THE USE OF CONTROLLED ATMOSPHERES FOR THE STORAGE OF GRAIN

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This paper deals with aspects of the use of controlled atmospheres for the storage of cereal grains with a moisture content not exceeding 13%. Controlled atmosphere storage of high moisture content grain has been dealt with by other authors [1].

The term "inert atmospheres" has also come to be used for atmospheres of the types we shall be discussing, namely those consisting of nitrogen or mixtures of air and carbon dioxide. We prefer not to use this terminology as carbon dioxide and air mixtures are not physiologically inert.

Two distinct approaches can be applied to the use of such atmospheres. The first is to use them as a method of slow-acting fumigation that does not leave any toxic residues. The second is to hold the grain in the atmosphere for the whole of its storage life thus gaining the advantage of a residue-free grain protectant.

Historically, airtight storage, which is a form of controlled atmosphere storage has probably been used, wittingly or unwittingly, in many parts of the world for a very long time. Thus, Hyde and Daubney [2] state that "Underground storage has been practised from time immemorial in certain countries, particularly those of the Middle East and Central Mediterranean regions". Underground storage does not necessarily produce atmospheres that will control insect pests but in those instances where the structures were built with sufficient care to prevent the ingress of soil moisture it is likely that true hermetic conditions were achieved. Thus in recent trials with the fossae that were constructed in Malta three hundred years ago, and whose capacities ranged from 50 to as much as 500 tons, the oxygen content of the intergranular air at the bottom dropped to almost zero within three weeks of filling with grain.

In most recent times large-scale installations have been employed in South America and a system of "Waller" bins, originally built in Cyprus has been extended to East Africa. All these are commercial scale projects, and reports suggest that in general, and in the context of the trading situation in which they exist, they are successful in reducing or eliminating losses due to insect pests. They may, however, leave much to be desired in terms of grain handling convenience.

Smaller-scale trials have been conducted using bitumenised construction [3] and more recently plastic and butyl rubber containers. These latter have not always proved very successful owing to rapid weathering, mechanical damage or the activity of rodents.

The scientific background of underground or hermetic storage has been investigated since about 1918 when the pioneering studies of Dendy [4], produced a great deal of information concerning changes in the composition of the intergranular air among grain stored in airtight conditions and of the effects of such changes on a range of insect and mite pests. Bailey [5], showed that the depletion of oxygen due to the respiration of the insect and of the grain played a dominant role in the control of pests when normal air was the starting point, and that, further, it was necessary for the oxygen to be depleted to about 2% before the insects died. Oxley and Wickenden [6] demonstrated the effects of small leaks on insect pest populations, arguing that in practice, commercial size storage structures were not likely to be airtight in the absolute sense.

Subsequently, a number of workers have examined aspects of the mortality of insects due to changes in concentration of atmospheric gases. Notable among these are Knipling, Harein and Press, Lindgren, Vincent, AliNiazee Spears and Zettler, Person and Sorenson, Jay, Marzke and Pearman and others.

Much of the recent work has been carried out at a time when the newer organophosphorus grain protectants were achieving an excellent result in many countries. During this time, at least so far as Australia was concerned, the grain industries showed little interest in controlled atmosphere storage as they believed their insect control problems had been largely solved for the foreseeable future.

We are now experiencing a notable change in attitude. Major problems arising from the development of pesticide resistance and the lack of suitable alternatives and continuing problems with pesticide residues have greatly increased interest in more satisfactory solutions to insect pest problems.

SUMMARY OF MORTALITY DATA DUE TO CONTROLLED ATMOSPHERES: With the apparent great potential of the controlled atmosphere method of grain storage, it is remarkable that the exact limits of dosage and mortality for stored product pests has been so inadequately defined. Work has concentrated largely on the adult stage which appears to be the most susceptible and has largely ignored pupae and eggs which, from the limited data available, appear to be the most tolerant stages [7]. Data are also deficient in observations at temperatures lower than about 23°C.

The potential controlled atmospheres which could be utilized for grain storage are those with reduced oxygen tension or those with an increased CO_2 content. These appear to exert their action differently and high CO_2 mixtures, typically around 60% are reported as being more rapid in their action than low oxygen atmospheres. In order to present the current information for mortality/dosage relationships, as obtained by laboratory experiments using constant gas atmospheres we shall review the published material.

Table I presents data for adult *Tribolium castaneum*, the most widely studied insect. The exposure time given is the minimum used by the particular experimenter to produce a high mortality,

TABLE 1. The mortality of adult *Tribolium castaneum* exposed to different atmospheric gases

Gas Mixture %				Mortality %	Exposure Days	Temp. °C	r.h. %	Test Number x Replicates	Age of Adults In Days	Commodity	Ref.
O_2	N_2	CO_2	Air								
3	97			100	14	32	72	10 x 10	0-7	wheat	[8]
	100			100	5	22.2	-	30 x 3	-	-	[9]
1	99			94	14	15.6	64	10 x 3	11-17	cornmeal	[10]
0.3	99.7			100	2	26.7	59	10 x 3	0-7	cornmeal	[11]
2.5	97.5			100	7	26.7	61	10 x 3	0-7	cornmeal	[11]
		36	64	99	4	15.6	64	10 x 3	11-17	cornmeal	[10]
		48	52	92	7	15.6	64	10 x 3	11-17	cornmeal	[10]
		46	54	84	7	26.7	54	771 x 1	-	peanuts	[12]
		46	54	93	7	26.7	61	10 x 3	0-7	cornmeal	[11]
		97	3	100	2	26.7	59	10 x 3	0-7	cornmeal	[11]

and the results have been based on low numbers of test insects, generally less than 100.

However, it can be seen that the parameters influencing mortality are insect species, level of oxygen, level of carbon dioxide, temperature, and relative humidity.

High mortality was obtained at oxygen concentrations below 3% in nitrogen and with carbon dioxide concentrations exceeding 35% in air. Table II shows similar data for adults of the 3 species of *Sitophilus* and miscellaneous stored product Coleoptera. Because different authors have used different experimental procedures, direct comparison of their results is difficult. It is clear, however, that when mixtures of nitrogen and oxygen are used, the oxygen concentration must not exceed about 4.5% for even the most susceptible species.

Lindgren and Vincent [7] made a detailed study of *Sitophilus granarius* and *S. oryzae*. This study is the only one available with detailed investigation of all stages, probit analysis of mortality values, comparison of gases and mixtures, and of temperature effects. They showed that using oxygen-free gases there was little difference in effect of helium, nitrogen or carbon dioxide either at different temperatures or on different stages. They also demonstrated the change in LT_{95} with change in carbon dioxide concentration for *S. granarius* and *S. oryzae*. *S. oryzae* shows increasing mortality to concentrations of carbon dioxide up to 90%, while atmospheres containing above 60% carbon dioxide appear to be only marginally more effective for *S. granarius*.

There is a pronounced temperature effect on the length of time required to achieve a given level of mortality when using low oxygen concentrations (Fig. 1). No temperature study has been published on immature stages nor on high carbon dioxide/air mixtures though taking all the available observations into account there seems to be a similar effect. The effect is an important one since in a grain bulk that is not thermally homogeneous there will be parts in which pests may survive an otherwise adequate treatment and the economics of the method may be strongly dependent on the exposure time for treatment.

Immature stages have received little attention. The data are summarised in Table III. It is important to note that the LT_{95} for *S. oryzae* pupae, the most tolerant stage of the more tolerant species, is 18.4 days at 26.7°C [7]. If pupae have a similar temperature relationship as adults they would require an exposure of three months at 18°C. We have noted survival after 30 days at less than 1% oxygen in nitrogen at about 18°C for *S. granarius* in field trials. Unpublished laboratory experiments have given survival from mixed age cultures of *S. granarius* after 10 weeks at 1.3% oxygen at 18.3°C.

THE PRODUCTION OF CONTROLLED ATMOSPHERES: Classically, the production of controlled atmospheres has been carried out simply by making the grain container airtight. The respiration of any insect pests that may be present together with the respiration of grain embryos,

TABLE II. The mortality of adults of three species of *Sitophilus* and other important grain pests exposed to different atmospheric gases.

Species	Gas Mixtures %		Mortality %	Exposure Days	Temp. °C	r.h. %	Test Number x Replicates	Age of Adults In Days	Commodity	Ref.
	O_2	N_2								
Sitophilus granarius	2.5	97.5	100	17	25	72	10 x 10	0-7	wheat	[5]
S. oryzae	4	96	100	14	29	72	10 x 4	0-7	wheat	[13]
S. oryzae	1	100	94	2	32.2	60	30 x 2	-	sorghum	[14]
S. zeamais	4.2	95.8	100	14	29	72	10 x 4	0-7	wheat	[15]
S. zeamais		100	93	1.5	32.2	60	30 x 2	-	sorghum	[14]
S. zeamais	0.5	99.5	100	2	32.2	60	30 x 2	-	sorghum	[14]
Cryptolectes pusillus	1	99	100	2	32.2	60	30 x 2	-	sorghum	[14]
Cryptolestes ferrugineus	4.5	99.5	100	14	32	72	10 x 10	0-7	wheat	[8]
Tribolium confusum	0.3	99.7	99	2	26.7	59	10 x 3	0-7	cornmeal	[11]
Rhyzopertha dominica	3	97	100	14	32	72	10 x 10	0-7	wheat	[8]
Oryzaephilus surinamensis	4.5	95.5	100	14	32	72	10 x 10	0-7	wheat	[8]

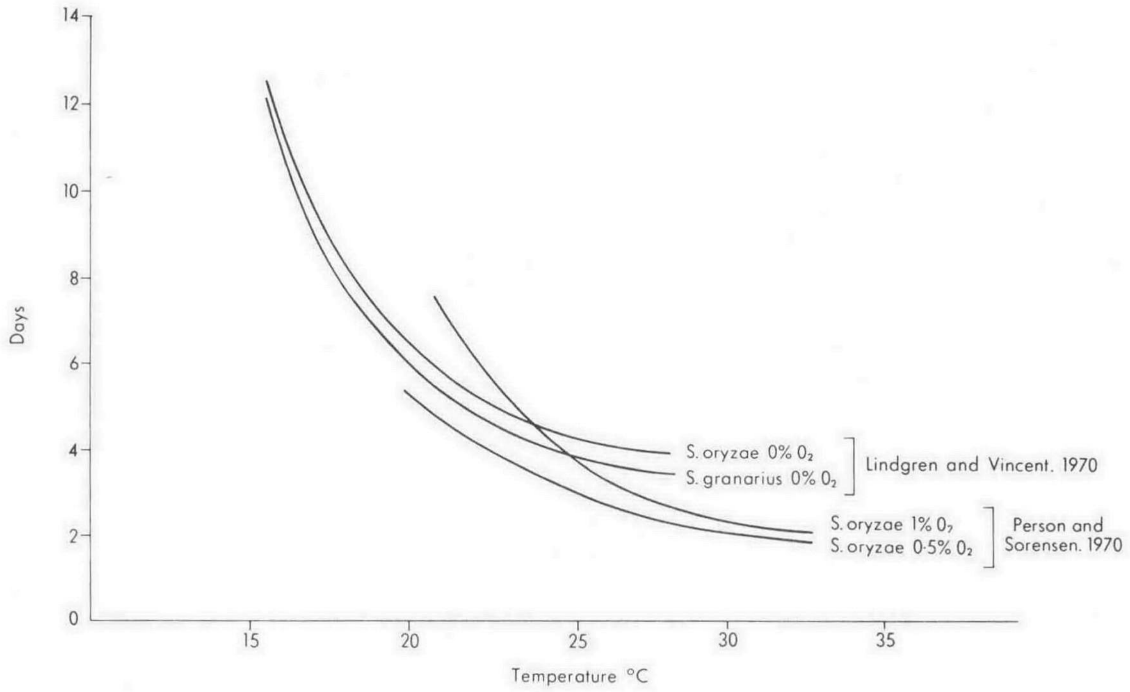

FIGURE 1. The relationship between temperature and the time required to give 95% mortality of adults of *Sitophilus granarius* and *S. oryzae*.

use up the oxygen and produce carbon dioxide, thus bringing about conditions that are lethal to the insect. Typically, the respiratory quotent of the system is a little less than unity, hence, even if all the oxygen present is used up the carbon dioxide concentration does not exceed about 18%.

The above oxygen consuming process has sometimes been hastened by the burning of candles or straw in the space above the grain. However, this is of limited benefit because candles are extinguished when the oxygen concentration falls to about 18% leaving the greater part of the oxygen to be consumed by respiratory processes. The simple sealing of stores, with or without the use of candles or straw, may suffice for the use of grain that is to be used by the grower, or sold at a village level. For grain that is to enter trade channels, particularly that for export markets, the number of grains damaged by the insects before they are killed, and the number of dead insect bodies that result may not be acceptable. This would certainly be the case with many of Australia's overseas wheat markets. It is therefore necessary to examine the possibility of producing lethal atmospheres by other means. Such means include the purging of silos by nitrogen or carbon dioxide gas from tankers or the use of oxygen free atmospheres produced by burners or catalytic generators. The trials we have carried out in Australia and which are described later, have all used tanker gases as these are immediately available.

Tanker gases have disadvantages. Carbon dioxide is often

TABLE III. The mortality of immature stages of important grain pests exposed to different atmospheric gases.

Species	O_2	N_2	CO_2	Air	Mortality %	Exposure Days	Temp. °C	r.h. %	Test Number x Replicates	Stage	Commodity	Ref.
Tribolium castaneum	2.5	97.5			97	7	26.7	61	10 x 3	2nd instar larvae	cornmeal	[10]
Tribolium castaneum			59	59	87	14	26.7	61	10 x 3	2nd instar larvae	cornmeal	[10]
Tribolium castaneum			46	54	83	7	26.7	71	582 x 1	-	peanuts	[12]
Sitophilus granarius	-	100			LT95	8.7	26.7	60-70	50 x 5	eggs	wheat	[7]
Sitophilus granarius		100			LT95	4.7	26.7	60-70	50 x 5	larvae	wheat	[7]
Sitophilus granarius		100			LT95	8.3	26.7	60-70	50 x 5	pupae	wheat	[7]
Sitophilus oryzae		100			LT95	9.3	26.7	60-70	50 x 5	eggs	wheat	[7]
Sitophilus oryzae		100			LT95	8.9	26.7	60-70	50 x 5	larvae	wheat	[7]
Sitophilus oryzae		100			LT95	18.4	26.7	60-70	50 x 5	pupae	wheat	[7]

expensive, nitrogen less so, though its present costs may be unrealistically low, as it is partly an unwanted by-product from oxygen manufacture. By its mode of action the use of controlled atmospheres is a slow effect process hence its use is sensible only in the country storage areas where speed of action is unimportant. Yet carbon dioxide and nitrogen are produced by industry situated in the capital cities. This necessitates long hauls of some hundreds of kilometers to the site of use. It seems therefore that we might do better to generate our atmospheres at the storage site rather than carry them in tankers.

On-site generation may be possible in a number of ways. The fresh fruit industry employ propane burners to produce low oxygen atmospheres., this can be done using a flame generator or by catalytic combustion. Other possible ways, not commercially available, might use agricultural waste fermented by aerobic bacteria or fungi in a digestion chamber through which the gaseous contents of the silo would be circulated or the use of enzyme systems, the use of a paramagnetic oxygen pump or a gaseous diffusion pump to separate the components of the air or the use of a regenerative electrolytic reduction cell based, for example, on chromium salts. This last works well for a time in the laboratory and might be very inexpensive to run as the oxygen among one ton of wheat would require the expenditure of only about 2 kilowatt hours of electrical energy to remove it. In practice, an electrolytic cell runs at a very high efficiency rating. In Australian country areas this system would cost about 6¢ per ton.

In this connection, it is interesting to note that we have reason to believe that cobalt salt solutions may be in current use in China for the controlled atmosphere storage of bagged rice under plastic sheeting.

FIELD TESTS WITH INERT ATMOSPHERES: We have carried out field trials in parallel with laboratory studies. Nitrogen has been used as the gas because it is conveniently available and is best covered by supporting literature. The goal of the field trials has been to maintain an atmosphere of less than 1.5% oxygen indefinitely at reasonable economic cost. This long term storage approach contrasts with published trials with such atmospheres, which have all used an approach similar to a fumigation. The toxic atmospheres in these was maintained only for a brief period with relatively high make-up rates of gas. Our approach has been to minimise the maintenance rates by sealing the grain silo as well as possible. Effective sealing eliminates the possibility of the existence of local areas of high oxygen and substantially reduces the gas required to be introduced on a continuing basis for maintenance of the atmosphere within acceptable oxygen limits. Our general aim at sealing has been to achieve the standard whereby two inches of water gauge pressure drops to one inch in not less than 15 minutes.

Five full scale controlled atmosphere field trials have been carried out, three in steel bins and two in concrete bins, Table IV. The nitrogen was generated from liquid by passing it through a heat exchanger which raised the temperature of the gas stream close to ambient temperature. The gas was then introduced into the base of the bin either through specially designed ducting, existing aeration facilities, modified outloading valves, or directly into the bin through ports in the bin wall. The gas introduction was divided into two phases, the purge and the maintenance. In the first phase the gas was introduced quickly e.g., at 2.5 m^3/min. In the second phase the flow was reduced until maintenance of the lower oxygen concentration was just achieved.

(a) Purging - It is important to be able to purge the grain mass efficiently as, under well sealed conditions, this may be the main gas usage. Contrary to other workers we have found that purging can be carried out with the grain *in situ* and this is advantageous since mixing of the purge gas with air is restricted. Figure 2 shows the rise of a nitrogen front up a vertical bin. The rate at line 3, the central sampling line is higher than at the edge, lines 1 and 5, but at all points a definite front was observed. In the head space the oxygen decay was exponential as would be expected from free mixing. Since the latter method is a less efficient method of oxygen removal than direct displacement by a front it follows that to minimise purge gas usage the head space should be minimised. In four of the five trials a usage of about 1 m^3 nitrogen per tonne gave an acceptable atmosphere. This value is close to theoretical for frontal displacement in the intergranular spaces and exponential mixing in the head space.

(b) Maintenance - After a suitable atmosphere has been created it is necessary to add further gas to maintain it against leakage. The main factors giving rise to gas loss in a leaky bin are wind effects and the "chimney" or "stack" effect. The latter effect is caused by the difference in gas density inside and outside of a grain bulk because of differing temperatures and relative humidity. A 20°C difference in this temperature for a 30 metre high bin will cause a standing pressure difference of about 10 Pascals. Standing values of this order have been observed for air. Under conditions of extreme density differences, such as are encountered with the use of carbon dioxide the standing pressure difference may rise to over 250 Pascals. These forces may cause substantial gas loss through leaks especially if at the base. Early trials in 1968 with carbon dioxide were unsuccessful because of this. The effects may be overcome by more efficient sealing.

Under well-sealed conditions long period effects such as diurnal barometric changes and solar heating become significant. Substantial pressures of over 200 Pascals have been observed in a steel bin due to heating of the roof and head space. These effects could be reduced by insulation or shelter from the sun.

The two trials carried out in the relatively leaky concrete bin required high ingress rates. For the second of these the maintenance rate required was about 700 m^3/day, added at the bin base. This rate could possibly have been reduced if the gas had been added at the apex. The rate is too high to contemplate

TABLE IV. Summary of Field Scale Nitrogen Swamping Trials.

Trial No.	Bin Construction[1]	Floor	Dimensions		Contents	Pressure Tests		Head Space to volume ratio	Gas required to give 1% O_2 m^3/tonne	Air ingress rate %/day
			Hgt. at apex m	Dia. m	Commodity	Flow giving 250 Pa. (l/min)	Pressure drop 500-250 Pa. (secs)			
I	welded steel	Concrete	25.2	23.1	7035T wheat	-	370	0.08	1.04	2.6
II	welded steel	concrete	25.2	23.1	5220T wheat	-	<690	0.26	-	4.0
III	concrete	concrete	30.5	10.9	2040T wheat	-	96	0.15	1.18	12.4
IV	concrete	concrete	30.5	10.9	2040T wheat	1280	102	0.15	1.09	-
V	welded steel	concrete	22.4	13.9	1809T barley	250	~1000	0.20	1.82	3.9*

* Estimated from maintenance rate.

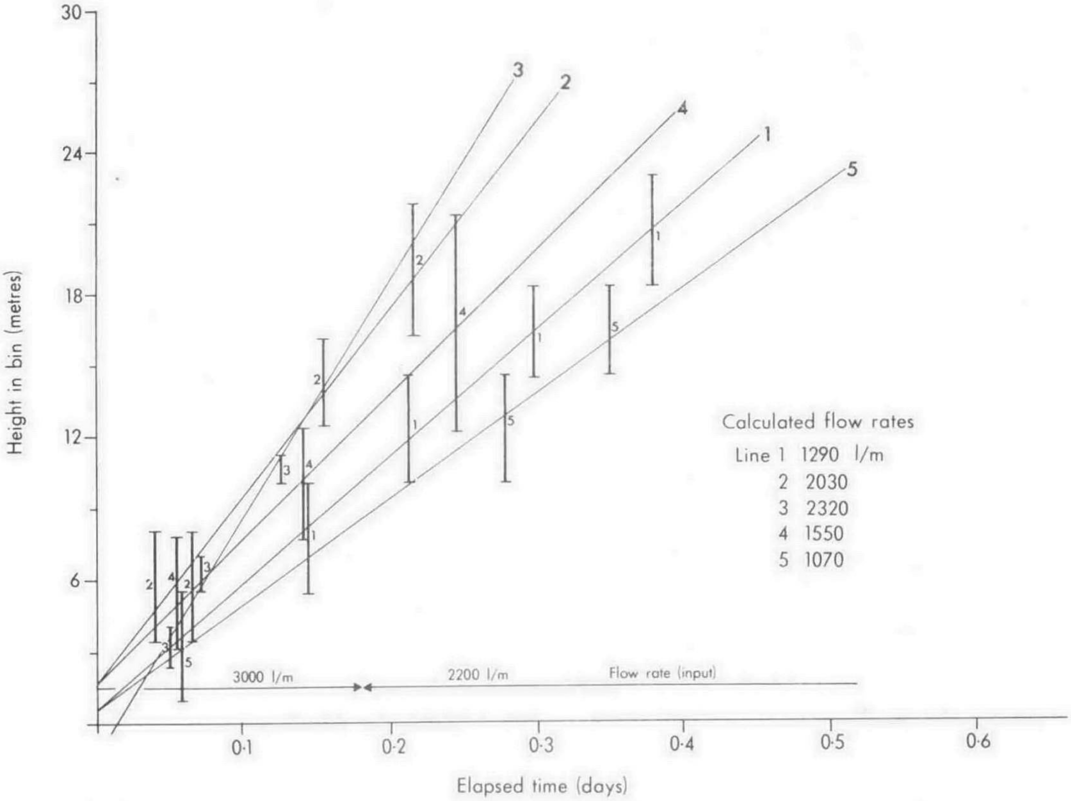

FIGURE 2. The rise of a nitrogen front in a vertical silo bin.

for routine use. In the third trial of the steel bins a nitrogen maintenance rate of 70 m^3/day gave an acceptable atmosphere of less than 1.1% oxygen throughout the 26 day period of the trial.

LIMITATIONS TO THE USE OF CONTROLLED ATMOSPHERES: It is clear that there are limitations to the use of controlled atmospheres for the storage of grain. Some are inherent in the system, others are due to the current situation.

At present the largest limitation in Australia is the lack of storage facilities that can be made sufficiently gastight for the purpose. An enormous amount of capital is invested in a great variety of existing storage structures. These include only a small number of welded steel silos which can be modified relatively easily, Table V. The remaining silos are vertical concrete cells, with or without caps, and a bewildering variety of horizontal storage types, most of which would be very difficult to seal sufficiently. Development work might enable some of the existing structures to be effectively sealed and we are exerting continual pressure on the designers of storage structures to incorporate sealability in all new building programmes.

Among the limitations that are inherent in the use of controlled atmospheres is the relatively long time for an existing insect population to be killed, especially at low grain temperatures. This, in Australia, limits the use of the method to country storage areas, where time is not an important factor and where,

TABLE V. Capacities and types of wheat and barley storage facilities in Australia.

Capacity	
Country storages	14.2 million tonnes
Terminal storages	4.2 million tonnes

Types	% of total
Horizontal	66
Vertical concrete	24
Vertical steel	9

in fact most of our grain is stored. The shipping terminal silos at the seaboard are designed for rapid transit of grain and the use of controlled atmospheres would not be appropriate in such situations.

It is interesting to consider whether any form of resistance by the insects to controlled atmospheres could develop. The authors feel that an increase in the time required to kill could be selected over many generations or that some species might enter into a form of diapause. In either case effective control would still be achieved as reproduction would be extremely slow or would cease. The possibility of an increase in the time factor is a good reason to aim the development programme at continuous storage under controlled atmospheres, rather than looking upon their use as a form of fumigation. It is possible also that there might evolve strains able to tolerate lower oxygen levels. This would not appear to be serious as we are, in any event, aiming at atmospheres containing not more than about 1% oxygen. The possibility of the insects altering their biochemistry to one based entirely on anaerobic respiration is not considered likely.

Changes will also be required in some attitudes of the industry. At present visual inspection of grain is relied upon to a very great extent, but this would have to be dispensed with as it would defeat the purpose of the system if stores were opened for inspection purposes. Furthermore, the atmosphere within the stores would be lethal to man as well as to insects. Either automatic sampling techniques would need to be devised, or more likely, remote reading instruments, monitoring such factors as temperature and oxygen concentration would come to be relied upon.

When one surveys the field of grain storage it is difficult to see a role in the long term for chemical pesticides other than in local crisis situations. Resistance and residue problems appear inevitable so long as chemical pesticides are depended upon. Research and development work leading to the large-scale use of a few non-chemical means of insect control offer the promise of eventual permanent solutions to storage problems. We think it most important that stored product entomologists should be looking for grain storage methods that provide inherent in-built insect control operating on principles that will be as effective a hundred years from now as they are today. Among these means we would place controlled atmosphere storage very high on the list.

REFERENCES:

[1] Hyde, M. B., Oxley, T. A., Experiments on the air-tight storage of damp grain, Ann. appl. Biol. 48 (1960) 687.
[2] Hyde, M. B., Doubney, C. G., A study of grain storage in fossae in Malta, Trop. Science. 11 3 (1960) 115.
[3] Winterbottom, D. C., Weevil in wheat and storage of grain in bags, (Govt. Printer; Adelaide) (1922).
[4] Dendy, A., Report on the effect of air-tight storage upon grain insects. Part I, Rep. Grain Pest Comm. Roy. Soc. 1 (1918).
[5] Bailey, S. W., Air-tight storage of grain; Its effects on insect pests. I. *Calandra granaria* L., Aust. J. Agric. Res. 6 (1955) 33.
[6] Oxley, T. A., Wickenden, G., The effect of restricted air supply on some insects which infest grain, Ann. appl. Biol. 51 (1963) 313.
[7] Lindgren, D. L., Vincent, L. E., Effect of atmospheric gases alone or in combination on the mortality of granary and rice weevils, J. econ. Ent. 63 (1970) 1926.
[8] Bailey, S. W., Air-tight storage of grain; Its effects on insect pests. IV. *Rhyzopertha dominica* (F.) and some other *Coleoptera* that infest stored grain. J. stored Prod. Res. 1 (1965) 25.
[9] Knipling, G. D., Sullivan, W. N., Fulton, R. A., The survival of several species of insects in a nitrogen atmosphere. J. econ. Ent. 54 (1961) 1054.
[10] Harein, P. K., Press, A. F., Mortality of stored-peanut insects exposed to mixtures of atmospheric gases at various temperatures, J. stored Prod. Res. 4 (1968) 77.
[11] Jay, E. G., Pearman, G. C., Susceptibility of two species of *Tribolium* to alternations of atmospheric gas concentrations. J. stored Prod. Res. 7 (1971) 181.
[12] Pearman, G. C., Jay, E. G., The effect of relative humidity on the toxicity of carbon dioxide to *Tribolium castaneum* in peanuts, J. Georgia Ent. Soc. 5 (1970) 61.
[13] Bailey, S. W., Air-tight storage of grain; Its effects on insects pests. II. *Calandra oryzae* (small strain), Aust. J. Agric. Res. 7 (1956) 7.
[14] Person, N. K., Sorensen, J. W., Use of gaseous nitrogen for controlling stored product insects in cereal grains, Cereal chem. 47 (1970) 679.
[15] Bailey, S. W., Air-tight storage of grain; Its effects on insect pests. III. *Calandra oryzae* (large strain), Aust. J. Agric. Res. 8 (1957) 595.