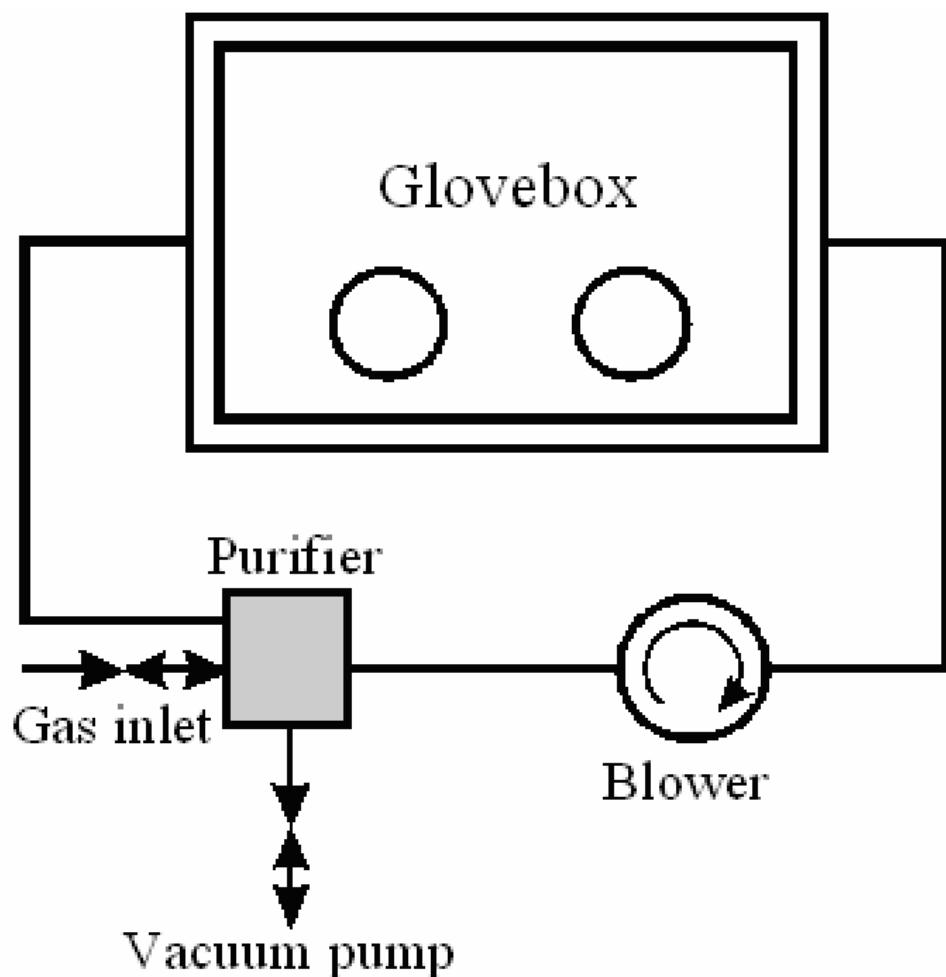


Peter Senn, Ph.D.

# Gloveboxes



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**Peter Senn, Ph.D.  
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## ABSTRACT

Current trends in the design and use of containment workstations or simply "*gloveboxes*" are discussed. Various designs of commercial glovebox systems are discussed with comments which will put them into perspective for potential applications. Criteria for the assessment and the appropriate design of containment workstations are provided, especially for workstations with recirculatory inert atmosphere systems for handling air-sensitive materials and anaerobic micro-organisms. A simple but fairly realistic mathematical model of gloveboxes with recirculatory gas purification systems is presented. It is shown, that the performance of containment work stations with gas purification systems can be predicted fairly accurately from a performance index  $\alpha$ , which is inversely proportional to the average time a particle of impurity spends inside the box before being adsorbed. Interactions among gloveboxes in glovebox lines with a single recirculatory gas purification system are also analyzed within the confines of the proposed mathematical model.

The performance of protective enclosures with controlled atmospheres might be substantially improved in the foreseeable future by using highly efficient gas purification systems in conjunction with laminar flow techniques similar to the ones used in systems with particulate filters.

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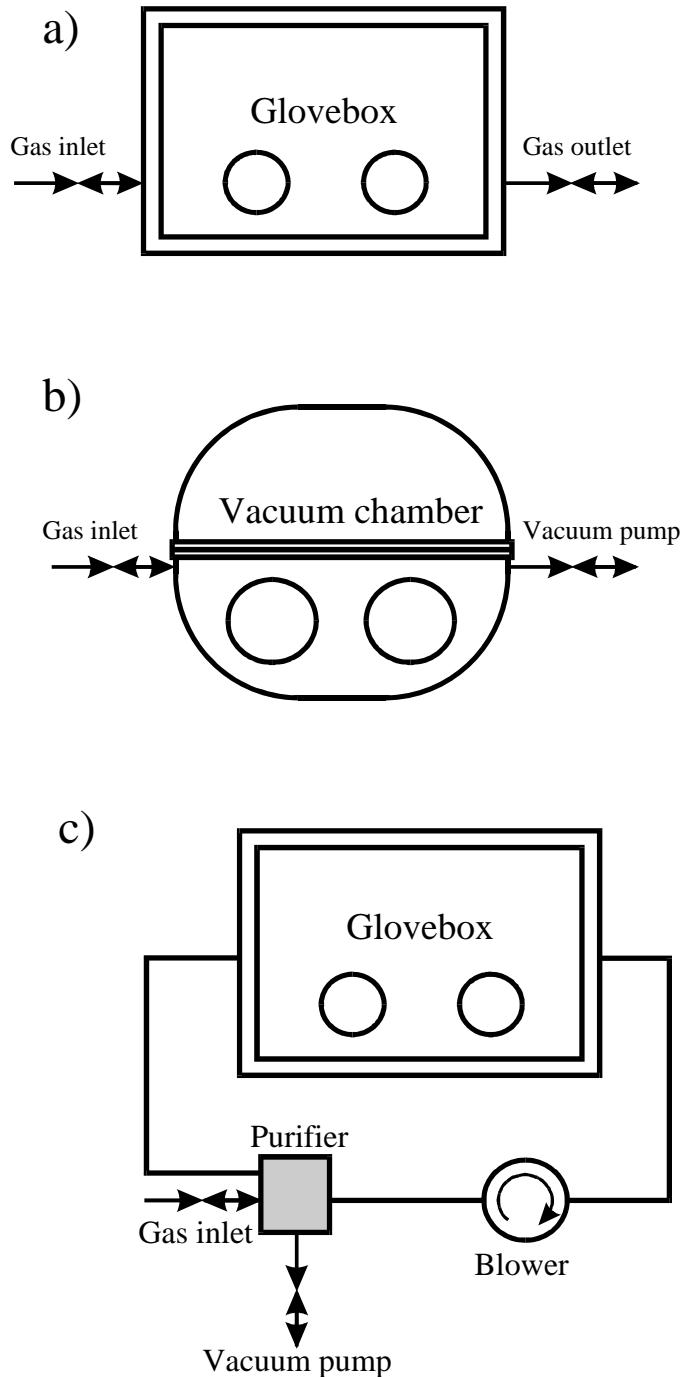
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## **1. Introduction**

The use of *gloveboxes* has a long tradition in fields with obvious hazards such as the chemical and nuclear industries. For a vast number of reasons the use of *containment workstations*, frequently referred to as *gloveboxes*, has spread dramatically in the past few years to industries and types of research which previously were not too much concerned with maintaining controlled environments. There are two principle reasons for using gloveboxes in facilities for small-scale manufacturing and/or research. In many cases gloveboxes are used for working with air sensitive materials [Shriver & Drezdzon, 1986; White & Smith, 962]. In some cases they are used to protect personnel handling harmful chemicals or micro-organisms which themselves are not air-sensitive. There may be considerable overlap of these objectives. The present discussion considers mainly applications where work has to be performed in a protective atmosphere in order to prevent damage to items being handled inside the box.

There exist three basic modes of operation for gloveboxes as follows:

- a) Preparation of an inert atmosphere by flushing an enclosure with inert gas or by flooding it with inert gas after being evacuated. In this mode of operation most of the equipment needed for the intended work is placed inside the box before applying vacuum. Some additional tools and substances which cannot tolerate full vacuum may be transferred into the box through an antechamber, which can be flushed with inert gas. After applying the vacuum the atmosphere inside the box is slowly contaminated by substances in the atmosphere infiltrating the box through tiny leaks or by diffusion, mainly through the gloves and rubber seals. After some time concentrations of contaminants inside the box will reach unacceptable levels.
- b) A box with flexible plastic walls is deflated around its contents to force air out through a tube, and then is inflated with inert gas.
- c) Maintaining an inert atmosphere by continuous flushing of an enclosure with inert gas, thereby keeping it in operation at all times. Some equipment may be installed inside the box permanently whereas some items may be transferred in and out of the box through antechambers which can be evacuated in most cases.
- d) Maintaining an inert atmosphere indefinitely by circulating the gas over an adsorbent bed which removes harmful impurities. Suitable gas purification systems are offered for sale by most manufacturers of gloveboxes.



**Fig. 1-1.** Illustration of three modes of operating a box with an inert gas atmosphere.

Three of the above modes of operation are illustrated in Fig. 1-1. The three modes illustrated are as follows: Continuous rinsing with inert gas (a); flooding with inert gas after evacuating the air inside the box (b); and maintaining an inert atmosphere indefinitely with a recirculatory gas purification system (c). A wide variety of gloveboxes are offered for sale by numerous manufacturers. The simplest and least expensive version of this type of enclosure frequently referred to as *glove bag* consists simply of a plastic bag with gloves. This kind of enclosure is considered "disposable" by many of its users. Based on specialized uses of gloveboxes we can distinguish seven major types as follows:

### **Controlled Atmosphere Glovebox:**

Enclosure with a controlled atmosphere. The control of the atmosphere can be achieved either by continuous flushing with inert gas or filtered air or by circulating the atmosphere inside the box through appropriate scrubbing devices. The box protects the operator from hazardous substances while allowing him to handle items inside the box via sealed gloves. At the same time the box may protect air sensitive materials from the surrounding atmosphere. Besides using gloveboxes there are three more techniques in current use for handling air-sensitive materials as follows [Salzer, 1996]:

- a) Vacuum line technique.
- b) *Schlenk technique.* (This technique requires specially adapted glass ware and a vacuum line).
- c) Septum technique. (This technique uses vials closed with rubber membranes through which liquids can be transferred with syringes).

In addition techniques have been reported where for short periods of time open vessels filled with argon are used [Quigley, 1991]. Many users of gloveboxes also apply the above techniques for handling air-sensitive materials. Boxes used for synthesis and purification procedures often need special feedthroughs for liquids used for cooling, preparative liquid chromatography, etc [Weidmann, 1987].

### **Anaerobic Glovebox:**

Enclosure for handling and cultivating *anaerobic micro-organisms* in research and clinical diagnostic laboratories. The oxygen which gets into the box by diffusion must be removed. In the majority of cases the inert gas used in the box contains a few percent of hydrogen gas ( $H_2$ ) which reacts with the traces of oxygen in the atmosphere when brought into contact with a suitable catalyst such as Palladium and/or Platinum. (Inert gas with an admixture of hydrogen is called *forming gas*). In some cases the atmosphere must be dried with a suitable desiccant. Anaerobic boxes are frequently equipped with UV lamps in order to sterilize the box. Some anaerobic boxes contain a few percent of carbon dioxide ( $CO_2$ ) in their atmospheres. A typical "*anaerobic mixture*" consists of 80%  $N_2$ , 10%  $H_2$ , and 10%  $CO_2$ , which is essential or stimulatory to the growth of many anaerobes [Willis & Phillips, 1988]. Many users do not use carbon dioxide in the box due to the risk of cross-contamination. They use carbon dioxide inside vessels with cultures or inside incubators. For *anaerobic boxes* designs with flexible vinyl films are very popular [Aranki & Freter, 1972]. Small quantities of redox indicator are usually added to the cultures in order to make sure that the concentration of oxygen is sufficiently low.

### **Biological Safety Cabinets:**

There are three classes of biosafety cabinets, all of them designed to protect the worker and the environment by laminar flow of air directed away from the user. The exhaust from the cabinets is usually through a HEPA filter. Biosafety cabinets are used for protecting the operator, and in most cases also the environment, from infectious agents. Biosafety cabinets of class II and III also protect the materials inside the cabinet. Only biosafety of class III are actual gloveboxes, although biosafety cabinets of class I and II in some cases have sliding front panels with gloves, iris diaphragms, or simply with a pair of holes. [CRC Handbook of Laboratory Safety, 1995; Kuehne *et al.*, 1995]. Since biosafety cabinets are not equipped in general with filters for capturing gaseous effluents, they are usually unsuitable for handling materials releasing toxic fumes.

### **Radioisotope Glovebox:**

Enclosure for handling low level radioactive materials. The enclosure must have adequate radiation shielding. If  $\alpha$ -emitters are handled shielding may not be necessary. Energetic  $\beta$ -emitters cannot be handled in normal glove boxes, partly due to the formation of *Bremsstrahlung*. The atmosphere in most of these boxes is circulated through scrubbing devices for removing particles and/or gaseous radioactive materials such as tritium. Pressure control devices are used in most cases in order to maintain a subatmospheric pressure whereby the emission of hazardous components can be reduced considerably. The window design frequently consists of a sandwich type construction utilizing layers of lead glass, safety-plate glass, and plastic to achieve adequate radiation shielding. Some windows have layers of water for neutron shielding. A maximum of 20 cm is considered the upper limit for working through glove ports. In some boxes the filtered exhaust from vacuum pumps is recycled into the enclosure. Manipulation is frequently effected partly or exclusively by operating mechanical tongs through the wall of the enclosure or by using master-slave manipulators. Glove ports are closed by iron and lead plugs when not in use.

### **Glovebox for Handling Hazardous Materials:**

Enclosure designed specifically for handling potentially hazardous chemical substances or pathogens. In general this type of box is equipped with HEPA (high efficiency particulate air) filters and a charcoal filtering system. Some boxes of this type are equipped with sliding front panels with gloves. They can then be used either as ordinary fume hoods or as gloveboxes.

## **Isolators and Growth Chambers:**

Enclosures for raising and keeping plants or laboratory animals in controlled surroundings. In some cases these organisms must be kept in a sterile environment or they must be isolated and kept under surveillance for some time in so-called *quarantine chambers*.

## **Glovebox for Manufacturing and Quality Assurance:**

This type of box may be used for a wide range of activities such as:

- welding, drilling, milling and cutting in an inert atmosphere.
- hazardous sample preparation, weighing, packaging etc.
- handling asbestos samples.
- assembly of electronic or micromechanical devices, e.g., pace makers.
- nanotechnology.
- handling of materials introduced into a manufacturing process.
- metallurgical sample preparation.
- removing sensitive or hazardous items from containers used for transport.
- opening and repairing sealed micromechanical devices.
- handling of materials leaving an industrial process as hazardous waste products.
- battery technology; handling of extremely air sensitive materials such as Lithium.
- manufacture of electronic component materials, e.g., semiconductor precursors.
- safe handling of new materials with unknown, potentially hazardous properties.
- welding of titanium alloys in inert atmospheres (aerospace industries).
- filling stations for pharmaceutical products.
- quality testing of components under controlled conditions, e.g., temperature and humidity.
- crystal growth.
- fabrication of optical fibers.
- packaging fruits or meat in controlled atmospheres.
- handling of low level radioactive waste.
- nuclear fuel fabrication.
- nuclear fuel reprocessing.
- vitrification of nuclear waste.
- retrieval of radioactive waste from interim storage.
- disassembly of irradiated samples.
- fusion research.
- plasma research.
- thin layer coating.
- housing of mass spectrometers, VUV spectrometers, etc.
- x-ray diffraction of air sensitive samples.

In many cases this type of box serves as *interface* or *safe buffer area* connected to an installation used for manufacturing or quality control. Some boxes are equipped with conversion front panels for the conversion of open-fronted cabinets to gloveboxes and vice versa. Using special seals enclosures can be obtained with performances similar to those of ordinary gloveboxes. Wilson *et al.* described a design for rapid box-to-hood conversion with an evacuated space in between twin inflatable seals [Wilson *et al.*, 1988].

## **2. Putting a Glovebox into Operation**

Boxes which can be evacuated generally are put into operation for a specific task of limited duration. Boxes which cannot be evacuated must be flushed with several volumes of inert gas before they can be used. Shriver and Drezdzon[Shriver & Drezdzon, 1986] distinguish three limiting cases according to the pattern of flow of the inert gas used for flushing as follows:

- a) **Perfect displacement:** The air in the box is removed in a plug flow mode where the inert gas pushes out the air without appreciable mixing. This type of situation may arise mostly in enclosures of elongated shape.
- b) **Perfect mixing:** The incoming inert gas and the gas in the box mix thoroughly. The concentration  $c$  of a given harmful component of air decreases exponentially as follows:

$$c(t) = c(0) e^{-V/V_B} \quad (2-1)$$

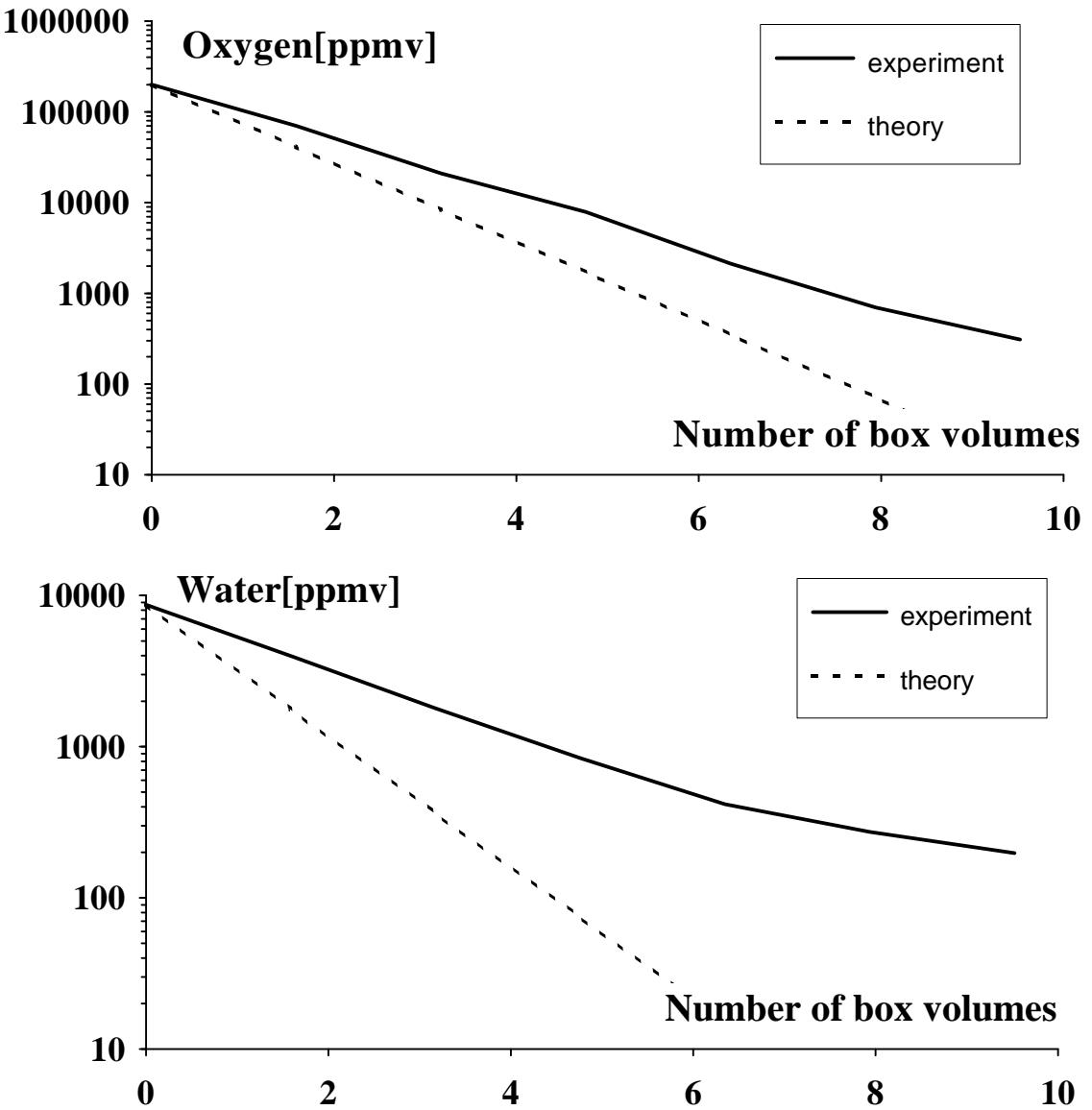
where  $V_B$  is the volume of the enclosure and  $c(0)$  is the concentration  $c$  at a time  $t = 0$ .

- c) **Short circuit:** The incoming inert gas mixes barely with the gas enclosed in the box, but unlike in the limiting case termed *perfect displacement* the inert gas passes from the inlet straight to the outlet whereby some regions inside the box are not being flushed at all and large quantities of impurities from the original atmosphere remain trapped in the box.

These three limiting cases of flushing are illustrated in Fig. 2-1.

**Fig. 2-1.** Three limiting modes of flushing a box with inert gas.

The three limiting modes of flushing are as follows: Perfect displacement (a), perfect mixing (b), and short circuit (c). White and Smith [White & Smith, 1962, Fig. 7.5] have reported experiments where a box has been flushed with carbon dioxide in different ways corresponding largely to the limiting cases outlined above. What is observed in practice is that the rate at which the air is purged from the box is considerably lower than the theoretical rate for perfect mixing. This is illustrated for oxygen in the upper part of Fig. 2-2 which shows the concentration of oxygen,  $O_2$ , (upper part) and water vapor,  $H_2O$ , (lower part) inside a box with a volume of 630 liters while being flushed with nitrogen,  $N_2$ , at a rate of roughly 3 liters per second. The volume of the gas used for flushing is expressed on the horizontal scale with "box volumes"; ten box volumes would correspond to a volume of 6300 liters. Also shown as dashed lines are the concentrations computed from (2-1). The concentration of oxygen follows an exponential decay. However, the dilution is less than one would expect from the rate of flushing for the limiting case referred to as perfect mixing. The reason probably lies in the fact that in the corners of the box the rate of mixing of the gas is relatively low. In many cases vessels and electrical instruments are placed inside the box when it is put into operation. Inside such items large quantities of air may remain trapped. In boxes with gas purification systems elevated concentrations of impurities are observed for some time after starting the circulation of the inert gas through the adsorbent column. This is due to outgassing of impurities adsorbed in the walls and various items located inside the box. Water is known to cover most materials, especially glasses, with a thin film which evaporates only very slowly [Thiel & Madey, 1987]. In order to quickly decontaminate items transferred into the box some antechambers can be heated. The lower part of Fig. 2-2 shows the concentration of water vapor in a box during and after flushing with inert gas. The data shown in Fig. 2-2 were obtained for flushing a box with a volume of 630 liters at a rate of approximately 3 liters per second.



**Fig. 2-2.** Concentration of oxygen and water vapor in a box during flushing with inert gas.

The expense of putting a glovebox into operation can be obtained as follows:

$$\begin{aligned} \text{total cost} &= \text{cost of inert gas used for flushing} + \text{cost of adsorbing residual impurities} \\ &= \varepsilon_i V + \varepsilon_a \frac{C}{C_o} V_B = \varepsilon_i V + \varepsilon_a e^{-V/V_B} V_B \end{aligned} \quad (2-2)$$

where  $\varepsilon_i$  and  $\varepsilon_a$  represent the cost of 1 liter inert gas used for flushing and the cost for adsorbing the impurities in 1 liter of air respectively. The latter quantity can be obtained by dividing the cost for regeneration or replacement of the adsorbent bed by its capacity expressed in terms of the number of liters of air which could be purged of its impurities by a fresh adsorbent bed. From this we obtain for the most economical use of inert gas for flushing the following:

$$\frac{V}{V_B} = \ln \frac{\epsilon_a}{\epsilon_i} \quad (2-3)$$

This means that the start-up of the box would be least expensive if the purification system were put into operation after the box had been flushed with a volume  $V$  of inert gas as indicated in (2-3). However, some adsorbents can overheat when exposed to gas with excessive levels of contamination. For this reason the most economical procedure may not be "economical" at all. (See also chap 15). Some manufacturers sell large balloons which can be inflated inside the box with inert gas. [Senn, 1993; Sherfey, 1954]. The box is then flushed with the inflated balloon inside, thus reducing the amount of inert gas needed for flushing. After flushing the balloon is deflated and taken out of the box.

When flushing a box the locations of the inlet and outlet should be such that the pattern of flow would unlikely approach the limiting case referred to as "short circuit". Some gases used in boxes differ considerably from the density of air. This fact can be exploited to more closely approach the most favorable case referred to as perfect displacement. White and Smith [White and Smith, 1962, pp 148-149] published results obtained from flushing a box with carbon dioxide for various setups.

In some cases a line of gloveboxes has to be put into operation. In this case it may be advantageous to flush the boxes in series

$$[c_1] \rightarrow [c_2] \rightarrow [c_3] \rightarrow [c_4] \rightarrow [c_5] \rightarrow [c_6] \rightarrow [c_7] \dots \dots \quad (2-4)$$

After flushing with a volume  $V$  of inert gas the concentration in the  $n$ -th box in a series of boxes with equal volume  $V_B$  is obtained for "perfect mixing" as follows:

$$\frac{c_n(V)}{c(0)} = \sum_{k=1}^n \left( \frac{V}{V_B} \right)^{(k-1)} \frac{e^{(-V/V_B)}}{(k-1)!} \quad (2-5)$$

where  $c(0)$  is the concentration of the contaminant in air. For example, in order to obtain a dilution of 0.00001 a single box would have to be flushed with roughly 11.5 box volumes. For two boxes in series "only" 14.2 box volumes would be needed in order to obtain a dilution of 0.00001 in the box last in line. For three or four boxes in series 16.6 and 18.7 box volumes, respectively, would be required. For five or more boxes in series the volume of inert gas needed would increase by less than two box volumes per box added (beyond four boxes).

### **3. Criteria for Performance Evaluations of Gloveboxes with Inert Atmospheres**

There are two main aspects relating to the performance of gloveboxes for working in inert atmospheres as follows:

1. The ability to provide quickly an atmosphere with low concentrations of contaminants.
2. The ability to maintain a low concentration of contaminants.
3. The ability to return to normal operating conditions after a contamination, where the term contamination shall denote throughout an event where relatively large quantities of contaminants enter the box within a short period of time, for example due to the transfer of items into the box which are hard to decontaminate.

For different applications the above criteria are relevant to various degrees. If it is important for a user to establish an atmosphere with low concentrations quickly then his needs may be served best with an evacuable glovebox. With this type of box concentrations of oxygen as low as 15 ppmv (parts per million by volume) may be reached within minutes. However, if no gas purification system is being used the concentration of oxygen and other contaminants would rise quickly, due to outgassing and diffusion processes. This type of box is suitable for working for two or three hours at concentrations of oxygen below 200-300 ppmv. Frequently this type of box is being operated as a *dry box* by keeping an open tray filled with a strong drying agent inside the box at all times. In addition to the above criteria a number of additional aspects such as the illumination of the box may be deemed relevant by the user. Appendix B shows some additional criteria which may be relevant in an evaluation of the performance of gloveboxes. Also shown are measures for optimizing the performance of a box.

## **4. Adsorption of Gaseous Impurities by Reactive Materials**

The damage resulting from brief exposure to elevated concentrations of contaminants may be due to chemical reactions or reversible adsorption. In the first case the substance may be altered and the cumulative damage may be in terms of a corresponding loss of yield in chemical reactions or the damage may be such that the sample must be considered spoiled. If the contaminants are merely adsorbed by the sample the damage may be remedied. However, if substances which themselves are unreactive towards contaminants later are used as reactants in processes involving air-sensitive substances the preceding contamination may represent a damage nonetheless.

If the sensitive substance undergoes chemical reactions with impurities in the inert atmosphere then the total amount  $\Delta m$  of material lost by the reaction can be computed as follows:

$$\Delta m \approx k \int_0^t [c(t) - \Lambda \Delta m] dt \quad (4-1)$$

where  $c(t)$  is the concentration of the corresponding impurity,  $k$  is the transfer coefficient for the adsorption,  $\Lambda$  is the partition coefficient and the integration is over the time of exposure. We shall consider here the case, where the amount of contamination adsorbed by the sample is far from equilibrium, i.e., in (4-1) we take  $\Lambda \approx 0$ . This gives

$$\Delta m \approx k \int_0^t c(t) dt = k C(t) \quad (4-2)$$

where  $C(t)$  is the concentration of the contaminant,  $c(t)$ , integrated over time, i.e.,

$$C(t) = \int_0^t c(t) dt \quad (4-3)$$

The quantity  $C(t)$  will be referred to as *exposure* throughout. It is related to the *transfer index*,  $T_\infty$ , first proposed by Lidwell [Lidwell, 1960] which is defined as follows:

$$C(\infty) = c(0) T_\infty \quad (4-4)$$

where  $c(0)$  is the concentration of the contaminant at the time  $t = 0$ .

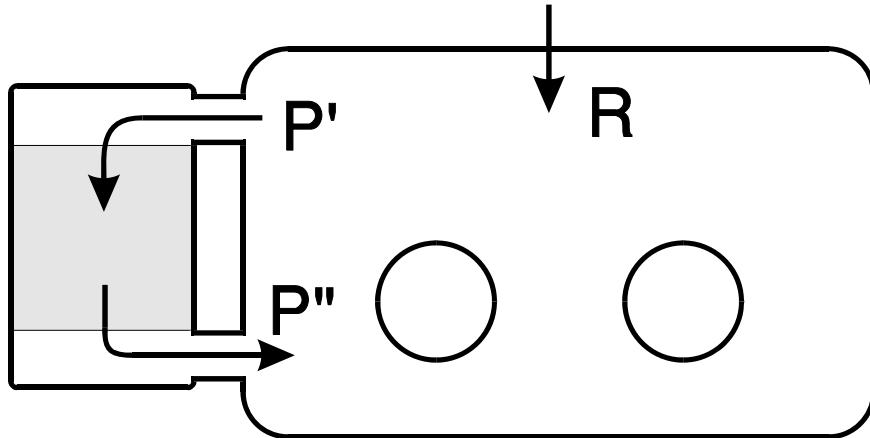
If the sample were saturated with the impurity at a given average concentration  $\bar{c}$  then the amount of impurity adsorbed would be obtained as follows:

$$\Delta m \approx \frac{\bar{c}}{\Lambda} \quad (4-5)$$

where  $\bar{c}$  is the average concentration of the corresponding impurity or it may be the most recent concentration of the impurity if the equilibrium between adsorption and desorption is reached quickly.

## **5. A Simple Mathematical Model for a Glovebox with a Recirculatory Gas Purification System**

In what follows we shall assume throughout that the concentrations of contaminants are the same everywhere in the box, i.e., the atmosphere is mixed thoroughly by ventilation. Fig. 5-1 shows a schematic picture of a glovebox with a recirculatory gas purification system.



**Fig. 5-1.** Illustration of the mathematical model used for boxes with a recirculatory gas purification system.

The change of mass per unit time of an impurity in the box can be obtained as follows:

$$\dot{m} = \frac{dm}{dt} = R - P' + P'' = R - P \quad (5-1)$$

where  $R$  is the rate at which the impurity leaks into the box from the outside and  $P$  is the rate at which the impurity is adsorbed in the adsorbent bed of the gas purification system. The rate  $R$  remains roughly constant in general. The rate  $P$  can be computed as follows:

$$P = P' - P'' \approx K \dot{V} c \quad (5-2)$$

where  $\dot{V}$  is the flow of the gas through the adsorbent bed,  $c$  is the (average) concentration of the impurity in the box and the parameter  $K$  represents the probability for a molecule of impurity of being adsorbed in the adsorbent bed. In a first approximation the "survival

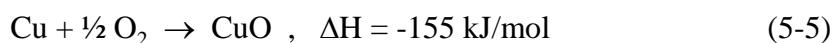
"probability" of a molecule of impurity in the adsorbent bed decreases exponentially with time and we can write

$$K \approx 1 - e^{-\beta \tau} \quad (5-3)$$

where  $\tau$  is the average time the molecule spends in the adsorbent bed. From (5-2) and (5-3) it is apparent that on one hand the efficiency of the gas purification system increases with an increase of the flow of gas through the reactor but it decreases with the concomitant decrease in  $\tau$ . The parameter  $\beta$  is related to the *half life time*,  $t_{1/2}$ , of gaseous particles passing through the adsorber as follows:

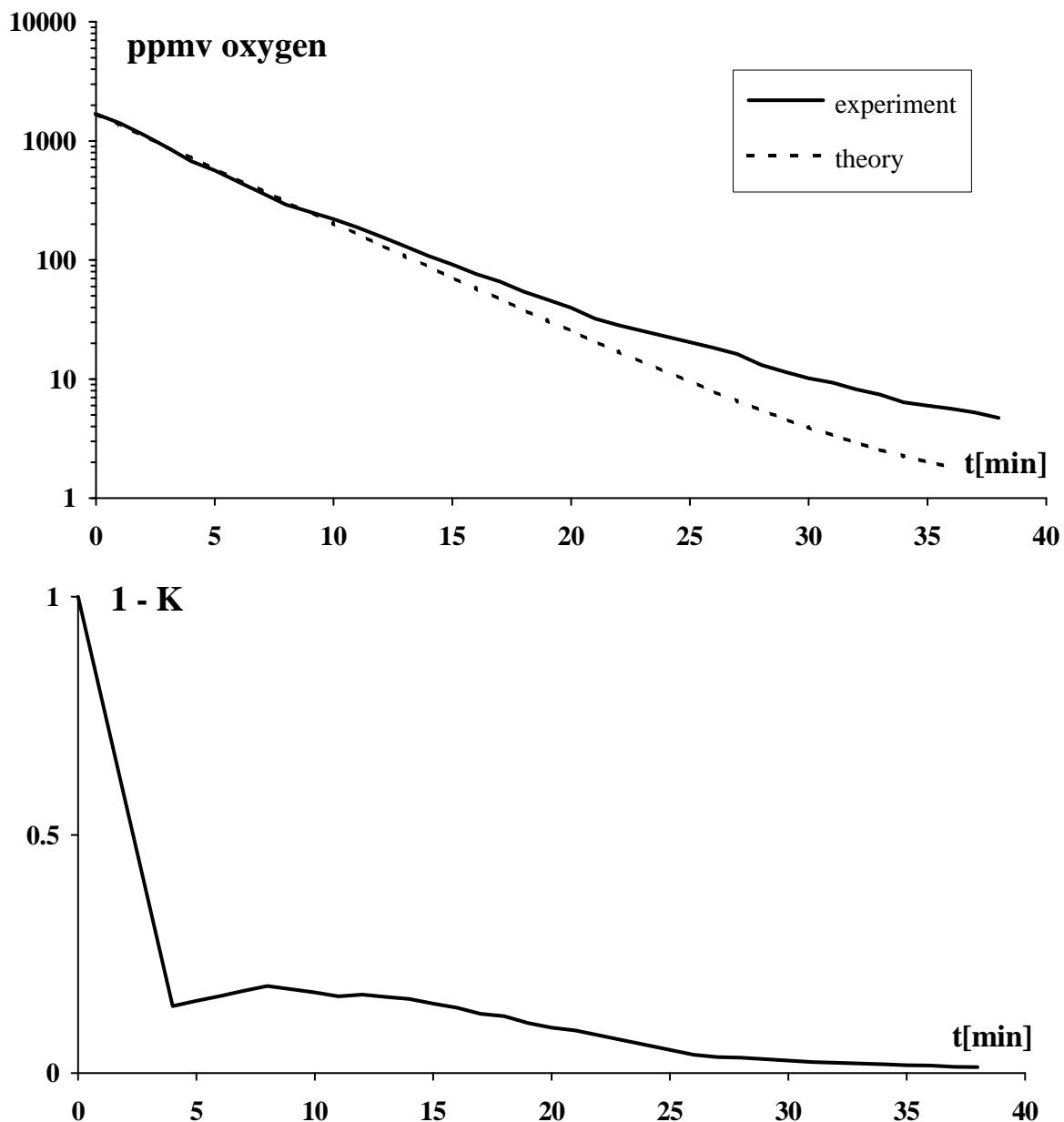
$$\beta = \frac{\ln 2}{t_{1/2}} \quad (5-4)$$

In most purification systems more than 80% of the oxygen passing through the adsorbent bed is adsorbed, i.e., for oxygen we have  $K \approx 1$ . Fig. 5-2 shows the concentration of oxygen at the inlet and the outlet of an adsorber system connected to a box with a volume of 630 liters and a recirculatory gas purification system with a rate of gas flow of  $8\text{m}^3/\text{h}$  after the injection of 5 liters of air. The computed concentration of oxygen is also shown as a dashed line. In the lower part the ratio of concentrations at the inlet and at the outlet is shown. This ratio, which corresponds to  $1 - K$ , is less than 0.2 in the example shown in Fig. 5-2. The fact that at the beginning this ratio is close to unity has to do with the fact that the instrument for measuring oxygen needs some time to adjust to sudden changes in the concentration. In most purification systems for the removal of oxygen copper sponge is being used which adsorbs oxygen by forming copper oxide



The oxidized copper sponge can be regenerated with hydrogen as follows:

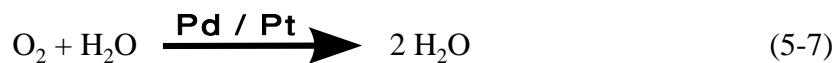




**Fig. 5-2.** Purification curve for oxygen in a box with a volume of 630 liters.

Since the net reaction of (5-5) and (5-6) corresponds to the formation of water from its elements, mediated by the presence of copper, the adsorbent is often referred to as "*copper catalyst*". Older models of gas purification systems for removal of oxygen use manganese oxide instead of copper sponge. For the regeneration of manganese oxide hydrogen is used as well. In practice forming gas consisting of a mixture of hydrogen and inert gas is used for this purpose, one reason being that pure hydrogen gas brought into contact with a depleted catalyst may cause an excessive increase in temperature. Temperatures exceeding 350°C will damage the copper catalyst irreversibly. The use of forming gas instead of pure hydrogen also lessens the danger of the formation of explosive mixtures in the box if large quantities of oxygen have contaminated the box. Hydrogen can form explosive mixtures with air at concentrations ranging from 4.5 to 74% of hydrogen by volume. In gloveboxes which are referred to as anearobic boxes forming gas is used inside the box instead of inert gas.

For this type of box precious metal catalysts containing *Platinum* and/or *Palladium* are used. The precious metal catalysts convert the traces of oxygen inside the box by accelerating its reaction with the hydrogen present in the atmosphere of the box.



Anaerobic enclosures are used mainly for the cultivation of anaerobic micro-organisms. The catalyst may be inactivated reversibly by the adsorption of moisture or it may be irreversibly poisoned by products of bacterial metabolism, especially hydrogen sulfide ( $\text{H}_2\text{S}$ ). The catalyst must be rejuvenated at regular intervals by heating it in a sterilizing oven at  $160^\circ\text{C}$ . [Jones, Whaley & Dever, 1977]. The exposure of the catalyst to harmful chemicals may be reduced to some extent by scrubbing the atmosphere with activated charcoal. Although palladium beds do not require regeneration, they are more easily poisoned by impurities than the  $\text{Cu}/\text{CuO}$  systems, mainly by halogenated hydrocarbons and compounds containing sulfur. Adsorbent columns which can be regenerated have been prepared also using  $\text{MnO}/\text{MnO}_2$  systems [White & Smith, 1962, pp 41-48]. Many users use nitrogen as inert gas. A minority of users do not consider nitrogen to be an "inert gas". There is a growing need to work in atmospheres free of nitrogen. The levels of contamination with nitrogen might be kept sufficiently low in a rare gas atmosphere by intermittent flushing due to pressure regulation and antechamber use when the box is in operation. Users who wish to operate their boxes with atmospheres containing less than 200 ppmv nitrogen need a gas purification system for the adsorption of nitrogen. In commercial systems the favored method is the adsorption with titanium sponge at elevated temperatures of  $750 - 900^\circ\text{C}$ . At these high temperatures the titanium forms a nitride ( $\text{Ti}_2\text{N}_3$ ) with nitrogen. Some users simply attach a small thermally insulated tube furnace containing titanium sponge to the box in order avoid excessive accumulation of nitrogen. The author is not aware of any reliable reports in the literature concerning the performance of this type of system for nitrogen absorption without the use of a circulation blower. The reactivation of the depleted getter material by a simple process is not possible. Gibb [Gibb, 1957] described a glovebox with a gas purification system circulating the gas through a high voltage sodium arc.

The common cobalt-impregnated desiccants, which change color when saturated with water, undergo this color change at far too high moisture levels to be useful for handling moisture-sensitive materials. Removal of moisture can be effected by molecular sieves which can be regenerated by boiling off the adsorbed moisture under vacuum at elevated temperatures. In most gas purification systems molecular sieves of type 13X are used which also adsorb solvent vapors [Shriver & Drezdzon, 1986, Sect. 2.3.E]. Some solvent vapors can poison the catalysts used for oxygen scavenging. Solvent vapors are removed most effectively with activated charcoal. Adsorbers for the removal of solvent vapors with activated charcoal can be regenerated by stripping with steam or low pressure gas at elevated temperatures [White & Smith, 1962, pp 77-85; Wadden & Scheff, 1987]. Regeneration with

steam is very effective in general. However, after regeneration with steam the wet activated charcoal would have to be dried thoroughly. When used for gloveboxes saturated adsorbent beds with activated charcoal are usually replaced rather than regenerated. The vapors adsorbed by the activated charcoal tend to migrate downstream by desorption into the gas stream where they are carried over some distance before being re-adsorbed [White & Smith, 1962, Fig. 4.20]. This tendency of solvent vapors to spread in the adsorbent column may reduce significantly the service life of the adsorbent bed.

## **6. Concentrations of Impurities at Equilibrium**

According to (5-1) the concentration of the impurity is at its equilibrium when  $R - P = 0$ , i.e., if  $\dot{m} = 0$ . From (5-2) we then obtain for the equilibrium concentration,  $c_{eq}$ , the following:

$$c_{eq} = \frac{R}{K} \quad (6-1)$$

According to this the concentration of impurity at equilibrium does not depend on the volume of the box. The rate  $R$  at which the impurity invades the box is expected to be larger for boxes of greater size, however.

Gloves of current design have rates of permeation of oxygen not below  $1 \text{ cm}^3/\text{h}$  per glove and the rate of circulation,  $\dot{V}$ , of the atmosphere is currently limited to around 60,000 liters/h. Even the use of gloves which are continuously flushed with inert gas does not change the situation a great deal. Using in (6-1)  $K = 1$  we would compute an equilibrium concentration,  $c_{eq}$ , of 0.033 ppmv for an "ideal box" with a pair of gloves of current design. From this it is readily apparent that, when using a glovebox, a user would ordinarily expect to be working at a ppmv level of impurities.

Notice that the volume of the box has no immediate effect on  $c_{eq}$ , the concentration of the impurity at equilibrium. The volume of the box affects this concentration only indirectly due to the fact that the influx of impurities tends to increase with the size of the box.

## **7. The Damage Resulting from a Contamination**

A further criterion for the performance of a gas purification system is the time needed to remove a contamination to a certain extent or, conversely, the average life time of a particle of impurity from the time it enters to the box to the time it is being adsorbed in the reactor. If

there is a contamination inside the box the rate  $R$  at which the impurity enters the box is largely irrelevant. From (5-1) we obtain an exponential decay of the impurity as follows:

$$c(t) = c(0) e^{-\alpha t} + c_{eq} [1 - e^{-\alpha t}] \quad (7-1)$$

where  $\alpha = KV/V_B$ . In the upper part of Fig. 5-2 the theoretical curve for the removal of a contamination in a box with a volume  $V_B$  of 630 liters and with a rate of gas flow through the adsorber,  $V$ , of approximately  $8m^3/h$  is shown as a dashed line. Using  $K \approx 1$  the value of  $\alpha$  is  $(8000/60 \cdot 630) \text{ min}^{-1} \approx 0.21164 \text{ min}^{-1}$ . The influx of oxygen was such that  $R \approx 6 \text{ ppmv/h}$  from which  $c_{eq} \approx (6 \cdot 630/8000) \text{ ppmv} = 0.47 \text{ ppmv}$  would be obtained. The actual value used for the computed purification curve was 1 ppmv for  $c_{eq}$ . The term "purification curve" refers to the removal of a contaminant by a recirculatory gas purification system after a contamination. The computed curve is in fair agreement with the experimental curve, except at low concentrations. This is due to the fact that instruments had been placed in the box. It appears that the removal of traces of oxygen which had infiltrated these instruments took longer than one would expect from the present model. The fact that the present model does not consider the retention of contaminants by reversible adsorption of surfaces inside the box is distinctly apparent in the purification curve for water shown in Fig. 7-1. Fig. 7-1 shows the purification curve for water after injection of  $7000 \text{ cm}^3$  of air into a box with a volume of 630 liters.

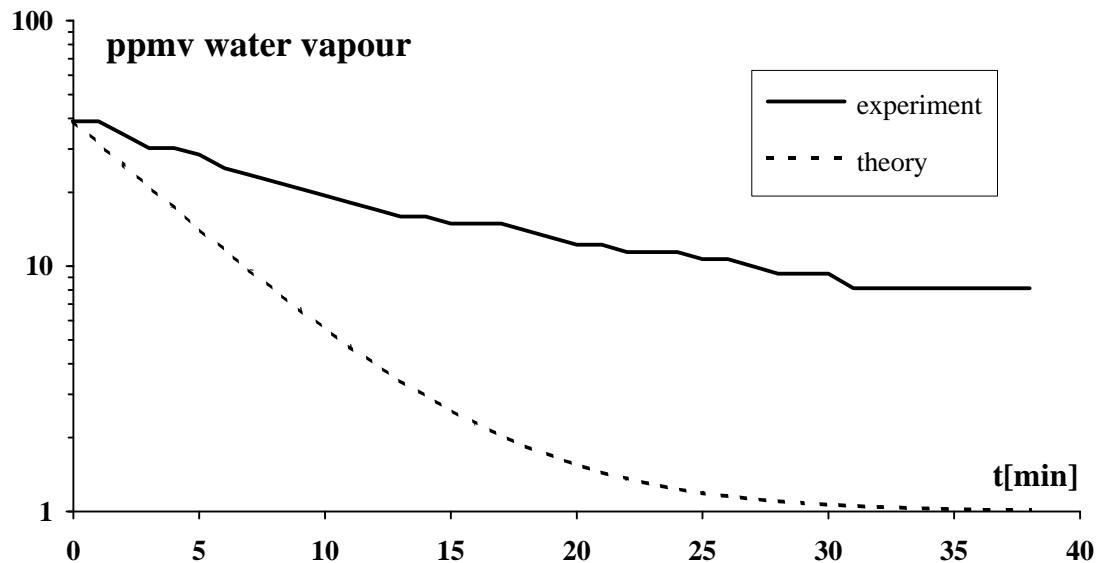


Fig. 7-1. Purification curve of water vapor after injection of  $7000 \text{ cm}^3$  of air.

The large discrepancies between experimental and computed concentrations are believed to be due to the tendency of water to adsorb tenaciously on almost any available surface [Thiel & Madey, 1987]. After a contamination water may therefore persist at elevated concentrations inside a box for extended periods of time, depending on the time it takes the contents of the box to dry out. There is not much point in incorporating adsorption of contaminants into the model because the corresponding aftereffects of a contamination depend strongly on what is inside the box. The values used for  $\alpha$  and  $c_{eq}$  for the computed purification curve in Fig. 7-1 were the same as those used for Fig. 5-2. In what follows the small second term will be neglected, i.e., we will use

$$c(t) \approx c(0) e^{-\alpha t} \quad (7-2)$$

According to (4-2) the damage resulting from a contamination with a relatively high concentration  $c(0)$  can then be expressed approximately as follows:

$$\Delta m \approx k c(0) \int_0^{\infty} e^{-\alpha t} dt = \frac{k c(0)}{\alpha} = \frac{k V_B c(0)}{K} \quad (7-3)$$

The initial mass of impurity equals  $V_B c(0)$ . We therefore can write

$$\Delta m \approx \frac{k m(0)}{K} \quad (7-4)$$

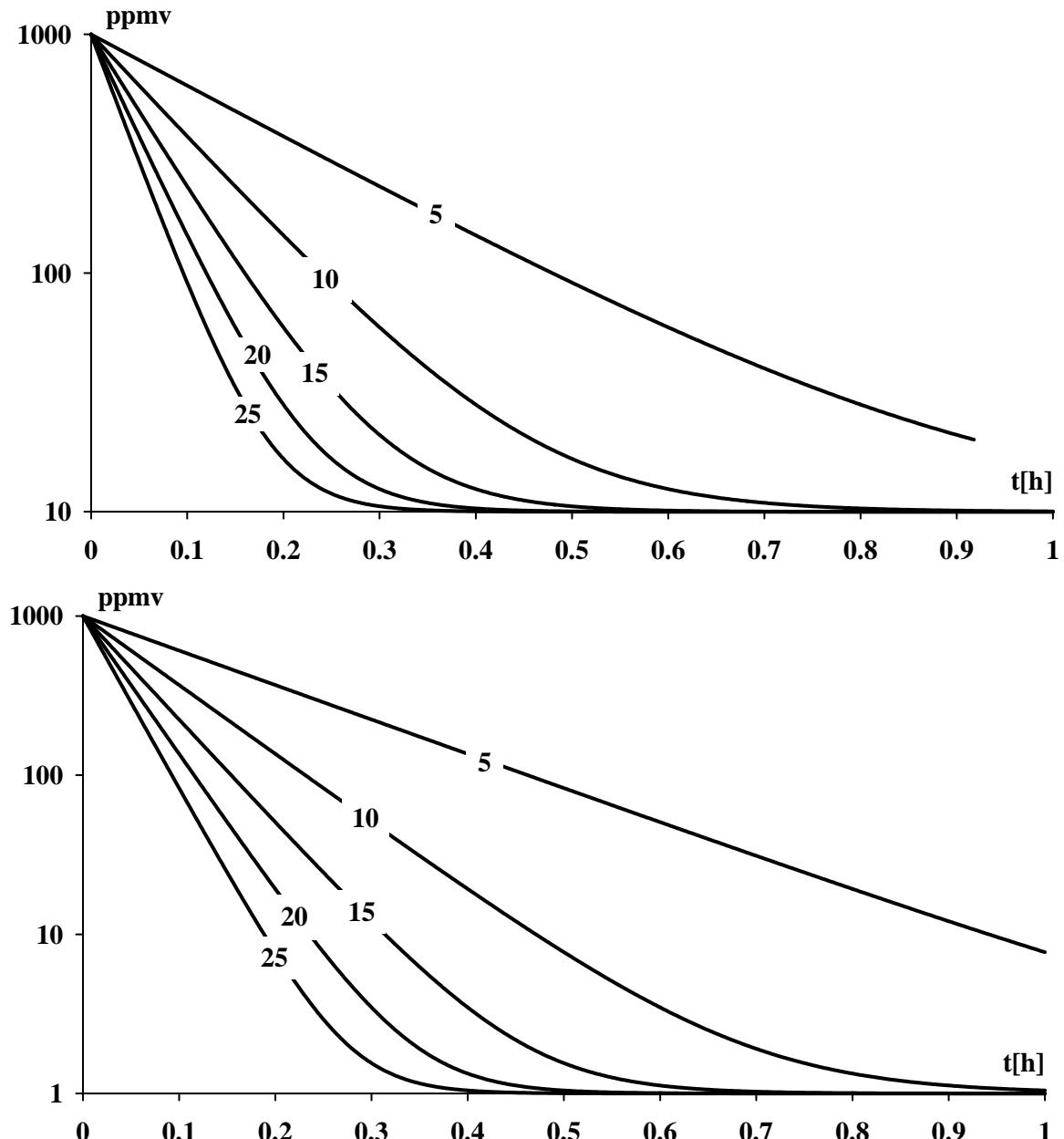
where  $m(0)$  is the mass of impurity present in the box at  $t = 0$ . According to the above the damage resulting from a contamination with a certain amount of air does not depend on the size of the box, even though the contamination will be diluted to a greater extent in a larger box. The exposure to lower concentrations of impurities during a contamination is compensated by the longer exposure to the impurity.

The average lifetime of a particle of impurity in the atmosphere can be computed as follows:

$$t_{ave} = \frac{\int_0^{\infty} t c(t) dt}{\int_0^{\infty} c(t) dt} \approx \frac{\int_0^{\infty} t e^{-\alpha t} dt}{\int_0^{\infty} e^{-\alpha t} dt} = \frac{1}{\alpha} = \frac{V_B}{K} \quad (7-5)$$

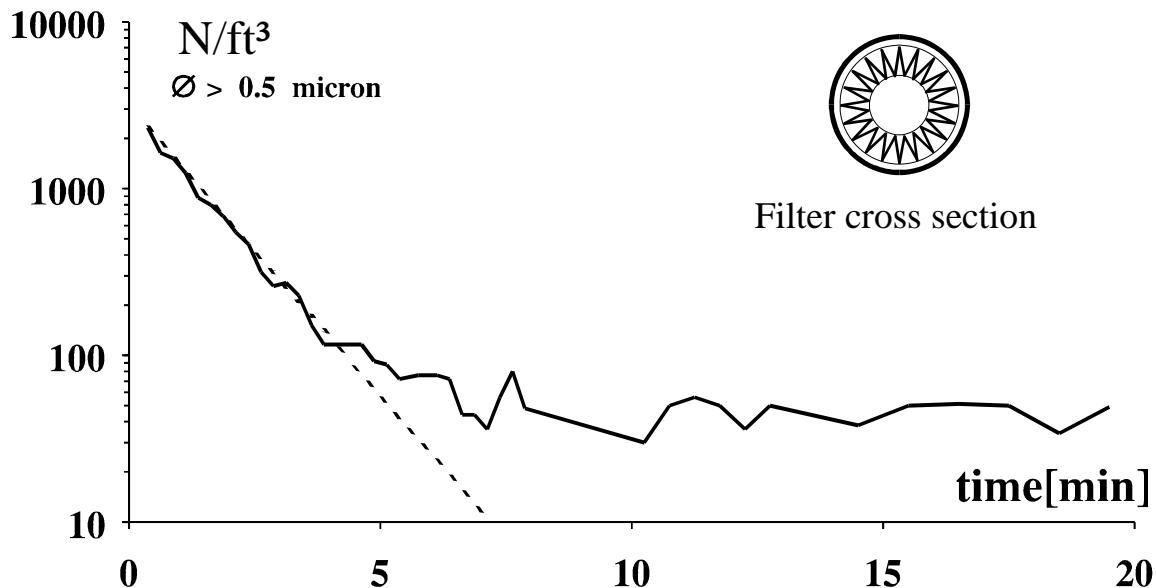
It then turns out that a molecule of impurity entering a box survives on average  $n$  times longer if the volume of the box is increased by a factor of  $n$ . The damage resulting from a contamination in a system with a gas purification system is then the same as if the air-sensitive sample were exposed to an impurity with a constant concentration  $c(0)$  in the

atmosphere during the time  $t_{ave}$ . Fig. 7-2 shows purification curves computed according to (7-1) for five different ratios  $\alpha = KV/V_B$  ranging from 5 to  $25 \text{ h}^{-1}$  and equilibrium concentration,  $c_{eq}$ , of 10 ppmv (in the upper part of Fig. 7-2) and 1 ppmv (in the lower part of Fig. 7-2).



**Fig. 7-2.** Computed purification curves for a series of values of the *performance index*  $\alpha$ .

The adsorption of particles in a purification system with gas circulation through particulate filters can be modeled in an analogous fashion. However, in contrast to contamination by gaseous substances, the solid particles do not infiltrate the box from the outside in general and with respect to the release of particles the interior of the box can be viewed as a reservoir with virtually infinite capacity. Fig. 7-3 shows the number of particles per cubic foot with diameters greater than  $0.5\mu\text{m}$  in a box made of acrylic glass with a volume of roughly 275 liters.



**Fig. 7-3.** Purification curve for dust particles exceeding  $0.5\mu\text{m}$  in diameter.

The rate of gas circulation through a small HEPA filter was roughly 0.8 box volumes per minute. The theoretical curve has been computed according to (7-2) using  $\alpha = 0.8 \text{ min}^{-1}$ . The filter was of cylindrical shape with a diameter of roughly 10 cm and a height of approximately 15 cm. The particulate filter was pleated inside its housing as shown schematically in Fig. 7-3. The initial concentration of particles was roughly  $2500/\text{ft}^3$ . The dashed line shows the approximate expected decrease with time of the particle count after the ventilator was turned on, i.e.,

$$N/\text{ft}^3 \approx N_0/\text{ft}^3 e^{-(\dot{V}/V_B)t} \approx (2500/\text{ft}^3) e^{-0.8 t/\text{min}} \quad (7-6)$$

After about ten minutes the particle count remained stable at roughly  $50/\text{ft}^3$ . Assuming the HEPA filter to be 100% effective this corresponds to a rate of particle emission in the undisturbed box as follows:

$$R \approx N_{eq} \dot{V} \approx \frac{50 \text{ particles}}{\text{ft}^3} \left( \frac{48}{\text{h}} \right) \left( \frac{275 \text{ liters}}{28.3 \text{ liters}/\text{ft}^3} \right) \approx \frac{389 \text{ particles}}{\text{min}} \quad (7-7)$$

For comparison; a person sitting motionless on a chair releases roughly 100,000 particles with  $\varnothing > 0.3 \mu\text{m}$  per minute. Maintaining a particle count of  $50/\text{ft}^3$  would correspond to a clean room of class 100, according to US Federal Standard No. 209, [US Federal Standard No. 209D].

## **8. Maintaining Low Concentrations of Impurities by Continuous Rinsing of the Glovebox with Inert Gas**

Boxes with inert atmospheres which are operated by continuous rinsing can be analyzed quite readily within the confines of the proposed mathematical model. For a box with a volume  $V_B$  which is rinsed at a rate of  $\dot{V}_r$  with inert gas containing traces of the impurity at a concentration of  $c_i$ , the following is obtained:

$$V_B \frac{dc}{dt} = R - \dot{V}_r (c - c_i) \quad (8-1)$$

In general the concentration  $c_i$  is negligible and (8-1) becomes largely analogous to (5-1)

$$V_B \frac{dc}{dt} = R - \dot{V}_r c \quad (8-2)$$

The rate of rinsing with inert gas needed in order to maintain an equilibrium concentration  $c_{eq}$  by continuous rinsing of the box is obtained for  $\frac{dc}{dt} = 0$  from (8-1) and (8-2)

$$\dot{V}_r = \frac{R}{c_{eq} - c_i} \approx \frac{R}{c_{eq}} \quad (8-3)$$

If 4 ml of oxygen get into a box per hour, then the rate for rinsing needed to maintain an equilibrium concentration of 10 ppmv oxygen would be

$$\dot{V}_r = \frac{0.004 \text{ l/h}}{0.00001} = 400 \text{ l/h} \quad (8-4)$$

The current discussion may also be of some concern to users working with rare gas atmospheres in systems without an adsorbent column for nitrogen. Let us assume that the influx of nitrogen,  $N_2$ , into the box is less than 10 ml per hour and that the average use of rare gas is at least 20 liters per hour. From this we would infer that the equilibrium

concentration of nitrogen should not exceed 500 ppmv under ordinary circumstances. This is obtained as follows:

$$c_{eq} < 1,000,000 \text{ ppmv} \frac{0.01 \text{ l/h}}{20 \text{ l/h}} = 500 \text{ ppmv} \quad (8-5)$$

## **9. The Effect of the Rate of Leakage on the Concentrations of Impurities at Equilibrium**

The leak rate is usually defined as follows [Roth, 1976]:

$$Q_L = V_B \left( \frac{dp}{dt} \right) \quad (9-1)$$

For the volume of gas leaking into the box per unit time we can write

$$\frac{dV}{dt} = \frac{Q_L}{p} \quad (9-2)$$

where  $p$  is the absolute pressure inside the box. The rise of concentration of impurities per unit time is then

$$\frac{dc}{dt} = c_a \frac{Q_L}{pV_B} \quad (9-3)$$

where  $c_a$  denotes the concentration of the impurity in the surrounding atmosphere. A common unit used for the leak rate is *lusec* corresponding to one  $\mu$  (micron of Hg) · liter / second. (An influx of  $1\text{cm}^3$  air at STP per hour would correspond to a leak rate of 0.211 lusec). Expressing the concentration of oxygen in ppmv we would obtain for a box with a volume of 700 liters and a rate of leakage of 150 lusec for the rate of increase of the concentration the following:

$$\frac{dc}{dt} = 200,000 \text{ ppmv} \frac{0.15 \text{ Torr} \cdot \text{liter} / \text{s}}{760 \text{ Torr} \cdot 700 \text{ liters}} \left( \frac{3600 \text{s}}{\text{h}} \right) = 203 \frac{\text{ppmv}}{\text{h}} \quad (9-4)^*$$

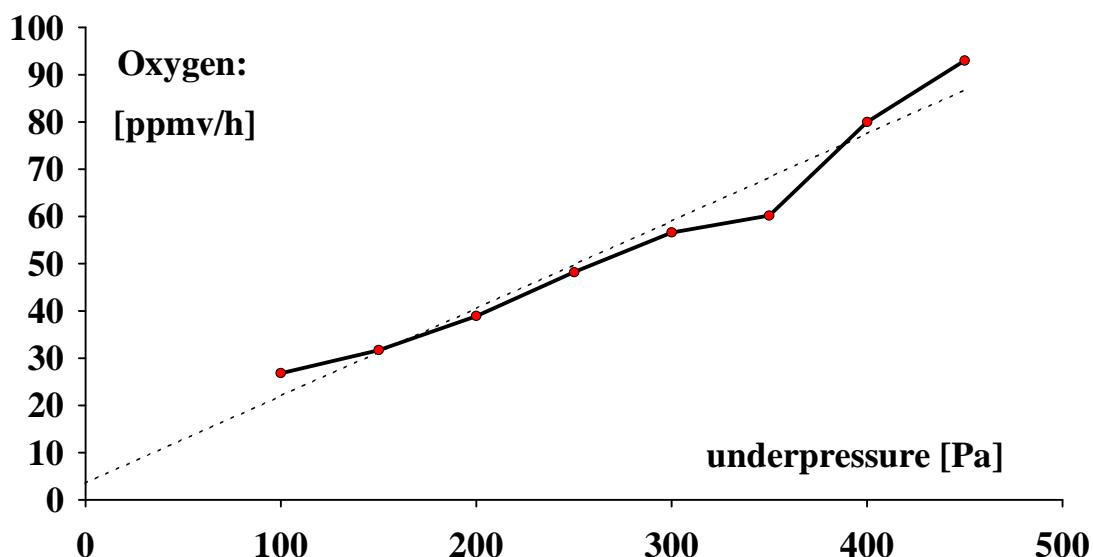
The influx of air is proportional to the underpressure inside the box. If the leak rate of 150 lusec was measured at an underpressure of 10 mbar, then operating the box at an underpressure of 1,5 mbar would give rise to an hourly increase of the concentration of oxygen of merely 15% of the value computed above, i.e.,

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\* Torr is a unit of pressure. 1 Torr = 133,3 Pa.

$$\frac{dc}{dt} = \frac{1,5 \text{ mbar}}{10 \text{ mbar}} \cdot 203 \frac{\text{ppmv}}{\text{h}} = 30.45 \frac{\text{ppmv}}{\text{h}} \quad (9-5)$$

Proper sealing of boxes is crucial. [P. Senn, 1992]. Some materials used for sealing deteriorate after a short period of time due to oxidation and the effects of exposure to mechanical vibrations and chemicals. Fig. 9-1 shows how the rate of increase of the concentration of oxygen increases with increasing underpressure. The experimental data have been obtained from a box with a volume of 630 liters. The dashed line is for a linear regression computed from the experimental data. The rate of leakage of a glovebox may fluctuate considerably, possibly due to variations in temperature.



**Fig. 9-1.** Rates of increase of the concentration of oxygen inside an idle glovebox as a function of underpressure.

The damage most frequently incurred to gloveboxes is the puncturing or tearing of the hand portion of the gloves. Schrock and Liu [Schrock & Liu, 1987] describe a procedure for replacing the hand portion of long-sleeved gloves.

## 10. The Rate of Influx of Contaminants by Permeation

The influx of contaminants by permeation takes place mainly through the gloves and to a lesser extent through the rubber seals of the windows and feedthroughs. There are four types of materials used for the manufacture of gloves with vastly different permeability coefficients

$$Q_A = \left( \frac{\Delta V}{\Delta t \Delta p} \right) \left( \frac{cm^2 \cdot Torr \cdot s}{N cm^3 \cdot cm} \right) \quad (10-1)^*$$

	<b>Butyl</b>	<b>Chloroprene**</b>	<b>Hypalon</b> Ø	<b>Viton</b> Ø
Oxygen (O <sub>2</sub> )	0.13 · 10 <sup>-10</sup>	0.4 · 10 <sup>-10</sup>	0.28 · 10 <sup>-10</sup>	0.15 · 10 <sup>-10</sup>
Water (H <sub>2</sub> O)	20.00 · 10 <sup>-10</sup>	180.00 · 10 <sup>-10</sup>	120.00 · 10 <sup>-10</sup>	5.20 · 10 <sup>-10</sup>
Nitrogen (N <sub>2</sub> )	0.03 · 10 <sup>-10</sup>	0.1 · 10 <sup>-10</sup>	0.1 · 10 <sup>-10</sup>	0.03 · 10 <sup>-10</sup>
Carbon dioxide (CO <sub>2</sub> )	0.52 · 10 <sup>-10</sup>	2.50 · 10 <sup>-10</sup>	2.08 · 10 <sup>-10</sup>	0.78 · 10 <sup>-10</sup>

where  $\Delta V$  denotes the volume of gas passing through a membrane of area  $A$  and thickness  $\Delta x$  per time  $\Delta t$  for the gradient of the partial pressure,  $\Delta p$ , across the barrier. The rate of permeation through a membrane of thickness  $\Delta x$  and area  $A$  depends on the difference of partial pressure,  $\Delta p$ , on both sides of the membrane as follows:

$$\frac{\Delta V}{\Delta t} = Q_A A \left( \frac{\Delta p}{\Delta x} \right) \left( \frac{N cm^3 \cdot cm}{cm^2 \cdot Torr \cdot s} \right) \quad (10-2)$$

The above data were measured from membranes of the corresponding elastomer at room temperature (20°C) [Jung Gummitechnik, GmbH]. Many gloves are manufactured from layers of the above common types of rubber. For example gloves made from butyl rubber are difficult to manufacture and uncomfortable to wear. For this reason butyl gloves frequently are laminated with Chloroprene. The thickness of most gloves being used varies between 0.4 and 0.8 mm. The best performances with respect to permeability are obtained with gloves made from *Butyl* and *Viton* [Barton, 1963]. Some manufactures offer gloves with layers of lead sandwiched between two layers of rubber for radiation shielding. In the selection of gloves used for working with radioactive materials there is obviously a tradeoff between radiation shielding and tactile feel. Besides permeability and comfort of wear there may be additional criteria for judging the suitability of various types of gloves [Barton, 1963]. In some cases the resistance to high temperatures, mechanical damage and aggressive chemicals may be of considerable importance. In addition, gloves made from latex are used for manipulations where greater touch sensitivity is needed. There may be some problems with hand irritation when wearing latex gloves. This problem may be overcome with a barrier foam applied to the hands before wearing the gloves or by wearing silk liner gloves underneath the latex gloves. There is also evidence that significant quantities of cytotoxins can permeate through thin gloves [Hart, 1989].

Some users operate their boxes using gloves with only sleeves, thereby permitting the handling of items inside the box with bare hands. Some boxes are equipped with an iris

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\* Torr is a unit of pressure. 1 Torr = 133,3 Pa.

\*\* Chloroprene is often referred to as Neoprene.

diaphragm, mainly for the transfer of elongated items which do not fit in the antechamber. In most of these boxes the iris diaphragm can be closed from the inside with a cover.

The effect of the relatively high permeability of the elastomers of the gloves can be reduced to some extent by flushing with inert gas [Billing *et al.*, 1973, Figs. 3 and 4; White & Smith, 1962, chap 11]. This can be done simply by using three gloves on each hand, the outermost glove being a normal rubber glove and the innermost glove a rubber glove with a gas inlet and outlet. An additional pair of gloves, made of a suitable fabric, is sandwiched between the rubber gloves, thereby serving as a separator between the rubber gloves. This technique is also useful for protecting personnel working at boxes, for example if there is Tritium or tritiated water inside the box. Some manufacturers offer gloves with rubber tubing for flushing the interior of the gloves with inert gas. Bennellick [Walton, 1958, p 13] claims that the service life of rubber gloves can be extended by covering them such that they are protected from the attack of gaseous reagents.

For boxes operated at overpressure the rate of influx of contaminants can be computed relatively accurately by an expression as follows:

$$\frac{dc}{dt} = \frac{gG + wW + b}{V_B} \left( \frac{1000 \text{ liter} \cdot \text{ppmv}}{h} \right) \quad (10-3)$$

where  $G$  and  $W$  denote the number of gloves and windows respectively. The parameter  $g$  depends on the type of gloves being used and the parameter  $w$  depends on the type of material used for the windows and their sealings. The windows are usually manufactured of safety glass, Macrolon or Lexan. The parameter  $b$  depends on the material of the walls of the box and of fittings and tubing. For a box of reasonable quality the three parameters  $g$ ,  $w$  and  $b$  should not exceed a value of 3. For a given box the influx of impurities through leaks is proportional to the underpressure applied. If the box is operated at an overpressure the influx of impurities through leaks is negligible in general. However, the rate of influx through leaks is much less predictable than the rate of permeation.

The influx of oxygen and water vapor by permeation through the gloves limits their lowest possible concentration in boxes with gas purification systems with a rate of circulation of around 20 box volumes per hour to not much less than 1 ppmv. This limitation also affects the design of gloveboxes. Excessive demands on the tightness of seals and tubing often don't make much sense.

The gloves must be fastened securely to the glove ports. This can be done with two or more o-rings made of rubber. The use of metal strips for this purpose is not necessarily advantageous because the rubber of the gloves tends to deteriorate and oxidize fastest at those locations which are subjected to mechanical stress.

Users of gloveboxes with inert atmospheres sometimes observe that the concentrations of impurities follow diurnal cycles. This is due mainly to variations in temperature. Permeability coefficients for contaminants such as oxygen and water vapor depend strongly

on temperature [Hwang, Choi & Kammermeyer, 1974]. Furthermore, the efficiency for the adsorption of contaminants by the adsorbent also depends on temperature. The adsorption of oxygen increases with increasing temperature, thereby compensating to some extent for the increased rate of permeation. However, the rate of adsorption of water vapor by molecular sieves decreases with increasing temperature. For this reason it may be rather difficult to maintain low levels of humidity at elevated temperatures. Perspiration and body heat can increase by a factor up to ten the rate of penetration of water vapor through the gloves[Ayers, Mayfield & Schmitt, 1960]. The permeation of water vapor through gloves can be reduced somewhat by wearing surgical gloves underneath the gloves of the glovebox [Johnson, 1960].

## **11. Transfer of Items through the Antechamber**

There are many techniques for transferring items in and out of a box. In the nuclear industries techniques with heat sealing of polyethylene bags are widely used. In safety cabinets of class III the transfer of items frequently takes place via a "dunk tank" filled with disinfectant. With conventional air locks there are basically two methods of using antechambers for the transfer of items. With the first method items to be transferred are simply placed into the antechamber which thereafter is being flushed. In this case the discussion in chap 2 on flushing a box largely applies. However, many items contain cavities where air gets trapped. The trapped air will later be released slowly, thereby contaminating the atmosphere inside the box over an extended period of time. For this reason the use of an evacuable antechamber is to be preferred. A prerequisite of course is that the items to be transferred can be exposed to vacuum. Many items retain most of their moisture while being transferred even if exposed to vacuum. In order to speed up the drying process some evacuable vacuum chambers can be heated. The heat being applied also speeds up the outgassing of adsorbed gases. Billing *et al.* [Billing *et al.*, 1973, pp 163-164] report a technique where items contaminated with tritium are exposed to steam prior to outgassing.

Let us assume that an antechamber with volume  $V_A$  which can be evacuated down to a pressure of  $p_f$  is attached to a box with volume  $V_B$ . The evacuation proceeds in  $N$  cycles involving evacuation of the antechamber followed by flooding with inert gas. Let  $\varsigma$  denote the ratio of the final pressure  $p_f$  after evacuation and the pressure  $p_o$  after flooding, i.e.,  $\varsigma = p_f / p_o$ . Let us assume that the inert gas used for flooding the antechamber has the same concentrations of impurities as the inert gas in the box. For the rise in concentration of contaminants after opening the inner door of the antechamber the following holds:

$$\Delta c \geq \frac{V_A}{V_A + V_B} \left( \frac{c_{initial}}{\varsigma^N} \right) \quad (11-1)$$

where  $c_{\text{initial}}$  is the concentration of the corresponding contaminant in the air. The above formula holds only approximately. Since  $V_B \gg V_A$  in general, the contamination of the box due to a transfer through the antechamber is expected to be roughly proportional to the size of the antechamber. The main reasons that the model with repeated dilution of contaminants fails are expected to be the following:

- Items placed into the vacuum chamber release adsorbed contaminants by outgassing or by slow release from cavities through leaks.
- During evacuation of the antechamber there is an influx of contaminants through leaks in the antechamber.
- The assumption that the inert gas used for flooding the antechamber has the same concentrations of contaminants as the inert gas inside the box may not be valid.

By flooding with only a portion of the gas needed to raise the pressure inside the antechamber to atmospheric pressure significant savings in inert gas can be attained. Tests at *Mound Laboratory* [Billing *et al.*, 1973, p 134] showed that 5 evacuations with back-fillings to  $\frac{1}{3}$  atmosphere were roughly twice as effective as the standard procedure with three cycles. The former procedure also needed one third less inert gas. The start-up of evacuable gloveboxes is quite similar to transfers via antechambers except that the items being "transferred" can be left in place. The author used with considerable success a technique for putting evacuable gloveboxes into operation with only one evacuation and then rinsing with a small gas flow at low pressure with the vacuum pump running. It turned out that it is quite important that the box was flushed quickly after the final evacuation in order to reduce leakage which is proportional to the differential pressure. Some manufacturers of gloveboxes sell inserts for the antechamber which allow a reduction in the amount of gas needed for flooding. For an antechamber of cylindrical shape these inserts have typically the shape of cylinders cut in half along the cylinder axis with each half cylinder half as long as the antechamber. The minimum space available is then one fourth of the volume of the antechamber.

For the evacuation of an antechamber with a vacuum pump with pumping speed  $s$  the pressure changes with time as follows:

$$p(t) = p_s + (p_o - p_s) e^{-\eta t} \quad (11-2)$$

where  $p_o$  and  $p_s$  are the initial and the base pressure of the vessel respectively and  $\eta = s / V_A$ . Assuming  $p_s = 0$ , we obtain for the time needed for  $N$  evacuations from an initial pressure  $p_o$  to a final pressure  $p_f$  the following:

$$t = \frac{N}{\eta} \ln \frac{p_o}{p_f} \quad (11-3)$$

From the above it is readily apparent that by reducing the initial pressure  $p_o$  by a factor of 3 will have the additional advantage that the time needed for the evacuations is reduced by a few percent. Good results have also been obtained by slightly opening the valve for inert gas after the final pressure had been reached in an evacuation thereby flushing the antechamber with a small flux of inert gas for a short period of time.

## **12. Using Inert Gas of "Low" Purity**

Some users believe that the gas used for operating a box with a purification system has to be at least as pure as the desired working atmosphere inside the box. However, gases of high purity are rather expensive. The actual maximum contaminations of gases are usually much lower than those which the manufacturers guarantee. When a box is in use the average consumption of inert gas does not exceed 2 liters per minute in general. Let us assume that the manufacturer guarantees that the concentration of a given impurity does not exceed 5 ppmv. Taking an influx of 120 liters per hour this would amount to

$$\dot{V}_i < \frac{5}{1,000,000} 120 \text{ liters/h} = 0.6 \text{ cm}^3/\text{h} \quad (12-1)$$

A "good" box with this volume usually has an influx of oxygen, mainly by diffusion through the gloves, which is 2 - 4 times larger than what has been computed above for the "worst possible case". The inert gas used for pressure regulation of the box is usually injected at the inlet of the adsorbent column, such that only a few percent of the impurity actually get inside the box. From this we would infer that the expense for inert gas of high purity cannot be justified in most cases.

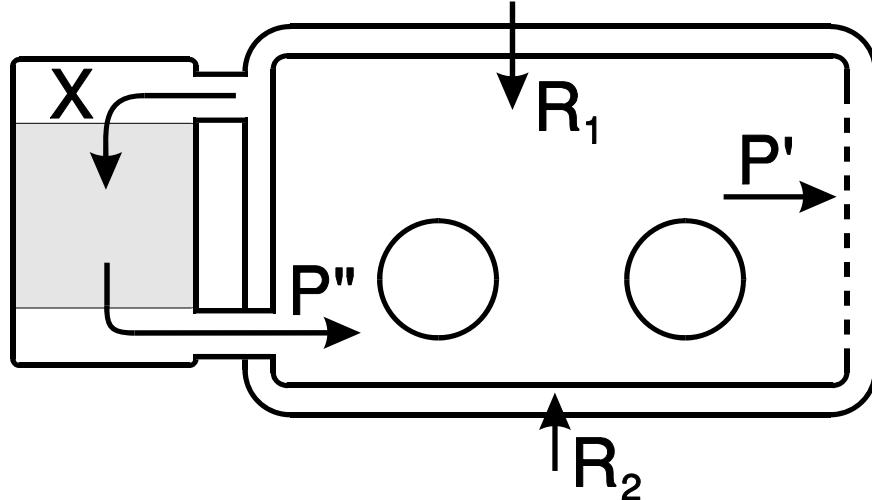
## **13. Double-Walled Containment**

Fig. 13-1 depicts schematically a box with two walls and one purification system. Let  $c$  be the concentration of a contaminant inside the box and  $z$  the (average) concentration in the space in between the two walls. The space inside the inner wall is referred to as the "*secondary containment*". For the model shown in Fig. 13-1 the following applies:

$$(1 - \chi)V_B \frac{dc}{dt} = R_1 + P'' - P' = R_1 - \dot{V}c + (1-K)\dot{V}z \quad (13-1)$$

$$\chi V_B \frac{dz}{dt} = R_2 + P' - X = R_2 + \dot{V}c - \dot{V}z \quad (13-2)$$

where  $\chi$  is the ratio of the volume  $V_B$  of the box which is located in between the walls.



**Fig. 13-1.** Schematic illustration of a double-walled containment.

From (13-1) and (13-2) the following is obtained for the concentrations at equilibrium:

$$c_{eq} = \frac{R_1 + (1 - K) R_2}{K} \quad (13-3)$$

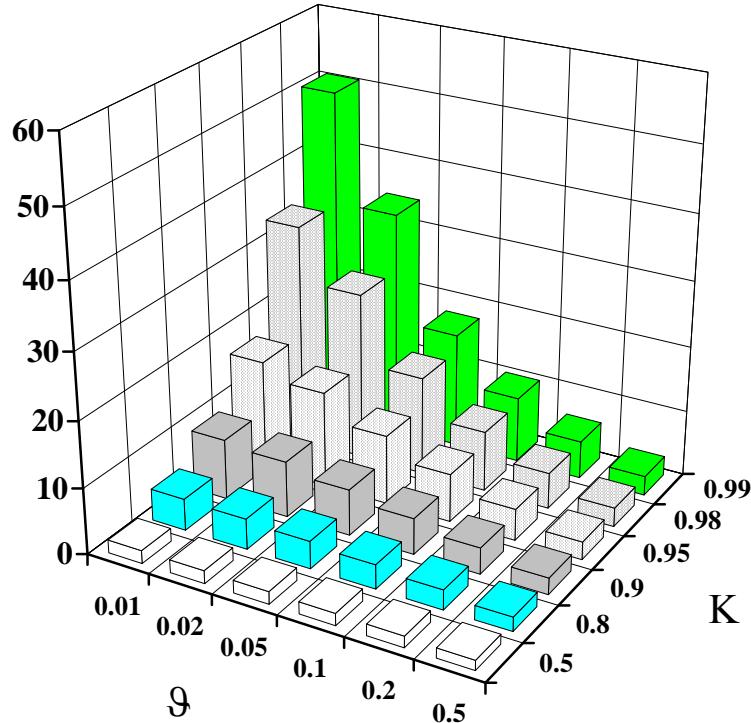
$$z_{eq} = \frac{R_1 + R_2}{K} \quad (13-4)$$

From this it is apparent that the concentration  $c$  of the impurity can be kept low by maintaining a high rate  $\dot{V}$  of circulation as is the case in a containment with one wall. A further reduction can be achieved by virtually eliminating the flux of impurity into the secondary containment and using an adsorber which eliminates a high portion of the impurity from the inert gas, i.e.,  $R_1 \rightarrow 0$  and  $K \rightarrow 1$  respectively. Notice that the concentration of the contaminant between the walls is the same as its concentration in a conventional box with the same purification system and an influx of impurity  $R = R_1 + R_2$ . For the ratio of  $z_{eq}$  and  $c_{eq}$  the following is obtained:

$$\frac{z_{eq}}{c_{eq}} = \frac{1 + \vartheta}{1 + \vartheta - K} \quad (13-5)$$

where  $\vartheta = R_1 / R_2$ . Fig. 13-2 shows a graph of the ratio  $z_{eq} / c_{eq}$  for different values of  $\vartheta$  and  $K$ . The ratio  $z_{eq} / c_{eq}$  also indicates how much better the double-walled containment performs when compared to an analogous system without the secondary containment. From

Fig. 13-2 it is readily apparent that in order to boost the performance of a box with double-wall containment by a factor of ten, using the design shown in Fig. 13-1 the ratio  $\vartheta = R_1/R_2$  should not exceed 0.1 and more than 90% of the contamination should be retained in the adsorber, i.e.,  $K > 0.9$ .



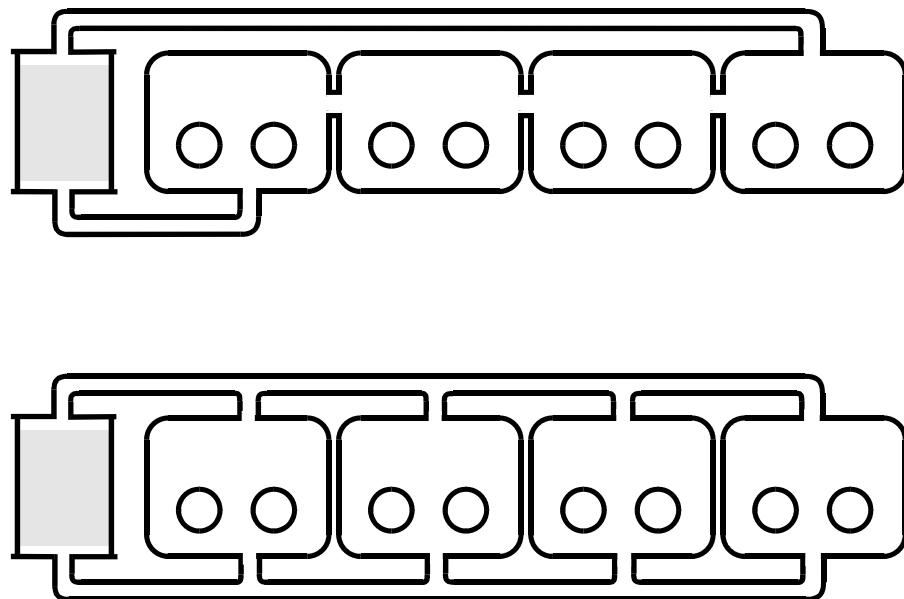
**Fig. 13-2.** Ratio of the concentrations in the primary and secondary containment of a double-walled containment.

If the adsorbent column is lengthened in order to increase  $K$ , then the rate of gas flow  $\dot{V}$  would inevitably decrease. It therefore appears that the increase of performance using a double-walled containment as depicted in Fig. 13-1 is not quite as dramatic as might be expected. However, the secondary containment and the space in between the wall can be operated with separate gas purification systems. It is also possible to continuously flush the space in between the walls, either with gas drawn from the secondary containment or with fresh gas from the gas supply. This kind of design makes it possible to operate the secondary containment at an overpressure and the space between the walls at an underpressure or vice versa. The system illustrated in Fig. 13-1 could also be operated such that purified gas gets from the exhaust of the adsorbent column into the secondary containment via the space between the walls. This mode of operation would be appropriate when working with hazardous materials.

Double-skinned enclosures frequently fail to fulfill the expectations of their manufacturers, mainly because it is rather difficult to make them leak-tight. For enclosures used for handling hazardous materials a secondary containment can be effected by installing the box inside a ventilated room with a window with holes at the glove ports [Walton, 1958, pp 90-96].

## 14. Glovebox Lines

Gloveboxes in glovebox lines with a single gas purification system are usually arranged such that the gas circulates through the boxes and the adsorbent bed as illustrated in the lower part of Fig. 14-1. This kind of assembly will be referred to as *connexion in parallel* whereas a configuration as illustrated in the upper part of Fig. 14-1 will be referred to as *connexion in series*.



**Fig. 14-1.** Illustration of two configurations of a glovebox line.

For a connexion of  $n$  glove in parallel a system of  $n$  linear differential equations as follows is obtained:

$$\dot{V}_j c_j + V_j \dot{c}_j = \left( \frac{1 - K}{V_j} \right) \sum_{i=1}^n \dot{V}_i c_i + R_j, \quad j = 1, 2, \dots, n \quad (14-1)$$

The rates of gas flow  $\dot{V}_1, \dot{V}_2, \dot{V}_3, \dot{V}_4, \dots, \dot{V}_n$  are for the individual boxes with volumes  $V_1, V_2, V_3, \dots, V_n$ , and  $\dot{V} = \dot{V}_1 + \dot{V}_2 + \dot{V}_3 + \dots + \dot{V}_n$ . At equilibrium we have  $\dot{c}_1 = \dot{c}_2 = \dot{c}_3 = \dots = \dot{c}_n = 0$  and the following is obtained for the equilibrium concentrations:

$$(c_j)_{eq} = \left( \frac{R_j}{K} \right) + \left( \frac{1}{K} - 1 \right) \left( \frac{R}{V} \right), \quad j = 1, 2, \dots, n \quad (14-2)$$

where  $R$  is the total rate of permeation into the containment with  $n$  boxes, i.e.,  $R = R_1 + R_2 + R_3 + \dots + R_n$ .

Consider the following example:

In a configuration of 5 equal boxes with equal rates of gas flow one of the boxes is defective. The rate of permeation is 8 times higher for the defective box than for the others. The retention ratio of the adsorbent column is 0.8.

For this example we obtain from (14-2) that the equilibrium concentrations are increased by a factor of 6.88 and 1.28 in the defective box and the other boxes respectively, when compared with the values which would be obtained without the defective box.

Taking  $R_1 = R_2 = R_3 = \dots = R_n = 0$  the differential equations in (14-1) can be rearranged to give the following:

$$-\dot{V}_j c_j = V_j \dot{c}_j + \left(\frac{1 - K}{K}\right) \dot{V}_j \sum_{i=1}^n V_i \dot{c}_i, j = 1, 2, \dots, n \quad (14-3)$$

By integrating from 0 to  $\infty$  we can obtain the exposures in the different boxes resulting from a "contamination" with the initial concentrations  $c_1(0), c_2(0), \dots, c_n(0)$  at  $t = 0$ .

$$C_j(\infty) = \int_0^\infty c_j(t) dt = \frac{(V_j c_j(0))}{\left(\frac{1 - K}{K}\right)} + \left(\frac{1 - K}{K}\right) \sum_{i=1}^n V_i c_i(0), j = 1, 2, \dots, n \quad (14-4)$$

The term *exposure* has been defined in (4-3).

Let us consider the following example:

In a glovebox line with five equal boxes with equal rates of gas flow a "contamination" takes place in one of the boxes. The retention ratio of the recirculatory gas purification system is 0.8. How are the different boxes affected by the contamination?

For this example we obtain from (14-4) that the exposure due to the contamination is 21 times stronger in the box in which the contamination took place than in the other boxes.

For  $n$  boxes with volumes  $V_1, V_2, V_3, \dots, V_n$  in series we obtain the following set of differential equations:

$$\dot{V} c_1 + V_1 \dot{c}_1 = (1 - K) \dot{V} c_n + R_1 \quad (14-5)$$

$$\dot{V} c_j + V_j \dot{c}_j = \dot{V} c_{j-1} + R_j \quad (14-6)$$

where  $j = 2, 3, \dots, n$ . It is assumed that the box with index "1" is the first box in the line, i.e., the gas flow is as depicted in (2-4). The rate of gas flow is  $\dot{V}$  for all boxes. At

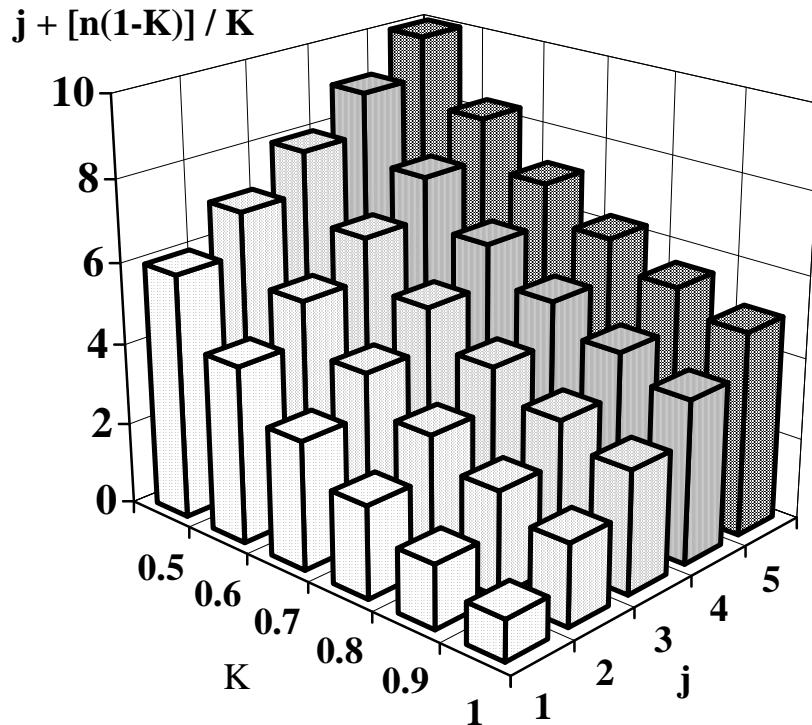
equilibrium we have  $\dot{c}_1 = \dot{c}_2 = \dot{c}_4 = \dots = \dot{c}_n = 0$  and for the concentrations at equilibrium we obtain from (14-5) and (14-6) the following:

$$(c_j)_{eq} = \frac{1}{R} \left( \left( \frac{1}{K} - 1 \right) R + \sum_{i=1}^j R_i \right), j = 1, 2, \dots, n \quad (14-7)$$

where  $R = R_1 + R_2 + R_3 + \dots + R_n$ . For a glovebox line with  $n$  equal boxes with equal rates of permeation  $R_o$  we can write

$$(c_j)_{eq} = \frac{R_o}{n} \left( n \left( \frac{1}{K} - 1 \right) + j \right), j = 1, 2, \dots, n \quad (14-8)$$

where  $j$  denotes the number of the box. Fig. 14-2 shows the quantity in the brackets in (14-8) as a function of the retention ratio  $K$  of the adsorbent column for  $n = 5$ . The parameters  $j$  and  $K$  denote the number of the box and the retention ratio of the adsorbent column respectively.



**Fig. 14-2.** Relative equilibrium concentrations of a contaminant in five equal boxes connected in series.

In order to obtain from the quantities shown the corresponding equilibrium concentrations  $R_o$  they would have to be multiplied by a factor of  $\frac{R_o}{n}$  as indicated in (14-8). If  $K = 0.8$  the equilibrium concentration in a single box would be greater by a factor of 1.25 than for  $K = 1$ . However, according to (14-8), for the glovebox line with  $n = 5$  the equilibrium concentrations in the first box would differ by a factor of 2.25 for adsorbent columns with  $K$

$= 0.8$  and  $K = 1$ . The boxes most heavily affected by the incomplete removal of contaminants in the adsorbent column are the first boxes in the line. The last box in the glovebox line behaves as though the entire influx of contaminants took place in this box.

Next let us look at what happens during "contaminations" of the boxes with initial concentrations of the contaminant in the boxes of  $c_1(0), c_2(0), \dots, c_n(0)$ . As before we assume again that the ordinary influx of contaminants is irrelevant for the duration of the "contamination", i.e.,  $R_1 = R_2 = R_3 = \dots = R_n = 0$ . Following a procedure largely analogous to the one used for the gloveboxes line with connexion in parallel we obtain the following for the exposure:

$$C_j(\infty) = \int_0^{\infty} c_j(t) dt = \frac{1}{K} \left( \sum_{i=1}^j V_i c_i(0) + (1 - K) \sum_{i=j+1}^n V_i c_i(0) \right), j = 1, 2, \dots, n \quad (14-9)$$

According to (14-9) a contamination taking place in the  $j$ -th box affects all boxes downstream as though the contamination took place inside those boxes whereas the boxes located upstream would be affected less by a factor of  $1 - K$ . Connexion in series of gloveboxes is usually less expensive than connexion in parallel. Its main drawback is that contaminations can propagate downstream in the glovebox line.

Let us now look at two configurations of a glovebox line with four boxes with equal volumes  $V_o$  operated with a circulatory gas purification system. In one case the gas flow is separated into four pipes leading to the individual boxes. Let  $R_o$  denote one fourth of the gas flow passing the adsorbent column. The rate of influx of contaminant into each box is the same, i.e.,  $R_1 = R_2 = R_3 = R_4 = R_o$ . Assuming  $K = 0.8$  the following is obtained from (14-2) and (14-8) for the equilibrium concentrations:

$$\text{Connexion in parallel: } (c_1)_{eq} = (c_2)_{eq} = (c_3)_{eq} = (c_4)_{eq} = \frac{R_o}{K} = 1.25 \frac{R_o}{K} \quad (14-10)$$

$$\text{Connexion in series: } (c_1)_{eq} = \left(1 - \frac{3}{4} K\right) \frac{R_o}{K} = 0.5 \frac{R_o}{K} \quad (14-11)$$

$$(c_2)_{eq} = \left(1 - \frac{1}{2} K\right) \frac{R_o}{K} = 0.75 \frac{R_o}{K} \quad (14-12)$$

$$(c_3)_{eq} = \left(1 - \frac{1}{4} K\right) \frac{R_o}{K} = \frac{R_o}{K} \quad (14-13)$$

$$(c_4)_{eq} = \frac{R_o}{K} = 1.25 \frac{R_o}{K} \quad (14-14)$$

From this we can see that for connexion in series none of the boxes has a higher equilibrium concentration of the contaminant than the boxes connected in parallel. As indicated previously this advantage is overcompensated in general by the disadvantage that in glovebox lines with connexion in series contaminations will spread to boxes downstream. Let us now

look at the exposures in the different boxes due to a contamination with the initial concentration  $c_j(0) = c_o$  in one of the four boxes. Again using the same example as above we obtain from (14-4) and (14-9) the following:

Connexion in parallel:

$$\text{Box with the contamination: } C_j(\infty) = \left( \frac{1 + 3K}{4K} \right) \frac{V_o c_o}{V_o c_o} = 1.0625 \frac{V_o c_o}{V_o c_o} \quad (14-15)$$

$$\text{Other boxes: } C_i(\infty) = \left( \frac{1 - K}{4K} \right) \frac{V_o c_o}{V_o c_o} = 0.0625 \frac{V_o c_o}{V_o c_o} \quad (14-16)$$

$$C_1(\infty) + C_2(\infty) + C_3(\infty) + C_4(\infty) = 1.25 \frac{V_o c_o}{V_o c_o} \quad (14-17)$$

Connexion in Series:

$$c_o = c_1(0): \quad C_1(\infty) = C_2(\infty) = C_3(\infty) = C_4(\infty) = \frac{V_o c_o}{4K} = 0.3125 \frac{V_o c_o}{V_o c_o} \quad (14-18)$$

$$C_1(\infty) + C_2(\infty) + C_3(\infty) + C_4(\infty) = 1.25 \frac{V_o c_o}{V_o c_o} \quad (14-19)$$

$$c_o = c_2(0): \quad C_1(\infty) = (1 - K) \frac{V_o c_o}{4K} = 0.0625 \frac{V_o c_o}{V_o c_o} \quad (14-20)$$

$$C_2(\infty) = C_3(\infty) = C_4(\infty) = \frac{V_o c_o}{4K} = 0.3125 \frac{V_o c_o}{V_o c_o} \quad (14-21)$$

$$C_1(\infty) + C_2(\infty) + C_3(\infty) + C_4(\infty) = \frac{V_o c_o}{V_o c_o} \quad (14-22)$$

$$c_o = c_3(0): \quad C_1(\infty) = C_2(\infty) = (1 - K) \frac{V_o c_o}{4K} = 0.0625 \frac{V_o c_o}{V_o c_o} \quad (14-23)$$

$$C_3(\infty) = C_4(\infty) = \frac{V_o c_o}{4K} = 0.3125 \frac{V_o c_o}{V_o c_o} \quad (14-24)$$

$$C_1(\infty) + C_2(\infty) + C_3(\infty) + C_4(\infty) = 0.75 \frac{V_o c_o}{V_o c_o} \quad (14-25)$$

$$c_o = c_4(0): \quad C_1(\infty) = C_2(\infty) = C_3(\infty) = (1 - K) \frac{V_o c_o}{4K} = 0.0625 \frac{V_o c_o}{V_o c_o} \quad (14-26)$$

$$C_4(\infty) = \frac{V_o c_o}{4K} = 0.3125 \frac{V_o c_o}{V_o c_o} \quad (14-27)$$

$$C_1(\infty) + C_2(\infty) + C_3(\infty) + C_4(\infty) = 0.5 \frac{V_o c_o}{V_o c_o} \quad (14-28)$$

Due to the greater rate of gas flow the duration of contaminations are considerably shorter if the boxes are connected in series. Connexions in series are useful in cases where in a glovebox line a "flow" of items being processed starts at one end of the glovebox line, with consecutive stages of processing requiring working atmosphere with decreasing levels of contamination. Besides the pair of patterns for connecting boxes considered here a wide

variety of modified designs are possible of course. Interconnected gloveboxes may also be serviced by more than one gas purification system.

## **15. The Optimum Design of Adsorbent columns**

The static pressure generated by a blower decreases roughly linearly with the rate of gas flow

$$\Delta p \approx \Delta p_o (1 - \cdot) \quad (15-1)$$

where  $\dot{V}_m$  is the maximum flow and  $\Delta p_o$  is the static pressure when there is no flow of gas. Frequently the static pressure of a blower depends considerably on whether the gas is pushed or "pulled" through the adsorbent column. When the gas is pulled through the adsorber the associated underpressure may cause additional leakage of contaminants. The average speed, v, of the gas through the adsorber depends on the applied pressure as follows [Wadden & Scheff, 1987]:

$$\Delta p \approx \Xi v^2 (h + h_o) \quad (15-2)$$

where  $h$  is the height of the adsorber. Let  $V_i$  be the interstitial volume of the adsorbent bed. Then the following applies:

$$\dot{V} \approx v \left( \frac{V_i}{h} \right) \quad (15-3)$$

From (15-1) and (15-2) we obtain

$$KV \approx \dot{V} \left( 1 - e^{-\beta V_i / \dot{V}} \right) \quad (15-4)$$

where

$$\dot{V} = \left( \frac{\sqrt{1 + 2B} - 1}{B} \right) \dot{V}_m \quad (15-5)$$

$$B = \left( \frac{2\Xi h_o^3}{\Delta p_o V_i^2} \right) x^2 (x + 1) \quad (15-6)$$

and  $x = h/h_o$ .

Let us now look at a specific example. A blower with  $\dot{V}_m = 290 \text{ m}^3/\text{h}$  and  $\Delta p_o = 350 \text{ Pa}$  pushes nitrogen gas through adsorbent columns with constant volumes and variable

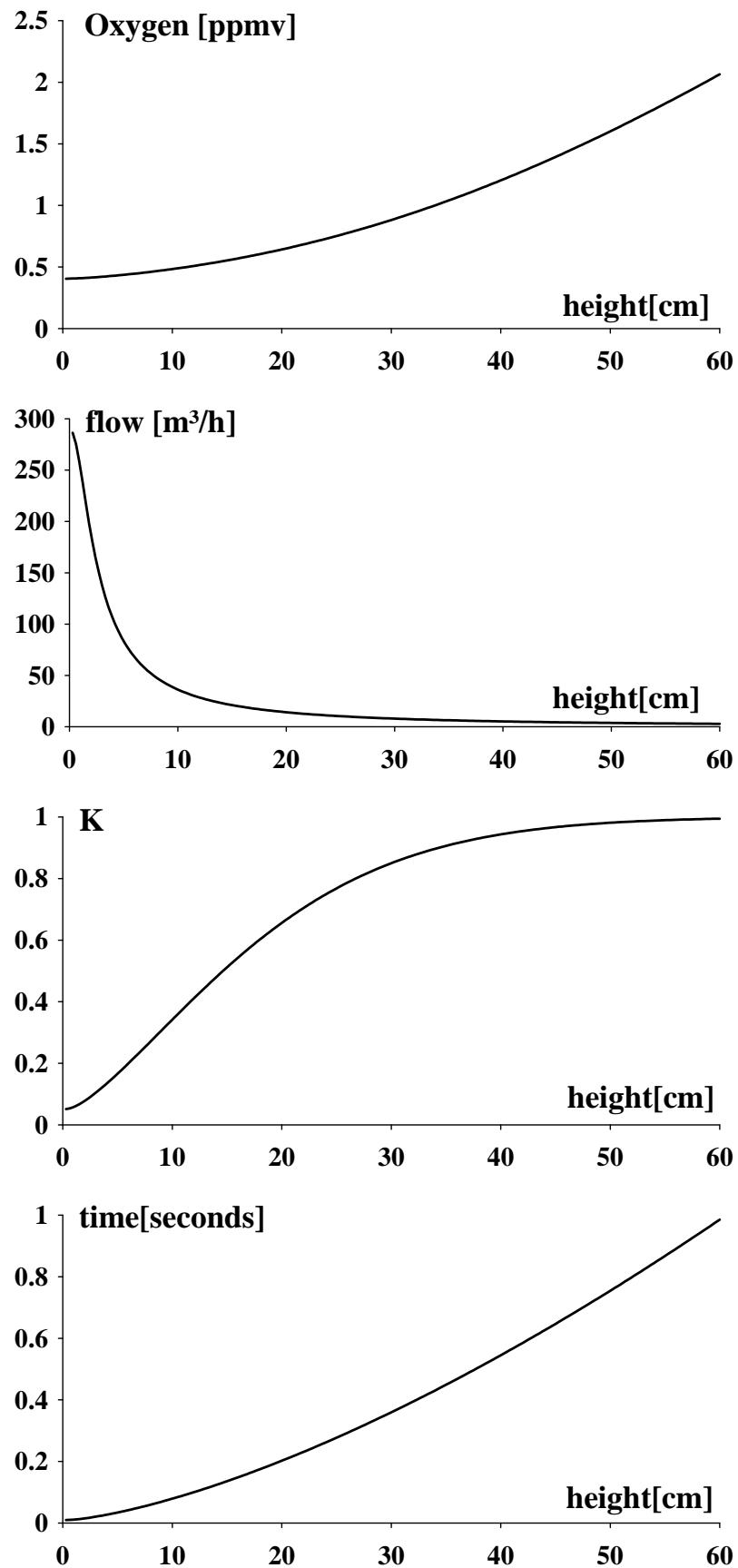
heights  $h$ . Let the interstitial volume of the columns be  $V_i = 800 \text{ cm}^3$ . For an actual adsorbent column with a height of 30 cm it was known that the blower generates a static pressure  $\Delta p$  of 340 Pa and the rate flow of gas was  $8\text{m}^3/\text{h}$ . Then estimating  $h_o = 3 \text{ cm}$  we obtain for  $\Xi$  from (15-2) and (15-3) the following:

$$\Xi = \left( \frac{340 \cdot 0.0008^2 \cdot 3600^2}{8^2 \cdot 0.3^2 \cdot 0.33} \right) \frac{\text{kg}}{\text{m}^4} = 1484 \frac{\text{kg}}{\text{m}^4} \quad (15-7)$$

At low concentrations the adsorber removed roughly 85% of the oxygen.

$$0.85 = \left( 1 - e^{-\beta \cdot V_i / \dot{V}} \right) = \left( 1 - e^{-\beta \cdot 0.0008 \cdot 3600s / 8} \right) \quad (15-8)$$

from which we obtain  $\beta = 5.27 \text{ s}^{-1}$ . Fig. 15-1 shows how the equilibrium concentrations of oxygen as a function of the height of the adsorbent column,  $h$ , would turn out for a box with a volume of 1070 liters for which the concentration of oxygen would increase at a rate of 6ppmv/h when the gas purification system is shut down. The column contained roughly 2 kg of molecular sieve and an equal mass of copper catalyst, whereof roughly 600 g were copper metal as a fine powder. The graph on top shows the computed equilibrium concentration of oxygen.



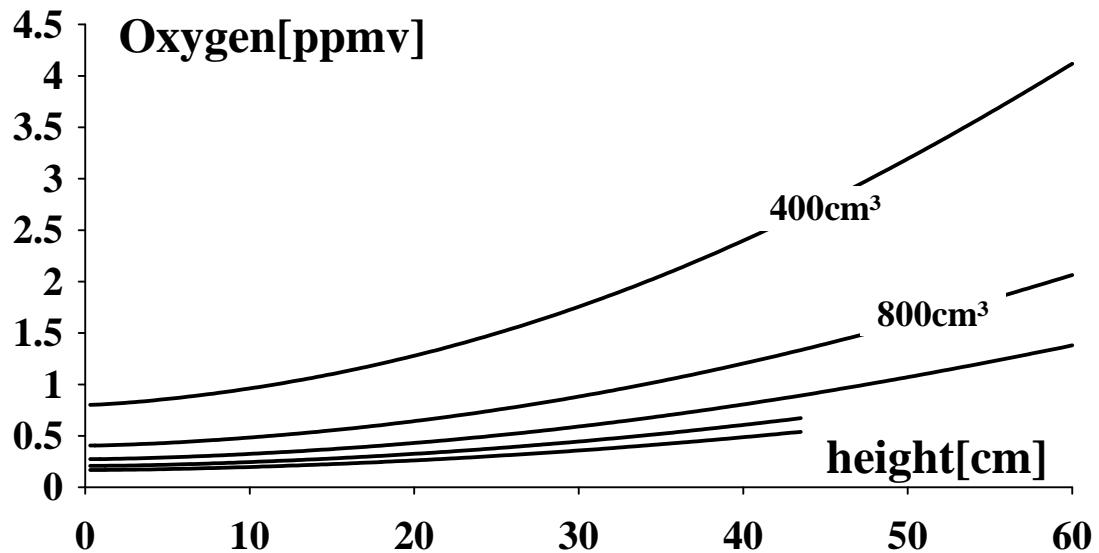
**Fig. 15-1.** Computed performance of a purification column as a function of its height.

The two graphs in the middle illustrate, that  $K$ , the probability of adsorption increases with increasing height of the column. This effect is offset, however, by a sharp drop in the rate of gas flow through the adsorber. It turns out that the optimum solution would be one where the adsorber is very short, i.e.,  $h \rightarrow 0$ . For short columns the present model would predict a reduction by a factor of 2 of  $c_{eq}$  when compared to the actual height of 30 cm. However, long columns are less susceptible than short columns to a reduction in efficiency due to a phenomenon referred as *channeling*. In the context of the present model the interstitial volume,  $V_i$ , is also a measure of the amount of adsorbent in the adsorbent column. The ratio  $V_i / V$  is a measure of the average time spent by a particle of contaminant in the adsorbent column. For this reason the "interstitial volume" used in the calculations is considerably smaller than the actual interstitial volume, which was estimated from bulk densities to be roughly  $1500 \text{ cm}^3$ , i.e., nearly twice as big as the value of  $800 \text{ cm}^3$  which has been used. In the adsorbent bed the flow of gas takes place mainly through the largest cavities. It is their combined volume that is relevant when estimating the interstitial volume for the present model.

For short columns the percentage of oxygen adsorbed becomes small. However, this effect is offset by the higher throughput of gas by the blower, which, for  $h \rightarrow 0$  approaches  $\dot{V}_m$ , the upper limit of the rate of gas flow, which in the present example equals  $290 \text{ m}^3/\text{h}$ . A high throughput can also be achieved by a reduction of the resistance against flow,  $\Xi$ , by using a loosely packed adsorbent bed. This will have an adverse effect on the value of  $\beta$  of course. The bottom part of Fig. 15-1 shows the average time spent by a particle of impurity in the adsorber which can be obtained as the ratio of interstitial volume and the rate of gas flow,  $\tau \approx V_i / \dot{V}$ .

Fig. 15-2 shows the equilibrium concentration of oxygen for various quantities of adsorbent, i.e., different values of  $V_i$ . In the context of the present mathematical model for the adsorbent column the interstitial volume of the adsorbent bed represents a measure of the amount of adsorbent in the adsorbent column. For the column actually used the interstitial volume was estimated to be around  $800 \text{ cm}^3$ . An interstitial volume of  $400 \text{ cm}^3$  would therefore correspond to an adsorbent bed of half its size. Five curves have been computed for adsorbent columns with interstitial volumes of  $400, 800, 1200, 1600$ , and  $2000 \text{ cm}^3$ . It turns out that  $c_{eq}$  is roughly inversely proportional to  $V_i$ .

The ultimate reason for the superior performance of "short" columns rests in the fact, that in short columns practically all of the adsorbent is exposed to gas with the level of contamination prevailing in the box, whereas in long columns a significant portion of the adsorbent remains practically idle, being shielded by upper layers of adsorbent. In view of this fact one might surmise, that the short column can maintain its level of performance for a much shorter time than a long column and that columns with equal quantities of adsorbent would become similar in performance after some time or that a long column might even perform better in a long run.

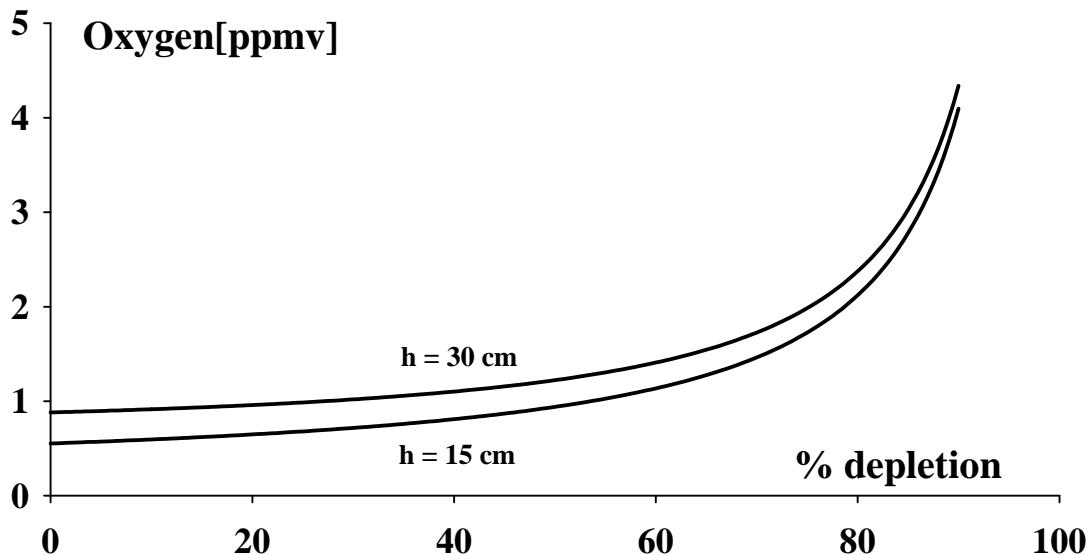


**Fig. 15-2.** The equilibrium concentration of oxygen as a function of the amount of adsorbent.

The half life time of a gaseous particle of contaminant is inversely proportional to the number of active sites. According to (5-4) the parameter  $\beta$  is therefore proportional to the remaining capacity of the adsorbent.

$$\beta = \beta_0 \left( \frac{\text{remaining capacity of the adsorbent}}{\text{original capacity of the adsorbent}} \right) \quad (15-9)$$

Fig. 15-3 shows for the example from the beginning of this chapter ( $R = 6 \text{ ppmv/h}$ ) the equilibrium concentration,  $c_{\text{eq}}$ , for oxygen as a function of the percentage of depletion for two adsorbent columns with equal volumes, but different heights of 15 and 30 cm. Fig. 15-3 reveals that the short column maintains its superior performance when compared to the column with twice its height. However, the difference between the performance of the two columns diminishes as the adsorbent is depleted. Fig. 15-3 also shows that the equilibrium concentration of oxygen rises sharply as the depletion passes 80%. Practical experiences with the system characterized in the beginning of this chapter agree with Fig. 15-3 inasmuch as the equilibrium concentration of oxygen could not be kept under 1 ppmv with a depletion of the adsorbent in excess of 40%.



**Fig. 15-3.** Equilibrium concentration of oxygen as a function of the percentage of depletion.

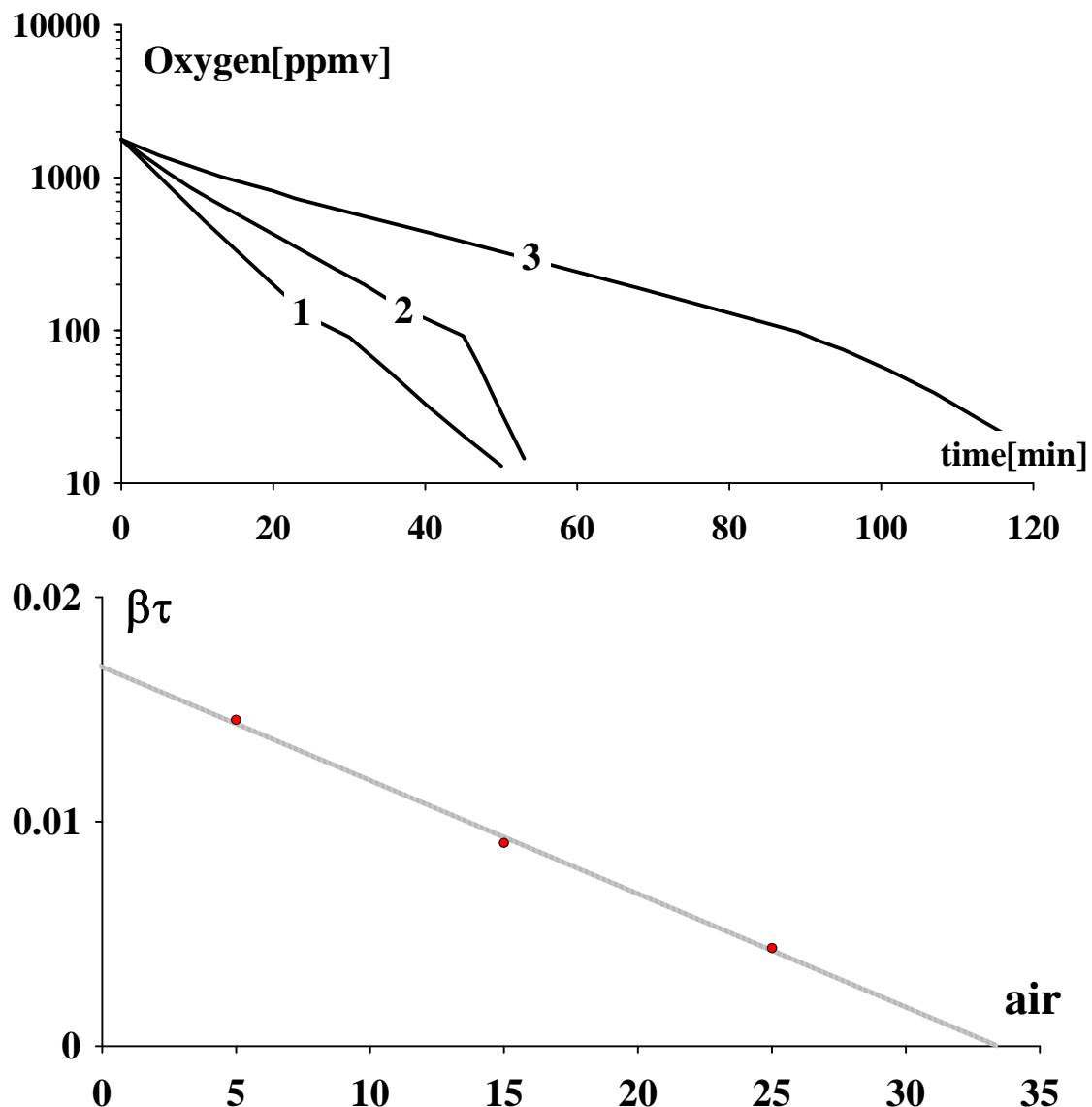
Fig. 15-4 shows purification curves of three successive injections of 10 liters of air into a box with a volume,  $V_B$ , of 1070 liters. From the initial slopes of the purification curves the quantities  $\beta\tau$  have been computed for each injection. The lower part illustrates the linear decrease of  $\beta\tau$  as a function of the volume of oxygen adsorbed or, equivalently, as a function of the percentage of depletion of the adsorbent column as proposed in (15-9). Using (7-2) the performance index  $\alpha$  was computed from the slope of the oxygen concentration at  $t = 0$  as follows:

$$\alpha = - \frac{d}{dt} \ln\left(\frac{c}{c(0)}\right) \quad (15-10)$$

from which the product  $\beta\tau$  was computed using (5-3) and  $\alpha = KV/V_B$  as follows:

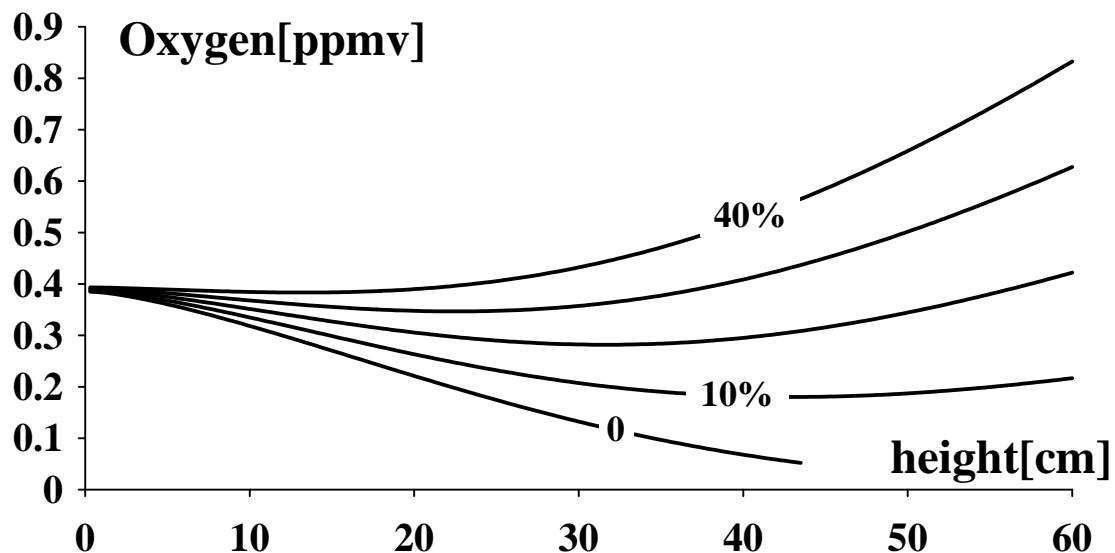
$$\beta\tau = - \ln\left(1 - \alpha \frac{V_B}{V}\right) \quad (15-11)$$

The lower part of Fig. 15-4 shows the product  $\beta\tau$  computed as a function of the volume of air injected. The parameter  $\tau$  should remain constant. The value of  $\beta$  decreases indeed linearly with the depletion of the adsorbent column as indicated in (15-9). The volumes of air injected have been taken at half of the volume injected in each portion of 10 liters, i.e., at 5, 15, and 25 liters of air. Fig. 15-4 indicates that the total adsorption capacity of the adsorbent column would be roughly 33 liters (where  $\beta$  becomes zero). This is in fair agreement to the manufacturer's specifications according to which the adsorption capacity of the column should be roughly equivalent to 40 liters of air. (In the present example there was also some uncertainty concerning the previous load on the adsorbent column).



**Fig. 15-4.** Purification curves following three injections of air into a box.

A short column is not necessarily the best solution for double-walled containments. For this type of system the effectual removal of contaminants in the adsorber is of greater importance than maintaining a high rate of circulation. Fig. 15-5 shows the computed equilibrium concentration of oxygen in the interior containment for the example discussed in the beginning of this chapter ( $V_B = 1070$  liters) for various ratios  $R_1/R_2$  with  $R_1 + R_2 \approx 6$  ppmv/h as a function of the height of the adsorbent column. The volume of the adsorbent column is kept constant and the total influx of oxygen is such that the concentration of oxygen would increase at a rate of 6 ppmv/h after shut-down of the gas purification system with a rate of gas flow of 8 m<sup>3</sup>/h. Five curves have been computed for  $\vartheta \cdot 100\% = 0, 10\%, 20\%, 30\%$ , and  $40\%$ , where  $\vartheta = R_1/R_2$ . It is readily apparent that for example for  $R_1/R_2 = 0.1$  a "long" adsorbent column with a length of approximately 40 cm length would be best.



**Fig. 15-5.** Computed equilibrium concentration of oxygen inside the secondary containment.

Usually the adsorbent bed should be regenerated or replaced when roughly 60% of its maximum adsorption capacity still remains. This is the case when

$$t \leq 0.4 \cdot 10^6 \left( \frac{V_{\max}}{\bar{\theta}} \right) \quad (15-12)$$

where  $V_{\max}$  denotes the maximum volume of gaseous contaminant the adsorbent bed can retain and  $\bar{\theta}$  is the average of  $\theta$ , the concentration of the contaminant expressed in ppmv

$$\bar{\theta} = \frac{\int_0^t \theta dt}{t} \quad (15-13)$$

The need to replace filters for particle adsorption is usually inferred from their resistance to gas flow.

## 16. Sources of Heat in Gloveboxes

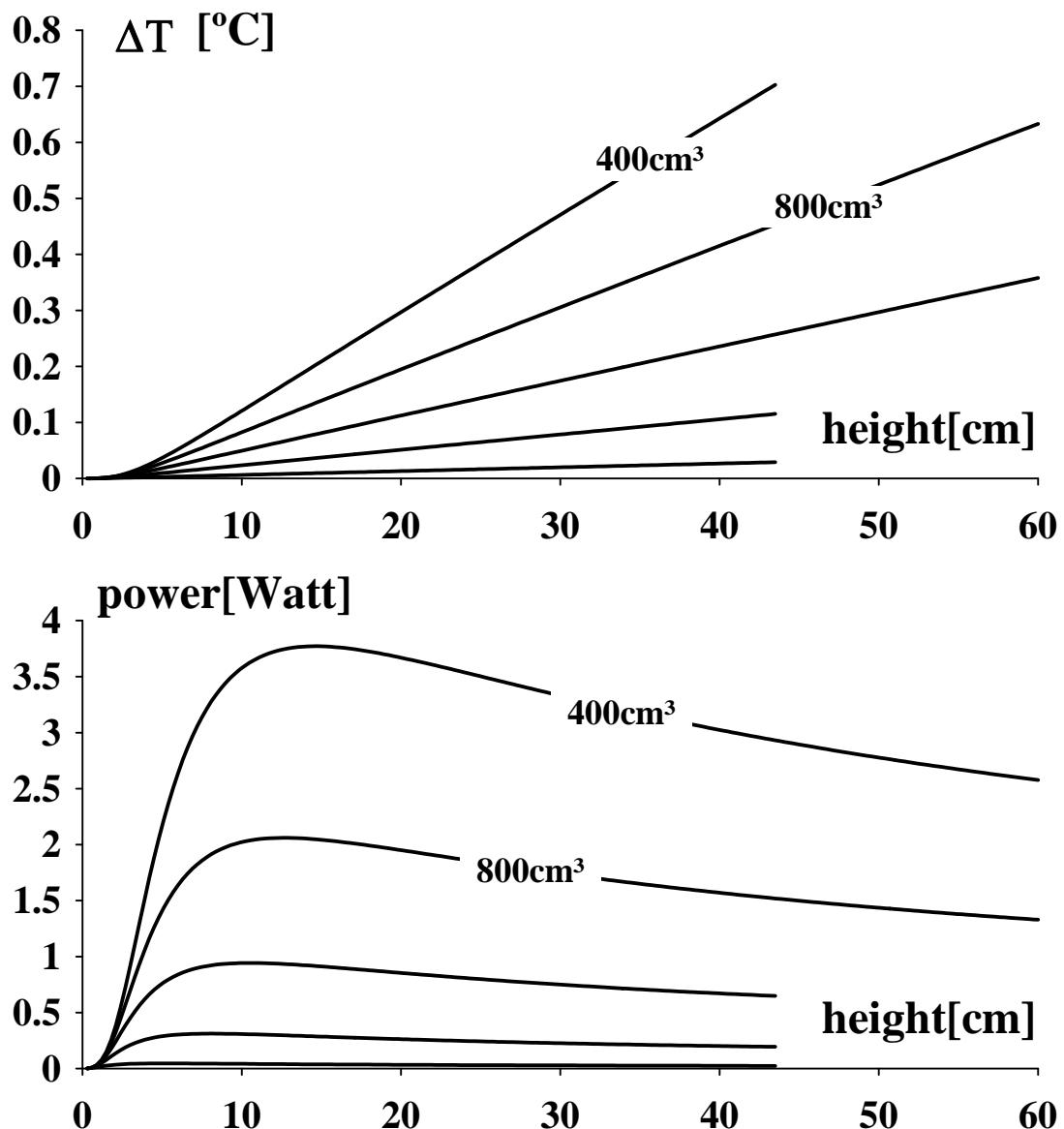
Temperatures inside gloveboxes are usually higher than ambient temperature. This is due in part to a *green house effect* whereby visible light passing through the window is converted to heat. Electrical instruments and light sources can further increase the temperature inside the box. In boxes with gas purification systems the flow of the viscous gas in the adsorbent bed will also rise the temperature. The following holds:

$$\Delta p \dot{V} = \dot{V} \rho c_p \Delta T \quad (16-1)$$

where  $\rho$  and  $c_p$  are the density and the specific heat capacity of the inert gas respectively. Using for  $\Delta p$  the expression in (15-2) the following is obtained for the rise in temperature of the gas.

$$\Delta T = \Xi \left( \frac{h^3}{V_i} \right)^2 \frac{(h + h_o)}{\rho c_p} \quad (16-2)$$

For the example discussed in the previous chapter the rise of temperature of the nitrogen gas is shown in the upper part of Fig. 16-1 for different values of the interstitial volume  $V_i$ , where  $\rho c_p = 1300 \text{ J/m}^3 \text{ K}$  was used.



**Fig. 16-1.** Computed increase of the box temperature and power converted to heat in the adsorbent column.

Five curves have been computed for interstitial volumes of the adsorbent bed of 400, 800, 1200, 1600, and 2000 cm<sup>3</sup>, where the interstitial volume represents a measure of the amount of adsorbent in the adsorbent column. The rise in temperature of the nitrogen gas in the adsorbent column increases roughly linearly with the height of the adsorbent column. However, due to the decrease of flow with increasing height, the rate at which heat is generated by internal friction in the adsorber goes through a maximum as can be observed in graph shown in the lower part of Fig. 16-1. In the present example the rate of heat input is quite small. However, in more efficient gas purification systems the increase in temperature may be appreciable. This is one of the reasons that boxes with efficient gas purification systems are sometimes equipped with heat exchangers or heat pumps.

The adsorption of contaminants in the adsorbent column also releases small quantities of heat. Let  $\Delta H_{\text{mol}}$  denote the heat released per mole of contaminant being adsorbed. Then the rate of heat release can be computed as follows:

$$\dot{Q} = \frac{\Delta Q}{dt} = \dot{V} \Delta H_{\text{mol}} \left( \frac{K\theta}{1,000,000 V_{\text{mol}}} \right) \quad (16-3)$$

where  $\theta$  is the concentration of the contaminant expressed in ppmv and  $V_{\text{mol}}$  is the molar volume. Assuming that all of the heat is given off to the inert gas, the rise in temperature could be computed from  $\dot{Q} = \dot{V} \rho c_p \Delta T$  as follows:

$$\Delta T = \frac{\Delta H_{\text{mol}}}{\rho c_p} \left( \frac{K\theta}{1,000,000 \text{ ppmv } V_{\text{mol}}} \right) \quad (16-4)$$

Ordinarily the concomitant rise in temperature is quite negligible. However, if a massive contamination takes place the rise in temperature is of considerable concern. Users who want to replace the adsorbent bed usually must inactivate the old adsorbent prior to exposure to air. This can be done by flushing the adsorbent bed with "diluted" air. From a given maximum temperature rise  $\Delta T$  an upper limit for the percentage of air in the gas mixture can be computed using (16-4). For example taking  $\Delta T \leq 150\text{K}$  and using for the depletion of the copper catalyst with oxygen  $\Delta H_{\text{mol}} = 310 \text{ kJ/mol O}_2$  and assuming  $K \approx 1$  we would obtain from (16-4) the following:

$$\theta \leq \frac{10^6 \text{ ppmv } \Delta T \rho c_p V_{\text{mol}}}{K \Delta H_{\text{mol}}} \approx \frac{10^6 \cdot 150 \cdot 1300 \cdot 0.0224}{1 \cdot 310,000} \text{ ppmv} \approx 14,090 \text{ ppmv} \quad (16-5)$$

where for nitrogen  $\rho c_p \approx 1300 \text{ J/m}^3 \text{ K}$  and  $V_{\text{mol}} \approx 22.4 \text{ liters}$  has been used. The concentration computed in (16-5) would correspond to an admixture of roughly 7% air by volume.

## **17. Conclusions**

The performance of gloveboxes with inert atmospheres of current design is limited mainly by the permeability of the gloves for the main constituents of air. Substantial progress toward gloveboxes with significantly enhanced performance might be anticipated mainly from designs based on laminar flow patterns using recirculatory gas purification systems with low residual concentrations of contaminants at the outlet. In this type of glovebox considerably lower levels of contamination "upstream" from the gloves should be attained.

# APPENDICES

## A. Expressing Concentrations in PPM

Residual concentrations of impurities in gloveboxes are usually expressed in *parts per million* (ppm). In "ppm" the "parts" correspond to the weight in general, i.e., with "ppm" we mean *parts per million by weight*. For example 5 ppm would indicate that in 1 kg sample there are 5 mg impurity. However, for gases the "parts" usually refer to the volume, i.e., the proper expression would be *parts per million by volume* (ppmv). This is largely equivalent to a corresponding number of molecules of impurity being present per one million molecules in the sample. For example 5 ppmv impurity in nitrogen ( $N_2$ ) would mean that of one million molecules there are five molecules of impurity on the average and 999,995 molecules  $N_2$ . Concentrations expressed in parts per million by weight and in ppmv can be converted into each other as follows:

$$\text{ppmv} = \frac{\text{density of inert gas}}{\text{density of impurity}} \text{ ppm} \quad (\text{A-1})$$

The ratio of molecular weights can be used instead of the ratio of densities. The unit *parts per billion* (ppb) is used in an analogous manner. The unit ppb is obviously smaller than ppm by a factor of 1000. For the most widely used inert gases and the most common impurities the ratios (density of impurity)/(density of inert gas) are as follows:

Contaminant	Inert gas		
	Nitrogen ( $N_2$ )	Argon (Ar)	Helium (He)
Oxygen ( $O_2$ )	1.142	0.801	7.994
Water vapor ( $H_2O$ )	0.643	0.451	4.500
Carbon dioxide ( $CO_2$ )	1.571	1.102	10.994

The volume of a contaminant inside a box with volume  $V_B$  can be computed from its concentration, expressed in ppmv, as follows:

$$V_c = \frac{V_B}{1,000,000} \left( \frac{c}{\text{ppmv}} \right) \quad (\text{A-2})$$

For example in a box with volume  $V_B$  of 600 liters a contamination with  $c = 200$  ppmv would amount to 120 cm<sup>3</sup> of gaseous impurity. Its approximate weight can be obtained from its molecular weight as follows:

$$m_c = \left( \frac{pV_c}{R_g T} \right) (\text{molecular weight}) \quad (\text{A-3})$$

where  $p$  and  $T$  are the absolute pressure and the absolute temperature (inside the box) respectively, and  $R_g$  denotes the universal gas constant;  $R_g = 8.314 \text{ J / (mol K)}$ . For 1 atmosphere pressure, where 1 atm = 101,325 Pa, and a temperature of 20°C this becomes

$$m_c = 0.04157 \text{ mol} \left( \frac{V_c}{\text{dm}^3} \right) (\text{molecular weight}) \quad (\text{A-4})$$

Using for molecular oxygen,  $O_2$ , a molecular weight of 32 g/mol, its total weight (inside a box with  $V_B = 600$  liters and a residual concentration of 200 ppmv oxygen) would be 0.1596 g.

A major concern is the amount of impurity entering the box per unit time. Let  $\dot{c}$  denote the rise in concentration of the impurity expressed in ppmv/h. The influx  $\dot{V}_c$  can then be computed from  $\dot{c}$  as follows:

$$\dot{V}_c = \frac{V_B}{1,000,000} \left( \frac{\dot{c}}{\text{ppmv}} \right) \quad (\text{A-5})$$

For example an hourly rise in the oxygen concentration of 6 ppmv in a box with a volume of 600 liters, according to (A-5) would amount to 3.6 cm<sup>3</sup> of oxygen per hour. According to (A-4) this would correspond to 4.79 mg of  $O_2$  per hour.

Next let us look at what happens when an impurity is diluted in a certain volume. If, for example, residual impurities in the antechamber with volume  $V_A$  are diluted when the inner door is opened after decontamination of an item to be transferred into the box. Let  $c_B$  and  $c_A$  denote the concentrations of the impurity in the box and in the antechamber respectively. Then  $c_{\text{mix}}$ , the concentration after mixing, can be obtained as a weighted average as follows:

$$c_{\text{mix}} = \frac{V_B c_B + V_A c_A}{V_B + V_A} \quad (\text{A-6})$$

Let us assume that the concentration of an impurity inside a box of volume 600 liters is 0.8 ppmv and the residual concentration of the impurity in an antechamber attached to the box is 11 ppmv. Then for a volume of 60 liters of the antechamber we obtain from (A-6)  $c_{\text{mix}} = 1.73$  ppmv.

## **B. Aspects of Performance**

Depending on the type of applications the performance of containment work stations can be judged based on different criteria of varying importance. Below different aspects are listed together with desirable design features and working procedures which can improve the performance of containment work stations. Depending on the type of application some of the recommendations may be nonsensical or even counterproductive. Most users of gloveboxes should nevertheless find a few useful hints in the list below.

<b>Gas Purification</b>	Elimination of gaseous contaminants infiltrating the box from the outside and/or being generated by various processes inside the box.
	<ul style="list-style-type: none"><li>• High flow rate of gas through the adsorbent column.</li><li>• High rate of adsorption of the gaseous impurities by the adsorbent in the adsorbent bed.</li><li>• High capacity of the adsorbent column for the adsorption of impurities.</li><li>• Low concentration of residual impurities at the outlet of the adsorbent column. (High retention ratio).</li><li>• Low flow resistance of the adsorbent column.</li><li>• Inside the box there should not be surfaces adsorbing large quantities of humidity or gases.</li><li>• Inside the box there should be no surfaces which are porous or rough.</li><li>• Installation of an acid trap (coarsely granulated soda lime). [Sherfey, 1954].</li><li>• Installation of a ventilating device in the gloves in order to reduce humidity from perspiration. [Sherfey, 1954].</li><li>• The inlet and the outlet of the adsorbent column are positioned far apart such that the formation of stagnant "pockets" with elevated levels of contamination is largely avoided.</li><li>• Operations are scheduled such that the risk of exposing highly sensitive materials to excessive levels of contamination is minimized.</li><li>• Operations are scheduled such that there is minimal risk of cross-contamination of samples.</li></ul>
<b>Regeneration or Replacement of the Adsorbent</b>	Reactivation or replacement of the adsorbing material in the adsorbent column.

## Gloves

The gloves form the weakest part of the box assembly. They should be chosen with great care and they should be examined at regular intervals when in use. Individual gloves can be tested for leaks using glove port covers. The gloves are first inflated and then covered with glove port covers. A quick collapse of an inflated glove is a sure sign of leakage. Damage to the portion wrapped over the glove port may go unnoticed, however. The gloves should be subjected to visual examination frequently by illuminating the interior with an electric torch. In order to make gloves less sticky they can be dusted with talcum powder.

- High tensile strength.
- Puncture resistant.
- Chemically inert.
- Heat resistant.
- Very flexible.
- High resistance to permeation of chemicals and gases in particular.
- High resistance to permeation of moisture.
- High resistance to swelling when in contact with liquids.
- High resistance to the formation of pin-holes and cracks.
- High resistance to abrasion.
- The gloves form airtight seals with the materials of the glove port.
- Long service life under the conditions prevailing at the location of use.
- High resistance to ozone and other pollutants in the atmosphere.
- No air bubbles have been introduced in the rubber during manufacture.
- Uniform thickness and tensile strength of the rubber. Areas with exceptional stress may be reinforced.
- High resistance to ionizing radiation.
- Strong absorption of ionizing radiation.
- Comfortable to wear.
- "Right" size for all users of the enclosure.
- Good tactile feel when handling items.
- Easy to replace.
- Only gloves which have passed an approved electrical test for material continuity are used.
- Sharp edges in the work area are covered with adhesive tape.
- Gloves are not stored in natural or artificial bright light.
- Gloves are not stored at temperatures above 25°C.
- Gloves are stored in a dry location.
- During storage gloves are not subjected to excessive pressure for example by excessive stacking.

- Soiled gloves are washed with soapy water (soft detergent). After washing they are rinsed copiously with distilled water and then dried in a cool air draft.
- Disinfection must be done using a method and disinfectants that are not harmful to the rubber of the gloves. (Do not use halogen products such as chloroform, javel water, etc., especially in concentrated form).
- Before putting on the gloves always take off your watch and any rings you may be wearing.
- When sterilizing gloves with chemicals such as ethylene oxide or peracetic acid vapors make sure that the disinfectant comes into contact with all surfaces of the glove. Turn the gloves over in the middle of the sterilization cycle if necessary.
- Keep an old pair of gloves in order to test on them resistance of the gloves which are being used to any operation which might damage them.

## **Pressure Regulation**

The pressure increases when a user reaches inside the box with gloves and it decreases if the gloves are withdrawn from the interior of the box. Due to changes in the ambient pressure the pressure difference between the box and its surroundings may vary. In many cases the pressure in the box has to be kept slightly above or below ambient pressure. For the aforementioned reasons the pressure inside the box has to be regulated. In some cases this is done by simply attaching a kind of balloon to the box which acts as an inflatable reservoir of gas. More refined systems for pressure regulation keep the pressure difference at a constant value by injecting or sucking off gas. Some users install foot switches for sucking off gas in order to ease entering the box with the gloves.

- Low gas consumption. (There is a tradeoff in general between low consumption of gas and narrow margins for the range of permissible pressures).
- Large volume of the box. (The variations in pressure due to users entering the box with their hands are inversely proportional to the volume of the box).
- The filtered exhaust from vacuum pumps is returned to the enclosure.

## **Temperature Control**

Instruments inside the box frequently give off significant quantities of heat. In addition radiation entering the box through the glass window and/or transparent walls may also increase the temperature of the box. In such cases the box may have to be cooled. Some applications, especially in the field of microbiology, require temperatures slightly above room temperature. In this case the box may have to be heated.

- Thermal insulation of the box.
- Circulation of the atmosphere in the box with a blower reduces thermal inhomogeneities.

## **Rate of Permeation**

Gaseous particles seep through the walls of the enclosure and especially through the gloves and sealings.

- Use of thick gloves made of a material with a low *permeation constant*.
- Avoiding the use of parts made of plastic or rubber.

<b>Rate of Leakage</b>	<p>Contaminations may enter the box also through leaks. This type of influx may be reduced to some extent by maintaining an overpressure inside the box. In some cases it may be desirable to prevent highly toxic and infectious agents from leaving the box. When working with an inert atmosphere at subatmospheric pressure the rate of leakage may greatly affect the purity of the atmosphere. Seals tend to deteriorate as time passes by. The aging process should not be excessive.</p>
<ul style="list-style-type: none"> <li>• Use of durable seals of high quality.</li> <li>• Maintenance of the box such that leak-tightness is assured. Seals may deteriorate due to aging of the sealant and gloves may be punctured.</li> <li>• Periodic testing of the enclosure for leaks.</li> <li>• The methods used for repairing leaks are durable and approved of.</li> <li>• There is no chance of abrasive dirt accumulating in seals.</li> <li>• Seals on doors and covers are protected from scratching by rims.</li> </ul>	
<b>Radiation Shielding</b>	<p>Radiation from radioactive decays may get through the gloves and walls. In addition particles from radioactive aerosols may escape from the box. In this case the criteria referred to in the "<i>rate of leakage</i>" apply. Build-up of radioactive contaminations inside the box could result in chronic exposure of personnel to excessive levels of radiation and material cross-contamination. Ionizing radiation may cause sensitive electrical equipment to malfunction [Betti, Rasmussen, Hiernaut, Koch, Milton &amp; Hutton, 1994].</p>
<ul style="list-style-type: none"> <li>• Thick gloves and walls made of material which strongly absorb radiation emitted in radioactive decays.</li> <li>• The box should be easy to decontaminate (rounded corners).</li> <li>• Installation of filters for particle absorption which prevent radioactive dust particles from settling in adsorbers used for gas purification. (There may be danger of radioactive contamination of the environment of the box when particulate filters must be replaced).</li> <li>• Rotating shafts, bearings, and other components which require lubrication may trap dust. They are avoided inside the glovebox.</li> <li>• Installation of isolation valves in pairs with which a contaminated box can be hermetically sealed.</li> <li>• Isolation valves have been installed in pairs between the box and components attached to the box.</li> <li>• There should be glove port covers available with which idle gloves can be covered.</li> <li>• Installation of a pressure regulation system with which a subatmospheric pressure can be maintained in order to prevent radioactive particles from escaping into the environment.</li> <li>• There should be some kind of safe with adequate radiation shielding in which strongly radioactive items can be stored when not in use.</li> <li>• Sensitive electrical instruments are shielded adequately against ionizing radiation.</li> <li>• The radiation shielding is such that personnel exposure is "as low as reasonably achievable".</li> </ul>	

- The sources of radiation are located as far away from users of the enclosure as possible.
- The gloves are securely fastened on the glove ports with metal straps.
- Adequate precautions against criticality accidents when working with fissile materials.
- Adequate precautions against introducing moderating materials in the proximity of a subcritical assembly of fissile materials.
- In the event of breach of containment gas is drawn from the box into a high depression extract at a rate high enough to prevent radioactive materials from escaping through the breach.
- Spread of radioactive contamination via gas transfer pumps or vacuum pumps is reduced as much as possible by appropriate filters [Billing, 1973, pp 166-170]. (Oil-free pumps may have to be used).
- Closed containers containing radioactive materials are monitored in order to detect pressurization due to radiolysis.
- Lubricants used exhibit low rates of isotopic exchange and they are largely unaffected by radiation.
- Lubricants can be replaced easily and the risk of radioactive spills is minimal.
- The rotary motion seals used provide adequate protection against spread of radioactive contamination.
- The filtered exhaust from vacuum pumps is returned to the enclosure.
- A high proportion of mechanization and remote handling is typical for the work done inside the enclosure.
- Persons with skin injuries on their hands do not use the gloves. When suffering skin injuries while operating the enclosure work is terminated as soon as possible and wounds are carefully cleaned and sterilized.
- Materials to be used for the construction of the enclosure have been tested. Their surfaces have been exposed to corrosive solutions of typical radioactive fission products for a specified length of time and the residual activity has been monitored over a well defined sequence of decontamination procedures.

## **Resistance Against Aggressive Substances and Mechanical Damage**

The walls may be scratched, dented or even cracked by mechanical action. For this reason the walls should be thick and they should be manufactured from material which can withstand the type of mechanical action the walls are expected to be subjected to.

- Use of materials which are resistant to mechanical damage.
- Use of materials which are chemically inert.
- Reinforcement of the box in places where damage by mechanical action is expected.
- Use of coatings of the walls which make them less susceptible to mechanical damage and corrosive attack by aggressive substances.
- Protection of the box by structural elements which protect the walls and or windows from mechanical damage and from damage due to aggressive chemicals. (For example placing a steel pan at the bottom of boxes made of acrylic glass in order to protect them from mechanical damage or from spills of aggressive chemicals).
- Whenever possible protective coatings should terminate outside the protective enclosure in order to prevent corrosive attack upon the substrate or the adhesive.

## **Resistance Against Heat**

Boxes made of acrylic glass begin to melt at places where the temperature exceeds 70°C.

- Avoiding the use of materials which melt or deteriorate at high temperatures.
- Shielding local sources of heat with insulating material.
- Cooling areas affected by heat sources.
- Installing transparent shields or wire meshes retaining sparks from processes such as welding.
- Installing a system for cooling the whole box.

## **Particulate Filtration**

Elimination of particles and/or bacteria from the gas inside the box. For the sterilization of the atmosphere special bacteria adsorbing filters may be used. In order to maintain a sterile atmosphere UV light can be used.

Some users install bacteria reducers with circulation blowers which pump the gas over a UV light source. In boxes with an atmosphere containing oxygen the accumulation of large quantities of dust in the atmosphere may have to be avoided for safety reasons. If there is too much flammable dust in the atmosphere devastating dust explosions may ensue. In order to extend the service life of expensive HEPA filters some boxes have primary and secondary filters, the primary filters serving the purpose of retaining particles of relatively large size. Some boxes used for performing work under clean room conditions are equipped with a laminar flow system with filters covering the left and right wall or the floor and the ceiling.

- High rate of gas flow through the filter.
- Laminar gas flow everywhere in the box.
- No obstacles and heat sources disrupting laminar flow patterns inside the box.
- High capacity of the filter for the absorption of impurities.
- Low concentration of residual impurities at the outlet of the filter. (High retention ratio).
- Low flow resistance of the filter.
- Facile and safe replacement of filters which are exhausted, leaking or clogged.
- Operations are scheduled such that the risk of exposing highly sensitive materials to excessive levels of contamination is minimized.
- Areas of the box where particles can settle down should be easy to decontaminate.
- Aerosol introduction and sampling ports are permanently installed for reproducible tests of the filters. (In systems with more than one duct feeding a filter bank, aerosol introduction should be such that defective filters can be identified).
- Where a reversal of gas flow cannot be entirely excluded additional filters may have to be installed.
- If the supply of gas is disrupted for some reason there should be no excessive pressure drop across filters.
- Where gas flow must be maintained at all times two filters of small size are installed rather than a single filter of large size. This will allow filter replacement at roughly one half normal flow.
- Pressure gauges are installed measuring the pressure drop across each filter system in order to identify exhausted filters.
- Fans and ejectors can be adjusted to allow for an increase in the resistance to gas flow of particulate filters as they become exhausted.
- In order to quickly identify defective filters, multiple filter units should be avoided.

<b>Illumination</b>	<p>Some boxes with transparent walls are sufficiently illuminated by the light sources illuminating the room in which the box is installed. Most boxes need their own light sources, however.</p>
	<ul style="list-style-type: none"> <li>• Proper positioning of one or more light sources of adequate strength.</li> <li>• Surface treatment of the walls in order to scatter the light of the light sources.</li> <li>• Avoiding the influx into the box of excessive amounts of heat released by the light source.</li> <li>• Use of a diffuse light source.</li> <li>• Critical process areas in the box are well illuminated and vision is not obstructed.</li> <li>• Spot lamps have been installed where necessary.</li> <li>• Mirrors have been installed where necessary.</li> <li>• The walls of the enclosure are transparent.</li> </ul>
<b>Ergonomics</b>	<p>Some workers spend large periods of time working in gloveboxes. In so doing they should feel as comfortable as possible. It may be that the design of the glovebox has to be matched to the size of its users. For example the ideal size of the gloves may depend on whether the box is used exclusively by females or also by males.</p>
	<ul style="list-style-type: none"> <li>• Most places inside the box should be within easy reach.</li> <li>• Sufficient shelf space should be provided in order to avoid the working area from being crammed.</li> <li>• The switches on the control panel and the displays of readings from measurement should be readily visible for the user while working.</li> <li>• Switches and valves on the box should be within easy reach.</li> <li>• The box should be constructed such that the user faces minimal risks of making serious mistakes.</li> <li>• The box should be easy to clean and to decontaminate.</li> <li>• Installation of a ventilating device in order to reduce discomfort from perspiration. [Sherfey, 1954].</li> <li>• The shape of the gloves should be such that, with his or her hands relaxed inside the gloves, the user should feel as comfortable as possible.</li> <li>• The temperature inside the box is such that its user feels comfortable. (The box may have to be cooled if there are too many sources of heat).</li> <li>• The enclosure is flexible. (When the user reaches for something the wall in front of him will recede).</li> <li>• The enclosure is collapsible and of small size. It can be easily transported to wherever it is needed.</li> <li>• The enclosure provides ample work space.</li> <li>• The enclosure has several work spaces where users can assist each other, for example a pair of work spaces facing each other.</li> <li>• The glove ports are of oval shape.</li> <li>• The pressure control is such that the user can get his hands easily in and out of the enclose. (Foot switches may be needed in order to adjust the differential pressure).</li> <li>• For work in the enclosure physical efforts are kept at a minimum as the operator works at a mechanical disadvantage.</li> </ul>

<b>Automation</b>	<p>In most boxes with gas purification systems which can be regenerated the regeneration process runs fully automatic. When working with gloveboxes a considerable amount of time is needed when transferring items through the antechamber. For this reason some boxes have a control device for the cycles of evacuations and floodings with gas needed for decontamination.</p>
	<ul style="list-style-type: none"> <li>• The automated procedure should limit the range of modes of operation as little as possible.</li> </ul>
<b>Feedthroughs</b>	<p>Most users of gloveboxes need supplies of gases, liquids and electrical power inside the box. The corresponding feedthroughs can be a source of considerable leakage. On some boxes all feedthroughs are installed on a removable sealed panel such that feedthroughs can be added or replaced without transporting the whole box to the machine shop. A great number of users need to have a close look at small items they handle inside the box. For this purpose a microscope can be installed inside the box with the upper part sticking out of the box. For this a special feedthrough is needed.</p>
	<ul style="list-style-type: none"> <li>• There should be no detectable leaks at the feedthroughs.</li> <li>• Feedthroughs for cables for transmission of electrical power or electrical signals should have sufficient capacity.</li> <li>• Feedthroughs for cables and tubes which have to be connected and disconnected frequently should be within easy reach and easy to operate.</li> </ul>
<b>Transfer Through the Antechamber</b>	<p>The antechamber serves as a barrier for contaminants or hazardous agents passing from the outside into the box or vice versa. Items placed inside the antechamber are decontaminated by flushing or by repeated evacuations and floodings with inert gas. The antechamber should leak as little as possible and it should be of sufficient size to accommodate all items which are to be transferred while the box is operating. However, the time needed for decontamination with a vacuum pump is proportional to the size of the antechamber and inversely proportional to the pumping speed of the vacuum pump used. For this reason many boxes are equipped with two or more antechambers of vastly different sizes. Some antechambers are used for the transfer of items between neighboring boxes. Antechambers of this kind are usually constructed with three doors, two doors leading to the boxes and one to the outside. Most antechambers are of cylindrical shape. For large antechambers a rectangular cross section may be more economical. The issues referred to below also apply to some extent to the start-up of evacuable boxes.</p>

- A pressure gauge has been installed on the antechamber.
- The vacuum chamber should have no detectable leaks.
- The vacuum chamber should be of sufficient size to accommodate all items the user wants to transfer.
- Evacuable antechambers should be constructed such that items can be decontaminated for transfer by either applying vacuum or simply by flushing. (Some items cannot be exposed to vacuum).
- The mechanisms for opening and closing the doors of the antechamber should be easy to operate and "snap" into position such that there is little danger of a door being closed improperly.
- Massive contaminations of the box taking place due to both doors of the antechamber being open simultaneously should be minimized as much as possible by a fail-safe mechanism.
- The amount of gas needed for flushing, the time needed for evacuation and the amount of contamination are roughly proportional to the size of the antechamber. The antechamber should therefore not be larger than absolutely necessary.
- In order to speed up drying and outgassing of items being transferred heating may be needed. The antechamber functions then like a vacuum oven.
- Depending on the nature of contaminants being removed in the antechamber its effluents may have to be scrubbed. (Vacuum pumps attached to antechambers are sometimes equipped with an oil mist condenser).
- The purge/backfill sequencer will activate an alarm if something went wrong while it was active, e.g., no vacuum or disruption of the supply of inert gas used for flooding.
- There is no interference from discharges into the vacuum line from other sources during evacuation of the antechamber.
- After evacuation the antechamber is flooded with inert gas immediately. (The antechamber is not left in an evacuated state for an extended period of time).

## **Use of Volatile Solvents Inside the Enclosure**

Vapors from solvents can accumulate in the box and condense at the colder parts of the walls. Halogenated solvents can quickly damage adsorbent beds used for the removal of oxygen. Molecular sieves used primarily for the removal of water are in most cases of a type which can also adsorb small amounts of organic vapors. The adsorbent bed of the gas purification system should therefore be regenerated more frequently at times when volatile solvents are being used inside the box. Older boxes are equipped with cold traps for the removal of solvents. Newer boxes tend to use adsorbers with activated charcoal for the same purpose. Charcoal made from coconut shells is especially well suited for the adsorption of solvent vapors. In some cases it is best to remove solvent vapors by flushing with inert gas.

- If the box is equipped with a gas purification system, it should be possible to operate the solvent adsorber with the gas purification system shut down in order to get rid of great quantities of solvent vapors having been released at one time.
- The complete removal of condensed solvent vapors is be readily accomplished.

- The removal of solvent vapors is quick.
- The removal of solvent vapors is complete, i.e., only small traces are left in the atmosphere.
- When removing solvents trapped in the adsorber or cold trap the user is not exposed to toxic fumes.
- The walls of the box are solvent resistant. (Vapors of many solvents cause *crazing* of acrylic glass. As a precaution a stainless steel pan might be installed in boxes made from acrylic glass in order to minimize the damage from spilled solvents).

## **Inspections and Maintenance**

Malfunctions and serious losses of performance of protective enclosures can be reduced considerably by scheduled maintenance. Maintenance may also be done at any time following detection of adverse changes.

- The manufacturer provided complete circuit diagrams, operation instructions, maintenance instructions, spare parts listing, and a listing of expendable replacement parts, e.g., filters.
- The user can estimate fairly accurately the expected service life of critical components of the glovebox.
- Gloves are examined visually at regular intervals for signs of cracking, tears, and punctures.
- Records of inspections and maintenance are kept in order to demonstrate that accepted standards of performance have been met.
- For the proper performance of the enclosure and its components testing procedures have been established. The acceptable deviations are clearly defined.
- A rigorous program of inspections and scheduled maintenance involving replacements of parts prone to failure has been implemented.
- Repaired and reworked products are reinspected prior to use.
- Controls are established to make sure that equipment that failed to pass the required inspections is not inadvertently installed and operated.
- Personnel performing inspections and maintenance have demonstrated competence for their assigned duties.
- Tools used for maintenance inside the enclosure do not pose a risk to the gloves.
- Provision is made to facilitate replacement of oil in diffusion or rotary vacuum pumps.
- Maintenance operations are closely supervised.
- The maintenance schedule takes into account the fact that deterioration of some materials such as rubber may be much faster than usual under laboratory conditions.

## Safety

Some gloveboxes are used for handling hazardous substances such as rocket fuel. This type of box poses a threat for their environment. On the other hand boxes which are used for handling sensitive items may malfunction and lose their protective function completely or to some extent. There are basically four types of hazards encountered by users of gloveboxes as follows:

1. Fires and explosions.
2. Release of hazardous substances or infectious agents by rupture of the containment.
3. Failure of the containment resulting in irreversible damage of items kept in the box.
4. Implosions due to vacuum applied to the box.

For installations with a high degree of complexity the safety of a glovebox has to be judged in view of its surroundings and the intricate interplay of interlinked systems.

- Installation of a device which prevents the formation of excessive underpressure or overpressure (for example a pressure relief bubbler).
- Design which minimizes the risk that vacuum is applied to the box. (For example a device preventing the antechamber from being "evacuated" when the inner door is open).
- Installation of a locking device which renders impossible the opening of a door of the antechamber when the other one is open. (An alarm to this effect may be sufficient).
- Installation of a control mechanism which conveys the system into a safe state if there are signs of serious malfunctions. (For example automatic shutoff of the vacuum pump).
- Providing a secondary containment inside the box in which items can be stored while they are not being used. (For instance using a fire-proof airtight safe for storing items inside the box. Even keeping items in closed jars may reduce significantly the damage from massive leakage).
- An efficient particulate filter is installed in order to prevent dust explosions.
- Valves are of a type such that power failure leaves them in a safe condition.
- Evacuable boxes are covered with a safety net during evacuation.
- For the regeneration of the adsorbent bed with hydrogen so-called *forming gas* is used. The content of H<sub>2</sub> in the forming gas should be sufficiently low to render the formation of explosive mixtures impossible (For mixtures with air at room temperature and atmospheric pressure: Inert gas containing less than 5% H<sub>2</sub>).
- Use of laminated safety glass on the window in order to prevent serious injuries if the glass should break.
- Instruments which withdraw gas from the box for monitoring are shut down if the pressure inside the box is dangerously low. (This type of situation may arise if the gas supply to the box is disrupted).
- Adsorbent columns which overheat if exposed to air are equipped with heat sensors which shut down the influx of the heavily contaminated inert gas if the containment should fail.

- When regenerating the adsorbent bed no vacuum can be applied during break-out if one or both valves at the ends of the adsorber are still open.
- Hazardous items are kept in a suitable secondary containment and they are kept in the box only as long as absolutely necessary.
- The design of the box does not encourage "dirty" habits by inexperienced staff.
- Difficult operations involving hazardous materials are first practiced with harmless substitutes.
- Users doing different types of work in a box inform each other about necessary precautions.
- There are permanent records of the conditions in the box (on magnetic storage or on paper). Items exposed to excessive levels of contamination can be unambiguously identified and they can be removed from further processing if necessary.
- No oil baths are used for heating inside the box.
- Users of the enclosure are protected from exposure to harmful UV radiation which can cause "*welder's eye*".
- Hazardous waste is encapsulated inside suitably sealed containers before being taken out of the box.
- When dealing with hazardous materials, only material currently processed is kept inside the box.
- No liquefied gases are used inside the box. Spilling of liquefied gases may give rise to a sudden rise in pressure.
- The glovebox laboratory can act as a confinement for hazardous material accidentally escaping the box.
- The air from the laboratory is exhausted through efficient nonflammable filters.
- Chemical filters with inflammable materials such as activated charcoal are fitted outside and some distance from the enclosure.
- Inquiry into causes of accidents and learning from the findings.
- Scheduled maintenance of the box at regular intervals.
- The user interface of automated boxes points out risks associated with different operations.
- Users of the enclosure are given binding instructions and rules for its proper and safe use.
- Danger signals are attached to the box at appropriate locations.
- Users can post signs on the box indicating that it should remain undisturbed or that an operation is in progress.
- External heat resistant glove port covers to block fires inside the enclosure are nearby.
- The gloves are secured properly.
- Stocks of emergency equipment are always available.
- The electrical wiring of the box is corrosion resistant.
- Defective electrical equipment is removed. The cause of its breakdown is determined.
- Heat sensors have been installed in electrical devices which might overheat.
- Robust electrical plugs are installed rather than miniature versions.
- In enclosures filled with argon the electrical wiring should have double the clearances recommended for air. (The breakdown voltage of Argon is lower than in air, a condition which may be worsened by ionizing radiation).

- Inflammable solvents are removed from the vicinity of the enclosure when not in use.
- Inflammable solvents are stored and transported in approved containers. The user does not handle quantities far in excess of what is actually needed.
- Waste is disposed of in different categories, e.g., combustible and non-combustible.
- Scrap from metal working is never stored in the enclosure but is removed as soon as practicable.
- Chips from pyrophoric metals are stored in chip pans of high thermal conductivity and heat capacity.
- The inside of the enclosure is not cramped and crowded.
- Disruption of the supply of cooling water does not cause equipment to overheat.
- Surfaces on the outside of furnaces are never hotter than 80°C.
- Materials to be used to combat fires should be safe. (When used on burning metals some materials such as water, carbon dioxide and carbon tetrachloride may actually help spread the fire. The use of unsuitable materials may even give rise to violent explosions).
- In the event of power failure there is no danger of breach of containment.
- Hazardous items which may break when dropped are not lifted higher than necessary. Whenever possible hazardous items are moved by sliding.
- Near the enclosure communication equipment is available for contacting specialists capable of dealing effectively with various types of emergencies.
- A label listing emergency phone numbers is affixed to the enclosure.
- The manufacturer of the box has supplied a detailed description of the tests the box had to pass before being released for sale and he provided the results of the tests for the box delivered to the user.
- The user is aware of the durability of critical components of the box such as magnetic valves and he knows the consequences of their failure. (Scheduled replacement of components liable to failure).
- The gloves do not come into direct contact with aggressive chemicals. As an added precaution an additional pair of disposable gloves, carefully matched to the chemical and its use, may be worn underneath the gloves of the box.
- The box has been constructed from non-flammable materials.
- The access to a box is sufficiently restricted to safeguard it against unauthorized manipulations or vandalism.
- Load limits of lifting devices and support rails are clearly posted.
- Each glove on the enclosure is tagged and a glove change program based on the projected service life of each type of glove is rigorously enforced.
- Users of the glovebox are encouraged to identify problems and recommend improvements.
- Obsolete versions of documents relating to the use of the glovebox should be removed from circulation.
- Auditory warnings are installed. In a glovebox line the glovebox which is defective should be identifiable.
- Switches and valves are color-coded for quick identification.
- Critical components of the glovebox are labeled for quick identification and for referencing in reports and guidelines.

- Cryogenic traps are not operated with liquid nitrogen in a box filled with argon gas.
- The required resistance to chemicals, radiation, heat, and mechanical stress of the materials used for the construction of the enclosure has been evaluated prior to use.
- Fire extinguishers of adequate design are installed.
- Hazardous waste remaining inside the enclosure during extended periods of time is labeled as such according to an appropriate scheme of classification.
- High-speed rotating systems must be properly encased.
- The glovebox and its understructure are expected to maintain structural integrity during an earthquake.
- Heavy equipment installed in the glovebox is supported inside the glovebox. If it should topple over nevertheless, the ensuing damage would be such that there is no breach of containment.
- In case of fire ventilation can be halted readily or it will stop automatically.
- Explosionproof electrical wiring inside the box (DIN 42005).
- The working atmosphere is selected to be nonreactive with materials within the protective enclosure.
- Use of an inert atmosphere inside the box in order to prevent the formation and spread of fire. (Inerting).
- Use of monitoring equipment, preferably with alarms, thereby increasing the probability that a malfunction of the box is detected early.
- Sharp objects which may puncture gloves are in the working area only at those times when their use is essential.
- Supplementary puncture resistant gloves are worn when necessary.
- Hot objects which may melt gloves are in the working area only at those times when their use is essential.
- Supplementary heat resistant gloves are worn when necessary.
- The design of the box is such that it actively encourages safe practices.
- Assurance that users of the box are thoroughly familiar with standard operating procedures.
- Assurance that users of the box are thoroughly familiar with emergency procedures.
- Contingency plans have been prepared if containment is lost during critical operations such as changing gloves.
- Adequate precautions are taken when unloading hazardous materials from the antechamber.
- When unloading contaminated items from the antechamber the worker is adequately protected from toxic fumes or infectious organisms.
- Users of the enclosure are wearing protective clothing.
- Preference for simplicity in the design and operation of the enclosure.

## **Electrostatics**

The very dry atmosphere in many gloveboxes is conducive to the buildup of static electricity, which can scatter powdered materials. Highly sensitive balances must often be placed inside some metal frame functioning as a *Faraday cage*. The ill effects of electrostatic charges on microcircuits are well known. There are many devices on the market for avoiding and removing electrostatic charges, many of which can be used in gloveboxes[Steinman, 1994]. Positive and negative ions which ordinarily would be present in the atmosphere are stripped out by the high efficiency air filtration. These charges would help to reduce build-up of static charges.

- The box should be grounded as much as possible.
- The box should be manufactured mostly from materials which are good conductors of electricity.
- Instruments inside the box can be grounded with wires.
- An electrostatic monitor has been installed in the box.
- An electrostatic eliminator (ionizing blower or gun) has been installed in the box.
- The work area is covered with grounded mats which dissipate electrostatic charges.
- "Problem areas" are covered with conductive grid tape.
- Users of the box wear suitable wrist straps underneath the gloves.
- An antistatic cabinet for handling sensitive items has been installed in the box.

## **Humidity Control**

In most boxes the humidity has to be kept low. This may be accomplished by simply placing drying agents in open trays inside the box. In most boxes with gas purification systems molecular sieves are used for this purpose. Molecular sieves can be regenerated by baking them out under vacuum. Even boxes used for handling and cultivation of micro-organisms usually are kept as dry as possible in order to avoid contamination of cultures with alien micro-organisms thriving in a humid environment. Some boxes, however, are operated with humid atmospheres (environmental chambers, climatic chambers). The removal of humidity is often much more time consuming than the removal of gaseous contaminants due to the fact that water tends to cover most solid surfaces with a thin film [Thiel & Madey, 1987].

- Adequate sealing of tubes with cooling water inside the box.
- Use of moisture absorbing material which can easily be regenerated or replaced.
- The box should not be manufactured from materials absorbing large quantities of humidity.

## **Durability**

At the time of purchase the user should be aware of the fact that under normal use most types of gloveboxes can be kept in service for decades. The parts most often in need of repair or replacement are gloves, seals, circulation blowers, windows and the adsorbent beds of gas purification systems.

- The enclosure has been purchased from a reputable manufacturer who provides written guarantees.
- Use of rubber seals which age slowly. (Anti-oxidant additives).
- Robust design.
- Design with few rubber seals. (With respect to durability welds are better than rubber seals).
- Protective coatings and surface treatment.
- Slow running circulation blower.
- Windows are made of material which cannot be scratched or cracked easily.
- Solvents used for cleaning and decontamination are compatible with glovebox shell, gloves, and window material.
- Cleaners containing more than 0.025 % by weight of *chlorides* are not used on stainless steel. (Corrosion cracking may result over time).
- Gloveboxes and glove bags made of plastic are not exposed to sunlight for extended periods of time.
- Cleaners leaving little or no residues are used.
- The design of the pressure control system and its settings are such that the average frequency of activation of magnetic valves is minimal and the solenoids are de-energized most of the time.
- Machines and instruments used in the enclosure are of a proven standard design. (Special designs may have unsuspected weaknesses which only show up after the equipment is heavily contaminated).
- Materials used for the construction of the enclosure are non-absorbent, non-adsorbent and resistant against chemicals, heat and high energy radiation.

## **Mechanical Vibrations**

Sensitive instruments, especially balances, must be protected from mechanical vibrations.

- No direct contact of the box with rotating machinery such as circulation blowers.
- Installation of platforms, such as marmor plates on rubber pads, for damping mechanical vibrations originating from components inside the box. (Centrifuges!).
- Putting up the whole box on a foundation which damps mechanical vibrations.

## **Serviceability**

The maintenance of boxes is usually done on site.

- Parts prone to failure should be easily accessible.
- Parts on the box should be of standard execution and readily available from the supplier.
- Parts which need periodic replacement should be kept in stock by the user.
- Parts which need periodic service should be ordered in duplicate by the user in order to minimize down-time.
- Items in frequent need of replacement or repair are installed outside the enclosure.
- Bulbs for microscopes which need frequent replacement are located in so-called *cold lights* outside the enclosure.

## **Monitoring Instruments**

Boxes with inert atmospheres need to have instruments for monitoring the purity of the atmosphere if the user wants to be sure that the box is working properly. The same applies to boxes where work is to be done under clean room conditions. Some users just use an incandescent lamp with the glass bulb removed in order to confirm that the concentration of oxygen is sufficiently low. If the concentration of oxygen is below 5 ppmv the incandescent lamp should keep burning for one to four weeks [Eubanks& Abbott, 1969; Hipps and Mazur, 1984]. An incandescent lamp used in this manner can serve as a switch for an alarm or some other device. Some organometallic compounds which smoke when exposed to traces of oxygen and/or moisture can be used for monitoring the atmosphere of the box [Shriver & Dezdzon, 1986, Sect. 2.3.F].

Users of anaerobic boxes have similar low cost solutions available for making sure that the atmosphere in the box is sufficiently anaerobic. They use redox indicators. Redox indicators are also added routinely to cultures of anaerobic microorganisms. (For example 0.1% by volume of a stock solution containing 10 g/l of *resazurin*). In some cases trace impurities other than oxygen and moisture have to be monitored. Hipps and Mazur [Hipps & Mazur, 1984] report a setup, where gas from the box was fed through a controlled leak into a mass spectrometer.

- While working in the box the user should be able to take readings from the instruments.
- The instruments should have to be calibrated rarely.
- The instruments are equipped with alarms indicating serious malfunctions of the box. (Nitrogen adsorbers, which usually work at high temperatures, can be seriously damaged if the concentration of oxygen rises too high. Instruments for measuring oxygen can be used to shut off these units automatically if the concentration of oxygen approaches dangerous levels).
- The instruments should need little maintenance.

## **Start-Up**

Most boxes are in continuous use. The procedures for putting a box into operation are therefore of little concern. However, some boxes, especially if operated by inexperienced users in academic settings, have to be put into operation quite frequently. The boxes most convenient to use under these circumstances are those which are evacuable.

- The amount of gas and the time needed for establishing suitable conditions for working in the box should be as small as possible.
- The site where the glovebox is to be installed should be prepared well in advance.
- Critical tests of the enclosure have been performed in the configuration that will actually be used.
- Before start-up the box is tested for leaks.

<b>Sterilization and Decontamination</b>	<p>Most boxes are equipped with filters for particle adsorption. For many boxes the need for cleaning arises only rarely. However, some boxes need to be decontaminated at regular intervals. In the nuclear industries the decommissioning of radioactively contaminated boxes has to be planned carefully.</p>
	<ul style="list-style-type: none"> <li>• The inside surfaces of the walls and especially of the floor of the box are smooth and easy to clean. (In order to reduce glaring reflections from the light sources, some manufacturers roughen the surface of the walls. This practice may be counterproductive with respect to the ease of decontamination).</li> <li>• If gaseous or liquid disinfectants are used a separate feedthrough may be useful.</li> <li>• The box has radius edges and corners.</li> <li>• Operations are scheduled such that there is minimal risk of cross-contamination of samples.</li> <li>• The frequency of transfer operations is kept at a minimum.</li> <li>• The frequency of changing gloves is kept at a minimum by using gloves which last longer.</li> <li>• Gaps and crevices are filled with suitable non-porous materials to facilitate cleaning and decontamination.</li> <li>• The design of the enclosure should preclude accumulation of hazardous materials in relatively inaccessible areas including ductwork.</li> <li>• When cleaning with cloth, cloths are changed frequently in order to prevent spread of contaminations.</li> <li>• Structural supports of cranes, monorails, hoists etc should be external to the enclosure in order to minimize surfaces to be cleaned.</li> <li>• The fans and motors are located in the clean part of the gas circuit to ensure safe access for repair should a fan failure occur.</li> <li>• Suitable anti-blowback devices prevent the reversal of gas flow due to wind back pressure or transient pressure changes in the ventilation system.</li> <li>• Interconnected boxes are equipped with one vacuum pump for each box.</li> <li>• There is a drain in the base of the enclosure leading to an effluent tank.</li> <li>• The inside of the enclosure is protected from the attack of corrosive chemicals. (Corroded surfaces are difficult to clean).</li> <li>• Effluents from leaking pumps are captured in suitable containers.</li> <li>• The site where the enclosure is operated is separated into clean and active zones.</li> <li>• Users of the enclosure are wearing sterile clothing.</li> </ul>
<b>Ease of Later Additions and Alterations</b>	<p>Frequently the type of use of a box undergoes changes.</p> <ul style="list-style-type: none"> <li>• The design of the box is such that it can readily be modified.</li> <li>• Modular design.</li> <li>• The box is equipped with blind flanges for extensions likely to be added to the box in the future.</li> <li>• The box is equipped with a removable sealed panel where additional feedthroughs can be installed.</li> <li>• The size of the box can be increased by removing a detachable side wall and adding an additional box.</li> </ul>

- Blind flanges have been installed for doors or transfer tunnels leading to neighboring boxes to be installed at some later time.
- The performance of the box has been chosen such that increasing demands in the foreseeable future can be met.
- The box is equipped with lifting lugs for transport to a new location.
- The glovebox and the antechamber have an identification nameplate furnished by the vendor.
- The manufacturer supplied blueprints for the box and all of its components.

<b>Ease of Recovery</b>	Breach of containment can be simply a nuisance causing delays and
<b>After Breach of Containment</b>	possibly minor repairs or it may have disastrous consequences.

- The enclosure is equipped with a system for the detection of breach of containment.
- Sensors for the detection of harmful effluents are installed at the site where the enclosure is being operated.
- The box is equipped with a powerful system for continuous removal of contaminants.
- The box is subdivided into separate containment work stations which are interconnected, but contaminations of one part do not spread among work stations under ordinary circumstances. (The workstations can be connected via antechambers).
- Inside the box there is a secondary containment for sensitive or hazardous items. (They might be kept in an airtight fireproof safe).
- Double-walled containment.
- The whole box can be evacuated, thereby allowing quick recovery from a contamination.
- The box is kept at all times at a subatmospheric pressure. Hazardous effluents are retained by filters of large capacity in the ventilation system.
- The box is kept at all times at an overpressure so that leakage is outward.
- Glove port covers are kept ready for quick installation at all times. Idle glove ports are covered with glove port covers.
- Instruments are installed for monitoring the atmosphere inside the box thereby providing early warnings of imminent danger.
- Instruments for monitoring the atmosphere are equipped with alarm relays which draw the operators attention to conditions indicating a malfunction of the box.
- The box undergoes automatically a transition into a safe state if readings indicate breach of containment.
- Gas purification systems which can suffer permanent and serious damage, for example by overheating, are automatically shut down if there is a breach of containment.
- After breach of containment make sure that affected personnel are monitored before they disperse.
- After breach of containment control entry into the contaminated area until it has been decontaminated.
- Provide facilities such as eye wash bottles and showers for quick dilution and neutralization of aggressive chemicals on the skin of workers.
- Provide facilities to shower and change clothes in a place nearby.

- A diesel-operated backup extract system will be activated in case of a mains-power failure.
- The enclosure is cleaned before commencement of critical operations such as changing gloves. While the operation is in progress hazardous and air-sensitive items are kept in sealed containers.
- Interconnected boxes can be separated quickly in case of an emergency such as fires or contaminations.
- Transfer tunnels between boxes can be closed with doors in order to minimize the spread of contaminations.
- Gas purification systems servicing several boxes can be disconnected quickly from boxes which are heavily contaminated.
- A contaminated box connected to other boxes can be disconnected and removed without interference with the work in progress in neighboring boxes.
- In the event of a power failure the oil in the vacuum pumps is prevented from being forced into the vacuum system.
- Blind flanges used for servicing equipment in the enclosure can be covered with a gas-tight PVC sheet tent.

## **Limitations in the Range of Applications**

Frequently a user decides to purchase a containment system for a specific purpose. Later on he may want to do another kind of work with the box.

- Prior to purchase the user has prepared a list of work he plans to do with the box.
- Prior to purchase the user sought advice from workers in his field who are experienced in working with containment workstations.
- The user has some idea on whether the box will be used sporadically with lengthy periods of time where the box stands idle or whether the box will be used continuously. (In the first case he might want to purchase an evacuable box which can be put into operation quickly).
- The user requested from manufacturers catalogues detailing the design and performance of various products and he talked personally to salesmen.
- The user requests that devices such as blind flanges and feedthroughs are being installed on the box which will ease the installation of additions for overcoming limitations. He knows of at least one manufacturer who can supply the needed equipment and he has a fairly good idea about the costs involved.

## **Compliance With Rules and Regulations**

The use of containment work stations has increased extensively over the past years. Increasingly tighter regulations have enhanced awareness for the appropriate containment of products and protection of working staff and the environment. In many cases containment workstations are the only practical and economic solution when handling hazardous and/or sensitive products.

- The containment workstation meets applicable quality standards required by law. (The manufacturer provided written guarantees to this effect).
- As long as it is used for the intended purpose, the box provides protection which would be considered adequate in a court of law.
- The manufacturer supplied adequate documentation concerning the proper use and the safety of the box.

- The box has not been subjected to alterations and extensions which compromise its safety and/or haven't been approved by the manufacturer.
- The box is operated and serviced by qualified staff.
- The box is inspected at regular intervals in order to make sure that it is fully functional and safe to operate.
- Evacuable gloveboxes are constructed and operated in conformance with applicable boiler and pressure vessel codes.
- All applicable codes should be followed during installation and operation of the box.
- Disposal of hazardous wastes is approved by proper authorities.
- Genetically altered organisms used in the box are destroyed in an autoclave when they are no longer needed.

## C. Accessories for Gloveboxes

**Access panel:** Detachable panel for equipment service and repair.

**Accordion sleeve**

**gloves:** Gloves with creased sleeves.

**Acid trap:** Adsorber for volatile acidic substances.

**Adsorbents:** Substances used mostly in adsorber columns for the elimination of impurities in the atmosphere inside the box.

**Adsorbent**

**work mat:** Embossed paper backed by a thin sheet of polyethylene for protecting work surfaces against spills of liquids.

**Air lock:** See *antechamber*.

**Air lock inserts:** Air lock inserts are used to fill empty space in antechambers during flushing or evacuation/flooding in order to reduce the consumption of gas.

**Aerosol generator:** Nebulizers for the inspection of flow patterns and performance. Microbiological tests use samples containing single-celled test organisms.

**Air sterilizers:** See *gas sterilizers*.

**Anaerobic chamber:** Airtight enclosure for the cultivation of anaerobic micro-organisms.

**Anaerobic jars:** Jars used for culturing anaerobes. Modern anaerobic jars use palladium catalyst, which removes oxygen in the presence of excess hydrogen.

**Antechamber:** Chamber with at least two doors, one leading to the outside and one leading to the interior of the box. Antechambers are used for the safe transfer of objects into and out of the box.

## **Anti-blowback**

### **device:**

Devices such as valves which prevent intermittent flow reversals due to wind back pressure or transient pressure changes in the ventilation system.

### **Bag port:**

Port for transfer of materials into or out of the box by means of plastic bags and heat sealing apparatus without breach of containment. Polyvinyl chloride, PVC, is the most common bagging material. Bags may be sealed or tightly twisted and wrapped with adhesive tape [White & Smith, 1962, Fig. 7.2].

### **Ball manipulator:**

Tongs for manipulations through a shielding wall of a hot cell. The tongs can be operated from the outside with a handle. Lateral movements are transmitted through a sealed ball swivel assembly.

### **Bare-hand entry:**

Enclosure with an *iris port* used for working with bare hands in a controlled atmosphere.

### **Bench:**

The glovebox may be installed on a special bench, which may be mobile. This bench may have structural elements for fastening varied accessories of the box.

### **Blind flanges:**

Blind flanges are installed on boxes for the transfer of bulky items in and out of the box or they may be installed in view of later additions and modifications.

### **Blow-off gun:**

Blow-off guns are used for eliminating dust from surfaces. When equipped with an ionizing unit a blow-off gun can also be used for static control.

### **Bursting disk:**

Device to limit the differential pressure on the box.

### **Catalyst box:**

A box with a blower for circulating the gas inside the box through a catalyst, usually a Pd-catalyst. This device is used mainly in so-called anaerobic boxes for the continuous removal of oxygen, by using forming gas inside the box.

### **Chair:**

Chair of a design suitable for working at the protective enclosure.

<b>Changing gloves:</b>	Gloves can be changed with a relatively small disturbance of the atmosphere inside the box if glove port covers are used and if the space inside the new glove is rinsed thoroughly with an appropriate gas. Some users recommend that the new glove be popped onto the glove it replaces which is then loosened and drawn into the box. Most procedures for changing gloves are relatively improvised.
<b>Charcoal filter:</b>	Filter used for removing gaseous impurities, mainly solvent vapors.
<b>CO<sub>2</sub> analyzer:</b>	See <i>gas analyzer</i> .
<b>CO<sub>2</sub> controller:</b>	Instrument for maintaining a constant concentration of carbon dioxide inside the box. This kind of instrument is used mainly in growth chambers of organisms metabolizing carbon dioxide.
<b>Cold light:</b>	A light source which is usually located outside the box. Its light is used to illuminate heat sensitive items inside the box via optical fibers. This kind of light source is used mainly for the illumination of slides in microscopes.
<b>Contractor:</b>	The organization which has contracted to supply specified equipment to the customer. Usually the term also covers any of his sub-contractors.
<b>Conversion front panel:</b>	Front panel with gloves for the conversion of open-fronted cabinets to gloveboxes. [Kuehne <i>et al.</i> , 1995, pp 145-170].
<b>Cold trap:</b>	Cold traps are used to remove solvent vapors by condensation on cold surfaces. The use of a cold trap is appropriate mainly for intermittent handling of volatile solvents.
<b>Conductive grid tape:</b>	Tape for removing static charges via a conducting grid sandwiched between two layers of antistatic plastic.
<b>Conveyor:</b>	Transport system in glovebox lines, such as chain propelled dollies, overhead chain carriers, belt conveyors, battery powered dollies, etc.
<b>Copper catalyst:</b>	An adsorbent for molecular oxygen in gases which can be regenerated with molecular hydrogen (or carbon monoxide). The temperature of the

catalyst should never exceed the recommendation of the vendor. Higher temperatures will cause the copper particles to splinter which will reduce their oxygen scavenging efficiency.

**Cryogenic trap:** See *cold trap*.

**Deepfreezer:** See *refrigerator*.

**Desiccant**

**dryer wafer:** Wafer containing desiccant in a gas purification system.

**Dip basin:** Basin filled with liquid disinfectant for decontamination. See also *dunk tank*.

**Dissipative mats:** Dissipative mats are used to remove static charges from the work area.

**Dosimeter:** Portable instrument for monitoring exposure to radiation of personnel.

**Dry box:** Enclosure for handling materials under exclusion of moisture. Also used as a synonym for "*glove box*".

**Dryer:** Special adsorbers used for removing exclusively moisture from the box. Some of these adsorbers can be regenerated simply by boiling off the water under vacuum.

**Drying train:** Recirculating gas purification system with desiccant for the removal of moisture.

**Dunk tank:** Special type of air lock filled with liquid disinfectant. Dunk tanks are used mainly in biosafety cabinets of class III. See also *dip basin*.

**Electric loop**

**sterilizer:** Instrument for the sterilization of devices used for the inoculation of culture media.

**Electrostatic**

**charge:** See *static eliminators*, *electrostatic monitors*, *dissipative mats*, *conductive grid tape* and *wrist straps*.

<b>Electrostatic monitor:</b>	Instrument for measuring static charges.
<b>End panel:</b>	Side wall of an antechamber of modular design.
<b>Extract fan:</b>	Fan which draws gas from the box in the event of breach of containment. (See also <i>high depression extract</i> ).
<b>Feedthrough:</b>	Penetrations through glovebox barriers for supply of power, gas, etc.
<b>Fire detection:</b>	Device to detect fires. Some detection systems combat fires by releasing some type of chemicals such as carbon dioxide, powders, etc.
<b>Fire extinguisher:</b>	Device for extinguishing fires. Some metals may react vigorously with carbon dioxide at elevated temperatures [Barton, 1963]. For this reason special designs might be needed.
<b>Floor pan:</b>	Floor cover, usually made of stainless steel, which protects the floor from spills of liquids and powders.
<b>Flow indicator:</b>	When rinsing a box, an antechamber or an adsorber a prescribed flow of gas must be maintained in many cases. For this purpose flow indicators or flowmeters are needed.
<b>Flowmeter:</b>	See <i>flow indicator</i> .
<b>Foot switch:</b>	Foot switches are sometimes installed in order suck off gas from the box, thereby facilitating the user getting with his hands into the box, especially in small boxes without a pressure regulating system. However, foot switches can be installed also for other purposes such as activating a cryogenic trap.
<b>Forming gas:</b>	Gas used for the regeneration of adsorbent columns, for example mixtures of inert gas and hydrogen to regenerate the "copper catalyst". These mixtures are usually designed such that the depleted adsorbent column does not overheat when it comes into contact with the gas. In addition the content of the active component must be kept sufficiently low to prevent the formation of explosive mixtures with air. Users working with rare gases can use a relatively inexpensive mixture of hydrogen and nitrogen as "forming gas" in general. In most gas

purification systems the gas is expelled from the adsorbent column by applying vacuum at the end of the regeneration procedure.

**Fume extraction:** Some operations performed inside the box, especially welding, generate harmful fumes which also may impair vision. These fumes would have to be sucked off into a funnel for filtering or discharge from the box.

**Fumigation:** Sterilization of an enclosure with gaseous chemicals or aerosols. Formaldehyde is one of the most effective agents. [Newsom, 1982].

**Gas:** Supply of gas used for working inside the box in a controlled atmosphere.

**Gas analyzer:** Instruments for continuous monitoring of the degree of contamination of the atmosphere inside the box.

**Gas mixer:** Device for continuous mixing of gases, mostly used for work with micro-organisms. Some users prepare their own "forming gas" using a gas mixer. This kind of unsafe practice is not recommended. Gases which can form explosive mixtures with air should be applied in "diluted" form, whenever possible.

**GasPack® pouch:** Disposable system for anearobic culture outside an anaerobic box; useful for field work.

**Gas pump:** Pump for gases, used mainly for drawing gas from the box for continuous monitoring of its contamination.

**Gas purification:** Gas purification systems can be classified in two types as a recirculating system or as a "last pass stripper" used for scrubbing effluents from the protective enclosure. For both types the mode of operation can be either continuous or they can be operated in a batch mode. The latter is usually the case for cryogenic traps used for the removal of solvent vapors. Most gas purifiers contain molecular sieve for the adsorption of moisture and an oxygen reactant.

**Gas sterilizer:** Device for continuous sterilization of the atmosphere in the protective enclosure, usually by heat treatment (400°C during at least 3 seconds) or with *germicidal lamps*. Some gas sterilizers use infrared heaters.

- Germicidal lamp:** See *ultraviolet lamps*.
- Glove bag:** Bag made of translucent PVC with glove sleeves sealed to the body of the glove bag.
- Glove bag port:** Ports with a (disposable) glove bag. This type of port is used frequently for replacing critical components of the containment such as gloves or windows.
- Glove box:** Any enclosure with one or more flexible gloves sealed to the container to allow handling materials inside a sealed enclosure with gloves.
- Glove change:** There are various techniques in use for replacing gloves [White & Smith, 1962, pp 143-144]. Systems can be purchased for changing gloves without breach of containment. A new glove attached to an ejectable support ring with an o-ring seal is loaded into a device used for changing gloves. The device is docked to the glove port and the old glove is ejected together with its support ring. The o-ring seal on the support ring of the new glove will have made contact with the port before the old glove loses its sealing contact. Similar systems can be installed also for changing filters.
- Glove ports:** Circular or oval-shaped holes in the glovebox face and protuberances around them for attaching gloves.
- Glove port clamps:** For secure fastening of the gloves on the glove ports metal strips can be used. They tend to decrease considerably the service life of gloves, however.
- Glove port covers:** Covers for glove ports. Most glove ports can be covered on both sides. Frequently different covers are needed for closing from the inside or the outside of the box. For evacuable gloveboxes glove port covers have to be installed from the inside in order to prevent rupture of the gloves. Some glove port covers have feedthroughs for gas for evacuating or flushing the outsides of the gloves.
- Gloves:** Gloves for working in gloveboxes. Various types of materials are used for their manufacture. The gloves can be of varied thickness and they can be either left hand, right hand or ambidextrous. Regarding their size there are three relevant measures as follows: size, cuffs, length.

Gloves should be marked with the vendor's name, the date of manufacture, and a symbol to indicate that the glove has passed an approved electrical test for material continuity.

**Glove sleeves:** These are used either with interchangeable hands or they are equipped with an iris diaphragm, thereby allowing the user to work with bare hands inside the box. In some cases short rubber gloves, called "hands" are attached to the sleeves.

**Hands:** Gloves which can be fastened to *glove sleeves*.

**Heat exchanger:** Heat exchangers are used mainly to lower the temperature of a box with a lot of heat sources inside. They may also be used in order to recover heat used for the adsorption of impurities in the gas. (See also *nitrogen furnace*).

**Heat sealing:** Sealing of polyethylene bags for the transfer of items in and out of the box. This technique is used mainly for work involving materials emitting  $\alpha$  or  $\beta$  radiation.

**Heating:** Some boxes have to be operated at elevated temperatures. Heating can be done in most cases using a fan heater and thermal insulation of the box if necessary. Some antechambers can be heated in order to speed up drying and outgassing of items being transferred into the box. See also *vacuum oven*.

**Heat pump:** The use of heat pumps is similar to the one of heat exchangers.

**Heat sealing:** Heat sealing of polyethylene bags is used in a technique for the transfer of items without breach of containment.

**HEPA filter:** *High efficiency particulate air filter.* These filters can remove particles from gases with diameters down to a fraction of a micron. The filters can be constructed from a variety of fibers with diameters of roughly one micron. Today only fibers made from inorganic materials are used. In gloveboxes a single HEPA filter is installed in general. (HEPA® is a registered trade mark of HEPA Corporation).

**High depression extract:**

In the event of breach of containment the pressure within the box rises to atmospheric pressure. This pressure rise is detected by instrumentation and air is drawn from the box into a *high depression extract* to achieve an inflow of air through the breach (approx. 1 meter/second) sufficient to virtually eliminate the risk of significant quantities of hazardous materials escaping from the box.

**Hot cell:** Enclosure with radiation shielding for handling radioactive materials.**Humidifier:** Device to increase the humidity inside the box. (Used mainly in environmental chambers).**Humidistat:** Device to maintain a prescribed humidity.**Hydrogen leak detector:** Device for detecting leaks using hydrogen. (This device is used mainly for anaerobic chambers with flexible walls).**Hygrometer:** Instrument for measuring the humidity, either the absolute humidity, (dew point), or the relative humidity.**Incubator:** Secondary enclosure with temperature control and in some cases with a separate controlled atmosphere, for example with carbon dioxide.**In-drum compactor:** Device for compacting solid waste in cylindrical storage containers.**Inert gas:** Supply of gas used for working in inert atmospheres inside the box.**Inertization:** Using inert gas inside the glovebox in order to prevent fires and explosions.**Inflatable balloon:** When a box is put into operation a large balloon can be inflated with inert gas inside the box thereby driving out most of the air inside the box [Senn, 1993; Sherfey, 1954].**Input sphincter:** Device with sliding seals for transferring cylindrical objects through the protective barrier without breach of containment and without use of air locks. [Am. Glovebox Soc., Figs. 5.8 and 5.9].

**Interlocking lead**

**bricks:** Lead bricks for building walls shielding against  $\gamma$  radiation.

**Iris port:**

Hole in the wall of the box or its antechamber for the transfer of items with only limited transfer of contaminants. These holes are closed by rubber membranes. In most cases they can be covered from the inside with a gas-tight lid when they are not being used.

**Iris diaphragm:**

See *iris port*.

**Isolation valves:**

In order to detach some components, such as solvent adsorbers or cold traps, from the box without disturbing its operation *isolation valves* may have to be installed.

**Junction box:**

Power supply for instruments inside the glovebox.

**Laminar flow:**

Laminar flow is defined by the US Federal Standard 209 [US Federal Standard No. 209D] as flow of air in which the entire mass of air moves inside a room with uniform speed and parallel streamlines. In turbulent flow particles may remain trapped in stationary whirls for extended periods of time. The same applies in principle to gaseous contaminants. However, laminar flow is used mainly for particle adsorption where suitable flat filters can readily be manufactured. Usually a pair of filters is used the first one serving a rough cleansing of the gas by adsorbing particles of large diameter. The condition of the filters is inferred from the differential pressure needed to keep up a desired rate of gas flow.

**Lamps:**

Lamps for illumination of the box. Generally the installation of lamps inside the glovebox should be avoided for obvious reasons of safety and service.

**Lead glass bricks:**

Bricks for building transparent walls shielding against  $\gamma$  radiation. See also *interlocking lead bricks*.

**Lead glass window:** Window which protects the operator of the enclosure against  $\gamma$  radiation.

**Lead storage safe:**

Safe for storing materials which emit  $\gamma$  radiation.

- Leak detectors:** Mobile instrument for detecting leaks on the containment work station. A wide variety of techniques for leak testing are in use, some of them involving measurements of differential pressure. Leaks may be located with ultrasound, with mass spectrometer leak detectors, or by a "sniffing" method using gaseous chemicals containing halogens.
- Lining:** Usually a fluoropolymer sheet material bonded to the substrate by means of a suitable adhesive system. Coatings should provide the required resistance to the environment typically found in nuclear, chemical, and pharmaceutical processing.
- Liquid transfer feedthrough:** Feedthrough for liquids used for cooling, column chromatography, etc [Weidmann, 1987].
- Manipulator:** Mechanical device for handling materials without direct contact. In the nuclear industries manipulators are sometimes used for handling objects submerged in water.
- Master-slave manipulator:** A pair of manipulators on vertical shafts linked together horizontally. The master and slave ends have similar differential knuckle joints. Operations performed with the master end are replicated by the slave.
- Metal finishes:** Modifications of metal surfaces with finishing tools such as side grinders, buffing wheels, polishing pads, etc.
- Metal strips:** See *glove port clamps*.
- Microscope:** A close visual examination of objects inside a glovebox is often rather difficult. For this and other reasons some boxes are equipped with (stereo) microscopes. The upper part of the microscope usually sticks out of the window of the glovebox. For this reason a special type of windows with an appropriate feedthrough has to be installed.
- Microscope lamp:** See *cold light*.
- Mini antechamber:** Antechamber of small size which can be quickly evacuated and needs relatively small quantities of gas for decontamination.

**Mist generator:** Ultrasonic generator of droplets of liquids with diameters down to a few microns; used in growth chambers and for testing with aerosols.

**Modular design:** Design of protective enclosures which facilitates later modifications.

**Moisture analyzer:** See *gas analyzer*.

**Molecular sieves:** Molecular sieves are universally employed for the removal of moisture in gloveboxes. Types 4A, 5A, and 13X are commonly used. Molecular sieves can also adsorb solvent vapors. They can be regenerated by heating to a range of 200 to 330°C, purging with gas, and/or break-out under vacuum.

**Nitrogen adsorber:** See *nitrogen furnace*.

**Nitrogen furnace:** Adsorber for molecular nitrogen, N<sub>2</sub>. Most of these adsorbers work with titanium sponge at elevated temperatures of 750-900°C. Some simple versions of this type of furnace simply consist of a heated protrusion at the top of the box containing titanium sponge.

**Non-return valve:** See *anti-blownback device*.

**Oil bubbler:** Device for maintaining a constant overpressure inside the box which is continuously flushed with gas. (Contains usually vacuum pump oil).

**Oil mist eliminator:** Device for the condensation of microscopic droplets of oil in the exhaust from vacuum pumps.

**O-rings:** The gloves can be fastened to the glove ports with O-rings made of rubber.

**Oxygen analyzer:** See *gas analyzer*.

**Particle analyzer:** Analyzer for the determination of the number of dust particles per unit volume of gas. Some instruments can discriminate among particles of different ranges of size. See also *gas analyzer*.

**Particulate filter:** See *HEPA filter*.

**Prefilter:** In order to lengthen the service life of HEPA filters so-called prefilters are installed for the adsorption of particles of relatively large size.

**Pressure gauge:** Instrument for measuring pressure, mainly inside antechambers.

**Pressure controller:** Device for maintaining a constant pressure difference between the interior of the box and its surroundings. The pressure difference is maintained at a constant value in general by two position action on a pair of magnetic valves leading to the gas supply and to the vacuum line.

#### **Pressure relief**

**bubbler:** Device with one or two chambers partially filled with vacuum pump oil. It is used to prevent the formation of excessive overpressure or underpressure in the box [Wilson *et al.*, 1988]. Barton [Barton, 1963] described some other devices for the same purpose, such as blow-off valves.

**Pressurized seals:** Tubes which can be inflated with air or inert gas which serve as tight seals. Wilson *et al.* [Wilson *et al.*, 1988] report a type of use where the space between a pair of pressurized seals is being evacuated.

**Pressurized suit:** Sealed clothing with fresh air supply. In some cases only the upper part of the body is inside a pressurized suit sealed to a protective enclosure.

#### **Purge/fill**

**sequencer:** The proper decontamination of objects in the antechamber using a purge/backfill mode of operation can be quite time consuming. For this reason a device is sometimes installed on the antechambers which performs the necessary sequence of operations automatically by opening and closing a series of magnetic valves.

**Radiation detector:** Instrument for the detection of radioactive contamination of the box.

**Refrigerator:** Freezer installed in the glovebox for storing perishable items or volatile liquids.

## **Remote handling**

**tool:** Tool for manipulating radioactive materials, chemicals, explosives and other hazardous items at safe distances.

**Replacement seals:** Seals on the box or on its accessories which need replacement at regular intervals.

**Robotics system:** Manipulators which can be used in an autonomous or interactive mode in a glovebox environment mainly in order to reduce operator exposure in hazardous operations.

**Rubber bellows:** A kind of a "rubber balloon" attached to the box which represents a low cost alternative for an automatic pressure regulator.

**Safe:** Fireproof and airtight secondary containment which may have to be lockable.

**Sampling vessel:** A vessel used to retrieve samples of process fluids, without breach of containment.

**Servomanipulator:** See *manipulator* and *robotics system*.

**Service panel:** Removable plate fastened to the exterior of the glovebox with utility feedthroughs installed. The advantage of using a service panel is that it can be removed and modified in a machine shop without having to transport the whole glovebox.

**Shelving:** In order to free some work space on the floor shelves can be installed inside the box. Some boxes have swiveling shelves fastened on vertical poles.

**Shielded cell:** See *hot cell*.

**Shielding pond:** Pond, usually filled with deionized water, which is used for shielding ionizing radiation. The water may be cleaned by a recirculation system through filter bags and ion exchange columns.

**Silica gel:** Moisture adsorbent which is used for reducing the humidity inside the box and which can be regenerated by heat treatment. In general these

adsorbents undergo a color change as they have adsorbed quantities of moisture close to their full capacity.

**Solvent adsorber:** Solvent adsorbers are used to remove solvent vapors. Usually activated charcoal or a suitable molecular sieve is used as adsorbent.

**Sparge pipe:** Sparge pipes are used at the inlets and outlets of gas purification systems in order to suppress the formation of stagnant pockets with elevated levels of contamination in the atmosphere in the enclosure.

**Stand:** Understructure of the containment work station.

**Static eliminators:** Devices for the reduction of electrostatic charges. (Blowers or guns. The ions used for neutralization are typically generated by corona discharges or ionizing radiation from radioactive sources).

**Sterilization:** A wide variety of techniques for sterilization are in use. Microbiologists use devices known as *flaming bacteriological loops* which can be operated by a foot switch. Protective enclosures can be sterilized with suitable gaseous chemicals. UV lamps can be used to reduce undesired growth of organisms. HEPA filters can be sterilized with vaporized hydrogen peroxide.

**T-antechamber:** Transfer tunnel connecting two neighboring boxes with three doors, two at both ends of the tunnel, and a third door leading to the outside.

**Titanium sponge:** Titanium is the only element that burns in nitrogen. Titanium sponge is used for the adsorption of nitrogen gas at temperatures around 900°C.

**Tong-change station:** Specially designed station for changing tong heads of manipulators.

**Transfer tunnel:** Tunnel connecting two adjacent boxes. Usually at least one end of the tunnel can be closed with a door.

**Ultraviolet lamps:** Sources of ultraviolet light used mainly for continuous sterilization of the box. Some of these light sources are located inside an opaque housing through which the atmosphere to be sterilized is circulated by a blower.

- Vacuum oven:** Secondary enclosure for heat treatment of objects. It is usually used for drying and degassing objects. In most cases it is attached to the outside of the box and functions simultaneously as an antechamber.
- Vacuum pump:** The vacuum pump may be used for operating the antechamber and/or for maintaining a constant relative pressure inside the box.
- Velcro closures:** Makeshift closures for protective enclosures.
- Vent tubes:** Effluents from the glovebox may have to be safely discharged into the ventilation system. For this purpose vent tubes must be installed. In some cases the effluents must be filtered prior to discharge into the ventilation system.
- Vibration damping:** Stone plates, frequently made of marble, which rest on rubber pads dampen vibrations. The whole glovebox can be shielded from mechanical vibrations in this manner.
- Vortex amplifier:** Fluidic throttling valve for pressure regulation of ventilated containment work stations.
- Waste basket:** Some boxes have special antechambers for the discharge of solid or liquid waste. Some "waste baskets" are simply circular openings with a cover. In some boxes the refuse is discharged into a plastic tube which can be heat sealed for airtight packaging of hazardous items.
- Window:** A visual access to the interior of the protective enclosure that maintains primary containment. The glove ports may be window-mounted or they may be installed on the wall of the box below the window.
- Wipe:** Wipes are usually taken from a wipe dispenser. If they are transferred into the box without adequate decontamination they can contaminate the atmosphere by outgassing for extended periods of time.
- Wrist straps:** Wrist straps are worn by users underneath the gloves in order to reduce the buildup of static charges.

**Zinc bromide**

**window:** Window with aqueous solution of zinc bromide in between a pair of laminated glass plates; used for radiation shielding [Walton, 1958, pp 330-344].

## D. Symbols

$b$	Parameter for computing the rate of permeation of a contaminant into a box.	$g$	Parameter which indicates the rate of permeation of a contaminant through one glove.
$B$	Parameter which indicates the rate of gas flow through an adsorbent column.	$G$	Number of gloves of the containment work station.
$c(t)$	Concentration of a contaminant in a containment work station as a function of time; $c(0)$ is the concentration at a time $t = 0$ .	$h$	Height of an adsorbent column. (Also "hour" when used as unit of time).
$C(t)$	Concentration of an impurity integrated over time as shown in (4-3). This quantity is referred to as <i>exposure</i> throughout.	$h_o$	An increment to the height of the adsorbent bed corresponding to the resistance to gas flow due to tubing, particulate filter, etc.
$\dot{c}$	Rate of change of the concentration of a contaminant.	$k$	Transfer coefficient for the reaction of a gaseous contaminant with a liquid or solid sample.
$\bar{c}$	Average concentration of a contaminant.	$K$	Probability of adsorption of a particle of impurity when passing through the filter. ( $0 \leq K \leq 1$ ).
$c_a$	Concentration of an impurity in the ambient atmosphere.	$m(t)$	Mass of a contaminant as a function of time; $m(0)$ is the mass at a time $t = 0$ .
$c_A$	Concentration of a contaminant in the antechamber.	$\dot{m}$	The rate of change of the total mass of contaminant inside the box.
$c_B$	Concentration of a contaminant in the containment work station.	$m_c$	Mass of a contaminant.
$c_{eq}$	Concentration of a contaminant at equilibrium when the total rate of influx and the rate of adsorption are equal.	$n$	The number of boxes in a <i>glovebox line</i> .
$c_i$	Concentration of an impurity in inert gas used for continuous flushing of a box.	$N/\text{ft}^3$	Number of dust particles per cubic foot.
$c_{mix}$	Concentration of a contaminant of a mixture of gases (with concentrations $c_A$ and $c_B$ ).	$N_{eq}/\text{ft}^3$	Number of dust particles per cubic foot at equilibrium.
$c_p$	Specific heat capacity of a gas.	$N_o/\text{ft}^3$	Number of dust particles per cubic foot at a time $t = 0$ .
		$p$	Pressure.
		$P$	Rate of flow of a contaminant expressed in mass per unit time.

	Different rates are labeled as P, P', and P".	$t_{ave}$	Average life time of a particle in the box prior to adsorption.
$p_o$	Pressure inside the antechamber after flooding.	$T_\infty$	<i>Transfer index.</i>
$p_f$	Final pressure during evacuation of the antechamber.	v	Speed of gas flow.
$p_s$	Base pressure for the evacuation of the antechamber. (Pressure for $t \rightarrow \infty$ ).	V	Volume of gas (used for flushing).
Q	Heat.	$\dot{V}$	Rate of gas flow through the adsorbent column; $\dot{V} = \frac{DV}{Dt}$ .
$\dot{Q}$	Heat input.	$V_A$	Volume of gas inside the antechamber.
$Q_L$	Leak rate.	$V_B$	Volume of gas inside the containment work station.
$Q_A$	Permeability coefficient.	$V_c$	Volume of a gaseous contaminant.
R	Rate of total influx of a contaminant into the containment work station.	$\dot{V}_c$	Rate of influx of a gaseous contaminant expressed in volume per unit time.
$R_1, R_2$	Rates of influx of contaminants into a double-walled containment. When referring to glove box lines the quantities $R_1, R_2, R_3, \dots$ denote the rate of permeation of contaminant into individual boxes and $R_o$ denotes the average rate of permeation of the boxes in the line.	$V_i$	Interstitial volume of the adsorbent bed. $V_i$ also denotes the amount of adsorbent in the adsorbent column.
$R_g$	Universal gas constant; $R_g = 8.314 \text{ J/(mol K)}$ .	$\dot{V}_i$	Rate of flow of impurity into the box due to residual impurities in the inert gas used.
s	Pumping speed of the vacuum pump used for the evacuation of the antechamber. (Also <i>second</i> , when used as a unit of time).	$\dot{V}_m$	Maximum rate of gas flow of a ventilator.
t	Time.	$V_{max}$	The maximum volume of gaseous contaminant the adsorbent column can retain.
T	Absolute temperature.	$\dot{V}_r$	Rate of rinsing a box with inert gas.
$t_{1/2}$	Half life time a particle of impurity in the filter.	w	Parameter which indicates the rate of permeation of a contaminant through one window and its seal.
		W	Number of windows on the containment work station.

$x$	Ratio of $h$ and $h_o$ ; $x = h/h_o$ .	$\varepsilon_a$	Cost of adsorbing the amount of contaminant in one unit of volume air.
$z$	Average concentration of a contaminant in between the walls of a double-walled containment.	$\eta$	Parameter related to the pumping speed, $s$ , of the vacuum pump used for the evacuation of the antechamber with volume $V_A$ ; $\eta = s/V_A$ .
$z_{eq}$	Average concentration of a contaminant in between the walls of a double-walled containment at equilibrium.	$\vartheta$	Ratio of $R_1$ and $R_2$ in double-walled containments; $\vartheta = R_1/R_2$ .
$\alpha$	A parameter indicating the performance of a gas purification system for a box with volume $V_B$ ; $\alpha = KV/V_B$ . The parameter $\alpha$ is inversely proportional to the average time a particle of impurity spends inside the box before being adsorbed.	$\theta$	The concentration of a contaminant expressed in ppmv.
$\beta$	A parameter which is related to the half life time of a particle of impurity in an adsorbent column.	$\bar{q}$	Average concentration of a contaminant expressed in ppmv.
$\beta_o$	The parameter $\beta$ for an adsorbent column at the time it is put into operation.	$\Lambda$	Partition coefficient of a contaminant.
$\Delta H$	Heat of reaction.	$\Xi$	Parameter indicating the resistance to gas flow of the adsorbent column.
$\Delta H_{mol}$	Molar heat of adsorption (of a contaminant in the adsorbent column).	$\rho$	Density of a gas.
$\Delta m$	Loss of mass of a reactive compound due to reaction with a contaminant.	$\varsigma$	Ratio of $p_o$ and $p_f$ in the evacuation of the antechamber; $\varsigma = p_f/p_o$ .
$\Delta p$	Pressure difference.	$\tau$	The average time a particle of impurity spends inside the adsorbent bed.
$\Delta p_o$	Maximum static pressure of a ventilator when the rate of gas flow is zero.	$\chi$	Portion of the volume which is located in between the walls in boxes with double-walled containment.
$\Delta T$	Change of absolute temperature.		
$\varepsilon_i$	Cost of one unit of volume of inert gas.		

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