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

MAY 18 1988

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#8F3606/FAP#8H5553. EPA Reg. No. 7969-58.
Poast[R] Herbicide for Use in or on Citrus Fruits and Blueberries.
MRID Nos. 404926-01 through 404926-05.
RCB Nos. 3436, 3437.
Evaluation of Analytical Method and Residue Data.

FROM: Michael T. Flood, Ph.D., Chemist
Tolerance Petition Section I
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

MTF

TO: Robert J. Taylor/Vickie K. Walters, PM#25
Fungicide-Herbicide Branch
Registration Division (TS-767C)

and

Toxicology Branch
Hazard Evaluation Division

THROUGH: Charles L. Trichilo, Ph.D., Chief
Residue Chemistry Branch
Hazard Evaluation Division (TS-769C)

BASF Corporation Chemical Division requests the establishment of tolerances for residues of the herbicide Sethoxydim {2-[1-(ethoxyimino)butyl]-5-[2-(ethylthio)propyl]-3-hydroxy-2-cyclohexen-1-one) and its metabolites containing the 2-cyclohexen-1-one moiety (calculated as the herbicide) in or on the following raw agricultural commodities (racs):

Citrus Fruits	0.5 ppm
Blueberries	4.0 ppm

and the following animal feeds:

Molasses	1.5 ppm
Dry Pulp	1.5 ppm

The registration of Poast[R] Herbicide, EPA Reg. No. 7969-58, would be amended by this petition.

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Tolerances have been established in or on the following racs under 40 CFR 180.412: alfalfa forage and alfalfa hay (40 ppm); cottonseed (5 ppm): flaxseed (5 ppm); flax straw (2 ppm); peanut hulls (5 ppm); peanuts (25 ppm); soybean hay and soybeans (10 ppm); sugar beets, roots (0.1 ppm); sugar beets, tops (3 ppm); and sunflower seeds (7 ppm). A tolerance of 2.0 ppm has been established under the same section for meat, fat, and meat byproducts of cattle, goats, hogs, horses, poultry and sheep. The tolerances for eggs and milk are 0.5 and 0.05 ppm, respectively. Tolerances are pending on certain other racs, including raspberries and strawberries (both 10 ppm).

Feed additive tolerances have been established under 21 CFR 561.430 for cottonseed soapstock (15 ppm); flaxseed meal (7 ppm); peanut soapstock (75 ppm); sugar beets, molasses (0.05 ppm); and sunflower meal (20 ppm).

There is no Registration Standard on sethoxydim.

Conclusions

1. The nature of the residue in plants and animals is adequately understood. The residue which is regulated consists of parent plus metabolites containing the 2-cyclohexene moiety (calculated as parent).

2. Adequate analytical methods are available for enforcement purposes. The method used for this petition is a minor variant of Method No. 30, which underwent successful EPA method validation for soybeans, milk and liver. Method No. 30 appears in the Pesticide Analytical Manual, Volume II (PAM II) as Method I.

3. Sethoxydim and its metabolites are not recovered using any of the four multiresidue protocols of PAM I.

4. Storage stability data on various racs are sufficient to support the submitted residue analyses. However, there was a time period between extraction of the rac and final analysis. If extracts from fortified samples were stored under the same temperature/time conditions prior to final analysis, no additional work is necessary. Otherwise, the petitioner will have to demonstrate the stability of sethoxydim residues in extracts by appropriate fortification studies.

5a. The proposed tolerance of 4.0 ppm for residues of sethoxydim in/on blueberries is appropriate. This conclusion is provisional pending adequate response to our storage stability question (Conclusion 4).

5b. There are insufficient residue data reflecting the proposed maximum use conditions -- a total of 2.5 lb ai/A/season, PHI = 10 days -- to support the proposed tolerance of 0.5 ppm for residues of sethoxydim in/on citrus. The petitioner should conduct additional field trials in California and Florida for each citrus crop. Poast should be applied at levels reflecting maximum use conditions.

5c. The submitted processing study demonstrates that residues in citrus concentrate threefold when converted to dried pulp or molasses.

6. Processed citrus products are animal feed items. Based on the proposed tolerances for citrus pulp and molasses and results from animal feeding studies, RCB concludes that tolerances on meat, milk, poultry and eggs will not be exceeded from the proposed use. This conclusion is provisional, however, pending submission of acceptable field trial data.

7. An International Residue Limit (IRL) Status Sheet is appended to this review. There are no established IRLs for sethoxydim or its metabolites in or on citrus fruits or blueberries. Therefore, the compatibility of tolerance levels does not arise.

Recommendations

RCB recommends against the establishment of the proposed tolerances for reasons stated in Conclusions 4 and 5b:

4. The petitioner should confirm that extracts from fortified samples were stored under the same temperature/time conditions as were extracts from the field trial samples. If this was not the case, the petitioner must demonstrate the stability of sethoxydim residues in extracts by appropriate fortification studies.

5b. The petitioner should conduct additional field trials in California and Florida for each citrus crop. Poast should be applied at levels reflecting maximum use conditions.

Detailed Considerations

Manufacture and Formulation

The manufacturing process is outlined in PP#0G2396 (E. Zager, memo of 12/4/80). Technical sethoxydim has a minimum

purity of 95%. The major impurities are listed in E. Zager's review. No residue problems with the impurities are anticipated.

Poast^{R} Herbicide is an emulsifiable concentrate containing 20% sethoxydim (1.52 lb ai/gal).

Proposed Use

Poast^{R} Herbicide is a selective broad spectrum postemergence herbicide for control or suppression of annual and perennial grasses. Poast does not control sedges or broadleaf weeds. A nonphytotoxic oil concentrate should always be added to the spray tank.

Citrus. For broadcast application apply 1 1/2 pts - 2 1/2 pts per acre (0.3-0.5 lbs ai/A), depending on the height of the grass. For spot treatment application, apply a 1% - 1 1/2% solution of Poast. Ten gallons per acre (0.25 lbs ai/A) is recommended. Spray gallonage should not exceed 20 gal/A.

Do not apply Poast within 10 days of harvest.

Do not apply more than a total of 12 1/2 pints of Poast per acre (2.5 lbs ai/A) per season.

Apply by ground equipment only.

Blueberries. Application rate is identical to that for citrus.

Do not apply Poast within 30 days of harvest.

Do not apply more than a total of 5 pints of Poast per acre (1.0 lb ai/A) per season.

Apply by ground equipment only.

Nature of the Residue

No new metabolism studies have been submitted in this petition. Studies have been previously submitted and reviewed for soybeans (PP#0G2396, memo of E. Zager, 12/4/80; PP#3F2904, memo of K. Arne, 6/26/85), alfalfa (PP#3F2904, memo of K. Arne, 6/26/85), sugarbeets (PP#3F2950/FAP#3H5413, memo of K. Arne, 2/2/84) and tomatoes (PP#5F3284/FAP#5H5475, memo of C. Deyrup, 10/9/85).

The structure of sethoxydim is shown in Attachment 1.

Metabolism of sethoxydim in plants is complex. The following reactions are known to occur:

1. The sulfur atom is oxidized to the sulfoxide and sulfone. The sulfoxide is frequently the major metabolite (Attachment 1), but its concentration as well as the concentrations of the other metabolites are dependent on PHI.

2. The ring is hydroxylated in the 5-position.

3. The imino group is de-ethoxylated.

4. An oxazole is formed as a result of a Beckman rearrangement.

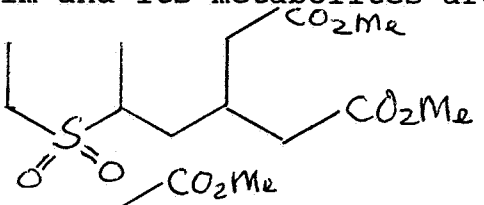
Metabolism studies of sethoxydim in animals have been submitted for rats, lactating goats and poultry (PP#0G2396, memos of E. Zager, 12/4/80, 5/12/81; PP#3F2904, memo of K. Arne, 6/26/85, memo of S. Malak, 12/2/86). Metabolism of sethoxydim in animals is similar to that in plants, but -- at least in goats -- sethoxydim may also be demethylated to form an analogous series of "nor" metabolites (Attachment 1) (PP#3F2904, memo of K. Arne, 6/26/85).

The nature of the residue in plants and animals is adequately understood. The residue which is regulated consists of parent plus metabolites containing the 2-cyclohexene moiety (calculated as parent).

Analytical Methods

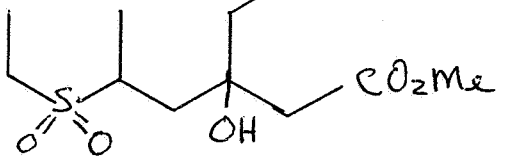
Residues in or on citrus fruits and blueberries were analyzed by BASF Agricultural Chemicals Method No. 30G, January, 1985. The method is an addendum to Method No. 30 entitled "Determination of BAS 9052 H and Its Metabolite Residues in Alfalfa Forage and Soybean Forage", originally submitted in PP#3F2904. The authors are R.C. Paulick, L.A. Sears and E.J. Panek. No claim of confidentiality has been made for this method.

Sethoxydim and its metabolites are oxidized to



"DME"

or to



"DME-OH" (from the 5-OH-metabolite)

DME and/or DME-OH are analyzed on a gas chromatograph equippped with a sulfur specific flame-photometric detector (FPD).

Samples are extracted directly with methanol. After a calcium hydroxide precipitation step, the aqueous methanol extracts are concentrated and oxidized with basic hydrogen peroxide. The oxidation products are then esterified (H^+, MeOH) and purified by silica gel chromatography prior to analysis by GC.

Blueberries were analyzed by unmodified Method 30G. Specific modifications were made for citrus fruits and processed oranges:

1. Citrus Fruits. Barium hydroxide was added to the extract before oxidation with hydrogen peroxide in order to maintain basic conditions. Silica gel chromatography was followed by solid phase extraction (C_{18} column).

2. Orange Process Fractions. The "wet" process fractions (unwashed and washed fruit, juice, prewash and afterwash rinse, oil emulsion water, chopped peel, peel fruits, finisher pulp, press liquor and molasses) were extracted directly with methanol, as above. Orange oil samples were dissolved in hexane and extracted with acetonitrile. Dried peel was subjected to an initial aqueous soaking procedure prior to methanol extraction.

   All matrices except the dried peel required C_{18} column purification. A Florisil column was used for the dried peel samples. For press liquor and molasses, additional purification was achieved with an alumina column prior to the C_{18} column.

Reported limits of quantitation are 0.05 ppm for sethoxydim residues in/on blueberries and citrus fruits and 0.10 ppm for residues in most of the orange process fractions (0.05 ppm for prewash and afterwash rinse and the oil emulsion water).

When fortified with 0.05, 0.5, 1.0 10.0 and 20.0 ppm sethoxydim, recoveries from blueberries averaged 91±16%. Recoveries of MSO and 5-OH-MSO_2 (Attachment 1) at the same fortification levels averaged 92±14 and 83±10%, respectively. Recoveries for citrus are given in the following table. Fortification levels were 0.05, 0.5, 1.0 and 5.0 ppm.

Table I
Recoveries from Citrus

	Sethoxydim	MSO	5-OH-MSO_2
Oranges	95±9	87±8	85±9
Lemons	92±9	90±8	85±13
Grapefruit	88±10	91±5	78±7

Various orange process fractions were fortified with MSO and 5-OH-MSO_2 at 0.50 ppm and either 0.05 or 0.10 ppm (depending on the quantitation limit for the particular matrix). Percent recoveries varied from 76-110% for MSO and 72-110% for 5-OH-MSO_2.

The basic analytical method has been tested against known residues obtained by radioactive counting of sethoxydim/metabolites in or on soybean forage, hay and seed and alfalfa hay. Methanol or methanol/water extracts 63-93% of the total radioactivity. The lower recoveries were obtained using weathered samples (PP#3F2904, memo of K. Arne, 6/26/85). For weathered samples the analytical method will yield low values of the total toxic residue even when the residue is corrected for recoveries obtained in the fortification studies.

BASF's Analytical Method No. 30, which differs from 30G in that a dichloromethane partition follows silica gel purification, underwent a successful method trial for soybeans, milk and liver (PP#2F2670, K.F. Kissler, memo of 4/1/83). Method 30 appears as Method I in PAM II. EPA method validation has been requested for the same method with the "nor" metabolites of sethoxydim (PP#3F2904, memo of S. Malak, 8/15/86). In the meantime, the petitioner has obtained acceptable recoveries in analyses of these metabolites.

Sethoxydim and its metabolites were analyzed using the four FDA multiresidue protocols of PAM I (PP#6F3452, memo of M. Nelson, 9/22/87). The petitioner was unable to achieve acceptable recoveries using these methods and claims that the labile nature of these compounds makes them very difficult to analyze without any derivatization procedures.

Residue Data

Storage Stability. Storage stability data are available for soybeans (PP#3F2904 -- 15°C, 24 months), soybean forage (PP#3F2904 -- 15°C, 27 months), tomatoes (PP#5F3284, "frozen", 15 months), strawberries (PP#6F3383, frozen 31 months) and potatoes (PP#3529, frozen, 26 months). The storage stability study on soybean forage has been extended to 53 months (PP#8F3577). The storage stability study on strawberries has been extended to 60

months. Recoveries of DME and DME-OH at 60 months were 89% and 74%, respectively.

A storage stability study on oranges is reported in this petition in a footnote. Details of the study are said to appear in BASF Notebook No. 92, which is not available to us. Orange samples were fortified 11/8/84 with 1.0 ppm each of MSO and 5-OH-MSO_2 and were then stored at <-5°C until analysis. Results are given in Table 2. Recoveries at "0-days" indicate the efficiency of the method for that day.

Table 2
Storage Stability of Sethoxydim and Its Metabolites in Oranges

	Analysis Date	Storage Period (months)	Sethoxydim Equivalents DME(ppm)	DME-OH(ppm)
0-day	12/16/86	0	0.82	0.76
11/8/84	12/16/86	25	0.82	0.63
0-day	8/5/87	0	0.75	0.92
11/8/84	8/5/87	31	0.78	0.80

RCB concludes that sethoxydim residues are stable in/on oranges at temperatures <-5°C for periods up to 31 months. We note, however, that because many of the citrus samples analyzed for this petition were stored for times greater than 31 months, this stability study alone is not enough to support the residue data on citrus.

Residue data are submitted in five reports:

Zehr, R.D., Magnitude of the Residue of Sethoxydim and Metabolites in Blueberries, BASF Document No. 87/5084, February, 1987. (MRID No. 404926-04)

Single, Y.H., Magnitude of the Residue of Sethoxydim and Metabolites in Oranges, BASF Document No. 87/5072, June, 1987. (MRID No. 404926-01)

Eswein, R.P., Magnitude of the Residue of Sethoxydim and Metabolites in Lemons, BASF Document No. 87/5071, June, 1987. (MRID No. 404926-02)

Eswein, R.P., Magnitude of the Residue of Sethoxydim and Metabolites in Grapefruit, BASF Document No. 87/5070, June, 1987. (MRID No. 404926-03)

Single, Y.H., Magnitude of the Residue of Sethoxydim and Metabolites in Orange Process Fractions, BASF document No. 87/5078, July, 1987. (MRID No. 404926-05)

Blueberries. Twenty field trials were conducted in nine states: Connecticut, Georgia, Illinois, Maine, Michigan, Minnesota, North Carolina, New Jersey and Washington. According to the Foods and Food Production Encyclopedia, 1982, these states account for most of the blueberry production in this country.

Samples were frozen after harvest and shipped to BASF Corporation Chemicals Division in Fairfield, NJ (before 6/1/86) or Research Triangle Park, NC (after 6/1/86). Upon arrival at the analytical laboratories, stems and other foreign matter were removed and the frozen blueberries were finally ground. Samples were stored frozen (<-5°C) until analysis. A maximum of 30 months elapsed between harvest and analysis of the samples. The residue data are supported by the storage stability study on strawberries. Analyses were done at Morse Laboratories in California and at Nippon Soda Co., Ltd. in Japan.

From examination of sample histories and the dates on the chromatograms, it appears that sample extraction occurred up to four weeks before final chromatographic analysis. The petitioner must demonstrate that sethoxydim residues are stable in extracts for the temperature/time conditions prior to final analysis.

Results are shown in the following table. Residue values have not been corrected for recoveries.

Table 3
Sethoxydim Residues in/on Blueberries

Test Site	Application Rate (lb ai/A)	PHI	Sethoxydim Equivalents DME(ppm)	DME-OH)ppm)
E. Lansing, MI	2 x 0.5	30	0.06-0.08	<0.05
Kalamazoo, MI	2 x 0.5	32	<0.05-0.08	<0.05
Haslett, MI	2 x 0.5	30	<0.05	<0.05
Mays Landing, NJ	2 x 0.5 3 x 0.5	31	0.76-0.88 1.1 -1.5	0.12-0.14 0.18-0.38
Mays Landing, NJ	2 x 0.5 3 x 0.5	32	1.2 -2.4 3.0 -3.2	0.26-0.60 0.59
Castle Hayne, NC	2 x 0.5	33	<0.05-0.11	<0.05
Castle Hayne, NC	2 x 0.5	33	<0.05	<0.05

Deblois, ME*	2 x 0.5	29	2.2 -2.4	0.36-0.42
	3 x 0.5		4.8 -6.3	0.50-0.57
Deblois, ME*	2 x 0.5	29	2.2 -3.0	0.33-0.41
	3 x 0.5		4.8 -5.3	0.22-0.40
Deblois, ME*	2 x 0.5	29	1.3 -1.8	0.20-0.29
	3 x 0.5		4.0 -5.0	0.32-0.35
Blairsville, GA	2 x 0.5	30	<0.05-0.11	<0.05
Blairsville, GA	2 x 0.5	30	0.23-0.28	<0.05
Brush Prairie, WA	2 x 0.5	30	0.65	0.13
	3 x 0.5		1.2	0.67
Simpson, IL	2 x 0.5	31	<0.05	<0.05
		60	0.06	<0.05
Windsor, CT*	1 x 0.5	15	0.43	<0.05
	1 x 1.5	15	1.0	0.08
Montrose, MI	2 x 0.5	14	0.08	<0.05
Windsor, CT	2 x 0.5	39	<0.05	<0.05
Becker, MN**	2 x 0.5	22	2.1 -3.7	0.26-0.29
Jonesboro, ME*	0.5 + 0.3	31	1.9 -2.6	0.24-0.49
Windsor, CT	0.5 + 0.3	31	0.11-0.16	<0.05-0.05
	1.0	50	<0.05-0.14	<0.05

* Lowbush variety. ** Hybrid of highbush and lowbush varieties.

The petitioner has excluded the fourth sample, having a total of 14.4 ppm, from the Minnesota field trial because the residue was atypical when compared to samples from the same trial as well as to all other trials. Replicate analyses of the same sample indicate that the actual analyses were not in error. The petitioner states that data from the cooperator conducting the trial indicated a heavy infestation of tall weeds during spraying for the fourth sample of the trial. The weeds were tall enough to interfere with the spray boom, causing uneven herbicide application.

The data show that the highest residues are found in the lowbush varieties. The petitioner reports that in at least one case (WA field trial) the entire bush was sprayed with herbicide.

Discounting the high value, the data support a tolerance of 4.0 ppm.

Citrus

Oranges. Nineteen field trials were conducted in California, Florida and Texas. According to Agricultural Statistics, 1985, in 1983 these three states accounted for over 98% of the U.S. orange production.

Samples were frozen after harvest and shipped to BASF Corporation Chemicals Division in New Jersey where they were stored frozen. Extraction for analysis occurred 15-49 months after sampling. Judging from the dates on the submitted chromatograms, actual analysis occurred two weeks after extraction.

As stated above, storage stability data on oranges extend to only 31 months. These data would support only about one quarter of the analyses. However, in conjunction with the other storage stability studies on strawberries and peas, RCB can conclude that no major storage stability problem is expected. However, the petitioner must demonstrate that sethoxydim residues are stable in extracts over the time period between extraction and analysis. Storage stability is discussed further in the "Summary and Conclusions for Citrus" section in this memo.

Residue data are summarized in the following table. Values have not been corrected for recoveries.

Table 4
Sethoxydim Residues in/on Oranges

Test Site	Application Rate (lb ai/A)	PHI	Sethoxydim Equivalents DME (ppm)	DME-OH (ppm)
Reedley, CA	4 x 0.5	3	<0.05	<0.05
Reedley, CA	1 x 0.5	3	<0.05	<0.05
Reedley, CA	5 x 0.5	14	<0.05	<0.05
Riverside, CA	4 x 0.5	15	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	6 x 0.5	38	<0.05	<0.05
Bradenton, FL	5 x 0.5	28	<0.05	<0.05
Palmetto, FL	1 x 0.5	14	<0.05	<0.05
	6 x 0.5	38	<0.05	<0.05
Bradenton, FL	1 x 0.5	14	<0.05	<0.05
	6 x 0.5	38	<0.05	<0.05
Weslaco, TX	1 x 0.5	20	<0.05	<0.05
	6 x 0.5	21	<0.05	<0.05
Weslaco, TX	5 x 0.5	35	<0.05	<0.05
Bradenton, FL	6 x 0.5	38	<0.05	<0.05
Weslaco, TX	5 x 0.5	10	<0.05-0.09	<0.05-0.06
	5 x 1.0		<0.05-0.46	<0.05-0.14
Dinuba, CA	5 x 0.5	14	<0.05	<0.05
	5 x 1.0		<0.05-0.25	<0.05
Fort Pierce, FL	6 x 0.5	25	<0.05	<0.05
	6 x 1.0		<0.05-0.06	<0.05
Lake Alfred, FL	5 x 0.5	11	<0.05	<0.05
	5 x 2.0		0.11-0.12	0.08-0.09
Lake Alfred, FL	6 x 0.5	11	<0.05	<0.05
	6 x 2.0		0.14-0.21	0.09-0.12

In most of the trials, sethoxydim residues were not found at the quantitation limit. In these trials the herbicide was

directed toward the grasses beneath the trees. Samples represented composites from the entire tree. According to the petitioner, in other trials both the application and sampling methods were modified to produce samples containing measurable residues. Samples with finite residues were needed for the processing study (discussed below), and in other trials applications were made to simulate spray drift. In these trials spray was directed toward the lower limbs, and samples from various portions of the tree were analyzed in addition to a composite.

The nineteen "field trials" are more accurately nineteen experiments in ten field trials. The six trials at Bradenton, FL, for example, are really two field trials. For each experiment within a field trial, pesticide was applied at the same time at the same site. For example, in the third and fourth Bradenton "trials" listed in Table 4, the respective application dates were 3/31, 5/28, 7/21, 9/18, 11/10/83, 1/5/84 and 3/31, 5/28, 7/21, 9/18, 11/10/83. The only difference is that an additional application was made in the third experiment.

Grapefruit. Fifteen field trials were conducted in three states: California, Florida and Texas. According to Agricultural Statistics, 1985, these three states produced over 95% of the grapefruit produced in the U.S. during the 1982-83 growing season.

Samples were frozen after harvest and shipped to BASF Corp. Chemicals Division in New Jersey. After grinding, aliquots of the samples were removed and shipped in dry ice to Nippon Soda Co., Ltd. in Japan for analysis, which occurred up to 42 months after sampling. Data from only six of the field trials were obtained under time conditions that could be supported by the stablity study on oranges. Our other comments are identical to those for oranges.

Results are summarized in Table 5. Residue values are uncorrected for recoveries.

Table 5
Sethoxydim Residues in or on Grapefruit

Test Site	Application Rate (lb ai/A)	PHI (days)	Sethoxydim Equivalents DME	DME-OH
Pailier, CA	4 x 0.5	3	<0.05	<0.05
Reedley, CA	4 x 0.5	3	<0.05	<0.05
Reedley, CA	5 x 0.5	14	<0.05	<0.05
Riverside, CA	4 x 0.5	15	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Bradenton, FL	4 x 0.5	53	<0.05	<0.05
Weslaco, TX	5 x 0.5	34	<0.05	<0.05
Weslaco, TX	5 x 0.5	35	<0.05	<0.05
Fort Pierce, FL	5 x 0.5	11	<0.05-0.18	<0.05-0.11
Weslaco, TX	5 x 0.5	10	<0.05-0.49	<0.05-0.11
Dinuba, CA	5 x 0.5 5 x 1.0	13	<0.05 <0.05	<0.05 <0.05

The six "field trials" in Bradenton (on three varieties) are more accurately described as six experiments in one trial, for dates of application are identical. (The two experiments on oranges having PHI 53 held in Bradenton were from the same trial also.) Likewise, the first two Weslaco trials in Table 5 were really two experiments in one trial. Therefore, threr were really nine field trials conducted.

The field trial data from the Fort Pierce, FL trial are notable in that there was no indication that the herbicide was sprayed directly on the canopy. This was the only citrus field trial in which detectable residue was observed when label instructions were followed.

Lemons. Seven field trials were conducted in two states: Arizona and California. According to Agricultural Statistics, 1985, these two states account for virtually 100% of the U.S. lemon production.

Samples were frozen after harvest and were maintained in a frozen state until analysis. Lemon samples were shipped to BASF Corporation Chemicals Division in New Jersey, where they were ground. Aliquots of samples were removed and shipped in dry ice to Nippon Soda Co., Ltd. for analysis. Analysis occurred up to 42 months after sampling. Data from the storage stability study on oranges would support the residue data from five of the seven field trials. Otherwise, our comments are identical to those for oranges, above.

Residue data for lemons are summarized in Table 6:

Table 6
Sethoxydim Residues in or on Lemons

Test Site	Application Rate (lb ai/A)	PHI (days)	Sethoxydim Equivalent DME	DME-OH
Parlier, CA	4 x 0.5	3	<0.05	<0.05
Reedley, CA	4 x 0.5	3	<0.05	<0.05
Reedley, CA	5 x 0.5	14	<0.05	<0.05
Riverside, CA	4 x 0.5	15	<0.05	<0.05
Phoenix, AZ	6 x 0.5	10	<0.05	<0.05
	6 x 1.0		<0.05	<0.05
Fresno, CA	3 x 0.5	12	<0.05-0.28	<0.05
	3 x 1.0		<0.05-1.0	<0.05
Dinuba, CA	5 X 0.5	13	<0.05	<0.05
	5 X 1.0		<0.05	<0.05

The only samples showing residues above the limit of quantitation were from trees receiving direct canopy applications of herbicide to establish the utility of the method for detecting spray drift. No residues were detected, even with exaggerated doses when Poast was applied to grasses beneath the trees.

Orange Processing Study. Orange samples from one of the Lake Alfred, FL field trials (5 x 2.0 lbs ai/A) were processed according to commercial standards. After processing, all of the fraction samples were frozen and shipped in dry ice to BASF Corporation Chemicals Division in New Jersey. Upon arrival, samples were homogenized and stored frozen until analysis. A

maximum of 18 months elapsed between harvest and final analysis of the process fractions. The storage stability study on oranges would support the analyses under these conditions.

Oranges were processed to juice, dry peel, oil and molasses. (A description of the process is given on pp. 7-8 of the report.) Results are given in the following table:

Table 7
Sethoxydim Residues on Orange Process Fractions

	Sethoxydim Equivalents	
	DME	DME-OH
Washed Fruit	0.12	<0.10
Juice	<0.10	<0.10
Orange Oil	<0.10	<0.10
Chopped Peel	0.12	<0.10
Peel Frits	0.10	<0.10
Finisher Pulp	<0.10	<0.10
Dried Peel	0.36	0.24
Press Liquor	<0.10	<0.10
Molasses	0.34	0.17

Examination of DME results indicates about threefold concentration from fruit to dried peel or molasses. DME-OH levels in fruit were below the quantitation limit, so it is impossible to determine its concentration from analysis alone. It is reasonable to assume threefold concentration also.

Summary and Conclusion for Citrus

Although many of the residue data are not covered by the 31 month storage stability study on oranges, RCB concludes that the 53 month study on soybean forage, the 52 month study on peas and the 60 month study on strawberries are sufficient to guarantee that the submitted residue data are accurate. There is a question concerning the stability of sethoxydim residues in extracts before analysis. If extracts from fortified samples were stored under the same temperature/time conditions prior to final analysis, no additional work is necessary. Otherwise, the petitioner will have to demonstrate the stability of sethoxydim residues in extracts under these conditions by appropriate fortification studies.

Overall, when Poast was applied according to the label instructions, no detectable residue was observed in citrus except in one trial on grapefruit. However, although the number of field trials would be sufficient to set a tolerance on citrus -- there were a total of 26 actual field trials by our count --the number of trials in which Poast was applied at levels and PHI corresponding to the proposed maximum use conditions is quite small. Because detectable residues were found in one of the grapefruit trials which did correspond to the proposed maximum use conditions, the petitioner should conduct additional field trials in Florida and California for each citrus crop. A seasonal total of 2.5 lb ai/A should be applied, and the PHI should be ten days.

The orange processing study is acceptable. There is a threefold concentration of residues when citrus fruits are processed to dried pulp or molasses.

Meat, Milk, Poultry and Eggs

According to our Residue Chemistry guidelines, citrus pulp, dehydrated can constitute 33% of the diet of beef and dairy cattle and 1% in the diet of swine; citrus pulp, wet can constitute 20% in the diet of beef cattle, 10% in the diet of dairy cattle and 10% in the diet of swine; and citrus molasses can constitute 15% in the diet of beef cattle and 10% in the diet of dairy cattle.

Results of feeding studies have been discussed in PP#3F2904 (memo of K. Arne, 6/26/85; memo of S. Malak, 12/2/86).

Goats were dosed with the most prominant plant metabolite of sethoxydim, MSO, at levels up to 300 ppm in the diet for 28 days. A fourth group was fed hay carrying weathered sethoxydim residues at 75 ppm in the diet. Residue levels are summarized in the following table:

Table 8
Sethoxydim Residues (ppm) in Goat Milk and Tissues

Feeding Level	Milk	Fat	Kidney	Liver	Muscle
25 ppm	<0.01-0.03	<0.05	<0.05	<0.05	<0.05
100	<0.01-0.07	<0.05	<0.31	<0.05	<0.05
300	<0.01-0.11	0.07	<0.45	0.17	<0.05
75	<0.01-0.18	<0.05	<0.44	0.14	<0.05

In an earlier study cows were dosed with sethoxydim at 50

ppm in the diet for 30 days. Results are given in Table 9, below.

Table 9
Sethoxydim Residues (ppm) in Cow Milk and Tissues

Feeding Level	Milk	Kidney	Liver	Muscle
50 ppm	<0.05-0.06	<0.15-0.16	<0.15-0.20	<0.03

Laying hens were dosed with up to 250 ppm MSO in the diet for 29 days. Residues in eggs and tissues are given in Table 10

Table 10
Sethoxydim Residues (ppm) in Eggs and Tissues

Feeding Level	Eggs	Fat	Kidney	Liver	Muscle	Skin
25 ppm	0.31-1.08	<0.05	0.75	0.40	0.06	0.10
80	1.04-3.9	0.05	1.04	0.42	0.10	0.23
250	2.15-11.9	0.17	1.46	1.26	0.21	0.60

In PP#3F2904 the reviewer estimated that, based on existing or pending tolerances, the maximum concentration in the diet of sethoxydim residues would be 27.25 ppm for cattle and 12.25 ppm for poultry (S. Malak, memo of 12/2/86). It was concluded that tolerances on meat, milk, poultry and eggs would not be exceeded by these levels of sethoxydim residues. Because the maximum possible contribution to the diet of sethoxydim residues due to citrus feed items is lower than that due to any of the feed items used to calculate the above diet residues, we can conclude that tolerances in meat, milk, poultry and eggs will not be exceeded due to the proposed tolerances. This conclusion is provisional pending evaluation of data from additional field trials.

Other Considerations.

An International Residue Limit (IRL) Staus Sheet is appended to this review. No IRL has been established for residues of sethoxydim/metabolites in or on any of the racs of this petition. Therefore, compatibility of tolerance levels is not an issue.

Attachments: two

Attachment 1: Structures of Sethoxydim and Three of Its Typical Metabolites.

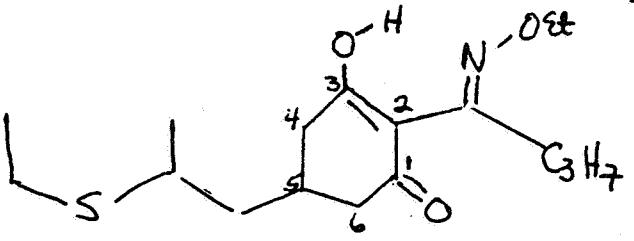
Attachment 2: International Residue Limit Status Sheet.

cc: Reviewer(Mike Flood), RF, Circu., TOX, PM#25, PP#8F3606/FAP#8H555
ISB/PMSD(Eldredge).

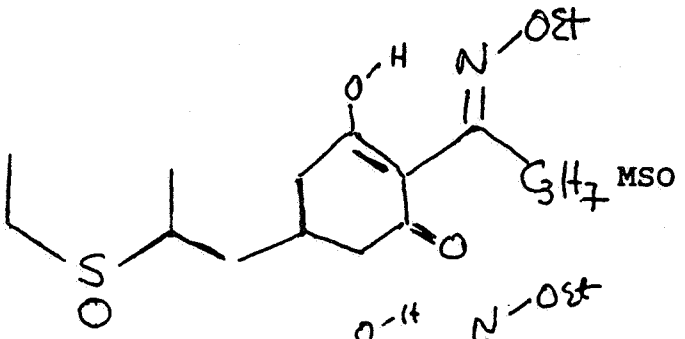
TS-769C:RCB:Reviewer(MTF):CM#2:Rm810:557-4362:typist(mtf):5/18/88.
RDI:SectionHead:RSQuick:5/17/88:DeputyChief:RDSchmitt:5/17/88.

Attachment 1

Structures of Sethoxydim and Three of Its Typical Metabolites



SETHOXYDIM



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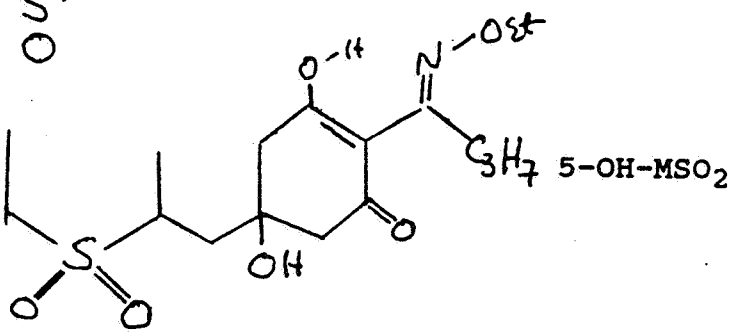
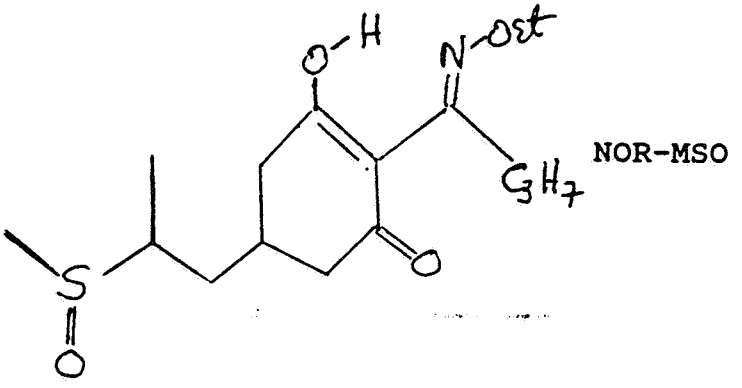


5-OH-MSO$_2$



NOR-MSO

Attachment 2

F. Kover 4/1/88

INTERNATIONAL RESIDUE LIMIT STATUS

CHEMICAL Sethoxydim

CODEX NO. _____

CODEX STATUS:

☑ No Codex Proposal Step 6 or above ~~(on these commodities)~~

Residue(if Step 8): _____

Crop(s)	Limit (mg/kg)

PROPOSED U.S. TOLERANCES:

Petition No. 8F3606/8H5553

RCB Reviewer FLOOD

Residue: Sethoxydim and its metabolites containing the 2-~~ecto~~cyclohexen-1-one moiety

Crop(s)	Limit (mg/kg)
Blueberries	4.0
Citrus Fruits	0.5
Animal Feeds	
Citrus Molasses	1.5
Dry Pulps	1.5

CANADIAN LIMITS:

☑ No Canadian limit (On these commodities)

Residue: As per proposed U.S.

Crop(s)	Limit (mg/kg)

MEXICAN LIMITS:

☑ No Mexican limit

Residue: _____

Crop(s)	Limit (mg/kg)

NOTES: