PLANT QUARANTINE AND FUMIGATION OF IMPORTED CEREALS IN JAPAN

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ABSTRACT:
In 1982, more than 30 million tonnes of cereals were imported into Japan. As much as 60% of these imports were fumigated at the port of entry, either in the silo, warehouse, container or barge, in order to prevent the entry of injurious insect pests. Remarkable progress has been achieved since 1950 in techniques of cereal fumigation with methyl bromide and phosphine for "quarantine fumigation". Most silos and warehouses that are currently approved by the Plant Protection Station are sufficiently gas tight to be suitable for fumigation. The high level of sealing results from the development of suitable construction and sealing techniques.

Practical aspects of quarantine fumigation in Japan and Japanese research on factors affecting fumigation and fumigant residues in cereals are described. Japanese quarantine fumigation schedules are given. Dosage-response data for methyl bromide treatment of the most tolerant stages of several stored product pests is summarised.

HISTORY AND SYSTEM OF PLANT QUARANTINE SERVICE

Plant Quarantine Service in Japan was initiated in 1914 by the promulgation of the Plant Quarantine Law. At first only live plants were inspected. With the increase in international exchange of agricultural products, the objects of inspection have been expanded. Import quarantine of cereals was initiated in 1950 under the current Plant Protection Law.

At present, the Plant Quarantine Service in Japan is carried out by the Plant Protection Station under jurisdiction of Plant Protection Division, Agricultural Production Bureau, Ministry of Agriculture, Forestry and Fisheries. Japan is divided into five blocks under the headquarters which are located in Yokohama, Nagoya, Kobe, Moji and Naha respectively. Each division has branches and sub-branches at major sea and air ports, where plant inspectors are stationed. The total number of the stations including branches and sub-branches in 1983 is 96, and about 600 plant inspectors are engaged in import and export inspection.

INSPECTION AND TREATMENT OF IMPORTED CEREALS

The quantity of grains and beans imported has increased year after year. In 1982, 25 million tonnes of grains and 5 million tonnes of beans were imported, largely from Australia, Canada, U.S.A. and Argentina (Table 1). Together with timber, inspection of grains and beans occupies a great share in the total work load of import quarantine.

Table 1.
Amount of grains imported into Japan and proportion treated by plant quarantine fumigation in 1978-1982

	Grains		Beans	
Year	Imported (tonnes)	Fumigated (%)	Imported (tonnes)	Fumigated (%)
1978	24,150,000	67.6	4,850,000	76.5
1979	25,710,000	66.4	4,620,000	71.0
1980	25,550,000	60.7	5,070,000	70.2
1981	25,120,000	56.2	4,710,000	67.7
1982	25,480,000	62.7	4,730,000	77.2

In parallel with this large increase in imported grains and beans, interceptions of insects of quarantine concern have been also on the increase. More than 100 species of stored-product insects were intercepted by the inspection in past five years, including many species of economic importance not known in Japan. These include, *Sitophilus granarius* , *Trogoderma granarium*, *Callosbruchus rhodesianus* and *Zabrotes subfasciatus*.

When injurious pests are found upon inspection of grains and beans, their import is permitted only after complete disinfestation. As much as 60% of imported cereals were fumigated at the port of entry in the silo, warehouse, container or barge in order to prevent the entry of injurious insect pests.

METHODS OF QUARANTINE TREATMENT OF IMPORTED CEREALS

Since the first trial of fumigation of stored products with carbon disulphide performed in 1897 in Japan, a variety of fumigants such as chloropicrin, hydrocyanic acid, ethylene oxide, methyl bromide and aluminium phosphide have been used in Japan. Methyl bromide was introduced in Japan in 1948. Since it can be used at low temperatures, it has gained the status of a major fumigant for quarantine treatment since 1950 when import quarantine of cereals began. Aluminium phosphide was introduced as a quarantine fumigant in 1971. Its use is limited only to cereals which sorb much methyl bromide, since it requires a much longer exposure period than methyl bromide. Production and importation of these fumigants in Japan are shown in Table 2.

Table 2.
Quantities of some fumigants (tonnes) produced in or imported by Japan

Year	1977	1978	1979	1980	1981
Methyl Bromide	4791	6091	6223	6609	5630
Aluminum phosphide	25	37	51	44	47

Fumigation Facilities:

Warehouses and silos for plant quarantine fumigation must be approved by Plant Protection Station. Facilities for quarantine fumigation are divided into three classes according to their gastightness. To test gastightness, methyl bromide gas is introduced into an empty fumigation facility at a dose rate of 10 g m^{-3}. The facility is classified "A" when the concentration of methyl bromide averaged from three different points at upper, middle and lower of the facility is more than 7 g m^{-3} 48 hours after dosing; "B", 7 - 5.5 g m^{-3}; "C", less than 5.5 g m^{-3} respectively. Alternatively a pressure test is applied to test the gastightness of silos. The internal pressure of a silo is raised to 5,000 Pa. If the time taken to fall to 2,000 Pa. is longer than 20 minutes, the silo is classified "A".

During the early years of the quarantine fumigation of cereals, the gastightness of the warehouses and silos was not sufficient for fumigation. However, with the development of the economy and increase of international trade of agricultural products, the storage facilities have been completely modernized and many multi-storied ferroconcrete warehouses have been constructed. As much as 80% of the total number of warehouses that are currently approved by the Plant Protection Station are sufficiently gastight for fumigation.

In 1950, only a few silos were available for fumigation. Moreover, no silo was equipped with a gas circulation system. However, at present, most silos are sufficiently gastight for fumigation and are equipped with a vapourizer for methyl bromide and a modern recirculation system (Table 3). Most silos are of ferroconcrete or steel. The average capacity of a bin is about 500 tonnes but there are some bins of more than 4000 tonnes capacity.

Table 3.
Number of warehouses and silo bins in 1982 in Japan approved by the Plant Protection Station for fumigation

Classification	Warehouses	Silo Bins
A	2,121	8,994
B	454	7
C	123	22
Total	2,698	9,023

Fumigation Schedules

The current dosages and exposure periods set for quarantine fumigation with methyl bromide have been established on the basis of both basic and practical experiments. In plant quarantine work the object of fumigation is to obtain complete mortality of all stages of the insect pests against which treatment is directed. For this reason, the dosage based on the susceptibility to methyl bromide of *T. confusum* or *T. castaneum* pupae, relatively tolerant species and stages to methyl bromide, have been applied against almost all insect pests. In 1977, a lower dosage against *Sitophilus* spp., which are very sensitive to methyl bromide, was established. Factors that require to be taken into account in methyl bromide fumigation are as follows; a) fumigation facilities b) gastightness c) kinds of commodities d) load e) temperature f) susceptibility of insects to methyl bromide g) exposure period h) gas circulation.

The dosage of methyl bromide used and exposure period for quarantine fumigation of imported cereals in the facilities classified "A" are shown in Table 4. The dosage of aluminium phosphide and exposure period for quarantine fumigation of imported cereals in the facilities classified "A", "B" and "C" are shown in Table 5.

Apparatus for Measuring Gas Concentrations

In 1956, the Riken interferometer was introduced into quarantine fumigation for measuring gas concentrations. This indicator is now commonly used to check the validity of each quarantine fumigation by the plant inspector. Gas detector tubes, Kitagawa, Gastec and Drager, have been used for the detection of methyl bromide and phosphine in the TLV region. A halide leak detector is used for the detection of leaks of methyl bromide from warehouses. Gas chromatography is commonly used for laboratory fumigation experiments for estimation of both methyl bromide and phosphine.

Table 4.
Dosage and exposure period for methyl bromide for quarantine fumigation of imported cereals

Fumigation facility	Species of insect pest	Exposure period (hours)	Temperature (°C)	Dosage ($g\ m^{-3}$) Cereal type (*a*) (1)	(2)	(3)	(4)
Warehouse with gas circulation system (Bagged cereals)	*S. zeamais* and *S. oryzae*	24	20 and above	15	18	20	23
			10 - 19	20	24	28	32
			below 9	35	42	49	56
	T. confusum and others (*b*)	24	20 and above	21	25	29	33
			10 - 19	29	34	40	45
			below 9	35	42	49	56
	"	48	20 and above	13	17	21	25
			10 - 19	17	23	29	34
			below 9	21	28	35	42
	"	72	20 and above	12	15	19	25
			10 - 19	16	21	27	34
			below 9	21	27	34	42
Silo bin with gas recirculation system	*S. zeamais* and	24	20 and above	20	25	30	-
			10 - 19	29	34	41	-
			below 9	49	59	59	-
	T. confusum (*b*)	24	20 and above	29	35	35	-
			10 - 19	41	48	48	-
			below 9	49	59	59	-
	"	48	20 and above	18	24	25	-
			10 - 19	24	32	34	-
			below 9	29	39	42	-
	"	72	20 and above	16	22	23	-
			10 - 19	22	30	31	-
			below 9	28	38	41	-

a) Cereals are divided into four groups according to their degree of methyl bromide sorption as follows; (1) Low : rice, wheat, barley, barley malt (2) Relatively low : maize, sorghum, millet (3) Relatively high : soybean, ground nut, rape seed (4) Very high : buckwheat, safflower seed, powder products, pellets.

b) Double the dosage for *T. confusum* and others is used against *Trogoderma granarium*.

Fumigation Practice

Fumigation is not carried out directly by the plant quarantine inspectors. Quarantine inspectors check the application of fumigants into the fumigation facilities and the effectiveness of the treatment by measuring gas concentrations and by bioassay. Usually *T. confusum* is used as a test insect.

Table 5.
Dosage of phosphine and exposure period used for quarantine fumigation of cereals (*a*)

Fumigation facility	Classification	Dosage ($g\ m^{-3}$ as PH_3)	Temperature (°C)	Exposure period (days)
Warehouse (bagged cereals)	A	0.5	20 and above	5
	B	0.75	10 - 15	6
	C	1.0	5 - 9	7
Silo bin	A, B, C	2.0	20 and above	3 - 5
			15 - 19	4 - 6
			10 - 14	5 - 7
			7 - 9	6 - 8
			5 - 6	7 - 9

a) This schedule does not apply for cereals infested with *Sitophilus zeamais*, *S. granarius*, *S. oryzae* and *T. granarium*.

At present, quarantine fumigation of cereals is performed by commercial fumigation companies, equipped with such instruments and materials required for fumigation (eg. gas measuring equipment, gas masks, sealing materials and first-aid equipment). Employees in charge of fumigation operations must attend a special training course organized by the Plant Protection Station. The course includes the following subjects: techniques of warehouse fumigation, silo fumigation and safety arrangements related to fumigation. Persons working regularly with fumigants must have blood tests and physical examinations.

RECENT RESEARCH ON QUARANTINE FUMIGATION OF CEREALS

Some aspects of fumigation as a plant quarantine treatment still remain to be fully developed. Studies are required still on safe and efficient fumigation. Remarkable progress on fumigation techniques and gas measuring apparatus, such as gas chromatograph, have occurred in the past ten years. Also, many laboratory experiments have been conducted on the susceptibility of insect pests to methyl bromide, sorption of methyl bromide to cereals and residues in commodities in order to improve fumigation schedules. The research for quarantine in Japan are summarised below.

Susceptibility of Stored-Product Insects to Methyl Bromide

Methyl bromide has been used as a fumigant to control stored-product insects for more than forty years, but basic data on the susceptibility of major insect pests to this fumigant under long exposure periods was insufficient.

The effects of temperature and exposure period in tests with methyl bromide on the developmental stages of *Tribolium confusum*, *Sitophilus zeamais*, *Trogoderma granarium*, *Callosobruchus maculatus*, *C. rhodesianus* and *Ephestia kuehniella* were studied. These test insects were reared at 26°C, 70% R.H., except *T. granarium* which was reared at 30°C, 70% R.H. Laboratory experiments based on the recommended FAO Method No. 16 (FAO 1975) were conducted at various temperatures (5, 15 and 25°C) and exposure periods (5, 24 and 48 hours). Methyl bromide gas was injected into the specially designed 5-litre glass jar. The gas concentrations present were measured using a gas chromatograph equipped with a flame ionization detector and concentration-time products (*CT*-products) were calculated, based on measured gas concentrations. The mortality of immature stages of insects was determined by adult emergence and the mortality of adults was as sensed after a post-exposure interval of two weeks. Mortality data was analysed by probit analysis. LC_{50} and LC_{99}'s were estimated from dose-probit mortality curves.

LC_{50} and LC_{99}'s for the most tolerant stages of six species of stored-product insects at various temperatures and exposure periods are shown in Table 6. The results indicated that the *CT*-products required for 99% mortality increased with an increased exposure period at 25°C in the six species of insects tested and at 15°C in *T. granarium*, *C. maculatus* and *E. kuehniella*, respectively. Whereas, they were almost constant at any exposure period at 15°C in *T. confusum* and *S. zeamais* and at 5°C in *C. maculatus*, *C. rhodesianus* and *E. kuehniella*. In temperatures ranging from 5°C to 25°C, the greatest susceptibility was shown by *S. zeamais* pupae and *E. kuehniella* pupae when the exposure period was 48 hours. The susceptibility of *S. zeamais* was higher than that of *T. confusum* at any exposure period at 15°C and 25°C, but there were little differences between two species at 5°C. The susceptibility of *C. maculatus*, *C. rhodesianus* and *E. kuehniella* was higher

Table 6.
Susceptibility to methyl bromide of the most tolerant stages of some stored-product insect pests at various temperatures and exposure periods.

Species	Stage	Exposure period (hours)	Temp. (°C)	LC_{50}		LC_{99}	
				$g\ m^{-3}$	$g\ h\ m^{-3}$	$g\ m^{-3}$	$g\ h\ m^{-3}$
<u>Tribolium confusum</u>	Pupae	5	25	12.9	64.5	15.3	76.5
		5	15	28.2	141.0	35.2	176.0
		5	5	39.6	198.0	63.1	315.5
		24	25	3.7	88.8	4.2	100.8
		24	15	5.7	136.8	7.6	182.4
		24	5	11.3	271.2	17.4	417.6
		48	25	2.7	129.6	3.2	153.6
		48	15	2.7	129.6	3.4	163.2
		48	5	3.3	158.4	5.5	264.0
<u>Sitophilus zeamais</u>	Pupae	5	25	5.2	26.0	10.9	54.5
		5	15	10.7	53.5	17.1	85.5
		5	5	19.1	95.5	80.7	403.5
		24	25	1.6	38.4	2.9	69.6
		24	15	1.3	31.2	4.2	100.8
		24	5	2.7	64.8	15.5	372.0
		48	25	1.2	57.6	2.9	139.2
		48	15	1.2	57.6	2.1	100.8
		48	5	1.8	86.4	6.8	326.4
<u>Trogoderma granarium</u>	Diapausing larvae held for 2 months at 30°C	5	25	14.7	73.5	27.5	137.5
		5	15	32.9	164.5	73.2	366.0
		24	25	6.4	153.6	8.9	213.6
		24	15	8.5	204.0	25.7	616.8
		24	5	22.7	544.8	40.5	972.0
		48	25	4.0	192.0	6.5	312.0
		48	15	6.2	297.6	8.7	417.6
		48	5	10.6	508.8	18.6	892.8

Callosobruchus maculatus	Pupae	5	25	3.0	15.0	4.9	24.5
		5	15	6.0	30.0	9.4	47.0
		5	5	9.0	45.0	16.4	82.0
		24	25	1.4	33.6	2.2	52.8
		24	15	0.9	21.6	3.0	72.0
		24	5	2.0	48.0	3.5	84.0
Callosobruchus rhodesianus	Pupae	5	25	4.6	23.0	8.3	41.5
		5	15	5.3	26.5	12.6	63.0
		5	5	7.1	35.5	25.6	128.0
		24	25	1.5	36.0	2.5	60.0
		24	15	1.4	33.6	2.5	60.0
		24	5	3.0	72.0	5.6	134.4
Ephestia kuehniella	Pupae	5	25	5.8	29.0	7.5	37.5
		5	15	8.6	43.0	11.7	58.5
		5	5	23.2	116.0	32.1	160.5
		24	25	1.8	43.2	3.1	74.4
		24	15	1.9	45.6	2.7	64.8
		24	5	4.5	108.0	6.8	163.2
		48	25	0.9	43.2	2.0	96.0
		48	15	1.1	52.8	1.8	86.4
		48	5	2.2	105.6	3.3	158.4

than that of *T. confusum* at any temperatures and exposure period. Diapausing larvae of *T. granarium* showed exceptional tolerance to methyl bromide among six species tested.

Residues of Fumigants in Cereals after Quarantine Fumigation

Consumer's concern on fumigant residues in imported cereals after quarantine fumigation has increased in recent years. Work on the residue level of some fumigants in commodities resulting from quarantine treatment has been carried out in our laboratory in response to this concern.

Methyl Bromide

Effects of aeration and processing on methyl bromide and total bromide residues in wheat and soybean fumigated with methyl bromide under quarantine treatment conditions were investigated. Methyl bromide residues were analyzed by the method of Asaka and Seguchi (1974). Methyl bromide, liberated by a modified sweep codistillation method (Malone 1969) or acid reflux method, was converted to O,O-diethyl dithiophosphate in acetone. This derivative was determined by gas chromatography using a flame photometric detector. The maximum detectable level was 0.002 ppm with a 50 gram sample. Total bromide residues were analyzed according to modifications of the recommended FAO/WHO method. The minimum sensitivity of this method was 1 ppm in a 5 gram sample.

The residues of unchanged methyl bromide in wheat were over 1 ppm one day after treatment at 5°C and 15°C, and then decreased rapidly as shown in Table 7. However, the residues were only 0.08 ppm one day after treatment at 25°C, and then decreased gradually. Very little amount of methyl bromide retained in fumigated wheat over 1 to 3 months. The residue level of total bromide was almost constant at any storage period. After baking, no residual methyl bromide was detected. No residual methyl bromide was detected and only 1 ppm total bromide remained in tofu (bean curd) made from treated soybeans (Table 8).

Aluminium Phosphide

Effects of fumigation conditions and storage on the residues of phosphine in some raw cereals fumigated with phosphine generated from aluminium phosphide were studied. Phosphine residues were analyzed using a modification of the method of Bruce *et al.* (1962). The phosphine liberated by distillation was oxidized to phosphoric acid with bromide and determined colorimetrically. With a 500 gram sample and with the phosphate in 5 ml of solution, 0.002 ppm of phosphine could be determined. Recoveries of phosphine from untreated wheat by adding 0.1 and 0.05 ppm phosphine were 73.2% and

Table 7.

Effect of aeration on the residues of methyl bromide and total bromide in wheat fumigated with methyl bromide under various conditions (*a*).

Variety (Origin)	Treatment temperature	Dosage $g\ m^{-3}$	Days after (*b*) treatment	Residues (ppm w/w)	
				Methyl bromide	Total bromide
Red Spring	9	Untreated	-	ND[(c)]	5
(Canada)	5	29	1	1.48	13
			7	0.132	12
			14	0.030	14
			21	0.014	-
			28	0.011	14
Red Spring	9	Untreated	-	ND (*c*)	7
(Canada)	15	24	1	1.29	27
			7	0.040	21
			14	0.031	23
			91	0.031	22
Hard Winter	9	Untreated	-	0.004	7
(USA)	24	18	1	0.080	29
			7	0.051	30
			14	0.042	28
			28	0.045	29
			62	0.027	30

a) Load : 0.54 - 0.56 $t\ m^{-3}$, exposure period : 48 hours, data averaged from three replicates.

b) Samples were stored at the treatment temperature in the paper bag.

c) ND: not detected.

Table 8.
Effect of processing on the residues of methyl bromide and total bromide in wheat and soybeans fumigated with methyl bromide (*a*)

Commodity (Origin)		Residues (ppm w/w)			
		Before processing		After processing	
		Methyl bromide	Total bromide	Methyl bromide	Total bromide
Wheat, hard red winter (USA)	Untreated	ND	10	ND	11
	Treated	0.006	17	ND	15
Soybean (China)	Untreated	0.003	4	ND	ND
	Treated	0.004	29	ND	1

a) Bread was made from wheat and tofu (bean curd) was made from soybean. Data averaged from three replicates. Fumigation conditions were as follows; Wheat: Dosage 24 g m^{-3}, Temperature 15°C, Exposure period 48 hours, Load 0.56 t m^{3}. Soybean : Dosage 34 g m^{-3}, Temperature 15°C, Exposure period 48 hours, Load 0.5 t m^{-3}. Samples were stored at the treatment temperature.

b) Analyzed 3 days after treatment.

c) Analyzed 7 days after treatment.

d) Analyzed 14 days after treatment.

Table 9.

Residues of phosphine on some cereals fumigated with aluminium phosphide under various conditions in the laboratory (*a*)

Commodity (Origin)	Moisture content %	Fumigation conditions: Temperature (°C)	Fumigation conditions: Exposure period (days)	Days after treatment	Phosphine residues found (ppm w/w)
Maize (USA)	12.6	25	5	7	0.034
	12.6	25	5	14	0.012
	13.6	25	5	1	0.043
	13.6	25	5	6	0.014(*b*)
	12.6	15	8	6	0.014
	12.6	15	8	6	0.025
	12.6	15	6	7	0.028
	12.6	5	7	7	0.022
	13.6	5	9	1	0.030
	13.6	5	9	6	0.006
Wheat (Canada)	10.6	25	5	1	0.010
"	10.6	25	5	6	0.009
(USA)	12.1	25	5	7	0.008
"	12.1	15	6	7	0.018
"	12.1	5	7	7	0.005
(Canada)	10.6	5	9	1	0.010
"	10.6	5	9	6	0.011
Sorghum (Argentina)	12.5	25	5	7	0.026
	12.5	15	6	7	0.054
	12.5	5	7	7	0.023
Malt (Belgium)	6.3	25	5	7	0.037
	6.3	15	6	7	0.021
	6.3	5	7	7	0.002
Soybeans (USA)	10.6	25	5	1	0.047
	10.6	25	5	6	0.007
	10.6	5	9	1	0.058
	10.6	5	9	6	0.023

a) Dosage : 2 g m^{-3} as phosphine, Load : 0.17 kg L-1. Data averaged from 2 or 3 replicates.

b) Milled.

72.2% respectively.

The residues of phosphine in maize, wheat, sorghum, malt and soybeans ranged between 0.002 ppm and 0.058 ppm, 1 to 7 days after treatment under any fumigation conditions (Table 9). No consistent relationship was found between temperature, exposure period and residues. Aeration and milling proved very effective in removing any residues present.

Maximum Residue Limit of Fumigants in Japan

The maximum residue limit of unchanged methyl bromide has not been established. In Japan the MRL of total bromide in raw wheat fumigated with methyl bromide is 50 ppm. The MRL of phosphine in rice, wheat, beans and other raw cereals fumigated with aluminium phosphide is 0.1 ppm. Both total bromide and phosphine residues in wheat after plant quarantine fumigation did not exceed these MRLs.

REFERENCES

Akiyama, H., Kawamoto, N. and Tsukuda, Y. 1977.
Fumigant residues in imported agricultural products fumigated with hydrogen cyanide or aluminium phosphide. Res. Bull. Pl. Prot. Japan. 14:38-46.

Akiyama, H., Tsukuda, Y., Yasumoto, J. and Kawamoto, N. 1980.
The tolerance to methyl bromide of stored product insects. 1. The effects of temperature and exposure periods in tests with methyl bromide on *Tribolium confusum* Jacquelin du Val and *Sitophilus zeamais* Motshulsky. Res. Bull. Pl. Prot. Japan 16:77-84.

FAO. 1975
Recommended methods for the detection and measurement of resistance of agricultural pests to pesticides. Tentative method for adults of some major pest species of stored cereals with methyl bromide and phosphine - FAO Method No. 16. FAO Pl. Prot. Bull. 23:12-25.

Asaka, S. and Seguchi, K. 1974
Gas chromatographic determination of methyl bromide. Noyaku Kagaku 2:107-111.

Bruce, R. B., Robbins, A. J. and Tuft, T.O. 1962.
Phosphine residues from Phostoxin-treated grain. J. Agric. Fd Chem. 10:18-21.

Malone, B. 1969.
Analysis of grains for multiple residues of organic fumigants. J. Ass. Off. Anal. Chem. 52:800-805.