US EPA ARCHIVE DOCUMENT

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

(85)

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

JUL 31 1989

Ms. Alice Marcotte
Technical Editing Group (HFF-40)
Food and Drug Administration
200 C Street, S.W.
Washington, DC, 20204

Dear Ms. Marcotte,

Enclosed are materials for a new entry in PAM II. The subject compound is methyl bromide per se, for which tolerances have not yet been established. Only the inorganic bromide residues have been regulated in the past.

Regulatory authorities are in need of a monitoring method for methyl bromide, despite the lack of established tolerances for this residue, because as one of the few remaining fumigants, it is registered for use on virtually every raw agricultural commodity and processed product.

A previous submission sent by W.J. Hazel on 6/5/87, described a headspace method for the analysis of methyl bromide (Method I). The present submission, Method A, describes further modifications of the headspace method.

If there are any questions regarding the enclosed materials, please contact me at (FTS) 557-1681.

Sincerely,

Cynthia Deyrup

Cynthia Deyrup
OPP/HED/DEB (H7509C)
U.S. Environmental Protection Agency
401 M St. SW
Washington DC, 20460

Attachments: 1. PAM II Cover Sheet
2. Methyl Bromide Industry Panel modification of King et al method

cc (FDA letter plus attachments 1-2): E. Eldredge (ISS/ISB/PMSD), M. Bradley (DEB)

cc with Attachment 1: Deyrup (DEB), Thompson (RTP-NC), EAB, Dean Hill (NEIC/Denver), E. Claussen (WH-562-B), Methyl bromide SF, Methyl Bromide Registration Standard File-Boodee

cc without Attachments: RF, Circu

RDI:D. Edwards:7/25/89:R. A. Loranger:7/25/89
TS-769:CM#2:RM810:X7484:C. Deyrup:cd:7/31/89

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Attachment 1

PAM II Cover Sheet

Acceptable Name: Methyl Bromide

Pesticide Reg. Section: No tolerances yet established for the parent compound.

Structure: CH_3Br

Other Names: Bromomethane, monobromomethane, Embafume®, Brom-O-Gas®, Celfume®, Meth-O-Gas®, Terr-O-Gas®

Petitioner: Methyl Bromide Industry Panel
L. Vernon White, Chairman
Great Lakes Chemical Corp.
P.O. Box 2200
West Lafayette, IN, 47906

Method: King, J.R., C.A. Benshoter, and A.K. Burditt. 1981. Residues of methyl bromide in fumigated grapefruit determined by a rapid, headspace assay. J. Agric. Food Chem., 29(5):1003-1005 (with modifications made by the Methyl Bromide Industry Panel).

Pesticide Petitions: PP #5F3300, PP #5F3198, Methyl Bromide Registration Standard Follow-up

Product Application: Various Commodities

Detection Limit: 0.002 ppm

Attachment 2

LABORATORY PROCEDU: 3 DATE: 12/1/88

METHOD 1

MeBr by Headspace GC
Modified King Headspace Method (1)

1. Place open blender jar in a MeBr-free environment.

2. Chop up commodity to be analyzed. Weigh 50.00 g (± 0.20 g) of commodity into weighing dish. Transfer contents of dish to blender jar (see Figure 1 and 2). Add distilled H_2O according to the following chart. Seal Jar.

AMOUNT OF H_2O ADDED TO COMMODITY TYPE*

Commodity Type	Amount of H_2O (ml)
Fresh Fruits and Vegetables	100
Lettuce	150
Broccoli, Cauliflower	150
Potatoes	100
Dried: Nuts, Wheat, Corn	200
Walnuts (10g)	100

* For commodities not listed, amount of water established by experimentation.

3. Blend commodity and water for 3 minutes at high speed using Waring Blender (Waring 7010 Model 31BL91).

4. After blending, allow sample to sit for 17 minutes in a 26° (80°F) water bath (liquid in bath should not rise above lid on jar).

5. Set Column temperature to 100° C and set detector temperature to 300°C. Column flow rate should be around 20 ml/min. Check instrument parameters (see attachment).

6. Using tubing connected to an Ultra zero grade air outlet, purge sample syring 3 to 5 times with air.

7. Collect 20 ml of ultrapure air into syringe. Inject this into blender jar. Pump syring 4 to 5 times to mix vapors well. Extract sample (allow internal jar pressure to push plunger up on syringe) and load sample into sample loop.

8. Set run table to inject sample onto column (see instrument parameters)

9. Test the samples of commodity immediately upon receipt. Order of analysis of samples: treated commodity (4 replicate samples); control commodity (4 replicate samples); and standard curve/recovery (minimum of 4 pts).

10. Prepare standard curve samples using a composite of control samples. Prior to blending sample, withdraw 20 ml of headspace from jar (do not discard). Add appropriate amount of methyl bromide vapor to jar with syringe. Satisfy vacuum in jar with removed headspace. Proceed with steps 3-8..

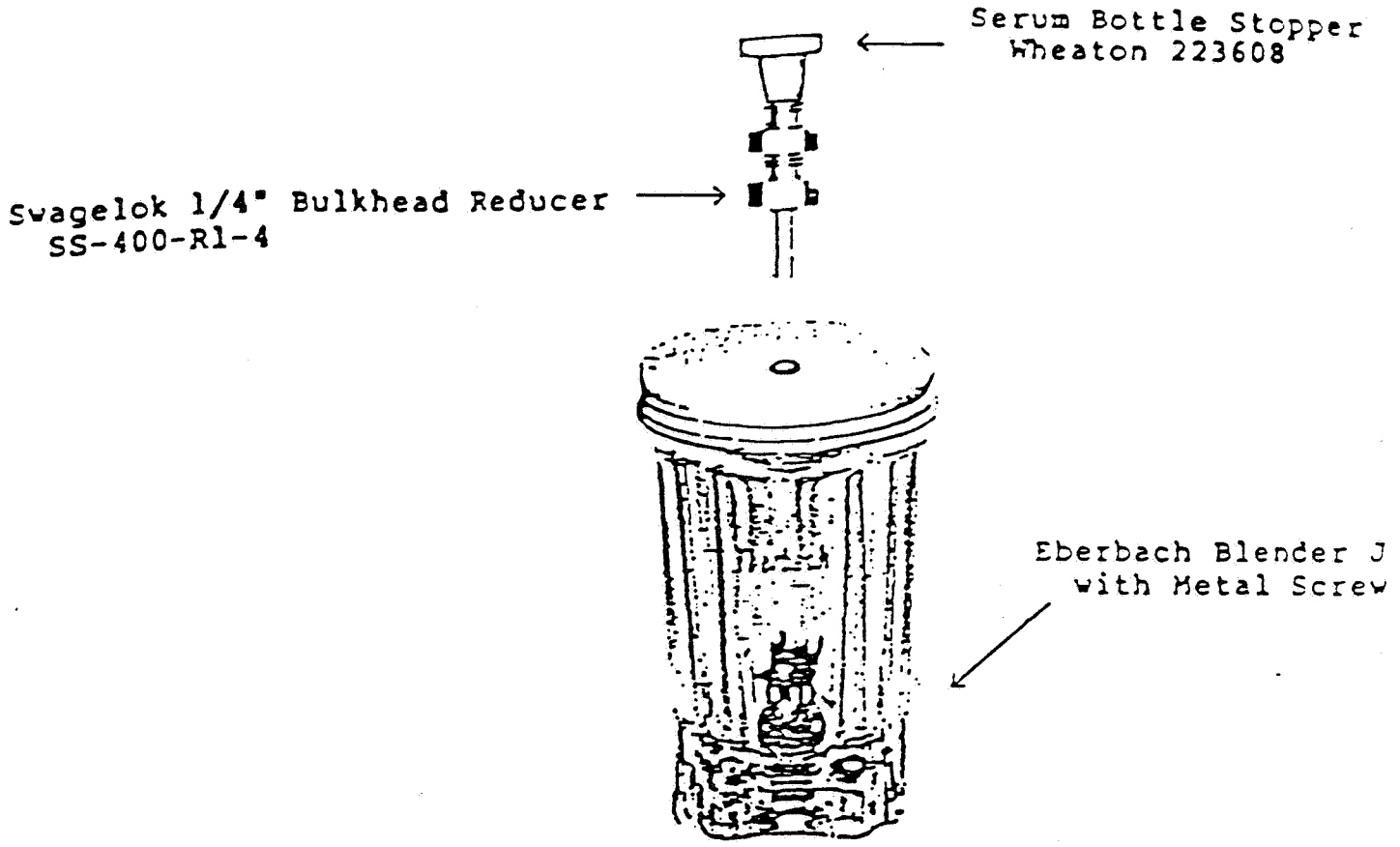

Figure 1: Blender Jar Assembly

Lid of the blender jar is lined with 2 pieces of 1/8" Neoprene rubber prior to inserting bulkhead union. A small piece of neoprene was made into a gasket to seal the top of the lid opening. Washers are placed on the outside of the neoprene sheets around the bulkhead union to provide a good seal. (See figure 2.)

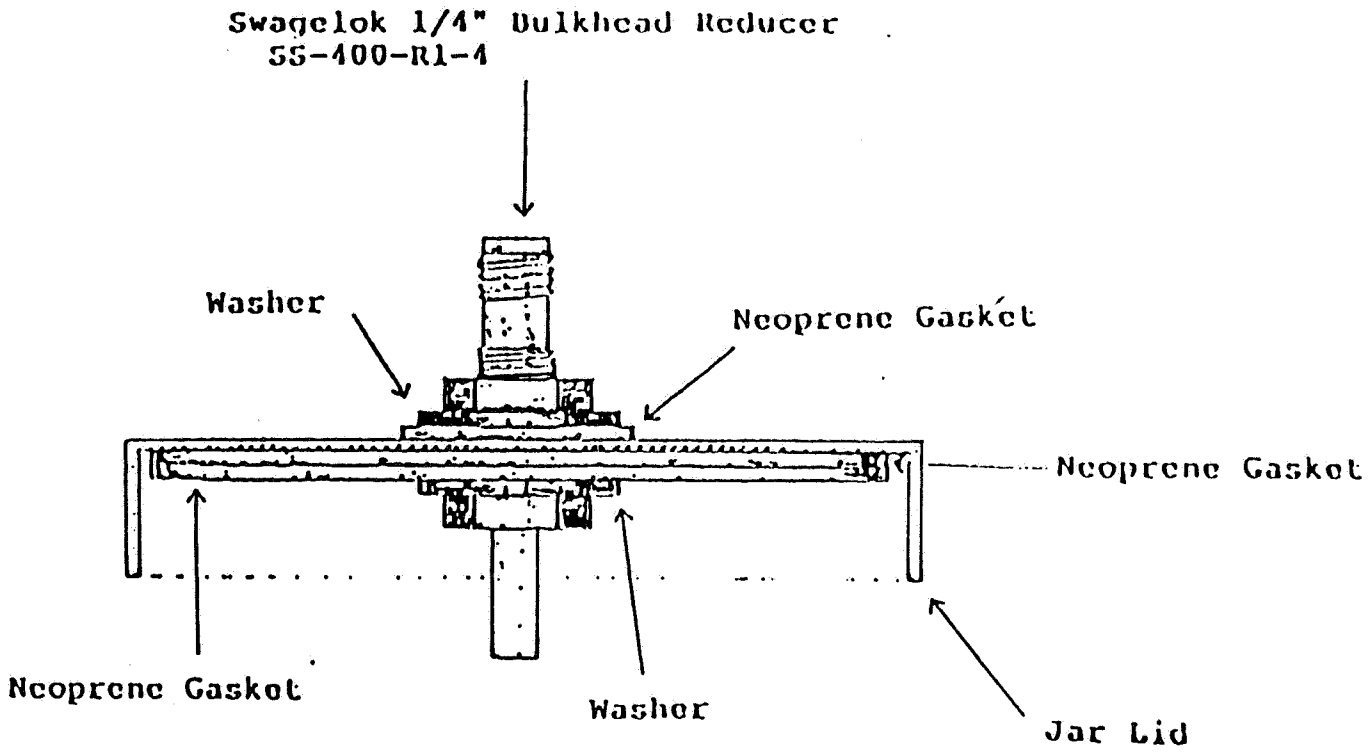

Figure 2: Exploded View of Blender Jar Lid Assembly

INSTRUMENT PARAMETERS

Hewlett Packard 5880

Instrument Parameters:

Column Type:	Poropak GSQ 30m x 540 um
Oven Temperature:	70°C to 100°C (dependent on commodity)
Detector Type:	Ni-63 Electron Capture Detector (ECD)
Detector Temperature:	300° C
Injector Type:	Gas Sampling Valve (GSV); 1cc loop
Injector Temperature:	Oven temperature + 5° C
Flow Rates and Type:	
Carrier:	20 ml/min; Zero grade N_2
Attenuation:	2 ↑ 6
Run Table	0.10 Valve 1 off 8.00 Stop