Carl Garner/PMSD/ISB
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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

OCT 9 1986

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#4F3007/PP#4F3074. Tilt in Various Commodities. Reevaluation of analytical Methods

FROM: A. Smith
Alfred Smith, Chemist
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

TO: Henry M. Jacoby, PM#21,
Registration Division (TS-767C)

and

Robert Coberly
Toxicology Branch
Hazard Evaluation Division (TS-769C)

THRU: Charles Trichilo, Chief
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

EPA laboratory personnel have audited the Tilt residue methods (Methods AG-356 and AG-359) and have raised questions which could render the methods unacceptable for regulatory purposes (Ronald F. Thomas, ACS, 9/9/86). The questions are discussed below.

1. In both methods a 16-hour reflux with concentrated nitric acid is required. As a result the entire sample assay would require greater than 24 hours for completion. The EPA Residue Chemistry Guidelines indicate that regulatory methods should require a maximum of 24 hours for completion.

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2. For Method AG-356 (PP#4F3007, pecans), the determinative step uses a combination of Gas Chromatography and Mass Spectrometry in the chemical ionization mode. This is a research-type instrument and is not generally available as a routine analytical tool. Consequently, field enforcement laboratories are not likely to have ready access to this instrumentation.

3. For Method AG-359(Tilt and its metabolites in eggs, milk, and meat of livestock, PP#4F3074), the chromatograms do not provide adequate information for residue purposes. The crop background prevents consistently reliable and adequate determinations of the peaks of interest. The peaks are poorly resolved, and constructing a baseline for quantitation is very difficult.

In view of the foregoing discussion RCB, concludes that the methods AG-356 and AG-359 are inadequate for enforcement purposes. The procedures should be modified, or new procedures should be submitted. Adequate validation data should also be submitted to support the new or modified methods. The parent and metabolites should also be run through the FDA multiresidue protocols (see below).

Method trials were requested for Tilt on pecans (PP#4F3007, A. Smith, 6/18/86) and meat, milk, and eggs (PP#4F3074, A. Smith, 6/18/86). In view of the questions raised on the adequacy of the methods, the EPA laboratory (Analytical Chemistry Lab, ACS, Warren Bontoyan/Ronald F. Thomas) should defer action on the method trials until these questions have been resolved.

The petitioner should be informed that the residue methods (AG-356 and AG-359) are not adequate for regulatory purposes due to items 1,2, and 3 above. The methods should be modified, or new methods should be submitted. Adequate validation data should be submitted in support of the new or modified methods.

The petitioner should also be informed that the Residue Chemistry Data Requirements in 40 CFR 158.125(b)(15) require that regulated pesticide residues be subjected to one or more of the multiresidue procedures published in an Addendum to Pesticide Assessment Guidelines Subdivision O - Residue Chemistry Data Requirements for Analytical Methods in 40 CFR Part 158.125, Multiresidue Protocols. To our knowledge, such testing has not been conducted on Tilt residues. Therefore, the following data are required:

Residues of Tilt and its metabolites, including 1,2, 4-triazole, in or on crop samples and meat, milk, and egg samples must be subjected to analysis by the multi-residue protocols.

Protocols for Methods I, II, III, and IV are available from National Technical Information Service under Order No. PB 86 203734/AS.

Note to the PM: For the Convenience of the petitioner, the Federal Register Notice announcing the availability of the FDA multiresidue analysis protocols and actual FDA protocols for Methods I, II, III, and IV are attached, and RCB request RD send these with their response letter.

ATTACHMENT I: Federal Register Notice 6560-50, "Pesticide Assessment Guidelines Subdivision O - Addendum"

ATTACHMENT II: "Multiresidue Method Testing"

cc: Attachment I and II to PM#21

cc: A. Smith, Circu., R.F., PMSD/ISB, TOX, PM#21, PP#4F3007, PP#4F3074, D. Marlow/BUD
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