PP# 0F2277: Naphthaleneacetic Acid on Apples, Pears, and Olives. Evaluation of analytical method and residue data.

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The Union Carbide Agricultural Products Company, Inc. proposes that the established tolerances (§180.155) for residues of the plant regulator α-naphthaleneacetic acid, or 1-naphthaleneacetic acid (NAA), be amended to reflect the use of the free acid or the ethyl ester forms as follows.

1.0 ppm in or on apples and pears from the application of α-naphthaleneacetic acid or the ethyl ester of α-naphthaleneacetic acid.

0.1 ppm in or on olives from the application of α-naphthaleneacetic acid or the ethyl ester of α-naphthaleneacetic acid.

This proposal includes no changes in the established tolerance levels for apples, pears, or olives.

Tolerances for α-naphthaleneacetic acid are also established in quinces at 1.0 ppm and in pineapples at 0.05 ppm (§180.155). A tolerance is pending for NAA residues in citrus (PP# 7E1956).

The proposed uses on apples, pears and olives reflect application when no fruit are present whereas the already registered uses are for application when fruits are present.

Conclusions

1. The nature of the residue is adequately understood.

2. The analytical method is adequate for the determination of NAA residues.

3. No real residues are expected in olives, apples, pears, or their byproducts. Therefore, the established tolerances for apples, olives, and pears will not be affected by the proposed uses.

4. No residues are likely to occur in eggs, milk, and meat of livestock § 180.6(a)(3).

Recommendation

Toxicological and FFE considerations permitting, we recommend for the proposed tolerance amendments.
Detailed Considerations

Formulation

AMCHEM Sprout Inhibitor A112, a liquid mixture containing the ethyl ester of 1-naphthaleneacetic acid at 15.1% (1-naphthaleneacetic acid equivalent of 1.0 lb/gal. or 13.2% by weight), is proposed for use on apples, pears, and olive trees to control sprout or sucker growth.

The formulation is made from

The impurities are not likely to be a residue problem.

All inert ingredients are cleared for use under 3780.1001.

Proposed Use

Apples and Pears

Formulation is to be applied during the dormant period before resprouting or when new shoots are 6-12 inches long. On bearing trees do not treat suckers during bud swell, bloom, or fruit set (period from start of growth to 4 weeks after petal fall). Do not repeat applications on same trees for at least one year.

The solution is applied once at a rate of 8.7 lb act/100 gallons.

Olives

Do not treat scaffold branches or pruning cuts on olives. Treat once when sprouts are no more than 10 inches tall at the above rate. Do not treat during olive bloom or fruit set.

The foliage of trees (olive, apples, pears) is not to be sprayed.

Nature of the Residue

The metabolism of 1-naphthaleneacetic acid (NAA) in plants (citrus, olives, Coleus, wheat, peas, and various weed varieties) has been fully discussed in previous reviews (PP# 7E1956, PP# 1E1099). Plant residues consist primarily of the parent, NAA, and the conjugated NAA components 1-naphthaleneacetyl aspartic acid and 1-B-D-Glucose–naphthaleneacetate. The plant metabolism of NAA is adequately understood.

Rats were administered single doses of radiolabelled C-14-NAA, and 37-97% of the doses were excreted in the urine and feces within 72 hours. The major portion of the residue consisted of esters of NAA with glycine, (naphthaceturic acid), and glucuronic acid, (naphthamethyl-gluconisduroonic acid). Small amounts of unchanged NAA and two unidentified metabolites were also noted.
Because of the proposed use (no application to foliage of trees; no treatment during bloom or fruit set), no residues of NAA are likely to occur in the fruit of treated trees.

Analytical Method

A ground sample is extracted by blending with methanol and filtering. The filtrate is mixed with a buffer solution and partitioned with hexane. The aqueous phase contains the NAA and the hexane phase contains the ethyl ester of NAA.

The aqueous phase containing NAA is washed with methylene chloride which is discarded. The aqueous phase is acidified, and NAA is extracted into methylene chloride which is evaporated to dryness. The residue is taken up with methanol which is concentrated and digested with methanolic perchloric acid. (This converts NAA to methyl NAA).

The residue is extracted into hexane, cleaned up on a Florisil column, and eluted with an ethyl ether: hexane mixture. The eluate is evaporated to dryness, and the residue is taken up with methanol. This solution contains any NAA (as methyl NAA) which may have been present in the sample.

For ethyl NAA analysis, the initial hexane extract (see paragraph one above) is evaporated to dryness, and the residue is cleaned up on a Florisil column. The residue is eluted with an ethyl ether: hexane mixture which is evaporated to dryness. The residues of ethyl NAA are taken up with methanol for determination.

The NAA and ethyl NAA solutions are each examined by liquid chromatography using an ultra-violet detector.

Untreated (control) samples of olives, apples, and pears had no NAA (<0.01 ppm) or ethyl NAA (<0.02 ppm) equivalent residues. Control samples of olives, apples, and pears were fortified with NAA, ethyl NAA, and methyl NAA at levels of 0.1 and 0.2 ppm. Recoveries were 70-106%.

The method is adequate for the determination of NAA and ethyl NAA residues in olives, apples, and pears.

Residue Data

Olives

Samples of fresh or brined olives had no detectable residues of NAA (<0.01 ppm) or ethyl NAA. The fresh samples were obtained from trees in California which had been treated as proposed. The samples were collected at intervals of 39-52 days after treatment.

Pears

Samples were obtained from trees in California which had been treated as proposed and harvested at intervals of 93 days after treatment. No detectable residues (<0.01 ppm) of NAA or ethyl NAA were noted.
Apples

Samples were obtained from crops in Michigan, New York, Oregon, Indiana, Minnesota, Pennsylvania, and Canada which had been treated as proposed. The samples showed no detectable residues of NAA (< 0.01 ppm) or ethyl NAA (< 0.02 ppm) at periods of 10-212 days after treatment.

The data indicate that no detectable residues (< 0.02 ppm) are likely to occur in olives, apples, and pears from the proposed use. These data support our conclusion (see Nature of the Residue) that no real residues are expected in olives, apples, and pears from the proposed uses. Further, the absence of residues in apples precludes the presence of residues in the livestock feed item, apple pomace. Also, no residues are expected in the juices, jellies, and canned fruits.

We conclude that the established tolerances for apples, olives, and pears are adequate to cover residues of NAA resulting from the proposed and registered uses.

Meat and Milk

Apple pomace may be used as a livestock feed. We have indicated that no residues are expected in apple pomace from the proposed use. Therefore, no residues are expected in eggs, milk, and meat of livestock (§180.6(a)(3)).