(7-8-97)

MEMORANDUM

Subject: Thifensulfuron Methyl. Product Chemistry, HCB DCI. DuPont Response to Deficiencies in HCB DCI Submission. CBRS 17275. DP D226480. MRID None.

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Dupont Agricultural Products previously submitted a report on the analysis of technical thifensulfuron methyl for hexachlorobenzene and pentachlorobenzene (K. Dockter, CBRS 13423, DP D200743, 09/19/95). Certain deficiencies were noted, and the registrant is now responding to those deficiencies in a letter of 04/24/96 Tony Catka, Product Registration Manager, to Dennis Utterback, SRRD.

Guideline	MRID	Acceptability	Additional Requirements
62-1,-2,-3	43147301 43147302	Fully Acceptable	Submit a CSF that incorporates the findings for HCB and PCB.

Conclusions

DuPont has responded in a satisfactory manner to all substantial deficiencies noted in the report on the analysis of technical thifensulfuron methyl for HCB and PCB, and that study is upgraded to fully acceptable.

Recommendation

CBRS recommends that no additional data be required for fulfillment of the HCB DCI for thifensulfuron methyl. The registrant should submit a CSF that includes upper certified limits for the impurities HCB and PCB.

Detailed Considerations

Deficiency 1:

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A copy of anayltical method NS-37-003 must be supplied.

DuPont response:
The method is now submitted as Attachment 1.

CBRS evaluation:
The method was developed by Lancaster Laboratories. The sample (0.1 g) is dissolved in acetone (10 ml) and is added to sodium borate buffered water (1 l). The solution is extracted with hexane (3 X 60 ml), and the hexane extract is concentrated to 10 ml. The extract is shaken with sulfuric acid, and the hexane layer is stored at -15° C until analysis.

The extract is analyzed by GC (DB-608 30 m X 0.53 mm megabore column; confirmatory column, DB-1701, 30 m X 0.53 mm; ECD). Calibration is by five point external standards, and the calibration data are fitted to a linear calaibration curve.

Quality control samples consist of a reagent blank, a matrix spike sample (MS), and a matrix spike duplicate sample (MSD), each included with a group of 20 or fewer samples. The HCB and PCB contents of the blank must each be <100 ppb. The recoveries from the MS and MSD must be within 20% of the average method validation recoveries. The MS and MSD were run at a fortification level of 200 ppb each. A level of 100 ppb was preferable, but CBRS agreed to the higher level in conversation with Lancaster Laboratories.

The method is fully acceptable. GC/MS confirmation was required only where GC analysis indicated target analytes at concentrations >100 ppb. HCB and PCB were <100 ppb in all samples. The quality control measures meet mimimum requirements.

Deficiency 2:
Sampling plan not detailed. Production represented by the samples not specified.

DuPont response:
The sampling was conducted from 06/14/93 until 07/23/93. During this time [redacted] lbs. of product were produced, or 81 batches. Batch 5 (6/13) was selected as representative of early production. Seven additional batches were sampled, specifically, every tenth batch from batch 20 through batch 80.

CBRS evaluation:
The samples represent a significant segment of production and were collected during the entire production campaign. The samples should provide adequate representation of typical production and variations. The deficiency is resolved.

Deficiency 3:
Before the analysis of samples, the analytical method must be validated for accuracy and precision.

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DuPont response:
The validation data are summarized in Attachment II. Four replicates of thifensulfuron methyl were spiked at 30 - 40 µg/g each with PCB and HCB. PCB average recovery was 131% ± 8.0%, range 123 - 142%. HCB average recovery was 117% ± 4.5%, range 113 - 120%. Based on these results, MDL's of 14 ppb and 5 ppb were calculated for PCB and HCB, respectively. LOQ's of 43 ppb and 16 ppb were calculated for PCB and HCB, respectively.

Accuracy and precision were demonstrated by quadruplicate analyses of thifensulfuron fortified at about 100 ppb each with HCB and PCB. PCB recovery was 111% ± 3.4%, range 108 - 116%. HCB recovery was 107% ± 1.7%, range 104 - 108%. In both cases, the average plus/minus two standard deviations is in the range 70% - 130%. Accuracy and precision are acceptable. The deficiency is resolved.

deficiency 4:
For calibration, the curve must encompass the DCI-mandated LOQ (100 ppb). It cannot be determined if this condition was met.

DuPont response:
The low level standard for both HCB and PCB was 0.5 µg/l. This corresponds to 100 µg/kg, based on a 0.5 g sample and a 10 ml final extract, per the method.

CBRS evaluation:
The deficiency is resolved.

cc: S. Funk, RF, Subject File, Circ., Dennis Utterback (SRRD, 7508).
RDI:F. B. Suhre:07/08/97:
7509C:CBRS:S.Funk:305-5430:CM#2:RM803:SF(0696.3):06/27/96:07/08/97.