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OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Methyl Bromide Registration Standard Follow-up.
Protocols for Postharvest Fumigation of Dried
Fruits and Nuts. (EPA ID No. 53201-1, Record No.
251192, DEB No. 5774)

FROM: Nancy Dodd, Chemist *Nancy Dodd*
Tolerance Petition Section II
Dietary Exposure Branch
Health Effects Division (H7509C)

THRU: Debra Edwards, Ph.D., Chief *Debra Edwards*
Tolerance Petition Section II
Dietary Exposure Branch
Health Effects Division (H7509C)

TO: Walter Francis, Acting PM 32
Antimicrobial Program Branch
Registration Division (H7505C)

and

Lois Rossi, Chief
Reregistration Branch
Special Review and Reregistration Division (H7508C)

Background

The Methyl Bromide Industry Panel (MBIP) submits protocols for postharvest fumigation of raisins, prunes, dates, figs, walnuts, almonds, pistachios, pecans, and bridging data on cut fruits, hazelnuts, macadamias, and Brazil nuts.

Previous DEB reviews of postharvest protocols for the MBIP were dated July 14, 1988 and November 3, 1988 (both by C. Deyrup). Postharvest fumigation issues were also discussed in meetings on November 10, 1988 (see memorandum dated December 8, 1988, C. Deyrup, Ph.D. and Nancy Dodd) and on December 15 to 18, 1988 (see memorandum dated January 5, 1989, C. Deyrup, Ph.D.). Postharvest fumigation requirements were also discussed in the Residue Chemistry Chapter dated

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March 28, 1986 of the Methyl Bromide Registration Standard, in PP#5F3300 (W. Hazel, review dated February 19, 1986), and in Registration Standard follow-up reviews.

Conclusions

1. Commercial size chambers should be used. This conclusion may be reconsidered if additional data are provided which show that residues in commodities in large scale and small (28.3 l) chambers are similar.
2. Triplicate chambers and triplicate samplings from each chamber at each sampling time should be kept in the protocols.
- 3a. The dosage rates in the protocols are consistent with the registered rates.
- 3b. The 24-hour exposure times in the protocols are appropriate.
- 4a. Additional data comparing residues resulting from different treatment temperatures and the same exposure period are needed for dried fruits and nuts. The worst residue case may involve a high temperature for exposure (absorption) and low temperatures for aeration and storage (desorption).
- 4b. Since temperature is an important factor affecting residues, DEB concludes that the labels for dried fruits and nuts will need to specify treatment and aeration temperatures. For example, the labels will need to limit the fumigation and aeration temperatures to 50 degrees F or above if low temperatures result in higher residues and the studies are therefore conducted at 50 degrees F.
5. DEB concludes that a 30 percent load factor will be appropriate provided the labels are revised to limit fumigations to chambers with load factors of 30 percent or higher.
- 6a. Labels for dried fruits should be revised to prohibit vacuum fumigation.
- 6b. Data reflecting both normal atmospheric pressure (NAP) and vacuum treatment of nuts should be provided since DEB cannot determine from one comparison study on walnuts that use of NAP on

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nuts will cause higher residues than use of a vacuum chamber.

7. Concerning the King et al. headspace method, recoveries from spiked substrates and the limit of detection for each commodity must be reported.
8. The first sampling should occur when the methyl bromide concentration reaches 5 ppm. Perhaps the timing of the fumigation could be scheduled so that the 5 ppm level is reached during the day. It should be noted that this initial sampling time will require a label statement such that commodities may not be removed from fumigation chambers until MeBr concentration in chamber air is 5 ppm or less.
- 9a. Samples should be analyzed within 12 hours of sampling, or handled as described in the excerpt from the Residue Chemistry Chapter of the Methyl Bromide Registration Standard below:

"Analyses must be conducted as soon as possible (perhaps within 12 hours) after sampling and/or samples must be stored in impermeable containers. If stored in leakproof containers, analysis of headspace samples as well as the sample itself may be required if preliminary studies indicate that a significant amount of methyl bromide in the treated sample volatilizes into the gaseous phase of storage containers during a typical sample storage period. A problem may arise estimating the volume of the gaseous phase once the sample has been introduced. To increase confidence in residue determinations, spiked samples of each crop should be handled just as the treated samples are to determine the loss between treatment and analysis."
- 9b. Residue data "must be accompanied by data regarding storage length and conditions of storage of samples analyzed. These data must be accompanied by data depicting stability of residues under the conditions and for the intervals specified."
10. The protocol regarding one fumigation of packaged commodities is acceptable provided that the labels will be revised to restrict the number of fumigations of packages to one. This would mean that packages which are fumigated with methyl bromide immediately after packaging could not be refumi-

gated if returned to the packager. This may also mean that some recordkeeping system (such as stickers or codes on the package labels) would be needed by the methyl bromide fumigators so that the restriction is practical.

11. The MBIP should select a minimum time interval between refumigations for each commodity and revise labels so that refumigations will not occur within that time interval. Then the proposed studies should be conducted with refumigations occurring at the selected minimum time intervals.
- 12a. The numbers of fumigations of bulk commodities as specified in the dried fruits and nuts protocols are acceptable except for walnuts.
- 12b. Since walnuts may be fumigated five or more times before packaging, the walnut studies should be conducted with five fumigations of bulk walnuts.
13. The following information should also be reported on Form A: commodity moisture, chamber humidity, method of MeBr introduction, rate of air circulation, and mechanism for air circulation.
- 14a. Thompson seedless, which accounts for approximately 98 percent of the U.S. raisin crop, is an acceptable variety of raisins for the studies.
- 14b. The French prune variety, which accounts for approximately 90 percent of the U.S. prune crop, is an acceptable variety for the studies.
- 14c. The Deglet Noor date variety, which is the most common variety, is an acceptable variety for the studies.
- 14d. The Adriatic variety of fig is an acceptable variety for the studies.
- 14e. Residue data on both the Eureka variety and the Hartley variety of walnuts are needed.
- 14f. The Nonpareil variety of almond is acceptable for use in the studies.
- 14g. The Kerman variety of pistachio, which accounts for approximately 99 percent of the U.S. pistachio crop, is an acceptable variety for the studies.

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- 14h. The Stewart variety of pecan is an acceptable variety for the studies.
- 15a. Residue data are needed for raisins, prunes, and figs in bulk bins (such as wooden bins), cartons with liners, consumer packages with liners, and consumer packages without liners.
- 15b. Residue data are needed on bulk nonpitted dates in bins (such as wooden bins), nonpitted dates in lined cartons, and dates in consumer packages with and without liners.
- 15c. Residue data are needed on walnuts, almonds, pistachios, and pecans in bulk bins (such as wooden bins), in cartons with liners, and in consumer packages with and without liners.
- 16a. To establish a tolerance on most nuts, only nutmeats must be analyzed. For almonds, hulls as well as nutmeats must be analyzed according to DEB's Residue Chemistry Guidelines.
- 16b. Since the MBIP has indicated that almonds in hull are not fumigated with MeBr, DEB concludes that a restriction on the labels should be added which prohibits fumigation of almonds in hulls so that DEB can be sure that this will never occur. Otherwise, residue data on almond hulls resulting from fumigation of almonds in hull would be needed (see above).
17. Residue data for both inshell and shelled walnut nutmeats are needed.
18. Since residues in shelled almond nutmeats are higher than in inshell nutmeats, only data on shelled almond nutmeats are needed.
19. Residue data for both inshell and shelled pecan and pistachio nutmeats are needed unless data are provided which show that residues are higher from one type of treatment.

20. Residue data should be provided for each dried specialty or cut fruit unless crop group tolerances are sought. However, data for peaches may be used to support use on nectarines. If crop group tolerances are desired, data on representative crops as discussed in 40 CFR 180.34(f)(9) would be needed. Other requirements discussed under 40 CFR 180.34(f) must also be met.
21. Residue data should be provided for each nut. A crop group tolerance for the tree nuts group as discussed in 40 CFR 180.34(f) is probably not appropriate since the number of fumigations for the nuts of concern varies from 2 to 6.

Recommendations

DEB recommends that the MBIP modify their protocols to incorporate the above Conclusions into their proposed studies.

Detailed Considerations

Raisins

The 28.3 liter atmospheric chambers located at the USDA-ARS station in Fresno will be used for all fumigations. (These are small laboratory-size chambers instead of commercial chambers.) Each fumigation will be conducted on triplicate chambers plus one control (untreated) chamber. The treatment will be conducted at a rate of 24 g/m³ methyl bromide for 24 hours at a temperature of 10 degrees C (50 degrees F). The aeration temperature will be the same as the treatment temperature. Raisins will be fumigated in bulk with liner and in package with liner. Packaged raisins will be fumigated once. (The MBIP indicates that it is commercial practice to fumigate packaged raisins once.) Bulk raisins will be fumigated 10 times. (The MBIP indicates that this number is based on general raisin handling practices.) Thompson seedless variety will be used. (The MBIP indicates that Thompson seedless accounts for approximately 98 percent of the U.S. raisin crop. Normal atmospheric pressure (NAP) will be used in the chamber during treatment. (The MBIP indicates that vacuum fumigation is not used on raisins.) Load factor (volume/volume) will be 30 percent. (The MBIP indicates that commercial fumigations will not be conducted with less than a 30 percent load factor.) To keep the 30

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percent load factor for multiple fumigations of bulk raisins, raisins from an extra fumigated chamber will be added to chambers after raisins have been extracted for sampling. After treatment, the chambers will be aerated with forced air. Methyl bromide concentrations in chamber air will be determined from one sample location in the free air space at 0, 1/2, 1, 2, 4, 8, and 24 hours. The first raisin sample will be taken when the methyl bromide concentration in air in the chamber decreases to 5 ppm. Periodic raisin sampling (in triplicate) for at least five times and until a 0.01 ppm residue level in raisins is reached will be conducted for runs 1, 5, and 10. Refumigation time intervals will be based on the time from treatment until residues in raisins decline to 0.01 ppm. That time interval is determined from treatment No. 1 for treatments Nos. 2, 3, and 4 but is determined from treatment No. 5 for treatments Nos. 6, 7, 8, and 9. After the tenth fumigation, both raisins and raisin waste will be analyzed. Raisin samples will be put on dry ice immediately after collection. Samples will then either be analyzed the same day as collected or frozen until analysis is possible. The MBIP indicates that residues of MeBr in processed raisins are stable in frozen storage for 4 weeks:

<u>Frozen Storage Interval</u>	<u>ppm MeBr</u>
24-hour aeration	2.5 ± 0.1
1 week	2.3 ± 0.1
2 weeks	2.0 ± 0.0
3 weeks	1.9 ± 0.1
4 weeks	2.1 ± 0.1

The standard curve will be validated each day that samples are analyzed by running a spiked sample. All individual data will be reported, including representative chromatograms, calculations, standard curves, etc. Postharvest fumigation data and postharvest analysis data are to be recorded on Forms A and B (see Attachment 1 and 2, respectively).

DEB's Comments/Conclusions re: The Raisin Protocol

1. Chamber Size

All of the dried fruit and nut protocols use a 28.3 l chamber, which is a small, laboratory-size chamber instead of a large commercial chamber.

One study on raisins compares residues in raisins in a commercial chamber (3976 m³) to the 28.3 l chamber. This one comparison study indicates that residues in raisins on day 1 may be slightly higher in a small chamber, but the residues in raisins may decrease faster in a small chamber than in a large one (i.e., by day 4, residues are slightly lower for raisins in the small chamber than in the large one).

Two other studies compare air concentrations of MeBr in various size chambers filled with inshell walnuts or inshell almonds. The air concentrations of MeBr for the same load factor were similar after 4 hours fumigation.

Another study on nectarines fumigated in 0.0283 m³ or 2.83 m³ chambers is available. These are both small chambers.

DEB concludes that commercial size chambers should be used. This conclusion may be reconsidered if additional data are provided which show that residues in commodities in large scale and small (28.3 l) chambers are similar.

2. Triplicate Analyses

All dried fruit and nut protocols involve triplicate chambers and triplicate samplings from each chamber at each sampling time.

Preston Hartsell, USDA, in a phone conversation with Nancy Dodd on September 21, 1989, asked whether duplicate sampling of each of three chambers would be sufficient. This would mean that a total of six treated samples instead of nine would be taken at each sampling time.

DEB considers three analyses of each of three runs to be more reliable.

DEB concludes that triplicate chambers and triplicate samplings from each chamber at each sampling time should be kept in the protocol.

3. Rates and Exposure Times

The rates in the protocols are 24 g methyl bromide/m³ (1.5 lb/1000 cu ft) for dried fruit and 56 g methyl bromide/m³ (3.5 lb/1000 cu ft) for

nuts. Exposure times in all the protocols are 24 hours.

For dried fruit, registered rates for 98 and 100 percent pressurized gas formulations as stated in the "EPA Compendium of Acceptable Uses" issued March 7, 1984 for dried fruits (including apple, apricot, cherry, date, fig, peach, pear, prune, and raisin) were 1 to 1.5 lb per 1000 cu ft, with exposure time of 12 to 24 hours.

For nuts, registered rates for 98 and 100 percent pressurized gas formulations as stated in the "EPA Compendium of Acceptable Uses" issued March 7, 1984 for walnuts, almonds, pistachios, pecans, Brazil nuts, macadamia nuts, and hazelnuts were 1 to 3.5 lb per 1000 cu ft. The Residue Chemistry Chapter of the Methyl Bromide Registration Standard dated March 28, 1986 indicates that the exposure time for all the nuts listed above is 24 hours.

DEB concludes that the rates in the protocols are consistent with the registered rates.

DEB also concludes that the 24-hour exposure times in the protocols are appropriate.

4. Temperature

All of the protocols indicate that the temperature for exposure and aeration will be 50 degrees F (10 degrees C).

The boiling point for methyl bromide is 4.5 degrees C.

Data are provided by the MBIP which indicate that almonds exposed to MeBr for a longer time at a lower temperature have higher residues. Data are also provided on walnuts which indicate that lower storage temperatures after fumigation result in higher residues.

Another preliminary report (E.J. Bond and A.N. Starratt, Research Centre, Agriculture Canada, May 1986) indicates that apples have higher residues at 25 degrees C than at 10 degrees C.

Length of exposure can affect residue levels.

DEB concludes that additional data comparing residues resulting from different treatment

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temperatures and the same exposure period are needed for dried fruits and nuts. The worst residue case may involve a high temperature for exposure (absorption) and low temperatures for aeration and storage (desorption).

Since temperature is an important factor affecting residues, DEB also concludes that the labels for dried fruits and nuts will need to specify treatment and aeration temperatures. For example, the labels will need to limit the fumigation and aeration temperatures to 50 degrees F or above if low temperatures result in higher residues and the studies are therefore conducted at 50 degrees C.

5. Load Factor

All the dried fruit and nut protocols indicate that load factor in the fumigation chambers in the studies will be 30 percent.

The MBIP indicates that commercial fumigations will not be conducted with less than a 30 percent load factor.

A low load factor is expected to result in higher residues.

DEB concludes that a 30 percent load factor will be appropriate provided the labels are revised to limit fumigations to chambers with load factors of 30 percent or higher.

6. NAP/Vacuum

All the dried fruit and nut protocols indicate that the studies will all be conducted using NAP instead of vacuum fumigation.

The MBIP indicates that dried fruits (raisins, prunes, dates, and figs) are not vacuum fumigated.

The MBIP indicates that nuts (walnuts, almonds, pistachios, and pecans) can be vacuum fumigated, but the proposed studies will use NAP. The MBIP also indicates that it wants to retain labeling for vacuum fumigation on the labels.

The MBIP indicates that residues in bulk walnuts treated inshell are higher with NAP than with vacuum.

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DEB concludes that labels for dried fruits should be revised to prohibit vacuum fumigation.

DEB also concludes that data reflecting both NAP and vacuum treatment of nuts should be provided since DEB cannot determine from one comparison study on walnuts that use of NAP on nuts will cause higher residues than use of a vacuum chamber.

7. Analysis Method

All the dried fruit and nut protocols indicate that the method which will be used is based on the King et al. headspace GC/EC method (J. Agric. Food Chem. 29(5), 1003-1005, 1981).

DEB has previously determined (W. Hazel, May 26, 1987) that the King et al. headspace method is suitable for data collection provided that a calibration curve is prepared for every commodity analyzed.

DEB now concludes that recoveries from spiked substrates and the limit of detection for each commodity must be reported.

8. Initial Sampling Time

All the dried fruit and nut protocols involve a first sampling when the air concentration reaches 5 ppm methyl bromide.

Preston Hartsell, USDA, in a phone conversation with Nancy Dodd on September 21, 1989, asked if sampling could be delayed until morning if the 5 ppm level was reached at night. Aeration would continue, so the residues at the first sampling time would be lower.

DEB concludes that the first sampling should occur when the methyl bromide concentration reaches 5 ppm. Perhaps the timing of the fumigation could be scheduled so that the 5 ppm level is reached during the day. It should be noted that this initial sampling time will require a label statement such that commodities may not be removed from fumigation chambers until MeBr concentration in the chamber air is 5 ppm or less.

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9. Sampling to Analysis Interval

The MBIP indicates that residues of methyl bromide in raisins and prunes are stable for 4 weeks in frozen storage. Therefore, the MBIP plans to analyze raisins, prunes, and similar commodities (dates and figs) as soon as possible but not necessarily the same day.

The MBIP indicates that residues of methyl bromide in almond meats and walnut meats degrade in frozen storage. Therefore, the MBIP plans to analyze almonds, walnuts, and similar commodities (pistachios and pecans) within 24 hours of sampling.

The Residue Chemistry Chapter (dated March 28, 1986) of the Methyl Bromide Registration Standard stated that "analyses must be conducted as soon as possible (perhaps within 12 hours) after sampling and/or that samples must be stored in impermeable containers. If stored in leakproof containers, analysis of headspace samples as well as the sample itself may be required if preliminary studies indicate that a significant amount of methyl bromide in the treated sample volatilizes into the gaseous phase of storage containers during a typical sample storage period. A problem may arise estimating the volume of the gaseous phase once the sample has been introduced. To increase confidence in residue determinations, spiked samples of each crop should be handled just as the treated samples are to determine the loss between treatment and analysis."

The Residue Chemistry Chapter also indicated that all residue data "must be accompanied by data regarding storage length and conditions of storage of samples analyzed. These data must be accompanied by data depicting stability of residues under the conditions and for the intervals specified."

DEB concludes that samples should be analyzed within 12 hours of sampling, or handled as described in the Residue Chemistry Chapter excerpt above. DEB also concludes that storage stability data are needed as described above.

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10. Number of Fumigations of Packaged Dried Fruits and Packaged Nuts

All the dried fruit and nut protocols indicate that the packaged commodities will be fumigated once in the studies because it is commercial practice to fumigate packaged commodities once.

DEB concludes that the protocol regarding one fumigation of packaged commodities is acceptable provided that the labels will be revised to restrict the number of fumigations of packages to one. This would mean that packages which are fumigated with methyl bromide immediately after packaging could not be refumigated if returned to the packager. This may also mean that some record-keeping system (such as stickers or codes on the package labels) would be needed by the methyl bromide fumigators so that the restriction is practical.

11. Minimum Intervals Between Refumigations

All the protocols for the dried fruit and nuts indicate that refumigations of bulk commodities in the studies will be conducted when residues resulting from previous fumigations decline to 0.01 ppm for dried fruits or 0.1 ppm for nuts. The MBIP also indicates that it does not expect commercial refumigations to occur before residues in walnuts, almonds, and pecans decline to 0.1 ppm.

DEB has previously indicated that factors affecting residues should resemble actual commercial practices. Residues will not be measured between commercial fumigations.

DEB concludes that the MBIP should select a minimum time interval between refumigations for each commodity and revise labels so that refumigations will not occur within that time interval. Then the proposed studies should be conducted with refumigations occurring at the selected minimum time intervals.

12. Number of Fumigations of Bulk Dried Fruits and Nuts

DEB concludes that the numbers of fumigations of bulk commodities as specified in the dried fruits and nuts protocols are acceptable except for walnuts. (See "Walnuts" below.)

13. Additional Factors

DEB concludes that the following information should also be reported on Form A: commodity moisture, chamber humidity, method of MeBr introduction, rate of air circulation, and mechanism for air circulation.

14. Variety

DEB concludes that Thompson seedless, which accounts for approximately 98 percent of the U.S. raisin crop, is an acceptable variety for the studies.

15. Fumigation Containers (Bulk/Cartons/Liners/
Packaging)

The MBIP intends to fumigate raisins in bulk with liner and in package with liner.

The MBIP has submitted data comparing residues in raisins fumigated in bulk in wooden bins, lined cartons, unlined cartons, lined consumer packages (CPL), and unlined consumer packages (CPU). The highest residues between 2 and 13 days after treatment were found in CPU, but the highest residues 1 day after treatment were in CPL. (Mean values 1 day posttreatment were 3.4 ppm in CPL and 3.3 ppm in CPU.) Residues in lined cartons were higher than in unlined cartons between 1 and 8 days posttreatment. Residues 1 day after treatment were slightly higher in bulk wooden bins than in cartons (with or without liners); however, residues in bulk wooden bins were lower or equal to residues in cartons after the first day.

DEB concludes that residue data on raisins should be obtained in bulk wooden bins, in bulk cartons with liners, and in consumer packages with and without liners.

Prunes

The protocol is similar to that described for raisins. Comments relating specifically to prunes are noted below:

The French prune variety (which comprises approximately 90 percent of the U.S. prune crop) will be used.

Prunes will be fumigated in bulk with liner and in package with liner.

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Prunes in bulk will be fumigated three times. (The MBIP bases this number on commercial practice.) Triplicate samples will be taken after all three fumigations for prunes fumigated in bulk with liner, starting when the methyl bromide concentration in air is 5 ppm and continuing until the methyl bromide residue in prunes is 0.01 ppm, with at least five sampling times.

Packaged prunes will be fumigated once. (The MBIP indicates that packaged prunes are fumigated once commercially.)

Vacuum fumigation is not used on prunes.

Pitted prunes will be used in the study. (The MBIP indicates that residues are similar in pitted vs. nonpitted prunes.)

The MBIP indicates that MeBr residues in unprocessed prunes are stable in frozen storage for 4 weeks:

<u>Frozen Storage Interval</u>	<u>ppm MeBr</u>
24-hour aeration	1.2 ± 0.1
1 week	1.1 ± 0.2
2 weeks	0.85 ± 0.10
3 weeks	0.81 ± 0.14
4 weeks	1.1 ± 0.1

DEB's Comments/Conclusions re: The Prunes Protocols

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to prunes.

2. Variety

DEB concludes that the French prune variety, which accounts for approximately 90 percent of the U.S. prune crop, is an acceptable variety for the studies.

3. Fumigation Containers (Bulk/Cartons/Liners/Packaging)

The MBIP plans to fumigate prunes in bulk with liner and in package with liner.

The MBIP has submitted data on prunes which compare residues in consumer packages and residues in bulk. The data indicate that residues in prunes in consumer packages are higher.

DEB concludes that residue data are needed for prunes in bulk bins (such as wooden bins), cartons with liners, CPU, and CPL.

Dates

The protocol is similar to that described for raisins. Comments relating specifically to dates are noted below:

The Deglet Noor variety will be used. (The MBIP indicates that this is the most common variety.)

Dates will be fumigated in bulk with liner and in package.

Dates in bulk will be fumigated eight times. (The MBIP bases this number on commercial practice.) Triplicate samples will be taken after the first, fourth, and eighth fumigations for dates in bulk with liner, starting when the MeBr concentration in air is 5 ppm and continuing until the MeBr residue in dates is 0.01 ppm, with at least five sampling times.

Packaged dates will be fumigated once. (The MBIP indicates that packaged dates are fumigated once commercially.)

Unpitted dates will be used in the study. (The MBIP compared residues in bulk nonpitted dates, bulk pitted dates, and packaged pitted dates. Residues in packaged pitted dates were highest. Residues in bulk pitted dates were lowest.)

The MBIP indicates that vacuum fumigation is not used on dates.

The MBIP expects residues of MeBr in dates to be stable in frozen storage.

DEB's Comments/Conclusions re: The Dates Protocol

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to dates.

2. Variety

DEB concludes that the Deglet Noor variety, the most common variety, is an acceptable variety for the studies.

3. Fumigation Containers (Bulk/Cartons/Liners/Packaging)

The MBIP plans to fumigate dates in bulk with liner and in package.

The MBIP has submitted data on dates which compares bulk nonpitted, bulk pitted, and consumer packaged pitted dates. The residues in packaged pitted dates were highest. Residues in bulk nonpitted dates were higher than in bulk pitted dates.

DEB concludes that residue data are needed on bulk nonpitted dates in bins (such as wooden bins), nonpitted dates in lined cartons, and dates in consumer packages with and without liners.

Figs

The protocol is similar to that described for raisins. Comments relating specifically to figs are noted below:

The Adriatic variety will be used in the residue studies. (The MBIP indicates that this variety has the highest residues.)

Figs will be fumigated in bulk with liner and in package with liner.

Figs in bulk with liner will be fumigated three times. (The MBIP bases this number on commercial practice.) Triplicate samples will be taken after each fumigation, starting when the MeBr concentration in air is 5 ppm and continuing until the MeBr residue in figs is 0.01 ppm, with at least five sampling times.

Packaged figs will be fumigated once. (The MBIP indicates that packaged figs are fumigated once commercially.)

The MBIP indicates that vacuum fumigation is not used on figs.

The MBIP expects residues of MeBr in figs to be stable in frozen storage.

DEB's Comments/Conclusions re: The Figs Protocol

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to figs.

2. Variety

The MBIP intends to use the Adriatic variety of fig.

The MBIP has submitted residue data on dried figs comparing Adriatic, Calimyrna, Kadota, and Mission figs. Mean residues in Mission and Adriatic figs were 3.6 and 3.3 ppm, respectively, on day 1. Residues in Calimyrna and Kadota figs were lower. By day 6, residues were higher in Adriatic (0.69 ppm) compared to Mission (0.35 ppm).

DEB concludes that the Adriatic variety is an acceptable variety for the studies.

3. Fumigation Containers (Bulk/Carton/Liners/Packaging)

The MBIP plans to fumigate figs in bulk with liner and in package with liner.

The MBIP has submitted data which compare residues in bulk and consumer packaged figs. Residues on day 1 were higher in bulk figs (3.4 ppm) than in consumer packaged figs (0.52 ppm). However, mean residues in consumer packaged figs increased to 1.44 ppm by day 6, while mean residues in bulk figs decreased to 0.62 ppm by day 6.

DEB concludes that residue data are needed on figs in bulk bins (such as wooden bins), in carton with liner, in CPU, and in CPL.

Walnuts

The protocol is similar to that described for raisins. Comments relating specifically to walnuts are noted below:

Hartley walnuts will be used. The MBIP indicates that Hartley leads in acreage and tonnage. Eureka, a minor variety, showed higher residues.

Walnuts will be fumigated in bulk with liner and in package.

Walnut nutmeats in bulk with liner will be fumigated four times. (The MBIP expects residues in shelled nutmeats to be higher than residues in inshell nutmeats. The MBIP indicates that it bases the number of fumigations on commercial practice.) The MBIP will take triplicate samples of bulk walnuts after each fumigation, starting when the methyl bromide concentration in air is 5 ppm and continuing until the methyl bromide residue in walnuts is 0.1 ppm, with at least five sampling times. (The MBIP indicates that commercial refumigation will not occur until MeBr residues in walnuts decrease to 0.1 ppm.)

Packaged walnuts will be fumigated once. (The MBIP indicates that packaged walnuts are fumigated once commercially.)

Dosage will be 56 g/m³ for 24 hours at 15.6 degrees C.

Vacuum treatments are done commercially, but the MBIP plans to conduct the residue studies at NAP. The MBIP indicates that higher residues in bulk walnut nutmeats were found with NAP than with vacuum fumigation.

Residues of MeBr in walnuts in frozen storage decrease with time as follows:

<u>Interval of Frozen Storage</u>	<u>ppm Residues in Walnut Meats</u>
After 24 hours aeration	68.1 ± 11.9
1 week	37.8 ± 7.6
2 weeks	37.0 ± 3.8
3 weeks	26.5 ± 3.7
4 weeks	24.6 ± 1.0

The MBIP will analyze residues as soon as possible but walnut samples may be frozen for as long as 24 hours before analysis.

DEB's Comments/Conclusions re: The Walnuts Protocol

1. Comments/Conclusions 1 through 11, and 13 under "Raisins" also apply to walnuts.
2. Variety

The MBIP intends to use the Hartley variety because it leads in acreage and tonnage.

The MBIP has submitted data which compares residues in Eureka, Hartley, Payne, and Franquette varieties. Residues in the Eureka variety are highest.

DEB needs data on the "worst case" residues to set tolerances. For risk assessment purposes, data on the leading variety would also probably be useful.

DEB concludes that residue data on both the Eureka variety and the Hartley variety are needed.

3. Number of Fumigations of Bulk Dried Fruits and Nuts

The MBIP intends to fumigate bulk walnuts with liner four times.

The MBIP has submitted a processing outline which indicates that walnuts can be treated before packaging five or more times (for shelled kernels).

DEB concludes that walnuts may be treated five or more times before packaging according to the "General Procedure for Walnuts." Therefore, the walnut studies should be conducted with five fumigations of bulk walnuts.

4. Fumigation Containers (Bulk/Cartons/Liners/Packaging)

The MBIP intends to fumigate walnuts in bulk with liner and in package.

The MBIP has submitted data which compares residues in bulk walnut meats and consumer packaged walnut meats. Residues 1 day after treatment were higher in bulk walnut meats. However, residues in packages apparently increased slightly from day 1 to day 2 posttreatment and then declined slowly.

DEB concludes that residue data are needed on walnut in bulk bins (such as wooden bins), in cartons with liners, and in CPU and CPL.

5. Inshell vs. Shelled Nut Meats

Comparison of residues of inshell nut meats vs. shelled nut meats for walnuts indicates that residues 1 and 6 days after treatment are higher for inshell walnut meats. Residues on days 2 and 9 are slightly higher for shelled walnut meats.

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DEB concludes that residue data for both inshell and shelled walnut nutmeats are needed.

Almonds

The protocol is similar to that described for raisins. Comments relating specifically to almonds are noted below.

Nonpareil almond will be used. The MBIP indicates that this variety leads in acreage and tonnage and had slightly higher residues.

Almonds will be fumigated in bulk with liner and in packages. The MBIP submits data that indicates that residues in almond meats in bulk (no container) were lower than residues in bulk carton without liner. Residues in bulk carton with liner were highest of the three.

Almond nutmeats in bulk with liner will be fumigated five times. (The MBIP indicates that shelled almond nutmeats have higher residues than inshell nutmeats. The MBIP also indicates that almonds are not fumigated in hull with methyl bromide. The MBIP bases the number of fumigations on commercial practice.) The MBIP will take triplicate samples of bulk almonds after the first, third, and fifth fumigations, starting when the methyl bromide concentration in air is 5 ppm and continuing until the methyl bromide residue in almonds is 0.1 ppm, with at least five sampling times. (The MBIP indicates that commercial refumigation will not occur until MeBr residues in almonds decrease to 0.1 ppm.)

Packaged almonds will be fumigated once. (The MBIP indicates that packaged almonds are fumigated once commercially.)

Dosage will be 56 g/m^3 for 24 hours at 15.6 degrees C.

Vacuum treatments are done commercially, but the MBIP plans to conduct the residue studies at NAP.

Residues in frozen almonds decrease with time as follows:

<u>Interval of Frozen Storage</u>	<u>ppm Residues in Almond Meats</u>
After 24 hours aeration	40.6 ± 3.8
1 week	24.0 ± 4.3
2 weeks	22.4 ± 3.2
3 weeks	13.3 ± 1.2
4 weeks	10.8 ± 0.4

Almonds will be analyzed as soon as possible but may be held up to 24 hours.

DEB's Comments/Conclusions re: The Almond Protocol

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to almonds.

2. Variety

The MBIP intends to use the Nonpareil variety of almond. The submitted data indicate that the Merced variety had slightly higher residues for 4 to 24 hours aeration time, but the difference was 2 ppm or less. Residues at 72 hours aeration were higher for Nonpareil.

DEB concludes that the Nonpareil variety of almond is acceptable for use in the studies.

3. Fumigation Containers (Bulk/Cartons/Liners/Packaging)

The MBIP intends to fumigate almonds in bulk with liner and in package.

The MBIP has submitted data on almonds which compares residues in bulk and in consumer packages. Initial residues (1 day after treatment) are higher in bulk almonds than in consumer packages. Residues in packages decrease slower.

The MBIP also submitted data comparing residues in almond meats in bulk, in cartons without liners, and in cartons with liners. Mean residues after 24 hours aeration were 25.2 ppm in bulk almonds, 24.4 ppm in cartons without liners, and 19.8 ppm in cartons with liners. After 48 hours aeration, residues of almonds in cartons with liners were higher than in cartons without liners. This indicates that a liner slows dissipation of residues from almonds.

DEB concludes that residue data are needed on almonds in bulk bins (such as wooden bins), in cartons with liners, and in CPU and CPL.

4. Almond Hulls

To establish a tolerance on most nuts, only nutmeats must be analyzed. For almonds, hulls as well as nutmeats must be analyzed according to DEB's Residue Chemistry Guidelines.

Since the MBIP has indicated that almonds in hull are not fumigated with MeBr, DEB concludes that a restriction on the labels should be added which prohibits fumigation of almonds in hulls so that DEB can be sure that this will never occur. Otherwise, residue data on almond hulls resulting from fumigation of almonds in hull would be needed.

5. Inshell vs. Shelled Nut Meats

Data comparing residues in inshell vs. shelled almond nutmeats indicate that residues in shelled almond nutmeats are higher.

DEB concludes that only data on shelled almond nutmeats are needed.

Pistachios

The protocol is similar to that described for raisins. Comments relating specifically to pistachios are noted below:

Kerman pistachio will be used. The MBIP indicates that this accounts for approximately 99 percent of the U.S. pistachio crop.

Pistachios will be fumigated in bulk with liner and in package.

Pistachio nutmeats in bulk with liner will be fumigated five times. (The MBIP expects residues in shelled nutmeats to be higher than residues in inshell nutmeats. The MBIP bases the number of fumigations on commercial practice.) The MBIP will take triplicate samples of bulk pistachios after the first, third, and fifth fumigations, starting when the MeBr concentration in air is 5 ppm and continuing until the MeBr residue in pistachios is 0.1 ppm, with at least five sampling times.

Packaged pistachios will be fumigated once. (The MBIP indicates that packaged pistachios are fumigated once commercially.)

Dosage will be 56 g/m^3 for 24 hours at 15.6 degrees C.

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Vacuum treatments are done commercially, but the MBIP plans to conduct the residue studies at NAP.

Pistachios will be analyzed as soon as possible after sampling but may be stored frozen for as long as 24 hours before analysis.

DEB's Comments/Conclusions re: The Pistachio Protocol

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to almonds.

2. Variety

DEB concludes that the Kerman variety of pistachio, which accounts for approximately 99 percent of the U.S. pistachio crop, is an acceptable variety for the studies.

3. Fumigation Containers (Bulk/Cartons/Liners/Packaging)

The MBIP intends to fumigate pistachios in bulk with liner and in package.

The MBIP has submitted data on pistachios which compare residues in bulk and in consumer packages.

Residues in pistachio nuts were higher in consumer packages than in bulk.

DEB concludes that residue data are needed on pistachios in bulk bins (such as wooden bins), in cartons with liners, and in CPU and CPL.

4. Inshell vs. Shelled Nutmeats

Data comparing residues of shelled vs. inshell pistachio nutmeats have not been submitted. Such data for walnuts and almonds are discussed in the walnut and almond sections of this review.

DEB concludes that residue data for both inshell and shelled pistachio nutmeats are needed unless data are provided which show that residues are higher from one type of treatment.

Pecans

The protocol is similar to that described for raisins. Comments relating specifically to pecans are noted below:

Stewart variety of pecan will be used. The MBIP indicates that the Stewart variety leads in acreage and tonnage.

Pecans will be fumigated in bulk with liner and in package.

Pecan nutmeats in bulk with liner will be fumigated two times. (The MBIP expects residues in shelled nutmeats to be higher than residues in inshell nutmeats. The MBIP bases the number of fumigations on commercial practice.) The MBIP will take triplicate samples of bulk pecans after each fumigation, starting when the MeBr concentration in air is 5 ppm and continuing until the MeBr residue in pistachios is 0.1 ppm, with at least five sampling times. (The MBIP indicates that commercial refumigation will not occur until MeBr residues in pecans decrease to 0.1 ppm.)

Packaged pecans will be fumigated once. (The MBIP indicates that packaged pecans are fumigated once commercially.)

Dosage will be 56 g/m³ for 24 hours at 15.6 degrees C.

Vacuum treatments are done commercially, but the MBIP plans to conduct the residue studies at NAP.

Pecans will be analyzed as soon as possible after sampling but may be stored frozen as long as 24 hours before analysis.

DEB's Comments/Conclusions re: The Pecan Protocol

1. Comments/Conclusions 1 through 13 under "Raisins" also apply to pecans.
2. Variety

The MBIP intends to use the Stewart variety of pecan, which leads in acreage and tonnage.

The MBIP has submitted data which indicate that mean residues 1 or 2 days after fumigation are higher in the Stewart variety than in Witchita or Western varieties. However, 6 days after fumigation, residues in the Stewart variety are lower than in the Witchita or Western varieties. Mean values at 6 days posttreatment are 3.9, 3.6, and 2.0 ppm for Witchita, Western, and Stewart varieties, respectively.

DEB concludes that the Stewart variety is an acceptable variety for the studies.

3. Fumigation Containers (Bulk/Cartons/Liners/
Packaging)

The MBIP intends to fumigate pecans in bulk with liner and in package.

The MBIP has submitted data on inshell pecans which compare residues in bulk and in consumer packages. On 1 day posttreatment, mean residues in bulk were higher than in packages (63.3 vs. 9.7 ppm, respectively). Residues in inshell pecans in consumer packages increased from 1 to 2 days posttreatment (from 9.7 to 24.9 ppm).

DEB concludes that residue data are needed on pecans in bulk bins (such as wooden bins), in cartons with liners, and in CPU and CPL.

4. Inshell vs. Shelled Nut Meats

Data comparing residues in shelled vs. inshell pecan nutmeats have not been submitted. Such data for walnuts and almonds are discussed in the walnut and almond sections of this review.

DEB concludes that residue data for both inshell and shelled pecan nutmeats are needed unless data are provided which show that residues are higher from one type of treatment.

Bridging Data

Dried Specialty or Cut Fruits (Apricots, Pears, Peaches,
and Nectarines)

The MBIP indicates that it considers apricots to be representative of the cut fruits. The MBIP does not plan to conduct residue studies on each cut fruit.

The MBIP considers residues in cut fruit to be similar to that of other dried fruits. The MBIP has submitted data which compares residues in cut dried apricots to those in figs, raisins, nonpitted prunes, and dates. The MBIP indicates that residue data for cut dried apricots are "not significantly different" from residue data for figs, raisins, prunes, and dates.

According to the MBIP, dried cut fruits can be fumigated after drying, in storage, and after packaging.

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DEB's Discussion re: Dried Specialty or Cut Fruits

Mean residues in consumer packaged cut, dried apricots were 5.4 ppm 1 day after fumigation. Residues in packaged figs were much lower (0.52 ppm) at that time.

No crop group is established under 40 CFR 180.34(f) for dried cut fruits (apricots, pears, peaches, and nectarines). These fruits include fruits from two crop groups: stone fruits and pome fruits.

DEB's Conclusions re: Dried Specialty or Cut Fruits

DEB concludes that residue data should be provided for each dried specialty or cut fruit unless crop group tolerances are sought. However, data for peaches may be used to support use on nectarines. If crop group tolerances are desired, data on representative crops as discussed in 40 CFR 180.34(f)(9) would be needed. Other requirements discussed under 40 CFR 180.34(f) must also be met.

Nuts

The MBIP indicates that residues decrease from commodity to commodity as follows: pecans, walnuts, hazelnuts, almonds, macadamia nuts, Brazil nuts, pistachios.

The MBIP does not intend to conduct residue studies on hazelnuts, macadamia nuts, or Brazil nuts since residues on those nuts fall within the range of residues on the other nuts.

DEB's Conclusion re: Bridging Data for Nuts

DEB concludes that residue data should be provided for each nut. A crop group tolerance for the tree nuts group as discussed in 40 CFR 180.34(f) is probably not appropriate since the number of fumigations for the nuts of concern varies from 2 to 6.

Attachment 1: Form A
Attachment 2: Form B

cc with Attachments: RF, SF, Circulation, PP#5F3300, N. Dodd (DEB), Methyl Bromide Registration Standard File - W. Boodee, E. Eldredge (ISB/PMSD), R. Schmitt (DEB)

RDI:D. Edwards:2/15/90:R. Loranger:2/15/90
H7509C:DEB:CM#2:Rm. 800D:X1681:N. Dodd:KENCO:2/16/90

POST-HARVEST FUMIGATION DATA

DATE _____ FUMIGATION CHAMBER NO. _____

DESCRIPTION OF CHAMBER
(size, NAP, vacuum) _____

PREFUMIGATION

COMMODITY TEMP. _____

FUMIGANT USED

DOSAGE _____ EXPOSURE PERIOD _____

COMMODITIES FUMIGATED

FUMIGATION STARTED

DATE _____ TIME _____

CONCENTRATION DURING EXPOSURE PERIOD:

START: _____

DURING: _____

END: _____

AERATION COMMENCED:

DATE _____ TIME _____

DURATION _____

COMMODITY SAMPLED

SAMPLE I.D.

FORM B

POST-HARVEST FUMIGATION ANALYSIS

SAMPLE I.D. NUMBER

HOW HANDLED

SHIPPING DATA

DATE _____ TIME _____

PACKING _____ LOCATION _____

SAMPLE RECEIVING DATA

DATE _____ TIME _____

CONDITION _____ DISPOSITION _____

ANALYSIS PERFORMED

RESULTS

SIGNATURE

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