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**FINAL CONTRACT REPORT**

**DEMONSTRATION OF ALTERNATIVES TO THE USE OF METHYL  
BROMIDE IN PALM DATE FUMIGATION AT THE SOCIETE  
MEDITERRANEENE FRUITIERE (MEDIFRUIT).**

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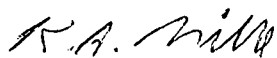
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I declare that this work was done under my supervision according to the procedures described herein and that this report represents a true and accurate record of the results obtained.



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Signature

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**Final Report**  
**UNIDO Project : MP/TUN/98/166**

**DEMONSTRATION OF ALTERNATIVES TO THE USE OF  
METHYL BROMIDE IN PALM DATE FUMIGATION AT THE  
SOCIETE MEDITERRANEENE FRUITIERE (MEDIFRUIT)**

**Periods covered: 5 March - 2 June 1999.**  
**1 - 18 April 2000**

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## **A. Summary of achievements.**

### **A 1. To 3 June 1999.**

1. Tests have been carried out in both vacuum chambers using carbon dioxide for 1, 2, 3 and 4 day exposure periods. The pump system and pipe work to the chambers were found to be excessively leaky for use with this gas and a further series of trials were carried out using re-dosing. The aim was to maintain the carbon dioxide concentration above 40%. Trials were carried out at CSL using a more gas-tight chamber to supplement the data obtained in Tunisia.
2. Tests have been carried out with the conventional aluminium phosphide formulation to generate phosphine in stacks at a dose of  $1.5 \text{ g m}^{-3}$ . Samples of bioassay insects were included and withdrawn after 3, 5, 7 and 10 days.
3. Tests have been carried out in sheeted stacks dosed with carbon dioxide to a minimum concentration of 40%. Samples of bioassay insects were withdrawn after 3, 5, 7 and 10 days exposure. It was difficult to make the stacks sufficiently gas-tight and so frequent topping up of the concentration was necessary. A PVC floor sheet rather than the polyethylene used is required. A repeat test is required using a PVC covering sheet on the floor to minimise the use of carbon dioxide.
4. Tests on the cylinderised formulation of phosphine in carbon dioxide in stacks and in freight containers were not possible due to the late arrival of the gas which had to be sourced in Cyprus and delivered via the UK. Three cylinders were subsequently delivered to Medifruit and these tests were carried out in 2000 when the containers were made sufficiently gas-tight.
5. Difficulties were encountered in preparing the two freight containers and installing the electrical heating system. The installation resulted in a loss of gas tightness and the original door seals were inadequate to hold gas. Leakage was also found from the floors and so remedial sealing has been requested from Medifruit for one container, initially. The heating system has been tested and shown to be capable of heating a fully loaded container. The test needs to be repeated when ambient temperatures are below the target treatment temperature of  $30^\circ\text{C}$ .
6. Fumigation tests have been carried out in containers with carbon dioxide and phosphine as for stacks.
7. It has been shown that dates do not absorb significant amount of both phosphine and carbon dioxide and that there is no detectable effect on the appearance and palatability of dates treated with either gas in all the trials.
8. Two insect monitoring exercises have been carried out in the Medifruit facility after taking advice on likely problem areas from the management. Medifruit staff were trained to continue the exercise over the Summer and Autumn of 1999 though this was not carried out.

9. Medifruit staff have been trained in the fumigation techniques and equipment and instrumentation used to date.

#### A.2. April 2000.

1. On arrival the remedial sealing work to the floor and door of container 1 was near completion though much needed to be done on container 2. Both pressure relief valves fitted previously to the containers had been broken by Medifruit staff when the containers were turned round to face the building for ease of loading. A single spare valve was available and this was fitted to container 1 and a trial the check the heating system and the sealing in the empty container was carried out. Trials with carbon dioxide and the phosphine-carbon dioxide mixture were carried out. Container 2 was unserviceable and not able to be pressure tested.
2. A trial of a 4-day exposure to the phosphine-carbon dioxide mixture was carried out in the fully loaded container 1 with a bioassay of infested dates. The heating system was used only before dosing. Excellent CT products were obtained throughout the container with little loss of gas and moth larvae were controlled. There was evidence of slight heating above 30 °C due to solar gain but there was generally a gradual reduction in temperature.
3. The same loaded container was dosed, again after heating, with aluminium phosphide tablets for a 4-day exposure. Again excellent CT products with little loss of gas were obtained with control of the bioassay. There was the same gradual reduction in temperature.
4. The container was dosed with carbon dioxide for a 4-day exposure. The heating system was run throughout the trial and carbon dioxide loss occurred. This was thought to occur mainly through the pressurised heating system. A daily application of carbon dioxide was required in order to maintain an adequate concentration of 40 %. The bioassay was controlled.
5. A small 8 m<sup>3</sup> stack wrapped with PVC sheet was treated with carbon dioxide. The stack was leaky due to its small size and 5 additions of gas were required to maintain 40 % carbon dioxide. A 7-day exposure was necessary to control moth larvae.
6. A larger 29 m<sup>3</sup> stack was treated with carbon dioxide for a 3-day treatment without a bioassay in order to demonstrate that it was possible to achieve a gas-tightness which would allow a single dosing.
7. A 28 m<sup>3</sup> stack was dosed with phosphine carbon dioxide mixture for a 7-day exposure with bioassays for 3, 5 and 7-day exposures. Control of moth larvae was achieved in the 3-day exposure which could be reduced to 2 days due to the rapid dosing. The stack retained gas sufficiently to be used for the control of moth eggs in 5 or 7 days according to temperature.

8. During this period Medifruit staff were re-trained in techniques and use of equipment:
  - a. The use of aluminium phosphide tablets for dosing stacks and freight containers and the safe desposal of residues by burying was demonstrated together with safe airing practice.
  - b. The dosing of carbon dioxide using an electrical vaporiser into chambers, stacks and freight containers was demonstrated together with safe airing practice.
  - c. The use of the portable carbon dioxide and phosphine monitors was taught. The use of detector tubes for phosphine and carbon dioxide at treatment and environmental safety concentrations was demonstrated during treatments and during airing. Personal respiratory protection apparatus and the required filters for use with phosphine was demonstrated.
  - d. The pressure testing of freight containers and the vacuum testing of stacks using a half-life test and how to remedy leaks.
  - e. The correct sheeting of stacks for use with phosphine and carbon dioxide.
  - f. The use of the electrical heating system for the freight containers. Drawing and written instructions were left on site.
  - g. The use of 3 different trap types and pheromone lures for monitoring insect infestation, particularly flying moths, was demonstrated and how this could be used as part of an ICM approach to infestation control.
  - h. The ICM approach was discussed in the context of Medifruit's current practice. Improvements were suggested as per this report.
  - i. The use of temperature monitoring by thermocouple and maximum-minimum thermometer was demonstrated and the importance of temperature to the success of fumigation explained and discussed.
  - j. The necessary improvements to the Medifruit chambers were described in order that they could be used with carbon dioxide. These involved repair to leaks on valves for vacuum use and improved door seals if the chambers are to be used at atmospheric pressure.
  - k. The siting of freight containers indoors with regard to the problem of solar heating and the need for security whilst under fumigation was discussed.
  - l. In addition to siting indoors, freight containers require some form of insulation in order to economise on heating.
  - m. The temporary heating system for the containers needs to be upgraded. The ducts should be seam-welded instead of riveted with a removable gasketed heater section



for ease of maintenance. Consideration should be given to an improved venting system in order to speed up re-dosing. This should include a 10 cm diameter chimney and the provision of a powerful pump to move fresh air through the volume.

## **B. Recommendations and economic assessment.**

### **B.1 Phosphine.**

Phosphine can be successfully used in stacks and unheated freight containers. The recommended dosage rate in the TOR of  $1.5 \text{ g m}^{-3}$  can be reduced to  $1.0 \text{ g m}^{-3}$  when treating dates containing moth larvae. However, it would be prudent to retain the full dosage rate when treating dates which may carry moth eggs.

The Medifruit chambers could be used with phosphine if the vacuum system is disabled before treatment. The system could be used for venting gas in conjunction with an air intake. There may be a requirement to check the gas tightness of the doors which would be reduced when the chamber operates at atmospheric pressure.

Exposure periods recommended for the following mean temperatures:

Larvae:

Mean temperature 15-30 °C - 3 days when dosed with aluminium phosphide and 2 days when dosed with phosphine-carbon dioxide mixture.

Eggs:

15 °C - 10 days exposure

20 °C - 7 days exposure

25 °C - 5 days exposure

30 °C - 3 days exposure

These exposure periods are the same for both phosphine producing formulations.

It should be noted that many insect species have the capacity to develop resistance to phosphine with high levels possible Price and Mills (1988). Therefore great attention should be taken to sealing, correct dosing and not shortening the recommended exposure periods. The frequent use of concentration measurement, especially towards the end of treatments is recommended. It may be unlikely that resistance can develop in *E. ceratoniae* since it will be mostly treated as relatively susceptible larvae and multiple treatments in a processing facility will be rare. However, there is the possibility of low quality fumigations in stacks in the producing areas and the risk that resistant moths could arise. It is possible to monitor for resistance and CSL can advise on this aspect, if required, in the future.

### **B.2 Carbon dioxide.**

It is recommended that the concentration does not fall below 40 % in any fumigation and, therefore, that 45 % is the target to give a safety margin. It is recommended that provided the pressure test standard is met in all enclosures the initial dose of carbon dioxide need not exceed that which produces a 65 % concentration after mixing.

Higher concentrations are wasteful, especially in leaky structures where higher concentrations are lost more rapidly. Recommended exposure periods are as follows:

Atmospheric pressure at mean temperatures 15-25 °C:

Larvae: 7 days exposure

Eggs : 10 days exposure

In heated containers: 4 days at 30 °C.

Vacuum at ambient temperatures above 15 °C.

Larvae and eggs: 6 days exposure (based on tests at CSL).

There appears little advantage in using carbon dioxide under vacuum for the control of larvae. PVC base and covering sheets are required for stack treatments.

### **B.3. Integrated Commodity Management (ICM) Strategy.**

It is recommended:

1. That fumigated and unfumigated dates are clearly labelled and segregated in the cold store so that the untreated ones can be eventually fumigated and that multiple treatments are avoided.
2. Trapping with moth flight traps with regularly replaced pheromone is routinely carried out in order to monitor the level of infestation within the facility and to identify possible breeding sites within the fabric of the building. Good records are kept.
3. That dates are processed as rapidly as possible after removal from the cold store and that any unprocessed dates are returned to the cold store overnight.
4. That sorted and known infested dates are ground rapidly or returned to the cold store for processing the next day.
5. That packs of finished dates are not left uncovered but are wrapped by the end of the working day.
6. That large amounts of dates are not left in the intake area to await fumigation but are rapidly put into the coldstore so that only those which can be fumigated within a day or two are left in the intake area.
7. That records are kept of the concentrations of phosphine or carbon dioxide used for treating the different batches of dates. Also that dates are examined 1-2 weeks after treatment to ensure that moth larvae are dead. This is particularly important for phosphine-treated dates since survival could be an early warning of resistance.
8. That areas of infestation discovered in the fabric of the building or in machinery are rapidly treated with insecticide, synergised pyrethroids are preferred.

#### **B.4. Economic assessment.**

Aluminium phosphide tablets in Tunisia cost 25.5 TDN for a 333 tablet pack. Each tablets gives off 1 g of phosphine gas and so the cost is 0.076 TDN per g.

The cost of the phosphine-carbon dioxide mixture is more difficult to estimate. CSL were given 3 cylinders via the Cyprus Grain Board for the project free of charge by BOC, Australia (now Cytec Inc.). The price to Cyprus was US\$ 60 per cylinder or 86 TDN. On the other hand, a recent quotation was \$US 160 for a large order. This is 229 TDN per cylinder. Each 30 kg cylinder contains 600 g of phosphine. It is probably not realistic to consider the price to Cyprus since it may be special for the introduction to a new market. The full price is not likely and it would be advisable to obtain a quotation for supply for the quantities required for Tunisia. Therefore the best estimate is an mean of these prices i.e. 157 TDN per 600 g or 0.262 TDN per g This makes it 3.44 times as expensive as the conventional phosphide formulation but there are advantages in being able to reduce the treatment by a day to 3 days for use against larvae.

Carbon dioxide is expensive in Tunisia. Tunisie Gaz Industriels quote 0.759 TDN per kg for a 30-35 kg cylinder. Air Liquide quote 26.909 TDN for a 30 kg cylinder or 0.897 TDN per kg. The mean is 0.828 TDN per kg.

For methyl bromide Tunisie Gaz Industriels quote 11.5 TDN per kg for a 100 kg cylinder.

#### **Methyl bromide.**

From CSL's trials, Medifruit used between 0.99 and 1.92 kg of methyl bromide to treat a single chamber load and our best estimate was 1.67 kg or 0.21 kg per tonne of dates with 8 tonnes loaded into the vacuum chambers. Therefore the cost is 2.415 TDN per tonne. If we take the dose which Medifruit say they dose i.e. 1 kg for the same tonnage the cost reduces to 1.446 TDN per tonne.

#### **Phosphine.** (Note when measuring concentrations that 70 ppm = 0.1 gm<sup>-3</sup>)

It was generally not possible to fumigate an enclosure filled with dates due to their unavailability in quantity for trials. The best approach is to consider a full enclosure.

Using the vacuum chamber containing 8 tonnes of dates a dose of 1.0 x 22 g m<sup>-3</sup> or 22 g of phosphine at a cost of 22 x 0.076 = 1.672 TDN or 0.209 TDN per tonne. This is cheaper than methyl bromide by a factor of 6.9 although the exposure period is much longer. If the chamber is dosed with the cylinderized formulation then the cost is 3.44 x 0.209 = 0.719 TDN per tonne and this is still 50 % cheaper than using methyl bromide on chemical alone for the treatment of larvae. The same costs per tonne apply to sheeted stacks and freight containers.

For the treatment of eggs the dosage rate is 1.5 times that for larvae and so the cost increases to 1.078 TDN per tonne i.e. still cheaper than methyl bromide for chemical alone.

### **Carbon dioxide.**

The cost of using carbon dioxide is more difficult to estimate. There is a need to improve the gas-tightness of the chambers and the freight container when the heating system is used during the treatment. It should be possible, with care and a large enough budget, to achieve the same standards of sealing obtained in the last stack trial where the stack had a vacuum half-life of 35.8 seconds. This was a 24.44 m<sup>3</sup> stack with 12 pallets. If the pallets had been fully loaded, the stack would have contained 10.8 tonnes of dates. A dose of 48 kg of carbon dioxide was used and it was estimated that sufficient gas would be present after 10 days to enable a successful treatment of dates infested with eggs without topping up. The volume of dates in a pallet is estimated as 40 % of the volume. If the 12 pallets of empty crates in the stack had been full, the dates would have occupied a volume of 9.8 m<sup>3</sup> (40 % of 22.4 m<sup>3</sup>). Ignoring the 1.515 tonnes of dates actually treated and the effect of sorbtion, it can be argued that the dose could be reduced by 40 % to give a dosage of 28.8 kg. This would cost 0.828 x 28.8 = 23.85 TDN for 10.8 tonnes or 2.202 TDN per tonne. This is 1.53 times the cost of methyl bromide. Even if the treatment has to be re-dosed with the same amount of carbon dioxide the cost is only 3.06 times the cost of methyl bromide.

However, there is a capital cost for the vaporiser and other equipment.

The capital cost of the modified containers was expensive (per container):

Purchase price - US\$ 4375 or 6270 TND

Modifications -1200 TDN

False floor (wood and labour) - 960 TDN

Parts and paint - 190 TDN

Electrical heating system - £ sterling 1808 or 3590 TDN

Grainguard pressure relief valve 205 TDN

**Total = 12415 TDN**

The heated container system was the one favoured by Medifruit and they had plans to install several in the facility and favoured a gas-fired heating system for economy.

There is still further development required, particularly on the heated freight container system which is likely to be the most popular in that it allows the shortest exposure periods. There is further trials required on the sheeted stack system since this would be the system used when the containers are fully utilised and dates require treatment.

It is anticipated that the information presented in this report will enable further trials to proceed. The authors are available to assist with technical queries.

## **1. Introduction.**

The Tunisian date industry is vitally important to the economy and is third in value to exports after olive oil and seafoods. 60-70 million metric tonnes are produced valued at 90-100,000 TD. Of the processing units, 31-33 of the total of around 40 are certified for the export trade and 25-29000 metric tonnes are exported annually. 'Bio dates' produced without the use of pesticides are increasingly important for some markets and command a price premium. Twelve of the processors are in the producing regions with the major ones near Tunis. The Carob Moth, *Ectomyelois ceratoniae*, is the major infestation problem with larvae found inside the date. Eradication of infestation by conventional insecticides, the use of natural predators, bacterial insecticides and the use of pheromone for mating disruption in the palmeries has helped tremendously with the infestation problem but has not proved the complete answer. Infestation levels in some batches can reach 20-25 % and there is a strong possibility of infested dates yielding adults which infest other dates in storage. There is a permitted 3-5 % tolerance of all infestation, according to season, but the aim is to drastically reduce this level, particularly for some competitive markets. The industry is very important in the South of the country where the date is the main cash crop. It is estimated that 50,000 people in this region and others throughout the country are supported by the date industry.

### **A. The use of fumigation.**

The use of atmospheric pressure and vacuum fumigation with methyl bromide is widely practised and producers with their own processing plant will fumigate on their premises. Three exporters already use phosphine either in the palmeries or in the processing plants either using block-built chambers or stacks under gas-tight sheets in the open where it is likely that the effect of wind will make fumigation difficult. Freezing or the application of heat to 70 °C has been tried but the heat treatment darkens the dates. There have also been problems with fermentation and so heat treatment is considered unacceptable.

The Tunisian Ministry of Agriculture is promoting the increased exportation of 'bio dates' and the use of netting to protect the dates on the palms is considered though there is concern about the cost of this approach. In the mid-term the production of 'bio dates' will increase appreciably. The use of irradiation is not acceptable to some European markets and so there is much interest in the use of carbon dioxide on 'bio dates' even though this will require longer exposure periods and will be more expensive. For non-'bio dates' phosphine will become very important. Both these treatment methods will require much longer exposure periods than does methyl bromide and there are severe logistical and capital investment problems to overcome in order to use them.

It may be possible that the results of the current project will form the basis for an application from the Tunisian Government to UNEP's Multilateral Fund for an investment or implementation project and fumigation efforts will go in parallel with the use of biological control methods to aid Tunisia's phaseout of methyl bromide.

## **B. Medifruit.**

The current demonstration project was hosted by Medifruit, a medium sized processor ,at their processing and packing facility at Borj Cedria near Tunis.

The company was described as having many features in common with other processors. It has two vacuum chambers and two large cold stores. The plant was technically very well run and relies heavily on the treatment of all dates entering the plant with methyl bromide under vacuum. The cold storage at 1-2 °C, even when available as at Medifruit, is not able to kill all larvae, hence the reliance on fumigation. Medifruit aim to fumigate all dates entering the plant, either on intake or after a period of cold storage. At busy intake periods dates go straight into cold storage without fumigation due to a lack of vacuum chamber capacity. There is the possibility that if dates are taken out of cold storage and held in the packing area for too long then adult moths can emerge, mate and lay eggs onto other dates. This may happen during the sorting process where infested dates are removed and held for eventual processing into animal food, especially if the known infested dates are not processed quickly. CSL staff were told that adult moths are seen in the facility, especially in Summer. Fumigation should eliminate all larvae but an inadequate treatment could result in contamination of dates with eggs from emerged adults just prior to packing.

## **C. Verification of methods by the use of a bioassay.**

No references could be found to the tolerance of *E. caratoniae* to phosphine and carbon dioxide though there is information on the effect of these on closely related Pyralid moth species, some of which are minor pests of dates. CSL's normal procedure is to test biological material in laboratory chambers under closely controlled environmental conditions. However, for this project it was expected that a bioassay in trials at Medifruit would suffice, especially where it could be backed up with published efficacy data on related species.

A good bioassay should contain the correct pest in the correct stage of development in sufficient numbers to give confidence in the results. CSL intended to use large cultures of two species of stored product beetles prepared in the laboratory but, even though these were cosmopolitan pests, they could not be imported into Tunisia and so they could not be used.

It was therefore decided to rely on selecting infested dates as bioassays. Fortunately, the larval stage of insects are relatively susceptible to the gases employed and this approach was considered adequate and would give results directly applicable to the main pest. Medifruit were able to supply sorted and infested dates for trials and, after training, CSL staff could readily obtain dates infested with larvae from stocks. Unfortunately, it was not possible to obtain eggs of *E. ceratoniae* due to the lack of adult moths. It was considered important to treat moth eggs should dates thought to have eggs recently laid from moths emerging in the facility ever require treatment before packing. The control of eggs was expected to be the most difficult to achieve

from the literature of the relative tolerance of the different life stages of stored-product moths.

Fortunately, good contacts have been established with Prof. Mohamed Habib Dhouibi of the National Institute of Agronomy in Tunis, an acknowledged expert on the infestation of dates. His department helpfully supplied 0-1 day old eggs of the closely related Pyralid species the Mill Moth, *Ephestia kuehniella* as a bioassay insect. These were either treated immediately or when they were a maximum of 48 hours old.. CSL take the view that if the eggs of *E. kuehniella* are controlled then the eggs of *E. ceratoniae* will be controlled. Medifruit management were able to supply dates infested with the larvae and a limited number were included in almost all trials.

#### **D. Work periods.**

CSL staff, including their sub-contractor, Igrox (a major UK commercial fumigation company) were on site at Medifruit as a team of two people with occasional single staff during some trials monitoring.

The work periods on site at Medifruit were:

5 – 30 March 1999.

11 April – 3 June 1999 (extended due to staff illness).

1 – 18 April 2000.

In addition, 6 trials including bioassays were carried out in CSL's own gas-tight vacuum chamber in the Summer of 1999 in order to provide data on the effect of carbon dioxide under vacuum not available due to the leakiness of the Medifruit chambers during long exposure periods.

Scheduled work on freight container and stack fumigation with phosphine at Kebili in the growing area immediately after the 1999 harvest was cancelled after discussions with UNIDO and on Medifruit's recommendation in order to concentrate resources, particularly on making freight containers at Medifruit sufficiently gas-tight to carry out meaningful trials in 2000.

#### **E. Integrated Commodity Management (ICM).**

The project Terms of Reference refers to the need for replacing the need for 'end of pipeline' methyl bromide treatment by maintaining a 'clean pipeline'. It is not known how far the Medifruit practice of fumigating as many incoming dates as possible is used throughout the processing facilities in Tunisia. Certainly this is the correct approach when dealing with dates which may have not been fumigated or fumigated by uncertain standards in the producing area. It may be that some processors depend on the initial fumigations and carry out little or no fumigation in their own premises.

For this project the ICM recommendations refer only to the Medifruit situation and it is hoped that they will apply equally well to other companies. It is important to identify the stages through which the dates pass as they are processed and packed, and defining the critical points that influence insect infestation. This is the well understood

Hazard Analysis Critical Control Point (HACCP) evaluation commonly adopted in the food sector.

The Medifruit facility was extremely clean and generally free of dates residues. There was frequent in-depth cleaning of equipment and power water jet washing of floors and equipment. A special effort was made to clean the facility and maintain the machinery in the Spring when the date stocks were exhausted prior to the next harvest. The manager was aware of a problem with a small inaccessible area under the ovens used to dry a glucose glaze on dates. This appeared not to be a source of *E. ceratoniae* but it yielded many microlepidoptera and fruit flies which may cause an infestation problem. This was dealt with by the application of special cleaning and occasional synthetic pyrethroid spray.

Medifruit aim to fumigate dates on intake. Dates not treated due to the lack of chamber capacity were put in a cold store as quickly as possible. Development of moth larvae in these at 1-2 °C would have been minimal though it is possible that some of the larvae entering the cold stores were near to pupation. Medifruit aimed to fumigate these dates when chamber time became available but this pre-supposes that the treated and untreated batches were correctly identified and that the untreated dates were identified if they had to move to another store in the event of a breakdown of the cooling system. It is possible that errors were made and that unfumigated dates were taken from the cold store for processing. It is also possible that dates were underdosed with methyl bromide in the chambers though overdosing appeared more likely from CSL's observations of the chamber use. However, it is important that fumigation is carried out correctly and a system needs to be in place to record the dosage and the batch number and the subsequent location in the cold stores. This will apply to the application of phosphine and carbon dioxide in the future.

The possibility of pupae developing and adults emerging from dates which have missed being fumigated is real and the risk increases with the time they are left out of the cold store before processing. Dates are sorted by hand and the infested ones are ground for animal feed, a process which is likely to destroy most of the larvae. The rejected dates require special attention. They should be processed as rapidly as possible and if this is delayed they should be returned to the cold store overnight or until they can be processed. Unsorted dates pose a lesser risk but they could have a significant level of infestation if they are unfumigated. Again only sufficient dates which can be processed should be taken from the cold store. This was not done and we saw dates waiting for processing for several days in some cases.

After removing infested dates they were re-hydrated by wetting with a water hose and placed in a steam room. This process may kill many larvae. After this the dates are glazed with a glucose solution which is then dried in a high temperature gas-fired oven. This final process is to enhance the appearance of the finished product but also should kill any surviving moth larvae and pupae.

It was known by the Medifruit management that adult moths flew in the facility in the Summer months and attempts were made, where possible, to place dates newly out from the cold store in the intake area near to the fumigation chambers though this was



far from consistent. In fact, an attempt had been made by using polyethylene sheeting to separate this area from the main sorting and processing area. This attempt would be of limited benefit since moths could readily fly between these areas. It would be better to rely totally on the procedures outlined above in order to prevent eggs being laid on finished product both in the crates or in the final packs before wrapping with cellophane film.

Medifruit made no use of the trapping of adult moths to quantify the problem, the seasonal numbers and relating this to the presence of large quantities of dates stacked in the intake area, the processing area or both. It would be very worthwhile to do this to demonstrate to staff risk areas and the need for care in minimising the time dates are out of the cold stores before processing or grinding in the case of known infested dates.

Various traps were purchased for the project and their use demonstrated to Medifruit staff. These were of three types. A floor based cardboard sticky trap could be placed around the facility in places where it would not be disturbed. Polystyrene delta traps incorporating a sticky surface could be hung from lights or placed high on internal dividing walls. Finally a plastic funnel trap incorporating a piece of DDVP-impregnated plastic to kill trapped moths to prevent escape could be placed in the same way as the delta traps. The performance of the latter two types could be enhanced by using a pheromone lure to attract flying male moths.

The major pheromone component for attracting *E. ceratoniae* is (Z,E)-9,11,13-tetradecatrienal but lures containing this are not commercially available. However, lures based on the closely related (Z,E)-9,12-tetradecadienyl acetate are available to attract males of the Pyralid moths *Ephestia cautella*, *E. figulilella*, *E. calidella* and *Plodia interpunctella* and they work satisfactorily for *E. ceratoniae*. It is recommended that traps with lures are placed indoors at the rate of one for every 100 m<sup>2</sup>. Moth flight depends on the temperature within the facility and sticky traps on the floor will detect non-flying adults. Ideally traps should be inspected weekly with the pheromone lures changed monthly. The sticky inserts on the delta traps and the whole floor trap should be changed every 6 weeks, but more frequently in areas of high infestation.

#### **Trapping exercise.**

In mid-March 1999, 20 traps of the various types were placed all round the facility in a high density pattern with Medifruit staff: the intake area, outside the cold stores and throughout the processing and packing area. Some traps only contained the pheromone lure in an attempt to demonstrate its effectiveness and the traps were numbered and any moths caught noted after 2 weeks. Very few dates were outside the cold stores and the ambient temperature was low. Consequently no moths were caught. There were persistent problems with employees removing the floor traps during cleaning operations but it was clear that the moths were not active at this time. The pheromone lures were changed and the traps left until the next CSL staff visit in mid-April 1999. On arrival no moths were noted but a regular 2-weekly check finally revealed 3 moths by the end of this second visit on 3 June 1999.

At this point the use of floor traps were abandoned due to persistent removal. Medifruit staff were asked to remove the sticky portion of the delta traps and put the moths caught in the funnel traps into labelled polythene bags and to send everything to CSL for identification and counting, changing the pheromone lures and DDVP holders also at monthly intervals. Unfortunately, this was not done and so there is no data available on the build-up of the moth population throughout the Summer and its predicted decline during the Autumn and Winter of 1999. On the return of the CSL staff in April 2000 the traps contained many moths, microlepidoptera and fruit flies with far more moths in the ones with the pheromone lure. This was clear evidence of the presence of moths in the facility during the intervening period though the numbers of moths caught may not represent the true total due to the loss if effectiveness of the sticky traps and the DDVP. The traps were replaced with new sticky portions, new DDVP holders and monitored during the remainder of the project. As in 1999, no moths were caught at this time of year.

Ideally, it would have been useful to trap when dates were outside the cold store and again when this situation had been deliberately avoided in order to demonstrate the effect of preventing moth development outside the cold stores. Unfortunately, CSL staff were not present at the time of maximum moth activity since this was in the Summer and Autumn, a time when the freight container modifications were not started. In any case, this period would not have been suitable for working in the freight containers outside due to the high ambient temperatures.

Medifruit staff had the monitoring technique demonstrated to them and it is recommended that they use the traps left with them in order to monitor the availability of adult moths and to find the sources of any hidden infestation. In general, in CSL's experience there is a reluctance by some U.K. companies to use traps since they do demonstrate a problem and, perhaps, a lack of progress in improving the situation. It is hoped that this attitude, if it was the reason for the lack of co-operation in this case, will be overcome and trapping seen as an essential pest management tool for monitoring the effect of changes in practice and for identifying problems. It is well worth the expense and effort necessary.

Finally, it should be repeated that the infestation problems within the Medifruit facility were few but there are still lessons to be learned. In other, less well run facilities, the benefits of a trapping programme would be greater. The programme would need to be carefully designed to minimise the number of traps required with effectiveness, economy of materials and staff time in mind.

CSL have no doubt that the fumigation of dates on intake as practised by Medifruit rather than the fumigation of finished product is the correct approach. Combined with the recommendations for segregating and handling the dates this will result in negligible infestation in the final product. There remains the option to fumigate finished product on which eggs have been laid if there is an unexplained increase in moths within the facility at any time. This report, therefore, has recommendations for the treatment of moth eggs.

## **2. Trials carried out at Medifruit and CSL, 5 March - 2 June 1999.**

### **Sorption of carbon dioxide by dates.**

Sorption of carbon dioxide can cause problems in the treatment of commodities such as cereal grains where the speed of absorption and the volume of carbon dioxide absorbed varies with the type of grain, the temperature and the moisture content. For example, at 24°C, wheat absorbs 50% of the carbon dioxide within 20-24 hours. After 72 hours, an equilibrium of 0.6 litres per kg is obtained. Oat seeds are absorbent whereas maize and barley seeds far less absorbent (Peng, Wu-Kang, 1990). In calculating the dose of carbon dioxide used for the treatment of dates it is important to study the sorption onto the dates.

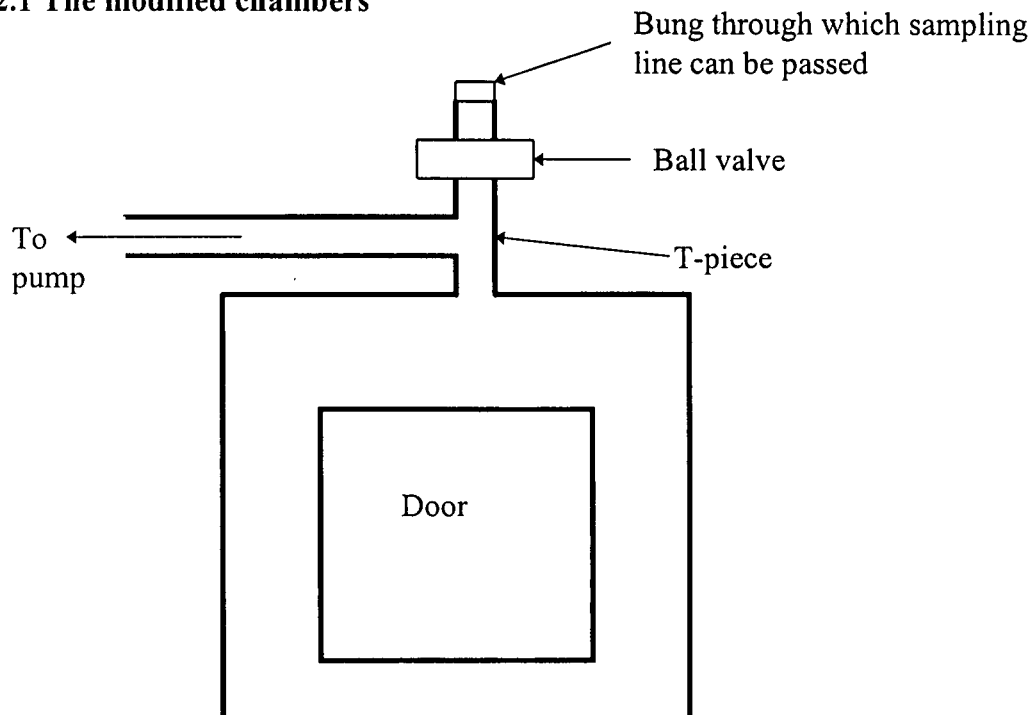
Chamber 1 was dosed with carbon dioxide using two pallets and 70 plastic crates both with and without dates at a temperature of about 16 °C where sorption be expected to be relatively high. The amount of dates used was 1616 kg. The average theoretical concentration of carbon dioxide was 35.5 % and the actual measured concentrations were 33.6 % with dates and 32.2 % without dates. Therefore dates do not absorb significant amounts of carbon dioxide and no allowance for this is required when dosing in the future.

Phosphine is known not to absorb significantly onto commodities and a similar test was considered unnecessary.

### **2.1. Chamber experiments.**

Two nominally 20 m<sup>3</sup> (measured by CSL to be 22 m<sup>3</sup>) chambers designed for use with methyl bromide under vacuum were present on site. The pumping system was capable of reducing the pressure in the chamber to about 100 mbar. The chambers were modified to include a T-piece and a ball valve on the chamber outlet to the pump so that pressures and concentrations could be monitored (Fig 2.1).

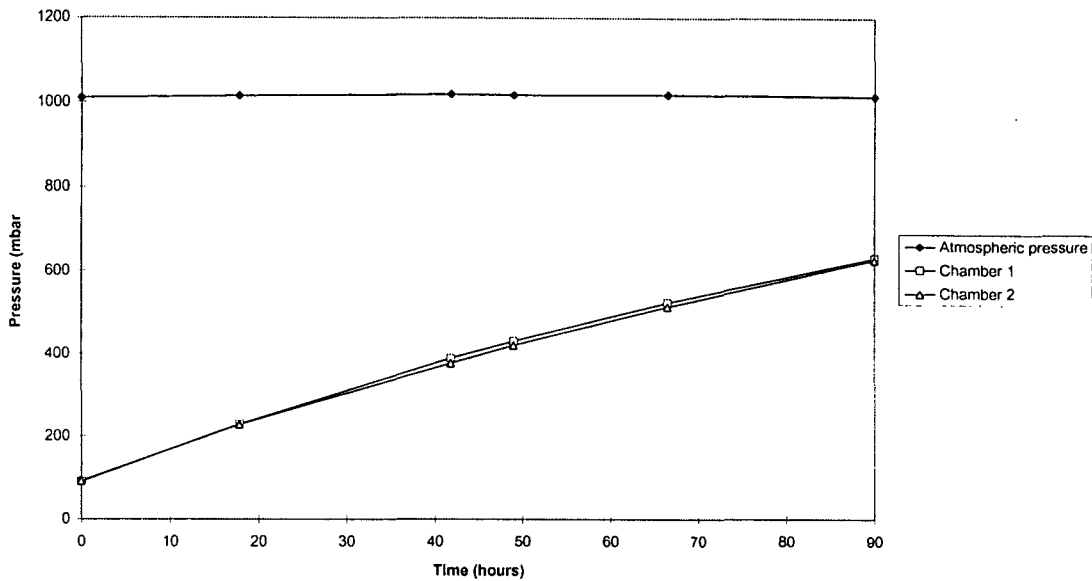
**Fig 2.1 The modified chambers**



The chambers were evacuated and checked for leaks. No leak would be expected from door gaskets which were in good condition and so would compress with the door under vacuum. However, a major leak was found in a ball valve between the vacuum pump and the chamber. The leak was made less severe by the use of vacuum grease on the ball of the valve.

Both chambers were then evacuated and the vacuum was monitored over four days using a digital manometer (Fig 3.2). In four days the pressure in the chamber had risen from 10 % of atmospheric to more than 60 % of atmospheric. Chambers 1 and 2 leaked at a similar rate of about 6 mbar/hour.

Fig 3.2. Ingress of air into the evacuated chambers.



Three tests were then undertaken using carbon dioxide held under vacuum for either one, two or three days. Two tests were carried out in chamber 1 and a single test in chamber 2. Two pallets of dates were placed in each chamber along with samples of *Ephestia kuehniella* eggs. A maximum-minimum thermometer was placed in one of the chambers for the duration of the tests. The chambers were evacuated and then dosed with carbon dioxide until atmospheric pressure was restored. The chambers were evacuated again and then left under vacuum for the required exposure period. The pressure in the chamber was monitored at each stage using a digital manometer and the theoretical concentration was calculated from the pressure data.

The starting pressures were 101, 148 and 200 mbar for the 1, 2 and 3 day tests respectively. The starting pressures used were greater in the longer tests so that the leakage of air into the chambers would have a smaller effect on the concentration of carbon dioxide per day.

At the end of the exposure period, the chamber was returned to atmospheric pressure by the introduction of air. The concentration of carbon dioxide was then measured. The chambers were then flushed with air and the insect samples retrieved.

Figs. 3.3 to 3.5 give the pressures and concentrations in the chambers against time in each test and Table 3.1 gives the amount of carbon dioxide used and the theoretical starting and average concentrations. The temperature in the chambers varied between 16 and 23 °C.

Fig 3. 3 Test 1 - One day carbon dioxide treatment in chamber 1.

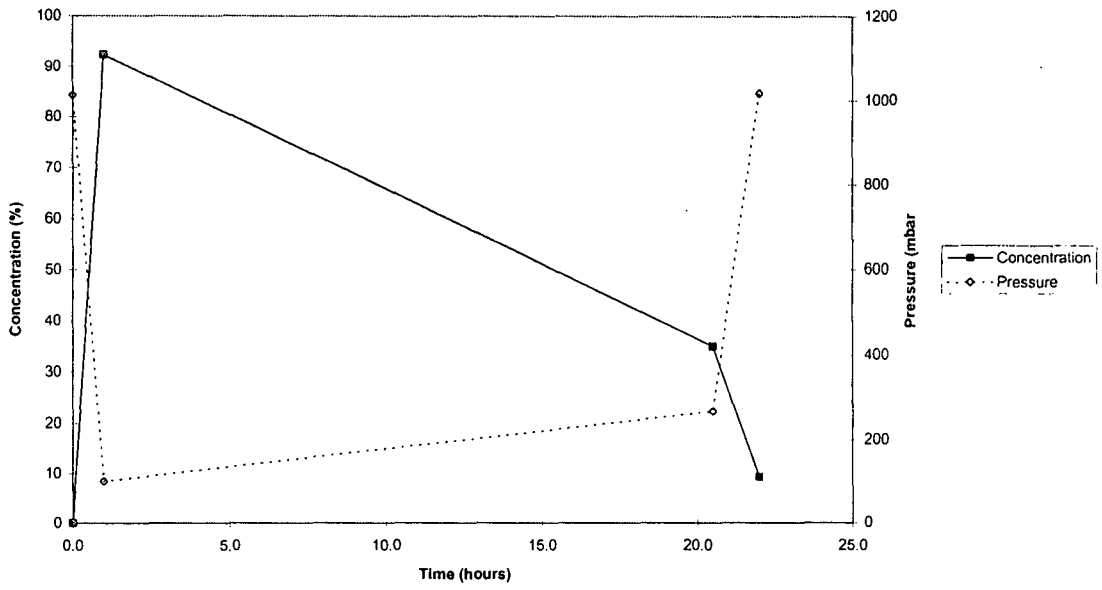


Fig 3.4. Test 2 - Two day carbon dioxide treatment in chamber 1 without re-dosing.

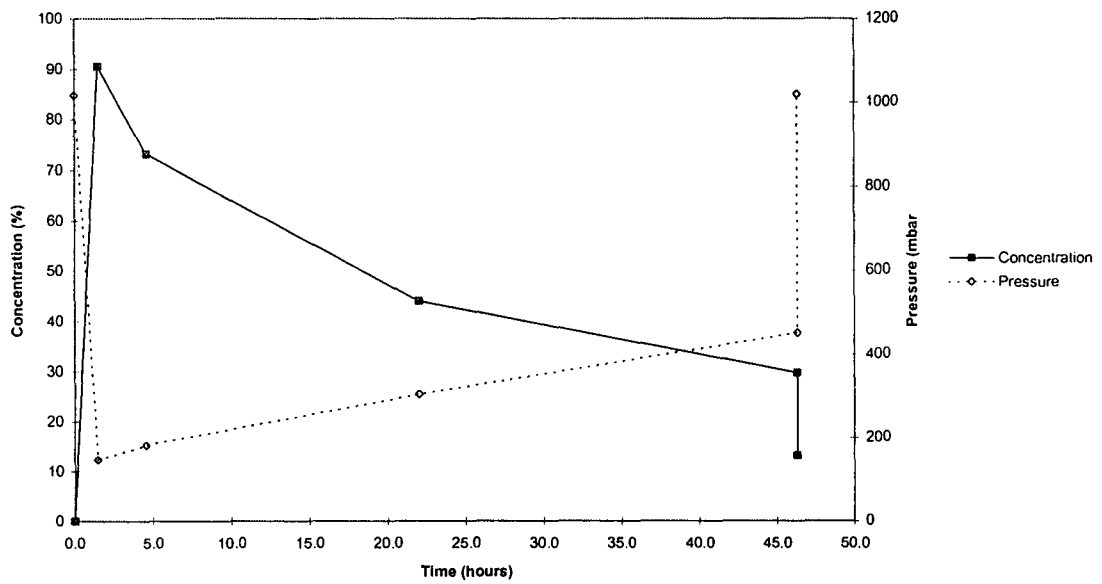


Fig 3.5. Test 3 - Three day carbon dioxide treatment in chamber 2 without re-dosing.

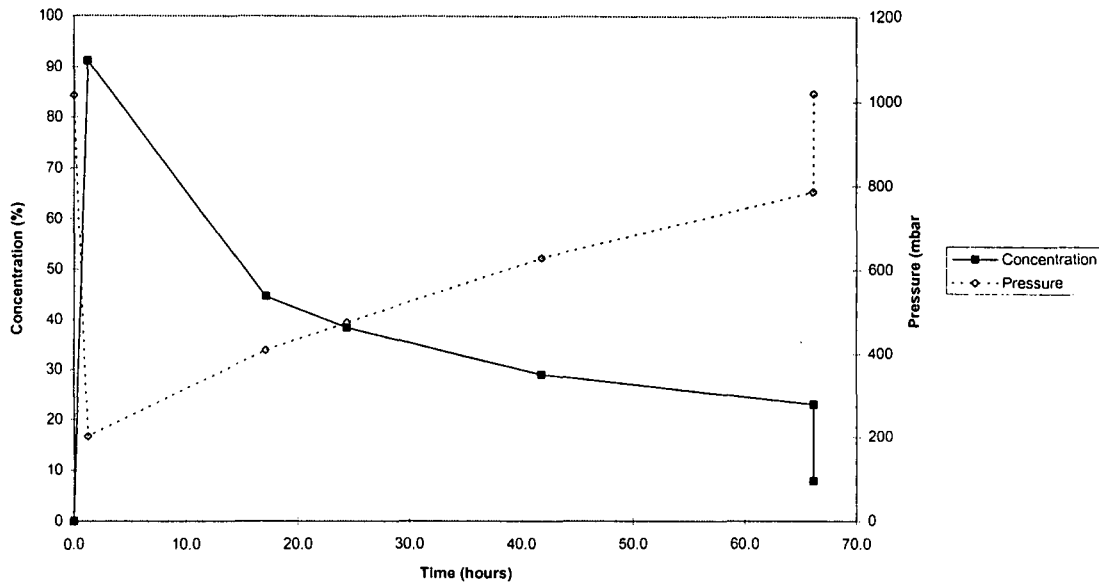


Table 3.1. The amount of carbon dioxide used, insect mortality the theoretical concentrations in the tests without re-dosing.

Test	Exposure (days)	Chamber	Amount of carbon dioxide used (kg)	Initial concentration (%)	Average concentration (%)	Starting pressure (mbar)	Egg survival
1	1	1	45	92.3	60.0	101.6	Survival
2	2	1	34	90.5	48.3	148	Survival
3	3	2	37	91.2	40.2	200	Survival

The leakage of air into the chambers had two consequences. These were the loss of vacuum and the decrease of the carbon dioxide concentration due to dilution. To overcome these problems in subsequent tests the chambers were re-dosed each day. Fresh bioassay insect samples were placed in the chambers before each test and the same date samples were left in the chambers. The initial dose and evacuation procedure was carried out as before and chambers were then left overnight at about 100 mbar.

Each day the chambers were evacuated to give the maximum possible vacuum, more carbon dioxide was added and a vacuum of about 100 mbar was re-established. The pressure in the chamber was measured at each stage and the theoretical concentration was calculated.

At the end of each test the chamber was brought back to atmospheric pressure and the concentration of carbon dioxide was then measured as before. The chambers were flushed with air and the insect samples were removed.

The power supply in the warehouse was occasionally interrupted during dosing. This meant that the cylinders could not be weighed and so some of the amounts of carbon dioxide had to be estimated from pressure readings.

Figs. 3.6 to 3.10 give the pressures and concentrations in the chambers against time in each test with re-dosing. Table 3.2 gives the amount of carbon dioxide used and the theoretical starting and average concentrations. The temperature varied between 17 and 27 °C. High numbers of *E. kuehniella* eggs hatched after both three and four day exposures. Emergence of adults of *E. ceratoniae* was noted from larvae treated at both these exposures.

Fig 3.6. Test 4 - Two day carbon dioxide treatment in chamber 1 with re-dosing.

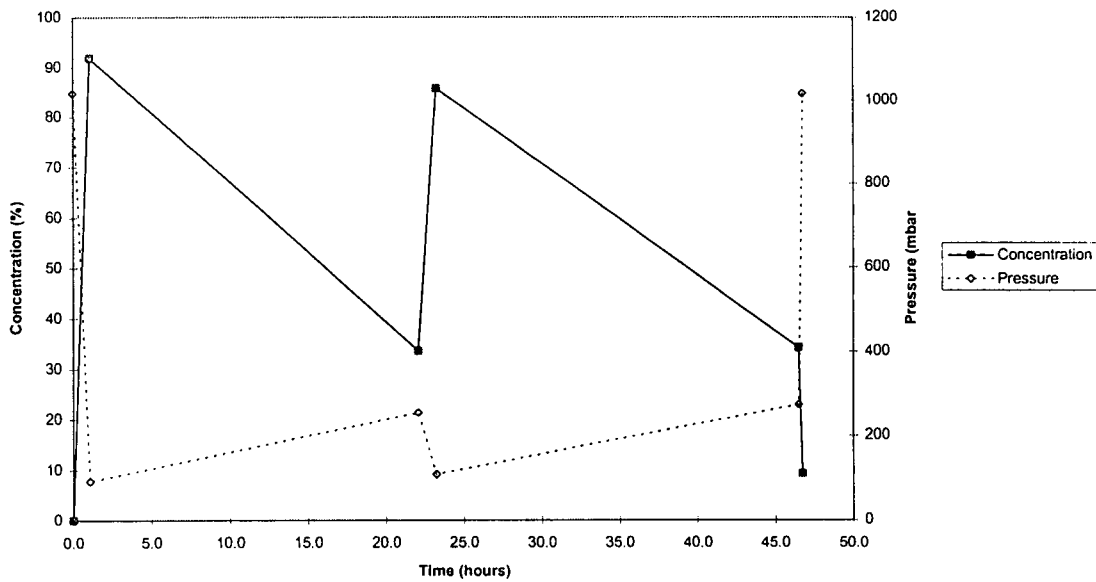


Fig 3.7. Test 5 - Three day carbon dioxide treatment in chamber 1 with re-dosing.

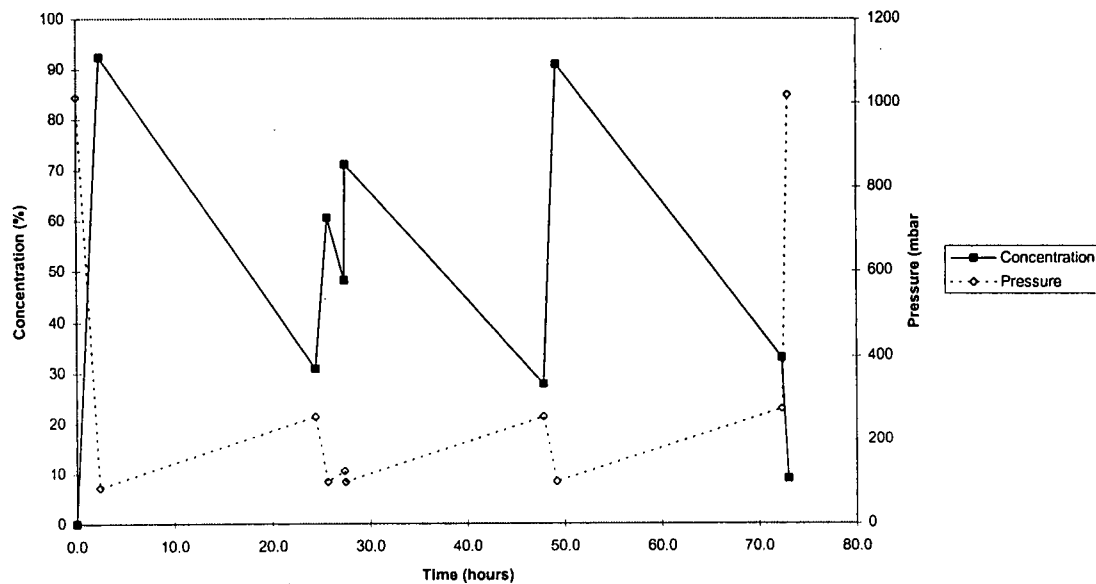




Fig 3.8. Test 6: Three day carbon dioxide treatment in chamber 2 with re-dosing.

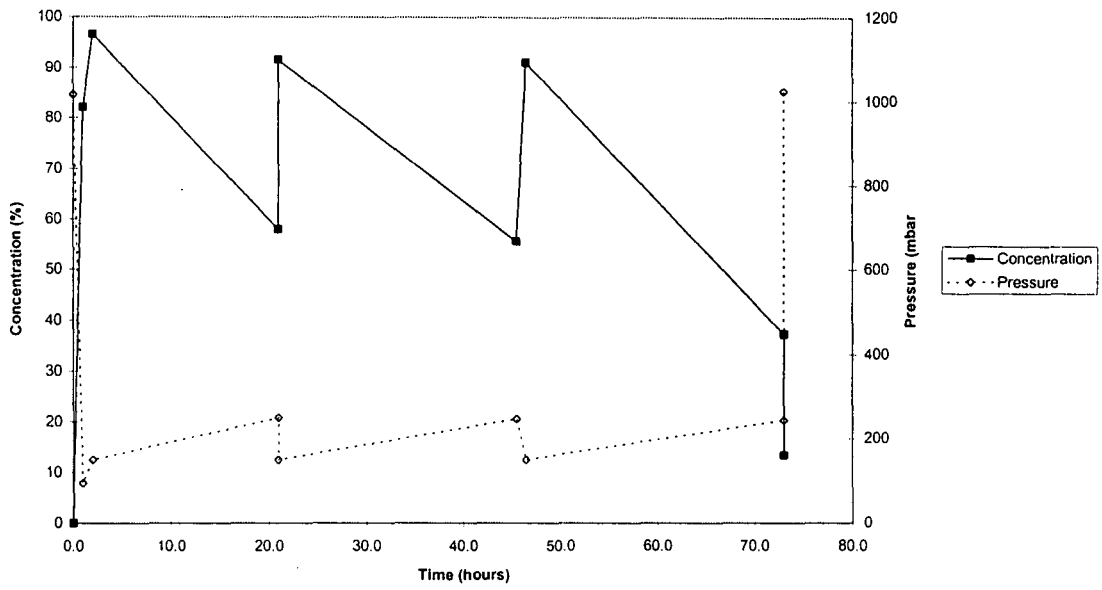


Fig 3.9. Test 7 - Four day carbon dioxide treatment in chamber 1 with re-dosing.

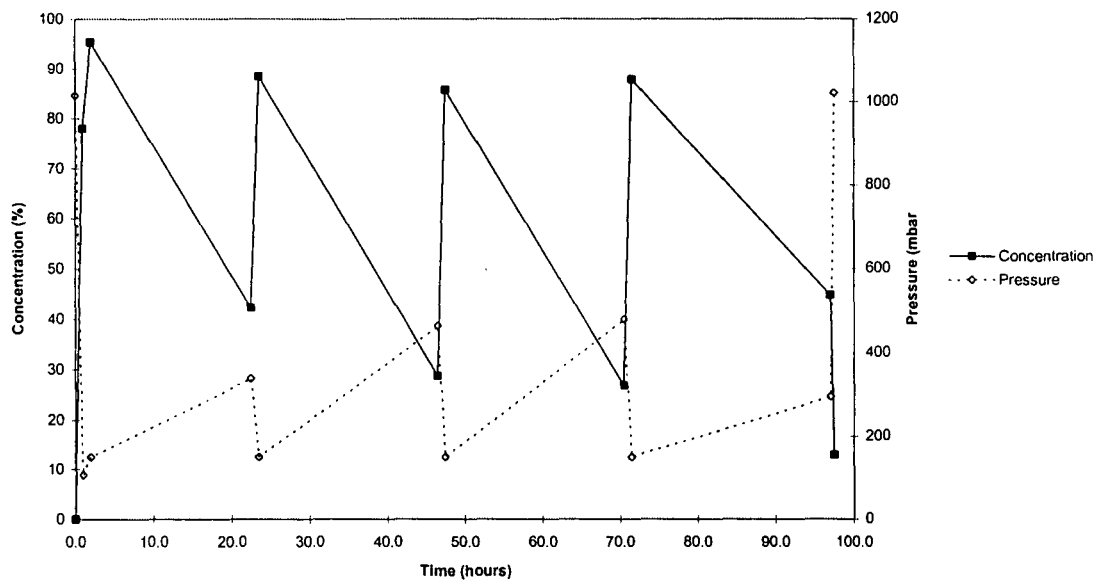


Fig 3.10. Test 8 - Four day carbon dioxide treatment in chamber 2 with re-dosing.

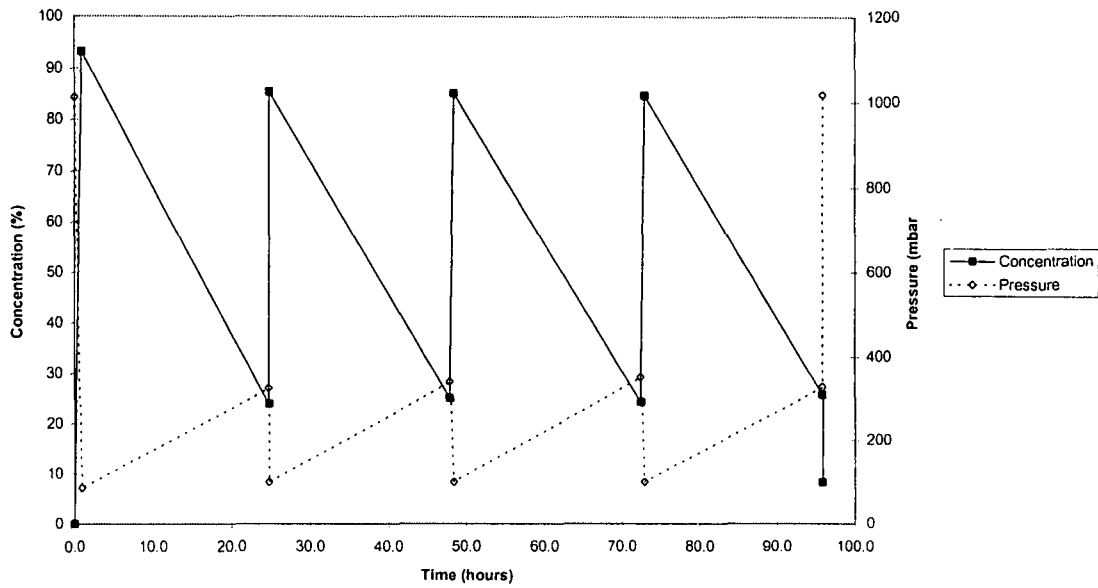


Table 3.2. The amount of carbon dioxide used and the theoretical concentrations in the tests with re-dosing.

Test	Exposure (days)	Chamber	Amount of carbon dioxide used (kg)	Initial concentration (%)	Average concentration (%)	Starting pressure (mbar)
4	2	1	47	91.8	60.7	93
5	3	1	55*	92.3	57.0	86
6	3	2	59*	96.6	70.9	150
7	4	1	71*	95.3	62.2	106
8	4	2	55*	93.2	55.8	86

\* Estimated from pressure readings

In all cases, except for test 5, the theoretical final concentration was within 15 % of the measured final concentration. In test 5 the theoretical final concentration was within 20 % of the measured final concentration.

### 3. Chamber tests in the United Kingdom.

A number of tests were conducted in a more gas-tight 1 m<sup>3</sup> vacuum chamber at CSL, York, UK in order to supplement the data from the tests at Medifruit. Liquid carbon dioxide (CO<sub>2</sub>) from a cylinder was used to dose the chamber in all the tests. After insertion of insect test samples, the chamber was evacuated to 10-20 mbar, charged with carbon dioxide and re-evacuated to the chosen starting pressure.

All life stages of the stored product beetles *Oryzaephilus surinamensis* and *Rhyzopertha dominica* were used in all tests. The cultures had been set up in plastic tubs on 22 January 1999 for use in Tunisia. In March, samples were flown to Tunisia and had returned to the UK unused because permission was not been given to use them at Medifruit. At CSL they were then kept at 20 °C and 50% rh until required for tests.

Five tubs of each species were used in each test. Three tubs of each species were kept in the same room for all the tests as controls. During the tests temperature ranged from 17.2 - 18.8 °C (av. ~18.0). After treatment insects were placed at 25 °C and 70% rh. All *R. dominica* samples were moved up to 30 °C and 60% rh after treatment. The cultures were checked for survivals periodically for up to 41 days after treatment. Survivors, seen as adults, were removed after each yield assessment count.

Table 3.3 shows the exposure time, pressures, theoretical concentrations of carbon dioxide and the survival of insects. A minimum of four days exposure was required for control of the most susceptible species.

**Table 3.3 Theoretical concentrations of carbon dioxide and insect survival for tests done at CSL.**

Test	Exposure (days)	Starting concentration (%)	Average concentration (%)	Starting pressure (mbar)	Survival of <i>Oryzaephilus surinamensis</i>	Survival of <i>Rhyzopertha dominica</i>
9	3	98.1	51.1	20	survivals	survivals
10	3	98.5	61.1	12	3 survivals	survivals
11	5	98	75.1	40	no survivals	survivals
12	2	98.9	74.6	37	3 survivals	survivals
13	4	98.9	75.6	42	no survivals	survivals
14	5	74.3	71.4	Atmospheric	1 survival	survivals

#### 4. Container trials.

Two 34 m<sup>3</sup> containers were to be modified to act as fumigation chambers suitable for use with phosphine and carbon dioxide. The first two containers delivered to Medifruit were not new and were considered to be unsuitable for the trials. They were replaced with two new containers fabricated in Tunisia. They were positioned outside the warehouse with container 1 nearest to the main door and container 2 to the right of container 1.

A two inch diameter hole was cut in one of the doors of each container and a piece of two inch pipe fitted with a ball valve was welded over each hole to act as an air inlet during airing and as a connection for pressure testing. Plates were welded over three out of the four ventilators on each container. The fourth ventilator was fitted with bulkhead fittings to introduce nylon gas sampling tubes and thermocouples used to measure temperatures in trials.

The pressure decay half lives of the containers could then be measured before they were modified further. The pressure was increased in each container by 6 mbar (600 Pa) over atmospheric pressure by connecting the outlet of a vacuum cleaner to the door inlet of the container. The valve on the inlet was then closed and the time taken for the pressure to reduce by half was then measured using a digital manometer and a stop watch.

In order to carry out phosphine or carbon dioxide treatments within a reasonably short exposure period the dates needed to be warmed above ambient temperatures and so an electrical heating system was installed into each container.

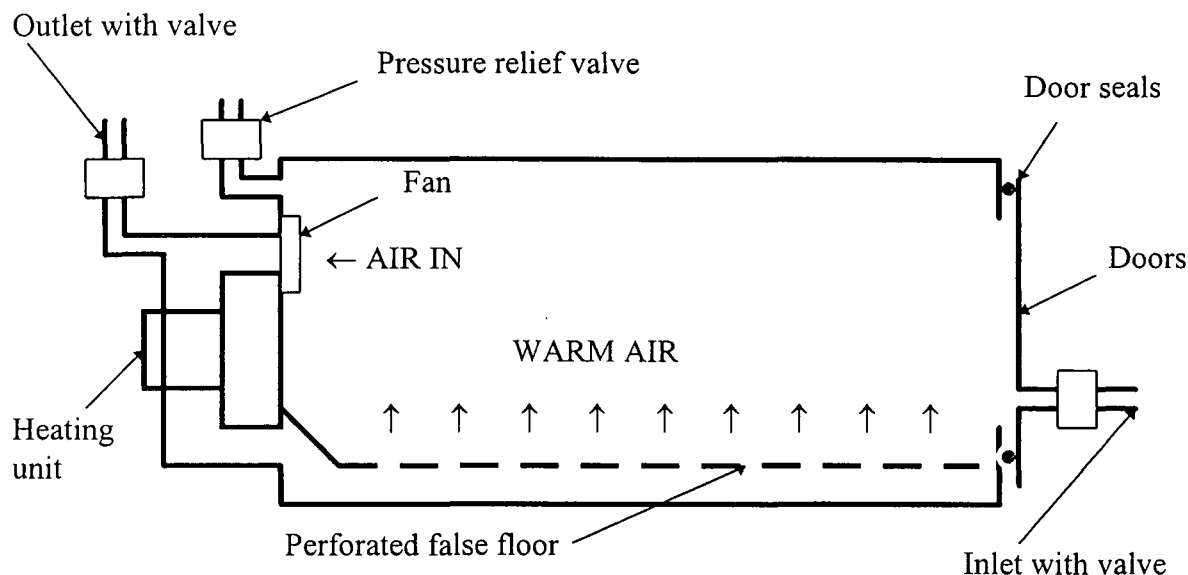
Two electrical heating units of 12 kW with the associated controls, square metal ducting and a high quality 50 Hz axial fan were fabricated in the U.K. and shipped to Medifruit. At a speed of 1420 revolutions per minute the fans were capable of a flow rate of up to 0.44 ms<sup>-1</sup> according the operating pressure (maximum 75 Pa). They were added to the rear of each container with the assistance of a local welder and electrician.

A perforated floor held clear of the container's metal floor was constructed from drilled sheets of plywood to spread the heat along the length of the container (Fig 4.1). The perforated floor contained 300 holes (18 mm diameter) in six rows of fifty. The height of the perforated floor and the number of holes was calculated to match the cross-sectional area of the inlet duct (0.1 m<sup>2</sup>).

The heating system would allow circulation of carbon dioxide with heating but this would not be possible in the case of phosphine which breaks down on the electrical heating elements. With phosphine the temperature in the container could be raised before the treatment but the heating unit would have to be switched off prior to dosing.

A two-inch outlet pipe, fitted with a ball valve, was added to the ducting of the heating system to act as a chimney when airing the container. A pressure relief valve containing oil was installed to the back of each container to prevent the build-up of pressure in the containers. These were used for pressure half-life tests.

**Fig 4.1. Modified container.**



The project required that the gas-tightness of the containers be assessed by a pressure half-life test so that a pressure of 500 Pa should not reduce to 250 Pa in less than 10 seconds.

The pressure half life was measured after modification using the Grainguard pressure relief valves designed for use on sealed grain silos and which were designed to actuate at 500 Pa. The level of liquid in the valve corresponding to 250 Pa had been marked by the manufacturer. The pressure in the containers was increased as before until the pressure relief valve actuated at 500 Pa. The valve on the chamber inlet was then closed and the time taken for the liquid in the pressure relief valve to drop to the 250 Pa mark was measured. To obtain a reasonable half life it was necessary to improve the door seals using grease. This was a temporary measure until the door seal could be modified in order to progress the trials.

The half lives given by the chambers before and after the addition of the heating system and the pressure relief valve are given in Table 4.1.

**Table 4.1. Pressure half lives of the chambers before and after modification.**

Container	Pressure half life given before modification (seconds)	Pressure half life given after modification (seconds)
1	13.4	16.2
2	6.9	2.0

The gas-tightness of container 2 decreased. Container 2 was also dosed with approximately 3 kg of methyl bromide to observe the loss of concentration and to search for leaks with an electronic leak detector. A source of leakage was found on the ducting of the heating system, around the door seals and from the container floor, a serious problem. The leaks on the ducting was sealed using mastic and PVC adhesive. This increased the half life to 4.2 seconds.

The heating system of container 1 was tested without dates by measuring the temperatures using thermocouples placed under the false floor at each end of the container. The ambient temperature in the container was 26 °C and so the thermostat was set at 40 °C. After 15 minutes the temperature at the front and the back was 33.1 and 37.2 °C respectively.

#### **Trials with aluminium phosphide tablets.**

Tests involving dates and insects began in May when it was not possible to use the heating system below 30°C because of the high ambient temperatures. A previous test on the empty container 1 showed that at a ambient temperature of 24°C and with the container heater thermostat set at 30°C, temperatures under the false floor were very similar at the front and rear of the container (average 29 °C) after an hour.

The floor of container 1 was covered in a layer of 10 pallets each containing 5 boxes of dates in a single layer, a total weight of 1004 kg. At an ambient temperature of 21.2 °C with the heater thermostat set at 30 °C, temperatures in boxes of dates at the front and rear were 25.3 and 26.3 °C, respectively after 3 hours of heating. The next day was much warmer and the solar heating caused the temperature in the container to rise. The thermostat read 37 °C at a setting of 25 °C.

Container 2 was dosed with 60 aluminium phosphide tablets to give a theoretical maximum concentration of 1.82 gm<sup>-3</sup> for a 7-day exposure. The actual maximum concentration achieved was well below this at 800 ppm or about 1.1 gm<sup>-3</sup> (60 % of theoretical) in this exceptionally leaky container. Container 1 was dosed with 54 tablets twice in separate trials to give a theoretical concentration of 1.64 gm<sup>-3</sup> over 3 and 5-day exposure periods. The actual maximum concentrations in these trials was 950 ppm or about 1.36 gm<sup>-3</sup> (83 % of theoretical) in the less leaky container. In all trials

dates infested with larvae and samples of *E. kuhniella* eggs and dates for tasting were included.

Figures 4.2 to 4.4 give the concentrations in the three container trials with aluminium phosphide.

Figure 4.2. Three day phosphine exposure in container 1.

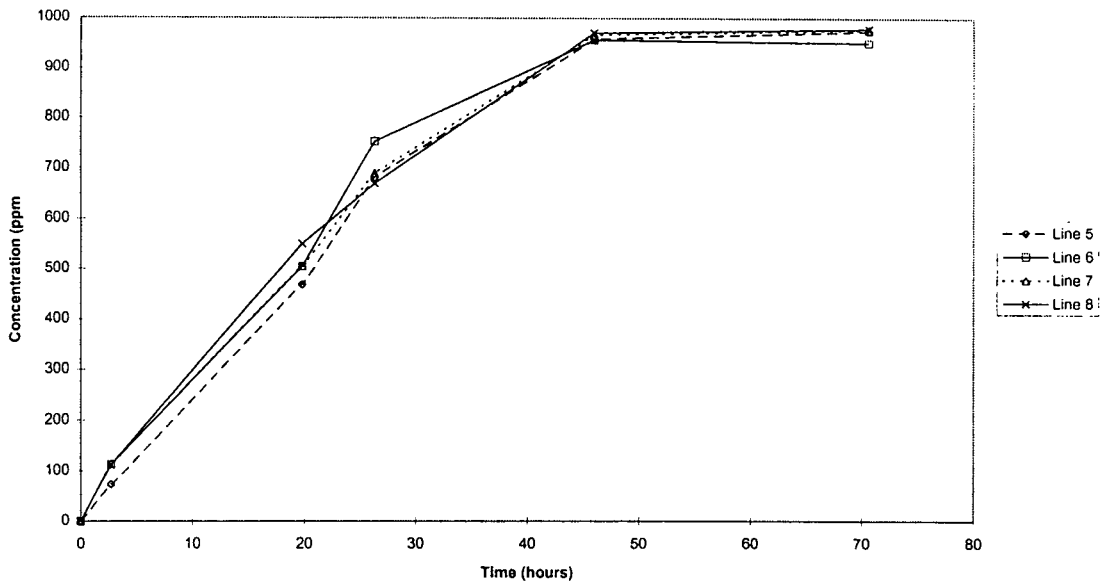


Figure 4.3. Five day phosphine exposure in container 1.

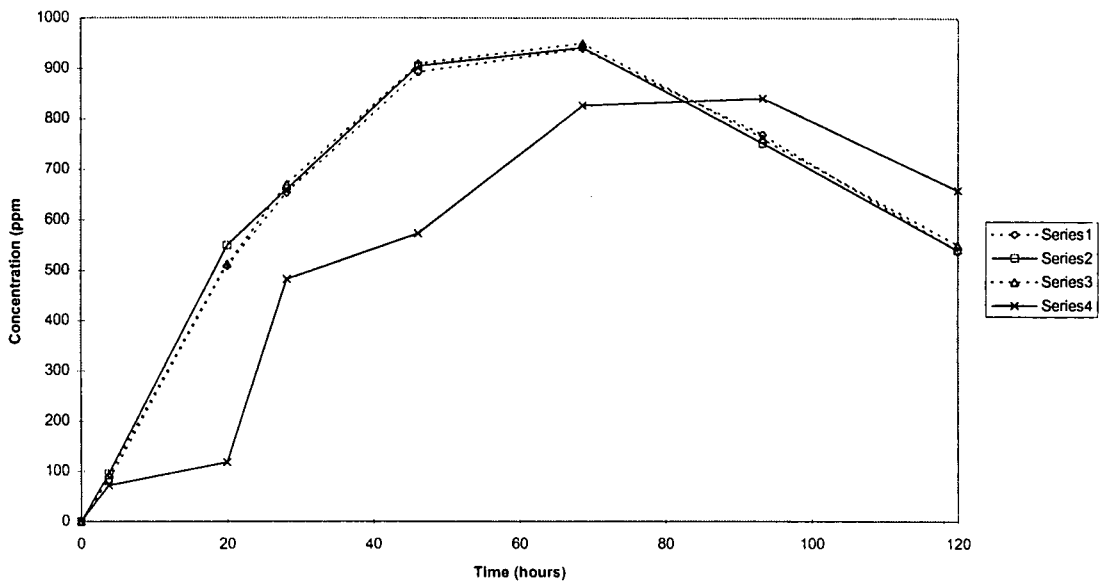


Figure 4.4. Seven day phosphine exposure in container 2

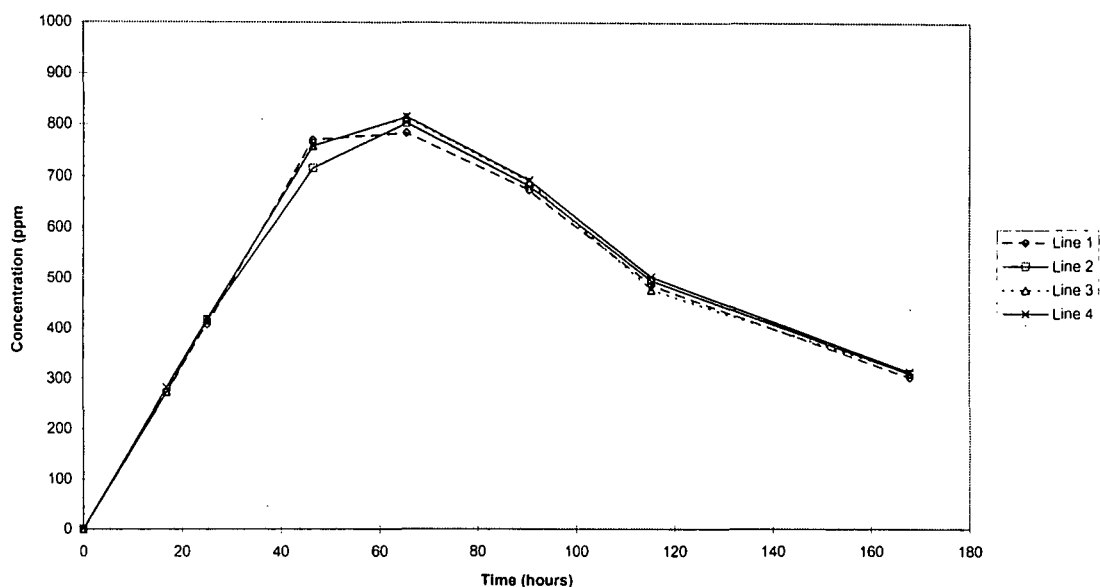


Table 4.2 gives the concentration-time products and average temperature for each test without heating using phosphine. The reduction in phosphine concentration was acceptable. Survival of *E. kuehniella* eggs was very low, only 3 eggs hatching after the 3-day exposure, 1 after 5-days and none at all after the 7-day exposure. No adults emerged from *E. ceratoniae* larvae treated for 3 days or longer.

Table 4.2 Concentration-time products and average temperatures for the phosphine tests in containers.

Length of treatment (days)	Container	Concentration-time product (g h m <sup>-3</sup> )	Maximum-Minimum Temperature (°C)	Average temperature(°C)
3	1	70	-	26.9
5	1	113	30-16	22.9
7	2	122	-	25.5

#### Trials with carbon dioxide.

Container 1 was fully loaded with approximately 9 mt of dates and Medifruit distributed 16 crates of infested dates in top and bottom positions down the left and right sides of the load. These were to be examined by Medifruit and there was no survival of larvae reported. Samples of *E. kuehniella* eggs and infested dates containing larvae of *E. ceratoniae* were located near the door for retrieval. The dates



were heated to between 21.5 and 32.9 °C using the heating system with the thermostat initially set at 35 °C. During the next morning, dates near to the ceiling had risen to a temperature of 42.4 °C. The container was dosed with carbon dioxide for a 4-day exposure period but despite careful manipulation of the heater controls, the temperature of the dates continued to exceed the required limit of 30 °C. A maximum-minimum thermometer in the container indicated temperatures of 21 - 42 °C.

The bioassay was retrieved and the container re-dosed with the same bioassay material for a 3-day exposure period. The same problem of ambient heating occurred with temperatures during the test being 24 - 37 °C. The dates were darkened in colour, fermented and unsaleable.

Figs 4.5 and 4.6 give the concentration against time and the average container temperatures for each test using carbon dioxide.

Figure 4.5. Average concentrations and temperatures for the three day carbon dioxide treatment in the container.

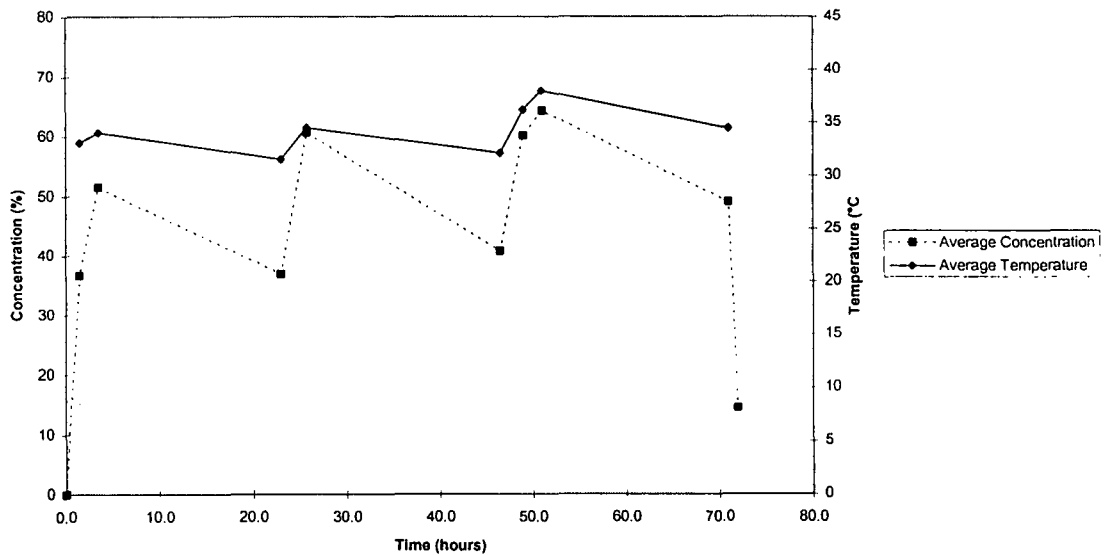


Figure 4.6. Average concentration and temperature in the four day carbon dioxide treatment in the container.

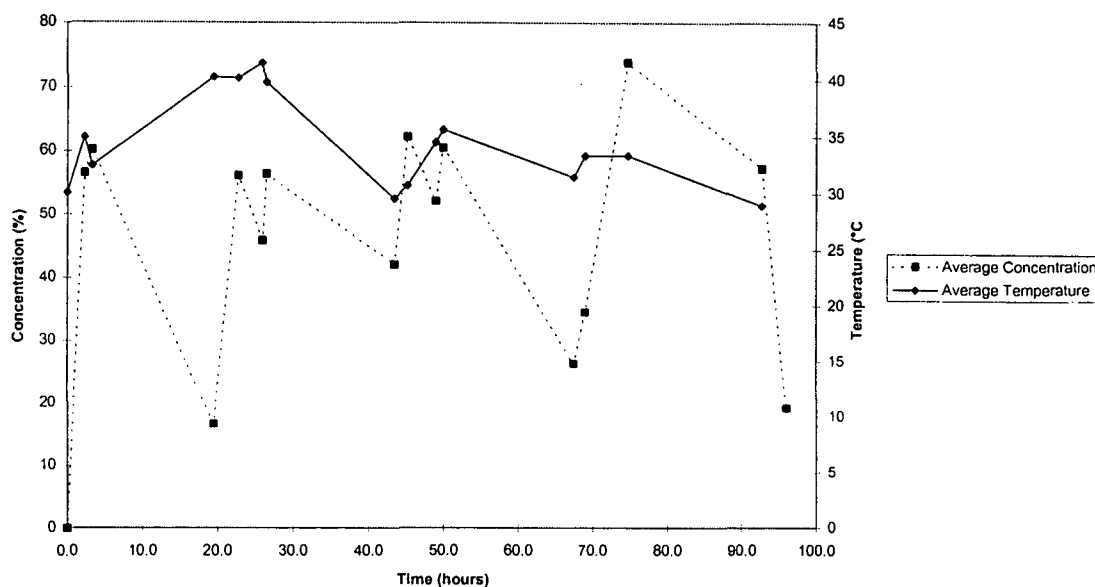


Table 4.3 gives the average temperature and the amount of carbon dioxide used. On day three of the three day test the electricity on the site was switched off and so the amount of carbon dioxide used on this day had to be estimated.

Survival of *E. kuehniella* eggs and emergence of moth adults was recorded after the 3-day but not the 4-day exposure.

Table 4.3 The amount of gas used and the average temperatures for the carbon dioxide tests in containers.

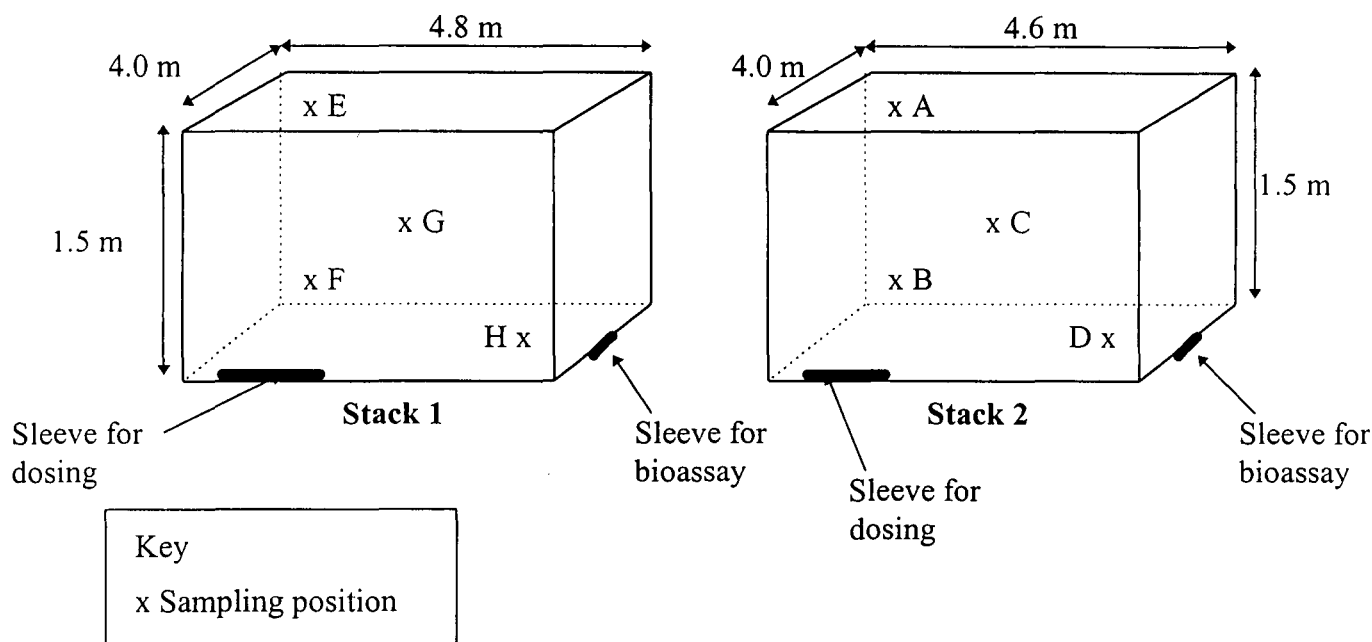
Length of treatment (days)	Container	Average Temperature (°C)	Maximum-Minimum Temperature (°C)	Amount of carbon dioxide used (kg)
4	1	34.4	21-42	235
3	1	34.3	24-37	120 (estimated)

This usage of carbon dioxide was excessive and loss was through the floors, doors and, particularly, the heating system. In fact the containers had to be re-dosed daily in order to maintain the concentration above the 45 % predicted to be reached during the night. Attempts were made to minimise gas loss by not running the leaky heating system during the day.

## 5. Stack trials.

Two stacks were built on 500 gauge (125 micron) polyethylene base sheets using a mixture of empty crates and crates filled with dates. Gas sampling lines and thermocouples were placed in various sampling positions in the stacks (Fig. 5.1).

Fig 5.1 Sampling positions in the stacks.



Specially fabricated PVC on mesh sheets were then placed over the stacks and fixed to the base sheet using PVC glue. Each sheet had two ports which could be sealed using screw on caps. One port was positioned on the top of each stack and the other was positioned near the bottom of one of the sides.

Two polyethylene sleeves were fashioned for each stack and placed between the base sheet and the covering sheet. One sleeve was to be used for dosing with phosphine and the other was to be used for the placement and retrieval of bioassay samples and samples of dates for taste testing. The ends of the sleeves were sealed using PVC tape.

The join between the base sheet and the covering sheet was then rolled and stuck to the side of the stack with PVC tape to leave as little of the polyethylene exposed as possible. This was done to minimise losses due to known permeation of carbon dioxide through the polyethylene.

The vacuum half lives of the two stacks were then measured by reducing the pressure in the stacks using an industrial vacuum cleaner and valve that had been fitted to one of the inlets. The valve on the inlet was then closed and the time taken for the pressure to increase half way to atmospheric was then measured using a digital manometer and a stop watch.

The half life given by both stacks was 7 seconds.

### Trials with carbon dioxide.

Eight tubes containing *E. kuehniella* eggs, samples of dates infested with *E. ceratoniae* larvae and samples of dates for taste testing were placed in both stacks using the polyethylene sleeve (Fig 5.1). Another two tubes of *E. kuehniella* eggs were placed in the warehouse office to act as controls. These hatched within 4 days.

The stacks were then dosed with carbon dioxide from cylinders using an electric vaporiser via the inlet on the side of the stack. The inlet on the top of the stack was left open throughout the dosing process to avoid the build-up of pressure. Stack 1 was dosed with 84 kg and stack 2 was dosed with 64 kg.

The concentration was monitored throughout the trials using an infra-red carbon dioxide analyser. Whenever the concentration threatened to go below 40 % the stacks were re-dosed. The day-time temperature in the bulk was measured using the thermocouples and the ambient temperature was monitored using a maximum-minimum thermometer.

The average day time temperature in the stacks was 21 °C and the ambient temperature varied between 15 and 26 °C.

Figs 5.2 - 5.4 give the carbon dioxide concentrations in the three trials.

Figure 5.2. Carbon dioxide treatment in stack one.

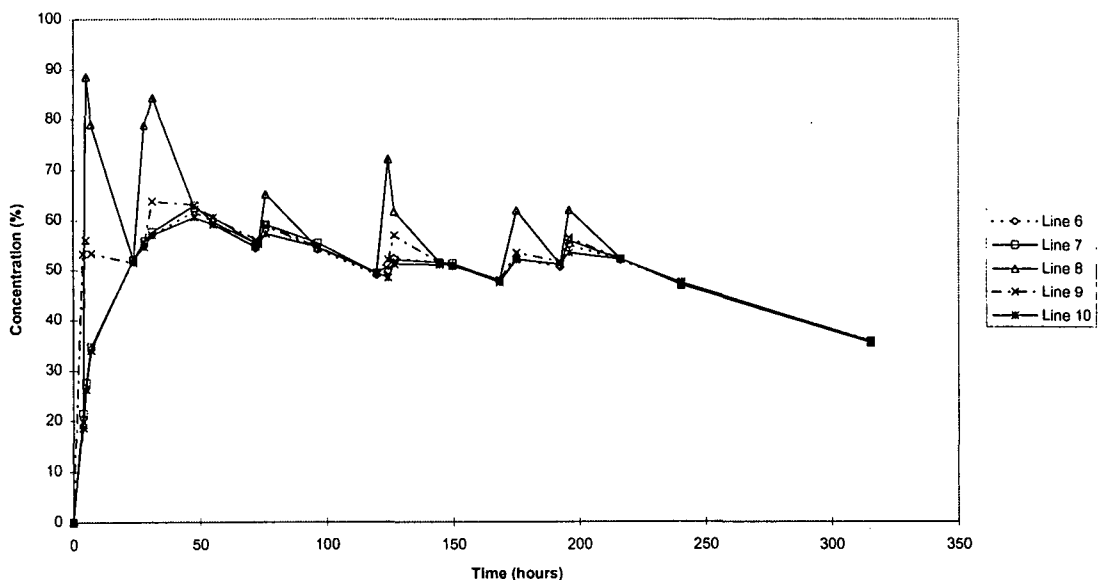


Figure 5.3. Carbon dioxide treatment in stack two.

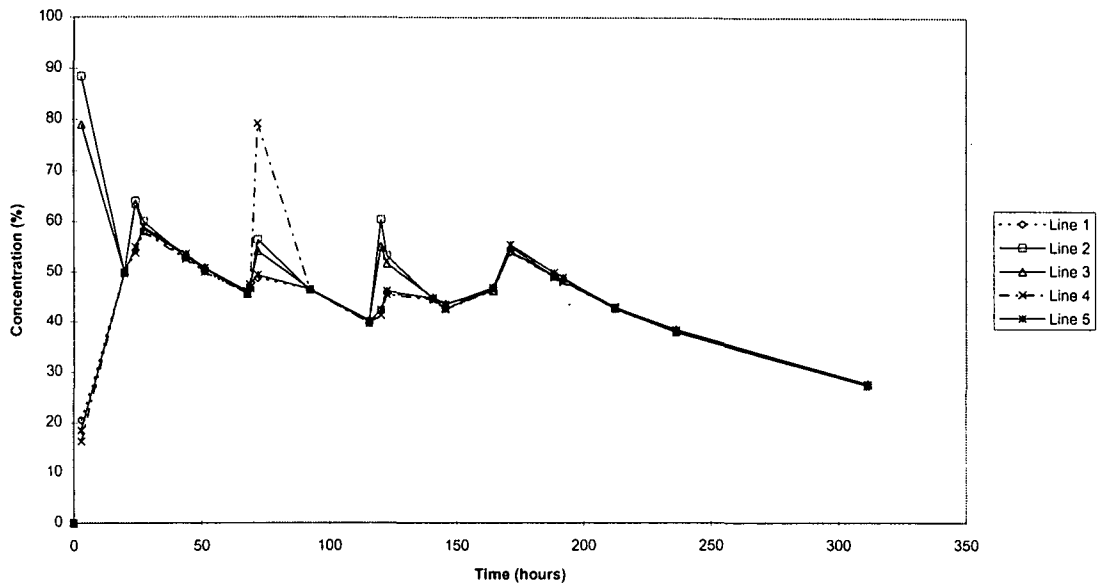


Table 5.1 The carbon dioxide used the concentrations and the survival of *Ephestia kuehniella* eggs from the first set of carbon dioxide stack experiments.

Test	Exposure (days)	Stack	Average concentration near the bioassay (%)	Average concentration in the stack (%)	Amount of carbon dioxide used (kg)	Survival of eggs
1	3	1	63.1	54.2	113	Survival
2	3	2	56.8	49.8	84	Survival
3	5	1	60.2	54.4	127	Survival
4	5	2	52.9	48.9	93	Survival
5	7	1	58.5	53.5	147	Survival
6	7	2	51.2	47.9	118	Survival
7	10	1	57.2	53.1	174	No survival
8	10	2	49.6	47.3	134	No survival

Insect samples and dates were removed from the stacks after 3, 5, 7 and 10 days exposure. The *E. kuehniella* eggs were placed in an incubator at 25 °C examined

periodically for 12 days after treatment. The infested dates were brought back to CSL for examination by breeding out the adults from surviving larvae of *E. ceratoniae*.

The experiment was then repeated in stack 2 using higher concentrations of carbon dioxide in an attempt to shorten the exposure period. In this case the average day time temperature was 23 °C.

In both trials carbon dioxide was added daily in order ensure that the concentration remained above 45 %.

Figure 5.4. Second carbon dioxide treatment in stack two.

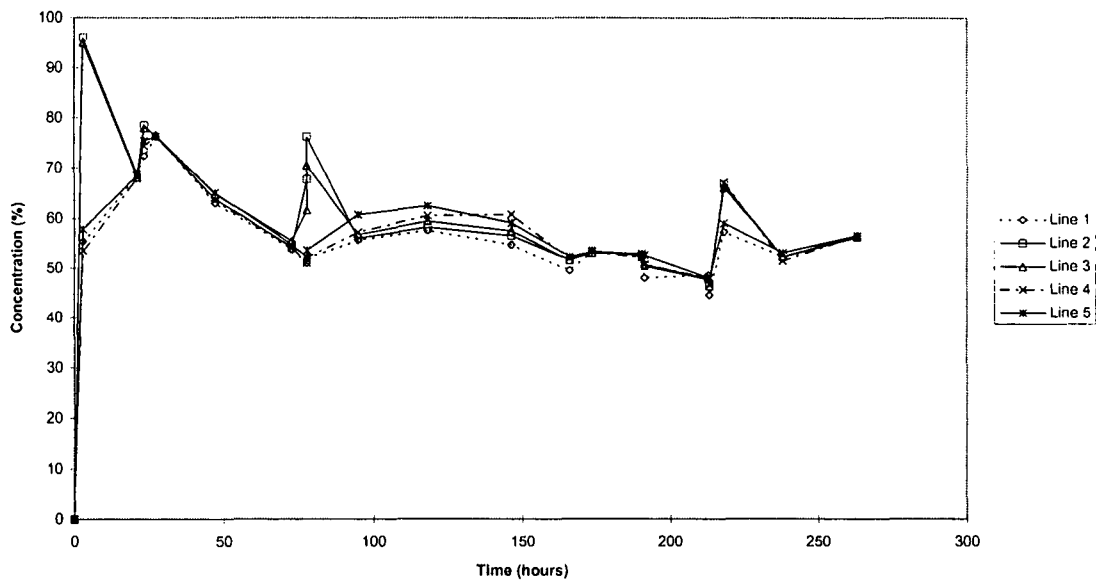


Table 5.2 gives the amount used and the concentration of carbon dioxide. Survival of *E. kuehniella* eggs was recorded up to 7 days of exposure. Adults of *E. ceratoniae* were obtained from larvae exposed for 3 and 5 days but not from those exposed for 7 and 10 days. There was no advantage noted from exposing larvae to a higher concentration, though results from a 6-day exposure may have shown a concentration effect.

**Table 5.2 Usage of carbon dioxide and concentration data from the second set of carbon dioxide stack experiments in stack 2 and survival of *Ephestia kuehniella* eggs.**

Test	Exposure (days)	Average concentration near the bioassay (%)	Average concentration in the stack (%)	Amount of carbon dioxide used (kg)	Survival of eggs
9	3	68.9	65.3	125	Survival
10	5	65.8	62.6	145	Survival
11	7	63.0	61.0	150	Survival
12	10	60.2	58.6	175	No survival

**Stack trial dosed with aluminium phosphide tablets.**

Bioassay samples and dates for taste testing were placed in stack 1 as before. The stack was then dosed using 46 tablets of aluminium phosphide through the polyethylene sleeve put in place for this purpose. The dose was equivalent to 46 g or 1.6 g m<sup>-3</sup> of phosphine. Samples were removed via the other sleeve after 3, 5, 7 and 10 days. The average day time temperature in the stack was 23 °C.

Table 5.3 gives the average concentration-time product (CTP) in the stack and the CTP by the insect samples. Survival of *E. kuehniella* eggs was recorded only for the 3-day exposure. Larvae of *E. ceratoniae* did not survive a 3-day exposure.

**Table 5.3 The concentration-time products in the stack during the tests with the aluminium phosphide formulation.**

Exposure (days)	Average concentration-time product near the bioassay (g h m <sup>-3</sup> )	Average concentration-time product in the stack (g h m <sup>-3</sup> )	Survival of <i>Ephestia kuehniella</i>
3	34	36	survivals
5	71	73	no survivals
7	109	112	no survivals
10	158	160	no survivals

## **6. Trials carried out in April 2000.**

No eggs of *E. kuehniella* were available for this work period due to the depletion of cultures at the Institute of Agronomy.

### **Estimation of the amount of methyl bromide used in the Medifruit vacuum fumigation chambers for the fumigation of dates.**

The primary objective in using vacuum fumigation is to hasten and improve the penetration of methyl bromide into the dates. It has a distinct advantage where a quick turnover of material is required such as the rapid post-harvest intake period. Medifruit aim to fumigate as much of the incoming dates as possible before they enter the cold store. There is an adverse physiological effect of high vacuum-induced rapid pressure changes on insects and vacuum also aids penetration into the insect and so improves efficacy. However, dosage schedules for vacuum fumigation are designed to achieve the CTPs required at atmospheric pressure by the use of shorter exposures than the normal 24 or 48-hour exposures used for methyl bromide. Vacuum fumigation should be viewed as a reliable high-quality treatment with a built-in efficacy factor. Medifruit did not carry out any atmospheric pressure fumigations with methyl bromide and so trials were confined to the vacuum chambers.

It was necessary to estimate the amount of methyl bromide used per tonne of dates treated in the normal fumigation procedure employed by Medifruit in their two vacuum chambers. Working with the regular fumigation operative enabled the chambers, their dosing system, the vacuum fumigation procedure to be assessed and any over or under-dosing quantified.

Medifruit fumigate using methyl bromide in the vacuum chambers, estimated to have an individual volume of 22 m<sup>3</sup> by CSL staff, in the following way. The chambers are evacuated to give a reading of 850 mbar on the vacuum gauge connected to the chambers. Methyl bromide is then dosed until a reading of 820 mbar on the vacuum gauge is achieved. During dosing the methyl bromide passes through an old electrically-heated vaporiser which is not switched on since vaporisation into a vacuum is very efficient. When dosing has finished the vaporiser is isolated from the chamber using a valve. The exposure period is a minimum of four hours but the period may be longer if the chambers were not busy or if the chamber is left under gas overnight. An old weighing balance adjacent to the chambers was not normally used but the regular operative estimated that approximately a kilo of methyl bromide was used in a typical fumigation.

At the end of the exposure period the chamber is restored to atmospheric pressure. The chamber is then vented by taking 2 successive vacuums of 850 mbar on the chamber gauge followed by restoration of atmospheric pressure with air diluting the remaining methyl bromide, a process known as 'air washing'. Finally, the door was opened and a period of at least an hour allowed before entering. This procedure was considered completely safe with the final atmospheric concentration estimated from detector tubes to be well below the EU Occupational Exposure Standard (OES) of 5 ppm (8-hour reference period).



The standard Medifruit method was monitored in three trials. In each trial atmospheric pressure, the pressure in the chamber before and after the introduction of methyl bromide and at the end of the exposure period were measured using a digital manometer. In each case the exposure period was the normal minimum of four hours. The concentration of methyl bromide was measured using a thermal conductivity meter once the chamber was restored to atmospheric pressure and before the chamber was evacuated. The chamber was vented using the standard Medifruit method of 2 consecutive 'air washes' already described.

In the first trial, chamber 1 was dosed in the usual way by the regular operative. The chamber contained packaging material and rolls of polyethylene film since no dates were available from the store. The amount of methyl bromide used in trial 1 was weighed as 2.5 kg by using the Medifruit balance although this was considered to be so inaccurate that it was not used in subsequent trials by CSL staff.

Trial 2 used chamber 1 again, this time loaded with 2 pallets of dates. The rubber hose from the methyl bromide cylinder split early during dosing. The split was repaired and dosing was re-started but the longer dosing time would have resulted so that the amount of methyl bromide entering the chamber was less than expected. A maximum-minimum thermometer in the chamber read 19-22 °C.

Trial 3 was in chamber 2 loaded with 2 pallets of dates.

In all three tests the atmospheric pressure was 1012 mbar. Table 5.4 gives the pressure readings and the theoretical concentration calculated from the pressure readings.

Table 5.4

<b>Trial</b>	<b>Vacuum chamber</b>	<b>Pressure before dosing (mbar)</b>	<b>Pressure after dosing (mbar)</b>	<b>4-hour Final pressure (mbar)</b>	<b>Theoretical concentration (g m<sup>-3</sup>)</b>
1	1	174	217	275	168
2	1	126	143	118.5	68.7
3	2	36.9	59.1	76.9	86.5

Table 5.5 gives the measured concentration of methyl bromide, the amount of methyl bromide used calculated from the concentration measurement and the estimated concentration-time product (CTP) for the three trials (concentration x 4).

Table 5.5.

Trial	Measured concentration (g m <sup>-3</sup> )	Mass of methyl bromide used (kg)	CTP product for a 4-hour exposure (g h m <sup>-3</sup> )
1	87.4	1.92	350
2	44.8	0.99	179
3	65.2	1.43	261
		Total = 4.34	

The amount of methyl bromide used was estimated from the final concentration readings and varied between 0.99 and 1.92 kg for the 22 m<sup>3</sup> chambers. It is clear that the dosing system was inadequate to guarantee accurate dosing. At an atmospheric pressure of 1012 mbar the concentration due to an increase in pressure of 30 mbar on the vacuum gauge should give a concentration of 2.96 % or 117 g m<sup>-3</sup>. It appears that the vacuum gauge is not sufficiently accurate for this purpose. The methyl bromide cylinder was weighed on the store weighbridge (readable to 1 kg) and a total of 6 kg was used for the 3 trials. This is broadly in line with the more accurate figures given in table 5.5 (total 4.34 kg) given the accuracy of the weighbridge and the methyl bromide lost in trial 2 when the hose split.

Table 5.6 gives the theoretical and measured concentrations of methyl bromide and the difference between them.

Table 5.6.

Trial	Theoretical concentration (g m <sup>-3</sup> )	Measured concentration (g m <sup>-3</sup> )	Difference	Difference as % of theoretical concentration.
1	168	87.4	2.04	48.0
2	68.7	44.8	0.61	35.1
3	86.5	65.2	0.54	24.7

The difference between measured and theoretical concentrations is due to two factors: sorption of methyl bromide and the inward leakage of air causing dilution. It was known that the vacuum system had a small leak and it was suspected that the vaporiser also leaked. When under vacuum the leakage would be inwards and would be greatest during dosing since the vaporiser is in use. The inward leakage would be unlikely to be consistent which explains the wide variation in the difference between measured and theoretical concentrations.

The effect of the vacuum would tend to minimise sorption of methyl bromide onto the dates and the wooden pallets in the second and third trials. However, some sorption would have occurred once the chambers were returned to atmospheric pressure at which point the concentration was measured. The highest difference of 48.0 % between theoretical and measured concentrations of methyl bromide can be partially explained by the effect of increased sorption on the materials in the chamber in trial 1.

The difference in these estimates of methyl bromide dosed in trial 2 compared with trial 3 may be due, in part to the increased dosing time and consequential loss of vacuum in trial 2. This is reflected in the difference in the amount of methyl bromide dosed i.e. only 0.99 kg in trial 2. The lowest figure for the difference between theoretical and measured concentrations of methyl bromide of 24.7 % determined in trial 3 This is the maximum possible figure for the effect of sorption if it is assumed that there was no inward leakage in this case. However, it is certain that there was leakage and so this must be an overestimate.

It should be noted that only 2 pallets of dates were fumigated in trials 2 and 3 since sufficient dates to load the fully chamber were not available. Dates have a relatively high oil content and are known to be a relatively sorptive commodity when constructing methyl bromide fumigation schedules. Anon. (1993) recommends, at 10-20 °C, a dosage of 50 g m<sup>-3</sup> for a fumigation of 48 hours for this commodity. In comparison, rice, peas, beans, cocoa beans and dried vine fruits require a dosage of only 15 g m<sup>-3</sup> for a shorter fumigation of 24 hours.

The 4 pallets of dates used in these trials were underweight since they weighed only a total of 2.824 tonnes, i.e. an average of 0.706 tonnes gross each. It had been determined previously that fully loaded pallets of dates can weigh an average of 0.901 tonnes gross and that deducting the weight of 35 plastic crates (total 70 kg) and the weight of an average wooden pallet (26.7 kg) indicates that the average pallet load is 0.804 tonnes net. Medifruit indicated that a chamber can hold up to 10 pallets of dates and so a fully loaded chamber would hold 8 tonnes net of dates and this loading would increase the concentration of methyl bromide by reducing the free gas space.

The amount dosed in trial 1 (1.92 kg) was almost twice what the operative intended. The average of this and the amount estimated to be dosed in trial 3 was 1.67 kg The Medifruit dosage rate was, therefore, estimated to be 0.21 kg of methyl bromide per tonne net of dates for a fully loaded chamber containing 8 tonnes.

The CTP obtained in chamber 2 of 261 g h m<sup>-3</sup> is excessive even for fumigations at atmospheric pressure at ambient temperatures. Bell (1976) determined the doses required for the control of pupae, larvae and eggs of four species of the family (Pyrilidae) to which *Ectomyelois ceratoniae* also belongs. At 15 and 20 °C the most tolerant stage was the pupae and those of the most tolerant species were controlled by a CTP of 64 g h m<sup>-3</sup> at both temperatures and by 53 g h m<sup>-3</sup> at 25 °C. The United States Department of Agriculture recommends a dose of 40 g m<sup>-3</sup> for an exposure period of 3 hours to a sustained vacuum above 15 °C for the treatment of dates and other loose dried fruit. If this concentration was achieved consistently in a 4-hour exposure there should not be any survival of *E. ceratoniae* larvae. If the chamber had been fully loaded the sorption of methyl bromide would have increased but the reduction in free space volume would have easily compensated for this effect.

The amount of methyl bromide routinely dosed in the Medifruit chambers indicates an unjustifiable safety margin for the control of *E. ceratoniae* larvae. This dose can safely be halved and this should be done as the process of phaseout continues in order to conserve stocks before alternatives are implemented. The presence of moths noted

by Medifruit staff and trapped in the facility during the current project may indicate that fumigation is not always carried out consistently to the Medifruit method. However, it is more likely that adult moths emerged from unfumigated dates which have been held in the cold store and await fumigation or processing for a period at ambient temperatures.

### **7. Trials in freight containers.**

The remedial sealing work on the floor and doors of both containers was not completed on arrival of the CSL team and they were disconnected from the electricity supply. Additionally, the containers had been moved so that the doors now faced the packing facility for ease of loading. In moving the containers both pressure relief valves had been damaged beyond repair. A single spare valve was fitted but this meant that the planned test schedule could not be adhered to since each container could only be used singly. In fact only container 1 was finished and available for tests.

### **Final modifications to containers.**

The containers had been modified under Medifruit's direction and with CSL's agreement. A flooring resin had been applied to the floors of both freight containers and was visible in container 2 as a layer 4-5 mm in thickness. It was found to be cracked in places but was considered to be an improvement to the floor seal, especially when a new floor was placed on top. Container 1 had been fitted with a metal floor of steel sheets of 2-3 mm thickness which had been welded together and to the sides of the container. Three I-beams were in place and welded in position to support the secondary perforated wooded floor. Two were located along the length of the containers sides and a third was located centrally along the length of the container. The space between the side I-beams and the wall was filled with sealant. The additional floor height meant that the top of the I-beams were level with the top of the false wall around the floor. The doors on container 1 had been welded together along the central joint with strengthening bars along the top and bottom of the door and hinged on the right. A thin metal flange had been welded round the perimeter of the container doorway to hold a high-quality compressible rubber gasket onto which the door could be tightly pressed with the aid of 3 threaded studs welded to the left side of the container doorway to receive stout metal bars and nuts tightened with a spanner. This arrangement provided a very gas-tight door similar to those necessary on fumigation chambers.

The completed container 1 was pressure tested. Initially, the pressure relief valve was used and a pressure decay half-life of 50 seconds estimated. Using a digital manometer, an initial pressure of 639 Pa reduced by 50% in 39.3 seconds and by 75% in 66.5 seconds. A second test produced a half-life of 36.4 seconds from a pressure of 642 Pa and a third test gave a half-life of 35.9 seconds from a pressure of 644 Pa. The average of these tests is a pressure half-life of 37.2 seconds on the container with the door seal untouched. This sealing standard is well in excess of the required standard of 500-250 Pa in not less than 10 seconds.

### **Heating test on empty container 1.**

The red-painted container situated outside experienced significant solar gain in temperature during the afternoon. Within 2 hours the temperature rose from a mean of 36.6 °C (mean of 4 locations, range 34.9-36.3 °C) to a mean of 40.2 °C (range 39.7-40.5 °C) when the heaters were on at 12 kW power with the fan running at a thermostat setting of 30 °C. This immediately had serious implications for using the containers when loaded with dates which were not permitted to be heated beyond 30 °C in order to preserve their quality. Medifruit planned to install modified containers inside the building but it was not possible to re-locate these two experimental containers in the shade nor to paint them white to reduce solar gain.

The heating thermostat was set at 30 °C and the heaters operated at 12 kW power with the fan running for an hour with the ambient temperature 23.9 °C and a wind speed of 2 m s<sup>-1</sup>. After an hour the heaters were switched off and the fan continued to run. After a further hour the mean temperature was 31.1 °C (range 30.6-31.6 °C). At this point the thermostat was re-set to 22.5 °C, in view of the previously high temperatures produced, with the fan running and an ambient temperature of 22.4 °C. The temperature of the container reduced to a mean of 25.0 °C (range 24.6-25.5 °C) after a further 3.25 hours.

The thermostat was then re-set to 30 °C, again at 6 kW heater power and the mean temperature was 24.3 °C (range 24.0-24.8 °C) after 30 minutes when the ambient temperature had reduced to 18.5 °C (20.15 h). It appeared that the 6 kW power setting was insufficient to produce rapid heating and so it was increased to 12 kW. After only 15 minutes the mean temperature started to increase to 26.0 °C (range 25.5-26.6 °C) when the power setting was again reduced to 6 kW to run overnight.

### **Phosphine trial in a loaded container 1 by dosing with phosphine in carbon dioxide.**

The following morning the temperature in the container was a mean of 33.9 °C (range 31.8-36.0 °C) showing that early morning solar heating together with a 6kW setting over-rode the thermostat setting of 30 °C. A maximum-minimum thermometer placed in the container at the beginning of the heating trial read 20/43 °C. The minimum temperature indicates that the 6 kW setting was insufficient to maintain temperature at low night ambient temperatures. It would, therefore, be necessary to insulate the containers, perhaps by a spray-on polystyrene foam, even if they were housed indoors for economy of electricity.

The heating system was switched off and 6.272 tonnes of dates were transferred from a chamber to container 1 held on specially sized metal pallets half the width of the container. The container temperature reduced to 22 °C within 30 minutes of the doors being opened. It was considered prudent to cover the roof of the container with wooden pallets and wooden planks in order to provide a shaded air space above it in case of strong sun. Gas sampling tubes and thermocouples were located within the container in positions 5-8 as follows:

- 5 - right rear bottom.
- 6 - right near door bottom.
- 7 - right near door top.
- 8 - right rear top.

A maximum-minimum thermometer was located at position 6. The temperature further reduced within the container as the solar heating reduced so that after 6 hours the mean temperature was 20.1 °C (range 19.4-20.8 °C) with the maximum-minimum thermometer reading 19 °C at 14.50 h. At this point the container was sealed and the heating system switched on with the thermostat set at 30 °C and a 6 kW power setting. After 50 minutes the mean temperature had remained virtually unchanged at 20.2 °C (range 19.4-20.8 °C) with an ambient temperature of 19 °C. The container temperature remained unchanged after a further hour when the ambient temperature reduced to 14 °C (16.50 h).

The following morning it was discovered that the container heating system had been mistakenly switched off by Medifruit staff for a short period. At 09:30 h the container temperature had reduced to a mean of 19.4 °C (range 18.6-19.3 °C) at an ambient temperature of 17 °C. The heating system was switched on at the previous settings resulting in a steady rise in temperature over 3 hours to a mean of 23.1 °C (range 21.1-25.2 °C) with clear evidence that the temperature at the bottom of the container exceeded those at the top by a mean of 3.3 °C, indicating that the improvised shading on the roof of the container was working. After a further hour the container temperature had risen to a mean of 24.1 °C (range 22.1-26.6 °C) with the top-bottom differential maintained at an ambient temperature of 24 °C. After a further 2.5 hours, during which the sun was obscured by cloud for an hour, the mean temperature was 26.5 °C (range 24.7-29.1 °C) with the ambient temperature reducing from 25 to 23 °C and the wind measured at 0.6 gusting to 1.5 m s<sup>-1</sup>. After a further 45 minutes the container temperature had hardly increased and so a 15 minute period at a thermostat setting of 40 °C was included in order to boost the temperature up to near 30 °C before dosing phosphine.

Before dosing, the temperature in the container was a mean of 27.4 °C (range 25.0-32.6 °C) at an ambient temperature of 19.4 °C. At this point it was decided to discontinue the heating since the highest temperature recorded had exceeded the recommended treatment temperature and the lower temperatures had to be accepted. A bioassay of infested dates together with uninfested dates for tasting were inserted into the container at gas sampling position 6 to remain for a 4-day exposure period.

The contained was dosed with cylinderized 2% w/w phosphine in carbon dioxide. To dose 1.5 g.m<sup>-3</sup> in a 34 m<sup>3</sup> container it is necessary to apply 1.5 x 34 g = 51 g of phosphine. Using a 2 % w/w mixture the weight of mixture required is 50 x 51 g = 2.55 kg. Using the weighing equipment available it would be difficult to weigh precisely the correct amount. It was estimated that 3 kg was dosed by using the Medifruit weighbridge and 2.6 kg by using ordinary bathroom scales, the more accurate estimate which equates to 52 g of phosphine. (N.B. The CSL accurate electronic balance was held in Syria following a UNIDO demonstration project). After dosing, the fan was run for 5 minutes without heating in order to mix the phosphine in

the container. Soon after dosing, initial concentration readings were 690, 1000, 840 and 1240 ppm at positions 5-8 respectively (17.17 h).

Table 5.7 gives the phosphine concentrations in ppm and the resulting CT products calculated after converting ppm to  $\text{g m}^{-3}$ . The concentration profiles are shown graphically in figure 5.5. An examination of the concentrations indicates that the distribution of the phosphine, while not completely uniform, is very satisfactory and that the loss of concentration with time is very low indeed. The average CT product was  $100.4 \text{ g h m}^{-3}$ .

After a lapsed time of 91.5 hours the container fan was switched on with the valves on the door and chimney open to 'air-wash' the atmosphere. After 3 hours the concentration had reduced to a mean of 145 ppm at which point the door was opened and the bioassay samples and tasting dates retrieved. The internal maximum-minimum thermometer read  $11/26 \text{ }^{\circ}\text{C}$ . Normal operation would demand that the concentration was lower than this, especially if the container were housed in a building, in order not to exceed the Occupational Exposure Standard (OES) of 0.3 ppm. This could be achieved by using a longer air-wash time aided by wider chimney and valve of 10 cm diameter. However, during busy periods it would be necessary to air, unload, re-load and re-dose as quickly as possible. The airing would be much more rapid with the connection of a vacuum pump to the chimney in conjunction with the fresh air intake on the door. The pump dedicated to the vacuum chamber would be suitable if re-furbished. Further trials would be required using a modified venting system.

Table 5.7. Phosphine concentrations and CT products obtained in a trial in the modified freight container 1 dosed with phosphine in carbon dioxide.

Date/Time	Lapsed time (hours)	Corrected phosphine concentration (ppm)			
		Position 5	Position 6	Position 7	Position 8
05/04/00 17:00	0	0	0	0	0
05/04/00 17:17	0.28	690	1000	1240	840
06/04/00 10:00	17.00	1250	900	980	930
06/04/00 12:00	19.00	1030	900	970	980
06/04/00 14:00	21.00	990	820	940	930
06/04/00 14:30	21.50	970	800	900	910
07/04/00 09:40	40.67	860	710	820	840
07/04/00 13:50	44.83	870	700	830	810
08/04/00 10:25	65.42	810	660	700	600
08/04/00 16:15	71.25	770	660	740	740
09/04/00 12:30	91.50	740	640	720	720
09/04/00 13:50	92.83	720	630	690	700
09/04/00 15:45	94.75	160	130	150	140
		<b>4-day CT product (ghm<sup>-3</sup>)</b>			
		<b>113.4</b>	<b>97.6</b>	<b>101.3</b>	<b>89.3</b>



Figure 5.5. Concentrations of phosphine in container 1 dosed with phosphine in carbon dioxide.

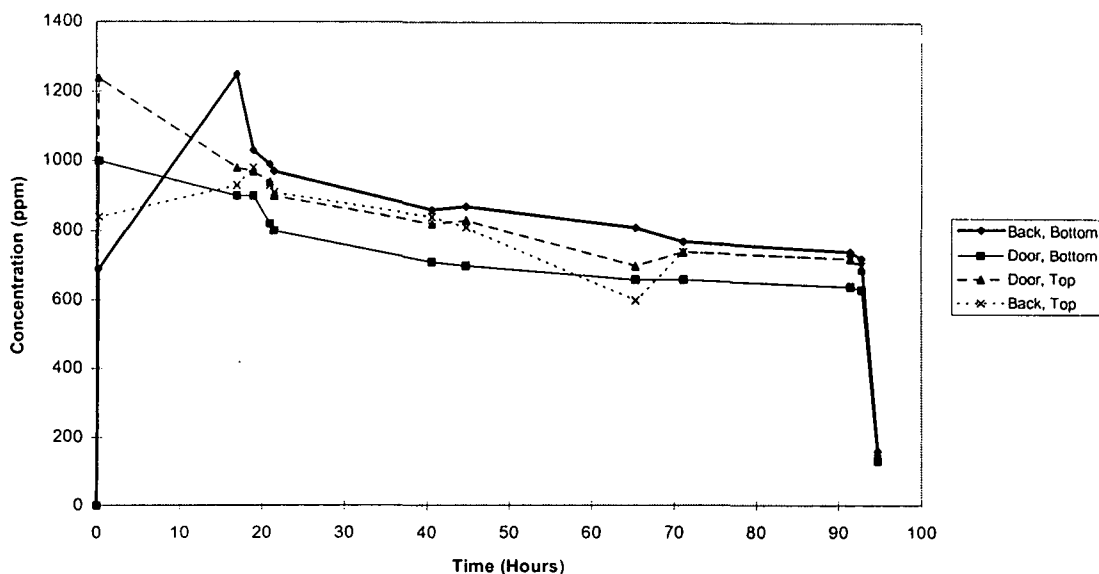


Table 5.8 gives the temperatures within the container and the ambient during the trial.

Table 5.8. Temperature in container 1 trial after dosing with phosphine in carbon dioxide.

Date/Time	Temperature °C				Ambient
	Position 5	Position 6	Position 7	Position 8	
05/04/00 17:00	26.2	32.6	25.7	25.0	
06/04/00 10:00	19.2	18.7	21.9	20.7	
06/04/00 12:00	20.1	19.4	24.2	21.1	
06/04/00 14:00	20.9	20.5	27.0	21.6	18.0
07/04/00 09:40	16.7	16.4	20.1	18.5	16.0
07/04/00 13:50	18.4	18.4	27.2	19.5	17.0
08/04/00 10:25	16.2	15.8	20.5	17.4	
08/04/00 16:15	17.2	17.5	20.3	18.6	
09/04/00 12:30	15.8	16.0	16.2	16.7	
09/04/00 13:50	16.1	16.1	17.3	16.7	
<b>Mean</b>	<b>18.7</b>	<b>19.1</b>	<b>22.0</b>	<b>19.6</b>	
<b>Internal maximum-minimum</b>		<b>11.0 - 26.0</b>			

As would be expected in an uninsulated container the temperature steadily reduced over the 4 days. There would be a different pattern of heat loss if the container was in the building since it would not experience the same low night temperatures and the high daytime temperatures and the pattern of heat loss would depend on the time of year. There is evidence in this trial of a significant solar heat gain noted in earlier container trials. This would not occur if the container was sited in the building and, therefore, it reinforces the need for insulation in order to conserve the heat supplied by the heating system. The temperatures within the container are very satisfactory for phosphine use. However, it is recommended that further trials be carried out once containers are sited indoors to measure the heat loss. This would be expected to be greater given the absence of solar heat gain here after the heater was switched off.

Before the venting of the container commenced another set of pressure half-life tests was carried out on the container in order to obtain the degree of seal during the fumigation trial. In view of the attention given to sealing the container floor it was expected that leakage points would be few.

For 600-300 Pa the mean half-life was 56.3 seconds (range 52.9-58.5 seconds) and for 300-150 Pa the mean half-life was a mean of 106.1 seconds (range 102.2-109.8 seconds). Again this indicated a very satisfactory sealing standard.

#### **Date samples.**

There was no survival from the 46 dates infested with *E. ceratoniae* larvae treated for a 4-day exposure and there was no adverse effect on the flavour or appearance of the uninfested dates.

#### **Trial in container 1 dosed with aluminium phosphide tablets.**

The same dates were fumigated in this trial. The heating system was turned on at 1630 hours when the mean temperature in the container was 17.0 °C (range 16.5-18.2 °C) using the same heater settings.

The heating system was switched on at 16.30 hours at the same settings used in the previous trial. The initial temperature in the container was a mean of 17.0 °C (range 16.5-18.2 °C). The ambient temperature was 17.1 °C and the wind speed was 2.5 ms<sup>-1</sup> with gusts up to 5 ms<sup>-1</sup>. At 0930 hours the following morning the mean temperature was 24.4 °C (range 22.3-25.9 °C) and 40 minutes it had changed little with a mean of 23.8 °C (range 22.0-25.3 °C). The temperature was considered adequate to proceed with the trial and the heating system was switched off in order to prevent breakdown of phosphine on the heating elements.

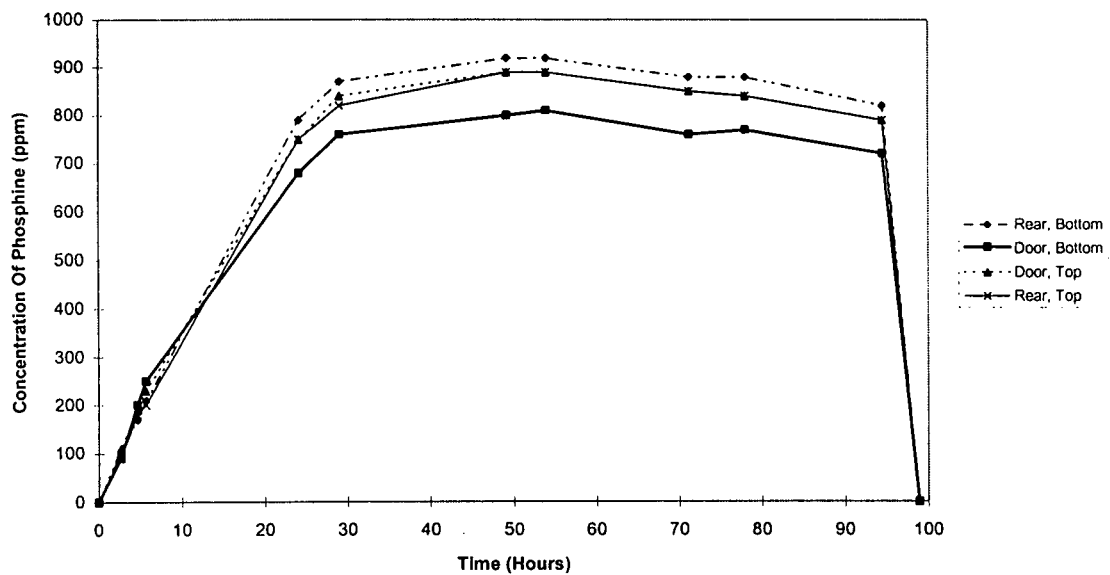
In order to calculate the number of aluminium phosphide formulation tablets to dose to a theoretical maximum concentration of 1.5 g m<sup>-3</sup> the volume was taken as 40 m<sup>3</sup> i.e. the volume to be allowed when stacking standard containers. In fact the project terms of reference (TOR) referred to the usable filling volume as 34 m<sup>3</sup> and the actual volume recorded on the loading plate was 33.1 m<sup>3</sup>. 60 tablets, each giving off 1 g of phosphine, of the aluminium phosphide formulation were dosed in the container. This would produce a theoretical maximum concentration of 1.81 gm<sup>-3</sup> (60/33.1) and not

the  $1.5 \text{ gm}^{-3}$  (60/40) as per the TOR. Table 5.9 and figure 5.6 give the concentrations at the same 4 gas sampling positions

Table 5.9. Phosphine concentrations in the container dosed with aluminium phosphide formulation.

Date/Time	Lapsed time (hours)	Corrected Phosphine Concentration (ppm)				Winds speed (m s-1)
		Position 5	Position 6	Position 7	Position 8	
10/04/00 10:00	0	0	0	0	0	
10/04/00 12:40	2.67	100	90	110	90	0.5-1.0
10/04/00 14:40	4.67	170	200	190	180	
10/04/00 15:40	5.67	210	250	230	200	
11/04/00 10:10	24.17	790	680	750	750	
11/04/00 15:03	29.05	870	760	840	820	
12/04/00 11:10	49.17	920	800	890	890	1.6-3.5
12/04/00 15:55	53.92	920	810	890	890	0.4-2.0
13/04/00 09:10	71.17	880	760	850	850	0.2-1.1
13/04/00 16:00	78.00	880	770	840	840	0.3-1.1
14/04/00 08:30	94.50	820	720	790	790	0.2-1.0
14/04/00 12:56	98.93	0	0	0	0	
<b>Mean</b>		<b>656</b>	<b>584</b>	<b>638</b>	<b>630</b>	
		<b>CT products <math>\text{g h m}^{-3}</math></b>				
		<b>101.3</b>	<b>89.3</b>	<b>97.9</b>	<b>97.1</b>	

Figure 5.6 Concentrations of phosphine in container 1 dosed with aluminium phosphide formulation.



The maximum concentrations were attained in about 48 hours in contrast to the rapid dosing seen by dosing with the phosphine-carbon dioxide formulation.

Table 5.10 gives the temperatures in the container during the trial.

Table 5.10. Temperatures within the container and ambient during the trial.

Date/Time	Temperature (°C)				
	Position 5	Position 6	Position 7	Position 8	Ambient
10/04/00 12:40	24.0	25.1	29.7	23.6	22.1
10/04/00 14:40	23.7	26.1	31.7	23.8	21.3
10/04/00 15:40	23.7	26.3	32	23.9	22.8
11/04/00 10:10	21.0	20.5	25.1	21.5	22.2
11/04/00 15:03	18.4	19.3	17	22.3	
12/04/00 11:10	17.0	16.8	21.7	17.4	20.2
12/04/00 15:55	18.5	20.6	28.4	19.7	
13/04/00 09:10	16.9	16.2	20.4	18.1	19.0
13/04/00 16:00	20.0	22.2	31.6	22.2	26.8
14/04/00 08:30	19.2	18.9		20.9	
14/04/00 10:40					26.0
14/04/00 14:05					30.9
<b>Mean</b>	<b>20.2</b>	<b>21.2</b>	<b>26.4</b>	<b>21.3</b>	<b>23.5</b>

A careful monitoring of the concentration at position 7 was undertaken to assess the efficiency of using the chamber fan with the valves on the chimney and door open. A pump was used from 180 minutes to increase the airing airflow. The concentrations are given in table 5.11.

Table 5.11. Concentrations in container 1 at position 7 during airing.

Lapsed Time (minutes)	Phosphine concentration (ppm)
0	790
12	750
15	700
16	680
20	530
38	490
52	410
55	400
77	300
105	220
127	160
155	100
168	90
172	110 *
219	50
240	50

\* Reading taken at the chimney.

Again there was evidence of solar heating though the highest temperature recorded at the top near the door was 31.7 °C though this may well have exceeded later in the day. The readings on the maximum-minimum thermometer adjacent to the bioassays were 15/26 °C. The phosphide formulation was fully decomposed at the end of the 4-day exposure and the residue was buried.

As in the previous phosphine trial the CT products were very good with an average of 96.4 g h m<sup>-3</sup>. This is slightly less than the average in the previous despite dosing 8 g of phosphine more. It would be expected that the slower dosing would result in less gas loss but the reverse was true and can be explained by a less effective seal. Pressure half-life tests were carried out before opening the door. The time for 660-330 Pa was a mean of 35.8 seconds (range 34.7-37.2 seconds). This was faster than when the container was sealed for the trial using phosphine in carbon dioxide though it was still very acceptable compared to the required standard of 500-250 Pa in not less than 10 seconds. The cause of this is unknown but the most likely explanations are a change in the door seal pressure and/or increased leakage in the heating system joints after heating and cooling.

Again the rate of airing was slow due to the exponential dilution of the phosphine concentration and a vacuum pump would be necessary to achieve rapid airing. The concentration was 50 ppm after 4 hours of dilution.

### **Date samples.**

There was no survival of *E. ceratoniae* larvae in the 50 infested dates treated and the uninfested dates showed no adverse effect on taste and condition from the treatment.

A soap bubble test on the heating ducting showed minor leaks which were repaired using silicone sealant.

### **Trial in container 1 with carbon dioxide.**

The container was aired of phosphine by having the door open for 1.5 hours after which no phosphine was detectable. The container was sealed and the temperatures recorded as a mean of 25.5 °C (range 22.9-31.1 °C). Three infested batches of dates were inserted near to position 6 together with a sample of dates for tasting and a maximum-minimum thermometer. The fan and heater was switched on at the 12 kW setting but after 40 minutes it was decided to lower the thermostat setting to 27.5 °C in view of the highest temperature recorded. The heaters and fan were used throughout the trial which lasted for a 4-day exposure shown necessary previously..

Carbon dioxide was dosed using a combination of cylinder types through the valve fitted to the door though the chimney valve remained closed in order to minimise loss of carbon dioxide. The dip tube cylinder type was used with a regulator setting of 60 or 80 psi together with the gas off-take type used in conjunction with the electrically heated vaporiser. The heater and fan were operated during the dosing to maintain the temperature and to mix the carbon dioxide in order to obtain concentration readings to assess the progress of the dosing. It was accepted that there would be loss of gas through leaks in the heating system when it was pressurised.

During the dosing the concentrations at rear bottom and rear top were measured and found to be 34.3 and 35.6 % respectively (mean 34.9 %) after 53 minutes of dosing when 28 kg had been dosed. 28 kg of carbon dioxide occupies approximately 14.25 m<sup>3</sup>. For an internal volume of 33.1 m<sup>3</sup>, assuming perfect displacement of air and no loss of carbon dioxide, the theoretical concentration would be 14.25/33.1 or 43.1 %. Therefore the loss of carbon dioxide during this phase of the dosing was  $(\{43.1 - 34.9\}/43.1) \times 100 = 19 \%$  of the theoretical. In total 63 kg were dosed in 2.3 hours.

Using the same calculation, 63 kg occupy approximately 32.1 m<sup>3</sup> and so the theoretical concentration would be 32.1/33.1 or 97 %. The heating thermostat was set back to 12 kW at 17.44 hours for the night when the container temperature was a mean of 26.6 °C (range 24.8-30.0 °C). The concentration after dosing stopped was very even at a mean of 65.2 % (range 64.4-65.7 %). The total loss of carbon dioxide during dosing was therefore  $(\{97.0 - 65.2\}/97.0) \times 100 = 32.8 \%$ .

For the economic assessment it would be more meaningful if the container was assumed to have a better sealed heating system without atmosphere circulation (see the stack dosing method) and dosed according to the volume. In order to obtain a concentration of 65.2 % the volume of carbon dioxide to be added would be  $0.652 \times 33.1 \text{ m}^3 = 21.6 \text{ m}^3$  or 42.4 kg. This would then be mixed by re-starting the heating and circulation system.

The following morning at 09.39 hours the benefit of running the heating system overnight was obvious. The mean temperature was high and uniform at a mean of 27.0 °C (range 26.0-27.8 °C) with the ambient temperature 20.3 °C and a wind speed of 0.2-0.9 ms<sup>-1</sup>.

For full efficacy, the concentration should not allow to fall below 40 % (45 % to give a suitable margin). Carbon dioxide is heavier than air and would readily escape from any leaks in the container, especially from the heating system when it was pressurised. After 16 hours the concentration fell to a mean of 50.3 % (range 50.0-50.6 %) from 65.2 %, a reduction of 14.9 % or 22.9% of the initial concentration i.e. 1.43 % of the initial concentration per hour. Dosing higher concentrations in the container in this condition would result in a greater amount lost per hour and so it was considered that dosing to about 65 % was an acceptable compromise target for economy which would give no risk of reducing to 40 % overnight. Further carbon dioxide was added on three occasions in 9, 17 and 13 kg increments whenever it was judged necessary in order to maintain adequate concentrations overnight, again with the chimney closed. Table 5.12 and figure 5.7 give the concentrations..

Table 5.12. carbon dioxide concentrations in container 1.

Date/Time	Time (hours)	Concentration (%)				Amount dosed (kg)	Wind Speed (ms <sup>-1</sup> )
		Position 5	Position 6	Position 7	Position 8		
14/04/00 15:30	0	0	0	0	0	63	
14/04/00 17:44	2.23	65.7	65.1	65.6	64.4		
15/04/00 09:39	18.15	50.0	50.3	50.6	50.4		0.2-0.9
15/04/00 12:33	21.05	48.6	47.8	47.8	48.0		
15/04/00 17:10	25.67	44.7	44.8	43.9	44.9	9	0.3-1.4
15/04/00 18:05	26.58	52.9	53.3	53.1	53.6		
16/04/00 09:31	42.02	44.0	43.7	43.5	43.6		0.3-1.9
16/04/00 15:17	47.78	41.2	40.7	40.7	41.0	17	0.8-3.5
16/04/00 16:44	49.23	56.4	56.4	56.6	57.9		
17/04/00 09:55	66.42	44.5	44.3	44.5	44.5	13	0.2-2.9
17/04/00 17:30	74.00	54.0	53.9	54.3	53.3		0.7-1.4
18/04/00 10:18	90.80	44.0	43.5	44.0	44.0		0.4-1.8
	<b>Average</b>	<b>49.6</b>	<b>49.4</b>	<b>49.5</b>	<b>49.6</b>		
	<b>Total:</b>					<b>102</b>	

Figure 5.7.

Fig. 3. Graph Showing Carbon Dioxide Concentrations in the Trial in Container 1.

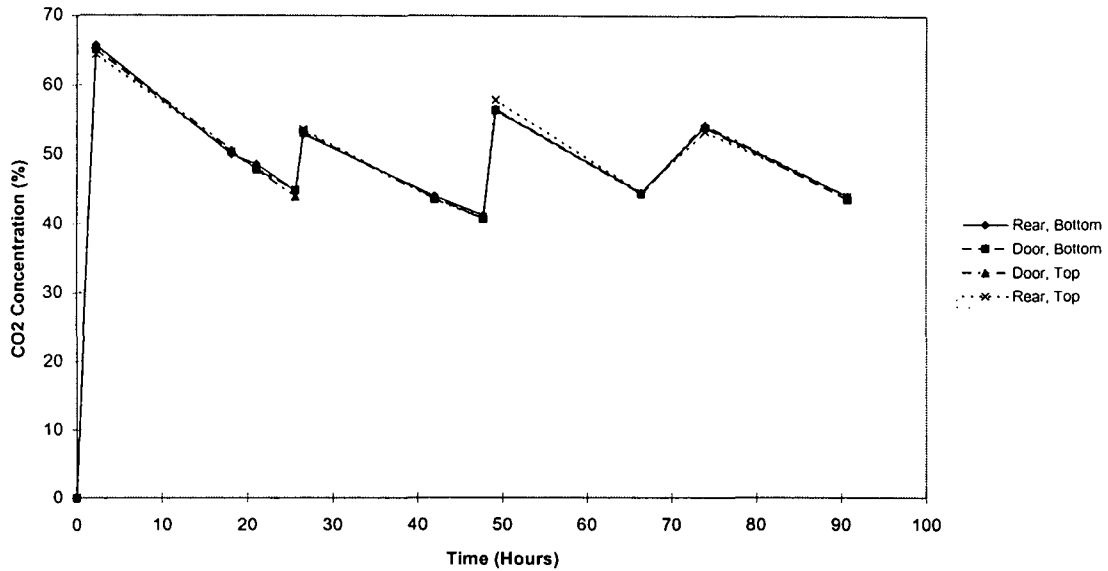


Table 5.13 gives the temperatures within the container during the trial.

Table 5.13.

Date/Time	Temperature (°C)				
	Position 5	Position 6	Position 7	Position 8	Ambient
14/04/00 17:44	25.5	26.3	30.0	24.8	
15/04/00 09:39	28.0	27.6	27.8	26.0	20.3
15/04/00 12:33	26.9	27.9	30.5	26.5	
15/04/00 17:10	26.7	29.5	30.8	27.9	22.0
16/04/00 09:31	30.5	30.9	32.2	29.3	23.0
16/04/00 15:17	29.7	31.9	35.3	30.0	34.2
16/04/00 16:44	29.5	31.8	34.1	30.1	
17/04/00 09:55	27.5	28.9	24.7	30.6	17.9
17/04/00 17:30	28.2	30.8	28.0	30.0	16.8
18/04/00 10:18	28.9	28.4	30.7	27.9	18.6
<b>Mean</b>	<b>28.0</b>	<b>29.3</b>	<b>30.4</b>	<b>28.3</b>	<b>21.8</b>
<b>Internal maximum - minimum</b>		<b>25-35</b>			

The trial went well in terms of the carbon dioxide concentrations achieved. The concentration was very uniform, as would be expected with the fan running continuously. The mean concentration over the trial across all positions was 49.5 %. With experience it was possible to judge the required dose increment though it would be prudent to be a little more generous in dosing since the concentration fell to an



average of 40.9 % (range 40.7-41.2 %). For regular use it would be advisable to not allow the concentration to fall below 45 % in order to give an adequate safety margin.

It is difficult to give a reliable estimate of the total amount of carbon dioxide required for the 4-day treatment in the container with a well sealed heating system and dosed without re-circulation by upward displacement of air. It would be certainly less than the total of 102 kg dosed in this trial. A reasonable estimate would be 50 kg which would give an initial concentration of 77 %. In this case it is likely that the concentration would not reduce to 45 % and so additional gas would not be required. 50 kg is the figure which will be used in the economic assessment but it should be emphasised that more trials will be necessary on a well sealed system in order to determine the true amount required. The container held 6.272 tonnes of dates.

The temperature data shows that there was solar heating of the container again. The maximum-minimum thermometer at the bottom front right read 25/35 °C. There had been a malfunction of the heating thermostat since on the 15 April at 12.33 hours it read 31 °C despite having been set at 27.5 °C.

The trial ended on the last working day of the project and so the container was not vented in a controlled manner but this was achieved by simply opening the doors.

#### **Date samples.**

There was no survival from the 46 dates infested with *E. ceratoniae* larvae. There was no adverse effect of the treatment on the taste and appearance of the uninfested date samples despite exceeding the 30 °C limit set by Medifruit by solar heating. No adverse comment was received from Medifruit on the condition of the loaded dates which must also have been overheated and had also experienced two phosphine treatments in the container. Part of the load had been fumigated in the vacuum chamber with methyl bromide prior to being loaded into the container.

#### **8. Trial with carbon dioxide in a small stack.**

It had been determined previously that polyethylene was permeable to carbon dioxide. The purpose of this trial was to study the saving in dosing a stack constructed with a PVC base sheet and to obtain further data on the effect of exposure period on *E. ceratoniae* larvae.

Only two pallets of dates containing crates partially full and weighing a total of 0.914 tonnes were available for the trial. Therefore a small stack was constructed on a PVC sheet in such a way that the stack was wrapped by the sheet which was sealed with PVC adhesive along the edges and the sealable nozzles placed one on the top and the other near the base. The stack consisted of single row of four pallets of date crates, two of empty crates with the two pallets with three rows of crates with dates in the opposite corners of the stack and the remaining space filled with empty crates to six crates high to maintain the shape of the stack. The stack was 4.4 m long, 1.22 m wide and 1.5 m high giving a volume of 8.05 m<sup>3</sup>. The usual sealable access was constructed

to remove infested dates and dates for quality assessment. Samples of each were provided to be exposed for 3, 5 and 7 days. A ambient maximum-minimum thermometer was placed on the stack. Gas sampling positions and thermocouples were located as follows:

- 6 - in dates in the corner half-way up the stack plus a thermocouple.
- 7 - in the free space at the top corner above 7.
- 8 - in the free space at the corner bottom near the bioassay and below position 6.
- 9 - in the free space at the stack centre.
- 10 - in the free space top in the opposite top corner to position 7 plus a thermocouple.

A vacuum half-life test 350-175 Pa was 9.61 seconds. The corners were taped with adhesive PVC tape in an attempt to improve the seal. A vacuum of 620 Pa could be attained. A half-life test 500-250 Pa gave an average of 8.25 seconds (8.42, 8.09). This was considered a good result in view of the high ratio of sealed edge to volume ratio.

The stack was dosed with carbon dioxide using both gas off-take and liquid off-take type cylinders. A total of 25 kg was dosed. This occupies a volume of 12.73 m<sup>3</sup> in a stack of volume 8.05 m<sup>3</sup>. Gas was dosed until it was detected at the top nozzle.

The stack proved to be very leaky and the pressure caused by addition of carbon dioxide probably opened up the sheet joints. Further additions of carbon dioxide were made:

Lapsed time (hours)	Carbon dioxide added (kg)
23	7
51	13
73	14
97	17
123	15
<b>Total</b>	<b>66</b>

The total carbon dioxide used in the stack was, therefore, an excessive 91 kg or 11.3 kg per m<sup>3</sup>. The trial failed to demonstrate the benefit of a PVC base sheet due to the gross leakage from the sheet joints.

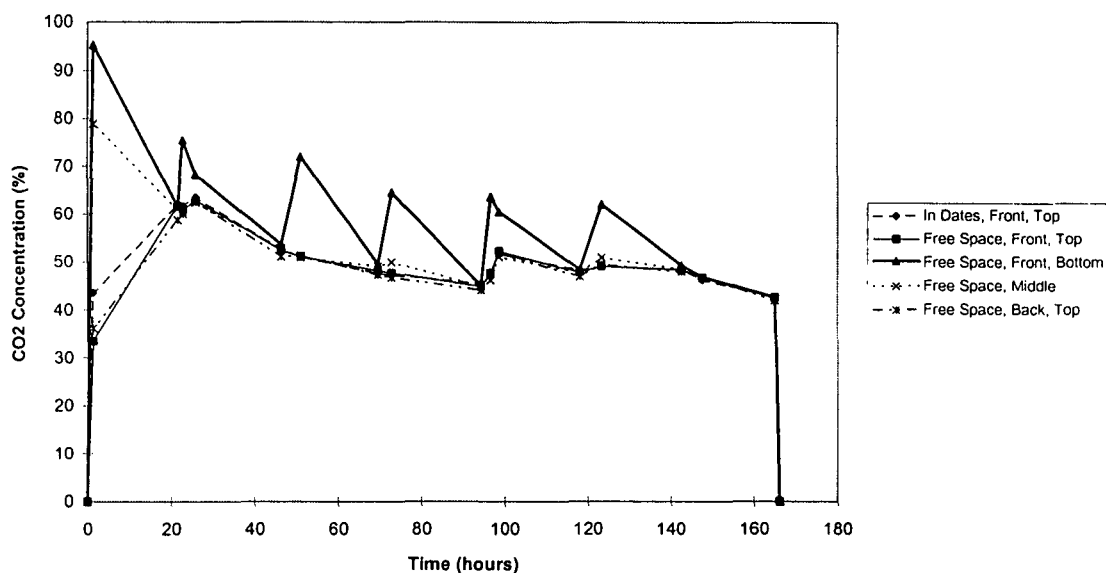
Table 5.14 gives the concentrations and temperatures within the stack. The ambient temperature within the store during the trial was 14-23 °C. The diurnal temperature changes would have caused a loss of gas by the 'bellows effect' resulting from gas expansion and contraction within the stack.

Table 5.14.

Real time (hours)	Lapsed time hours	Carbon dioxide %					Temperature (°C)	
		Position					Line 6	Line 10
		6	7	8	9	10		
06/04/00 11:50	0	0	0	0	0	0		
06/04/00 13:15	1.42	43.6	33.5	95.3	78.8	36.2	17	19.6
07/04/00 09:30	21.67	61.8	61.8	61.7	61.0	58.7	14.2	14.8
07/04/00 10:45	22.92	61.8	60.9	75.5	60.5	60.0		
07/04/00 13:45	25.92	63.6	62.9	68.3	62.4	62.6	16.0	17.8
08/04/00 10:15	46.42	52.5	52.5	53.7	52.9	51.1	14.0	15.3
08/04/00 14:55	51.08	51.1	51.2	72.0	51.0	51.4	15.7	17.2
09/04/00 09:30	69.67	47.6	48.2	49.6	49.1	47.3	15.2	15.2
09/04/00 12:45	72.92	47.3	47.7	64.6	50.0	46.7	15.3	15.6
10/04/00 10:15	94.42	45.5	44.9	45.0	44.9	44.2	14.8	16.3
10/04/00 12:30	96.67	47.5	47.7	63.7	46.2	47.5	16.0	19.3
10/04/00 14:30	98.67	51.8	52.3	60.6	51.1	52.0	17.4	20.4
11/04/00 10:00	118.17	48.4	47.9	48.5	47.9	47.0	16.7	19.5
11/04/00 15:11	123.35	49.2	49.2	62.2	51.0	49.6	18.4	16.9
12/04/00 10:33	142.72	48.5	48.5	49.4	48.6	48.0	14.9	16.1
12/04/00 15:30	147.67	46.3	46.8	47.0	46.9	46.9	17.0	19.3
13/04/00 08:50	165.00	42.7	42.7	42.7	42.8	42.0	15.1	16.0
13/04/00 10:00	166.17	0	0	0	0	0		
<b>Mean</b>		<b>50.6</b>	<b>49.9</b>	<b>60.0</b>	<b>52.8</b>	<b>49.5</b>	<b>15.8</b>	<b>17.3</b>

Figure 5.8 shows the concentrations within the stack graphically.

Figure 5.8.



The day time temperatures measured in the stack averaged a minimum of 15.8 °C at the bottom and after natural mixing of the gas had occurred the minimum concentration was 42 %.

#### Bioassay.

Exposure (days)	Mean concentration (%)	Number of infested dates	Number of moths emerged.
3	68.0	51	16
5	63.2	47	4
7	60.0	51	0

The mean concentrations for the three exposure periods were similar to those in another stack trial (see table 5.2) when the mean day time stack temperature was 23 °C. The same result was obtained with larvae surviving the 5-day exposure but not the 7-day exposure despite the temperature in the current trial being lower. This gives a good basis for a treatment schedule of a mean concentration of 65 % over 5 days provided that the temperature is >15 °C.

#### 9. Trial with carbon dioxide in a well sealed larger stack.

Previous trials indicate that a topping up of carbon dioxide was necessary for stacks with a PVC floor sheet and the previous sealing techniques. This is not only more expensive than a single dosing but it requires attention and labour.

A final attempt was made to produce a well-sealed stack. A PVC sheet was cut to the size of a 12 pallet stack with a 1 m excess all round. Nine pallets of empty crates were

placed on it and a row placed at one end containing three pallets containing three rows of crates filled with dates with empty crates above these to maintain the stack shape. They were covered with a PVC sheet cut to size with the dosing nozzle in the bottom centre of the width and the other nozzle in the far right corner at the top of the stack. The half loaded pallets contained 1.515 tonnes of dates (601, 421, 493 kg).

Gas sampling positions and thermocouples were inserted as follows:

- 6 - top of stack in the dates half-way up the stack right plus thermocouple.
- 7 - in free space at top of stack above 6.
- 8 - in free space at bottom of stack below 6.
- 9 - free space in stack centre.
- 10 - in free space at top in the opposite corner to 7 plus thermocouple.

The stack was sealed by a modified method of applying two, not a single, band of PVC adhesive to join the two sheets. The corners were well sealed and folded down and weighed down with empty pallets leaned against the stack. The sealing took two men one hour to seal a perimeter of 17.1 m and the four corners.

A vacuum half life test on the stack showed it to be extremely gas-tight due to the double bands of adhesive. Triplicate tests at 660-330 Pa gave a mean of 35.8 seconds (range 34.7-37.2 seconds). This is more than three times the required standard from the TOR. The volume of this 12 pallet stack was 29.07 m<sup>3</sup> (length 4.6 m, width 3.95 m, height 1.6 m).

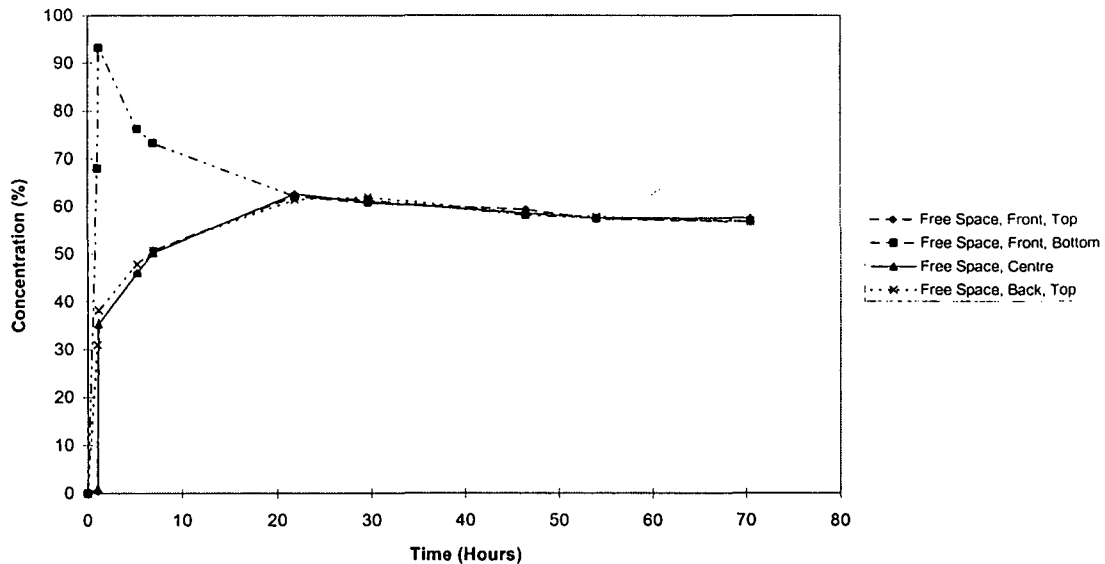
It was planned to dose the stack with 48 kg of carbon dioxide or approximately 24.44 m<sup>3</sup>. This would give an expected concentration of  $24.44/29.07 \times 100 = 84\%$  in order to try for a single application treatment. Unfortunately, an error was made and the 3-position valve on the dosing position was left so that gas was vented to atmosphere rather than entering the stack. Finally 60 kg of gas was dosed from cylinders. It is estimated from the average concentration of 62 % measured after 22 hours when the gas had mixed that the amount dosed was  $62 \times 29.07 / 100 = 18.02 \text{ m}^3$  or approximately 35 kg.

The stack concentrations and temperatures were measured. and are given in table 5.15. and graphically in figure 5.9. The sampling tube for position 7 soon became blocked inside the stack, probably from date debris and no further readings were possible.

Table 5.15. Trial in a large gas-tight stack with a PVC base sheet..

Date / Time	Lapsed Time (hours)	Carbon dioxide %					Temperature (°C)		
		6	7	8	9	10	6	10	Ambient Max-Min
15/04/00 11:15	0	0	0	0	0	0			
15/04/00 12:18	1.05	34	0.4	68.0	0.9	31.0			
15/04/00 12:25	1.17			93.2	35.3	38.2	22.9	17.5	
15/04/00 16:30	5.25		46.0	76.2	46.0	47.8	21.2	19.3	
15/04/00 18:12	6.95		50.7	73.3	50.2	50.7			
16/04/00 09:10	21.92		62.1	62	62.6	61.4	18.9	16.1	13-25
16/04/00 16:55	29.67		60.7	61.3	60.9	61.9	23.4	19	
17/04/00 09:45	46.50		59.3	58.1	58.5	58.5	17.8	16.7	16-26
17/04/00 17:17	54.03		57.3	57.4	57.5	57.8	17.8	17.1	
18/04/00 09:46	70.52		56.7	56.9	57.5	56.7	17.5	15.7	14-20
	<b>Mean</b>		<b>56.1</b>	<b>67.4</b>	<b>47.7</b>	<b>51.6</b>	<b>19.9</b>	<b>17.3</b>	

Figure 5.9



There was no time at the end of the project to extend the exposure period further and so no bioassay or test dates were treated. The mean concentration after 71.5 hours of 56.9 % showed a loss of only 5.1 % from the mean of 62 % at 22 hours. This indicates a percentage loss of  $5.1/62.0 \times 100 = 8.2$  % or about 4 % per day. At this rate the concentrations would have remained above 40 % after 10 days. It would be prudent to dose initially for a concentration of 80% rather than 62 % as a safety factor. This would require 23.3 m<sup>3</sup> of carbon dioxide or 45.7 kg. There is the potential for using stacks with a single dosing, even for the control of *E. kuehniella* eggs and hence eggs of *E. ceratoniae* provided that a similar level of gas tightness is achieved. These

insects as well as larvae of *E. ceratoniae* would have been controlled at the temperatures in the stack which were well above 15 °C.

#### 10. Trial on the fumigation of a stack with phosphine-carbon dioxide mixture.

A stack was constructed using a polyethylene base sheet and PVC cover sheet to accommodate a stack of 12 pallets of crates 6 crates high in 3 rows of 4 pallets. Again, due to the shortage of dates, only 6 of the pallets contained dates in 3 or 4 layers of crates and made up to 6 layers of crates with empty crates to maintain the stack shape. The six pallets with dates were positioned in an L-shape in a row of 4 along the length of the stack and a row of 3 along the width. The dosing inlet was in the centre of the left hand length. The usual sealable location for the insertion and removal of infested dates and dates for quality testing was positioned at the floor in the centre of the front width in front of the pallets with dates. Samples of these dates were inserted for withdrawal after 3, 5 and 7 days. The dimensions of the stack were 5.2 x 3.47 x 1.55 m giving a volume of 28 m<sup>3</sup> and it contained a total of 2.96 tonnes of dates.

Gas sampling locations and thermocouples were:

- 1- top corner free space opposite the corner with dates.
- 2- centre of stack free space.
- 3 - corner bottom free space near dates.
- 4 - top free space above 3 plus thermocouple.
- 5 - top in the dates near 4 plus thermocouple.

The stack was well sealed and a vacuum half-life test gave a mean of 8.91 seconds (range 7.72-9.68 seconds) for 150-75 Pa.

According to the TOR the stack was to be dosed to obtain a theoretical maximum concentration of 1.5 gm<sup>-3</sup> of phosphine. This would require 28 x 1.5 or 42 g of phosphine equivalent to 42 x 50 g = 2.1 kg of the 2 % w/w mixture. Dosing was rapid though it was difficult to accurately weigh the cylinder with the bathroom scales available. On re-checking the weight later it was found that only 1.1 kg of mixture or 22 g of phosphine had been dosed but this was fortuitous since it provided an opportunity to assess the efficacy of a lower concentration which would not be expected to reduce excessively, given the degree of seal. The amount theoretical concentration of phosphine was estimated as  $1100/(50 \times 28) = 0.786 \text{ gm}^{-3}$  or 550 ppm and it is probable that from an examination of the early gas concentration readings (a mean of about 300 ppm) it was even less.

The trial took place at the same time as the trials of stacks dosed with carbon dioxide and the indoor ambient temperatures were the same.

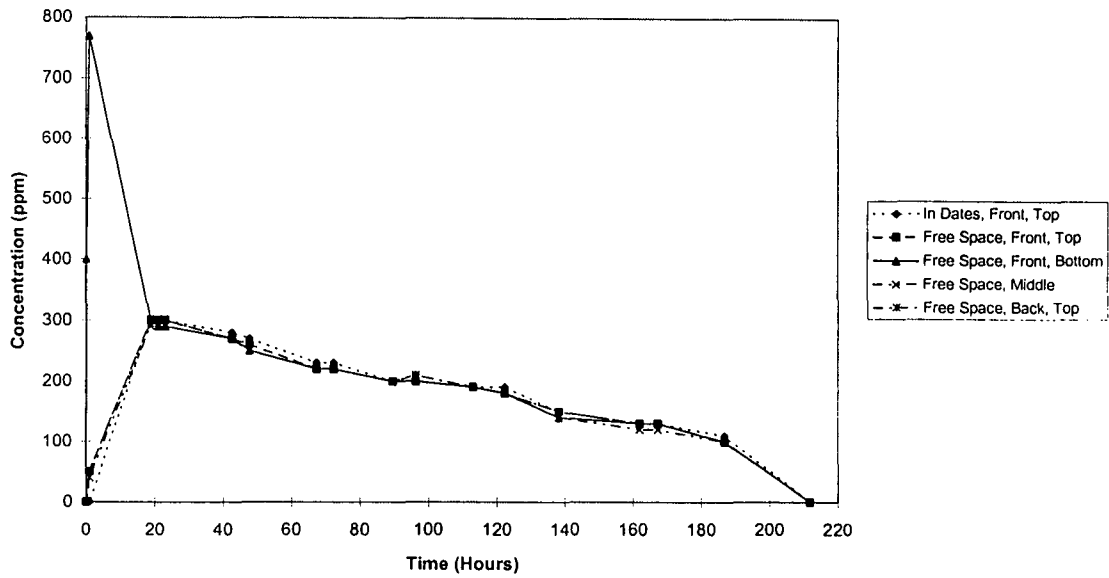
Table 5.16 gives the concentrations and the CTP products resulting.

Date/Time	Lapsed time (hours)	Phosphine concentration (ppm)				
		Position				
		1	2	3	4	5
09/04/00 15:20	0	0	0	0	0	0.0
09/04/00 15:34	0.23	0	0	400	0	0
09/04/00 16:15	0.92	0	50	770	40	50
10/04/00 10:15	18.92	300	300	300	290	300
10/04/00 12:30	21.17	300	300	290	290	290
10/04/00 14:30	23.17	300	300	290	290	300
11/04/00 10:00	42.67	280	270	270	270	270
11/04/00 15:11	47.85	270	260	250	250	250
12/04/00 10:45	67.42	230	220	220	220	220
12/04/00 15:45	72.42	230	220	220	220	200
13/04/00 09:00	89.67	200	200	200	200	200
13/04/00 15:40	96.33	210	200	200	200	210
14/04/00 08:29	113.15	190	190	190	190	190
14/04/00 17:45	122.42	190	180	180	180	180
15/04/00 09:40	138.33	150	150	140	140	150
16/04/00 09:23	162.05	130	130	130	120	130
16/04/00 14:40	167.33	130	130	130	120	130
17/04/00 10:17	186.95	110	100	100	100	100
18/04/00 11:10	211.83	0	0	0	0	0
	<b>3-day CTP</b>	<b>24.1</b>	<b>24.2</b>	<b>33.6</b>	<b>23.6</b>	<b>24.0</b>
	<b>5-day CTP</b>	<b>38.5</b>	<b>38.2</b>	<b>47.6</b>	<b>37.6</b>	<b>38.2</b>
	<b>7-day CTP</b>	<b>48.0</b>	<b>47.6</b>	<b>56.7</b>	<b>46.4</b>	<b>47.6</b>

The concentrations are shown graphically in figure 5.10.



Figure 5.10.



The temperatures within the stack are given in table 5.17.

Table 5.17.

Date/Time	Lapsed time (hours)	Temperature (°C) Position		
		4	5	Max/Min (°C)
09/04/00 15:20	0			
09/04/00 15:34	0.23			
09/04/00 16:15	0.92	16.6	16.0	
10/04/00 10:15	18.92	16.9	15.3	14-21
10/04/00 12:30	21.17	19.6	15.6	
10/04/00 14:30	23.17	20.2	16.0	
11/04/00 10:00	42.67	19.5	16.4	14-23
11/04/00 15:11	47.85	16.8	17.2	
12/04/00 10:45	67.42	17.1	15.5	13-23
12/04/00 15:45	72.42	19.1	16.0	
13/04/00 09:00	89.67	17.4	15.7	
13/04/00 15:40	96.33	21.8	17.0	
14/04/00 08:29	113.15	17.8	19.2	17-25
14/04/00 17:45	122.42	22.9	19.9	
15/04/00 09:40	138.33	20.1	18.5	15-28
16/04/00 09:23	162.05	20.2	17.7	
16/04/00 14:40	167.33	23.6	18.1	
17/04/00 10:17	186.95	17.2	19.5	
18/04/00 11:10	211.83			14-20
<b>Mean</b>		<b>18.1</b>	<b>17.1</b>	

The concentrations at the end of nearly 9 days were at least 100 ppm at all positions, indicating a successful fumigation at the temperatures in the stack which were an average of 17.6 °C in the day time and which would have averaged at least 15.0 °C throughout the fumigation.

### **Bioassay.**

There was no survival of *E. ceratoniae* larvae in the minimum of 50 dates exposed at any exposure period. Bell (1976) found that larvae of Pyralid moths were killed at very low CTPs over just 2 days of 3.7 g h m<sup>-3</sup> at 25 °C and 13.8 g h m<sup>-3</sup> at 15 °C. The CTPs obtained in the trial are more than sufficient and the minimum exposure period for the control of *E. ceratoniae* larvae could be reduced to 2 days at this dosage rate though it would be prudent to retain the 3-day exposure period when dosing with aluminium phosphide formulation.

The eggs of moths of related species are much more tolerant in short exposure periods. Bell (1976) showed that at 15 °C CTP of more than 248 g h m<sup>-3</sup> is required for control of some young eggs in a 7-day exposure but that this can be reduced to 10.3 g h m<sup>-3</sup> over 5 days at 20 °C and 3.9 g h m<sup>-3</sup> at 25 °C. The previous trial using aluminium phosphide formulation gave control of eggs of *E. kuehniella* in a 5-day exposure to a CTP of 71 g h m<sup>-3</sup> at a mean temperature of more than 20 °C.

### **11. Conclusions.**

Phosphine and carbon dioxide are suitable replacements for methyl bromide if used carefully. There are economic implications in terms of logistics and the capital investment required since both treatments take considerably longer than does vacuum fumigation with methyl bromide.

The project was concluded completely successfully in regard to developing methods and recommendations for the use of phosphine in heated freight containers and sheeted stack and carbon dioxide in heated containers, sheeted stacks and vacuum chambers. Attention to the valving on the chambers is required to prevent loss of vacuum and dilution of carbon dioxide when used under vacuum. The current vacuum chambers could be readily used at atmospheric pressure both for phosphine and carbon dioxide with attention to the door seals.

All or some of these fumigation systems are available elsewhere in Tunisia and there are brickwork chamber also available for atmospheric pressure treatments.

Technically the project suffered from three main problems:

1. Insufficient dates being made available for trials. Normally enclosures contained some dates with the remaining volume being empty crates. This resulted in a tendency to overestimate the amount of phosphine and carbon dioxide required though attempts have been made in the economic assessment to overcome this problem.

2. The production of a fully effective heating system in the time scale before the first trials took place in Tunisia was difficult. The heating system heated dates effectively though there were severe operational difficulties in working outside due to solar heating. The containers will be used indoors by Medifruit and CSL agree with Medifruit that a gas rather than an electrical system will be more economical. Another problem with the CSL system was the construction of the system's ducting which was riveted and, therefore, insufficiently gas-tight, especially when pressurised by the circulation fan running. There was some delay in obtaining suitable containers and in making them sufficiently gas-tight through the use of more materials and labour than was first envisaged. A commercial system will require gas-tight welded ducting with arrangements for servicing the heaters and some form of heat insulation (spray-on polystyrene is preferred) for energy economy.

3. The project Terms of Reference in regard to phosphine doses were restrictive. The recommendation to use a concentration of  $1.5 \text{ gm}^{-3}$  was excessive. There is an excessive safety margin on concentration which could be reduced with properly designed laboratory toxicity study against larvae and eggs of *E. ceratoniae* at a range of temperatures. This is recommended. However, the economic assessment indicates that even at a concentration of  $1 \text{ gm}^{-3}$  the use of the aluminium phosphide formulation is seven times cheaper and the phosphine-carbon dioxide formulation is 50 % cheaper than methyl bromide on chemical alone.

The cost of using carbon dioxide was more difficult to estimate due to the severe impact of gas-tightness on the amounts required. However, our best estimates are that it is about 1.5 to three times the cost of methyl bromide and thus a very economic alternative, especially for 'bio dates'.

Unfortunately, the project failed to demonstrate that the use of trapping moths in the Medifruit facility could be used as part of an ICM strategy. Specifically, it was expected that it could demonstrate the benefits of correctly identifying fumigated and non-fumigated dates in the cold stores and of allowing dates to stand only for short periods before processing. There was much which could have been done in this area but, unfortunately, Medifruit management were not committed to the necessary effort for the trapping exercises. However, specific recommendations have been made on the assumption that minimising the opportunity for adult moths to emerge and lay eggs on the finished product will reduce infestation to the consumer.

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