to Sandoz Basel's method were necessary.

Percent recoveries from soybean grain and forage averaged 90.5±1.9% (n=4) and 93.5±0.6 (n=4), respectively. Supporting chromatograms are included. Recoveries from fortified corn grain and fodder were more variable. Percent recoveries from corn grain averaged 100.5±26% (n=4) with a range of 82-140%. The first try with corn fodder yielded low recoveries -- an average of 63.5±18% (n=4) with a range of 40-82%. A second try yielded an average of 94±4% (n=3), but this average does not include a high recovery of 800% which we agree is probably the result of either contamination or an incorrect fortification. Submitted chromatograms show well resolved peaks.

We conclude that Sandoz's method for SAN-582H is now acceptable for EPA method validation.

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