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

JUL 21 1994

OFFICE OF
PREVENTION, PESTICIDES AND
TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: Response to the Methyl Bromide Reregistration Standard: Residue Data. (MRID # 43166201, CBRS No. 13428, Barcode No. D200922).

FROM: R. B. Perfetti, Ph.D., Chemist
Reregistration Section 2
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Health Effects Division (7509C)

THRU: William J. Hazel, Ph.D., Section Head
Reregistration Section 2
Chemistry Branch II: Reregistration Support
Health Effects Division (7509C)

TO: Esther Saito, Chief
Reregistration Branch
Special Review & Reregistration Division (7508W)

Attached is a review of residue data submitted in response to the methyl bromide Reregistration Standard. This review was completed by Dynamac Corporation under supervision of CBRS, HED. It has undergone secondary review in the branch and has been revised to reflect Agency policies.

1. The submitted dried fruit and nut study is inadequate and cannot be upgraded because of deficiencies in the methodology. The following deficiencies were noted in the methodology: (i) residue values were calculated using standard curves that were generated months prior to the analysis of the actual residue samples, and concurrent recoveries from fortified controls indicated that the standard curves were no longer valid; (ii) the linear regressions calculated for the low standard curves were skewed by inclusion of an arbitrary zero value; (iii) maximum residue values in nuts exceeded the range of the high standard curve by a factor of 5-9x; (iv) the matrix standard curve for raisins was inappropriately used to determine methyl bromide residues in raisin waste; and (v) only a single sample from each of the duplicate test chambers was analyzed, rather than the two samples per chamber that were specified in the approved protocols. In addition, only 25% of the treated nut

samples were analyzed within one day of collection as specified by the protocols. Over 40 of the 107 samples were held at -10 C for ≥4 days prior to analysis. The available storage stability data on nuts indicate that residues of methyl bromide in nuts declined by ~45% after 7 days of storage at -15 C; data reflecting shorter storage intervals are not available. Given the rapid decline in methyl bromide residues in nuts, storage stability data reflecting the actual storage intervals of the residue samples will be required if samples are held frozen for more than 12 hours.

2. As the deficiencies cited in the methodology cannot be corrected for this study, the registrant must conduct new residue studies depicting the residues of methyl bromide in/on dried fruits and nuts following fumigations with methyl bromide at the maximum rate using the minimum retreatment interval. For guidance on how to conduct the study, the registrant should refer to the previously reviewed protocols (CB No. 7993; 5/24/91) and note the deficiencies cited in this review. In addition, the new residue studies need reflect sampling only at 0, 1 and 3 days postfumigation/-aeration.

A revised Tentative Residue Chemistry Summary sheet is included.

If you need additional input please advise.

Attachment 1: MeBr Residue Data Review.

cc (With Attachment 1): RBP, MeBr Reregistration Standard File, MeBr Subject File, RF, Circ., and Dynamac.

DYNAMAC
CORPORATION
Environmental Services

Final Report

METHYL BROMIDE
Shaughnessy No. 053201
Case No. 0335
(CBRS No. 13428, DP Barcode D200922)

TASK 4
Registrant's Response to Residue Chemistry Data Requirements

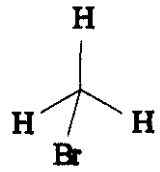
June 21, 1994

Contract No. 68-D2-0053

Submitted to:
U.S. Environmental Protection Agency
Arlington, VA 22202

Submitted by:
Dynamac Corporation
The Dynamac Building
2275 Research Boulevard
Rockville, MD 20850-3268

METHYL BROMIDE



Shaughnessy No. 053201; Case 0335

(CBRS Nos. 13428, DP Barcode D200922)

Task 4

REGISTRANT'S RESPONSE TO RESIDUE CHEMISTRY DATA REQUIREMENTS

BACKGROUND

In response to the Methyl Bromide Residue Chemistry Chapter (3/86), the Methyl Bromide Industry Panel (MBIP) submitted protocols for magnitude of the residue studies on postharvest fumigation of dried fruits and nuts. These protocols have undergone several rounds of review and revision by the Agency (CB No. 5774, 2/22/90, CB No. 6879, 7/30/90, and CB No. 7993, 5/24/91, N. Dodd). In conjunction with the MBIP, the Dried Fruit Association of California and the USDA, ARS have submitted data (1993; MRID 43166201) depicting residues of methyl bromide in/on dried fruits and nuts following postharvest fumigation with methyl bromide. These data are reviewed here to determine their adequacy in fulfilling residue chemistry data requirements. The Conclusions and Recommendations in this document pertain only to the magnitude of the residue in plant commodities.

The qualitative nature of the residue in plants is adequately understood; the residue of concern is methyl bromide *per se* (R. Perfetti, CBRS No. 8601, 9/24/91). The nature of the residue in animals is not adequately understood. Tolerances for residues of methyl bromide in/on food and feed commodities are currently expressed in terms of inorganic bromide [40 CFR §180.123, §180.199 and §185.3480]. However, the Agency has determined that inorganic bromide is not of toxicological concern and is requiring the registrant to propose tolerances for methyl bromide to replace the inorganic bromide tolerances. As there are no Codex MRLs for residues of methyl bromide, there are no questions with respect to Codex/U.S. tolerance compatibility.

An adequate method is available for enforcement of the current tolerances for inorganic bromide and is listed in PAM, Vol. II as Method I. For determining residues of methyl bromide *per se*, a GC/ECD headspace assay method [King et al., *J. Agric. Food Chem.*, 29(5), 1003-1005, 1981] is available for data collection and tolerance enforcement. The limit of detection for methyl bromide is 0.01 ppm. This method has been forwarded to the FDA for inclusion in PAM, Vol. II as Method A.

CONCLUSIONS/RECOMMENDATIONS

1. The submitted dried fruit and nut study is inadequate and cannot be upgraded because of deficiencies in the methodology. The following deficiencies were noted in the methodology: (i) residue values were calculated using standard curves that were generated months prior to the analysis of the actual residue samples, and concurrent recoveries from fortified controls indicated that the standard curves were no longer valid; (ii) the linear regressions calculated for the low standard curves were skewed by inclusion of an arbitrary zero value; (iii) maximum residue values in nuts exceeded the range of the high standard curve by a factor of 5-9x; (iv) the matrix standard curve for raisins was inappropriately used to determine methyl bromide residues in raisin waste; and (v) only a single sample from each of the duplicate test chambers was analyzed, rather than the two samples per chamber that were specified in the approved protocols. In addition, only 25% of the treated nut samples were analyzed within one day of collection as specified by the protocols. Over 40 of the 107 samples were held at -10 C for $\geq$4 days prior to analysis. The available storage stability data on nuts indicate that residues of methyl bromide in nuts declined by ~45% after 7 days of storage at -15 C; data reflecting shorter storage intervals are not available. Given the rapid decline in methyl bromide residues in nuts, storage stability data reflecting the actual storage intervals of the residue samples will be required if samples are held frozen for more than 12 hours.

2. As the deficiencies cited in the methodology cannot be corrected for this study, the registrant must conduct new residue studies depicting the residues of methyl bromide in/on dried fruits and nuts following fumigations with methyl bromide at the maximum rate using the minimum retreatment interval. For guidance on how to conduct the study, the registrant should refer to the previously reviewed protocols (CB No. 7993; 5/24/91) and note the deficiencies cited in this review. In addition, the new residue studies need reflect sampling only at 0, 1 and 3 days post fumigation/aeration.

DETAILED CONSIDERATIONS

Residue Analytical Methods

In conjunction with the methyl bromide magnitude of the residue study, the registrant submitted method descriptions (1993; MRID 43166201) for the analysis of methyl bromide residues in/on dried fruit and nuts. Residues of methyl bromide were determined using the modified King GC/ECD headspace method #93-001. This method was reviewed by the Agency (CB No. 3890, 7/14/88; and CBRS No. 4399, 11/3/88, C. Deyrup) and deemed adequate as an enforcement method for analysis of methyl bromide *per se* on plants.

Briefly, frozen samples are blended with water in a sealed container equipped with a sampling port. Residues are released by allowing the blended sample to equilibrate for 10 minutes at ambient temperature. The headspace is sampled and residues are determined by

GC/ECD. The residues are quantitated by comparison with a matrix standard curve, which is generated through the analysis of fortified control samples of each commodity.

In the current submission, matrix standard curves were generated for each commodity, excluding raisin waste. The matrix standard curves generated for raisins were used to calculate residues in raisin waste. High (1.6-4.8 ppm) and low (0.013-1.6 ppm) standard curves were generated only once for each commodity, except raisins, at the beginning of the sample analyses. For raisins, the registrant generated a second set of standard curves following the first raisin fumigation because of instrument calibration problems. These standard curves were used to calculate methyl bromide residues in all subsequent raisin and raisin waste samples.

The interval between the generation of the matrix standard curves and the final analyses for the related commodities was between 3.4 and 18.7 months, and was typically about 10 months. CBRS notes that the maximum residue values determined for nuts (19.5-35.3 ppm) exceeded the maximum limit of the high standard curve by a factor of up to 9x. In addition, the registrant arbitrarily included a zero point (x=0, y=0) in their regression calculations for low standard curves, thereby skewing the low standard curves.

As indicated in the reviewed protocols (CB No. 7993, 5/24/91, N. Dodd), the registrant analyzed a single control sample fortified at 1.6 ppm concurrently with each analysis to validate the initial standard curves. Concurrent recoveries from the 16 to 34 fortified control samples of each commodity ranged from 38% to 230% (Table 1).

The reported limit of quantitation (LOQ) for each commodity is 0.01 ppm. The LOQ's are defined as the lowest acceptable fortification level in the matrix standard curve. Chromatograms and sample calculations were provided.

Residues of methyl bromide were <0.01 ppm in/on the 10 to 31 control samples of each commodity. Residues of methyl bromide were 0.02 ppm in/on one date and two raisin control samples, and were 0.07-0.91 ppm in/on one to six almond, pecan, pistachio, and walnut control samples. Residues in treated samples were quantitated by comparison to a matrix standard curve. These recoveries were inherently corrected for matrix effects, as residue levels were not quantitated against a solvent standard curve. The sample analyses were performed by the DFA of California, Fresno, CA.

As submitted, the analytical method is inadequate for collecting data on residues of methyl bromide in/on dried fruits and nuts. Although validation of a standard curve using fortified controls is acceptable, the use of a single set of standard curves over a 10-month course of analyses is inappropriate and unacceptable. The large number of recoveries outside the acceptable range (70-120%) indicate that the registrant's assumption that the standard curves were valid throughout the study is not correct. The submitted method also includes the following deficiencies: (i) the linear regressions calculated for the low standard curves were skewed by inclusion of an arbitrary zero value; (ii) maximum residue values in nuts exceeded

the range of the high standard curve by a factor of 5-9x; (iii) the matrix standard curve for raisins was inappropriately used to determine methyl bromide residues in raisin waste; and (iv) a single sample was analyzed in triplicate for each fumigation chamber, rather than 2 to 3 samples per chamber as specified in the reviewed protocols.

Table 1. Concurrent method recovery of methyl bromide from control samples fortified at 1.6 ppm.

Crop Grouping/ Commodity	% Recovery	Number of Samples [a]
Tree Nuts		
Almonds	54-188	29 (17)
Pecans	38-114	16 (6)
Pistachios	54-191	28 (9)
Walnuts	38-230	34 (21)
Dried Fruit		
Dates	51-172	16 (6)
Figs	61-140	25 (4)
Prunes	77-168	20 (3)
Raisins	69-169	24 (11)
Raisin waste	54-208	31 (21)

[a] Number of fortified control samples with recoveries outside of the acceptable 70-120% range are listed parenthetically.

Storage Stability Data

The Agency (CB No. 6879, 7/30/90, N. Dodd) has concluded that residues of methyl bromide in/on raw agricultural commodities are stable when samples are stored on dry ice for up to 12 hours, and that storage stability data are necessary only for samples stored in excess of 12 hours.

In the current submission (1993; MRID 43166201), the registrant presented data on the stability of methyl bromide residues in nuts and dried fruits stored at -15 C. To determine storage stability, the registrant fumigated almonds, walnuts, raisins, and prunes at 3.5 lb ai/1,000 ft^3 for 24 hours. Following a 24-hour aeration period, samples were collected and subsamples were immediately analyzed. The remaining samples were reanalyzed after 1, 2, 3, and 4 weeks of storage at -15 C. The storage stability data (Table 2) indicate that residues of methyl bromide are stable in dried fruits for up 4 weeks; however, residues are not stable in nuts. Residues declined by approximately 45% in nuts after 1 week of storage at -15 C, and declined by approximately 70% after 4 weeks of storage at -15 C.

As residues levels in the 0-day samples will serve as the basis for determining appropriate tolerance levels, storage data on these samples are critical. In the current submission, 0-day samples of dried fruits and nuts were stored at -10 C until analysis. Dried fruit samples (0-day) were analyzed within 1-14 days (Table 3), and nut samples (0-day) were analyzed within 1-9 days.

The available storage stability data support the residue data for dried fruits; however, additional storage stability data are required to support the current tree nut data. Because the majority of nut samples were analyzed within 1 week and methyl bromide levels in nuts decline appreciably (45%) within 1 week, additional data are required depicting the decline in methyl bromide residues in frozen nuts following shorter storage intervals so that residue values can be corrected accordingly.

Table 2. Frozen storage stability of methyl bromide in nuts and dried fruits.

	Methyl Bromide Residues (ppm) [a]				
	0	1 week	2 weeks	3 weeks	4 weeks
Almonds	40.6 -	24.0 (59%) [b]	22.4 (55%)	13.3 (33%)	10.8 (27%)
Walnuts	68.1 -	37.8 56%)	37.0 (54%)	26.5 (39%)	24.6 (36%)
Raisins (processed)	2.5 -	2.3 (92%)	2.0 (80%)	1.9 (76%)	2.1 (84%)
Prunes (unprocessed)	1.2 -	1.1 (92%)	0.85 (71%)	0.81 (68%)	1.1 (92%)

[a] Data are the mean of triplicate analyses of single subsamples.
[b] Percent recoveries are listed parenthetically.

Magnitude of the Residue in Plants and Processed Food/Feed Commodities

Postharvest fumigation of dried fruits and nuts. In conjunction with the MBIP, the Dried Fruit Association of California and the USDA, ARS have submitted data (1993; MRID 43166201) depicting residues of methyl bromide in/on dried fruits and nuts following repeated postharvest fumigations with methyl bromide. Protocols for these studies have undergone several rounds of review and modification by the Agency (CB No. 5774, 2/22/90, CB No. 6879, 7/30/90, and CB No. 7993, 5/24/91, N. Dodd).

Two methyl bromide end-use products (100% PrGs, EPA Reg. Nos. 5785-11 and -41) are currently registered for postharvest fumigation of dried fruits and nuts. Maximum labeled single application rates for dried fruits and nuts are 1 and 3.5 lb ai/1000 ft^3, respectively, for 24 hours. The maximum number of fumigations allowed and the minimum retreatment intervals are not specified.

In the current submission, commercially obtained dried fruits (unpitted dates, figs, pitted prunes, raisins, and raisin waste) and nuts (almonds, pecans, pistachios, and walnuts) were packed in both bulk cases (industrial) or in retail consumer packs for fumigation. Dried fruit were fumigated with methyl bromide at 1.5 lb ai/1,000 ft^3 for 24 hours at 10 C, and nuts were fumigated with methyl bromide at 3.5 lb ai/1,000 ft^3 for 24 hours at 15 C. Packaged fruit and nut samples were each fumigated only once simulating commercial practices. Bulk samples received multiple fumigations in accordance with the standard commercial practices for each commodity. The repeated fumigations of bulk samples were as follows: dates, 8 fumigations at 18- to 57-day intervals; figs, 3 fumigations at ~28-day intervals; prunes, 3 fumigations at ~34-day intervals; raisins, 10 fumigations at 23- to 112-day intervals; raisin waste, 5 fumigations at 29- to 67-day intervals; almonds, 5 fumigations at 21- to 68-day intervals; pecans, 2 fumigations at a 55-day interval; pistachios, 5 fumigations at 25- to 48-day intervals; and walnuts, 4 fumigations at ~90-day intervals.

Fumigations of dried fruits and nuts were conducted at normal atmospheric pressure (NAP) in 28.3 liter test chambers and samples occupied 30% of the chamber capacity, as specified in the approved protocols (CB No. 7993, 5/24/91, N. Dodd). CBRS has previously concluded that residue data from NAP fumigations will be sufficient to cover vacuum fumigations as residues resulting from vacuum fumigations would be lower. Each commodity was fumigated in duplicate test chambers. The concentration of methyl bromide in each chamber was monitored during fumigation. After each 24 hour fumigation, the chambers were forced-air vented (3-5 hours) until the concentration of methyl bromide was $\leq$5 ppm at which time the 0-day samples were collected. Samples were collected periodically post-treatment for a minimum of 5 sampling intervals.

For each commodity, a single sample was collected from each test chamber, frozen, and shipped to the analytical laboratory. All samples were stored at -10 C until analysis, and each sample was analyzed in triplicate or quadruplicate. The approved protocols specified that two to three samples from each chamber were to be analyzed. Sample storage intervals

are presented in Table 3 and were determined by the reviewer using the sample shipping forms and the dates of analysis from chromatograms.

Residues of methyl bromide in/on dried fruits and nuts were determined using the modified King headspace method (Method #93-001) described in the Residue Analytical Methods section. The validated LOQ for methyl bromide in dried fruits and nuts is 0.01 ppm.

Apparent residues of methyl bromide were <0.01 ppm in/on 10 to 31 control samples of each commodity, and were 0.02 ppm in/on one date and two raisin control samples. Apparent residues of methyl bromide were also detected at 0.07-0.91 ppm in/on one to six control samples of almonds, pecans, pistachios, and walnuts. Residues of methyl bromide in/on treated samples of each commodity are presented in Table 3.

The submitted dried fruit and nut study is inadequate and cannot be upgraded because of deficiencies in the methodology. The following deficiencies were noted in the methodology: (i) residues values were calculated using standard curves that were generated months prior to the analysis of the actual residue samples, and concurrent recoveries from fortified controls indicated that the standard curves were no longer valid; (ii) the linear regressions calculated for the low standard curves were skewed by inclusion of an arbitrary zero value; (iii) maximum residue values in nuts exceeded the range of the high standard curve by a factor of 5-9x; and (iv) the matrix standard curve for raisins was inappropriately used to determine methyl bromide residues in raisin waste. In addition, the registrant only analyzed a single sample from the each of the duplicate test chambers, rather than the two samples per chamber that were specified in the approved protocols.

The registrant also stated in the approved protocols that nut samples would be analyzed within 24 hours of sampling. However, only 27 out of the 107 treated nut samples were analyzed within 1 day of sampling, and over 40 samples were held for ≥4 days prior to analysis. The available storage stability data for nuts indicates that residues of methyl bromide in nuts declined by ~45% after 7 days of storage at -15 C; data reflecting shorter storage intervals are not available. Given the rapid decline in methyl bromide residues in nuts, storage stability data reflecting the actual storage intervals of the residue samples will be required if samples are held frozen for more than 12 hours.

As the deficiencies cited in the methodology cannot be corrected for the current submission, the registrant must conduct new residue studies depicting the residues of methyl bromide in/on dried fruits and nuts following fumigations with methyl bromide at the maximum rate using the minimum retreatment interval. For guidance on how to conduct the study, the registrant should refer to the previously reviewed protocols (CB No. 7993; 5/24/91) and note the deficiencies cited in this review. In addition, the new residue studies need reflect sampling only at 0, 1 and 3 days post fumigation/aeration.

Table 3. Residues in dried fruit and nut commodities following fumigation with methyl bromide at ~1x and in-chamber

Table 3 (*continued.*)

aeration.

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Dates (bulk)	1.5	24	10	0	2.6, 2.3	2
1st fumigation				3	1.4, 1.2	1
				7	0.16, 0.09	2
				13	0.01, 0.01	2
				15	<0.01, <0.01 [d]	1
4th fumigation	1.5	24	10	0	4.0, 3.7	2
				3	2.3, 2.2	1
				7	0.39, 0.40	7
				13	0.13, 0.07	1
				17	0.02, <0.01	1
8th fumigation	1.5	24	10	0	5.5, 4.5	3
				3	1.9, 2.0	4
				7	1.1, 0.79	6
				18	0.24, 0.16	5
				28	<0.01, <0.01	5
Dates (packaged)	1.5	24	10	0	4.1, 4.7	5
				3	2.1, 2.9	4
				14	0.25, 0.39	2
				17	0.09, 0.14	4
				30	0.01, <0.01	1
Figs, dried (bulk)	1.5	24	10	0	3.6, 3.7	1
1st fumigation				3	0.63, 0.76	3
				7	0.17, 0.22	6
				14	0.08, 0.07	5
				21	<0.01, <0.01	1
2nd fumigation	1.5	24	10	0	4.2, 4.5	1
				3	1.5, 1.0	5
				7	0.51, 0.41	1
				14	0.13, 0.18	1
				21	0.03, 0.05	6
				26	<0.01, <0.01	6
3rd fumigation	1.5	24	10	0	4.5, 4.5	2
				3	0.86, 0.81	2
				7	0.41, 0.67	3
				14	0.22, 0.20	2
				21	0.07, 0.03	2
				28	0.03, 0.04	1
				38	0.06, 0.01	1
				45	0.01, <0.01	5

(continued; footnotes follow)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Figs (packaged)	1.5	24	10	0	0.72, 1.9	6
				3	2.5, 0.29	4
				14	0.47, 0.79	2
				17	0.39, 0.25	4
				30	0.09, 0.02	4
				34	0.04, 0.02	1
				37	<0.01, <0.01	16
Prunes (bulk) 1st fumigation	1.5	24	10	0	7.3, 7.1	2
				3	3.4, 3.1	1
				7	0.89, 0.76	1
				14	0.15, 0.12	1
				23	0.04, 0.03	1
				27	<0.01, <0.01	7
2nd fumigation	1.5	24	10	0	7.2, 7.1	2
				3	4.0, 3.9	1
				7	0.36, 0.49	2
				14	0.17, 0.23	2
				21	0.05, 0.03	2
				25	<0.01, <0.01	3
3rd fumigation	1.5	24	10	0	7.1, 7.4	3
				3	4.7, 4.2	2
				7	1.1, 1.2	1
				14	0.19, 0.33	5
				21	0.01, 0.01	4
Prunes (packaged)	1.5	24	10	0	7.1, 6.9	2
				3	5.0, 4.8	3
				14	0.26, 0.33	2
				17	0.08, 0.04	4
				30	<0.01, <0.01	1
Raisins (bulk) 1st fumigation	1.5	24	10	0	1.7, 2.3	2
				3	1.4, 1.5	1
				7	0.55, 0.58	1
				15	0.03, 0.02	1
				17	0.02, 0.01	4
5th fumigation	1.5	24	10	0	5.22, 5.6	2
				3	1.94, 2.02	1
				7	1.42, 1.06	2
				14	0.45, 0.60	1
				21	0.28, 0.39	1
				36	0.05, 0.07	1
				50	<0.01, 0.01	1

(continued; footnotes follow)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Raisins (continued) 6th fumigation	1.5	24	10	0	3.9, 4.3	14
				3	2.0, 2.0	12
				14	0.55, 0.52	2
				28	0.12, 0.16	3
				50	0.03, 0.03	1
				64	<0.01, <0.01	2
10th fumigation	1.5	24	10	0	6.5, 4.5	ND [e]
				3	4.9, 4.6	2
				8	1.1, 1.5	6
				20	0.05, 0.21	ND
				27	0.01, 0.03	1
				33	<0.01, <0.01	3
Raisins (packaged)	1.5	24	10	0	5.9, 5.9	2
				10	1.2, 1.1	1
				38	0.07, 0.10	6
				48	0.01, 0.02	1
				58	<0.01, 0.01	6
Raisin Waste 1st fumigation	1.5	24	10	0	7.3, 7.5	5
				3	0.73, 0.92	4
				7	0.71, 0.70	1
				12	0.24, 0.17	9
				14	0.22, 0.16	7
				24	0.22, 0.30	4
				28	0.14, 0.14	12
				35	0.07, 0.05	27
				40	<0.01, 0.03	23
2nd fumigation	1.5	24	10	0	9.6, 10.1	8
				3	5.8, 5.4	5
				7	1.37, 1.58	6
				21	<0.01, <0.01	3
				34	<0.01, <0.01	1
3rd fumigation	1.5	24	10	0	4.3, 4.4	4
				3	5.3, 5.2	2
				7	1.3, 1.3	6
				14	<0.01, 0.01	6
				25	0.09, 0.13	2

(*continued; footnotes follow*)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Raisin waste (continued) 4th fumigation	1.5	24	10	0	7.1, 4.5	4
				3	5.0, 5.6	3
				7	2.2, 2.4	2
				14	0.64, 0.31	2
				26	0.11, 0.04	1
				35	0.12, 0.10	1
				55	<0.01, <0.01	13
5th fumigation	1.5	24	10	0	6.33, 4.9	1
				3	2.5, 4.8	7
				7	0.18, 0.49	4
				21	0.02, 0.13	ND
				33	<0.01, <0.01	ND
Almond, nutmeats (bulk) 1st fumigation	3.5	24	15.6	0	27.8, 27.5	2
				3	18.6, 17.9	1
				7	2.5, 2.8	2
				14	0.24, 0.21	1
				21	0.09, 0.03	3
3rd fumigation	3.5	24	15.6	0	28.7, 28.7	1
				3	16.8, 16.7	3
				7	14.5, 14.0	1
				14	1.38, 1.77	16
				31	0.06, 0.04	15
4th fumigation	3.5	24	15.6	0	35.3, 32.7	1
				3	27.8, 27.4	1
				7	16.4, 17.1	5
				14	4.67, 4.57	5
				28	0.26, 0.26	2
				36	0.13, 0.12	11
				57	<0.01, <0.01	6
5th fumigation	3.5	24	15.6	0	29.2, 29.4	2
				3	20.9, 21.4	6
				8	9.64, 9.95	2
				14	3.7, 3.2	5
				21	0.65, 0.66	2
				28	0.30, 0.26	2
				35	0.03, 0.06	2
Almond, nutmeats (packaged)	3.5	24	15.6	0	0.53, 0.77	7
				3	0.32, 0.35	4
				14	1.22, 1.25	1
				30	0.02, 0.03	11
				40	<0.01, <0.01	3

(*continued; footnotes follow*)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Pecan, nutmeats (bulk) 1st fumigation	3.5	24	15.6	0	22.9, 26.1	9
				3	12.5, 11.5	7
				14	0.37, 0.35	1
				24	0.19, 0.02	5
				30	<0.01, <0.01	13
2nd fumigation	3.5	24	15.6	0	22.7, 19.1	6
				3	7.1, 6.2	19
				16	0.17, 0.23	7
				24	<0.01, <0.01	1
				30	0.88, 0.75	<1
				45	<0.01, <0.01	13
Pecan, nutmeats (packaged)	3.5	24	15.6	0	0.44, 0.02	6
				3	<0.01, <0.01	21
				24	<0.01, <0.01	1
				30	<0.01, <0.01	1
				37	0.14, 0.03	1
Pistachio, nutmeats (bulk) 1st fumigation	3.5	24	15.6	0	13.4, 14.9	1
				14	0.88, 1.4	1
				21	2.58, 0.16	2
				28	<0.01, 0.03	2
				31	0.01, 0.10	4
3rd fumigation	3.5	24	15.6	0	17.7, 19.5	5
				3	11.9, 11.9	3
				14	0.73, 0.39	2
				21	0.06, <0.01	3
				28	0.01, <0.01	8
5th fumigation	3.5	24	15.6	0	17.9, 17.8	1
				3	10.6, 9.5	6
				8	3.0, 1.8	2
				14	1.14, 0.40	5
				21	0.20, 0.20	2
				28	0.10, 0.17	2
				35	0.01, 0.04	2
Pistachio, nutmeats (packaged)	3.5	24	15.6	0	13.7, 14.9	2
				4	6.9, 7.6	3
				7	5.0, 7.1	2
Test 1 [f]				28	0.06, 0.09	4
				31	0.86, 0.29	17

(*continued; footnotes follow*)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Pistachio, nutmeats (packaged)	3.5	24	15.6	0	17.9, 18.5	4
				3	12.5, 13.6	4
				7	8.0, 8.0	5
Test 2 [f]				14	1.5, 2.3	5
				30	0.11, 0.15	3
				40	0.02, <0.01	3
Walnut, nutmeats (bulk) 1st fumigation	3.5	24	15.6	0	24.6, 24.0	2
				3	19.2, 19.6	1
				7	15.8, 15.9	1
				14	10.7, 10.6	1
				28	2.24, 2.58	1
				67	0.17, 0.24	3
				74	0.09, 0.12	13
2nd fumigation	3.5	24	15.6	0	22.0, 24.0	3
				3	17.6, 18.6	8
				14	9.37, 8.46	1
				30	1.44, 1.55	5
				74	0.02, 0.03	5
3rd fumigation	3.5	24	15.6	0	23.5, 23.7	1
				3	17.0, 16.6	6
				8	10.8, 11.0	2
				14	5.0, 5.3	2
				28	2.2, 2.2	2
				74	0.16, 0.15	1
				80	0.12, 0.13	2
				85	0.08, 0.11	1
4th fumigation	3.5	24	15.6	0	26.1, 26.0	5
				3	20.7, 18.8	7
				14	7.9, 6.7	6
				28	1.39, 1.31	7
				60	0.48, 0.36	3
				84	0.12, 0.11	2
				90	0.08, 0.06	2

(*continued; footnotes follow*)

Table 3 (*continued.*)

Commodity	Application Data			Sampling Interval [a] (days)	Methyl Bromide Residues (ppm) [b]	Storage Interval [c] (days)
	Rate (lb ai/ 1000 ft^3)	Fumigation Interval (hrs)	Commodity Temp. (C)			
Walnut, nutmeats (packaged)	3.5	24	15.6	0	14.6, 15.6	<1
				3	15.4, 16.0	3
				12	7.1, 7.9	1
				35	0.33, 0.44	5
				45	0.15, 0.29	1
				55	1.38, 0.97	4
				60	0.03, 0.14	14

[a] The sampling interval represents the interval between completion of specified aeration to sampling of the commodity.
[b] Each value is the average of triplicate analyses of a single sample.
[c] The sample storage interval is the period that samples were stored at -15 C prior to analysis; this interval was calculated by the reviewer using sample shipping dates and the dates of analysis on chromatograms.
[d] The reported LOQ is 0.01 ppm for each commodity.
[e] ND = No data available.
[f] The submission contained two sets of data from a single fumigation of packaged pistachios nutmeats.

MASTER RECORD IDENTIFICATION NUMBERS

The citations for the MRID documents used in this review are presented below.

43166201 McKinney, J. (1993) Methyl Bromide Dried Fruit and Nuts Residue Study. Report ID. No. DFTN-1. Unpublished study prepared by USDA Agricultural Research Service and the Dried Fruit Association of California. 4318 p.

AGENCY MEMORANDA CITED IN THIS DOCUMENT

CB No: 3890
Subject: Follow-up to Methyl Bromide Registration Standard. Post Harvest Protocol, Interim Plant Metabolism Report, Analytical Methods, and Storage Stability.
From: C. Deyrup
To: J. Kempter
Date: 7/14/88
MRID(s): 40579501, 40607801, and 40618501.

CB No.: 4399
Subject: Follow-up to Methyl Bromide Registration Standard. Methyl Bromide Industry Panel Response (9/22/88) to DEB Review of 7/14/88 on Postharvest Protocol, Analytical Methodology, and Storage Stability.
From: C. Deyrup
To: J. Kempter
Date: 11/3/88
MRID(s): None.

CB No.: 5774
Subject: Methyl Bromide Reregistration Standard Follow-up. Protocols for Postharvest Fumigation of Dried Fruits and Nuts.
From: N. Dodd
To: W. Francis and L. Rossi
Date: 2/22/90
MRID(s): None.

CB No.: 6879
Subject: Methyl Bromide Reregistration Letter and Attachments from the Methyl Bromide Industry Panel Dated 5/25/90.
From: N. Dodd
To: W. Francis
Date: 7/30/90
MRID(s): None.

CB No.: 7993
Subject: Methyl Bromide Protocol for Postharvest Fumigation of Dried Fruits and Nuts.
From: N. Dodd
To: W. Francis and W. Francis
Date: 5/24/91
MRID(s): None.

CBRS No.: 8601
Subject: Methyl Bromide Industry Panel: Response to the Methyl Bromide Reregistration Standard: Metabolism Study.
From: R. Perfetti
To: W. Burnam and L. Rossi
Date: 9/24/91
MRID(s): None.