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GAS PROCESSES, CA AND FUMIGATION, FOR QUARANTINE AND BIOSECURITY

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ABSTRACT

Many countries rely on fumigation to eliminate insect pests of quarantine/biosecurity significance from durable goods in trade (import and exports). These goods include grains, wood and wooden materials and derived products. Biosecurity measures, giving an appropriate level of protection, are recognised as legitimate constraints to trade under the WTO SPS Agreement. Fumigation is also used to control pests that damage commodity quality in durable foodstuffs in trade, such as export grains. Some countries and importers may specify fumigation as a requirement for importation.

There is an urgent need to develop and deploy new gas processes (CAF, CA and fumigation) to replace the main fumigants, phosphine and methyl bromide, currently in use. Methyl bromide, though currently still permitted for Quarantine and Preshipment (QPS) uses, is a recognized ozone-depleting chemical. There is a stated policy to replace this fumigant where possible. Phosphine is under threat from development of resistance and the long exposure times required for full effectiveness are logistically inconvenient and costly. Potential alternatives for several major QPS uses include sulphuryl fluoride, cyanogen ('EDN'), carbonyl sulphide, ethyl formate and CAs (this Conference). Development of these alternatives is inhibited by many considerations including small market size, stringent requirements on effectiveness, lack of familiarity with their properties and application.

In postharvest treatment of durables, there is a particular need for locating and proving alternatives to methyl bromide for export grains and pulses against *Trogoderma granarium* Everts and for timber and solid wood packing materials against many pests but including longicorn beetles and pinewood nematodes. Methyl bromide treatments against *T. granarium* is an entrenched use. Its basis is not as firm as would be required of new treatments. Several treatments are under continued consideration for alternatives to methyl bromide (and heat) for ISPM 15.

Use of fumigants on exports requires a working procedure that renders the atmosphere within the treated enclosure safe to entry by personnel, including customs and quarantine officers. There have been several incidents with workers affected by residual fumigant in freight containers and surveys have detected excessive levels in a surprising number of containers in export trade. Several customs and quarantine authorities have adopted ventilation procedures and gas scrubbers that remove gas to safe levels before entry of fumigated containers.

Key words: biosecurity, quarantine, fumigation, methyl bromide alternatives, *Trogoderma*, ISPM 15, scrubbers, recapture, safety procedures.

INTRODUCTION

Many countries rely on fumigation to eliminate insect pests of quarantine/biosecurity significance from durable goods in trade (import and exports). These goods include grains, wood and wooden materials and derived products. The insect pests of concern may be directly infesting the durable goods or may be carried by other materials in particular consignments that are directly infestible. Some pests may be able to complete their life cycles on the goods (e.g., *Trogoderma granarium* Everts on cereal grains and *Cryptotermes brevis* (Walker) on some wooden materials) while others, such as many of the forest pests of biosecurity concern, cannot reproduce and multiply on the goods.

Application of biosecurity measures, such a fumigation of goods against pests of quarantine concern, are inconvenient, costly and are an interuption to free trade. They are subject to numerous constraints, not only to ensure they are used effectively and safely, but also to ensure they are not used excessively.

Biosecurity measures form an internationally recognised and permitted technical barrier to trade under World Trade Organisation (WTO) Agreements on the Application of Sanitary and Phytosanitary Measures (SPS) and Technical Barriers to Trade (TBT) agreements (WTO, 1995). These agreements, particularly the SPS agreement, impose important constraints on how and when quarantine measures can be applied to international trade. International biosecurity measures relating to plants and plant products, including durable commodities, are regulated in detail under the IPPC, with International Standard Phytosanitary Measures (ISPM). Two particular items, important to discussion below, follow as a result of these agreements. Individual countries must develop lists of pests of biosecurity concern and control measures applied against these pest must be set so they achieve an appropriate level of management of risk against establishment of the pest in the importing area.

The importance of fumigation, particularly with methyl bromide, for QPS (Quarantine and Preshipment) purposes is underlined by the exemption of methyl bromide from phaseout under agreed control measures under the Montreal Protocol. This continues, despite the high ozone depleting potential of methyl bromide and continuing discussion on its appropriateness. It is said that the specific exemption from phaseout for methyl bromide for QPS purposes was agreed, in 1992 (UNEP, 1992) and was because at that time it was argued that there were no technically available alternatives to methyl bromide where it was used for QPS purposes. This exemption is unique under the Protocol.

The exemption for QPS purposes under the control measures of the Montreal Protocol does come with some limitations. The International Plant Protection Convention (IPPC) has a stated policy (CPM, 2008) for member states on quarantine measures to reduce and avoid use of methyl bromide. Decisions VI/11 and VII/5 of the Montreal Protocol urge Parties to avoid using methyl bromide and to reduce emissions of methyl bromide where technically and economically feasible.

This paper discusses the potential alternative gas processes for quarantine and particularly to methyl bromide for QPS purposes, describes in detail the use of methyl bromide and alternatives against a key quarantine pest of durables (khapra beetle, *T. granarium*) and against forest pests potentially conveyed in wooden packaging material and the like as managed by ISPM 15, and finally looks at managing one of the problems that arise in use of toxic fumigants for QPS purposes. This is presence of residual gases in treated freight containers in international trade.

There is an urgent need to develop and deploy new or renewed gas processes (CAF, CA and fumigation) to replace the main fumigants, phosphine and methyl bromide, currently in use for QPS purposes on durables.

Methyl bromide, though currently still permitted for QPS uses, is a recognized ozone-depleting chemical and international policy requires its replacement if technically and economically feasible. Additionally, though it has a reputation for effectiveness against insect pests, it is not always very effective. If it were a new chemical fumigant and subject to the same rigorous assessment as these new fumigants, it may well not be found acceptable.

Phosphine is under threat from development of resistance and the long exposure times required for full effectiveness are logistically inconvenient and costly. Its very widespread use against pests of durable commodities such as grains is not only its strength but also a weakness. Introduction of highly phosphine-resistant strains of pests into areas at present having only easily manageable resistant strains would represent a major biosecurity breach.

The list of potential candidate gas treatments for QPS purposes is now quite long, as a result of programs to develop new gas processes for pest control. This contrasts with the situation a decade or so ago when fumigants were being lost to use at an alarming rate. This is not a criticism of the reasons for their loss, but an observation that the recent interest in development of new fumigants and revival of disused ones has been productive.

Development of a new process for a quarantine process from concept to approval for use is a long and complex process. Aside from requiring proof of efficacy to a high and reliable level, it also may require registration by regulatory authorities including national pesticide registration authorities and may often involve protracted negotiation between importing countries requiring appropriate biosecurity and exporters wishing to establish or continue a particular trade. In many situations, there is insufficient profit in the particular trade to support the work necessary to establish a new process.

Table 1 gives a listing of potential fumigants for durable commodities likely to be able to produce treatments on their own, or possibly in combination with helpers such as CO_2, heat or other fumigants. Some of these fumigants are still transiting the pesticide registration process, an uncertain, thorough and expensive process. The wide registration enjoyed by methyl bromide and phosphine seems unlikely to be achieved by the newcomers to the process.

It is interesting to compare the list in Table 1 with the fumigants in Table 2 described in Bond (1984).

Many of the papers at this conference describe developments in application of new and renewed fumigants, including some that relate to potential biosecurity treatments.

FUMIGATION AGAINST THE KHAPRA BEETLE, *Trogoderma granarium*

METHYL BROMIDE

The special biology of *T. granarium* poses particular problems with regard to quarantine treatments for this pest. As a larva, the pest may enter a very long lived diapause. In this form the pest seeks out crevices and other harbourage and can be very difficult to find by inspection or access by many normal control measures. Cast skins (exuviae) and trapping may give an indication of its presence, but in general the habits of the pest are such that fumigation treatments are carried out because of the risk of presence of the pest, rather than because of detection.

Table 1. Fumigants available in some countries or at an advanced but incomplete stage of registration

Fumigant	Comments
Carbon disulphide	Very flammable, out of patent
Carbonyl sulphide	Longer exposures than methyl bromide
Cyanogen ('EDN')	Water soluble, flammable
Ethyl formate	Very rapid action, poor penetration
Ethylene oxide	Carcinogenic, flammable, requires chambers
Hydrogen cyanide	Poor penetration particularly in wet commodities, out of patent
Methyl bromide	Wide registration, ozone depletor, permitted for QPS
Methyl iodide	Registrant withdrawing support from some applications
Methyl isothiocyanate	Poor penetration, may require other fumigant in combination
Ozone	Action needs further definition
Phosphine	Requires long exposure periods
Propylene oxide	Flammable, requires chambers
Sulphuryl fluoride	Poorly effective against insect eggs, leaves fluoride residues

Worldwide the fumigant of choice for quarantine treatment against *T. granarium* is methyl bromide (MB). This is despite its known unusually high level of tolerance to the fumigant, particularly when in diapause. Practical problems with fumigating some oily, high risk commodities, e.g., expeller cake, with methyl bromide further compounds the difficulties. With the very high dosages required for complete kill, there is a risk that residue limits for bromide ion may be exceeded in some treated commodities.

Table 2.'Bond's List.' Fumigants listed in Bond (1984) with threats, real or alleged, to continued use in 1984. From Banks (1994)

Fumigant	Threat
Acrylonitrile	Suspect carcinogen, residues
Carbon disulphide	Lack of interest
Carbon tetrachloride	Ozone depletor, residues
Chloropicrin	almost forgotten
Dichlorvos	Residues, alleged carcinogen
Ethylene dibromide	Environmental contaminastion, fertility effects, alleged carcinogen
Ethylene dichloride	Not very effective, alleged carcinogen
Ethylene oxide	Suspect carcinogen, residues
Ethyl formate	Almost forgotten
Hydrogen cyanide	Lapsed Codex Alimentarius registration
Methallyl chloride	No food registration
Methyl bromide	Ozone depletor, alleged carcinogen
Methyl formate	Almost forgotton
Phosphine	Resistance
Sulphuryl fluoride	No food registration
Trichlorethylene	Not very effective, residues

Historically, dosage recommendation for control of khapra beetle have been based on 'double the normal dosage' for typical stored product pest control (e.g., Bond, 1984 ; p.238-239). The latter are often aimed at *ct* = 200 g h m^{-3} at 20°C, implying *ct* = 400 g h m^{-3} at 20°C for *T. granarium*. This is close to the value of 480 g h m^{-3} for 100% kill at 20°C given by Bell et al. (1985), but less than the Russian quarantine dosage implied by Mordkovitch and Sokolov (1992) of 600 g h m^{-3}. Bogs (1976) give a dosage of 600 g h m^{-3} for >15°C, which may be the origin of this recommendation.

Modern 'khapra' dosages to meet quarantine requirements are typically very high. Australian quarantine requirements are for 80 g m^{-3} for 48 h at >21ºC (Dept. of Agriculture, Fisheries and Food, 2009), corresponding to a *ct*-product (CTP) of about 1700 g h m^{-3} on basis of exponential decay of fumigant concentration and end point retention of 20% of applied dosage. The USDA Treatment Manual (T302-c-1, USDA, 2012) specifies a dosage of 96 g m^{-3} with a retention of 30 g m-3 at 12 h at 15-21ºC, corresponding to a CTP of about 530 g h m^{-3}. It is of interest to see what level of mortality this actually corresponds to on the basis of published studies. There are no recent published studies as a check on current susceptibilities, so this analysis relies on studies from 20 years ago or more.

During the apparently successful khapra beetle eradication campaign in the 1950's in USA, the dosage recommendations finally adopted correspond to a minimum CTP of about 1200 g h m $^{-3}$ at unspecified temperatures (initial dosage 80 g m^{-3}, 32 g m^{-3} remaining after 24 h) (calculated from Armitage (1958)).

Bell et al. (1985) provide the most modern laboratory assessment of the effect of methyl bromide on *T. granarium* at low temperatures. Exposures used by Bell et al. (1985) are not stated but appear to have been 24-48 h with a few hundred larvae used per exposure and '100% kill' estimated by extrapolation to an undefined level of kill (>99%).

El-Lakwah (1977a) tested the effect of rearing of *T. granarium* at 25 and 28°C and various low temperature conditions on susceptibility of larvae to methyl bromide. Overall, his data shows that there is a range of susceptibility which can be induced by particular rearing temperatures and subsequent handling. Larvae allowed to enter diapause by holding at 25°C appear the most tolerant of the forms he investigated. From the point of view of quarantine control, the dosage should presumably be targeted at the most tolerant form that has a significant risk of occurrence at point of treatment.

El-Lakwah's (1977a) observations at 15°C correspond closely with those of Bell et al. (1985) (*ct*-products for the most tolerant material for 99% kill was 396 and 450 g h m^{-3} respectively) giving confidence that the two data sets can be treated as a single series. El-Lakwah's data also shows that material at least partly in diapause, i.e., held at 25°C or below for at least 8 days, showed increased tolerance to MB compared with material reared at 28°C and cooled quickly (over less than a few days) (e.g., reared at 28°C and fumigated at 12°C required 263 g h m^{-3}, or reared at 28°C, held at 25°C for one month and then fumigated at 12°C requires 420 g h m^{-3} for 99% kill.).

Figure 1 gives data points from various studies on *T. granarium* and methyl bromide, including those requiring highest dosage for kill under particular conditions. The envelope enclosing points of least susceptibility gives a estimate of *ct*-product for control of these forms. Test material at around 10°C appears anomalously susceptible, but larvae not fully in diapause may have been tested. At 21ºC the trend line shows a CTP of about 400 g h m^{-3} is required for 99% mortality. This supports modern quarantine dosage requirements where a much higher kill is required and CTPs of 1700 or more are set for 21ºC.

At this time no field resistance of *T. granarium* to methyl bromide has been recorded. However, slightly increased levels of tolerance to MB can be selected for in the laboratory with levels of 2x resistance achieved after many selections (Mordkovich and Sokolov, 1992). While this resistance level is low, it would jeopardise control with standard, already high dosages used currently with MB for quarantine purposes.

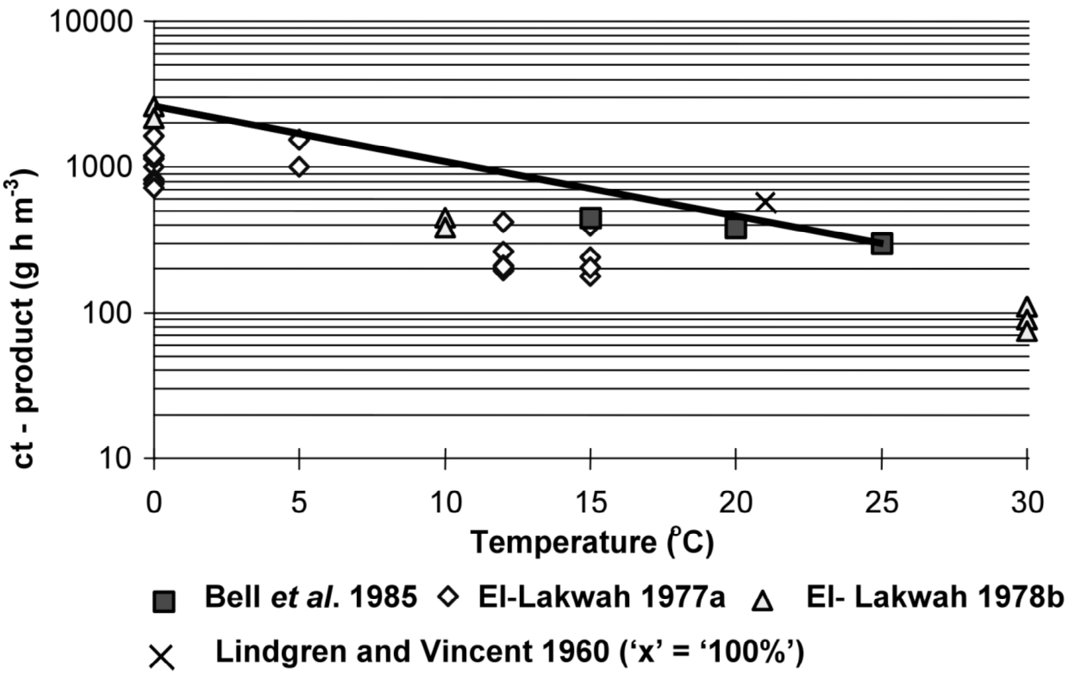

Fig. 1- *Ct*-products of methyl bromide (logarithmic scale) giving estimated 99% kill of *T. granarium* larvae, including those in diapause, for different temperatures. Line indicates envelope of maximum dosage required to give 99% kill of diapause larvae.

PHOSPHINE

Phosphine fumigation is widely used in countries where khapra beetle is common for its control. It is not listed as a quarantine measure against *T. granarium*. Reasons for this appear to be historical - with suitable precautions to prevent leakage and exposure times of 12 or more days, depending on conditions, data available for phosphine action on *T. granarium*, up until the early 1990s, supported consideration of its use as an alternative to MB for this pest. However, as noted below, resistance development may now have rendered this former option inappropriate.

Susceptible strains of *T. granarium*, even as a larva in diapause, is quite sensitive to phosphine. Typical long exposure dosage rates for control of *Sitophilus* species (e.g., 1.5 g m^3 for 7 days) are sufficient to control normally-susceptible *T. granarium* at >20°C, and appear to be so too for some resistant *T. granarium*.

The best studies on action of phosphine against *T. granarium* are those of Bell et al. (1983, 1985) and Hole et al. (1976). There are numerous other studies (e.g., Lindgren et al., 1958; El-Lakwah et al., 1989; Punj and Girish, 1969; Dhaliwal and Lal, 1973), but as these are carried out under conditions where diapause is absent or not adequately proven, they are, at best, indicative only of the relative susceptibility of *T. granarium* to phosphine.

Bogs (1975) testing (apparently) diapause larvae of *T. granarium* found LT_{99} at 0, 5, 10 and 15°C of 16, 9, 8 and 5 days, respectively at 0.7 g m^{-3} PH_3. El-Lakwah (1978a) using larvae reared at 28° (not in diapause) found LT_{99} at 29°C at 80 ppm (0.1 g m^{-3}) after only 34 h (86 h at 20 ppm). From apparently the same test stock (El-Lakwah, 1978b) the LT_{99} at 25°C at 50 ppm was 40 h, 12°C and 150 ppm was 140 h and at 0°C and 50 ppm was 565 h. He provides a variety of other data on this test sample involving effects of rising and falling concentration and other (higher) fixed dosages.

Bell et al. (1983) give a 6 day minimum exposure to give "100%" kill of diapause larvae at 20° using a CTP of 203 g h m^{-3} (dosage applied 1.5 g m^{-3}). They reinforce the conclusion of Hole et al. (1976) that extended exposure even at 30°C was required, with 0.4 g m^{-3} for 2 days giving only 99% control of freshly laid eggs.

The situation with phosphine against *T. granarium* has now changed. Various studies from the Indian subcontinent (Sharma and Kalra, 1998; Alam et al*.,* 1999; Sarfraz et al*.,* 2000; Ahmedani et al*.,* 2007) are showing a high level of resistance to phosphine even in active, non diapause larvae. Sarfraz et al. (2007) studied 3 strains of *T. granarium* that required 14 days exposure at 800 ppm (c. 1 g m^{-3}) phosphine at 34°C/65% r.h. With the expectation that the pests will be more tolerant still at lower temperatures or in diapause, this level of resistance would require excessive exposures to achieve quarantine levels of security.

OTHER FUMIGANTS AND CA

Hydrogen cyanide, chloropicrin and carbon disulphide show some promise as rapid treatments against *T. granarium* including diapause larvae (e.g., Lindgren et al., 1955; Lindgren and Vincent, 1960), but have not been developed to modern quarantine standards. They all will suffer from poor penetration of commodities under some conditions.

Low oxygen CAs show some promise for elimination of *T. granarium* but carbon dioxide-based atmospheres (< 70% CO_2) are less effective against *T. granarium* than most other stored product pests, requiring much prolonged exposure for control of diapause larvae. Annis (1987) concluded that 16 days exposure at 80% CO_2 (20-30°C) was required to eliminate *T. granarium* (data of Spratt et al. (1985), Verma and Wadhi (1978) and Le Torc'h (1983)).

Low-oxygen atmospheres however appear to be quite effective against *T. granarium,* including eggs and diapause larvae (Verma and Wadhi, 1978; Le Torc'h, 1983), requiring the same exposures as other tolerant stored product insect pests. Annis (1987) suggests 0.1% oxygen at 20-29°C for more than 20 days.

CAs have not been tested to quarantine standards for required levels of security.

DISINFESTATION OF WOODEN PACKAGING MATERIAL (ISPM 15)

Disinfestation of wood and wood packaging material remains the largest user of methyl bromide globally, despite widespread use of heat in place of methyl bromide for ISPM 15 treatment of wood packaging material in international trade. Work continues on the search for alternative fumigants to methyl bromide for ISPM 15. To date, no alternatives have been approved to a level that allows inclusion in ISPM 15 revisions.

Table 3 lists the fumigants that have been considered recently as alternatives to methyl bromide for ISPM 15.

Until recently there have been major impediments to approving alternatives to methyl bromide for ISPM 15, with methyl bromide itself not subject to the same level of scrutiny and requirements. There are signs that this is now changing. TEAP (2012) summarized the changes within the technical committees of the IPPC as:

Table 3. Fumigants under condsideration as alternatives to methyl bromide for ISPM 15 treatment of wood packaging material in international trade

Sulphuryl fluoride
Methyl isothiocyanate / sulphuryl fluoride (Ecotwin)
Hydrogen cyanide
Methyl iodide

Source: CPM. 2011.

"The Standards Committee [of the IPPC] made several recommendations in 2011 that may enhance the prospects for additional ISPM-15 treatments being developed and accepted. Firstly, the Committee recommended that the treatment must be shown to be effective against *Bursaphelenchus xylophilus* (pinewood nematode, PWN) and *Anoplophora glabripennis* (Asian longhorned beetle, ALB). Secondly, the Committee recommended that the current list of pests should be narrowed further to individual species if possible and should also focus on organisms to be eliminated at the point of treatment, i.e., the issue of infestation after treatment should not be considered. Thirdly, any new treatment was recommended to be at least as efficacious as heat and MB that are already approved for ISPM-15. As the efficacy of these two treatments might not be known, the Committee recommended that an expert judgement of their efficacy may be sufficient if exact scientific data were not available, as these data are urgently needed for the approval of new treatments. Fourthly, the International Forestry Quarantine Research Group at its meeting in September 2011 (CPM, 2012) agreed that Probit-9 was impractical for many wood pests and proposed an alternative approach to treatment that did not prescribe an efficacy target."

Some of the proposed replacements for wood fumigation, including for ISPM 15 and unsawn timber, have better penetrant ability than methyl bromide. This potentially addresses a continuing concern that methyl bromide only penetrates large section of wood slowly and may lose efficacy as a result. Phosphine, carbonyl sulphide, cyanogen ('EDN') and sulphuryl fluoride all are more penetrant than methyl bromide, and also show less loss by reaction (Ren et al., 1997, 2011).

Treatment of export whole logs continues to be a major user of methyl bromide as a QPS treatment. Some alternative processes are in use for particular trades. The use of phosphine in transit on logs going to China from New Zealand (Glassey, 2005) is well established. A mixture of methyl isothiocyanate and sulphuryl fluoride has been shown to be effective against a number of wood pests, including pinewood nematode (Soma et al., 2001), and is available for use on imported timber in Japan.

RESIDUAL GAS IN FREIGHT CONTAINERS

Continuing use of methyl bromide for QPS purposes brings with it some continuing safety hazards. This is also true for phosphine and, no doubt, will be so for new persistant fumigants as they are adopted. One such hazard of direct concern to quarantine and those involved in quarantine is the presence of residual fumigant gas in treated frieght containers. These may or may not have been well ventilated at point of treatment, but residual fumigant (and other toxic gases) may be commonly expected when the containers are opened by workers and inspectors including quarantine officials on import.

These are several studies that have been carried out to assess the frequency of presence of residual fumigant, particularly methyl bromide, in imported freight containers at levels dangerous to human health. In one such study, Baur et al. (2010) studied 2113 containers arriving at Hamburg port over a 10 week period in 2006 and found 294 of these with over 0.1 ppm methyl bromide with 23 of these with over 1 ppm methyl bromide. There was a similar incidence of high phosphine detections in other studies.

Some quarantine and customs agencies have deployed forced venting equipment, based on activated carbon recapture technology (Nordiko Quarantine Systems, pers. comm.) to assist in protecting their staff from undue exposure to residual fumigant.

It seems prudent to develop and deploy systems to avoid exposure of workers, inspector and bystanders to excessive fumigant concentrations. This is an important part of sustainable development of new fumigants for quarantine, as well as for continuing use of established ones.

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