



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

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OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Follow-up to Methyl Bromide Registration Standard.
Post Harvest Protocol on 12 Commodities.
(I.D. No. 53201-1; Record No. 228,380, 228,382-
228,390, 228,393, 228,395) RCB No. 4121-4130,
4133, 4135

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The Methyl Bromide Industry Panel (MBIP) has submitted protocols for generating residue data on 12 commodities. The protocols reflect postharvest and, where appropriate, preplant soil fumigations uses. The Methyl Bromide Registration Standard has cited the need for residue data on all commodities for which MeBr is registered for postharvest use.

Recommendations

DEB recommends that the protocols be modified to take into

account the Comments/Conclusions delineated under each commodity in the Detailed Considerations section of this review.

Detailed Considerations

DEB's Comments/Conclusions will follow the description of each protocol.

Apples

The apples are to be grown, where possible, on preplant fumigated soil. If preplant soil fumigated sites cannot be found, the study will use apples grown on non-preplant fumigated soil. The harvested apples will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities treated at the same rate.

Apples grown on preplant fumigated soil will be fumigated at a dosage rate of 3 lbs ai/1000ft³ for 2 hours. Before fumigation, the apples will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode method. The apples will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the apples will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The apples will be aerated for a total of 24 hours when additional samples will be withdrawn, again in quadruplicate. The apples will be subjected to one fumigation only.

Fruit bearing measurable residues of MeBr will be processed into apple sauce and juice. The registrant states that fumigated apples are intended for fresh market use only, and the protocol does not include processing into "...peel, oil, and molasses."

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Apples

1. The preplant fumigation rate was not specified. According to the Methyl Bromide Registration Standard, deciduous fruits may be planted in soil fumigated at a rate of ≤ 870 lbs ai/A or

2 lb ai/tree site, and fruit are not to be harvested for at least 2 years. DEB understands that it may be difficult to find an adequate supply of apples which have been treated in accordance with the maximum label rate for preplant use. The preplant data were required by the MeBr Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to fumigate apples grown in untreated soil could depend upon the results of studies aimed at determining residue levels in apples grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in apples grown in preplant treated soil prove to be insignificant, DEB has no objection to using apples grown in untreated soil. If bromide levels in apples grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

For the sake of completeness, the background levels of MeBr and iBr should also be determined in apples grown in untreated soil.

2. The dosage to be used in the protocol is 3 lbs ai/1000 ft³ for 2 hours. According to the Methyl Bromide Registration Standard, apples may be treated at a rate of 5 lbs ai/1000 ft³ for 2 hours. This is the same dosage rate and exposure time proposed in PP #5F3300. If it is MBIP's intent to lower the dosage rate to 3 lbs ai/1000 ft³, all labels describing the postharvest use of MeBr on apples would need to be revised.

The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of apples in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.

3. According to the Plant Protection and Quarantine Manual, apples may be fumigated at the following rates for 2 hours:

1.5 lbs ai/1000 ft³; 80-89°F
2.0 lbs ai/1000 ft³; 70-79°F
2.5 lbs ai/1000 ft³; 60-69°F
3.0 lbs ai/1000 ft³; 50-59°F
4.0 lbs ai/1000 ft³; 40-49°F

The proposed protocol would cover residues in imported apples only if all imported apples were treated under the conditions used to generate the residue data; i.e., 3.0 lbs ai/1000 ft³ at some unspecified temperature for 2 hours.

In order to cover residues in apples treated for quarantine purposes, it would first be necessary to determine the

worst case; the worst case is not immediately obvious because of the variation in the parameters affecting residue levels (dosage rate and temperature). Then residue data representing the worst case would need to be generated.

4. The temperature of the fumigation was not specified. It has been reported in the literature that the storage temperature prior to fumigation may effect the amount of fumigant absorbed by the commodity [W. B. Sinclair and D.L. Lindgren, "Factors Affecting the Fumigation of Food Commodities for Insect Control," J. Econ. Entomology, 51 (6): 891-900 (1958).] The residue data should reflect the worst case. The label submitted with PP #5F3300 limited fumigation temperatures to $\geq 50^{\circ}\text{F}$. If lower temperatures may occur during commercial operations and if lower temperatures lead to higher residue levels, MBIP will need to demonstrate that the 50°F restriction is feasible and generate the appropriate residue data. If higher temperatures lead to higher residue levels, the residue data should reflect the highest temperatures expected during commercial fumigation.

The registrant has the option of narrowing the range of fumigation temperatures in a revised label, but he will need to consider the practicality of the label and should support the contention that the temperatures used to generate the residue data would be applied commercially.

5. If apples are stored prior to fumigation, residue data should be generated on apples which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities. The registrant has the option of specifying the temperature of the commodities to be fumigated, if it can be demonstrated that this restriction is practical.
6. The aeration temperature should be specified. Again, if different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be needed.
7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition

of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of containers; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of apples were to be fumigated. Residue data on common commercial cultivars are needed.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for apples treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.
12. According to United Fresh Fruit and Vegetables, apples may be waxed before fumigation. Therefore, residue data on waxed and unwaxed apples are needed.
13. MBIP will need to support the position that apples receive only one fumigation commercially.
14. Residue data should reflect treatment of the apples at the stages of development during which the fruit are fumigated commercially. Sinclair and Lindgren (see Comment #4 under DEB's Comments/Conclusions-Apples) have reported that the amount of fumigant sorbed by a commodity could depend upon its maturity.
15. It has been shown that residue levels for some fumigants may be higher if the fruit are bruised or stemless. Therefore, the samples should include a representative proportion of bruised and stemless commodities.
16. If apples are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of apples in trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residues

may be higher after the fumigation of trucks. If the registrant contends that apples are not fumigated in trucks, this position would have to be supported with documentation.

In generating residue data reflecting the fumigation of trucks, the protocol should reflect commercial practice. If commodities are aerated in trucks which are then sealed, a similar procedure should be followed in the study. Caution should be exercised in reentering the truck to secure samples; DEB has been informed by a fumigator that residues in the air of the truck may rise to unsafe breathing levels if the truck is sealed after the initial aeration.

If it can be demonstrated that fumigation in trucks does not lead to higher residue levels in commodities belonging to at least three different crop groupings, truck fumigation would not be required for all commodities covered in the protocols in the present submission.

17. After consulting with the FDA (telecon with D. Reid, 8/16/88), DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. The FDA monitors samples as they move through commercial channels. Therefore, if several bins from the same fumigation travel together through commercial channels, the FDA would sample from several bins; in effect this practice would composite samples which originated from different sections of the fumigation chamber. On the other hand, if it is possible that the lot could travel through commercial channels by itself, then, of course, sampling from only the one lot is possible.

If it is commercial practice for bins from the same fumigation to travel together, samples should be drawn from different sections of bins (e.g., from the top, middle, and bottom) from different areas of the chamber. The samples should be composited before analysis. If it is possible that a single bin could travel through channels to a distributor, then samples should be drawn from different sections of the bins located in the areas of highest MeBr concentration in the chamber. Again, the samples should be composited before analysis.

18. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or that the apples may not be bagged before the aeration period has elapsed).
19. The Methyl Bromide Registration Standard states, "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." This statement also occurs in the 2/19/86 review of PP #5F3300 (memo of W. Hazel).

Therefore, a storage stability study is needed for each commodity. Data submitted by MBIP indicate that loss of MeBr during storage could depend upon the nature of the commodity. MeBr residues in rice declined by 22% after 8 hours of storage at dry ice temperature, -78°C , which is considerably colder than freezer temperatures; freezer storage or storage on dry ice is proposed in the current protocol. MBIP may want to store the commodities at -78°C instead of in freezers.

20. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

21. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.), and precautions taken to avoid loss of the analyte should be delineated. The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.
22. The registrant needs to submit residue data on wet and dry pomace in addition to juice. DEB is concerned that if it is necessary to fumigate infested imported apples, the importer may opt to send the apples to a processor if fumigation has an adverse effect on the shelf life of the apples. Also, as long as there is a possibility for processing fumigated apples into juice, it would appear that it is equally possible to process the apples into pomace. Details of the processing should be submitted so DEB can determine whether the processing conforms to commercial practice.
23. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
24. MBIP should heed RCB's comments contained in previous memos

and in the Registration Standard regarding the generation of residue data for post harvest use.

Asparagus

The asparagus protocol is identical to the apple protocol described above. The asparagus will also be subjected to one fumigation only. However, the dosage rate is described as "4 pounds of methyl bromide," and it is unclear whether the 4 lbs is to be used for the 885 ft³ chamber or whether the registrant meant 4 lbs ai/1000 ft³.

DEB's Comments/Conclusions-Asparagus

1. The preplant fumigation rate was not specified. The Methyl Bromide Registration Standard specified that residue data are required on asparagus from CA grown in soil fumigated at a rate of 400 lb ai/A and from MI and WA on asparagus grown in soil fumigated at rate of 240 lb ai/A. Data on asparagus grown in preplant fumigated soil will be required. The Methyl Bromide Registration Standard describes a study in which iBr residues in asparagus were found to range up to 2100 ppm iBr 5.3 months after the preplant treatment; iBr residue levels in check samples ranged up to 69 ppm. The contributions of preplant and postharvest treatments to the total iBr must be taken into account before establishing tolerances for MeBr and iBr on asparagus.

In generating the preplant residue data, the registrant should consider commercial planting practices, such as the latest growth stage at which transplantation occurs. In the case of asparagus, some residue data reflecting the shortest PHI after crown set would be needed.

For the sake of completeness, the levels of MeBr and iBr should also be determined in asparagus grown in untreated soil.

2. MBIP needs to clarify the treatment rate. DEB assumes that the dosage rate is 4 lbs ai/1000 ft³, but the protocol does not give the volume to be treated with the 4 lbs.
3. The most recently revised label (submitted with PP #5F3300) does not include a use for asparagus. In fact, according to the "Guidance for the Reregistration of Pesticide Products Containing Methyl Bromide as the Active Ingredient," there is no domestic postharvest use for MeBr on asparagus. If the petitioner intends to register MeBr for postharvest use on asparagus, MBIP will need to amend PP #5F3300 to include the use on asparagus. Since there is no registered use or proposed use, DEB cannot draw any conclusions on the adequacy of the submitted protocol. If the registrant revises the label to include asparagus, data reflecting the use of vacuum chamber would be needed. The present label implies that vacuum chamber fumigation may be used on asparagus. The registrant has the option of revising the label to delete the use of

vacuum chamber equipment.

4. If it is assumed that the protocol covers a proposed use of 4 lbs ai/1000 ft³ for 2 hours, the fumigation temperature would still need to be specified.

It has been reported in the literature that the storage temperature prior to fumigation may effect the amount of fumigant absorbed by the commodity [W. B. Sinclair and D.L. Lindgren, "Factors Affecting the Fumigation of Food Commodities for Insect Control," J. Econ. Entomology, 51 (6): 891-900 (1958).]

If the label submitted with PP #5F3300 is amended to include asparagus, the fumigation temperature would be limited to $\geq 50^{\circ}\text{F}$. MBIP needs to determine the range of temperatures under which commercial fumigation of asparagus may occur, determine which temperature represents the worst case, and carry out the fumigations at this temperature. If lower temperatures may occur during commercial fumigation, and if lower temperatures lead to higher residue levels, MBIP will need to demonstrate the practicality of the $\geq 50^{\circ}\text{F}$ label restriction. If higher temperatures lead to higher residue levels, the residue data should reflect the highest fumigation temperatures used commercially.

The registrant has the option of narrowing the range of fumigation temperatures in a revised label, but he will need to consider the practicality of the label and should support the contention that the temperatures used to generate the residue data would be applied commercially.

5. DEB notes that after harvest, asparagus are often hydrocooled to $34-36^{\circ}\text{F}$ for less than 15 minutes (Dr. B Benson, UC-Davis). Therefore, DEB needs to know whether asparagus are generally stored cooled before fumigation and the temperature at which they are stored. If asparagus are stored prior to fumigation, residue data should be generated on asparagus which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities (see Conclusion 4). The registrant has the option of specifying the temperature of the commodities to be fumigated, if it can be demonstrated that this restriction is practical.
6. The maximum treatment rate given in the Plant Protection and Quarantine Treatment Manual (PPQ) is 4 lbs ai/1000 ft³ for 2 hours at $60-69^{\circ}\text{F}$. DEB cannot determine if residues in imported asparagus would be covered by the protocol because the fumigation temperature was not specified. If the temperature selected by the registrant is different from that listed in the PPQ, DEB suggests that bridging data linking the fumigation temperatures be generated.

7. The aeration temperature should be specified. Again, if different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be needed.
8. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

9. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data. For example, if wax coated fiberboard boxes, polyethylene liners, or expanded polystyrene foam boxes are in commercial use, some data may need to be submitted reflecting the use of these containers. If asparagus may be fumigated while set on sphagnum moss or water-retentive fiber pads, data may be needed reflecting this use.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

10. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for asparagus treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.
12. MBIP will need to support the position that asparagus receive only one fumigation commercially.
13. It has been shown that residue levels for some fumigants may be higher if the fruit are bruised or stemless. There-

fore, the samples should include a representative proportion of bruised commodities. If asparagus are selected and packed so that no specimens are bruised, the registrant should support this contention with documentation.

14. If asparagus are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. (See Conclusion 16 under Comments/Conclusions-Apples for further details).
15. DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under Comments/Conclusions-Apples)
16. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed).
17. A storage stability study is needed for each commodity. MBIP may want to store the commodities at dry ice temperature instead of in freezers. (See Conclusion 19 under Comments/Conclusions-Apples)
18. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

19. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.) and precautions taken to avoid loss of the analyte should be delineated. The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.
20. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
21. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Cherries

The cherries are to be grown, where possible, on preplant fumigated soil. If preplant soil fumigated sites cannot be found, the study will use cherries grown on non-preplant fumigated soil. The harvested cherries will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities treated at the same rate.

Cherries grown on preplant fumigated soil will be fumigated at a dosage rate of 4 lbs ai/1000 ft³ for 2 hours. The fruit temperature will be 6-12°C.

Before fumigation, the cherries will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode method. The cherries will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the cherries will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The cherries will be aerated for a total of 6 hours when additional samples will be withdrawn, again in quadruplicate. The cherries will be subjected to one fumigation only.

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Cherries

1. The preplant fumigation rate was not specified. According to the Methyl Bromide Registration Standard, deciduous fruits may be planted in soil fumigated at a rate of ≤ 870 lbs ai/A or 2 lb ai/tree site, and fruit are not to be harvested for at least 2 years. DEB understands that it may be difficult to find an adequate supply of cherries which have been treated in accordance with the maximum label rate for preplant use. The preplant data were required by the Methyl Bromide Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to use cherries grown in untreated soil could depend upon the results of studies aimed at determining residue levels in cherries grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in cherries grown in preplant treated soil prove to

be insignificant, DEB has no objection to using cherries grown in untreated soil. If bromide levels in cherries grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

In any case, for the sake of completeness, levels of MeBr and iBr should be determined in cherries grown in untreated soil.

2. The dosage to be used in the protocol is 4 lbs ai/1000 ft³ for 2 hours. According to the Methyl Bromide Registration Standard, cherries may be treated at a rate of 5 lbs ai/1000 ft³ for 2 hours. This is the same dosage rate and exposure time proposed in PP #5F3300. If it is MBIP's intent to lower the dosage rate to 4 lbs ai/1000 ft³, all labels describing the use of MeBr on cherries would need to be revised.

The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of cherries in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.

3. According to the Plant Protection and Quarantine Manual, cherries may be fumigated at the following rates for 2 hours:

- 1.5 lbs ai/1000 ft³; 80-89°F
- 2.0 lbs ai/1000 ft³; 70-79°F
- 2.5 lbs ai/1000 ft³; 60-69°F
- 3.0 lbs ai/1000 ft³; 50-59°F
- 4.0 lbs ai/1000 ft³; 40-49°F

DEB concludes that the protocol (4 lbs ai/1000 ft³, 43-54°F) would cover residues on imported cherries treated at a rate of 4.0 lbs ai/1000 ft³ while maintained at 40-49°F, provided that this temperature is maintained at $\leq 45^\circ\text{F}$ during the fumigation interval. The protocol would cover all imported cherries, if it is demonstrated that residue levels increase with decreasing temperature.

4. The protocol should specify the fumigation chamber temperature, as this may affect residue levels. The residue data should reflect the worst case.

The registrant should consider commercial practice and will need to support the contention that the temperatures used to generate the residue data would be applied in commercial practice.

The label submitted with PP #5F3300 limited fumigation temperatures to $\geq 50^\circ\text{F}$. From the submitted protocol, cherries are apparently stored at 43-54°F. If lower storage and fumigation temperatures may occur during commercial operations and if

lower temperatures lead to higher residue levels, MBIP may need to revise the label to permit fumigation at a lower temperature and generate the appropriate residue data. If higher temperatures lead to higher residue levels, the residue data should reflect the highest temperatures expected during commercial fumigation.

5. If cherries are stored prior to fumigation, residue data should be generated on cherries which have been stored for representative periods at the temperatures used for commercial storage.
6. The aeration temperature should be specified. Again, if different temperatures are used commercially for the aeration of cherries than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical.
7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data. For example, if wax coated fiberboard boxes, polyethylene liners, or expanded polystyrene foam boxes are in commercial use, some data may need to be submitted reflecting the use of these containers.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of cherries were to be fumigated. Residue data on sweet and tart varieties are

needed.

11. One fumigation run would not serve as an adequate data base for establishing tolerances for cherries treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.
12. MBIP will need to support the position that cherries receive only one fumigation commercially.
13. It has been shown that residue levels for some fumigants may be higher if the fruit are bruised or stemless. Therefore, the samples should include a representative proportion of bruised and stemless commodities.
14. If cherries are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. (See Conclusion 16 under Comments/Conclusions-cherries for details)
15. After consulting with the FDA (telecon with D. Reid, 8/16/88), DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under Comments/Conclusions-Apples for further details)
16. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or that the cherries may not be bagged before the aeration period has elapsed).

The protocol states that if MeBr residue levels have not declined to about 0.01 ppm after 24 hours of aeration, the aeration will be continued so an aeration period can be recommended. Since the registrant intends to analyze cherries after 3 and 6 hours of aeration only, he needs to explain how he will know whether MeBr residue levels will have declined to about 0.01 ppm after 24 hours of aeration.

17. A storage stability study is needed for each commodity. MBIP may want to store the commodities at dry ice temperatures instead of in freezers. (See Conclusion 19 under Comments/Conclusions-Apples for details)
18. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction

and use of the standard curve and the determination of ppm iBr from the correct equation.

19. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during chopping, pitting the cherries, and compositing samples, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.) and precautions taken to avoid loss of the analyte should be delineated.

The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.

20. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
21. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Grapefruit, Lemons, and Oranges

The citrus are to be grown, where possible, on preplant fumigated soil. If preplant soil fumigated sites cannot be found, the study will use citrus grown on non-preplant fumigated soil. The harvested citrus will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities treated at the same rate.

Citrus grown on preplant fumigated soil will be fumigated at a dosage rate of 3 lbs ai/1000 ft³ for 2 hours.

Before fumigation, the citrus will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode method. The citrus will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the citrus will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The citrus will be aerated for a total of 24 hours when additional samples will be withdrawn, again in quadruplicate. The citrus will be subjected to one fumigation only.

Grapefruit, lemons, and oranges having detectable residues of MeBr will be processed into juice.

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours

of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Grapefruit, Lemons, and Oranges

1. The preplant fumigation rate was not specified. According to the Methyl Bromide Registration Standard, deciduous fruits may be planted in soil fumigated at a rate of ≤ 870 lbs ai/A or 2 lb ai/tree site, and fruit are not to be harvested for at least 2 years. DEB understands that it may be difficult to find an adequate supply of cherries which have been treated in accordance with the maximum label rate for preplant use. The preplant data were required by the Methyl Bromide Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to use citrus grown in untreated soil could depend upon the results of studies aimed at determining residue levels in citrus grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in citrus grown in preplant treated soil prove to be insignificant, DEB has no objection to using citrus grown in untreated soil. If bromide levels in citrus grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

For the sake of completeness, levels of MeBr and iBr should be determined in citrus grown in untreated soil.

2. There is no current domestic use for postharvest treatment of citrus except for fumigation for quarantine purposes. The treatment rates given in the PPQ per 1000 ft³ are:

2 lbs ai, 3.5 hours, $\geq 70^{\circ}\text{F}$
1.25 lbs ai, 2 hours, 80-85 $^{\circ}\text{F}$
1.5 lbs ai, 2 hours, 70-79 $^{\circ}\text{F}$
1.75 lbs ai, 2 hours, 64-69 $^{\circ}\text{F}$
2.5 lbs ai, 2 hours, 70-85 $^{\circ}\text{F}$

DEB cannot determine whether the protocol will cover residues on imported citrus because no temperature was specified in the protocol. In order to cover residues in citrus treated for quarantine purposes, it would first be necessary to determine the worst case; the worst case is not immediately obvious because of the variation in the parameters affecting residue levels (dosage rate, exposure time, and temperature). In order to cover residues in citrus treated for quarantine purposes, the worst case would first need to be determined, then the appropriate residue data should be generated.

3. The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of citrus in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.
4. The protocol should specify the fumigation temperature, as the fumigation temperature may affect residue levels. The residue data should reflect the worst case. The label submitted with PP #5F3300 limited fumigation temperatures to $\geq 50^{\circ}\text{F}$.

If lower temperatures may occur during commercial operations and if lower temperatures lead to higher residue levels, MBIP may need to demonstrate that the 50°F restriction is practical and generate the appropriate residue data. If higher temperatures lead to higher residue levels, the residue data should reflect the highest temperatures expected during commercial fumigation. The registrant has the option of narrowing the range of fumigation temperatures in a revised label, if such a restriction is commercially practical.

5. If citrus are stored prior to fumigation, residue data should be generated on citrus which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities. If it can be shown to be practical, the registrant may choose to revise the label to specify the temperature of the citrus to be fumigated and then generate the appropriate residue data.
6. The aeration temperature should be specified. Again, if different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, residue levels), if it can be demonstrated that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be needed.
7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. The size and composition of the containers may affect residue levels in/on the commodities. DEB has learned from R. Brown (Florida Department of Agriculture and Consumer Services) that gift citrus are fumigated in different containers than regular citrus. If a variety of

containers is used commercially, the most commonly used containers should be used to generate the residue data.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of citrus were to be fumigated. Residue data on common commercial cultivars are needed. Data should be generated on Florida and California citrus.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for citrus treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.
12. According to United Fresh Fruit and Vegetables, citrus may be waxed before fumigation. Therefore, residue data on waxed and unwaxed citrus are needed.
13. MBIP will need to support the position that citrus receive only one fumigation commercially.
14. Residue data should reflect treatment of the citrus at the stages of development during which the fruit are fumigated commercially. Sinclair and Lindgren (see Comment #4 under DEB's Comments/Conclusions-Apples) have reported that the amount of fumigant sorbed by a commodity could depend upon its maturity. Residue data on early harvest grapefruit, for example, may be required.
15. Citrus are fumigated in trucks; in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residues may be higher after the fumigation of trucks. (See Conclusion 16 under DEB's Comments/Conclusions-Apples for further details)
16. After consulting with the FDA (telecon with D. Reid, 8/16/88), DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under DEB's Comments/Conclusions-Apples for further discussion)

17. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or that the citrus may not be bagged before the aeration period has elapsed).
18. A storage stability study is needed for each commodity. MBIP may want to store the commodities at dry ice temperatures instead of in freezers. (See Conclusion 19 under Comments/Conclusions-Apples)
19. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

20. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. (see Conclusion 21 under Comments/Conclusions-Apples for further discussion)
21. The registrant needs to submit residue data on dried pulp, peel, oil, and molasses in addition to juice. If infested imported citrus are fumigated, the importer may opt to send the citrus to a processor, if fumigation has an adverse effect on the shelf life of citrus. Also, as long as there is a possibility for processing fumigated citrus into juice, it would appear that it is equally possible to process the citrus into dried pulp, peel, oil, and molasses. Details of the processing should be submitted so DEB can determine whether the processing conforms to commercial practice.
22. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
23. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Grapes

The grapes are to be grown, where possible, on preplant

fumigated soil. If preplant soil fumigated sites cannot be found, the study will use grapes grown on non-preplant fumigated soil. The harvested grapes will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities treated at the same rate.

Grapes grown on preplant fumigated soil will be fumigated at a dosage rate of 4 lbs ai/1000 ft³ for 2 hours.

Before fumigation, the grapes will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode method. The grapes will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the grapes will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The grapes will be aerated for a total of 24 hours when additional samples will be withdrawn, again in quadruplicate. The grapes will be subjected to one fumigation only.

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Grapes

1. The preplant fumigation rate was not specified. The Methyl Bromide Registration Standard has cited the need for residue data reflecting preplant soil fumigation at a rate of 600 lbs ai/A. No residue data reflecting the preplant use are available. The preplant data were required by the Methyl Bromide Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to fumigate grapes grown in untreated soil could depend upon the results of studies aimed at determining residue levels in grapes grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in grapes grown in preplant treated soil prove to be insignificant, DEB has no objection to using grapes grown in untreated soil. If bromide levels in grapes grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

Residue data are required on grapes harvested as soon as possible from treated sites.

For the sake of completeness, the background levels of MeBr and iBr should also be determined in grapes grown in untreated soil.

2. The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit the fumigation of grapes in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.
3. According to the Plant Protection and Quarantine Manual, grapes may be fumigated at the following rates for 2 hours:

1.5 lbs ai/1000 ft³; 80-89°F
2.0 lbs ai/1000 ft³; 70-79°F
2.5 lbs ai/1000 ft³; 60-69°F
3.0 lbs ai/1000 ft³; 50-59°F
4.0 lbs ai/1000 ft³; 40-49°F
2.0 lbs ai/1000 ft³; ≥70°F; 3.5 hours
2.0 lbs ai/1000 ft³; 65-69°F; 4 hours

The proposed protocol would cover residues in imported grapes only if all imported grapes were treated under the conditions used to generate the residue data; i.e., exposure for 2 hours to 4.0 lbs ai/1000 ft³ at some unspecified temperature. In order to cover residues in grapes treated for quarantine purposes, it would first be necessary to determine the worst case; the worst case is not immediately obvious because of the variation in the parameters affecting residue levels (dosage rate, exposure time, and temperature). Then residue data representing the worst case would need to be generated.

4. The protocol should specify the fumigation temperature. The label submitted with PP #5F3300 limited fumigation temperatures to ≥50°F. If lower temperatures may occur during commercial operations and if lower temperatures lead to higher residue levels, MBIP will need to demonstrate that the 50°F restriction is feasible and generate the appropriate residue data. If higher temperatures lead to higher residue levels, the residue data should reflect the highest temperatures expected during commercial fumigation. The registrant has the option of narrowing the range of fumigation temperatures in a revised label, if such a restriction is commercially practical.
5. If grapes are stored prior to fumigation, residue data should be generated on grapes which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities. The

registrant has the option of specifying the commodity temperature in a revised label; he will need to support the argument that such a restriction is practical.

6. The aeration temperature should be specified. Again, if different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can show that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be needed.
7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data. For example, if wax coated fiberboard boxes or expanded polystyrene foam boxes are in commercial use, some data may need to be submitted reflecting the use of these containers.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of grapes were to be fumigated. Residue data on common commercial cultivars are needed.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for grapes treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.

12. MBIP will need to support the position that grapes receive only one fumigation commercially.
13. It has been shown that residue levels in bruised or stemless grapes may be higher for some fumigants. Therefore, the samples should include a representative proportion of bruised and stemless commodities.
14. If grapes are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residues may be higher after the fumigation of trucks. If the registrant contends that grapes are not fumigated in trucks, this position would have to be supported with documentation. (See Conclusion 16 under DEB's Comments/Conclusions-Apples for further details)
15. The sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under DEB's Comments/Conclusions-Apples for further details)
16. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed).
17. A storage stability study is needed for each commodity. The registrant may want to store the samples at -78°C instead of in a freezer (See Conclusion 19 under DEB's Comments/Conclusions-Apples for further details)
18. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

19. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.) and precautions taken to avoid loss of the analyte should be delineated. The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.

20. If infested imported grapes are fumigated, the importer may opt to send the grapes to a processor if fumigation has an adverse effect on the shelf life of the grapes. DEB does not consider it likely that these grapes would be dried to yield raisins, but the registrant needs to submit residue data on wet and dry pomace, raisin waste, and juice; details of the processing should be submitted so DEB can determine if the procedure reflects commercial processing.
21. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
22. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Peaches and Plums

The fruit are to be grown, where possible, on preplant fumigated soil. If preplant soil fumigated sites cannot be found, the study will use fruit grown on non-preplant fumigated soil. The harvested fruit will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities receiving the same treatment.

Peaches and plums grown on preplant fumigated soil will be fumigated at a dosage rate of 3 lbs ai/1000 ft³ for 2 hours.

Before fumigation, the fruit will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode method. The peaches and plums will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the fruit will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The peaches and plums will be aerated for a total of 24 hours when additional samples will be withdrawn, again in quadruplicate. The fruit will be subjected to one fumigation only.

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Peaches

1. The preplant fumigation rate was not specified. According to

the Methyl Bromide Registration Standard, deciduous fruits may be planted in soil fumigated at a rate of ≤ 870 lbs ai/A or 2 lb ai/tree site, and fruit are not to be harvested for at least 2 years. DEB understands that it may be difficult to find an adequate supply of peaches and plums which have been treated in accordance with the maximum label rate for preplant use. The preplant data were required by the Methyl Bromide Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to fumigate fruit grown in untreated soil could depend upon the results of studies aimed at determining residue levels in peaches and plums grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in fruit grown in preplant treated soil prove to be insignificant, DEB has no objection to using fruit grown in untreated soil. If bromide levels in fruit grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

For the sake of completeness, the background levels of MeBr and iBr should also be determined in fruit grown in untreated soil.

2. The dosage to be used in the protocol is 3 lbs ai/1000 ft³ for 2 hours. According to the Methyl Bromide Registration Standard, peaches and plums may be treated at a rate of 5 lbs ai/1000 ft³ for 2 hours. This is the same dosage rate and exposure time proposed in PP #5F3300. If it is MBIP's intent to lower the dosage rate to 3 lbs ai/1000 ft³, all labels reflecting the use of MeBr on peaches and plums need to be revised.

The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of peaches and plums in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.

3. According to the Plant Protection and Quarantine Manual, peaches and plums may be fumigated at the following rates for 2 hours:

1.5 lbs ai/1000 ft³; 80-89°F
2.0 lbs ai/1000 ft³; 70-79°F
2.5 lbs ai/1000 ft³; 60-69°F
3.0 lbs ai/1000 ft³; 50-59°F
4.0 lbs ai/1000 ft³; 40-49°F

The proposed protocol would cover residues in imported peaches and plums only if the imported peaches and plums were treated

under the conditions used to generate the residue data; i.e., 3.0 lbs ai/1000 ft³ at some unspecified temperature for 2 hours.

In order to cover residues in peaches and plums treated for quarantine purposes, it would first be necessary to determine the worst case; the worst case is not immediately obvious because of the variation in the parameters affecting residue levels (dosage rate and temperature). Then residue data representing the worst case would need to be generated.

4. The temperature of the fumigation should be specified. The fumigation temperature may effect the amount of fumigant absorbed by the commodity. The residue data should reflect the worst case. (See Conclusion 4 under DEB's Comments/Conclusions-Apples)
5. If peaches and/or plums are stored prior to fumigation, residue data should be generated on fruit which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities. The registrant has the option of specifying the temperature of the commodities to be fumigated, if it can be demonstrated that this restriction is practical.
6. The aeration temperature should be specified. Again, if different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical.
7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data; for example, if plastic foam collars are used to cradle the fruit during fumigation commercially, then some residue data should reflect this use.

Alternatively, the registrant may provide bridging data which

indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of peaches and plums were to be fumigated. Residue data on common commercial cultivars are needed.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for peaches and plums treated according to the proposed use. Residue data from at least 3 fumigation runs are needed.
12. According to United Fresh Fruit and Vegetables, peaches may be waxed before fumigation. Therefore, residue data on waxed and unwaxed peaches are needed.
13. MBIP will need to support the position that peaches and plums receive only one fumigation commercially.
14. Residue data should reflect treatment of the fruit at the stages of development during which the fruit are fumigated commercially; information in DEB's files indicates that plums are generally picked green and that peaches are picked when the ground color is turning yellow. Sinclair and Lindgren (see Comment #4 under DEB's Comments/Conclusions-Apples) have reported that the amount of fumigant sorbed by a commodity could depend upon its maturity.
15. It has been shown that residue levels for some fumigants may be higher if the fruit are bruised or stemless. Therefore, the samples should include a representative proportion of bruised and stemless commodities.
16. If peaches and plums are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residues may be higher after the fumigation of trucks. If the registrant contends that peaches and plums are not fumigated in trucks, this position would have to be supported with documentation. (See Conclusion 16 under DEB's Comments/Conclusions-Apples for further details)

17. DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under DEB's Comments/Conclusions-Apples for further details)
18. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or that the fruit may not be bagged before the aeration period has elapsed).
19. A storage stability study is needed for each commodity. MBIP may want to store the commodities at -78°C instead of in freezers. (See Conclusion 19 under DEB's Comments/Conclusions-Apples for further details)
20. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

21. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during, pitting, chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.) and precautions taken to avoid loss of the analyte should be delineated. The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.
22. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
23. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Spices and Herbs

Domestic Herbs

Basil, chives, dill, marjoram, and sage (spice group 1) will be harvested from soil which has either been preplant

fumigated with MeBr or which has not been fumigated. Control samples will be taken from herbs grown in treated and untreated soil and analyzed in quadruplicate for residues of MeBr by the King headspace method and for iBr by the ion selective electrode method.

The herbs will be placed in an 885 ft³ atmospheric chamber which has been certified by the California Department of Food and Agriculture. The dosage rate will be 3 lbs ai/1000 ft³ for 24 hours, followed by a 24 hour aeration period. The domestic herbs will be fumigated only once.

Quadruplicate samples of the herbs will be taken for analysis after the 24 hour aeration. The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted.

Each time samples are analyzed, spiked samples will also be analyzed.

Imported Herbs and Spices

Spices will be secured as received into the US and divided into four identical lots. The herbs and spices chosen for investigation are black pepper, chilies, cassia, oregano, celery seed, sesame seed, and nutmeg. These spices were selected to represent spices derived from fruit, bark (cassia), leaf, and seed. Duplicate control samples of each lot will be analyzed for residues of MeBr and iBr as described above.

The spices (excepting the portions set aside as controls) will then be fumigated and aerated as described above. Following the initial fumigation and aeration, the spices will be refumigated and aerated 3 more times for a total of 4 fumigations and aerations.

Triplicate samples from each of the four lots of each commodity will be taken following each of the 4 aeration periods.

Samples will be stored as described above.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions

1. The protocol does not specify the preplant fumigation rate for the domestic herbs. DEB notes that in PP #5F3198, pre-plant soil fumigation use was proposed on a crop group basis at a maximum rate of 300 lbs ai/A. If it is MBIP's intent to fumigate soil at these rates, the herbs should be grown in soil fumigated at the maximum proposed rate.

2. The protocol does not specify whether the herbs (basil, dill, chives, marjoram, and sage) are to be dried before fumigation.

If it is MBIP's intent to fumigate dried herbs only, this should be clear in both the protocol and on the label. If dried leaves are to be used, MBIP will need to describe the drying of the herbs so that DEB can determine whether the drying process reflects common commercial practice. Basil, dill, and chives are often used fresh; when tolerances are established on the herb and spice group, residue data on both fresh and dried herbs are required. Therefore, if it is MBIP's intent to limit fumigation to dried herbs, MBIP will need to revise the label to specify "dried herbs" and spices. Otherwise residue data reflecting the fumigation of fresh herbs would be required.

3. MBIP has not described the physical state of the spices. Residue data are needed on both ground and unground spices.
4. According to the American Spice Trade Association, spices may be fumigated in trucks; in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residue levels may be higher after the fumigation of commodities in trucks.
5. MBIP will need to support the argument that domestic herbs are fumigated only once, and imported herbs and spices are fumigated up to 4 times. [DEB has received a protocol from the American Spice Trade Association; this protocol specifies 3 fumigations for domestic herbs.]

Fumigation practices in the country of origin should also be taken into account.

6. The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of herbs and spices in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.
7. The temperature of the fumigation should be specified. It has been reported in the literature that the storage temperature prior to fumigation may effect the amount of fumigant absorbed by the commodity. The latest proposed label (submitted with PP #5F3300) allows fumigation at $\geq 50^{\circ}\text{F}$. The residue data should reflect the worst case. (See Conclusion 4 under DEB's Comments/ Conclusions-Apples)
8. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

9. The aeration temperature should be specified. If different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be required.
10. MBIP will need to describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

11. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
12. DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion #17 under DEB's Comments/Conclusions-Apples)
13. DEB needs to know whether each lot of a particular spice is to be fumigated separately, i.e., four separate fumigation runs per spice are proposed. If that is the case, DEB has no objection, because several fumigation runs are needed to determine residue levels in spices from the postharvest use. One fumigation run would not serve as an adequate data base for establishing residues arising from the proposed use.
14. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or it will not be packaged before the aeration period

has elapsed).

15. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
16. Storage stability data are needed for each ground and unground spice. MBIP may want to store the commodities at -78°C instead of in freezers. (See Conclusion 19 under DEB's Comments/ Conclusions-Apples for further details)
17. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

18. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during, pitting, chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr.

The present protocol does not call for any maceration; so loss of residues in this step may not pose a problem. However, if it is necessary to process whole nutmegs before adding them to the Waring blender, care should be taken to avoid loss of the volatile analyte, MBIP should delineate the precautions taken to avoid loss of the analyte, and should demonstrate that MeBr is not lost during this process.

19. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Pears

The pears are to be grown, where possible, on preplant fumigated soil. If preplant soil fumigated sites cannot be found, the study will use fruit grown on non-preplant fumigated soil. The harvested fruit will be fumigated at the maximum label rate, which, according to MBIP, will be applicable to imported commodities receiving the same treatment.

Pears grown on preplant fumigated soil will be fumigated at a dosage rate of 3 lbs ai/1000 ft³ for 2 hours.

Before fumigation, the fruit will be analyzed for MeBr by the King headspace method and for iBr by the ion selective electrode

method. The pears will be placed in slatted bins and fumigated in an 885 ft³ chamber certified by the California Department of Food and Agriculture (CDFA).

The chamber will then be opened, and the fruit will be aerated for 3 hours, at which time quadruplicate samples of fruit will be taken for analysis. The pears will be aerated for a total of 24 hours when additional samples will be withdrawn, again in quadruplicate. The fruit will be subjected to one fumigation only.

The samples will be placed in one quart canning jars with screw top lids and stored on dry ice or in a freezer until they can be analyzed. Analysis of the samples will be completed within 18 hours of collection or a storage stability study will be conducted. If MeBr residues have not declined to about 0.01 ppm after 24 hours of aeration, aeration time will be extended so that an aeration period can be recommended.

Each time samples are analyzed, spiked samples will also be analyzed.

DEB's Comments/Conclusions-Pears

1. The preplant fumigation rate was not specified. According to the Methyl Bromide Registration Standard, deciduous fruits may be planted in soil fumigated at a rate of ≤ 870 lbs ai/A or 2 lb ai/tree site, and fruit are not to be harvested for at least 2 years. DEB understands that it may be difficult to find an adequate supply of pears which have been treated in accordance with the maximum label rate for preplant use. The preplant data were required by the Methyl Bromide Registration Standard because there are indications that persistent residues of iBr in soil may result in elevated iBr levels in the crop two years after the preplant treatment.

Whether or not it is acceptable to fumigate pears grown in untreated soil could depend upon the results of studies aimed at determining residue levels in pears grown in fumigated soil, a use which MBIP intends to support. If MeBr and iBr levels in pears grown in preplant treated soil prove to be insignificant, DEB has no objection to using pears grown in untreated soil. If bromide levels in pears grown in treated soil are significant, however, it would be necessary to establish a tolerance which would cover iBr from both preplant and postharvest uses.

For the sake of completeness, the background levels of MeBr and iBr should also be determined in pears grown in untreated soil.

2. The dosage to be used in the protocol is 3 lbs ai/1000 ft³ for 2 hours. According to the Methyl Bromide Registration Standard, pears may be treated at a rate of 5 lbs ai/1000 ft³ for 2 hours. This is the same dosage rate and exposure time

proposed in PP #5F3300. If it is MBIP's intent to lower the dosage rate to 3 lbs ai/1000 ft³, all labels describing the use of MeBr on pears need to be revised.

The label submitted with PP #5F3300 implies that MeBr fumigation in vacuum chambers is permitted. If it is not the registrant's intent to permit fumigation of pears in vacuum chambers, the label needs to be revised to delete this use. Otherwise, residue data reflecting vacuum chamber fumigation are required.

3. According to the Plant Protection and Quarantine Manual, pears may be fumigated at the following rates for 2 hours:

- 1.5 lbs ai/1000 ft³; 80-89°F
- 2.0 lbs ai/1000 ft³; 70-79°F
- 2.5 lbs ai/1000 ft³; 60-69°F
- 3.0 lbs ai/1000 ft³; 50-59°F
- 4.0 lbs ai/1000 ft³; 40-49°F

The proposed protocol would cover residues in imported pears only if all imported pears were treated under the conditions used to generate the residue data; i.e., 3.0 lbs ai/1000 ft³ at some unspecified temperature for 2 hours.

In order to cover residues in pears treated for quarantine purposes, it would first be necessary to determine the worst case; the worst case is not immediately obvious because of the variation in the parameters affecting residue levels (dosage rate and temperature). Then residue data representing the worst case would need to be generated.

4. The temperature of the fumigation should be specified. The fumigation temperature may effect the amount of fumigant absorbed by the commodity. The residue data should reflect the worst case. (See Conclusion 4 under DEB's Comments/Conclusions-Apples)
5. If pears are stored prior to fumigation, residue data should be generated on fruit which have been stored for representative periods at the temperatures used for commercial storage. The temperature at which commodities are stored has been shown to affect residue levels in the commodities. The registrant has the option of specifying the temperature of the commodities to be fumigated, if it can be demonstrated that the restriction is practical.
6. The aeration temperature should be specified. If different temperatures are used commercially than are observed in the residue study, higher residue levels may result. MBIP has the option of revising the label to specify a range of aeration temperatures, if it can be demonstrated that such a label restriction is practical. Residue data reflecting the worst case aeration temperature would then be needed.

7. MBIP needs to further describe the aeration (i.e., whether forced or unforced) and should support the position that this type of aeration is used commercially.

The type of aeration used to generate the residue data should be specified on the label.

8. MBIP will need to more fully describe the containers which hold the various lots. Since the size and composition of the containers may affect residue levels in/on the commodities, MBIP will need to support the position that the containers to be used are representative of containers used in commercial operations. If a variety of containers is used commercially, the most commonly used containers should be used to generate the residue data. For example, if Asian pears are fumigated while cradled by their plastic foam collars, some residue data reflecting the use of these collars are needed.

Alternatively, the registrant may provide bridging data which indicate which type of container represents the worst case, and residue data reflecting the use of that container could be generated. If it is practical to restrict the types of container used during fumigation, the label may be revised so that fumigation is limited to certain types of container; then residue data reflecting the use of those types of containers would need to be generated.

9. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
10. MBIP did not specify which varieties of pears were to be fumigated. Residue data on common commercial cultivars are needed.
11. One fumigation run would not serve as an adequate data base for establishing tolerances for pears treated according to the protocol. Residue data from at least 3 fumigation runs are needed.
13. MBIP will need to support the position that pears receive only one fumigation commercially.
14. Residue data should reflect treatment of the fruit at the stages of development during which the fruit are fumigated commercially; information in DEB's files indicates that pears are generally picked green. Sinclair and Lindgren (see Comment #4 under DEB's Comments/Conclusions-Apples) have reported that the amount of fumigant sorbed by a commodity could depend upon its maturity.
15. It has been shown that residue levels for some fumigants

may be higher if the fruit are bruised or stemless. Therefore, the samples should include a representative proportion of bruised and stemless commodities.

16. If pears are fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks, trailers, or vans should be submitted. Data in DEB's files indicate that residues may be higher after the fumigation of trucks. If the registrant contends that pears are not fumigated in trucks, this position would have to be supported with documentation. (See Conclusion 16 under DEB's Comments/Conclusions-Apples for further details)
17. DEB recommends that the sample selection process should mimic the FDA sample selection process as much as possible. (See Conclusion 17 under DEB's Comments/Conclusions-Apples for further details)
18. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed or that the pears may not be bagged before the aeration period has elapsed).
19. A storage stability study is needed for each commodity. MBIP may want to store the commodities at -78°C instead of in freezers. (See Conclusion 19 under DEB's Comments/Conclusions-Apples for further details)
20. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

21. DEB cautions the registrant that care must be taken to assure that residues of the volatile MeBr are not lost during pitting, chopping and compositing, as such a loss could result in grossly underestimating the levels of MeBr. The sample preparation should be described in detail (temperature, time needed for preparation, etc.) and precautions taken to avoid loss of the analyte should be delineated. The registrant will also need to demonstrate that there is no significant loss of MeBr during the sample preparation.
22. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies

underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.

23. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.

Wheat

A 75,000 bushel lot of hard red spring wheat which has not been previously fumigated will be placed in a flour mill elevator silo. The grain will be fumigated by a commercial applicator at the rate of 3 lbs ai/1000 ft³ for 24 hours. At the end of that time, the vents of the elevator will be opened, and air will be circulated through the grain for 4 hours.

One quart samples will be drawn as the silo is being filled with the untreated grain; the samples will be taken when the silo is 1/4 full, 1/2 full, 3/4 full, and when the silo has been filled with the grain. Throughout the course of the study, the secured samples will be placed in one quart canning jars with screw cap lids and stored in a freezer or on dry ice.

Three lots of 25,000 bushels will be milled. The first 25,000 bushels will represent time zero with regard to aeration. The second 25,000 bushels will be milled after 3 days of aeration, and the last 25,000 bushels will be milled after 7 days of aeration. Quadruplicate samples will be drawn in the following stages of wheat processing: 1) prior to entering the first break roll; 2) below the second break roll; 3) below the third middlings roll; 4) the low grade flour stream; and 5) the patent flour stream. The time of sampling will be spaced so that one sample is collected during each quarter of the mill run. Samples will be shipped to the laboratory by express freight. All residue data will be completed within 24 hours of receipt at the facility or a storage stability study will be conducted.

MeBr will be determined by the King headspace method, and iBr will be determined by the ion selective electrode method.

Each time samples are taken for analysis, spike samples will also be taken and compared to the standard curve.

DEB's Comments/Conclusions-Wheat

1. The label submitted with PP #5F3300 proposed fumigating wheat at a rate of 3 lbs ai/1000 ft³ for 24 hours at a temperature of $\geq 50^{\circ}\text{F}$. The protocol rate corresponds to that proposed use, but DEB cannot judge the adequacy of the

protocol because the fumigation temperature was not specified in the protocol.

The temperature of the fumigation and aeration should be specified. It has been reported in the literature that the storage temperature prior to fumigation may effect the amount of fumigant absorbed by the commodity. (See Conclusion 4 under DEB's Comments/Conclusions-Apples) The registrant will need to determine which temperatures and humidity in commercial practice would constitute the worst case and generate residue data reflecting those conditions.

In the case of wheat, fumigation and aeration would probably be carried out at the temperature at which the wheat is stored. In supporting the contention that wheat would not be fumigated at temperatures less than 50°, the registrant should consider the regions and seasons when fumigation occurs. If the registrant cannot demonstrate that the label restriction limiting application to ≥50°F is practical, residue data reflecting cooler temperatures may be required. Conversely, if higher temperatures result in higher residue levels, residue data reflecting the highest temperatures expected in commercial practice would be required.

2. DEB notes that the PPQ lists the following treatments for imported wheat:

2.5 lbs ai/1000 ft ³ ;	12 hrs;	90°F
3.5 lbs ai/1000 ft ³ ;	12 hrs;	80-89°F
4.5 lbs ai/1000 ft ³ ;	12 hrs;	70-79°F
6 lbs ai/1000 ft ³ ;	12 hrs;	60-69°F
7.5 lbs ai/1000 ft ³ ;	12 hrs;	50-59°F
9 lbs ai/1000 ft ³ ;	12 hrs;	40-49°F
8 lbs ai/1000 ft ³ ;	3 hrs;	≥60°F; 660 mm Hg
9 lbs ai/1000 ft ³ ;	3 hrs;	40-59°F; 660 mm Hg
10 lbs ai/1000 ft ³ ;	3 hrs;	30-39°F; 660 mm Hg

Considering the variations in the parameters affecting residue levels (dosage, temperature, exposure time, and pressure), DEB cannot judge which of the above conditions represents the worst case and whether or not the conditions in the protocol would cover MeBr/iBr residues from the USDA worst case.

3. The residue tests should represent actual commercial fumigation events in all respects, such as MeBr introduction, temperature, humidity, air circulation, packaging, load factor, and aeration and storage conditions. These details should be provided.
4. The protocol calls for only one fumigation run and for 3 processing studies. This protocol would demonstrate the effect that milling has on residue levels of the various fractions, but the results from one fumigation would not provide an adequate data base upon which to establish a tolerance. In generating

the residue data for a raw agricultural commodity and the processed commodities derived from it, the usual practice would involve a number of field trials or, in this case, fumigations. In the case of stored grain, fumigations under a range of conditions are required, in order to determine residue levels as a function of temperature, humidity, load factor, multiple applications, etc. Decline curves are usually generated during these studies.

Once the residue level from fumigation has been determined, a processing study is carried out to determine the concentration factors.

DEB recommends that the registrant incorporate at least 3 fumigation runs into the protocol before conducting the processing study. The fumigations should be designed so as to reflect the worst case commercial situations with regard to residue levels as a function of temperature, humidity, load factor, etc.

5. Fumigated wheat samples should be drawn from all areas of the elevator, composited, and analyzed.
6. For wheat processing, the commodities which need to be analyzed are: wheat/grain, bran, flour, middlings, shorts, and wheat germ.
7. The wheat grain to be analyzed should consist of the raw wheat as it comes from the elevator. The protocol calls for taking samples "prior to entering the first break roll." This description is ambiguous and could apply to wheat which has already been subjected to a separator, aspirator, disc separator, scourer, magnetic separator, washer-stoner, and/or Entoleter®. The wheat may also have been tempered at this point. Data on the raw agricultural commodity are required.
8. MBIP will need to support the position that wheat grain receives only one fumigation commercially. It seems to DEB that the wheat could be treated by each owner of the wheat as it moves through commercial channels. If multiple applications may occur, the residue data will need to reflect the maximum number of applications that wheat would receive as it moves through commercial channels.
9. If wheat is fumigated in trucks, then in addition to using the chamber to generate the residue data, some data reflecting the fumigation of trucks or boxcars should be submitted. Data in DEB's files indicate that residues may be higher after the fumigation of trucks. If the registrant contends that wheat is not fumigated in trucks, this position would have to be supported with documentation. (See Conclusion 16 under DEB's Comments/Conclusions-Apples for further details)

10. DEB reiterates that if tolerances are proposed on the basis of residue levels following a period of aeration, MBIP will need to demonstrate that the aeration period is appropriate (i.e., that the commodity will not be available for sampling by the FDA before the aeration period has elapsed).

The registrant will need to describe the aeration procedure in more detail. Will air be circulated through the grain for 7 days or just for the first 4 hours of aeration time?

11. A storage stability study is needed for each commodity. MBIP may want to store the commodities at -78°C instead of in freezers. (See Conclusion 19 under DEB's Comments/Conclusions-Apples) The storage stability study should cover the period from sampling to analysis.
12. The methods proposed for analysis of MeBr and iBr have already been reviewed by DEB (memo of C. Deyrup, 7/14/88), and the comments in that review still apply. DEB notes three different equations have been submitted to determine ppm iBr.

The registrant will need to resolve these discrepancies and provide sample calculations which illustrate the construction and use of the standard curve and the determination of ppm iBr from the correct equation.

13. Grain dust is collected at elevators and is sold to be added to feed. Residue data on grain dust are needed because data in DEB's files indicate that postharvest treatment of grain may lead to residue levels on grain dust that are much higher than on the grain itself. The apparent concentration of residues in grain dust occurs because of the large surface area of the dust, compared to that of whole grain (Dr. C. Parnell, Texas A & M).

The protocol should be modified to include residue data on grain dust. DEB recommends that at least 2 dust collectors be installed in the flour mill elevator; the use of a cyclone dust collector would ensure that the particles collected would be of the same size and density as commercially generated dust. Grain dust could be collected and analyzed, once an appropriate aeration period has been determined.

14. The nature of the residue in plants after postharvest fumigation is not yet adequately understood. If the metabolism studies underway identify other residues of concern, besides MeBr and iBr, additional residue data may be required.
15. MBIP should heed RCB's comments contained in previous memos and in the Registration Standard regarding the generation of residue data for post harvest use.