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# Inert Gases in the Control of Museum Insect Pests

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# The Getty Conservation Institute

The Getty Conservation Institute works internationally to further the appreciation and preservation of the world's cultural heritage for the enrichment and use of present and future generations. The Institute is an operating program of the J. Paul Getty Trust.

# Research in Conservation

The Research in Conservation reference series presents the findings of research conducted by the Getty Conservation Institute and its individual and institutional research partners, as well as state-of-the-art reviews of conservation literature. Each volume covers a topic of current interest to conservators and conservation scientists. Other volumes in the Research in Conservation series include *Oxygen-Free Museum Cases* (Maekawa 1998); *Stone Conservation: An Overview of Current Research* (Price 1996); *Accelerated Aging: Photochemical and Thermal Aspects* (Feller 1994); *Airborne Particles in Museums* (Nazaroff, Ligocki, et al. 1993); *Epoxy Resins in Stone Consolidation* (Selwitz 1992); *Evaluation of Cellulose Ethers for Conservation* (Feller and Wilt 1990); *Cellulose Nitrate in Conservation* (Selwitz 1988); and *Statistical Analysis in Art Conservation Research* (Reedy and Reedy 1988).

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# Foreword

The interest of the Getty Conservation Institute in the management of pest control reached a turning point in 1987 when Nieves Valentín, then with the Instituto de Conservación y Restauración de Bienes Culturales (now called the Instituto del Patrimonio Histórico Español) in Madrid, Spain, came to the GCI for a tenure as a Fellow to continue her conservation research on ways to stop biological attack on organic materials. Concerned about the impact that traditional pesticides have on both conservators' safety and the environment, the Institute recognized the need to contribute to the development of newer, safer techniques for halting the damage done by insects, fungi, and algae to museum collections. With this goal in mind, the GCI invited Dr. Valentín to join its staff and pursue her research in this important area of conservation. Her presence at the Institute and the enthusiasm she generated in her field of endeavor opened further avenues of exploration.

Since then, the GCI has continued research in this field both in-house and with associated institutions. This has resulted in a number of completed projects such as storage cases for the Royal Mummies of Egypt's National Museum; display cases to protect the Constitution of India developed in collaboration with the National Physical Laboratories of that country; cases for mummies in the Museo Balaguer in Barcelona, Spain; the disinfestation of Kienholz's *Back Seat Dodge '38,* done in collaboration with the J. Paul Getty Museum and the Los Angeles County Museum of Art; the display case for the Hudson Bay Charter, in Toronto; and technical advice for the conservation of the Declaration of Arbroath in Scotland. Additionally, the Institute has offered pest control management courses in various locations around the world.

*Inert Cases in the Control of Museum Insect Pests* is a compendium of information on the biological mechanisms by which nontoxic gases kill insects; the methods and materials needed to create and maintain an anoxic atmosphere; treatments; the construction and use of chambers and bubbles; and the procedures for treating objects.

This publication is a continuation of the Institute's Research in Conservation series, which aims to make available the findings of research conducted by the Institute and its partners as well as provide state-of-the-art reviews of conservation literature. From its inception the Institute has sought to open to the conservation professions the results of its research and experience in areas that bridge the gap between contemporary science and technology and current conservation practice.

In addition to Nieves Valentín, GCI senior scientist Shin Maekawa and GCI Fellows Kerstin Elert and Vinod Daniel have dedicated much of their time to this area, and a number of other institutions also have collaborated in our research efforts.

With his usual scientific rigor, organizational skills, and attention to detail, Charles Selwitz undertook the exacting task of reviewing the literature and the research done at the Institute and by other individuals and organizations to produce this publication. To him, to my colleagues, and to our partners, I extend my sincere acknowledgment.

I hope our readers will find in these pages the information and expertise that will provide them with further means to continue in our joint efforts to help conserve the world's cultural heritage.

—*Miguel Angel Corzo*
*Director*
*The Getty Conservation Institute*

# Preface

Over the centuries, one of the most persistent causes of loss of cultural property and museum collections has been damage done by insects. Prime targets are manuscripts on paper and parchment, natural history collections, and herbaria, but massive wooden objects also are often attacked and, occasionally, seriously harmed. Throughout the nineteenth and most of the twentieth centuries, the response to this biological assault has been to use an array of highly toxic chemicals, an approach that has resulted in many well-documented negative consequences. More recently, the conservation community has begun to appreciate that prevention is a better approach and that there are physical and biochemical procedures that can safely substitute for pesticides when prevention is occasionally inadequate. As embodied in a program of regular inspections, good housekeeping, and maintenance, prevention is designed to avoid providing pests with access to collections and is more commonly called *integrated pest management.* Better and safer killing methods to treat an established infestation include the use of biologically active agents such as pyrethrums and juvenile growth hormones, and physical means such as desiccation with nontoxic gases, freezing, and heating. Increasingly, well-established pest control programs at both small and large institutions are being built on the considered use of all of these options.

This book is about one option, the use of nontoxic gases (argon, nitrogen, and carbon dioxide) to rid museum objects of insect pests. Conservators are selecting this approach because they feel more comfortable and confident that using inert gases is much less likely to harm objects than other procedures. Curators have been known to insist that disinfestation be carried out by anoxia (which is defined as a deficiency of oxygen reaching bodily tissues of such severity as to cause permanent damage) in clear plastic bags so they can keep watch over their charges during treatment. Freezing and thermal methods are also effective when carried out properly. They are popular because they allow large quantities of material to be conveniently transported and treated. But freezing and heating, by their nature, must bring objects to unaccustomed temperatures where unwanted changes often occur. In contrast, treatment of cultural property in an inert atmosphere rather than air provides a more stable environment where deterioration is less likely. For example, the use of a low-oxygen environment will deter biological growth, prevent surface oxidation, and retard color fading. Studies by Burke (1992), Arney, Jacobs, and Newman (1979), and others show that the longevity of most organic colorants is substantially increased in a nitrogen atmosphere with less than 1000 ppm oxygen. One such study examined a number of inorganic pigments under these conditions and found that three of them—litharge (PbO), cinnabar (HgS), and sienna (mostly $Fe_2O_3$)—showed slight color changes after a one-month exposure. Additionally, Valentín (1990) found that low oxygen levels can be used to inhibit the growth of both bacteria and fungi.

In 1996 the remaining toxic fumigants generally approved for residential use in the United States were Vikane, sulfuryl fluoride, and methyl bromide. In museum facilities, however, the use of toxic gases has been abandoned, primarily for reasons of safety. But if it were necessary to find an additional reason for choosing nitrogen over Vikane, one might consider the visual effect of the two gases on painting materials. Koestler and coworkers (1993) assessed the impact of the two gases on thirty combinations of linen, rabbit-skin-glue size, lead-white oil ground, and oil-based paints employing eleven different inorganic pigments. The comparison was based on a visual evaluation made by two paintings conservators of color change, gloss change, blanching, topography change, and precipitation. Samples treated with Vikane by a commercial exterminator underwent the conventional treatment of two, well-spaced short exposures; anoxia samples were kept under nitrogen containing less than 1000 ppm oxygen for five months. Impurities in the Vikane (hydrogen chloride and sulfur dioxide) adversely affected ten of the eleven pigment samples, while nitrogen had no

observable effect on any sample. The inorganic colorants included lead oxide and sienna, but not cinnabar. These studies demonstrate that although there are many ways to rid museum collections of insect pests, none is likely to be safer or more protective of the integrity of objects than the use of a controlled atmosphere.

The preceding discussion helps to explain the rapid growth of anoxia and carbon dioxide fumigation as conservation procedures. In 1990 these methods were virtually unknown in the museum community, although studies were under way in a number of facilities. In most locations these studies quickly gave way to practice. Appreciation of this new preservation technology has since spread rapidly through journal publications, papers presented at meetings, and anoxia training courses. Nieves Valentín, of the Instituto del Patrimonio Histórico Español (formerly the Instituto de Conservación y Restauración de Bienes Culturales) in Madrid, conducted workshops in Spain and Latin America and brought this approach to pest control to Spanish institutions such as the Prado Museum, the Fundació Joan Miró, the Museum of Decorative Arts, and the National Palace of Fine Arts, as well as the National General Archives in Columbia and the National Palace of Fine Arts in Cuba. John Burke, with the Oakland Museum Conservation Center, provided training to conservators in California through a variety of formats, including a workshop at the San Diego Natural History Museum in March 1996. And the International Institute for Conservation—Canadian Group held a preconference course in May 1996 in Montreal entitled "Those Pests in Collections: Control of Insects and Fungi in Cultural Collections," which featured carbon dioxide fumigation. The Getty Conservation Institute offered programs on pest management and control for museums in Los Angeles in 1994 and in London in 1996.

Another important factor in the dissemination of this technology is the active sharing of new information by experienced conservators and conservation scientists, a practice not seen in most other disciplines, and the generous response to requests for help by the early workers in this field. Almost all of these people are still willing to answer questions and have permitted us to list their names in the Professional Contacts section of this book (Appendix A). Up to 1997 it was possible to track the institutions that were using nontoxic gases for pest control, but this is no longer possible because of the rapid growth in this field. It seems that most cultural institutions, large and small, in Europe, Latin America, Australia, and North America are now considering or actually employing this technology, while an increasing number of commercial vendors are making available materials and systems designed specifically for use with nontoxic gases.

The growing interest in anoxia and carbon dioxide fumigation has led to a large number of inquiries by museum personnel who are aware of this technology, and it is for this reason that this book was prepared. It is directed and dedicated to conservators who must handle problems of insect infestation, and it is intended to be a guidebook for this purpose. We attempt to do this by explaining how the proper use of these gases brings about 100% mortality of all life stages of the small number of insect species that are problems; by describing how to create anoxia systems in conservation facilities and providing examples of the way many prominent institutions are using this technology; by listing where supplies and operating systems can be purchased; and even by providing information on whom to telephone for help.

Chapter 1 describes the biological mechanisms by which nontoxic gases kill insects. The biochemistry of mortality by nitrogen and argon differs somewhat from that of carbon dioxide, but all of these gases owe their effectiveness to desiccation. It is important for conservators to understand this because factors that impact on desiccation, such as temperature and humidity, are important in the design and operation of treatment systems.

The second chapter discusses the pioneering research of conservation scientists Mark Gilberg, with the National Center for Preservation Technology and Training in Natchitoches, Louisiana, and Nieves Valentín. Working from different backgrounds, they demonstrated the absolute effectiveness of anoxia as a preservation tool and created practical systems for dealing with insect problems. This chapter also describes the early research by Michael Rust and Janice Kennedy of the University of California, Riverside. They conducted experiments with all life stages of twelve of the most destructive species, involving tens of thousands of specimens, to determine the minimum time necessary to ensure 100% mortality of each species under nitrogen containing less than 1000 ppm oxygen. The chapter further examines how factors such as oxygen concentration, type of gas (argon or nitrogen), temperature, relative humidity (RH), the embedment of insects inside objects, and the duration of treatment affect the success of the process.

Chapter 3 provides information about the methods and materials needed to create and maintain an anoxic atmosphere. In all cases, containment must be essentially free of oxygen; thus containers must be constructed of materials through which this gas cannot pass. This requires a nitrogen supply with only a few parts per million of oxygen, and container walls or plastic bags made of materials, called barrier films, that have very low oxygen permeability. This chapter also describes the use of powerful oxygen scavengers that can convert air to an acceptable grade of nitrogen. Three types of treatments have evolved from the manner in which these tools are used: maintaining a continuous flow of nitrogen over objects; sealing small items inside plastic pouches holding oxygen scavengers; and treating large quantities of material inside reusable chambers.

Chapter 4 provides guidance on aspects of carrying out treatments. The well-being of objects likely to suffer infestation generally depends on maintaining adequate humidity. Humidification is needed for continuous flow treatment, while the addition of water may not be necessary with static systems if the objects and their packaging provide adequate moisture buffering. Procedures for humidification are described. With anoxia systems the operator has a choice between nitrogen and argon, and the factors influencing that choice are discussed. Insect mortality is produced 30% faster with argon than with nitrogen. However, nitrogen is generally cheaper than argon, and most conservators have opted for this gas. Monitoring systems for oxygen and carbon dioxide concentrations, temperature, and RH are described in a comprehensive section by coauthor Shin Maekawa. Requirements for the safe use of nitrogen, argon, and carbon dioxide are also discussed. These gases are not toxic in the conventional sense, but they must be handled with adequate caution and monitoring. Nitrogen and argon are suffocants, and carbon dioxide will cause respiratory problems at very modest concentrations.

Chapter 5 provides details on the construction and use of plastic bags made of oxygen-barrier films. Suitable bags are commercially available, even for large, bulky projects. Rentokil, Inc., for example, has constructed barrier-film bags of 80 m$^3$ capacity for treating large numbers of oil paintings with infested wood frames. However, conservators doing a treatment generally make the pouches by hand in a wide range of sizes and shapes. The application of these bags to problems at institutions as diverse as the Metropolitan Museum of Art in New York, the Mendocino County Museum in Willits, California, the Los Angeles County Museum of Art, the Phoebe Apperson Hearst Museum of Anthropology in Berkeley, and the Oakland Museum is described. Treatments are occasionally carried out by maintaining a continuous flow of humidified, high-purity nitrogen about infested material for two or more weeks. This can be done by running the nitrogen into an artifact-containing bag that has a small, deliberate leak. More often this treatment is used with large, awkwardly shaped, and difficult-to-move objects. In such cases a complex barrier-film container is built around the object

with heat-sealed plastic panels. Chapter 6 describes the use of this procedure to disinfest ornate furniture from the decorative arts collection of the J. Paul Getty Museum, modern sculpture in the form of *Back Seat Dodge '38* by Edward Kienholz at the Los Angeles County Museum of Art, and a grand and regal Bartolomé March piano in Spain.

Many institutions conduct anoxia treatments in reusable systems that generally have a much larger capacity than do pouches. These fall into two classes: flexible bubbles and rigid chambers. Chapter 7 provides directions for making a 10 m³ barrier-film bubble, but most operators prefer to buy one of the commercial Rentokil units. Rigid chambers are perhaps more easily constructed in-house than are bubbles, and two examples are provided. Using a long, 18 in. (45.72 cm) diameter section of standard poly(vinyl chloride) pipe, Steven Pine of the Museum of Fine Arts in Houston built a chamber to treat items of some length but modest width, like rolls of fabric. Alan Johnston put together a simple but effective chamber for the Hampshire Council Museums using a polypropylene shell, a plate of polycarbonate, a large gasket, and several C-clamps. Another option is to locate a commercial fumigation chamber left over from the days when treatment with ethylene oxide was in vogue. The Los Angeles County Museum of Art has such a unit, which was easily converted for use with humidified nitrogen and is now run on a routine basis.

The final chapter of this book, chapter 8, deals with the use of carbon dioxide for treating infested objects. Although anoxia has found more widespread use, the amount of cultural property treated with carbon dioxide is larger. This gas requires less stringent control of concentration; it is cheaper to use; and the acidity created by carbonic acid formation has not been found harmful to treated objects. Although the biological mechanisms leading to desiccation and death differ between nitrogen and carbon dioxide, and nitrogen is generally more effective, the operating conditions and run times for carbon dioxide fumigation can be adjusted to achieve total disinfestation. Most treatments are done in relatively large, reusable commercial bubbles developed by Rentokil. Carbon dioxide fumigation has not found application with small bags or pouches. The earliest use of carbon dioxide fumigation was in England in the late 1980s, but its continued use there is deterred by regulations requiring that treatment be done only by licensed fumigators. Institutions in the United States with large collections of furniture and other decorative arts—such as the Henry Francis duPont Winterthur Museum in Winterthur, Delaware; Old Sturbridge Village in Sturbridge, Massachusetts; and the Society for the Preservation of New England Antiquities in Boston—have quickly moved to put carbon dioxide units on an operating basis. This technology is also being applied to safeguard the natural history collections at the Smithsonian Institution in Washington, D.C. In Canada the use of carbon dioxide was promoted by Thomas Strang of the Canadian Conservation Institute and was adopted by a number of Canadian museums such as the National Museum of Science and Technology in Ottawa and the Canadian Museum of Civilization in Hull. In Germany professional fumigator Gerhard Binker employs carbon dioxide on a massive scale to treat entire structures, primarily churches, that are either tented or leak-sealed.

This book contains many references to the published literature on anoxia and fumigation. They are indicated in the text with parentheses showing the author and year of publication. Complete publication information is listed in the References. Unlike most scholarly texts, and because this book is intended to be eminently practical, there is also an unusual number of informal descriptions of actual conservation experience that either have not been published or were published in limited-circulation periodicals. Most of these descriptions were directly communicated to the authors by letter or interview, in which case there is no parenthetical reference after the individual or institution is named.

# Acknowledgments

# Chapter 1

# Mechanisms of Insect Mortality

## Desiccation

A number of mechanisms have been proposed to show how oxygen deprivation (anoxia) causes increased mortality, but desiccation seems to offer the best explanation for the results obtained with the procedures and conditions used to eradicate museum pests. The evidence is threefold. First, insect physiology provides a well-defined respiratory system that leads to accelerated desiccation in the absence of oxygen or in the presence of modest amounts of carbon dioxide. Second, death rates generally increase as conditions move in directions favoring dehydration. Thus, increasing temperature or decreasing humidity typically makes anoxia a more effective killing procedure. The third important line of evidence is that mortality rates are positively associated with weight loss which under anoxic conditions can occur only by loss of water.

Insects are able to control two important processes—the exchange of oxygen and carbon dioxide, and the conservation of water—by a series of orifices known as spiracles (Fig. 1.1), which are part of a gas-transport system. A waxy epicuticular layer on the spiracles prevents water loss and maintains low oxygen permeability, thus putting the spiracles in control of the supply of these essential substances. Spiracles lie along the abdomen and thorax, where they are attached to regulatory organs (Fig. 1.2). The spiracles control the access time for the transport of gases and the size of the pathway through the body wall to the trachea. Hassan (1943) suggested that the successful evolution of insects as a group of terrestrial animals was largely facilitated by this regulatory system. The spiracles are normally kept closed to minimize water loss and are opened just enough for the insect to take in needed oxygen. When oxygen is scarce, however, they are forced to open more frequently and more widely, thus causing dehydration. An insect must get rid of carbon dioxide as well, and high concentrations of this gas in modified atmospheres, quickly sensed when the spiracles open, will also lead to sustained opening and, consequently, dehydration. These two conditions—very low oxygen levels and high concentrations of carbon dioxide—are the rationale for the two different types of modified atmospheres developed for pest control; both force the spiracles to open and remain open. This unnatural condition leads to high rates of water loss, as much as seven to ten times higher than when the spiracles are closed (Mellanby 1934; Wigglesworth and Gillett 1936; Bursell 1957; Jay, Arbogast, and Pearman 1971). Under conditions of high

**Figure 1.1**
Atriate spiracle with lip type of closing.
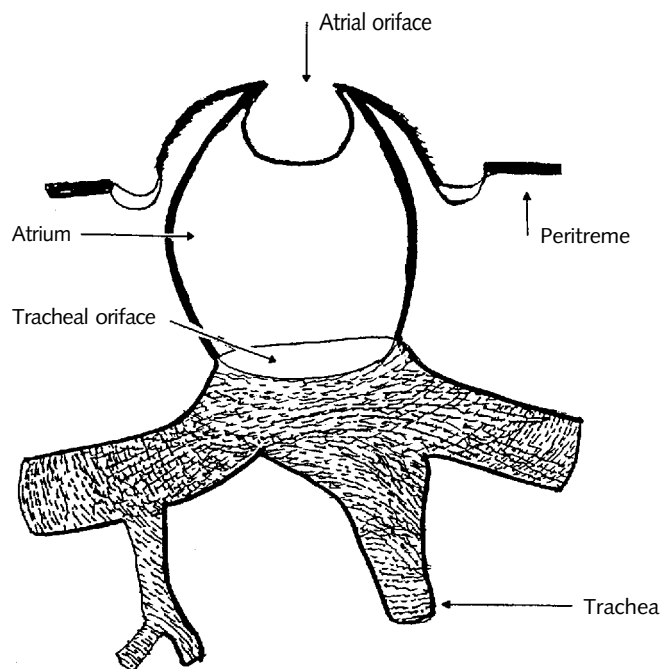


Atrial oriface

Atrium

Peritreme

Tracheal oriface

Trachea

Spiracles
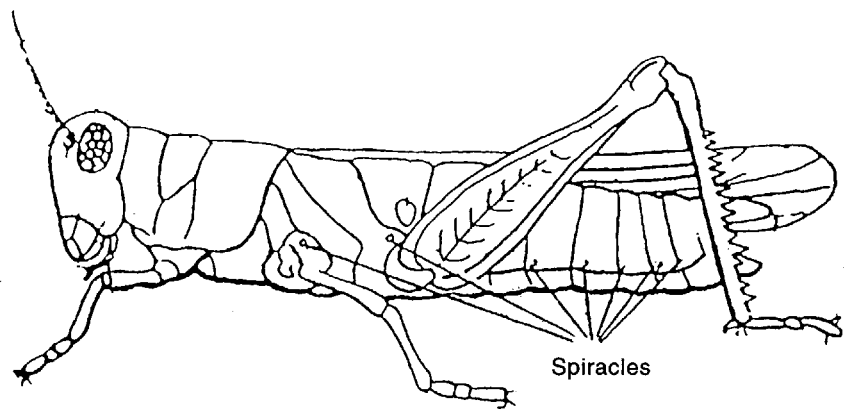
carbon dioxide concentrations accompanied by high humidity, the role of dehydration in causing death is less clear. The firebrat is an insect that typically lives at high temperatures and high humidity. In this species, high carbon dioxide concentrations relax the spiracle muscles, causing the spiracles to close by elastic recoil. There is low tracheal water loss in the firebrat as exposure to carbon dioxide brings on death (Noble-Nesbitt 1989). Donahaye (1990) also finds that other mechanisms must be invoked to describe the effects of high humidity and high carbon dioxide atmospheres on red flour beetles.

Rising temperatures increase insect respiration, resulting in a greater production and loss of water. This should increase mortality, and the work of Valentín (1993) demonstrates that it does. Figure 1.3a shows the minimum exposure times needed to achieve complete insect mortality at 40% relative humidity (RH) and 300 ppm oxygen in argon for eight different species at 20, 30, and 40 °C. Figure 1.3b provides the same comparison with nitrogen as the carrier gas. For each carrier an increase in temperature from 20 to 30 °C decreased the exposure time approximately 30%; an increase from 20 to 40 °C shortened the time about 90%. There was an even more dramatic decrease in exposure time for the most resistant species, the old house borer. Jay, Arbogast, and Pearman (1971) showed the relationship between mortality and RH by examining the death rate of red flour beetles and confused flour beetles in nitrogen atmospheres containing between 8000 ppm and 10,000 ppm oxygen after 24 hours at relative humidities of 9%, 33%, 54%, and 68%. As shown in Figure 1.4, both species showed a marked increase in mortality as the RH decreased. Decreasing the RH enhances the anoxic effect by increasing water transport and loss through open spiracles. When total body-water loss approaches 30%, most insects die.

Under anoxic conditions, any decrease in weight is due almost entirely to loss of water. In situations where there is a positive association between kill rate and weight loss, the mechanism responsible is highly likely to involve dehydration. Thus weight-loss studies are important for explaining a mechanism of mortality based on desiccation. One of the few such studies was done by Navarro (1978). For red flour beetle adults and *Ephestia cautella* pupae, he found that as the level of oxygen was uniformly lowered in 1% increments below 4%, the time to reach 95% mortality also fell in a constant manner. In the same study, however, the death rate for the rice weevil did not increase in a uniform way as the oxygen concentration was reduced in 1% increments from 3% to 0%. Navarro found instead a small dip in the time required for 95% mortality at 1% oxygen and surmised that this may be due to differences in spiracular movements at this particular concentration. He further determined the daily percentage weight loss for the rice weevil at each oxygen level and found that it was highest at 1%. Spiracles open as oxygen concentration falls, but this apparently reaches a maxi-

2

**Figure 1.3a,b**

Effect of temperature on the minimum exposure time to (a) argon and (b) nitrogen required to achieve complete insect mortality at 40% RH and 300 ppm oxygen (Valentín 1993). (Courtesy of Nieves Valentín.)



(a)



(b)
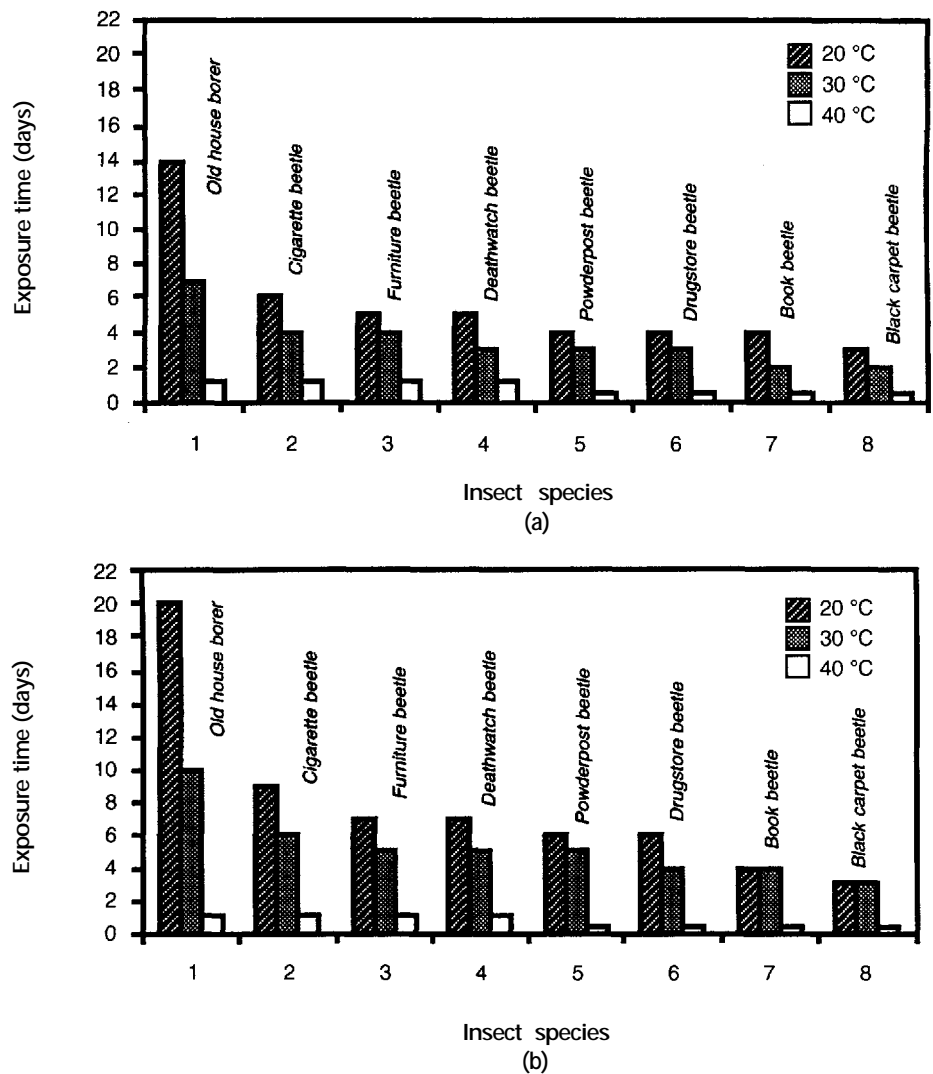
mum at 1%. As the oxygen content continues to drop, the spiracles tend to close. Thus, at 1% oxygen, as at other concentrations, desiccation is the cause of death, and there is no need to invoke an alternate mechanism.

There is a critical level of weight loss associated with mortality. Below this level, only weaker specimens succumb; above it, the general population rapidly perishes. Navarro found that for *Ephestia cautella* pupae, the critical range is from 17% to 25% weight loss. Jay and Cuff (1981) measured weight loss and evaluated mortality for three life stages of the red flour beetle at 1% and 3% oxygen. To achieve mortalities of 90% or higher, weight loss had to reach about 250 mg for twenty-five larvae and 150 mg for twenty-five pupae. Loss of weight was slower at 3% than at 1% oxygen, but when enough time was allowed to bring the weight loss to the critical level, the same high kill rate was achieved. This is additional evidence that anoxia kills by causing dehydration.

There is decreased susceptibility of flour beetles and other insects to anoxia as humidity increases. Studies showing a correlation between weight loss and mortality (Navarro 1978; Jay and Cuff 1981) at relative humidities of at least 54% in an anoxic nitrogen atmosphere suggest that the desiccation mechanism is still in play. Dehydration of the insect occurs even when it is surrounded by high levels of water vapor. The mechanism is twofold. First, nitrogen passes into the insect through its body wall, picks up water from the internal organs as it passes through the thorax and the abdomen, and then carries the water out of the

Figure 1.4

Mortality of red and confused flour beetles exposed 24 hours at 26 °C to 1 % oxygen in nitrogen at four relative humidities (Jay, Arbogast, and Pearman 1971).
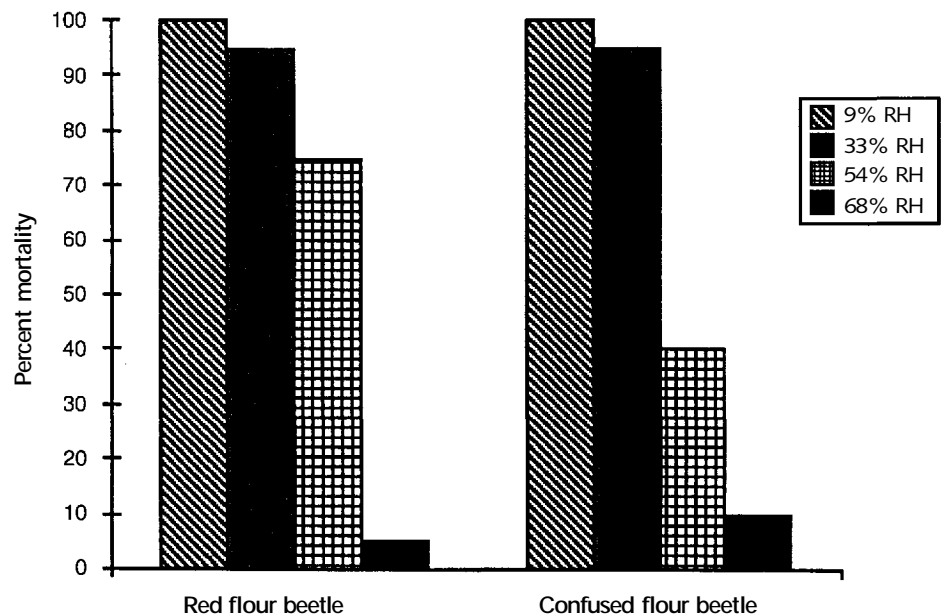


insect through the spiracles. Second, water is kept out of the insect by the epicuticle, that is, the waxy, waterproof covering of the spiracles (Edney 1967).

Argon and helium are even more effective atmospheres for anoxia than is nitrogen, and the explanation involves differences in the permeability of the insect's body wall to these gases. The previously cited work of Valentín, in addition to demonstrating a positive correlation between temperature and mortality rate, also compared the kill rates for eight insect species exposed to argon and nitrogen atmospheres. Under comparable conditions (40% RH, 300 ppm oxygen at 20, 30, and 40 °C), it generally took 50% longer with nitrogen than with argon to reach 100% mortality (Fig. 1.3). AliNiazee (1972) reported that helium also provides a much faster kill than nitrogen. This investigator found that helium generally took only half as long as nitrogen to achieve 97% mortality with red flour beetles and confused flour beetles (Table 1.1).

If these differences in effectiveness are due to the permeability of the inert gases through pores in the body walls of the insects, then these values are in line with the van der Waals radii of helium (1.22 Å), argon (1.91 Å), and molecular nitrogen (2.31 Å). Thus the smallest atoms (helium and argon) move the fastest through the minute pores and dehydrate with corresponding speed; the larger nitrogen molecule takes the longest.

## Anoxia at High Humidities

A mechanism other than desiccation takes over at extremely high humidities where anoxia is still effective, but slower. Donahaye (1990) found that loss of water from red flour beetles exposed to 0.5% oxygen in nitrogen at 95% RH occurred only after the insects were dead. Obviously, a biological pathway other than dehydration has to be involved in this lethal process. Donahaye's research, however, was primarily concerned with a different question: Could species such as the red flour beetle develop a resistance to modified atmospheres? He subjected the beetles to two modified atmospheres—0.5% oxygen in nitrogen, and 20% oxygen and 15% nitrogen in carbon dioxide—at 95% RH until 30-50% remained alive. (The extremely high humidity was employed to suppress the desiccation mechanism.) He repeated the treatment with the offspring of the survivors for over forty generations. Two resistant strains of beetle developed, each resistant only to the specific atmosphere to which it had been subjected. Compared to the original beetles, the resistant strains required nine times the

Table 1.1

Effect of temperature and inert-gas type on the exposure time required to produce 97% insect mortality at 38% RH and 0.05% oxygen concentration (AliNiazee 1972).

| | Temperature | | |
| --- | --- | --- | --- |
| | 15.6 °C | 21.1 °C | 26.7 °C |
| Confused flour beetle | | | |
|    Helium | 12 h | 9 h | 5 h |
|    Nitrogen | 30 h | 15 h | 12 h |
| Red flour beetle | | | |
|    Helium | 12 h | 9 h | 6 h |
|    Nitrogen | 24 h | 13.5 h | 12 h |

exposure before a 50% mortality level was observed. Additionally, insects removed from the modified atmospheres after thirteen generations retained 83% of their resistance after eight generations of exposure to ambient conditions. The type of compensations that enabled the red flour beetles to survive included a decrease in respiration rate, an increase in stored oxygen reserves, physiological changes to prevent water loss, and other biochemical adaptations.

The finding that strains of insects resistant to anoxia readily arise in high humidities raises concerns about generating resistant species both in natural environments and in the course of practicing pest control in museums and libraries. Insects, after all, are believed to have descended from aquatic ancestors that lived under anoxic conditions. A number of larvae and adult insects that are now at home in aquatic surroundings are recent returnees, like ocean-bound mammals, and they have at times readapted to anaerobic respiration. Insect survival without oxygen over extended periods of time is rare but not impossible, and there are examples of transitions from an aerobic to an anaerobic mechanism when the available oxygen drops below a critical level. When this happens, the energy-generating mechanism that sustains life causes the disappearance of body carbohydrates (in the form of glycogen) and fat, and the accumulation of ethanol by fermentation accompanied by less than the expected amount of lactate (AliNiazee 1972). In polluted, nutrient-rich bodies of water, and particularly in ice-covered ponds, the oxygen level may fall to zero for periods of weeks. Under these anoxic conditions, insect larvae living along the bottom depend on anaerobic energy production; several types of water midges, for example, are known to survive for longer than one hundred days in the absence of oxygen.

That these and other hypoxic survivals occur when insects are immersed in water or surrounded by ice suggests that, as in the Donahaye studies, evolution toward resistant species occurred when desiccation was overwhelmed by a supply of water. The evidence indicates that anaerobic ethanol formation in all of these species came about as rather recent adaptations to their unique environments (Zebe 1991).

More common and less drastic adaptations have produced the museum pests that take longest to eliminate by conventional anoxia treatment; these species are characteristically in environments tending toward confinement, dampness, and high carbon dioxide and low oxygen concentrations. Examples would be grain-storage insects, such as the cigarette beetle and dermestids, and insects that burrow deeply into books and wood. Still, they are amenable to anoxia; it just takes a little longer. Always, 100% mortality must be the goal for effective pest management. Total kill is far more important than trying to save time by shortening the treatment.

# Chapter 2

# Anoxia as a Conservation Procedure

Separate publications in the same year by Bailey and Gurjar (1918) and Dendy (1918), on the use of airtight storage to rid grain supplies of insect pests, marked the beginning of scientific inquiry into anoxia. The 1950s saw increased interest in and development of modified atmospheres for food preservation because of greater awareness of the environmental dangers posed by pesticides. An important stimulus was action by the U.S. Environmental Protection Agency in 1980 approving the use of nitrogen and carbon dioxide for all agricultural products. The degree of attention and development given this technology was summarized in a major review by Calderon and Barkai-Golan (1990). This methodology, however, is not widely used for preserving foodstuffs in large-scale commercial operations because of the expense of long treatment times and the development of safer procedures using methyl bromide and phosphine.

## From Food Preservation to Conservation

Custodians of cultural property also have had a long history of concern about the damage done by insects. In the 1980s, as interest in modified atmospheres for food preservation peaked, conservation scientists began to study how this technology could be adapted to museum needs. Fumigation with toxic gases is obviously not a safe option within public buildings, but the long treatment times required for nontoxic gases are generally acceptable. Although a carbon dioxide atmosphere had been favored for the preservation of foodstuffs, conservators saw more advantages in using nitrogen with low oxygen concentrations to treat cultural property because anoxia provides a higher degree of inertness and is easier to establish for small-scale operations. But the transfer of procedures from food preservation to museum use did not come easily; some important lessons had to be learned first.

Early recognition that modified atmospheres could be used to control pests for conservation purposes appeared in *Approaches to Pest Management in Museums,* a 1985 book by Keith Story. He wrote, however, that for practical considerations, modified atmospheres should be based on carbon dioxide rather than on nitrogen containing essentially no oxygen. Story did not describe any use of modified atmospheres other than those involving very large volume containments. Indeed, large-scale work is more readily done with moderate (50-60%) levels of carbon dioxide rather than with nitrogen having very low concentrations of oxygen because there is always the possibility of air leaks in large chambers. According to Story, such levels of carbon dioxide at 60% RH and 21 °C will kill all life stages of most insects in 4 days. Earlier, Arai and Mori (1980) had examined anoxia as a means of simultaneously treating fungal growth and insect infestation in cultural property. It took 20 days to achieve a 92% kill of rice weevils with nitrogen containing 1000 ppm to 2000 ppm (0.1-0.2%) oxygen in nitrogen at 55-70% RH. The fungal growth, on the other hand, was inhibited but not killed. This led the authors to recommend fumigation with insecticides for a more rapid extermination. However, their description of variations in the oxygen concentration in the laminated polymer bag they used suggests that the bag was excessively permeable to oxygen. In 1990 deCesare described in a library newsletter how books can be rid of insect pests by holding the works under an argon atmosphere in plastic bags or trash cans. Unfortunately, he gave no details on critical aspects of treatment such as insect types, oxygen concentration, temperature, length of treatment, or level of mortality achieved. The work by deCesare pointed in the right direction, but its qualitative nature did not offer a procedure that could be used by conservators with any assurance.

Conservation scientists at two separate institutions, the Australian Museum in Sydney and the Getty Conservation Institute in Los Angeles, first recognized and worked through these problems. Mark Gilberg joined the Conservation Division of the Australian Museum in the spring of 1987. His first assignment was to find

a substitute for ethylene oxide as a fumigant to rid museum objects of insect pests. At the time, there was considerable interest and activity in Australia in using modified atmospheres for suppressing insect infestations in grain storage (Annis 1987), and this appeared to be a promising direction for the scientists at the Australian Museum. Gilberg was able to enlist the help of Jonathan Banks, senior principal research scientist with the Stored Grain Research Laboratory in the Commonwealth Scientific and Industrial Research Organization. He had been involved in research on modified atmospheres for insect control for many years, with both low-oxygen systems and high carbon dioxide atmospheres (Banks 1979; Banks and Annis 1990). Banks quickly saw how the technology developed for grain storage could be adapted to the preservation of cultural property and made a number of valuable suggestions. He recommended the nitrogen anoxia approach because he felt that insects were more likely to build up a resistance to the toxicity of a carbon dioxide atmosphere. Banks also acquainted Gilberg with the properties of oxygen-impermeable plastic films for containment and with the use of commercial oxygen scavengers. The body of data that had been compiled over fifty years of food-preservation studies by many researchers was an invaluable information resource for museum personnel who were adapting anoxia to their needs. However, Banks's intercession and instructions provided an immediate, direct transfer of this technology between the two disciplines, saving conservation scientists several years of research effort.

## Early Museum Anoxia Studies

Two years after joining the Australian Museum, Gilberg published the first report describing effective control of insects by anoxia in a manner relevant to museum needs (1989a). He studied cigarette and drugstore beetles of the Anobiidae family and carpet beetles of the Dermestidae family, which are common museum and grain storage pests, as well as powderpost beetles and webbing clothes moths. Gilberg maintained insect colonies in glass containers at 0.4% oxygen concentration, 65-70% RH, and a slightly elevated temperature of 30 °C. He achieved 100% mortality with all species and life stages in 7 days. Gilberg considered this temperature, selected to shorten the required exposure time, to be the maximum safe level for museum objects. He also noted that studies with the rice weevil showed that this anoxia-resistant grain storage pest was completely killed in 1% oxygen at 29 °C in 3 weeks, and he suggested that this length of time might define an upper exposure limit for museum work. Gilberg (1991) repeated his work with these insects to confirm that 3 weeks of exposure would reliably provide total kill of all life stages. He further promoted the use of chemically reactive oxygen absorbers in enclosed systems as a convenient and effective way of bringing oxygen concentrations down below 1% (1990). Gilberg's work has led to the widespread use of an oxygen scavenger based on a highly reactive, high-surface-area ferrous compound that is commercially packaged and sold by the Mitsubishi Gas Chemical Company under the Ageless trade name. Details on using a scavenger to maintain a very low oxygen level are discussed in chapter 3.

When Gilberg started his anoxia studies, work was proceeding at the Getty Conservation Institute on the design and construction of nitrogen-filled, hermetically sealed display and storage cases, initially for holding the Royal Mummies of the Egyptian Antiquities Organization that are now on display at the Egyptian Museum in Cairo. It was hoped that by drastically reducing the level of oxygen in the cases, and then by controlling it as well as the RH, museums would have a cost-effective way to prevent deterioration of extremely old organic objects such as mummies. Such objects are affected by changes in humidity, biological attack, thermally and photochemically induced oxidation, and gaseous and particulate pollutants. The prototype cases developed by the Getty Conservation Institute were built so that under normal conditions they could be maintained at less than

2% oxygen concentration without major maintenance for up to ten years (Maekawa, Preusser, and Lambert 1993).

When Nieves Valentín joined the Scientific Program of the GCI in 1987, she was asked to evaluate the effectiveness of nitrogen anoxia for deterring the biological deterioration of ancient artifacts. Work presented in August 1989 (Valentín and Preusser) described studies in which colonies of bacteria and fungi on parchment were exposed to an atmosphere of 1% oxygen in nitrogen at 33-43% RH for 3 weeks. Both fungi and bacteria were deterred from further growth. The effect on insects was more dramatic: nitrogen anoxia eliminated all stages of the life cycle of the fruit fly. The exposure time required to do this decreased as the RH fell, but it was inversely related to temperature. At room temperature, 75% RH, and 5000 ppm oxygen, all life stages, including eggs, were killed in 80 hours. This time requirement was only 30 hours at 30 °C and 40% RH. Additionally, studies with a common museum pest, the drywood termite, demonstrated that infestations in wood could be totally eliminated by containment in aluminized plastic bags filled with nitrogen containing less than 1% oxygen at 25 °C and 40% RH.

## Determining Kill Times

These initial studies showed that by diligently maintaining a nitrogen atmosphere with a very low oxygen content, a complete kill of all life stages for some pests could be obtained in reasonable periods of time. A good start had been made in defining process requirements. Still, there were a number of material problems to be solved before general procedures could be developed that were easy and effective for conservators to carry out in most museums. Also of paramount concern was determining the minimum amount of time required for an assured 100% mortality rate for the most destructive museum pests and the relationship of this time to operating parameters. This assured kill time is critical for defining both the cost and usefulness of anoxia treatment. For example, the cost of monitoring oxygen concentration and maintaining airtight chambers and tents, two very important practical matters, can be reduced significantly if mortality rates at higher oxygen concentrations are known. These considerations had been examined for grain storage problems much earlier. In 1977 Banks and Annis, noting that the rice weevil was the most tolerant grain pest in low-oxygen atmospheres, reported the exposure times required for the complete kill of this species in 1% oxygen in nitrogen over a range of temperatures from 15 to 35 °C. An exposure of 6 weeks was recommended at 20 °C, but this fell to 2 weeks at 30 °C. Other pest groups, such as Tribolium, Oryzaephilus, and Rhyzopertha, required shorter exposures, perhaps half as long. However, if there were any doubt about the species present, it was suggested that the longer times be used. The addition of carbon dioxide appeared to allow some short, but undefined, reduction in exposure time.

The difficulty of answering questions about treatment times for art objects was first raised by Gilberg in 1990 and subsequently addressed in a study by Rust and Kennedy in 1993 (also Rust et al. 1996) that was sponsored by the Getty Conservation Institute. In selecting the pest species for their study, Rust and Kennedy used surveys from natural history and art museums that indicated that beetles belonging to the families Anobiidae and Dermestidae and moths belonging to the family Tineidae are major pests. Tables 2.1 and 2.2, taken from their report, summarize the common insect pests found in museums in North America. The twelve species that they studied were chosen primarily from these tables. Several other important species were not included in the study because they could not be legally brought into the United States. For this work, an anoxic atmosphere at 55% RH was produced by bubbling a sidestream of dry, prepurified nitrogen containing 20 ppm oxygen through water and then recombining

Table 2.1

Insect pests found in North American museums.[a]

| Order | Family | Species | Common name |
|---|---|---|---|
| Coleoptera | Anobiidae | *Lasioderma serricorne* | Cigarette beetle |
| | | *Stegobium paniceum* | Drugstore beetle |
| | | *Anobium punctatum* | Furniture beetle |
| | | *Xestobium* spp. | Deathwatch beetle |
| | Bostrichidae | *Dinoderus minutus* | Bamboo powderpost beetle |
| | Cleridae | *Necrobia rufipes* | Redlegged ham beetle |
| | Dermestidae | *Anthrenus flavipes* | Furniture carpet beetle |
| | | *Anthrenus verbasci* | Varied carpet beetle |
| | | *Attagenus megatoma* | Black carpet beetle |
| | | *Dermestes lardarius* | Larder beetle |
| | | *Thylodrias contractus* | Odd beetle |
| | | *Trogoderma indusum* | Cabinet beetle |
| | Lyctidae | *Lyctus* spp. | Powderpost beetle |
| | Tenebrionidae | *Tribolium confusum* | Confused flour beetle |
| Lepidoptera | Tineidae | *Tinea pellionella* | Casemaking clothes moth |
| | | *Tineola bisselliella* | Webbing clothes moth |
| Dictyoptera | Blattellidae | *Blattella germanica* | German cockroach |
| | | *Supella longipalpa* | Brownbanded cockroach |
| Thysanura | Lepismatidae | *Lepisma saccharina* | Silverfish |
| | | *Thermobia domestica* | Firebrat |

[a]Compiled from Kingsolver(1981) and Beauchamp, Kingsolver, and Parker (1981) as referenced by Rust and Kennedy (1993).

wet and dry streams with appropriate metering as described in chapter 4. Twelve insect exposure chambers built of quarter-inch-thick acrylic sheets (Fig. 2.1), each 1.5 ft$^3$ in capacity, were flushed with this gas. Each was an independent experimental chamber containing two packets of the commercial scavenger Ageless Z-1000, to maintain a very low concentration of oxygen, as well as a small pan of aqueous saturated magnesium nitrate solution, to ensure an RH of 55%. A Teledyne trace-oxygen analyzer with a fuel-type sensor was used to determine the oxygen level. Colonies of all life stages of the selected species were thus maintained at 25 °C and 55% RH in a nitrogen atmosphere containing less than 1000 ppm oxygen.

The kill rates were first determined for an expanded series of time periods likely to be long enough to provide total mortality. They were then reexamined using a

Table 2.2

Survey of natural history museums of North America.[a]

| Pests | Percentage of total respondents | |
|---|---|---|
| | Pests encountered | Greatest threat |
| Dermestid beetles | 70 | 49 |
| Silverfish | 30 | 2 |
| Cockroaches | 26 | 3 |
| Anobiid beetles | 13 | 6 |
| Tenebrionid beetles | 12 | 2 |
| Mites | 4 | — |
| Moths | 0 | 11 |
| Unspecified | 30 | — |

[a]Compiled from Bell and Stanley (1981) as referenced by Rust and Kennedy (1993).

Figure 2.1
Insect exposure chambers.
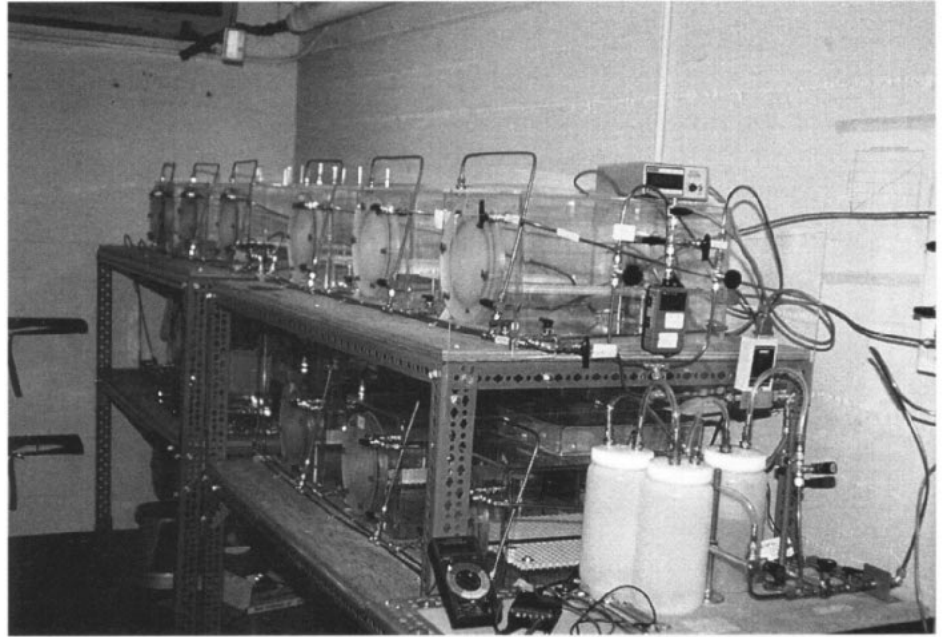
smaller number of exposure periods based on the time in the first series where 100% mortality of all life stages occurred. For example, the initial screening of the webbing clothes moth involved five exposure periods of 3, 24, 48, 72, and 96 hours. The 48-hour period appeared to provide 100% kill of all eggs, and in the subsequent confirming study, exposures were limited to 48, 72, and 96 hours. However, in one set the 72-hour period produced only 98.7% mortality of the cocoon stage. An exposure of 96 hours produced a total kill of all stages, and this was selected as the recommended time for the webbing clothes moth. In the case of the cigarette beetle, the only time selected for the second-stage evaluation was 192 hours, which equaled the minimum time required to obtain 100% mortality in the first screening, plus 6 hours. The 192-hour period was shown to provide complete kill in four separate tests.

Results obtained by Valentín, Gilberg, and Rust and Kennedy are summarized in Table 2.3. This shows that overall the time required to kill 100% of the insects varied among the species and especially among the developmental stages of a given species. Many of the pest species were completely exterminated with less than 72 hours of exposure, while the eggs of the cigarette beetle required 8 days to guarantee 100% kill. The addition of carbon dioxide to the anoxic gas slightly decreased the exposure needed for total kill, but increasing the temperature was more effective in reducing kill time than was adding carbon dioxide. In more recent work, Rust and Kennedy (1995) examined the effect of oxygen concentrations greater than 1000 ppm on the length of treatment for cigarette beetles and furniture carpet beetles. They found that within this limited sampling, results were moderately dependent on the species. Although the minimum assured kill time for the cigarette beetle is relatively long—8 days at 1000 ppm oxygen in nitrogen at 55% RH and 25 °C—this increases relatively slowly as the oxygen concentration is increased to 6200 ppm. In contrast, furniture carpet beetle larvae, which is the resistant stage for this species, require only 3 days for complete kill at 1000 ppm oxygen under these conditions, but this rises rapidly to 20 days at 6200 ppm oxygen (Fig. 2.2). Above this concentration of oxygen in nitrogen, minimum assured kill times rise sharply for both species. These data suggest that at the defined conditions of temperature (25 °C) and humidity (55%), timely and effective killing of pests by anoxia can be achieved at 3000 ppm oxygen in nitrogen, but not much higher.

**Table 2.3**

Minimum time recorded for assured 100% kill of all life stages of common museum pests.

| Species | Oxygen (ppm) | Inert gas | Temp. (°C) | % Relative humidity | Time (h) | Reference |
|---|---|---|---|---|---|---|
| American cockroach | <1000 | Nitrogen | 25 | 55 | 120 | Rust and Kennedy (1993) |
| Black carpet beetle | 300 | Nitrogen | 30 | 40 | 72 | Valentín(1993) |
| | 300 | Argon | 30 | 40 | 48 | Valentín (1993) |
| Book beetle | 300 | Nitrogen | 30 | 40 | 72 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 48 | Valentín (1993) |
| Brownbanded cockroach | <1000 | Nitrogen | 25 | 40 | 72 | Rust and Kennedy (1993) |
| Cabinet beetle | <1000 | Nitrogen | 25 | 55 | 120 | Rust and Kennedy (1993) |
| Cigarette beetle | 4200 | Nitrogen | 30 | 65-70 | 168 | Gilberg(1989a) |
| | <1000 | Nitrogen | 25 | 55 | 192 | Rust and Kennedy (1993) |
| | 300 | Nitrogen | 30 | 40 | 144 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 96 | Valentín (1993) |
| Confused flour beetle | <1000 | Nitrogen | 25 | 55 | 96 | Rust and Kennedy (1993) |
| Drugstore beetle | 300 | Nitrogen | 30 | 40 | 144 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 96 | Valentín (1993) |
| | 4200 | Nitrogen | 30 | 65-70 | 168 | Gilberg(1989a) |
| Drywood termite | 10,000 | Nitrogen | 22 | 40 | 360 | Valentín and Preusser (1989) |
| Firebrat | <1000 | Nitrogen | 25 | 40 | 48 | Rust and Kennedy (1993) |
| Fruit fly | 5000 | Nitrogen | 30 | 75 | 80 | Valentín and Preusser (1989) |
| Furniture beetle | 300 | Nitrogen | 30 | 40 | 168 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 120 | Valentín (1993) |
| Furniture carpet beetle | <1000 | Nitrogen | 25 | 55 | 72 | Rust and Kennedy (1993) |
| German cockroach | <1000 | Nitrogen | 25 | 55 | 24 | Rust and Kennedy (1993) |
| Larder beetle | <1000 | Nitrogen | 25 | 55 | 96 | Rust and Kennedy (1993) |
| Longhorn borer beetle | 300 | Nitrogen | 30 | 40 | 240 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 168 | Valentín (1993) |
| Powderpost beetle | 300 | Nitrogen | 30 | 40 | 120 | Valentín (1993) |
| | 300 | Argon | 30 | 40 | 72 | Valentín (1993) |
| | <1000 | Nitrogen | 25 | 55 | 120 | Rust and Kennedy (1993) |
| | 4200 | Nitrogen | 30 | 65-70 | 168 | Gilberg(1989a) |
| Rice weevil | 10,000 | Nitrogen | 20 | 12 | 1000 | Banks and Annis (1977) |
| | 10,000 | Nitrogen | 26 | 12 | 500 | Banks and Annis (1977) |
| Varied carpet beetle | 4200 | Nitrogen | 30 | 65-70 | 168 | Gilberg(1989a) |
| Webbing clothes moth | <1000 | Nitrogen | 25 | 55 | 96 | Rust and Kennedy (1993) |
| | 4200 | Nitrogen | 30 | 65-70 | 168 | Gilberg(1991) |
| Western drywood termite | <1000 | Nitrogen | 25 | 55 | 96 | Rust and Kennedy (1993) |

The second largest group in Table 2.3 was provided by Valentin (1993), who surveyed museums and archives in northern Europe and the Mediterranean area to determine the insects most prevalent in historic collections. The old house borer *(Hylotrupes bajulus,* which is known by several other common names, including longhorn borer beetle) and the furniture, drugstore, deathwatch, and black carpet beetles were found in cellulose materials in museums, while the furniture, drugstore, powderpost, and book beetles were isolated from books and bundles of documents taken from archives. The cigarette beetle was found frequently in plant collections. Samples of different life stages of each species were placed with wood or paper in low-permeability, poly(vinylidene chloride) bags and purged with pure humidified nitrogen or argon for 8 hours. Separate samples were held at 20, 30, and 40 °C for periods of 1-30 days. In all cases the oxygen

**Figure 2.2**
Effect of oxygen concentration on minimum time for assured 100% mortality of cigarette beetle and furniture carpet beetle at 25.6 °C and 55% RH (Rust and Kennedy 1995).
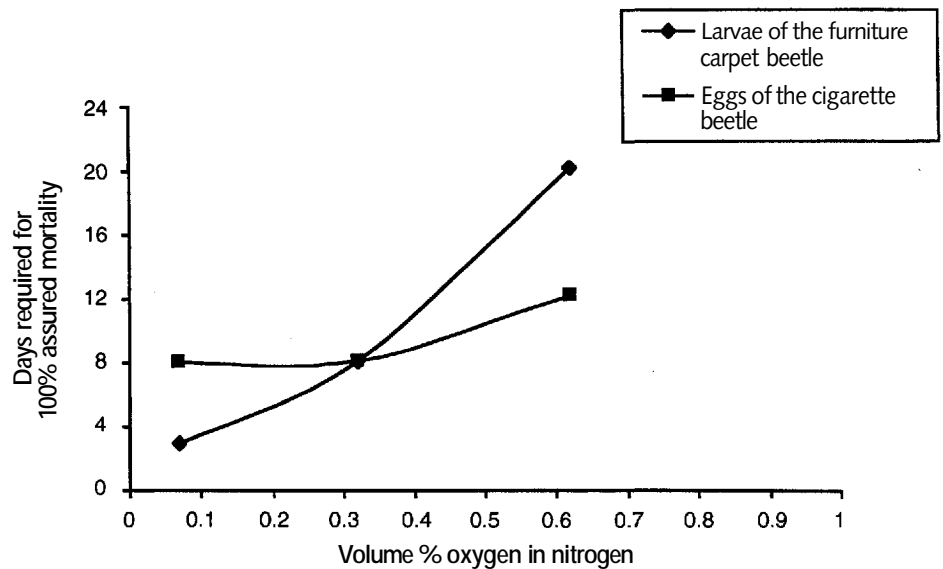


level was held between 200 ppm and 500 ppm. Following treatment, all samples were exposed to air under ambient conditions for seven months to disclose any sign of life. Valentín's results were in agreement with those of Rust and Kennedy. All life stages of the furniture and powderpost beetles, including eggs, were eliminated in 3-5 days with 300 ppm oxygen in nitrogen at 30 °C and 50% RH. Rust and Kennedy found that exposure times of 72 hours or less were sufficient to treat infestations of the furniture carpet beetle and the powderpost beetle. Both groups found that the cigarette beetle was more tolerant of anoxia and required longer exposure. Rust determined that this species was eradicated only after 8 days at 25 °C and 50% RH. As would be expected, Valentín found that longer exposures were needed at a lower temperature, for example, 9 days at 20 °C and 40% RH.

By using argon instead of nitrogen, the required exposure time is decreased about 30%, but a modest increase in temperature—going from 30 to 40 °C— will lower treatment time as much as 85-90%. Valentín determined that the complete kill of all life stages of the species she studied could be achieved in less than 24 hours by operating at 40 °C. Although much shorter treatment times resulted at elevated temperatures, little advantage is taken of this in current practice. Most treatments are done at room temperature, typically between 20 and 25 °C. Gilberg and Roach (1992) describe a program of treatment at the Australian Museum where objects were kept in bags under nitrogen in a thermal cabinet at 30 °C. Except for the Valentín study, there is little information about work done at temperatures as high as 40 °C.

## Thermal Techniques

There is a question of how high a temperature conservators can safely use in disinfestation treatments. Operating above ambient temperature requires more equipment and attention, but the principal reason for not doing treatments above 30 °C is concern for the well-being of objects. However, much of this fear may be without scientific justification. There is a growing body of literature suggesting that a large portion of general museum collections can be heated to temperatures approaching 60 °C without fear of damage, if adequate moisture is maintained (Strang 1992, 1995; Child 1994; Thomson 1995; Ertelt 1993). This has created an advocacy for the disinfestation of cultural property by strictly thermal means. Strang has shown that for a wide range of cellulosic materials, the risks from thermal techniques are eliminated by ensuring that the heating process does not drive water out of the object. Adequate humidification is

essential and is accomplished by enclosing objects in vapor barriers within the containment system. The Thermo Lignum Company sells equipment to do this and has defined the procedures it believes are required for the safe thermal disinfestation of museum objects. This is done by placing objects in a $4 \times 3 \times 2.7$ m chamber and slowly bringing the temperature to 52 °C. Even though most pest species are killed in one hour at this temperature, the treatment cycle takes about fifteen hours because the unit is deliberately heated and cooled slowly. In parallel, moisture is first added to and then removed from the chamber to keep the RH essentially constant. Undesirable thermal side effects in wood, such as shrinkage, cracking, and warping, are eliminated. The Thermo Lignum unit is also designed to operate in an atmosphere of 10% carbon dioxide or in nitrogen containing only 1000 ppm oxygen. If thermal treatment is safe for objects normally maintained in air, it should not pose a threat to them under anoxic conditions.

There are materials whose exposure to temperatures up to 60 °C should be considered with great caution; they include objects coated with soft waxes, ethnographic pieces containing deteriorated collagen, and animal specimens in natural history collections with body oils and fats that will migrate when warmed. The results of work with thermal techniques should encourage conservators, who are thoroughly aware of which objects are prone to heat damage, to conduct anoxia treatments at 30 °C and even somewhat higher, but they should be alert to the need for careful moisture control.

## Killing Burrowed Insects

There is natural concern that insects that have burrowed deeply into wooden objects will be slow to respond to an anoxic atmosphere, and that this could substantially increase the amount of time needed to reach 100% mortality. One approach to determining the additional treatment time is to measure the diffusion exchange rates of nitrogen, oxygen, and air through different types of wood. The porosity (air volume) of most woods when dry is about 45-50%. After a wooden block that has been standing in air is placed in a sealed chamber containing essentially pure nitrogen, the oxygen content within the chamber will rise as the oxygen diffuses out of the pores. The amount of time needed to bring the wood to anoxic conditions throughout can thereby be determined and then used to calculate the additional time required for treating infestation. Lambert and Maekawa evaluate the exchange rate through poplar, oak, and walnut in this manner (Lambert and Maekawa, forthcoming). Wooden blocks (3.8 X 3.8 x 50.4 cm, about 700 cm³), both uncoated and coated along the sides with shellac, were placed in a 32 l acrylic chamber containing 5000 ppm oxygen in nitrogen. The increase in oxygen was followed with a Teledyne trace-oxygen analyzer, which could detect changes to 1 ppm. The time required to get to equilibrium for the six samples, described in Table 2.4, ranged from 9 hours for uncoated poplar to 120 hours for coated walnut. However, the long exchange times are misleading because they were determined for blocks of fresh wood. Insect boring and tunneling open up the structure of wood and dramatically increase the rate of exchange. The time needed to get the oxygen out of

Table 2.4

Time required to completely displace air with nitrogen in dried wood[a] (Lambert and Maekawa, forthcoming).

| | Wood type | | |
| Sample condition | Poplar | Oak | Walnut |
|---|---|---|---|
| Uncoated | 9 h | 11 h | 34 h |
| Coated | 34 h | 48 h | 120 h |

[a]Wood block size: 3.8 x 3.8 X 50.4 cm; chamber size: 32 l; ambient temperature: 23 °C; starting $O_2$% in $N_2$:0.1-0.2%; equilibrium $O_2$% in $N_2$: 0.40%.

infested walnut, for example, fell to as little as one hour for untreated wood and four hours for a heavily coated sample.

In a more direct assault on this problem, Rust and Kennedy (1993) sealed powderpost beetles and western drywood termites inside blocks of wood and compared the time needed for their total demise with the time needed to kill unsealed insects. Beetles of the Lyctidae family, collectively referred to as true powderpost beetles, feed on hardwoods such as oak, walnut, and mahogany. Western drywood termites, one of the most destructive termites in the United States, establish themselves in almost any wood that is not decayed. Rust and Kennedy encased these insects in blocks of Douglas fir, measuring 8 x 8 x 14 cm, that had been cut in half and hollowed in the center to hold small, open-ended, glass cylinders containing the insects. To eliminate air leakage through the sectioning, a sheet of parafilm was sandwiched tightly between the two block halves. There were 1.3-2.5 cm of solid wood surrounding each vial. In control studies with exposed insects, it took 5 days to completely kill all life stages of the powderpost beetle. With the beetles placed inside the wood, 100% mortality was achieved in 5 and 6 days in separate tests. Nearly identical results were obtained with the western drywood termites with and without enclosure in the Douglas fir blocks. Rust and Kennedy concluded that the deep burrowing of insects into wood does not significantly increase the time needed for their complete extermination by anoxia.

Valentín (1993) investigated the exposure times needed to eliminate all insect infestation in wooden pieces, books, and other items in a collection of museum objects. Her results are summarized in Table 2.5. The species studied are active feeders in works of art made of pine, cedar, walnut, oak, mahogany, and chestnut. Column A shows the actual kill times for insects in the infested objects; column B shows the kill times for control insects exposed to comparable environmental conditions. There was little or no difference in required kill time between insects in infested objects and their controls for six of the eleven treatments. At the other extreme, a complete kill of the furniture beetle in polychrome sculptures and a piano ranged from 10 to 14 days, substantially longer than the 4 days needed for this species in the open.

Table 2.5

Environmental conditions used to achieve complete insect mortality in different historic objects exposed to an argon atmosphere (Valentín 1993).

| Infested object size (cm) | Insect species | T (°C) | % RH | $O_2$ (%) | Kill time (days) | |
| | | | | | A Insects in objects | B Control insects |
| --- | --- | --- | --- | --- | --- | --- |
| Books—35 x 25 x 16 | Drugstore beetle, powderpost beetle | 30 | 40 | 0.02 | 4 | 3 |
| Bundled documents—30 x 25 x 15 | Drugstore beetle, book beetle | 30 | 65 | 0.02 | 4 | 3 |
| Sculpture[a]—200 x 80 x 52 | Furniture beetle | 25 | 45 | 0.04 | 10 | 4 |
| Sculpture[a]—130 x 30 x 60 | Furniture beetle | 25 | 45 | 0.03 | 10 | 4 |
| Piano[a]—200 x 100 x 100 | Furniture beetle | 25 | 40 | 0.03 | 14 | 4 |
| Panel[a]—80 x 30 x 15 | Deathwatch beetle | 20 | 50 | 0.02 | 7 | 5 |
| Sculpture[a]—34 x 25 x 20 | Book beetle, furniture beetle | 20 | 50 | 0.02 | 10 | 4 |
| Textiles—135 x 87 x 43 | Black carpet beetle | 30 | 45 | 0.03 | 7 | 2 |
| Panel[a]—175 x 64 x 35 | Old house borer | 20 | 50 | 0.04 | 15 | 14 |
| Frame[a]—75 x 45 x 15 | Old house borer | 25 | 45 | 0.03 | 10 | 10 |
| Plants | Cigarette beetle | 40 | 35 | 0.03 | 1 | 1 |

[a] Polychrome

# Chapter 3

# Methods and Materials

An anoxic environment suitable for treatment of infested objects can be achieved in a variety of ways. There are two basic approaches: static and dynamic. With the static procedure, the more common approach, objects are held under high-purity nitrogen or argon in a tightly sealed container with as little transmission of gas as possible. The oxygen concentration is brought down to anoxic levels by one of three methods. The container is purged with many exchanges of high-purity nitrogen; the oxygen is removed using large quantities of an oxygen absorber; or a combination of purging and absorption is used. With the alternate dynamic approach, an inert gas is continuously passed through the system during the treatment. Oxygen-free nitrogen or argon is used to flush all of the air out of the container by initially using a high purge rate; then, when an oxygen concentration of less than 1000 ppm is reached, the flow is reduced to that needed to maintain the low oxygen level for the duration of treatment.

The use of an oxygen absorber, by itself, to bring the system to anoxic conditions may be the simplest static procedure. The need for cylinders of nitrogen and humidification systems is avoided. The availability of premade treatment bags with good oxygen-barrier properties and simple clamps that provide gas-tight seals should make disinfestation of cultural property by anoxia available at low cost to almost any institution. This method is best suited for treating limited quantities of small objects. More complex is treatment by nitrogen purging followed by sealing, with or without the additional use of an oxygen scavenger. This is the most widely used static method and applies to containments of all shapes and sizes, from small pouches to very large plastic tents as well as rigid metal or plastic chambers, large and small. Humidification is generally used, but not always. The dynamic method has found limited use in special situations, particularly when the object is too large or too awkwardly located to be enclosed in a standard container. In this situation, containment is provided by shaping a barrier film around the object. Because unusual or uneven surfaces, such as floor sections, often become part of the containment, it is generally difficult to obtain the required airtightness. However, gas leakage can usually be brought to a level low enough so that a slow, continuous flow of oxygen-free gas through the system can be maintained effectively and economically. Humidification must be used with this dynamic approach. What is sometimes considered to be a third procedure, called the static-dynamic method, is an intermediate approach where the purging gas is allowed to pass through the system very slowly.

A number of early workers in the field recognized the possibilities of these treatment methods and described and compared various approaches and systems in research papers. Getty workers (Hanlon et al. 1992; Daniel, Hanlon, and Maekawa 1993) described the characteristics of each method and first proposed the terms *static, static-dynamic,* and *dynamic.* Koestler (1992) used a dynamic system to treat a sixteenth-century Andrea del Sarto panel painting that was infested with drywood termites. The panel, resting on a flat table, was covered with a large sheet of barrier film taped to the surface of the table. This containment system was then flushed with humidified gas introduced through a port at one end and allowed to escape through another port at the far end. The treatment was successful but used forty cylinders of gas over twenty days. In the same article, Koestler described three static-dynamic treatments. These used a containment chamber fabricated from a utility storage cart made of heavy-gauge polyethylene; a Rentokil minifumigation bubble; and handmade pouches of a barrier film made with an ethylene-vinyl acetate copolymer. Treatments with the pouches combined the use of a nitrogen purge and oxygen scavengers.

Valentín (1993) also evaluated the efficacy of the static-dynamic method in three different containment systems: plastic bags of low oxygen permeability; a tiny (80 $cm^3$), rigid vacuum chamber; and a 6.2 $m^3$ fumigation bubble made of

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poly(vinyl chloride)-reinforced polyethylene. Humidification was used with each system, but oxygen scavengers (thirty packets of Ageless Z-2000) were added only to treatments in the fumigation bubble. Valentín claims that all three systems could provide satisfactory treatment.

Each variation in approach has its own advantages, disadvantages, and special requirements, but there are also critical needs in common. Each requires barrier films with low oxygen permeability; oxygen scavengers with high capacity and good absorption potential; inexpensive nitrogen supplies with a very low oxygen content; and effective methods of containment closure. These will be discussed in detail in the sections that follow.

### Barrier Films

Most work with modified atmospheres for insect control is done today with flexible containers—that is, in pouches or bubbles made of plastic film. After initial research using glass jars and rigid, thick-walled polymer boxes had demonstrated the feasibility of eliminating insect infestations using oxygen deprivation, polymer-film technology was ready, and products were available to make anoxia a convenient procedure. Vapor-barrier laminates had been used for some time on a vast commercial scale with perishables to seal in freshness and hold out oxygen and odorants and other causes of spoilage. From their earliest availability, these films were designed to be heat-sealable and good water barriers. John Burke (1992) has written a very useful review outlining the extent of the application of barrier films to conservation practice. He points out, for example, that museums have been lining shipping crates with aluminized polyethylene (Marvelseal 360) since the early 1980s. In 1986 the Oakland Museum sent on tour an exhibit of six hundred silver objects of local origin wrapped with this material. They were returned essentially untarnished after two years. Thus, vapor-barrier films had found their way into a wide range of conservation applications even before they were considered for insect control.

To carry out anoxia treatments in plastic bags it is necessary to get the oxygen out and keep it out of the bags; this makes low oxygen permeability an extremely important film property. It is just as important that only one surface of a film sheet soften sufficiently on heating to provide good adhesion to another surface and form a secure, gastight seal. Strength is also a critical property—the bag should be tear and puncture resistant and not likely to form pinholes. Polymer chemistry makes possible a high degree of property specificity for barrier films. This is achieved by the use of unique monomers; the correct design of polymerization conditions; and the appropriate physical processing of the films, for example, by annealing or stretch-orientation. However, there is a limit to the extent to which a combination of desirable properties can be built into a single film. On the other hand, there is a synergism that is created when several films chosen for specific values are sandwiched into a laminate. Barrier films used for pest control are all laminates of at least three single-composition polymer films that impart specific properties, the most important of which are low oxygen permeability, mechanical strength, and heat-sealability. Other properties, of varying importance, are low water-vapor transmission, transparency, printability, antistatic qualities, and fire resistance. Of course, low cost and availability are also important. Table 3.1 lists polymer films that are important for designing barrier laminates for insect control and shows their oxygen permeability. Table 3.2 shows how these films are combined into the barrier laminates that have become the most important for this work.

As seen in Table 3.1, polymers based on vinylidene dichloride and copolymers of ethylene and vinyl alcohol produce films of extremely low oxygen transmission.

Table 3.1

The composition and oxygen permeability of films.

| Composition | Abbreviation | Monomers | Oxygen[a] permeability | Outstanding quality |
|---|---|---|---|---|
| Vinylidene dichloride monopolymer and copolymers with vinyl chloride | PVDC | Vinylidene dichloride, vinyl chloride | 0.16-2.46 | Very low oxygen permeability |
| Ethylene-vinyl alcohol copolymers (dry) | EVOH | Ethylene, vinyl alcohol | 0.11-0.80 | Very low oxygen permeability |
| Ethylene-vinyl alcohol copolymers (100% RH) | EVOH | Vinyl alcohol, ethylene | 8-16 | Very low oxygen permeability |
| Nylon-6 | NYL | Caprolactam | 40 | Low oxygen permeability, good strength |
| Poly(ethylene terephthalate) | PET | Ethylene glycol, terephthalic acid | 56 | Low oxygen permeability |
| Poly(chlorotrifluoroethylene) | Aclar | Chlorotrifluoroethylene | 141 | Low oxygen permeability |
| Poly(vinyl chloride) | PVC | Vinyl chloride | 200 | Low oxygen permeability, good strength |
| Polypropylene (oriented) | PPO | Propylene | 7800 | Heat-sealable |
| Polypropylene (cast) | PP | Propylene | 3700 | Heat-sealable |
| Polyethylene (low density) | PE | Ethylene | 4800 | Heat-sealable |
| Polystyrene | PS | Styrene | 5200 | — |
| Polypropylene laminate with aluminum | PPAL | Propylene, aluminum | 3 | Heat-sealable, low oxygen permeability |

[a]Oxygen permeability in $cm^3$ x mil/($m^2$ x days x atm).

More recent laminates have been developed using these polymers as core oxygen barriers. A second group of films in a somewhat higher—but still effective—oxygen transmission range consists of poly(vinyl chloride); poly(chlorotrifluoroethylene), which is known commercially as Aclar; nylon-6; and poly(ethylene terephthalate). Films of nylon-6 and poly(ethylene terephthalate) are also tough with high mechanical strength and are used primarily for these properties. Although pure Aclar has good, but not outstanding, oxygen-barrier values, composites containing this material do have very low oxygen permeability. However, high cost has been a deterrent to the use of laminates containing Aclar. Additionally, some ambiguity has crept into the use of the term "Aclar." It properly refers to the polymer poly(chlorotrifluoroethylene), but a number of laminate

Table 3.2

Barrier laminates.

| Core (oxygen-barrier film) | Composition[a] | Source[b] | Trade name | Film thickness[c] (mil) | Oxygen permeability[d] |
|---|---|---|---|---|---|
| PVDC | PET/PVDC/PE | 1 | Keepsafe (bags) | 2.5 | 7.1 |
| PVDC | PET/adh/PVDC/adh/PE | 2 | Filmpack1193 | 4.9 | 0.28 |
| Aclar | PET/PE/Aclar/PE | 3 | Film-O-Rap 7750 Filmpack 1177 Aclar | 4.5 | 50 |
| Aluminum | NYL/Al/PE | 3 | Marvelseal 360 | 5.3 | 0.01 |
| Aluminum | PPO/PE/Al/PE | 4 | Shield Pack Class A | 5.0 | 0.01 |

[a]Adh = unspecified adhesive.
[b](1) Keepsafe Systems, Inc., Toronto, Canada; (2) Ludlow Corporation, Homer, La.; (3) Allied Signal Inc., Morristown, N.J.; (4) Shield Pack Inc., West Monroe, La.
[c]Measurements made at the Getty Conservation Institute.
[d]Oxygen transmission in $cm^3$ x mil/($m^2$ x days x atm), measurements made at the Getty Conservation Institute.

**Table 3.3**

Comparative cost of barrier films.

| Polymer | Cost (1996 US$/m$^2$) |
|---|---|
| Laminates based on Aclar | 6.10-11.00 |
| Marvelseal 360 | 1.66-2.00 |
| Filmpack1193 | 5.30-7.44 |

fabricators use this term broadly to designate composite barrier films containing poly(chlorotrifluoroethylene). The polyolefins, polyethylene and polypropylene, are poor oxygen barriers but are incorporated into laminates for their needed adhesion and sealing properties. A laminate formulation with a flexible aluminum film is included in Table 3.1 to show the effectiveness of this inexpensive material as a core barrier. Burke describes aluminum films as being formed of very small platelets that are deposited on top of each other to create an extremely long and difficult pathway for transiting gas molecules. Pinholes form easily in thin aluminum foil, so it is not a perfect solution.

Information on the chemical and physical properties of barrier films is important; it serves to educate and convince conservators of the large and significant differences in oxygen permeability that occur as a function of film composition. This is why the selection of an appropriate polymer is critical. Common polyethylene bags, for example, are useless because of their high oxygen permeability. Conservators who favor producing their own bags, or who must put together odd-shaped constructions out of barrier-film sheets and have to shop for the most cost-effective laminates, need precise information on composition and properties as well as on cost. The costs of three important commercial laminates are compared in Table 3.3. For those who buy ready-to-use laminate bags, there is still a need for this information in order to purchase wisely.

## Gas Supplies

Nitrogen is widely available from a number of vendors in convenient, but heavy, metal cylinders. The Linde Division of the Union Carbide Company is a prime producer of industrial- and research-grade gases, marketing them through a network of distributors. They offer a range of nitrogen grades: *research, ultra-high purity, prepurified, high purity, extra dry, oxygen free,* and zero grades. Argon is provided in about the same range of grades. The most suitable grades of nitrogen for insect control are those designated as "prepurified" and "high purity," which contain 5 ppm and 10 ppm oxygen, respectively (Table 3.4). A number of companies, particularly in industrial areas, market cylinders of nitrogen suitable for anoxia treatments under a variety of grade descriptions and will generally supply written analyses providing the oxygen content for any grade. Industrial-grade nitrogen, usually offered as 99.7% nitrogen, is widely available and is lower in cost than the research grades. Although its use may be limited to initial purging stages, this grade often shows a surprisingly low oxygen content on analysis (e.g., 20 ppm) and is certainly useful for treatments. For fumigation with carbon dioxide, which is typically used at 60-70% for insect control, the 99.7% concentration of the "bone dry" grade is quite adequate and provides a relatively low gas-volume cost. Other expenses associated with using gas cylinders, based on 1996 prices, are approximately US$240 for a regulator and a tank rental fee of about US$6 a month.

The use of larger containment systems requiring greater amounts of nitrogen will lead to the accumulation of a large number of cylinders during each treatment. Much of the awkwardness of handling a large quantity of tanks can be avoided

Table 3.4

Price comparison of argon, nitrogen, and carbon dixode.[a]

| Gas | Cylinder size | Grade | Purity (%) | Weight (lb) | Oxygen (ppm) | Water (ppm) | Gas volume[b] (ft$^3$) | Cost (US$/cylinder) | Approx. cost (US$/100 ft$^3$) | Approx. cost (US$/m$^3$) |
|---|---|---|---|---|---|---|---|---|---|---|
| Nitrogen | T | Prepurified | 99.998 | 22 | 5 | 3 | 304 | 46.00 | 15.00 | 5.10 |
| Nitrogen | T | High purity | 99.99 | 22 | 10 | 3 | 304 | 37.00 | 12.00 | 4.08 |
| Argon | T | Prepurified | 99.998 | 34 | 3 | 3 | 336 | 65.00 | 20.00 | 6.80 |
| Argon | T | High purity | 99.996 | 34 | 5 | 3 | 336 | 50.00 | 15.00 | 5.10 |
| Carbon dioxide | K | Bone dry | 99.7 | 50 | — | — | 430 | 15.00 | 3.50 | 1.19 |
| Liquid nitrogen | — | — | 99.99-99.999 | 273 | 10 | 3 | 3495 | 90.00[c] | 2.60 | 0.90 |

[a] Data and prices from Gilmore Cyrogenics, El Monte, Calif., February 1,1996.
[b] At standard temperature and pressure.
[c] Includes monthly rental for container.

by connecting them in parallel on a manifold. Even so, working with five to thirty heavy cylinders can be expensive, physically demanding, and dangerous. This problem can be circumvented by using a nitrogen generator, which provides large volumes of oxygen-free gas at low cost after purchase of the generator. Units, such as the Prism generator manufactured by the Air Products and Chemicals Corporation, are available on a turnkey basis in many countries. The Prism nitrogen generator, model 1300, uses a membrane to separate high-purity nitrogen from air and can supply 41 m$^3$ of nitrogen with less than 100 ppm oxygen per day. It consists of an air compressor, a series of filters, and a trace-oxygen analyzer in the output stream. The air is compressed and passed through an early filter system to remove water, dust particles, and gaseous pollutants, and then sent through the membrane filter where separation of the nitrogen stream occurs. The system can produce nitrogen of 95-99.99% purity, although the highest purity is achieved at the lowest flow rate. In 1996 the unit cost about US$18,000 with a 230 VAC 3 phase motor/compressor and incurred expenses of US$200-US$300 per year for replacement filters and the sensor in the oxygen analyzer. In studies at the Getty Conservation Institute, Maekawa and Elert (1996) found the Prism nitrogen generator easy to operate and reliable over ten weeks of continuous usage, although the compressor was somewhat noisy. The model was greatly oversized for the 10m$^3$ bubble under test but it would be quite satisfactory for a 100 m$^3$ chamber. Adding a pressurized storage tank would enhance the cost-effectiveness of this system.

Another convenient and inexpensive source of large volumes of nitrogen gas is liquid nitrogen. The conventional 160 l tank of liquid nitrogen supplies 9.9 x 10$^4$ l of gas containing 20 ppm oxygen. This volume is equivalent to that provided by twelve T-cylinders. The tank contains internal heat exchangers, which vaporize the cold liquid and produce flow rates of more than 100 l min$^{-1}$, although rates of around 30 l min$^{-1}$ are more convenient. One tank of liquid nitrogen easily purges a 10 m$^3$ barrier-film tent to under 1000 ppm oxygen. Using liquid nitrogen requires no mechanical components and little maintenance, and it is inexpensive. A considerable amount of ice forms on the top of the tank when a flow rate of 34 l min$^{-1}$ (49 m$^3$ per day) is maintained for a long period. This may be somewhat inconvenient but it is not a hazard. The tank has a limited shelf life of about one month because nitrogen gas is continuously and automatically released to avoid an excessive pressure increase in the tank at room temperature.

## Oxygen Scavengers

The availability of a powerful and effective oxygen scavenger, which is based on the conversion of iron to iron oxide, has been an important factor in the development of anoxia treatments for pest control. Among a number of companies offering similar products are Atco SA, a French company operating through distributors in Europe, and the Mitsubishi Gas Chemical Company, a Japanese firm that markets its Ageless oxygen absorbers worldwide. Ageless is the more widely distributed and discussed absorber and will be the basis for most of the discussion on oxygen scavengers in this book. This material was brought to the attention of Mark Gilberg in 1990, when he was at the Australian Museum, by scientists at the Stored Grain Research Laboratory in Canberra. After some evaluation of Ageless, Gilberg used it in his museum pest control projects and brought samples to the Getty Conservation Institute, where it was studied for use in hermetically sealed, inert gas-filled display and storage cases (Lambert, Daniel, and Preusser 1992). A Mitsubishi product bulletin issued in 1994 describes the properties, uses, and limitations of this material. Ageless is marketed for inclusion in products for retail consumption, primarily food in airtight containers, to bring the oxygen concentration below 0.01% and thereby stop oxidative deterioration. The bulletin claims that the low-oxygen atmosphere will eliminate mold, bacteria, and insect infestation and prevent the growth of aerobic microorganisms. No conditions are given. Small packets of Ageless can be found in sealed containers of common foods like coffee. The widespread use of this material in food packages is an indication of its nontoxicity and safety.

The Ageless packet contains a mixture of iron, potassium chloride, water, and some type of zeolite—one of a class of natural aluminosilicates that selectively absorb water and other small molecules. Oxygen is removed by reacting with a highly reactive form of metallic iron, which is probably made by the hydrogenation of iron oxide. The oxidation is exothermic, and packets can get very warm. Conservators who plan to work with Ageless should experience this thermal effect for themselves by cautiously feeling a fresh packet of Ageless Z-2000 over several hours of exposure to air.

Water is required for the reaction of iron with oxygen as seen in the following equations:

$$4Fe + 3O_2 + 6H_2O \rightarrow 4Fe(OH)_3$$
$$4Fe(OH)_3 \rightarrow 2Fe_2O_3 \cdot (H_2O)_3$$
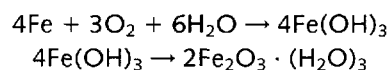
There is generally enough water available in the packets to enable Ageless to take up oxygen in most situations. However, in low humidities moisture will leave the packet, and the reaction will slow; it may essentially stop if the atmosphere is too dry. The French manufacturer, Atco, claims that this does not happen with its scavenger product.

Ageless is sold commercially in six forms, which are tailored to specific end uses. The product conventionally used for pest control is type "Z," designed for use in relative humidities of 50-85%. Ageless Z is supplied in nine different packet sizes, which are graded and labeled to show how many cubic centimeters of oxygen the packet will absorb. Thus, the largest size, Z-2000, will absorb 2000 cm$^3$ or about 16 g of oxygen, which is the amount of oxygen in 10 l of air. The Atco product is similarly designated; Atco LH3000 will absorb 3000 cm$^3$ of oxygen. The conservator can determine the number of packets required for a treatment by the oxygen-removal capacity of the scavenger. To maintain the oxygen concentration at a predetermined level with Atco LH or Ageless, it is also necessary to measure the rate at which oxygen leaks into a sealed unit filled with

an inert gas. This is easily done with a sensitive low-oxygen-concentration ana-lyzer. The number of packets, N, of absorber is then calculated from the equation

$$N = \frac{80 \times L \times V}{C \times D}$$

where $L$ is the leak rate of the unit (ppm $O_2$/day); $V$ is the volume of the unit (l); $C$ is the oxygen capacity of the absorbent ($cm^3$); and $D$ is the predetermined level of oxygen (ppm).

For example, the number of packets of Atco LH3000 needed to maintain an oxy-gen concentration of 1000 ppm in a 1000 l case leaking 300 ppm oxygen per day would be 8. It is also important to know the oxygen capacity of the scaven-ger when it is to be used by itself to create anoxic conditions—that is, when the treatment starts in air and does not involve purging with low-oxygen nitrogen. Determining how many packets of what size scavenger are needed to remove oxygen from a flexible bag filled with air is relatively simple. Because oxygen constitutes 20% of the volume of air, inserting enough packets to equal one-fifth the volume of the pouch will consume all of the oxygen in it. The gross vol-ume of the bag, at least for smaller ones, can be obtained by filling the bag with water and then measuring the water's volume. Although it may seem logical to throw in a few extra packets of absorber to ensure a good result, this isn't neces-sary. Ageless Z, as packaged and sold, has as much as a threefold excess capacity for combining with oxygen. (The exact amount should be verified by using an oxygen monitor.) This information should temper the use of too much material.

The intensity of oxygen removal by a scavenger is another matter. Lambert, Daniel, and Preusser (1992) studied the rate of reaction of oxygen with Ageless. They examined two factors that can affect the time needed to reduce the oxy-gen concentration to anoxic levels: how the packets were arranged in the chamber and the RH. In most experiments the Ageless packets were placed in a holder that allowed the atmosphere access to both sides. When the packets were arranged with one flat side in contact with the floor, the absorption rate slowed by about 20%, suggesting that this type of placement does not critically deter the effectiveness of treatment. However, operating at 33% RH, which is below the recommended range of 50-85%, did decrease the absorption rate sharply. The researchers concluded that Ageless would maintain the oxygen content at a very low level for several years in an inert gas-filled, hermetically sealed display case with a moderate leak rate. For purposes of pest control this study provides information on effective and economical ways to use Ageless. It also found that it is easier to remove oxygen at high concentrations than at low levels. The study found that the more absorbent used, the more rapidly the oxygen concentra-tions will be brought to kill levels.

It bears repeating that when Ageless reacts with air, in contrast to a nitrogen atmosphere containing oxygen at just a few thousand parts per million, the packets become quite hot. Thus, when used for oxygen removal from air, Ageless should never be in contact with thermally sensitive objects. Furthermore, air is 20% oxygen, and after Ageless has removed it from a containment bag during treatment, the flexible bag shrinks in volume by one-fifth. Delicate objects could be damaged by this partial collapse if they are placed in a tight-fitting plastic bag at the beginning of the anoxia treatment.

The aggressive use of Ageless was demonstrated by conservators at the Austra-lian Museum in Sydney in 1991, when a total of 1,290 objects were treated by sealing them in handmade, oxygen-impermeable, plastic-film bags and remov-ing all of the oxygen solely with Ageless Z-2000. Overall, 1,675 packets of the absorber were used (Gilberg and Roach 1992). Only about one-fifth of the items

were known or suspected to be actively infested. The cigarette beetle was the most prevalent pest, but the powderpost beetle, the webbing clothes moth, the drugstore beetle, and the furniture carpet beetle were also identified. Individual items and groups of objects, supported by matte board when needed, were placed in the bags along with the necessary amount of Ageless Z-2000 before heat-sealing. The objects consisted of a range of ethnographic materials including statues, bowls, blankets of bark, baskets, carved wooden panels, spears, headdresses, necklaces, and paintings on bark. Flushing the bags with nitrogen was not done, as the Ageless served to lower the oxygen in air to anoxic levels. The quantity of oxygen scavenger required was determined from the oxygen contained in a volume calculated from the length and width of the bag and the height created by the contained object, less the volume of the object. Bags containing objects of extreme fragility, such as feathered headdresses, or of exceptional size were first flushed with nitrogen, and a much smaller amount of Ageless was used. The bags were made from an ethylene-vinyl acetate copolymer, poly(vinylidene chloride), and nylon composite film that has an oxygen permeability of less than 5 $cm^3$ $m^2$ per day at 23 °C and 70% RH. This film was favored for its low oxygen permeability, good handling properties, and the ease with which it could be sealed using a bar sealer. All sealed bags were placed inside a temperature-controlled cabinet for 21 days at 30 °C. After use, the Ageless was discarded. Examination of infested items a year later failed to find any trace of continuing activity.

## Closures

### Heat Sealers

The construction of gastight containers using barrier films relies primarily on heat-sealing to make oxygen-impermeable unions. One surface of a heat-sealable barrier film must consist of a polymer, usually either polyethylene or polypropylene, that has a softening point below 177 °C and is fusible in a manner that forms a nonleaking, oxygen-tight seal. Creating the perfect seal requires a balance of hand pressure, sealer temperature, and press time. Doing this correctly is an acquired skill, but with practice and diligence most conservators develop a degree of skill that allows them to turn barrier film into perfect anoxia bags easily and competently.

Heat-sealing has long been used in the manufacture of industrial packaging for such diverse, atmospherically sensitive materials as food and electronic components. A wide range of heat-sealing equipment is commercially available. Sources include bagging supply companies, general equipment suppliers, and more specialized sellers of materials for conservation. Table 3.5 describes several different sizes and types of heat sealers. The handheld bar sealer is the most commonly used tool for conservation purposes. Tacking irons, such as the Sealector II or the Seal King, are less frequently used but they are cheaper. Some conservators claim that with experience, they can seal faster with tacking irons than with bar sealers, although the tacking irons must be used with a suitable soft and flat undersurface. A tacking iron's broader seal width—4.45 cm (1¾ in.) compared to 1.43 cm (⁹/₁₆ in.) for the handheld bar sealer—can be an advantage for making larger, flexible chambers. The automatic impulse sealer is more expensive, but it is cost-effective if there is a large amount of bag sealing to be done. Spatula and spade-tip tacking irons provide much smaller heating surfaces but can get into locations where the larger tools may not fit. A small tacking iron is especially suitable when a transparent window is called for in a metallized bag. It can be used to heat-seal the edges of a barrier-film patch to a metallized film into which a smaller window opening has been cut. Robert Hinerman, working with the conservation staff at the Mendicino County Museum in northern California, has fabricated a sealing tool for this purpose. It consists of a metal ring at

Table 3.5
Heat sealers.

| Type | Description | Model | Supplier | Cost (1996 US$) |
|---|---|---|---|---|
| Bar sealer | Handheld 1½ lb (0.68 kg) 6 in. (15.24 cm) long $^9/_{16}$ in. (1.43 cm) wide 100-260 °F (38-127 °C) | Futura Portable Barrier Model | Packaging Aids | 140 |
| Tacking iron | One surface thermostated 150-350 °F (65-177 °C) | Sealector II | Conservation Materials | 63 |
| Tacking iron | Heating area: 4 in. (10.16 cm) wide 1¾ in. (4.45 cm) long 150-400 °F (65-205 °C) | Seal King | McMaster-Carr | 49 |
| Automatic impulse sealer | 24 in. (60.96 cm), 2 jaws tabletop model automatic time set | 92-96348 | National Bag Company | 637 |
| Spatula tacking iron | Heating area: $^{11}/_{16}$ in. (2.38 cm) wide ¾ in. (1.91 cm) long temperature to 450 °F (230 °C) | Tacking tool 74535A2 | McMaster-Carr | 28 |
| Spade-tip tacking iron | Similar in size to the above | Adem C6 | Conservation Materials | 48 |

the end of an electric heating iron. The ring, which heats to over 177 °C, has an outer diameter of 5.08 cm and a bar width of about 1.27 cm, and conveniently heat-seals a very functional circular window into the metallized film.

## Zippers

The use of plastic zippers, or zip-locks, is another way of sealing a plastic bag. These fasteners are fabricated separately and then heat-sealed into the container. They appear in two applications: as closures on anoxia bags and as portals to much larger and permanent flexible chambers. Although zippers are convenient, they leak badly and can be used only on bags for dynamic anoxia treatments. In this mode, the leakage through the zipper allows the constant flow of humidified nitrogen through the bag. A heavier, better-designed zip-lock is built into large chambers. These have to be made more effective than bag zippers because repeated heat-sealing is not an option for reusable chambers. Even though they are much more gastight than small zippers, these closures are the part most prone to leakage on large bubbles. However, the application of generous amounts of petroleum grease through the zipper as it is closed has been found to reduce leakage.

## Clamps

Clamps for anoxia bags are plastic strips up to 183 cm long and about 1.25 cm wide, generally resembling a horseshoe in cross section. The film layers to be joined are placed between the two halves of the clamp, which form a tight seal when pressed together. There is some disagreement as to the effectiveness of

this closure, but most conservators who have worked with these clamps have found that they can provide leak-proof closure. In general, however, conservators continue to use heat-sealing. Clamps appear to work better with softer laminates, such as Marvelseal 360 or Film-O-Rap 7750, than with the less flexible Filmpack 1193. In Japan the Mitsubishi Gas Chemical Company markets small anoxia kits for food preservation consisting of 30.5 cm² (1 ft²) barrier bags, a small amount of Ageless, a single Ageless-Eye tablet (a passive oxygen monitor) for each bag, and a set of plastic clamps, which is some testimony to the utility of this form of closure. These kinds of clamps can be obtained from several of the companies supplying conservation needs.

### Adhesive Strips

Another way to make gastight seals between sheets of barrier film or to provide a closure for anoxia pouches is to employ one of several types of sticky tapes produced by Schnee-Morehead, Inc. These are used industrially to form high-vacuum packaging seals. Oxygen-impermeable closure is obtained by placing the tape along the inside rim of the bag opening and pressing firmly along the length of the bag-tape interlay. Another tape, SM 5144, available from Conservation Support Systems and other suppliers, is tinted yellow from a pigment that changes color when the tape is thermally cured (a process not required for insect anoxia treatment). The tape has a leachable component—liquid polyisobutylenes of extremely low volatility—that may be the basis for the gas impermeability of the seal in much the same way that petroleum grease helps to cut down on leakage through zip-locks on bubbles. John Burke confirmed the effectiveness of this tape in studies done at the Oakland Museum. He purchased from an Oakland hardware store a similar sealing tape that seemed to differ from SM 5144 only in its white color. After both tapes were used to seal anoxia pouches, the ability of the tapes to make gastight, oxygen-impermeable seals was tested with Ageless-Eye indicators and by leak-testing. Each tape was tested in triplicate. The yellow tape succeeded; the hardware tape failed. A major disadvantage of this type of closure, however, is the general difficulty experienced in working with a sticky tape. In addition, these materials do not pass the Oddy test, which detects the off-gassing of sulfur compounds. Although tape might be convenient for those without the tools or experience to carry out heat-sealing, conservators who construct bags on a routine basis feel that heat-sealing is still faster and easier.

### Portals

The passage of anoxia gases in and out of plastic containers is done through nonleaking portals generally made with plastic or brass fittings. The Swagelok brand is optimal and is available at hardware stores and laboratory supply houses. Conservators have found Swagelok O-Seal Straight Thread Male Connectors to be the most satisfactory fittings. These are illustrated in catalogs with the designation "[X-Y]-1 -OR," where X is the metal type of the adapter (B for brass, S for stainless steel) and Y is the tubing dimension (400 for ¼ in. [6.35 mm], 810 for 1/2 in. [12.7 mm]). The most popular models are the B-400-1 -OR and the B-810-1 -OR. Common laboratory cork borers can be used to cut precise holes in plastic bags to establish a perfect contact with the O-ring. Bradpoint drill bits should be used to make a clean hole in thicker polymer sections, such as the tough polypropylene caps of humidity bottles. It is helpful to wrap the brass threads with Teflon tape to get a tight seal. Figure 3.1 shows a cross section of a portion of the O-ring sealing area of the Swagelok adapter at the site where the gastight seal is made. After a circular hole is cut through a container wall, the O-ring is positioned against the outside surface; the flange is on the inside against a polyethylene or polypropylene surface. A good seal is obtained by tightening the flange nut. A problem encountered in using these

**Figure 3.1**
Schematic cross section of a portion of the
O-ring area of a Swagelok adapter.

Flange

Pressure

Surface A

Groove

Pressure

O-ring

Front nut

Outer surface of
barrier film

Inner surface of
barrier film
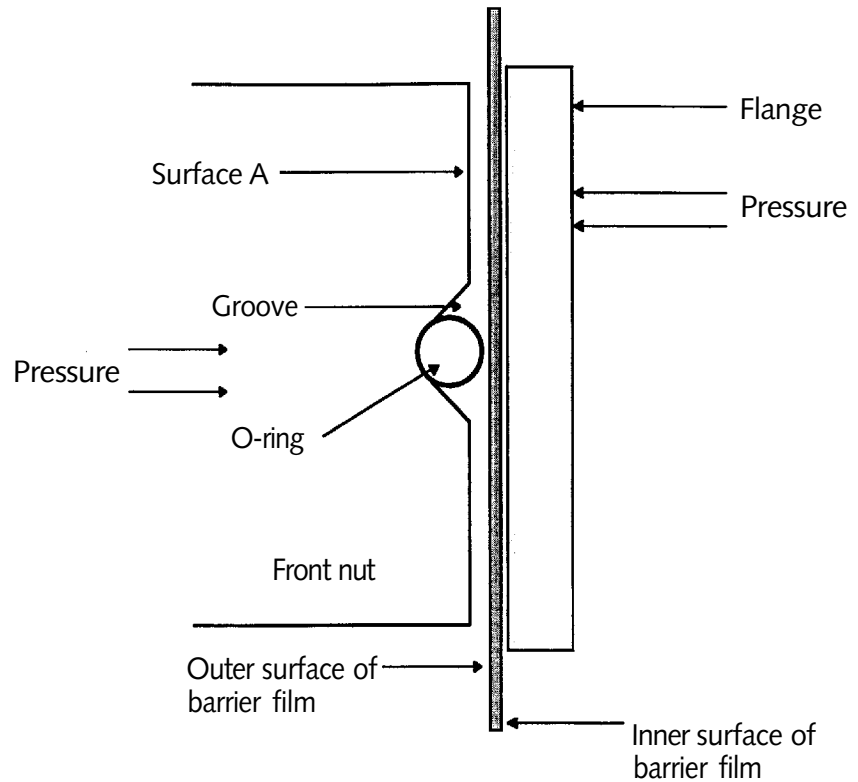
metal fittings with barrier films is that the O-ring does not protrude much above
the surface of the front nut (surface A in Fig. 3.1). This may cause the surface to
contact and rub against the barrier film on extended tightenings, thereby dete-
riorating the film. A variety of solutions has been used to solve this problem.
One is simply to use an O-ring with a larger cross section. Another is to put a
thin bed of epoxy resin in the bottom of the circular groove holding the standard
O-ring. Alternatively, the outer film surface can be made heavier and stronger in
the area of the fitting by adding layers of adhesive tape.

# Chapter 4

# Operational Problems and Practices

## Humidification

The compressed gases typically used for insect control—nitrogen, argon, and carbon dioxide—are supplied from commercial cylinders in "bone dry" condition—that is, their water content is in the range of 10-20 ppm. Moisture-containing and moisture-sensitive materials will slowly lose water and undergo dimensional and chemical changes if moved from an atmosphere with ambient levels of water vapor to a much drier one. These materials would include all cellulosics such as wood, cotton, linen, and paper as well as many protein-containing substances such as glue and leather. All of these are materials on which insects like to feed. Consequently, to avoid hygrometric shock to museum objects during fumigation, the gas streams may have to be humidified before being passed into the treatment chamber.

The three-jar humidification system is one method of providing moisture during treatments. It was developed by workers at the Getty Conservation Institute and the J. Paul Getty Museum (Hanlon et al. 1992; Rust and Kennedy 1993; Daniel, Hanlon, and Maekawa 1993) and is pictured in Figure 4.1. As seen in Figure 4.2, which is a diagram of the flow scheme, dry gas from the cylinder is split into two streams. The first is humidified by bubbling through a column of water held in a thick-walled polypropylene bottle of 2-4 l capacity. The stream is now saturated with water, although if the flow rate is too high and equilibrium cannot be established, it will be less than saturated. The humidified stream then passes into the second bottle, where it is mixed with the remaining dry stream, which is passed directly to this bottle. The mixing bottle also serves to knock down any spray. The combined streams next flow through a third bottle, which holds a relative humidity sensor, and then into the treatment chamber. A system of three needle valves, as shown in Figure 4.2, is used to adjust the ratio of flows and thereby the level of humidity in the treatment gas. The pressure within the three-bottle system is somewhat greater than the ambient pressure. This causes the gas, typically nitrogen, in the sensor bottle to have a higher RH than the exiting gas filling the anoxia chamber. At 50% RH and room temperature, the difference is about 10 percentage points—that is, 40% RH in the exiting gas with a system of the size indicated. One can compensate for this by creating a modestly higher RH in the humidification system than that required in the treatment chamber, and monitoring the humidity levels in both locations with a pair of relative humidity sensors. This works satisfactorily if both sensors have a short

Figure 4.1
Three-jar humidification system used with Vacudyne chamber at the Los Angeles County Museum of Art. (Photo courtesy of the Los Angeles County Museum of Art.)
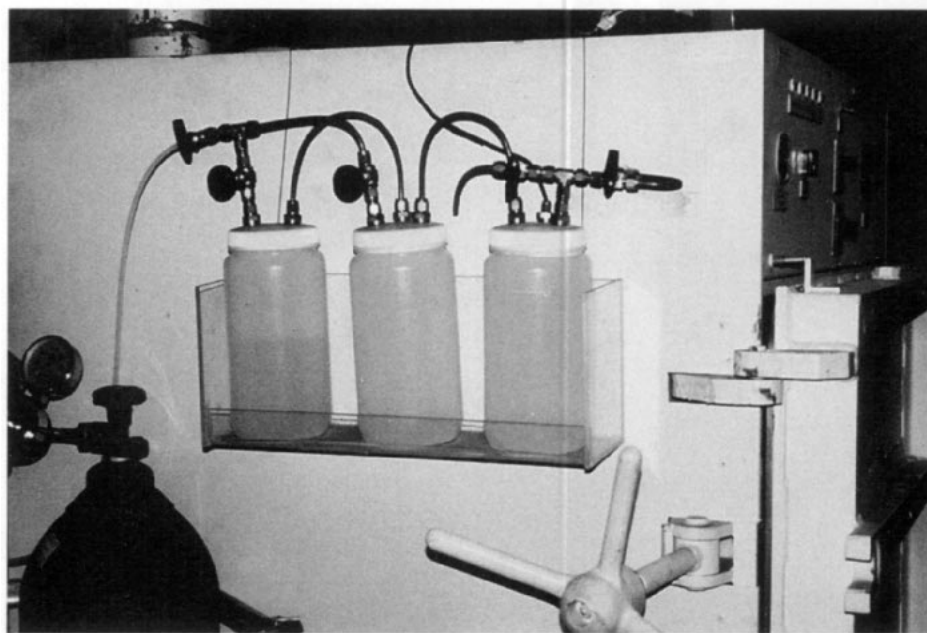
Figure 4.2
Schematic of three-jar humidification system.
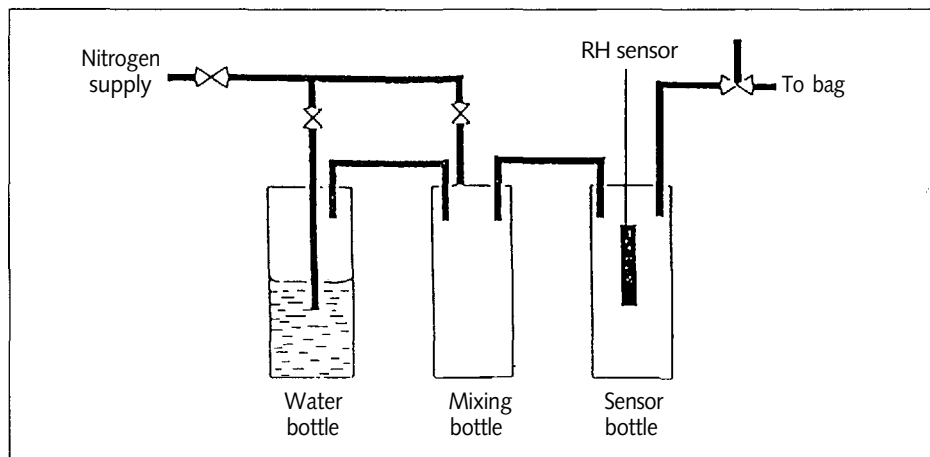
response time and are highly accurate. Such sensors, however, are expensive. Simple closure-type flow regulators, like needle valves, cannot be set to provide flow ratios with precision; without a pair of very good monitors, they could deliver a humidity level far from the desired mark. A way around this dilemma is to pass the separate gas streams through relatively inexpensive flow meters as shown in Figure 4.3. This provides an accurate determination of the ratio of wet and dry gas flows and, thus, of the RH entering the chamber. Then, only a single, simple humidity sensor in the chamber is needed.

Alternatives to the bubbler approach to humidification include placing pieces of wet cloth in the treatment bags or conditioning the objects at somewhat higher humidity than that at which they are normally kept prior to treatment. Placing humidifiers inside the treatment chamber may be called for when working with very large containment units where the need for delivery of substantial quantities of nitrogen or carbon dioxide in reasonable periods of time would require high inflow rates. This may be difficult to do with a conventionally sized bubbler system, where troublesome pressure buildups and less-than-saturated streams would result. At Canada's National Museum of Science and Technology in Ottawa, the staff carries out fumigation with carbon dioxide in a custom-built bubble that measures 6.7 × 2.4 × 2.7 m high. Humidification is done with a Bionaire ultrasonic humidifier placed inside the bubble. Distilled or deionized water should be used with this unit. Conservators at the Museum of Fine Arts in Houston do their treatments with nitrogen in a 30 $m^3$ bubble. This size requires the input of twenty to thirty cylinders of nitrogen per treatment. Humidification is provided by a 9.46 l (2.5 gal.) steam-type boiler. Two Cole-Parmer minihygro-thermographs are positioned on opposite sides of the interior of the chamber, next to viewing ports. The steam humidifier and a 41 cm (16 in.) fan are posi-

Figure 4.3
Schematic of new humidification module.
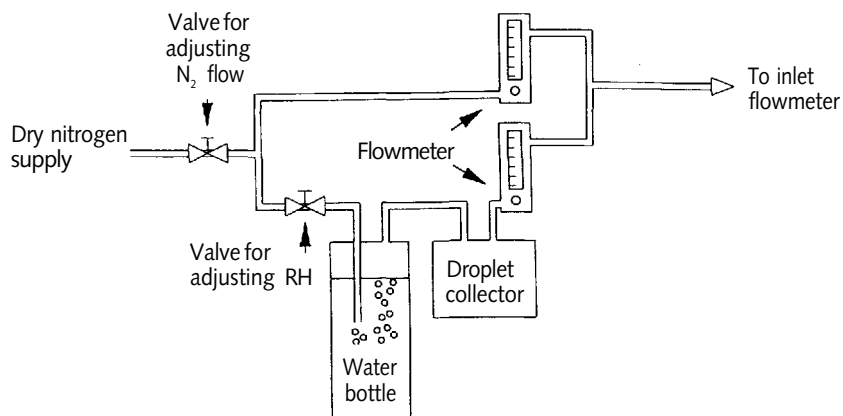
tioned along an interior side wall near the incoming nitrogen so that there is good mixing of moisture and nitrogen before the humidified gas enters the space containing the infested objects.

A very popular alternative is simply not to humidify. Many conservators do not run their equipment with humidification units, and the majority of materials being treated are not subjected to the deliberate introduction of additional water. The main factors allowing for this decision are the nature of the anoxic treatment and the objects being treated; whether nitrogen or carbon dioxide is being used; and the amount of moisture buffering that occurs. In a dynamic or flowing system, where dry nitrogen continuously passes over objects at a significant rate, humidification is a must to prevent hygrometric shock. This is less obvious in a static system where a fixed atmosphere, even one with near zero humidity, is in equilibrium with items undergoing treatment.

Objects invaded by pests contain primarily cellulosic materials, which have a significant and reversible water content. Buck (1952) has shown that wood types, including poplar, ash, oak, chestnut, and fig, ranging in age from less than one year to over thirty-seven hundred years, closely averaged 10% water content at 60% RH; 8% water content at 40% RH; and 6% water content at 20% RH. At 22.5 °C, the addition of one gram of water to a cubic meter of air will increase the RH by 5%. Thus, a kilogram of wood, conditioned to 60% RH, would lose only 10 of its 100 grams of water to dry nitrogen to bring the entire system to equilibrium at 50% RH. In smaller containments or in holding boxes of cardboard or wood or other buffering material, the loss of water from wooden artifacts could be much less. Kamba (1994), studying how fluctuations in humidity cause dimensional changes in wood kept in a closed case, concluded that wood enclosed with little air and without any moisture-buffering materials is dimensionally more stable than wood enclosed with conditioned silica gel. In the case of textiles, the major humidification concern is with constrained fabrics suddenly being exposed to high relative humidities (Michalski 1993). They can shrink dangerously in 75-100% RH, especially above 90% RH. Below 75% RH, constrained textiles show very little tension change. Humidity control is generally not a concern for unconstrained material such as clothing or carpets. Therefore, with fabrics the main caution for operators of anoxia chambers is that their humidification system does not accidentally supply too much water.

Valentín, in 1993, studied the buffering effect of paper conditioned at 40% RH and 80% RH and subsequently maintained in argon atmospheres of 0% RH and 50% RH. The results of the four experiments are shown in Figure 4.4. Plastic bags containing the conditioned paper were purged at 2.0-2.5 l min$^{-1}$ for 4 days and then sealed and allowed to stand an additional 6 days. The humidified argon maintained RH equilibrium in the two experiments where it was used. When dry argon was used, the system settled around 65% RH after starting at 80% RH. In the plastic bag with paper conditioned to 40% RH, the dry argon actually picked up enough water to provide a stable RH above 40%. Valentín concluded that museum holdings maintained in modest relative humidity ranges can be easily disinfested using a nonhumidified inert gas. However, works of art already exposed to high humidities, and sensitive objects such as musical instruments or polychromed wood, should be treated with humidified gas.

Valentín found that nitrogen and carbon dioxide behaved similarly to argon. Cipera and Segal (1996) demonstrated this with carbon dioxide on an operational scale. In fumigations in a 30 m$^3$ bubble containing wooden artifacts as well as textiles packed in cardboard boxes, the RH, after most of the air was replaced with dry carbon dioxide, would often drop as much as 10 percentage points, but it would return to initial values within forty-eight hours (Fig. 4.5).
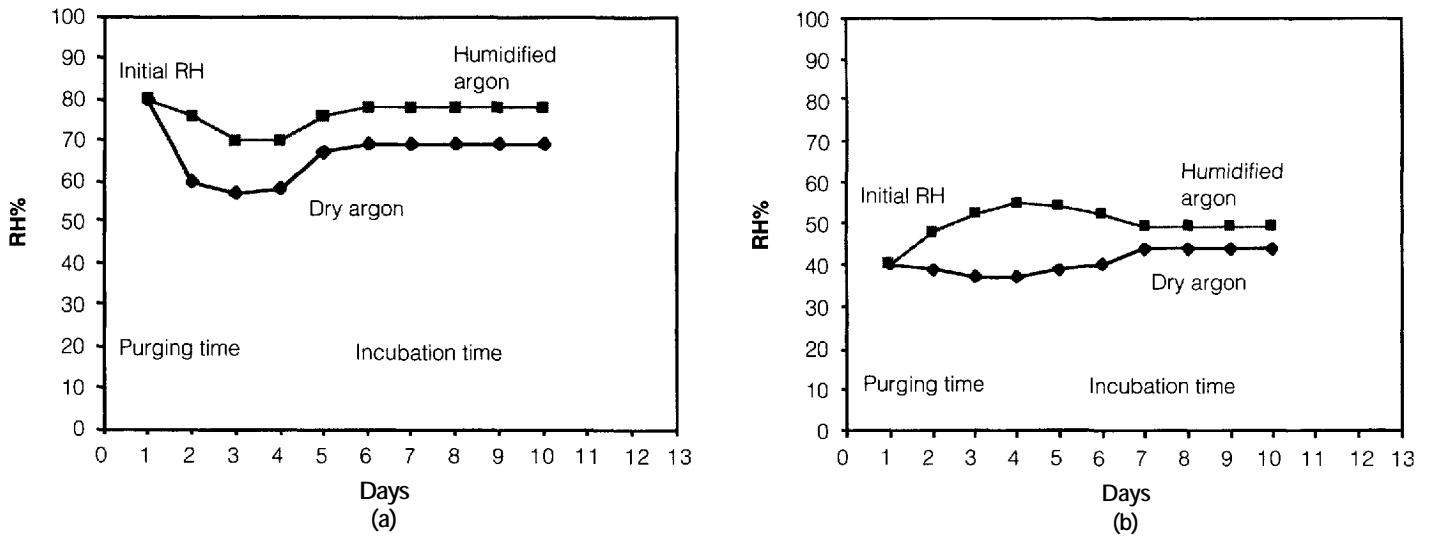
Figure 4.4a,b

RH change in plastic bags during treatment of paper. Bags were purged with dry argon and humidified argon (50% RH), after starting at (a) high (80%) initial RH, and (b) low (40%) initial RH (Valentín 1993). (Courtesy of Nieves Valentín.)

Conservators using carbon dioxide are generally less likely to use humidification than those employing anoxia. Carbon dioxide as a fumigant works most effectively at a concentration of 60-70%. The residual air still contains its water vapor; thus, one-third of the original humidity is maintained, and a totally dry environment is never present. In addition, the typically large carbon dioxide fumigation unit is usually filled with objects well-wrapped in paper or cotton and packed into cardboard boxes in which there is significant moisture buffering. Avoiding excess humidification helps to hasten and ensure a complete kill and allays concerns about the formation of carbonic acid.

Figure 4.5

Hygrothermograph of carbon dioxide fumigation in 30 m³ bubble showing humidity buffering by contents of bubble. (Courtesy of the Conservation and Technical Services Division, Canadian Museum of Civilization, Hull, Quebec.)
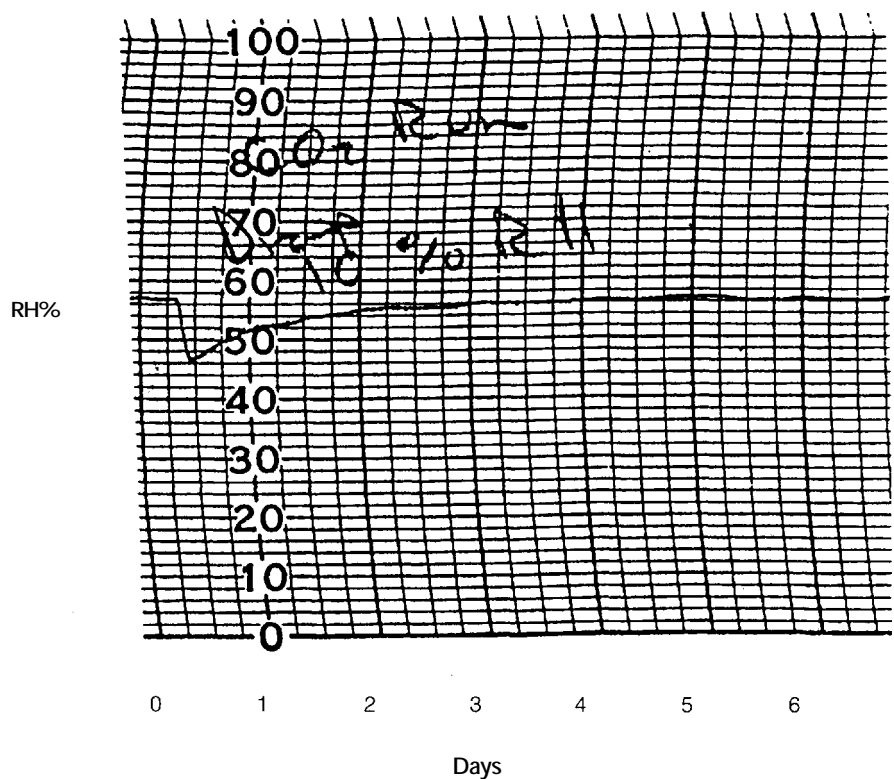
## Argon versus Nitrogen

While most anoxia treatments are being carried out with nitrogen, the use of argon deserves consideration. Although helium is also effective, this very light gas has a relatively high diffusion rate through most plastic films, creating containment problems; it is also expensive. Argon is attractive because its cost in many locations may be comparable to nitrogen. Additionally, studies by Valentín showed that the time necessary for 100% assured kill with argon was 65-70% of the time with nitrogen (Fig. 1.3). The species compared at 40% RH, 300 ppm oxygen in argon or nitrogen, and at 20, 30, and 40 °C included the old house borer and the cigarette, furniture, deathwatch, powderpost, drugstore, book, and black carpet beetles. The study showed, for example, that at 20 °C, it took 7 days to kill all life stages of the furniture beetle with nitrogen but only 5 days with argon. Argon has been the anoxant of choice for Valentín for pest control in humid regions where the cost of nitrogen and argon is generally comparable. She found that when using a 6.2 m$^3$ commercial bubble, based on a film made with poly(vinyl chloride) and reinforced with polyester, the oxygen leak rate after 11 days was 2.3% with nitrogen and 1.6% with argon. It is not clear why this should be the case, but the oxygen leak rate was also higher with nitrogen than with argon for poly(vinylidene chloride) bags—0.6% versus 0.5% over one year. The slightly slower influx of oxygen when argon is used may help explain the higher rates of insect kill with this gas.

Koestler and Mathews (1994) conducted a pilot program in 1992 in which they used argon to treat ancient documents, infested with book lice, from the library of the Great Lavra Monastery on Mount Athos in Greece. Seventy manuscripts were rendered free of lice by sealing them inside plastic bags based on nylon film as the oxygen barrier. The bags were flushed with humidified argon to bring the oxygen content to 700-1000 ppm before heat-sealing. While Koestler and Mathews claimed that 20 days of exposure would have been sufficient in a museum that maintains a constant room temperature, the variable and sometimes quite low temperatures at Mount Athos led to a minimum of 30 days of treatment. Unfortunately, the pilot program was not expanded to treat the entire, very large manuscript collection, although Rentokil has since done selective fumigations at the monastery with carbon dioxide (Smith 1996).

Whether the cost of argon is comparable to nitrogen or substantially higher may depend on the location and the availability of suppliers other than prime distributors of research-grade gases. The cost of the two highest purity grades of nitrogen, based on the February 1996 listed prices of prime suppliers, ran between $4.08 and $5.10 per cubic meter in Los Angeles, California, while the corresponding grades of argon ranged from $5.10 to $6.80 per cubic meter (Table 3.4). This is based on prices ranging from $37.00 to $65.00 for a T-cylinder of gas. An industrial grade of nitrogen, assayed at 20 ppm oxygen, can often be obtained from secondary suppliers for a fraction of the price of the research grade. The savings on industrial argon are less but still significant.

Proponents of argon cite advantages other than speed of kill, but the practical importance of these has yet to be demonstrated. For example, argon is somewhat heavier than oxygen and less dense than carbon dioxide (Table 4.1). This might lead to stratification that would create lower oxygen concentrations around insect-infested objects placed on the floor of a containment unit. However, the magnitude of the density difference is not likely to create dramatic stratification effects. Additionally, if there are diurnal temperature swings in the facilities where the bags are stored, then thermal convection currents within the bags are likely to eliminate any such stratification. Also, there is a perception that microbial or fungal growth on museum objects might feed on nitrogen, which would not occur with argon. However, this is not likely to happen on

Table 4.1

Density of common gases at 1.0 atmosphere and 0°C.

| Gas | Kg m$^{-3}$ |
| --- | --- |
| Nitrogen | 1.25 |
| Oxygen | 1.43 |
| Argon | 1.78 |
| Carbon dioxide | 1.98 |

wooden pieces in the absence of liquid water (Blanchette 1995) and does not pose a genuine threat to the use of nitrogen for anoxia.

## Monitors

This section provides a general description of the different types of monitors that are used with nontoxic gases for pest control. Monitoring equipment for conducting treatments can be either purchased directly from manufacturers or obtained from integrated-sensor dealers like Omega Engineering, Inc., the Cole-Parmer Instrument Company, and the Mitchell Instrument Company. (Only a few vendors are mentioned here. A more complete list of suppliers can be found in Appendix B. Conservators are urged to contact these companies for current prices as well as more precise information on specifications and availability than can be provided in this book.)

### Electronic Oxygen Monitors

Instruments that are commercially available for measuring oxygen concentration fall into two classes: trace analyzers and monitors. Trace analyzers determine very low levels of oxygen with a resolution of 1 ppm and are generally expensive. They are used to monitor combustible environments in process industries and in food-packaging quality-assurance programs, and they are critical for anoxia research.

Less sensitive and less expensive units, called monitors, are suitable for most work with anoxia. Instruments with an oxygen concentration range of 0-25% and a resolution of 1000 ppm are available. They are typically used for monitoring the safety of work places where oxygen deficiency is a possibility. For pest control, the monitor should have a range of 0-1% with a resolution of 200 ppm. It should be capable of being calibrated to 20.9% oxygen, and it should be temperature-compensated. Monitors can be used in two configurations, either as a remote sensor or with a sample aspirator. The remote sensor consists of probes, based on oxygen-sensitive, galvanometric membrane electrodes, which produce an electrical potential that is a function of the oxygen concentration. This electrical signal is converted to a digital or analog concentration value for display. If the monitor is battery operated, it can be left inside the enclosure, and readings can be made from the outside through a viewing window. Otherwise, cables need to be passed from inside the enclosure to an external readout unit through a hermetically sealed connection. The sample-aspiration configuration uses an internal or external pump to draw a gas sample from the anoxic atmosphere for analysis by a sensing cell in the monitor. The sample can be either vented or returned to the enclosure. Sensor probes deplete as they react with oxygen, losing sensitivity after six months in a normal (air) atmosphere. Teledyne Analytical Instruments, Delta F. Corporation, and Gas Tech, Inc., are well-established oxygen-sensor manufacturers in the United States.

### Passive Oxygen Monitors

A simple and inexpensive way to determine whether an atmosphere's oxygen concentration has been reduced to below 1000 ppm is to use a passive oxygen

**Figure 4.6**

Oxygen-related color change in the azine dye methylene blue.
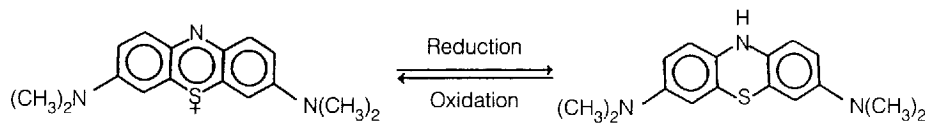


monitor. This is a ¼ in. (6.35 mm) diameter tablet that is placed inside the treatment container; it is blue in the presence of oxygen and red or pink under anoxic conditions. This color change is based on the chemistry of methylene blue, an azine dye employed in the formulation of the monitor (Fig. 4.6). Azine dyes can alternate between an oxidized or a reduced state, and this is indicated by color. Air keeps methylene blue in an oxidized condition with a corresponding blue color. In the absence of oxygen and in the presence of mild reducing compounds such as ferrous salts or aldehydes, the dye changes to pink.

Scientists at the Mitsubishi Gas Chemical Company have studied the chemistry of this color change and developed the Ageless-Eye oxygen indicator. They have obtained a variety of Japanese, European, and United States patents covering products marketed under the Ageless-Eye trade name. One of their early formulations was based on glucose as the reducing agent in a mixture with triethylamine, oleic acid, methylene blue, titanium dioxide, and glycerin (U.S. Patents 4,169,811 and 4,349,509). This formulation turned from blue to red at oxygen concentrations as high as 3.5%, which is much too high for killing insects. There were other problems as well with this early formulation. A relatively high moisture content was needed for the chemicals to interact and provide a color change. In addition, over time, the pink-red color would take on a bluish cast, even in a very low oxygen environment. A more recent patent (Inoue, Hatakeyama, and Yoshino 1994) disclosed that these problems can be overcome by substituting 1-amino-2, 3-propanediol for the triethylamine. This formulation provides a color transition that occurs under conditions closer to those needed for anoxia treatment. It illustrates how dependent accurate monitoring is on the nature and quality of the chemicals comprising the indicator, which may explain why conservators have had variable results using Ageless-Eye as an oxygen sensor. The composition of Ageless-Eye can be formulated to have the color transition occur at different oxygen levels, and two variations are marketed: type "C" and type "K." Type K changes from blue to pink at an oxygen concentration below 2500-3000 ppm. This is well above the transition zone of type C, which is between 1000 ppm and 1500 ppm. The color change appears to occur more slowly with C than K. Type C operates best at 15-35 °C and 60-90% RH, while type K is best at 5-35 °C and 30-92% RH. Type C Ageless-Eye has a deeper blue color than type K.

When exposed to an atmosphere containing 5000 ppm or more oxygen, Ageless-Eye is purple or blue. It will turn pink when the oxygen concentration falls sufficiently below this level. Quickly reexposing the Ageless-Eye to oxygen, within five to fifteen minutes, brings on the blue color. Going in the other direction, from pink to blue, is slower and more difficult and takes several hours after anoxic conditions have been reached. The return to pink is brought about by the weak reducing agent contained in the formulated tablet. The chemistry of the reduction requires water, a fact that is reflected in the moisture requirements for both types of Ageless-Eye. Gilberg (1989b) reported on the poor indicator properties at low RH. He found that at 0% RH, Ageless-Eye failed to turn pink even after prolonged exposure to nitrogen atmospheres of very low oxygen content.

When Ageless-Eye is used in a treatment, it should be positioned to avoid direct exposure to bright light. With time and exposure to light and heat, the capacity of the reducing agent diminishes. This deterioration is generally the cause of

premature failure of the indicator. The manufacturer recommends storing the tablets in a cool, dark, and oxygen-free atmosphere, conditions under which the capacity of the reducing agent is not used up. This is conveniently done by sealing packets of Ageless-Eye in a barrier-film bag containing the Ageless Z oxygen scavenger, and placing the bag in a refrigerator. With careful attention to handling and storage, Ageless-Eye has been reused nineteen times over a one-year period (Elert 1996).

Mitsubishi offers Ageless-Eye packaged three ways. Type K comes in single blue packets that have tiny openings in their windows to allow the treatment atmosphere access to the indicator. Type C is similarly packaged in single, colorless envelopes, but it also comes in a green-colored strip of ten envelopes. Here, too, the singles have openings to the atmosphere, but the strip sets must be punctured. The need for great care in storing Ageless-Eye made conservation supply houses reluctant to repackage the manufacturer's standard five-thousand-tablet container into smaller, more affordable units. However, the growing demand for this indicator appears to have overcome this problem, and inexpensive packages holding as few as twenty-five type C tablets can now be purchased.

Carbon Dioxide Monitors

A large number of carbon dioxide monitors have become commercially available to cope with recent stringent requirements for indoor air quality. While there are systems produced to cover any range of concentrations, environmental monitors normally provide measurements over two ranges: 0-2000 ppm and 0-5000 ppm. (The normal atmospheric concentration of carbon dioxide is approximately 350 ppm.) These ranges cover the safety limits set by the U.S. Office of Safety and Health Administration (OSHA). The monitors offer an accuracy that is 5% or better of full scale, but readings normally drift as temperature changes. Calibration must be done periodically and is based on pure nitrogen and a reference mixture of carbon dioxide and nitrogen. The determination of concentration is usually based on the absorption of infrared radiation at bands characteristic for carbon dioxide. The gas to be analyzed is passed through an infrared absorption cell operating over a narrow bandwidth where carbon dioxide absorbs strongly. Changes in radiation energy are detected, amplified, and sent to the signal-processing portion of the system for display or storage. The monitors contain either a diffusion cell, which provides low-power-consuming stable sensing at a low cost, or a sample aspirator, which has higher accuracy and long-term stability but at a higher cost.

An alternative to carbon dioxide monitors are oxygen monitors that can be used to determine concentrations of carbon dioxide in enclosures and chambers. When an enclosure filled with air is flushed with pure carbon dioxide, the oxygen concentration decreases. A reduction of oxygen from 20.9% to 10.45% corresponds to an increase in carbon dioxide concentration from about 0.04% to 50%; an oxygen reading of 8% indicates that the carbon dioxide level is at 60%, the level that is favored for fumigation. The output from the oxygen monitor can be configured to read as carbon dioxide concentration. Bruel and Kjaer Instruments Incorporated, Gas Tech, Inc., and Valtronics are established manufacturers of these sensors.

Relative Humidity Monitors

Many types of RH monitors are available, including indicator strips, hair hygrometers, and electronic instruments based on bulk polymer, capacitance, and chilled-mirror sensors. Most respond well in the range of 20-80% RH. More sophisticated and expensive instrumentation is needed outside this range. Selec-

tion of a monitor type is based primarily on range, accuracy, response time, size, and cost.

The atmosphere in small sealed bags and pouches, where the change in RH is minimal and occurs slowly, can be monitored using inexpensive indicator strips or dial-type hygrometers. These can be calibrated at only one RH and tend to be less accurate than electronic sensors, which can be calibrated at both a low and a high RH. A more accurate and faster-responding sensor is needed in large chambers and bubbles where high nitrogen input, for a variety of reasons, obscures a precise determination of RH. Among electronic monitors, resistivity bulk-polymer sensors are inexpensive but take up to five minutes to produce 90% of their response; monitors based on capacitance generally provide data in less than thirty seconds.

RH indicator strips are the cheapest sensors, while hair hygrometers can be found at a variety of prices. Electronic sensors range from a simple, low-cost, bulk-polymer-based indicator, to a highly sophisticated and expensive chilled-mirror dew point sensor. Low-cost electronic RH sensors are available from the HVAC monitoring industry. ACR Systems, Inc., Edge Tech Moisture and Humidity Systems, Hy-Cal Engineering, General Eastern Instruments, and Vaisala, Inc., supply most ranges of RH sensors.

## Temperature Sensors

A wide range of inexpensive temperature sensors can be purchased. These include temperature indicator strips and paints, bimetal and dial thermometers, glass thermometers, thermocouples, thermistors, resistive thermal devices, and infrared thermometers. All of the above thermometers, except temperature indicator strips and paints, deliver the accuracy needed for pest control work. Bimetal and dial temperature indicators and glass thermometers are inexpensive. Thermocouples and thermistors are electronic sensors that can be connected to chart recorders or data loggers for automatic monitoring. K-type thermocouples are the cheapest electronic thermometers. Thermistors are, in general, more accurate than thermocouples, but they cost more.

## Leak Detectors

Conservators embarking on a program of treatments with barrier-film containers, particularly when the containers are handmade, will find a leak detector to be a valuable tool. The detection of specific leak points can be accomplished in several different ways. It can be done by activating a battery-operated ultrasonic sound generator (35,000-40,000 Hz) placed inside a treatment container, and then searching for the leak point from the outside with an ultrasonic detector. A detection system based on fluorescence works in a similar way. A tracer gas is released into the pouch (for example, by injection, with a gas syringe), and when an ultraviolet light is played over the exterior surface, leaks are detected as a fluorescent glow. The cheapest and most popular device is the halogen leak detector, which is based on a refrigerant gas such as 1,1,1,3-tetrachloropropylene that is released inside the chamber. The detector nozzle senses changes in the thermal conductivity of the atmosphere around the leakage point and gives off a characteristic squawk or squeal.

## Monitoring Life Signs

Perhaps the most critical and the most difficult factor to be monitored is the extent and the completion of insect kill. Researchers have investigated a number of barely measurable manifestations of insect life signs but they have been unable to transfer their work into a monitoring methodology as satisfactory as

direct examination. This is a tedious process; it requires looking for any evidence of life after allowing enough time and appropriate conditions for all life cycles to continue and reveal themselves, should any life still exist. It has remained for a few landmark studies to make these determinations and establish the conditions where 100% mortality is achieved with a high degree of confidence. Valentín, Gilberg, and, particularly, the team of Rust and Kennedy have done this to create Table 2.3, which lists the conditions and minimum times required for 100% assured kills for a large number of the most common museum pests. In the study by Rust and Kennedy, the adult insects were generally examined for mortality after a few days; the other life stages were held for inspection for weeklong periods up to nine weeks and, in some cases, for as long as four months. The number of insects involved in this study ran into the tens of thousands. With the availability of information on the minimum number of days required for assured complete kill, conservators have confidently carried out effective fumigations—without concerning themselves with monitoring for life signs—by using much longer treatment times, occasionally even extending fumigation by four to five weeks.

Some interesting approaches to measuring mortality directly have evolved. Koestler (Koestler and Mathews 1994) adapted a procedure developed by the U.S. Department of Agriculture (USDA) to determine insect infestations in wheat. The USDA created a highly sensitive FTIR analysis that could detect an output of about one part of carbon dioxide in one million parts of total gas per minute from a single rice weevil in 350 g of red wheat. Koestler was able to use his adaptation to follow the anoxic termination of insect life in museum objects. A picture frame with suspected infestations was placed in a low-permeability pouch, and a steady increase in carbon dioxide was monitored for 5 days. The bag was then flushed with humidified argon until the oxygen content was below 0.1%, and new measurements were made. No carbon dioxide was detected after 16 days, and air was returned to the system. There was no additional formation of carbon dioxide over 11 days, which was taken to show that 100% mortality had been achieved.

A simpler, less sophisticated device to measure and monitor the respiration of a single insect was described by Carlson in 1995. The breathing of insects removes oxygen and releases carbon dioxide, but the net effect is a decrease in volume. Carlson designed an apparatus, contained in a constant-temperature bath, in which a glass tube holding an insect is connected to a capillary containing a tiny droplet of oil. Movement of the droplet provides a life-sign indication of metabolism. With some simple determinations of air pressure, temperature, and movement volume of the drop, it is possible to calculate the number of molecules the insect respires. The system is designed for amateur scientists and is inexpensive to put together, but a larger gastight container connected to a capillary could make the unit suitable for determining whether infested material still harbors living insects.

## Safe Use of Nontoxic Fumigants

Although gases such as nitrogen and argon are not pesticides, there are safety concerns about their use. Carbon dioxide is somewhat different. It is not conventionally considered a toxic material, but the presence of relatively small amounts in air causes breathing problems. Local regulations governing the use of carbon dioxide as a fumigant vary and should be ascertained by conservators planning to use this gas for insect control. In Canada, for example, carbon dioxide is not classified as a fumigant, and users do not have to be licensed. In California and in the United Kingdom, it is listed as a fumigant. In England, Rentokil has gone through the necessary testing and documentation with the appropriate governmental authorities and is licensed to do carbon dioxide fumigation. No

one else has done this; consequently, British conservators looking to treat infested material on-site with a modified atmosphere are likely to use anoxia with nitrogen or argon instead. The physiological effects of excessive concentrations of nitrogen and carbon dioxide differ. At moderate levels of carbon dioxide (e.g., 5000 ppm), they are unpleasant and serve as a warning for exposed personnel to leave the area. Modest oxygen deficiencies (e.g., 12-19% oxygen in nitrogen) will cause some individuals to experience a pleasurable sensation under conditions where unconsciousness can occur without warning. Persons succumbing to any of these gases need to be moved to fresh air and given resuscitation as quickly as possible.

OSHA literature warns that a drop in oxygen concentration as small as 1.4%, for example, from the normal 20.9% concentration in air to 19.5%, may start to have adverse effects on individuals. At 8-10% oxygen in nitrogen, the effect may be lethal. A general indication of what can happen with either nitrogen or argon that is deficient in oxygen is shown in Table 4.2, which has been provided by the Compressed Gas Association. The indications are for a healthy, average person at rest. Factors such as individual health, degree of physical exertion, and high altitudes can affect these symptoms and alter the oxygen levels at which they occur.

The Compressed Gas Association offers the following suggestions to individuals who may be subjected to oxygen-deficient atmospheres:

1. Never enter a suspected oxygen-deficient atmosphere without proper protective breathing apparatus and attendant support.
2. Analyze the atmosphere to determine if there is a deficiency of oxygen. Continue to monitor during the work process. If the oxygen level is less than 19.5%, ventilate to establish good air quality.
3. Be trained on what to expect and how to handle it.
4. Positively isolate to a confined area any incoming lines, and ventilate the area.
5. When it is necessary to work in any oxygen-deficient atmosphere, provide a self-contained breathing apparatus or airline-style breathing mask for all workers.
6. Use an established hazardous-work permit procedure in all confined-space activities.

**Table 4.2**

Safety concerns with oxygen-deficient atmospheres.

| Oxygen content (% by volume) | Effects and symptoms of acute exposure of humans (at atmospheric pressure) |
|---|---|
| 15-19% | Decreased ability to perform tasks. May impair coordination and may induce early symptoms in persons with heart, lung, or circulatory problems. |
| 12-15% | Breathing rate increases, especially on exertion. Pulse rate up. Impaired coordination, perception, and judgment. |
| 10-12% | Breathing increases further in rate and depth, poor coordination and judgment, lips slightly blue. At this oxygen content or less, anoxia will bring about unconsciousness without warning—so quickly that individuals cannot help or protect themselves. Lack of sufficient oxygen may cause serious injury or death. |
| 8-10% | Mental failure, fainting, unconsciousness, ashen face, bluish lips, nausea (upset stomach), and vomiting. |
| 6-8% | 8 minutes: may be fatal in 50-100% of cases; 6 minutes: may be fatal in 25-50% of cases; 4-5 minutes: recovery with treatment. |
| 4-6% | Coma in 40 seconds followed by convulsions, breathing failure, death. |

A similar tabulation of physiological effects for carbon dioxide is provided in Table 4.3 (Banks and Annis 1990).

These safety factors are usually not an issue with anoxia treatments carried out with reasonable prudence in barrier bags. They come into play during operations involving large units placed in relatively tight quarters where many cylinders of gas or containers of liquid nitrogen may be used. The well-known tendency of carbon dioxide to accumulate in low-lying and enclosed regions is a potential cause of safety hazards when high-level carbon dioxide atmospheres are used, particularly in leaky enclosures. A number of operators of carbon dioxide chambers have installed monitors that are activated when levels of this gas rise from the normal 350 ppm in air to 1000 ppm. At this point, either a forced ventilation system is turned on or an alarm goes off. These are not uncommon occurrences. Because their density is similar to that of air, low-oxygen/high-nitrogen atmospheres are unlikely to present a similar type of hazard. All large chambers require adequate ventilation in the rooms in which they are used to ensure that there is no external danger to personnel.

Table 4.3

Safety concerns with elevated carbon dioxide concentrations in air.

| Carbon dioxide content (% by volume) | Effects and symptoms of acute exposure of humans (at atmospheric pressure) |
| --- | --- |
| 0.1-1%[a] | Slight increase in lung ventilation. |
| 3% | 100% increase in lung ventilation. |
| 5% | Breathing becomes labored; maximum tolerable concentration for extended periods. |
| 10% | Upper limit of tolerance, with retention of consciousness for a few minutes. |
| 15% | Unconsciousness occurs within 1-2 minutes. |
| 25% | Rapid unconsciousness leading to death if person not removed within a few hours. |

[a]OSHA time-weighted average of threshold limit is 0.50% or 5000 ppm.

# Chapter 5

# Anoxia Treatment in Barrier-Film Bags

This chapter, as well as chapters 6 and 7, presents down-to-earth detail about the work of individuals and institutions leading to the development of procedures for insect anoxia. The failures as well as the successes are recounted. A snobbish conservator might call the tone terribly unsophisticated; this is true and it is intentional. A detailed and unvarnished history of actual experience in a new field is far more helpful to most people entering it than the usual scientific article that glosses over crucial obstacles that may have taken years to overcome. The goal of this book is not only to disseminate rigorously proved data but also to describe many varieties of practice, some perfected only after much experimentation and some still needing improvement, so that the conservator considering anoxia treatment will not have to reinvent the wheel.

## General Procedures

The most widely used procedure for the treatment of infested objects is anoxia in barrier-film bags. Quite suitable bags may be purchased from a growing number of suppliers who have been upgrading the quality of their products and are providing bags based on the latest, most oxygen-impermeable barrier composites available from film fabricators. When these bags are purchased, it is advisable to obtain a statement of specifications for the film properties, especially for oxygen permeability. Treatment pouches can also be handmade from the same barrier-film composites that are sold in rolls.

Making bags by hand, instead of purchasing them, may seem like a major investment in time, and perhaps too much trouble. But many conservators, once they have learned the procedures and techniques, prefer to buy rolls of film and make their own bags. The procedure is simple and, with time, so is the execution. The first step is to cut a rectangle of film from a roll that is somewhat larger than twice the desired size of the bag. The rectangle will be doubled over to prepare a pouch of the required dimensions. Care should be taken to cut straight lines to produce a true rectangle so that the edges line up precisely. The film should be placed on a flat, horizontal surface with the heat-sealable surface, usually a layer of polypropylene or polyethylene film, face up. The sheet is then doubled over, and a pouch is created by heat-sealing the two side edges, leaving the top edge open. Finally, the objects to be treated, along with some type of oxygen monitor, are placed in the pouch, and the container is thoroughly purged with a rapid flow of nitrogen gas introduced via a flexible tube inserted into the open end of the bag. Then, packets of oxygen absorber are placed in the pouch, and the remaining open end is heat-sealed closed. An important step is leak-testing the bag after the objects have been sealed inside. As described in chapter 4, this is done by using a hypodermic syringe to inject into the bag a small amount (a few cubic centimeters) of detectable gas such as 1,1,1,3-tetrachloro-propylene. The puncture hole is sealed with tape, and the nozzle of a halogen leak detector is run over the bag's exterior.

If an opaque, metallized film is used to make the bag, it is not possible to see inside to check the performance of the oxygen monitor. The solution to this, when the monitor is a small-diameter indicator tablet, is to install a small window in the opaque film before the open sheet is converted into a bag. The window is prepared from a small piece of clear barrier film; often the plastic wrapper holding the packets of Ageless is used. A 2-3 cm (1 in.) square opening is cut into the metallized film and covered with a 7-8 cm (3 in.) square of clear barrier film with the heat-sealable surfaces in contact. The envelope containing the Ageless-Eye indicator tablet is taped to this window with the envelope's small openings facing away from the wall. The window is then heat-sealed to the opaque film with a small tacking iron, with appropriate pressure. The window is created 5-10 cm from where the final closing seal will be made. This allows

the bag to be reused when the sealed end is trimmed off after the treatment is completed.

## Conservators' Experience

John Burke has been a force behind bringing to conservators the message and methodology of treating museum objects in anoxia bags from the earliest days of this technology. Burke had worked extensively with barrier films and had developed an expertise in their general use for art conservation. He understood well the technical advantages and drawbacks of the different types of polymer- and metallic-film laminate compositions. In a 1992 article in the *WAAC (Western Association of Art Conservation) Newsletter,* Burke described several simple experiments that demonstrated that barrier-film bags based on poly(chlorotri-fluoroethylene) film (Aclar) and containing silica gel as a moisture buffer will maintain a narrow RH range for many years. Conservation practice at the Oakland Museum in California, where Burke worked, had involved the use of such a barrier film to line oak display cases to block fatty acid outgassing, to protect silver objects shipped out for exhibition, to construct low-cost microclimate display cases, and to prepare humidity-controlled storage bags. Thus, Burke was well positioned to design and construct suitable barrier-film containers for the disinfestation of museum objects using anoxia, when Gilberg, with whom he and Valentín had worked in San Francisco, described the effectiveness and requirements of this procedure. Burke's earlier studies on the relative performance of different commercial films as moisture barriers were extended to compare the oxygen transmission rates through the same materials. Initially, he dealt with institutions in the San Francisco Bay Area. More recently, in workshops like the one held in early 1996 in San Diego, California, he has been bringing hands-on experience to a wider audience. At the Oakland Museum, he periodically runs loads of objects through a carbon dioxide treatment in a Rentokil bubble but prefers to use nitrogen anoxia in smaller laminated-film containers. Burke claims that nitrogen provides a higher degree of flexibility when working with individual objects and is faster, cheaper, and safer than carbon dioxide.

Transparent bags can be made using a laminate based on poly(chlorotrifluoro-ethylene), but this material is too expensive for general museum use. A thin layer of aluminum is less permeable to oxygen and moisture than are organic polymers, and a composite with a core of aluminum film is relatively cheap. Burke uses aluminized laminates purchased in 3 ft (91.44 cm) wide rolls to make bags up to 3 × 5 ft (91.44 x 152.4 cm) in dimension. Pinholes and cracks in the aluminum film form even in the most carefully crafted containers, and these sources of leakage can be seen by holding bags up to a strong light and examining the film from the inside out. Burke finds that relatively sizable objects can be rid of insects in less time under nitrogen in these bags than by carbon dioxide fumigation in his Rentokil bubble—generally 1-2 weeks in pouches compared to 4-5 weeks in the chamber. Typically, a customized bag is constructed to be just large enough to hold the infested object along with several packages of oxygen absorber. After all components are placed in the bag, it is sealed almost closed and flushed with nitrogen several times. The excess gas is then pressed out of the bag, and the seal is completed. After several days, the Ageless should bring the oxygen content down below 0.1%, and the Ageless-Eye indicator will turn pink. The bag stays sealed for several weeks, and the oxygen indicator is checked periodically to ensure that it is remaining pink and that leakage is not occurring. After treatment, the seal on one side of the bag is trimmed off, and the disinfested object is put into appropriate storage. The slightly shortened bag is generally reusable for several more treatments.

Burke favors using a quantity of Ageless several times greater than the calculated required amount. He believes this is cheaper than redoing the treatment if

a small leak occurs. The Ageless packages are reused, along with makeup oxygen absorber, in subsequent treatments. He is also redundant in his use of Ageless-Eye because a spent indicator may give a false reading of leakage. After treatment, both the absorber and the indicators are quickly stored for later use in tightly closed, nitrogen-flushed bags. Other conservators have suggested that the extended reuse of Ageless and Ageless-Eye should be done only by highly experienced workers.

Conservators in northern California quickly accepted this new, safe method for fumigating their objects and eliminating infestations. They were provided with instructions, demonstrations, and even a popular buying guide to sources and costs, *Materials and Equipment for Anoxic Fumigation,* which Burke compiled. An example of how this technology was put in place in a small museum is illustrated by the experience of the staff of the Mendocino County Museum in Willits, California. This museum has a fine ethnographic collection and, on occasion, encounters infestation problems. Rebecca Snetselaar, who is both curator and conservator, led a group from Willits on a field trip early in 1994 to the Oakland Museum conservation laboratories, where they were shown the procedures and equipment that Burke had developed for dealing with infestations. With the notes from the field trip and the cost data from Burke's guide, they were able to prepare a proposal that enabled them to obtain an Institute of Museum Services training grant to fund an insect control program. Burke conducted a training session in Oakland for the Mendocino staff and, as a final step, went to Willits to be sure that all procedures were done properly and effectively. This hands-on assistance by an anoxia specialist given at the conservator's facility appears to be important for getting programs into operation at institutions with limited personnel. Nitrogen anoxia for treating infested objects is now used on a routine basis at the Mendocino County Museum. In November 1995, for example, the museum received seven Pomo baskets on loan from a private collector, which it planned to use in a special exhibit. Upon examination, one of these baskets showed signs of infestation. There was a substantial quantity of frass in the basket's tissue-paper wrapping and a 6.35 mm hole in one of the willow foundation rows. The baskets were treated immediately, preventing further damage and protecting the other pieces in the exhibit.

At the Phoebe Apperson Hearst Museum of Anthropology in Berkeley, California, the conservation staff also treats infestations using nitrogen anoxia in bags, but their holdings are much larger than the Mendocino County Museum collection. Accordingly, there are more treatments carried out, and they are more diverse in nature. There is relatively little ongoing infestation with the internal collection. Older pieces contain residual pesticides from past treatments, and this is believed to be a deterrent to reinfestation. The principal source of insect problems is new acquisitions. These are generally held in isolation and periodically checked for signs of infestation to determine if treatment is necessary. In any event, if the nature of the object causes suspicion, treatment is carried out. Moths and dermestids are most often encountered, particularly where woolens, furs, and feathers are involved. Drugstore beetles are an ongoing, annual source of infestation in museum storage. They appear to be extremely selective in their attack of museum objects but travel widely. Dead adults are found in many storage cabinets even though they appear to have done no damage.

Madeleine Fang has been responsible for the conservation of the Hearst Museum collection. She had been using primarily freezing and No-Pest Strips (dichlorvos) to deal with infestation problems, when she became acquainted with insect anoxia through contacts with Mark Gilberg and with Thomas Strang of the Canadian Conservation Institute. Fang learned to make and use anoxia bags with John Burke at the Oakland Museum conservation facility. Because of Marvelseal's low cost, Fang opted to use this aluminized barrier laminate to

make bags with a built-in window to watch the Ageless-Eye monitor. However, after a year of dealing with bags that leaked because of poorly sealed windows, she dispensed with the window and the Ageless-Eye and decided to put her trust in well-sealed bags without oxygen monitoring. Now the improved procedure involves placing the objects inside a Marvelseal bag, flushing three times with prepurified nitrogen, adding the required amount of Ageless, heat-sealing the bag, and then allowing the bag to stand a minimum of three weeks. Leakage problems, which have decreased dramatically, are usually evident within a day, as shown by the collapse of the inflated bag. If leakage is detected within a few hours, the bag can generally be resealed with a portable heat sealer while the object is still in it. Conservators at the museum have dealt primarily with infestations of carpet beetles and casemaking and webbing clothes moths, but there has been at least one object infested with drugstore beetles. To date, the museum has not had a problem with any insects surviving. Freezing and treatment with dichlorvos are still done, however. The pesticide is used with severe infestations, particularly of large objects like a tule boat that was too big to be bagged. The museum owns a Vacudyne chamber designed for use with ethylene oxide (last used in 1989) and a standard 30 $m^3$ Rentokil bubble, but there has been no need to make these units operational for nitrogen anoxia.

At the Metropolitan Museum of Art in New York, Robert Koestler investigated both rigid and flexible reusable chambers and pouches. He found that in terms of ease of bagging, sealing, and maintaining a low-oxygen environment, the pouch was the most successful (1992). The sealing system was a major problem with both types of chambers. The flexible unit was a minifumigation bubble originally designed for use with methyl bromide. Koestler found that it was not possible to get the reusable seal to maintain a sufficiently low oxygen concentration for more than a day. The bags he uses, often as large as 1.1 × 2.3 m, are made from barrier films of low oxygen permeability. After filling a bag with objects, he purges it with argon to an oxygen concentration of 1000 ppm. The gas is humidified by equipment similar to the three-jar system described in chapter 4. Ageless is added for further oxygen control, and both Ageless-Eye and another oxygen monitor are used to follow the oxygen level. Koestler emphasized that bags can be used anywhere and that they are low in cost, easy to set up, and effective. Further, they can be reused until cracks and pinholes form in the wall, making the bags permeable to oxygen.

The Los Angeles County Museum of Art finds that its collection is most easily cared for by the combined use of a large reusable chamber and plastic pouches. Most of its anoxia treatments are done in a converted 1.3 $m^3$ metal Vacudyne chamber, but there is a need for custom-built bags for objects that will not fit inside the chamber—for example, 213.36 × 60.96 cm screens from its Japanese collection. The museum's bags are constructed of Film-O-Rap. Silica gel conditioned to 50% RH is used to control moisture, and the oxygen content is taken down to anoxic levels with packets of Ageless after the bags are purged with nitrogen. Treatments are also done in the transparent bags for those curators who prefer not to have their pieces treated in a metal box. These curators are concerned with the potential for accidents, particularly those that might be caused by humidity levels. They also feel a need to look in on their charges while the objects are undergoing disinfestation. While most pouches are heat-sealed closed, clamps also have been found to provide adequately leak-proof bags. The museum has not found Ageless-Eye to be a satisfactory oxygen monitor. The conservators there prefer to place in the bags a battery-operated, portable Teledyne oxygen analyzer that reads over a 0-1% range.

In Europe the leader in using anoxia in bags has been Nieves Valentín at the Instituto del Patrimonio Histórico Español in Madrid. There Valentín has continued the research she started at the Getty Conservation Institute, developing and

widely demonstrating the procedures and requirements for effective anoxic treatments. Valentín's studies of the minimum assured kill times for eight common insect pests using nitrogen and argon at various temperatures (Table 2.3) were all done with plastic bags. Her research results were tested at the archives of Reine de Galicia in Spain. This historic building has six floors with 9000 m of shelving holding archival material. Approximately 20% of the document collections were contaminated by furniture and powderpost beetles. A two-year program using insecticides had been only partially successful in reducing the number of adult insects and larvae found in documents. Treatment was switched to Valentín's procedure, in which infested bundles of documents were placed in large oxygen-barrier bags along with Ageless. The bags were purged with argon containing 200 ppm oxygen at 30% RH for 10 hours and then sealed and held at 30 °C for about 4 days. Most recently, this program involved preparing ten such treatment units each day. On average, the infested bundles contained twelve adults, twenty-six larvae, and eleven pupae, all of which were eliminated by anoxia. The procedures used in this work have also been extended to treat the collections in the archives of Spain's La Coruña.

In the United Kingdom, John Newton of the Research and Development Division of Rentokil Division PLC, David Pinniger of Central Science Laboratories, and Madelaine Abey-Koch of the National Trust have demonstrated the construction and use of a very large barrier bag for anoxic disinfestation. They have provided a well-documented description (1996) of its use in the treatment of a collection of curtains from the Mottisfont Abbey in Hampshire. The project was undertaken in 1995 both to provide insect control and to evaluate the parameters of the treatment. The curtains were commissioned in 1938-39 for the drawing room of the abbey as part of the decor created by Rex Whistler. The ten curtains, each 4.2 m long and 2 m wide, were made of silk trimmed with imitation ermine composed of combed white wool and cotton. The silk had deteriorated over the intervening years, and the curtains had been attacked by insects, primarily carpet beetles but also clothes moths and fur beetles.

A dual-purpose experimental design was developed that would determine the amount of time required for complete mortality of varied carpet beetles and webbing and casemaking clothes moths (three species typically found in fabric collections of this type) while, at the same time, achieving a total disinfestation of the curtains. The fabrics were maintained in nitrogen with less than 4000 ppm oxygen at 50-65% RH and 24 °C. The length of the treatment time was determined by placing larvae of each of the three test species in the bag along with the curtains and removing samples periodically to evaluate the mortality rate. The monitoring of insect mortality required a bag of unique design. The barrier film consisted of a 9 μm aluminum core bonded to a 60 μm layer of low-density polyethylene and a 12 μm layer of poly(ethylene terephthalate) to provide support and mechanical strength. A large section of film was folded about the curtains, which had been first cleaned, lined with acid-free tissue paper, wrapped in cambric, and secured with brass pins. The barrier film was folded so that the polyethylene layer was on the inside. The resulting unsealed container measured 5.7 × 2.5 m and held the curtains near the long fold on the left side. Before the film was folded, sampling and gas transit ports were installed in such a way that they would be positioned at opposite ends of the right side of the finished bag. The open sides were sealed with an impulse-type heat sealer whose 24 volt heating bar switches off automatically after a few seconds to avoid overheating, as all such sealers do. Continuous seams were made along the margins of the two short sides. Further sealing along the remaining edge was done in a manner that allowed small cages of insects to be sealed in the bag and exposed to its atmosphere. Subsequently, new seams were made that allowed samples of the insects to be removed periodically for examination while maintaining anoxic conditions in the bag.

The treatment was designed to achieve and maintain an oxygen concentration below 4000 ppm. The oxygen concentration within the bag was measured at intervals using a Bedfont Scientific Instrument BT 2000 trace-oxygen analyzer connected to a sampling port. Similarly, the RH was monitored using a Novasina MIK 3000 thermohygrometer. On the first day, the bag was taken through four flushing cycles during which some atmosphere was withdrawn, creating a slight vacuum, and then the bag was reinflated with humidified nitrogen containing no more than 20 ppm oxygen. This brought the oxygen level to a satisfactory 2500 ppm. Two more flushings were made on the second day. Then the corners on the left side of the bag were cut open, twenty-five packets of Ageless Z-2000 and twenty packets of Atco 3000 were inserted, and the unit was resealed. The oxygen concentration at this point was 5600 ppm. It fell to 1300 ppm after 7 days, 300 ppm after 11 days, and 100 ppm after 21 days. It remained at this level over a total holding period of 73 days. The RH was maintained at 50-70%, and the temperature in the unheated storeroom late in the year ranged from 4 to 20 °C. The insect mortality phase of the study showed 100% kill of all life stages in less than 12 days. Following treatment, the dead carpet beetle larvae in the curtains were removed, and the curtains were again cleaned. A week before the curtains were rehung, the display room was sprayed with bendiocarb wettable powder, an insecticide effective against carpet beetles; new carpets were installed; and a daily program of vacuuming was begun. The drapes show no sign of damage or change.

After its work at the Mottisfont Abbey, the Rentokil team went on to build even larger barrier-film bags, when it was called on to solve a pest control problem by the French Ministry of Culture (Smith 1995). The French agency was concerned about the infestation, primarily by carpet and lyctus beetles, of very large oil paintings that were distributed among churches in the Provence region of southern France. For the treatment, forty paintings were gathered in 1995 and brought to a heated, secure warehouse in Marseilles. The paintings, some as large as 5 × 3 m, were placed on specially designed racks and installed in one of three barrier-film bubbles prepared on-site. These containers, each over 88 m³ in volume, were the largest ever constructed for treatment by oxygen deprivation. They were made of a laminate composed of polyethylene, aluminum, and poly-(ethylene terephthalate) films, similar to the laminate used to make the anoxia bags for the Mottisfont Abbey curtains. Large sections of the film were cut and formed into a chamber by heat-sealing with a specially designed wide-jaw sealer. They were fitted on opposite sides with lead-in ports for gas sampling, as well as with gas inlets and outlets.

The treatment units were first partially evacuated over a 10-minute period and then refilled with high-purity nitrogen. The nitrogen was passed through a split-stream humidifier adjusted to provide ambient RH to the gas as it passed into the bubble. This partial evacuation and purging was repeated several times. With such large bubbles, ten to fifteen flushings may be necessary before a target oxygen concentration of 2000 ppm can be achieved. The general processing conditions defined for this project were developed from data provided by the 1993 Rust and Kennedy study. While the design called for maintaining an oxygen concentration below 2000 ppm at 24 °C for 7-14 days, the actual oxygen level was well below 1000 ppm all of this time. After five flushings, a Bedford 415 trace-oxygen analyzer was connected to one of the gas sampling ports. When the oxygen concentration was found to have fallen below 2000 ppm after additional flushings, packets of oxygen absorber were placed in the chamber. These effectively removed much of the small amount of oxygen entering the containment by diffusion, pulling the oxygen level down below 1000 ppm without difficulty. Levels below 500 ppm were attainable but required that the evacuation-purge cycle be repeated when oxygen levels moved into the 500-1000 ppm region. Generally, it was necessary to do this flushing primarily during

Figure 5.1

Anoxia treatment of large paintings in barrier bags, conducted in Marseilles in 1995.
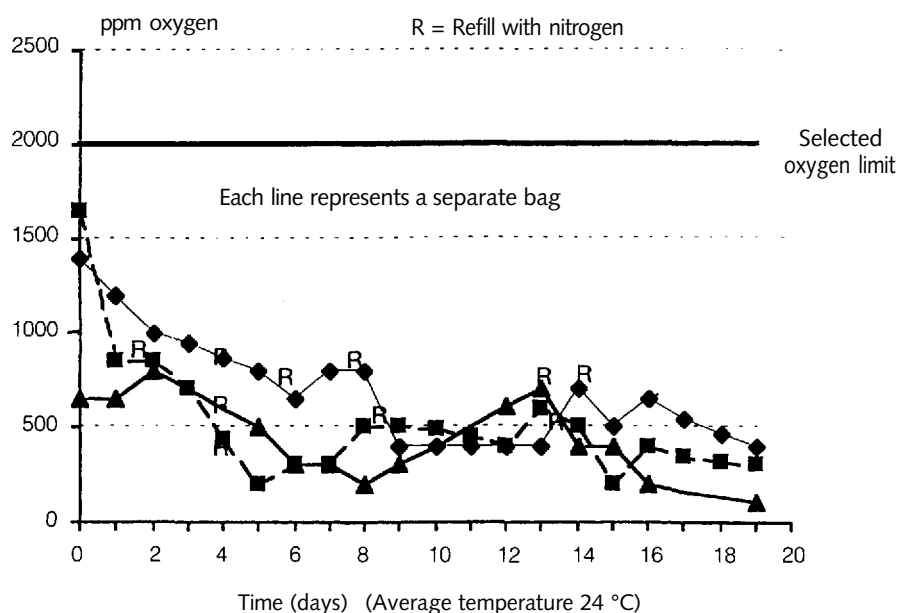
the early days of the treatment (Fig. 5.1). All paintings were rendered free of insects and were returned to their churches.

## Comments

In many of the treatments described in this chapter, and in general, conservators often became involved in unnecessarily long treatment times, complex bag constructions, and difficult insect examinations. Fortunately, the studies conducted by Rust and Kennedy on minimum assured kill times for the most resistant species provide the information needed to greatly simplify treatment procedures. The use of metallized films is an option to be used only when cost problems are pressing. Effectiveness of treatment should come first, and the use of clear plastic barrier films is recommended.

# Chapter 6

# Anoxia Treatment in a Dynamic Mode

Treating infested objects in a dynamic mode with a flowing stream of nitrogen is convenient and effective in situations with special needs or unique problems. This treatment approach has been done using small pouches, as well as larger chambers built around sizable or awkwardly proportioned objects. This method is often routinely carried out with pouches, while larger operations tend to be sui generis. The dynamic mode has some special features that are characteristic and are seen whenever this form of anoxia is used. When very dry nitrogen passes continuously over an object, with time and at ambient temperatures, deeply held water will be pulled out of the object, and irreversible damage could occur. Equilibrium buffering will not take place, and humidification must be used. An exception to this may be the treatment of waterlogged objects where both anoxia and drying may be desired. In addition, oxygen absorbers are not employed in the dynamic mode; removal of oxygen is achieved entirely by purging.

## Treatment in Small Pouches

Lesley Bone, conservator at the Fine Arts Museums of San Francisco, California, has been using the dynamic approach to nitrogen anoxia as her standard treatment. Bone became aware of its potential for treating infestation problems from her association in the early 1990s with Mark Gilberg, who was then working in the San Francisco area. Anoxic treatments at the Fine Arts Museums began in 1995 using reclosable zipper bags based on Aclar. The 1.2 $m^2$ bags were supplied by Conservation Support Systems. In a typical setup operation at the museum, prepurified nitrogen is first passed through a bubbler system to bring the gas to 50% RH. It then passes into the treatment bag via tubing attached by an O-ring Swagelok fitting. The bag also contains a low-range Teledyne oxygen sensor. The fitting is attached to an area of the film surface that has been strengthened with overlays of a stout transparent tape. After the infested object is placed in the bag, the zipper is only partially closed, to allow a rapid nitrogen inflow that brings the oxygen concentration down to 1000 ppm. The zipper is then sealed as tightly as possible, and the nitrogen flow is slowed to maintain the oxygen level below 1000 ppm. Runs of 2 weeks have been sufficient to kill species endemic to the museum's collection, namely powderpost beetles and webbing clothes moths. Incoming wooden ethnographic materials—for example, African carvings—may contain termites and are routinely treated in the same manner. Bone has made bags by hand from Marvelseal film. But she finds this to be more work than using zippered bags, which can be reused about three or four times.

David Casebolt, museum specialist in conservation with the San Francisco Maritime National Park, has used a variety of procedures to keep the park's archival collection free of insects. These include anoxia in refitted and gasketed aluminum containers from navy surplus, as well as oxygen deprivation with Ageless alone when treating small objects in handmade pouches. His facility is heavily involved with archival material that is most frequently infested with silverfish. The powderpost beetle, beetles in the Dermestidae family, and termites are other species often encountered. Casebolt uses nitrogen anoxia in the dynamic mode for more sizable objects, which go into larger bags. A major area of acquisition for the maritime complex is archival collections, which are often received in a damp condition. A bubbler system, which would normally be used in the dynamic mode, is usually not employed with these collections. In this way, the anoxia treatment is also used to dry the collection.

Christoph Reichmuth has used the dynamic method, which he calls "nitrogen flow fumigation," for projects ranging from studies of the effect of temperature on kill time to treatments of wooden sculpture and other objects. The mortality rate studies were directed at webbing clothes moths, subterranean termites, and a variety of beetles that are common museum pests (Reichmuth et al. 1993;

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Unger, Linger, and Reichmuth 1992). For these studies, infested objects as well as control insect specimens were placed in polyethylene bags that were purged one to three times with nitrogen. The pouches were inflated and kept at a 1% oxygen level with a slow nitrogen inflow at 5-10 Pa. The RH was held at 55-60% with aqueous solutions of glucose or calcium nitrate. The minimum kill times recorded by Reichmuth were substantially longer than those found by Valentín or Rust and Kennedy. However, he had used a higher oxygen level and lower temperatures than the other investigators. For example, Reichmuth determined a kill time of over 500 hours for the powderpost beetle at 25 °C and 1% oxygen, while Rust and Kennedy indicated that only 120 hours are needed at 0.1% oxygen at the same temperature. For the furniture beetle, Valentín found that 168 hours were needed to eliminate all life stages at 30 °C with 0.1% oxygen. Reichmuth required 500 hours at 35 °C and 1% oxygen; dropping the temperature from 35 to 16 °C led to an increase in treatment time of 2 weeks, for a total treatment time of 836 hours. Quite clearly, higher oxygen concentrations are responsible for extremely long treatment times. Additionally, as has now been thoroughly established, lower temperatures extend the treatment period even further.

Reichmuth describes several other successful treatments achieved with nitrogen flow fumigation, albeit with excessively long exposure times at the high oxygen level he uses. Two sculptures and a painting—all large, wooden pieces infested with furniture beetles—were rid of pests by runs conducted at 25 °C for 5 weeks. The painting, by Lucas Cranach the Younger, is a portrayal of the Last Supper on a 2.1 × 2.6 m wooden table. One of the treated sculptures was a polychrome altar, a shrine to the crowning of Mary. The altar holds three figures, each 1.1m high. The other sculpture was a Pietà, without mounting, made of limewood and measuring 85 × 55 cm. For these treatments, control samples of all life stages of the furniture beetle—some encased in wood to examine the diffusion of nitrogen into this medium—were added to the container along with the infested object. The control insects served as monitors of the treatment's effectiveness. Reichmuth noted that the disadvantages of nitrogen fumigation were the long treatment time at 1% oxygen and the lack of a residual protective effect.

## Treatment in Constructed Containments

### Objects at the J. Paul Getty Museum

In 1988, when Gordon Hanlon came to the J. Paul Getty Museum in Malibu, California, to work as a conservator in the Decorative Arts Conservation Department, he found that problems with insect infestation occurred infrequently. New acquisitions, primarily furniture from Europe, were fumigated and made free of insects close to their point of origin prior to shipment. When an in-house need for disinfestation arose, objects were treated with methyl bromide at a fumigation facility about thirty miles away. However, a variety of concerns about transportation, security, safety, and cost of this procedure were raised, making it desirable to find a procedure that could be done safely at the museum. These concerns came to a focus in 1991 when a 220-year-old French fire screen made of walnut was found to have a hole penetrating the wood with a small amount of frass beneath the hole. A pile of eggs was also uncovered, evidence of an infestation by furniture beetles. This event occurred at about the time that workers at the Getty Conservation Institute were demonstrating the effectiveness of nitrogen anoxia, and Rust and Kennedy were gathering data on the conditions generally needed to kill all life stages of the most common museum pests. Hanlon decided to try nitrogen anoxia on the fire screen. He believed that he could maintain the required treatment conditions by building a container around the object and purging the system with humidified nitrogen. An Aclar pouch was

Figure 6.1
Italian armchair under treatment.
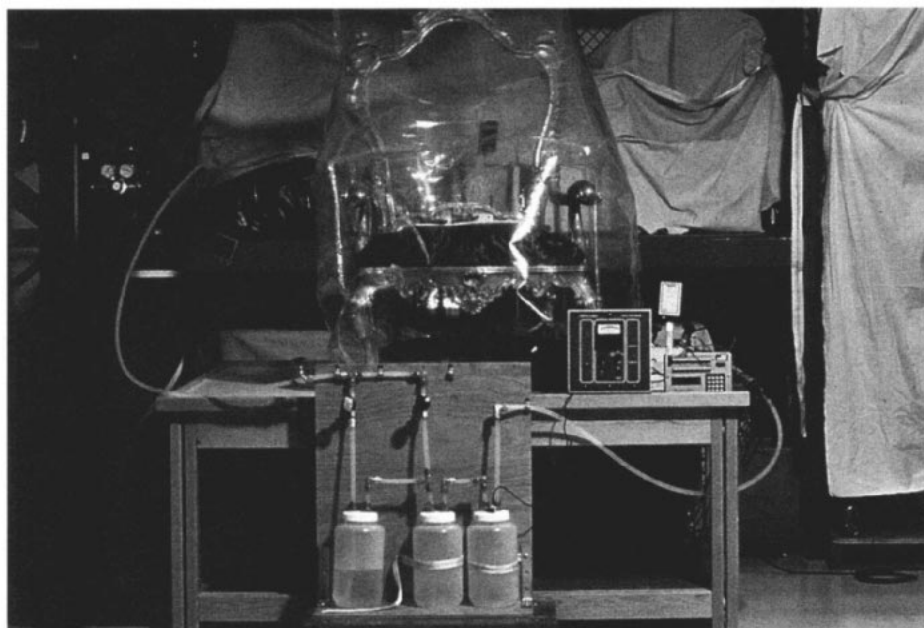
custom-tailored into a tapered, almost triangular, shape to fit the fire screen. The construction, done with a small, hand heat sealer, took only about two hours, although Hanlon believes that a larger bar sealer of the type used in paper conservation would have shortened the construction time. Dynamic nitrogen anoxia, as previously noted, requires humidification. This was done using the split-stream humidifier described in chapter 4 as well as Arten gel as a humidity buffer. The system was first flushed rapidly with nitrogen containing only 5 ppm oxygen until the oxygen level in the container fell to 1000 ppm. The nitrogen flow was then decreased and adjusted to maintain the oxygen concentration at 1000 ppm, and this was monitored by aTeledyne model 316 trace-oxygen analyzer over a two-week period.

Following the successful elimination of insects from the fire screen, the Aclar bag was reused to treat a small French console table that was infested with wood-boring insects. The third object in this early series of treatments was substantially larger, a 260-year-old Italian armchair that was infested with furniture beetles. A 1.5 m$^3$ container, again of Aclar laminate, was constructed to fit around the chair, which was placed on a table holding much of the monitoring equipment and the three-bottle split-stream humidifier (Fig. 6.1). The temperature and RH were measured with a Vaisala HMP 133Y monitor. The oxygen level was followed with two instruments: a GC Industries oxygen monitor, model GS502, with a 33-475 sensor that provides data down to 1000 ppm; and the Teledyne model 316 analyzer, which can determine oxygen levels to a few parts per million. Data were collected by a Campbell Scientific 21X data logger and stored. The careful, continuous recording of oxygen concentrations in this study provided insight into the mechanics of the gas flow needed to achieve and maintain oxygen levels below 1000 ppm during a dynamic run. In a preliminary experiment to determine the rate of oxygen leakage into the system, the bag holding the chair was first purged with nitrogen containing 5 ppm oxygen at 55% RH at a high input rate of 7 l min$^{-1}$, while temperature, oxygen concentration, and RH were monitored. This lowered the oxygen concentration to 400 ppm in 40 hours. At that point, the inlet and outlet valves were closed, and a slight bulging of the treatment container indicated that the pressure inside was somewhat higher than the pressure outside. Leakage began to equalize the pressures over 24 hours. This corresponded to a slow increase in oxygen concentration over this period, followed by a faster increase over the ensuing 72 hours until

**Figure 6.2**
Determination of oxygen leakage rate during treatment of Italian armchair.
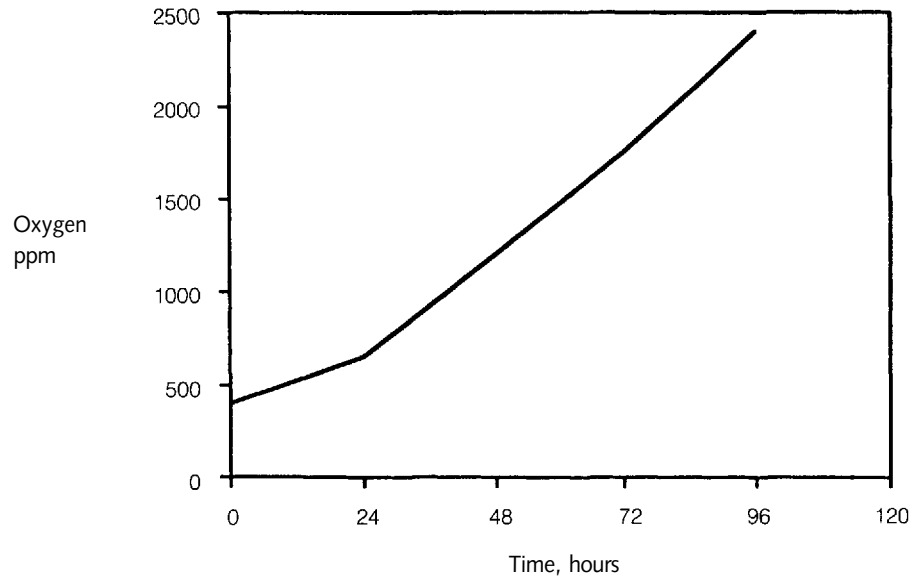


the pressures roughly equalized (Fig. 6.2). During this preliminary experiment, the oxygen leak rate was determined to be 490 ppm (735 ml) per day.

During the actual treatment of the chair, the bag was initially purged with nitrogen at 7 l min$^{-1}$ for 60 hours, and this brought the oxygen concentration to 800 ppm (Fig. 6.3). The nitrogen flow was then decreased to 0.5 l min$^{-1}$, and the exit valve was partly closed. The oxygen concentration remained relatively constant, increasing to only 900 ppm after an additional 120 hours. This demonstrated that a low flow rate of 720 l per day into a 1500 l bag can compensate for the leakage of 735 ml of oxygen per day into the bag. It was necessary to change the nitrogen cylinder at this point, and the flow was increased to 2.0 l min$^{-1}$, where it was maintained for the rest of the 2-week treatment. This higher rate brought the oxygen down to 400 ppm, a level where anoxia is even more effective than the recommended 1000 ppm for dispatching insects. Failure to find any sign of insect life after four years posttreatment is proof of the successful disinfestation of the fire screen, the wooden table, and the armchair. This led Hanlon to conclude that a continuous nitrogen flow into a large Aclar plastic bag can maintain an oxygen concentration well below 1000 ppm for the duration of treatment and provide a highly effective procedure for pest control.

**Figure 6.3**
Oxygen concentration in treatment bag during disinfestation procedure.
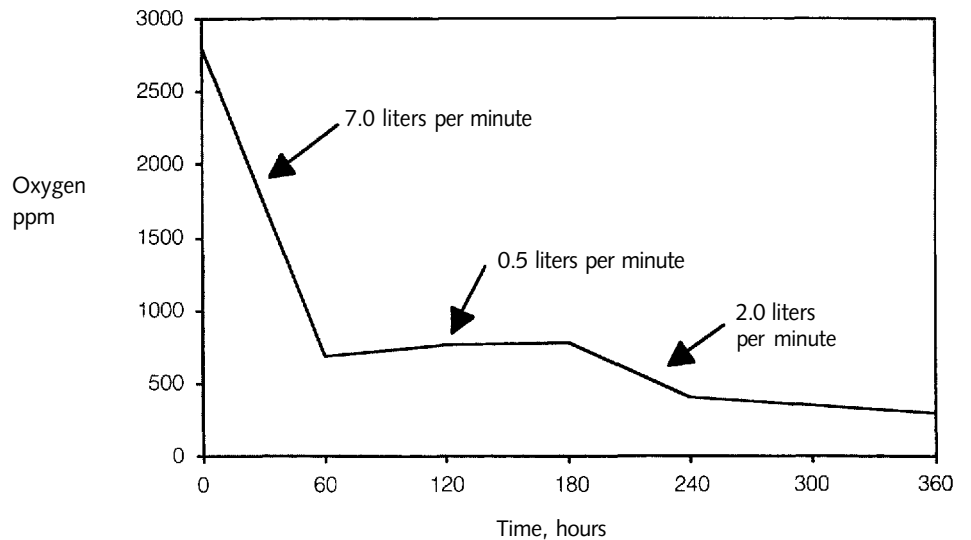
### Back Seat Dodge '38

Steven Colton, conservator at the Los Angeles County Museum of Art (LACMA), realized that he had a major infestation problem one day early in 1993 when he found six webbing clothes moths during a routine inspection of one of the museum's most popular exhibits, Edward Kienholz's *Back Seat Dodge '38*. Five juvenile insects were inside the car, eating floor carpeting, and a sixth moth flew out of the trunk when it was opened. The contemporary sculpture was controversial when it was acquired in 1966 because one door was open, revealing a sculptural portrayal of a drunken, life-size couple making love in the rear of the car. Los Angeles County supervisors denounced the work as revolting and blasphemous, and pressed the museum to remove it. The trustees unanimously rejected their appeal as an infringement on the museum's obligation to present works that represent an honest statement by a serious artist. The sculpture remained, although for a while, the car door was closed and a museum guard was posted to prevent viewers from peeking inside. Twenty-seven years after the uproar that rocked the art community, *Back Seat Dodge '38* suffered another attack. But, as described by the *Los Angeles Times,* "this time, the assailants weren't two footed politicians, they were winged moths and when they got a load of the Dodge, the wayward insects didn't see obscenity, they saw home and dinner" (Muchnic 1993).

The options for dealing with the problem at the time were limited. The sculpture was a complex piece containing a wide range of materials: leather, paper, cloth, metals, synthetic resins, glass bottles, flocking material, and a variety of adhesives. The museum was loath to send it off-site for pesticide treatment because transportation would risk damage to the fragile, deliberately dilapidated artwork, and conventional fumigation chemicals might harm the materials. The car was too large to fit into a pouch, and there were no big bubbles or chambers available similar to ones that others have found suitable for the anoxic treatment of large objects. LACMA turned to the Getty Conservation Institute for help. The museum's timing was good: GCI personnel Shin Maekawa and Vinod Daniel, working with Gordon Hanlon of the J. Paul Getty Museum, had just gone through the process of learning how to use barrier film to build containments about large, awkwardly sized objects for dynamic anoxia treatment. At the same time, a GCI-sponsored program at the University of California, Riverside, under the guidance of Michael Rust, was collecting the data needed to deal with webbing clothes moths. The studies by Rust and Kennedy found that holding this species of moth for 96 hours at 25 °C in nitrogen containing 1000 ppm or less oxygen was sufficient to create confidence that all life stages were exterminated. These data defined the conditions for the successful anoxia treatment of the *Back Seat Dodge '38* carried out by the LACMA and Getty teams.

The gallery of the Anderson Building, where the sculpture had been on display, was closed for ten days while the container was built and the treatment carried out. The car, on casters, was rolled onto a sheet of plywood placed over a larger sheet of transparent laminate film based on Aclar as the oxygen barrier (Fig. 6.4). Additional sections of this film were cut, assembled, and heat-sealed to the bottom film layer, creating a tight-fitting container completely enclosing the sculpture. At its maximum dimension, the container measured $6.1 \times 3.7 \times 1.7$ m. It had a surface area of 23 m$^2$, and construction required 44 linear meters of heat-sealing. The estimated volume of the bag was 5.5 m$^3$. However, void spaces such as the interior of the car and trunk, the area under the hood, and other spaces were filled with nitrogen-charged balloons to reduce the chamber volume to 4.7 m$^3$. This is now considered unnecessary. The oxygen leak rate through the bag was 167 ppm per day during the initial purge. Two industrial-grade nitrogen streams, each flowing at 5400 l h$^{-1}$, were passed first through separate split-stream bubblers, then into the bag, and out an exit duct. As the

Figure 6.4

Kienholz's *Back Seat Dodge '38* under treatment. (Photo courtesy of the Los Angeles County Museum of Art.)
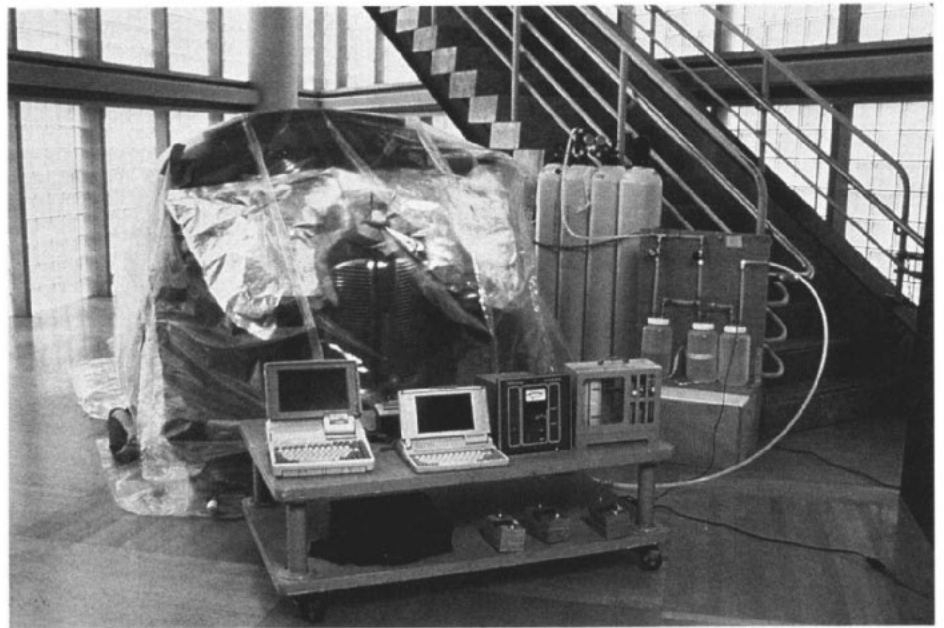
oxygen level dropped, the industrial grade was replaced with prepurified nitrogen. Continued rapid purging with nitrogen containing 5 ppm oxygen at 45% RH quickly brought the oxygen level in the bag to below 1000 ppm, where it was maintained for 8 days under a continuous gas flow at 30 l h$^{-1}$. Temperature, RH, and oxygen concentration were monitored continually. After treatment, dead larvae and adult insects were found in the car. When the project was completed, the equipment was dismantled, and the car was rolled out of the bag and put back on display.

**The Spanish Piano**

Early in 1992 inspection of a historic grand piano belonging to the Fundación Bartolomé Pons Cabrera in Valencia, Spain (Fig. 6.5), disclosed that the piano was host to a major infestation of furniture beetles, both adults and larvae, and lesser amounts of deathwatch beetles. The piano was treated by Nieves Valentín



Figure 6.5

Bartolomé March piano being prepared for treatment. (Photo courtesy of Nieves Valentín.)

of the Institute de Conservación y Restauración de Bienes Culturales (now called the Instituto del Patrimonio Histórico Español), Madrid. The first step was to build a large 6.3 m$^3$ tent around it from Saranex film (a laminate of polyethylene, poly[vinylidene dichloride], and ethylene-vinyl acetate copolymer made by Dow Plastics). To start the treatment, the tent was partly evacuated and refilled twice with high-purity argon to quickly lower the oxygen content. The argon, conditioned to 40% RH, was allowed to flow through the chamber for 8 days. The oxygen level fell to 100 ppm during this period, while the temperature was maintained at 24-25 °C. After 8 days, thirty packets of Ageless Z-2000 were put into the bag, the argon flow was terminated, and the unit was sealed so that it operated in a static mode for an additional 6 days. The effectiveness of the treatment was demonstrated by the complete mortality of the larvae of longhorn borer beetles that were placed in the system as monitors.

# Reusable Anoxia Systems

## Flexible Chambers

### The Getty Barrier-Film Tent

The craftsmanship required to make pouches can be applied to fabricating reusable containments of any size. Maekawa and Elert (1996), members of the Scientific Program at the Getty Conservation Institute, have described the construction of a prototype 10 m³ barrier-film chamber that they prefer to call a "tent." This tent, with accessories and a large-capacity nitrogen source, offers a system that provides convenient and effective treatment (Fig. 7.1). After purging, it will maintain a nitrogen atmosphere containing less than 3000 ppm oxygen at ambient temperature and at suitable moisture levels for a month.

A key parameter of this system is the oxygen permeation rate of the tent wall. This is important because it determines the amount of nitrogen needed over the treatment period to keep the oxygen level below critical concentrations. The authors have found that wall material based on poly(chlorotrifluoroethylene) or aluminum film is not suitable. Oxygen transmission rates of laminates using the fluoropolymer were too high (Tables 3.1 and 3.2), while films based on aluminum foil required great care to avoid forming pinholes on repeated use. Filmpak 1193, a strong laminate of somewhat limited clarity with an oxygen permeation rate of only 0.1 cm³ m⁻² per day, was chosen. A schematic of the tent is shown in Figure 7.2. The film was purchased as a 1 m wide roll. Sections of film were heat-sealed to form panels large enough to construct a tent measuring 2 m on each side and 2.5 m high. Sealing was done with a tacking iron, approximately 2 cm wide, instead of a bar sealer.

Stout loops of film were formed along two parallel top edges to attach a lifting device that made it possible to raise the tent for easy placement of objects. The tent was designed to fit over a support frame made of aluminum tubing. Sealing was done by extending the film outward from the bottom of the tent to create a 30 cm flange that pressed against a flat base. The base could be as simple as a smooth concrete floor coated with several layers of paint or a large flat sheet of plastic. To prepare a seal that would maintain the oxygen level below 3000 ppm for the duration of treatment, two lightly greased large square gaskets, prepared from self-adhesive rubber strips, were first placed in parallel on the flange area of the base. Then the tent was lowered, and its flange was adjusted to be wrinkle free. Lengths of cast acrylic, 1 cm thick and 15 cm wide, were placed on the flange perimeter, and these were weighted with bags of lead shot to provide a hermetic seal (Fig. 7.3). Over a one-month period, an oxygen leak rate of 50 ppm per day was experienced. This made it possible to keep the oxygen concentration well below 3000 ppm for more than four weeks without further purging, after starting at 1000 ppm. An adequate seal could not be obtained by taping the flange to the floor, despite many attempts and variations.

Figure 7.1
Schematic of anoxia treatment system.
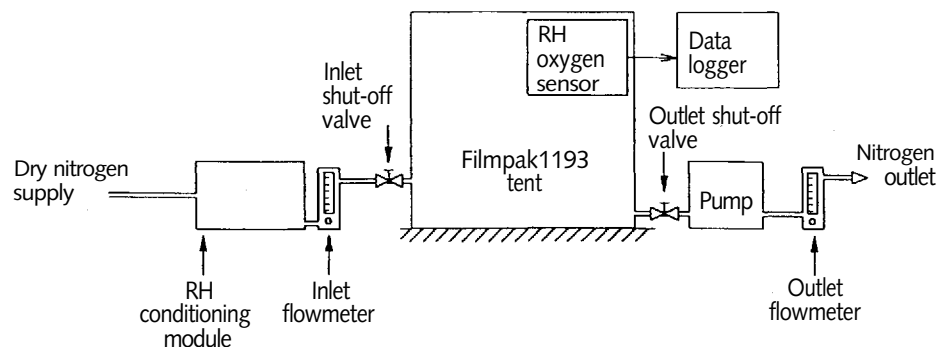
**Figure 7.2**
Schematic of anoxia tent.

Rope (lifting device)

Aluminum frame
(lifting device)

Nitrogen out

Tent cover
Filmpak 1193

Acrylic plates

Nitrogen in

Rubber strip

Acrylic sheet
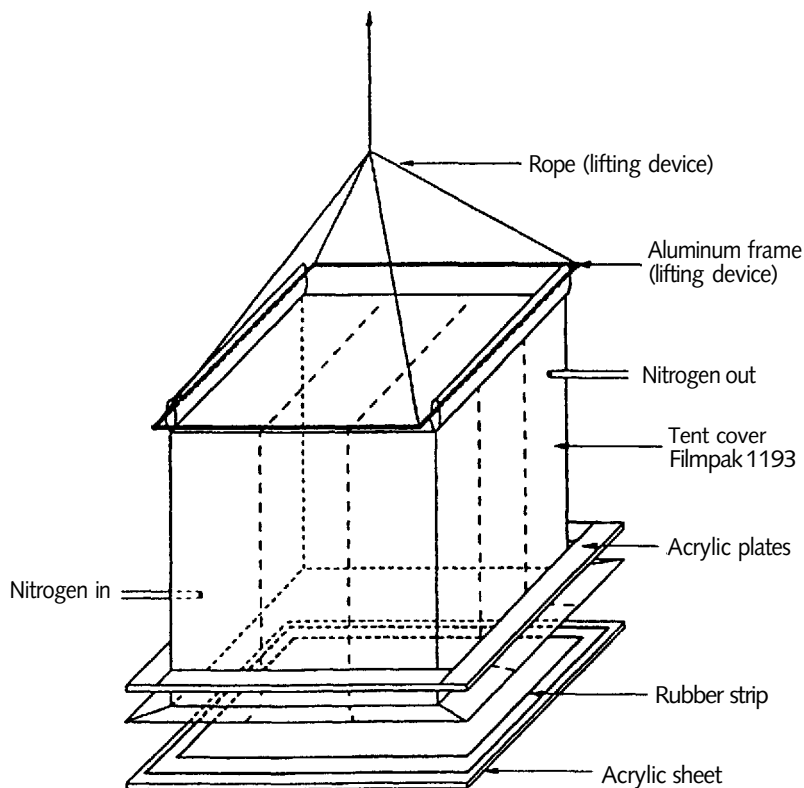
Large systems require substantial amounts of nitrogen during the initial purge, and this can be delivered from a bank of cylinders hooked up to a manifold. When especially large quantities of gas are required, an easier and cheaper approach is to use a tank of liquid nitrogen or the Prism nitrogen generator offered by the Air Products and Chemicals Corporation (chapter 3), which can supply 41 $m^3$ of gas per day containing less than 100 ppm oxygen. The chamber also requires the type of RH conditioning module described in chapter 4 and shown in Figure 4.1. Equivalent throughputs in the flowmeters would deliver an RH of 50% if the nitrogen bubbling through the water became saturated. At high gas flow rates this does not happen, and it is necessary to calibrate flow rates against measurements of RH inside the tent.

The treatment system, including the tent, was constructed in sixteen hours and cost US$2,500 for materials. The cost could be reduced by one-third by using poly(vinyl chloride) tubing instead of aluminum for the frame, and sand instead of lead for the weights. The leak rate of the 10 $m^3$ unit was only 50 ppm (500 ml) oxygen per day. This allowed the system to be purged to 1000 ppm oxygen and then left in a static mode without any oxygen absorber for more than 30 days before the oxygen level would rise to about 2500 ppm. The

**Figure 7.3**
Schematic of hermetic seal.



15cm

Filmpak 1193

Lead weight

Acrylic plate

1 cm

Self-adhesive rubber strips
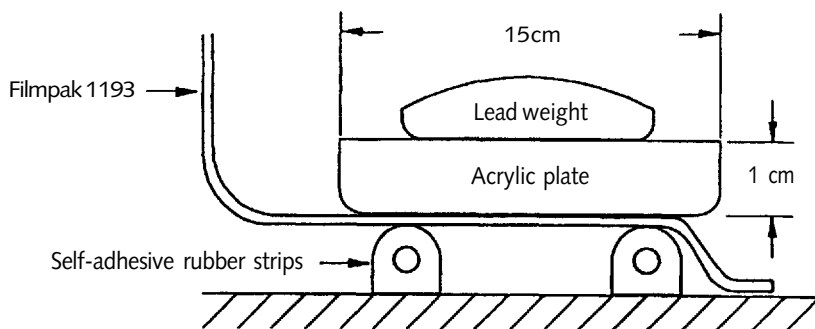
development of this flexible and reusable chamber resulted in a superior system. The prototype, however, was not used for any experimental study or for the treatment of insects. The goal of the Getty project was to demonstrate the production and testing of an anoxia chamber that could be built in a museum conservation facility.

## The Small Rentokil Bubble

In 1991 Rentokil offered a 6 $m^3$ off-the-shelf portable and reusable fumigation system for use with carbon dioxide. In 1997 Elert and Maekawa evaluated an updated model of the small Rentokil bubble for its ability to kill insects by nitrogen anoxia. This unit was similar in appearance to the earlier version, but the polymer films forming the flexible walls had been changed to provide lower oxygen permeability. The bubble measured 1.8 $m^2$ at the base and 1.7m high and had an overall surface area of 19 $m^2$. Two ports provided for gas passage, and hermetically sealed connectors were used for sensor signal lines. The shape was sustained by internal pressure instead of a frame. Satisfactory sealing of a well-greased zip-lock-type closure was confirmed by leak detection procedures. The bubble was easily purged to 1000 ppm oxygen using liquid nitrogen as the source of the nitrogen gas. A Teledyne trace-oxygen analyzer, model 317, was used in a series of tests to determine the oxygen permeation rate. Over one- to two-week test periods, the average oxygen leak rate was found to be 450 ppm per day. This is close to the value calculated from the oxygen transmission rates of the barrier films used to fabricate the bubble. Only 1.9 l $min^{-1}$ of nitrogen is required to maintain the oxygen concentration below 1000 ppm, and this makes the bubble very effective for anoxia. The evaluation of this unit did not extend to using it for studies with insects.

Dale Kronkright, senior conservator at the Museum of International Folk Art, part of the Museum of New Mexico (MNM) in Santa Fe, has adapted the 6 $m^3$ Rentokil bubble to treat the museum's collection with nitrogen anoxia. Kronkright chose this approach after an encounter with a severe infestation shortly after joining the MNM in November 1993. Clothing moths had attacked a collection of Turkish folk art containing a lot of woolen textiles, and this was dealt with by freezing all of the textiles. Fortunately, all of the objects were in one gallery, and the infestation was contained, but the episode was sobering. It forced the museum to close the show for three weeks, which was a severe disruption. The museum considered alternative procedures for future infestation problems and decided to order a chamber for anoxia treatments.

In September 1994, Kronkright negotiated with Power Plastics, the company that manufactures the bubbles for Rentokil, to prepare a flexible chamber to meet the museum's needs. He wanted a moderately sized unit that would operate effectively with nitrogen instead of carbon dioxide. He chose nitrogen because he believed it to be more efficient and faster than carbon dioxide. Speed of treatment was important because of the demands of the museum's program schedule. A bubble—essentially a 1.8 m cube—was constructed. The tent collapses to fit into a duffel bag, 0.6 m in diameter and 0.9 m long, and has been set up in a number of different galleries, collections areas, and loading docks. Infested objects, either boxed, supported in packing, or stacked on portable shelving or roll carts, are moved into the bubble through a front door. The tent is about one-fifth the size of the Rentokil bubble conventionally used with carbon dioxide. The size was selected so that the tent could be easily moved to any of MNM's four component museums, which are at separate locations in Santa Fe. This downsizing also substantially reduces the amount of nitrogen needed to purge or fill the unit. The need for nitrogen was further reduced by constructing the chamber walls of a composite film based on a chloropolymer. This has a

lower oxygen permeability than Rentokil's larger 30 m³ chambers, which are designed for use with carbon dioxide and built of reinforced polyester.

Once the unit was delivered, the staff had to do a great deal of adjusting and manipulation of exhaust valves and seals to reduce leakage to manageable levels so that runs could be made without the excessive use of nitrogen. A document pocket with a large hole had to be heat-sealed closed. The triple zip-lock seal leaked continuously and had to be caulked. Packing the main seal with house-hold petroleum jelly also reduced the rate of oxygen permeability.

The unit was used seven times in 1995. The chamber was initially fitted with an adjustable internal framework of PVC pipe. Tetrachloroethylene is used as the leak-detecting vapor with a TIF Instruments model 8800 gas detector. The bubble is inflated to a positive pressure with 99.95% nitrogen until the walls and ceiling of the bubble are distended. The gas detector is then used to find leaks along all closures, valve fittings, and seams. Buckled or improperly seated areas of the zip-lock seal are easily found using this method. After a proper seal has been ensured, the chamber is flushed with nitrogen until the oxygen level is 2000 ppm or less. One T-cylinder of nitrogen lowers the oxygen concentration from 20% to 3%. The level decreases from 3% to 1% with a second tank, from 1% to 6000 ppm with a third tank, and from 6000 ppm to 2000 ppm with an additional half tank. A Cole-Parmer 32014-13 direct-reading flowmeter is attached to the exit regulator and allows the flow of nitrogen to be reduced to the leak rate of the chamber, which is about 100 ppm in 24 hours. Kronkright has found that the lowest rate of oxygen diffusion into this bubble attained so far is about 500 ppm in 24 hours. With the slow dynamic flow of nitrogen made possible by the addition of the flow meter to the exit regulator, the MNM bubble can maintain an oxygen level of 2000-3000 ppm for a 72-hour period. When the oxygen exceeds that level, the bubble is flushed again to bring the level back down to 2000 ppm. Treatment runs last 10 days, and this requires the bubble to be repurged twice during the run. Each run requires six cylinders of nitrogen. When needed, the chamber is humidified with the conventional three-bottle bubbler system described in chapter 4.

The bubble was used to eradicate a number of species that had afflicted the museum's collections, including casemaking and webbing clothes moths as well as carpet, furniture, varied carpet, and odd *(Thylodrias contractus)* beetles. Now, objects showing signs of infestation, and all new acquisitions that are in any way suspect of carrying insects after a one-month quarantine, are treated. The museum has never found any species to survive ten days of anoxia.

### The Large Rentokil Bubble

Rentokil's standard large plastic chamber, known at one time as the "B & G Mini Bubble," was designed and widely used for fumigation with toxic gases such as methyl bromide. Since the late 1980s, this container has also found favor with museums for carbon dioxide fumigation. However, it has generally not been considered practical for nitrogen anoxia because of oxygen leakage problems that make reaching levels below 1000 ppm daunting, and the large number of nitrogen cylinders required per treatment.

Steven Pine, conservator at the Museum of Fine Arts in Houston, Texas, needed a large-volume fumigation system that he preferred to use with nitrogen rather than carbon dioxide (Pine, Daniel, and Maekawa 1993). He based his preference on data indicating that minimum assured kill times were better defined and shorter with nitrogen than with carbon dioxide, and on the feeling that nitrogen was more inert and safer. Despite the Rentokil bubble's apparent unsuitability for nitrogen anoxia, Pine decided to try to adapt it, and in 1991 a 30 m³ bubble was

purchased by the museum. This standard unit has a base 3.5 m square and a height adjustable to a maximum of 2.4 m. Entry to the bubble is gained through a horizontal zip-lock seal above floor level. The unit is equipped with three ports fitted with quick-release hose connections that have internal check valves that close under positive pressure from the interior. They are used for controlling the atmosphere within the bubble. Additional ports for connections to internal humidity, temperature, and oxygen monitors as well as for electrical extension cords were patched through the chamber wall using two-piece poly(vinyl chloride) couplings, rubber gaskets, and refrigeration putty for sealing. The Houston bubble was installed on a smooth, level surface, and cardboard was placed over the interior floor to protect the bottom plastic from puncture. The conversion from air to a low-oxygen atmosphere is facilitated by partially evacuating the bubble with a small vacuum pump before passing in high-purity nitrogen. A frame of poly(vinyl chloride) pipe was constructed inside the chamber to protect the contents from walls that might collapse during this partial evacuation. Typically, after the chamber is loaded with materials to be treated, cardboard is placed between the objects and the walls for protection. A clear film window provides a good view of the oxygen analyzer, hygrothermographs, and the objects behind them in the bubble.

Humidification is provided by an interior steam evaporator placed so that a 16 in. (40.64 cm) nonoscillating fan sends the vapor stream against the incoming nitrogen port and along the walls rather than directly at the objects. In trial runs, when the steam evaporator was not used, the RH fell to a level below the hygrothermograph's ability to measure—that is, below 10%. During treatment, an RH of 57% is maintained with the steam evaporator set at maximum, the fan on high, and the nitrogen at a delivery pressure of 414 kPa.

Before the bubble was put into routine operation, two steps were taken to ensure that the enclosure would hold oxygen below 1000 ppm for the time required for treatment. The first was to find and eliminate any leakage at the seams or the plastic zip-lock. To do this, the unit was fully inflated with nitrogen to slightly above atmospheric pressure and then injected with the contents of a can of spray used to dust off photographs. The gas contains Freon, and any leakage from the bubble would trigger beeping from a halogen leak detector. No leakage was found when the detector was passed over structural seams, but the test revealed that a portion of the zip-lock seal was not tightly closed and had to be reset. Testing with Freon was done only once to check the structural integrity of the bubble. It is not used as a seal check prior to each fumigation because the presence of halocarbons interferes with the accurate operation of the trace-oxygen analyzer. To test for leaks before most treatments, a slight vacuum is drawn on the enclosure at the beginning of each run and maintained for one hour. A system with large leaks would not be able to hold the vacuum. The second step in preparation for routine operation of the bubble was determining the rate of oxygen permeation into the chamber through its walls. This was done in conventional fashion, except that the chamber was partially evacuated prior to adding nitrogen containing 10 ppm oxygen. The evacuation and nitrogen recharging cycle were repeated until the oxygen concentration was reduced below 1000 ppm. The inflated bubble was allowed to stand for 24 hours while the oxygen concentration was followed on a Teledyne trace-oxygen analyzer. This established the rate of oxygen permeation, which can be expressed in ppm per day or per hour. Three runs revealed a rate of 800 ppm oxygen per day or 33 ppm per hour (Fig. 7.4). This means that with a chamber half full of objects (which would cause the oxygen buildup rate to be 66 ppm per hour) and having an atmosphere with 200 ppm oxygen, it would take about 12 hours to get to a maximum oxygen level of 1000 ppm. Depending on the volume of the load being fumigated and the gastightness of the zip-lock seal, the time between purging cycles could be as short as 8 hours or as long as 24 hours, but generally

Figure 7.4

Leak-rate measurement of oxygen concentration vs. time in Houston's Museum of Fine Arts bubble.

Oxygen ppm in nitrogen / Time, in hours
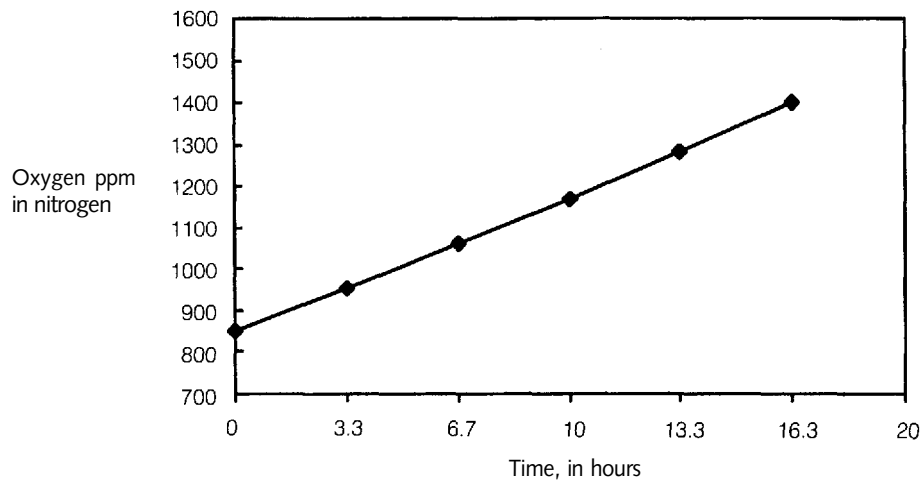
it was found to have a range of 10-14 hours. Had the bubble been operated at oxygen levels as high as 3000 ppm, purge cycles might have been spaced as much as two days apart.

In a typical run, objects are loaded into the chamber with the steam humidifier and the fan, which are positioned to avoid water vapor directly over the material being treated. The humidifier is filled with distilled water, and the oxygen analyzer is calibrated at 20.9% using air. The humidifier and fan are turned on only during the nitrogen entry period of the purge cycle. After the bubble is closed, a slight vacuum is drawn to remove some air (it is critical that the vacuum not be allowed to become excessive), and nitrogen is injected at no more than 60 psi until the bubble becomes full. The fan and humidifier are turned on again during this period. This completes one cycle. The nitrogen, fan, and humidifier are then turned off, and evacuation starts the second cycle. Cycles are repeated until an oxygen level of 200 ppm is reached. The system is rechecked for gastightness. The oxygen level will slowly rise, and when it reaches 1000 ppm, the purging routine is repeated.

Steven Pine is a highly creative craftsman. His perfection of this anoxia system and its frequent use at the Museum of Fine Arts in Houston demonstrate that the large Rentokil bubble can be used with nitrogen for the disinfestation of museum objects. The process is labor intensive, requiring purging cycles every 10-14 hours to keep the oxygen level below 1000 ppm. However, the bubble has been used to treat as many as twenty-four upholstered chairs or over one hundred rolls of textiles at one time, and the effort is considered well justified. The availability of low-cost, high-purity gas in the Houston area keeps expenses for nitrogen at a reasonable level.

## Rigid Chambers

### Homemade Containers

While a number of institutions carry out treatments in rigid and substantial plastic or metal containers, there are no units commercially available in the sense of being listed specifically for nitrogen anoxia. However, there are many commercial chambers or containers for other uses that could readily be adapted for anoxia. Museum personnel, with considerable skill and ingenuity, have built anoxia chambers out of pipes, boxes, water tanks, and units designed for use with toxic pesticides. Chambers formed from a single molded piece of plastic and an easily attached cover have generally worked well, while boxes assembled from flat panels and joined by gasketed rims have not. Koestler (1992) even built a treatment system out of a heavy-gauge polyethylene utility storage cart. A

Figure 7.5

Diagram of custom-made anoxia chamber used by Alan Johnston. (Courtesy of Alan Johnston.)

Side elevation          End elevation

Plan of chamber rim
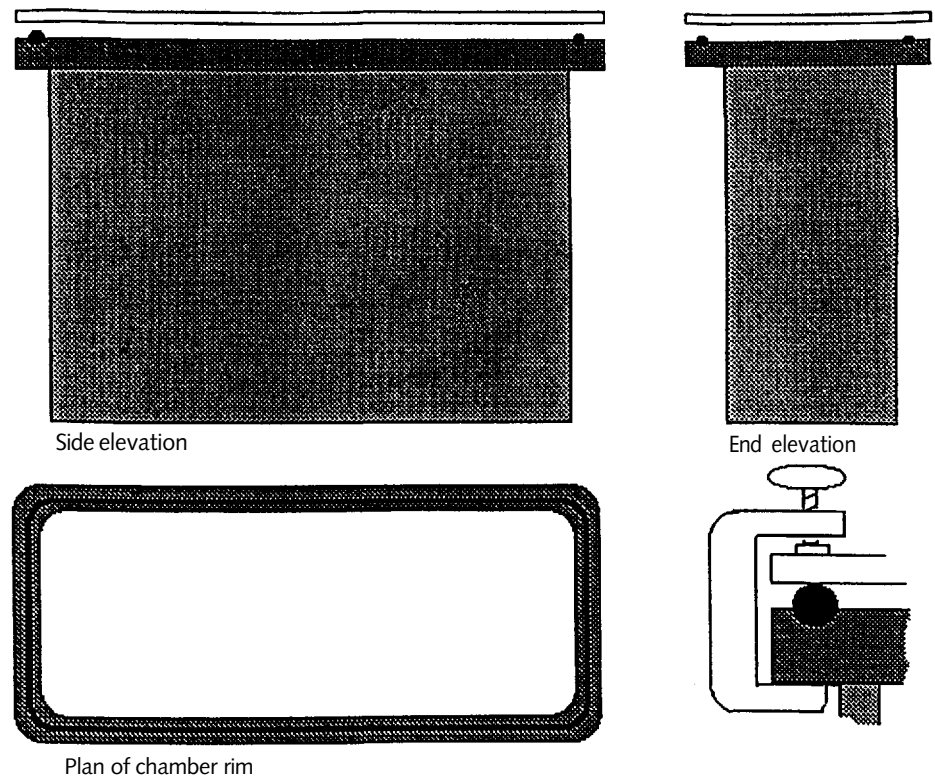
Plexiglas cover was sealed to a polyethylene chamber with silicone adhesive to create a container with an oxygen leak rate of only 50 ppm per day, which could be brought to an initial oxygen concentration of 50 ppm. Unfortunately, a new seal had to be prepared for each application.

Alan Johnston, of the Hampshire County Council Museum Service in the United Kingdom, put together a similar container (Fig. 7.5) using inexpensive off-the-shelf materials. It consisted primarily of a commercial polypropylene water tank, measuring $1 \times 0.5 \times 1$ m, and an oval lid cut from a 10 mm thick sheet of cast polycarbonate. Johnston (1996) was able to solve the sealing problem with ordinary woodworking C-clamps, a large neoprene O-ring designed for aircraft doors, and a smooth rim on the water tank. Nitrogen, humidified by passage through a three-bottle bubbler system, is fed into the chamber through a one-way truck tire valve set into the polycarbonate lid. This is connected to perforated rubber tubing that coils around the base of the tank and distributes the nitrogen. The gas leaves the chamber through a simple valve system either directly to the atmosphere or through a Teledyne oxygen monitor. The valve arrangement allows either evacuation or purging. Although the system can be brought to an oxygen concentration of 200 ppm when the unit is closed tightly, oxygen levels will climb to 1% over 168 hours. The system is most conveniently operated with the exit valves slightly open to allow a small flow of nitrogen to move through the tank. RH and temperature readings are made using a Meaco radio telemetric sensor within the chamber that transmits data to a central environmental monitoring computer. RH is also followed in the third mixing jar of the bubbler system with a simple digital Meaco meter.

Steven Pine, of the Museum of Fine Arts in Houston, has also designed and constructed several different types of rigid chambers for special applications. He converted a 20 ft (6.1 m) length of standard 15 in. (38 cm) diameter PVC pipe into a treatment chamber for long and awkward objects like spears and rolls of fabric (Fig. 7.6). Building this unit required a section of SDR 35 PVC gasketed sewer pipe, two 15 in. (38.1 cm) PVC caps, two 0.5 in. (1.27 cm) diameter

Figure 7.6
Anoxia chamber made of PVC sewer pipe.
(Photo courtesy of Steven Pine, The Museum
of Fine Arts, Houston.)
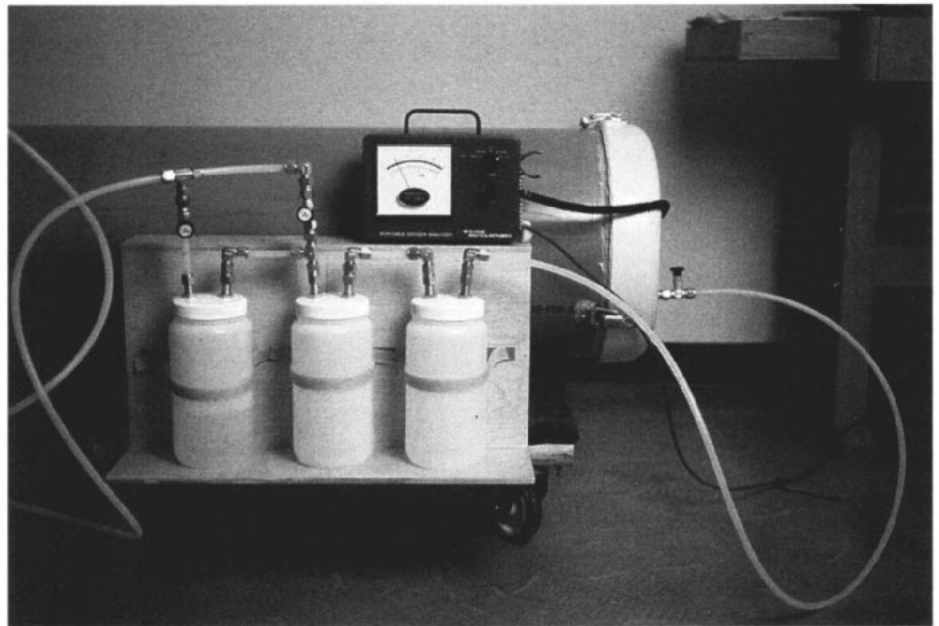


silicone O-rings, and eight latches. First, the flange end of the pipe was cut off,
then each PVC cap was cut perpendicular to the axis to provide both a collar and
a half cap. Collars were mounted at each end of the pipe with PVC cement, and
the half caps were attached with latches and made gastight to the collars with
the O-ring gaskets. Humidified nitrogen is brought in at one end of the pipe
through a cap and out the other end through the second cap, either directly to
the outside or through an oxygen monitor (Fig. 7.7).

Figure 7.7
Diagram of improved PVC prototype. (Courtesy of Steven Pine, The Museum of Fine Arts, Houston.)



Usual humidification system

A cap cut in half yields a half cap and a collar,
which is welded onto the pipe with PVC cement.

Digital hygrometer placed in third jar allows for
RH reading of the $N_2$ entering the tube.

$N_2$

Water

Large end of pipe is cut off.

Humidified $N_2$

Half cap

(1)

Four latches on each cap

(2)

(4)

(3)

$O_2$ monitor

T-valve $N_2$ outlet

Input

O-ring gasket made of silicone
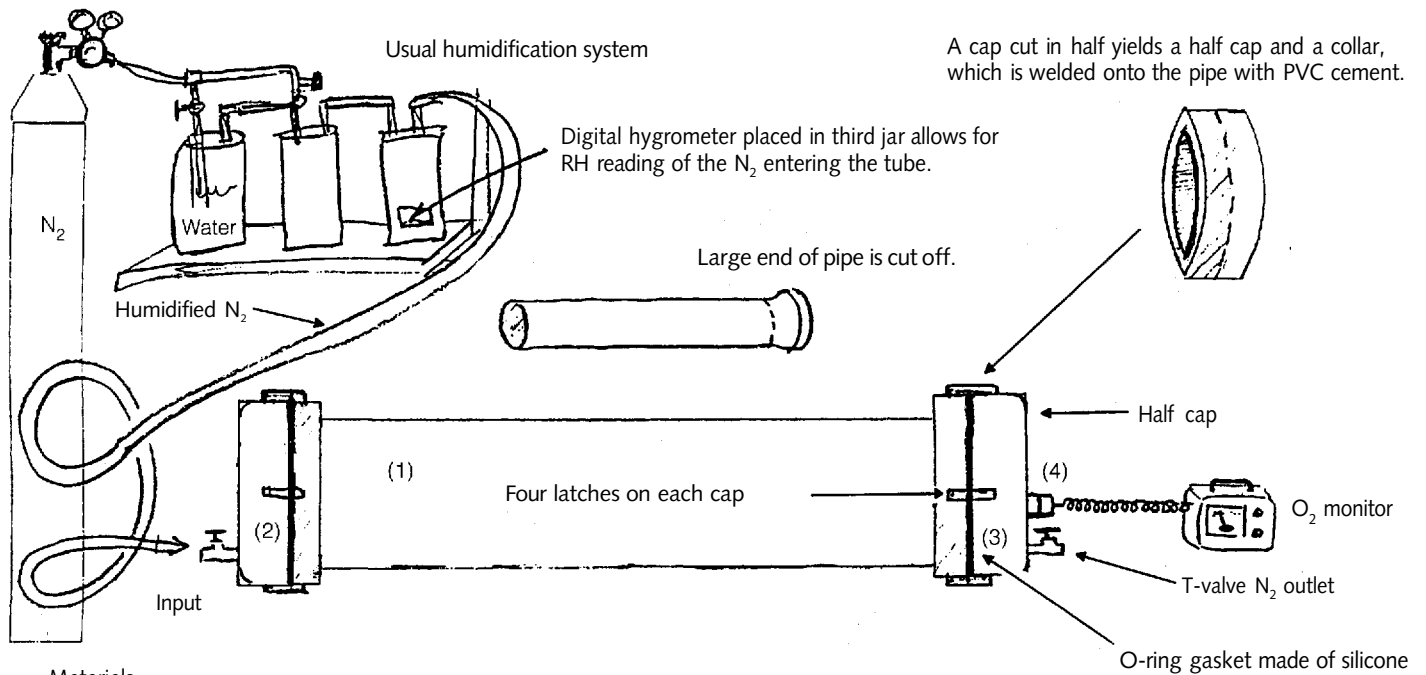
Materials
(1) 15 in. × 20 ft (38 cm × 6.1 m) SDR35 PVC gasketed sewer pipe
(2) 15 in. (38 cm) PVC gasketed sewer caps (two)
(3) 0.5 in. (12 mm) silicone gasket O-rings (two)
(4) Teledyne oxygen monitor

**Figure 7.8**
Hardigg case with taps. (Photo courtesy of Steven Pine, The Museum of Fine Arts, Houston.)

Any tightly sealable container with substantial walls that allow the installation of gas and utility lines should be convertible to an effective anoxia chamber. Hardigg Industries makes such reusable containers in a range of sizes, and Pine has converted a 9 ft$^3$ (0.3 m$^3$) case, shown in Figure 7.8 with gas ports and sensor connections, into a compact system with nitrogen supply and humidification (Fig. 7.9). This is easy to build and effective for the disinfestation of small objects in a standard dynamic process of allowing nitrogen to flow through the container for 5-20 days as needed, after the oxygen content has been lowered below 1000 ppm.

### Vacufume Chambers

Conversion of commercial metal fumigation chambers originally designed for use with gaseous pesticides can provide a unit in which anoxia with high-purity nitrogen is effectively carried out. An example of this is the Vacufume Fumigator-36, which was manufactured by the Vacudyne Company. Several thousand have been sold, mostly to hospitals for sterilization treatments, but a few were acquired by cultural institutions like the Los Angeles County Museum of Art (LACMA) and the Royal Ontario Museum in Toronto. Conversion of this chamber for anoxia treatments consists primarily of installing a humidification system and providing gastight entry to the box for humidity and oxygen monitoring cables. Vacudyne offers conversion kits, and a number of museums have made the change. LACMA purchased a Vacufume chamber in 1983 for use with ethylene oxide at about the time that this fumigant fell out of favor. Consequently, the unit was never used with ethylene oxide. Researchers at the Getty Conservation Institute proposed that the Vacufume chamber be converted to operate by anoxia. Shin Maekawa and Vinod Daniel of the GCI, along with Steve Colton of LACMA, designed and made the necessary modifications. The Vacufume Fumigator-36 is a large metal box with an inner capacity of about 1.02 m$^3$. It measures 0.91 m square at the base and is 1.22 m high. The large gasketed door is sealed with four wheel latches, one at each corner, and when tightly closed, the chamber has an oxygen leak rate of about 200 ppm per day. The ability to keep oxygen inflow to this level is a major reason that the Vacufume unit is an attractive candidate for conversion.

**Figure 7.9**
Anoxia system based on Hardigg case. (Photo courtesy of Steven Pine, The Museum of Fine Arts, Houston.)
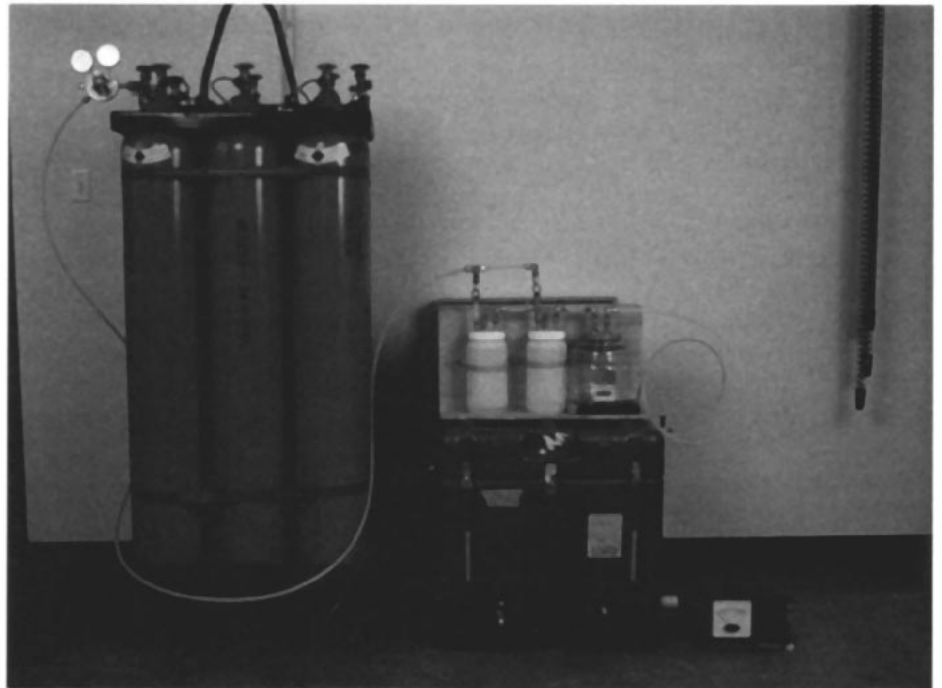
Infestations in the LACMA collection are not common, but the chamber finds frequent use treating new acquisitions and incoming pieces on loan with no fumigation history. In a typical treatment carried out in April 1996, the chamber was loaded with a large, well-wrapped, papier-mache construction held in a cardboard box and a small painting in a wooden frame that showed signs of infestation. After the door was closed, the latches were secured tightly, and a vacuum of 5 psig was pulled. The purpose of the vacuum is to remove some oxygen, to determine if the seal is holding, and to ensure that the chamber is made as gastight as possible. The reading on the oxygen monitor fell to 17.5%. This showed that there was a good seal and that it was possible to turn and tighten the latches just a bit more. The humidification system, consisting of three stout, half-gallon, polypropylene bottles, was attached to the left outside wall of the chamber as shown in Figure 4.1. Humidification is provided and measured as described in chapter 4. Nitrogen first sweeps out the air in the bubbler system via the purge line just before the entry valve to the chamber. Then the nitrogen is sent into the chamber, bringing it up to atmospheric pressure. A vent from the chamber is then opened to allow the unit to be flushed with nitrogen to achieve the required level of oxygen. In a typical run, the RH in the humidification system would quickly rise to 65%. This is higher than the RH in the chamber because the pressure there is higher. After the gas streams in the bubbler system were adjusted to send less nitrogen through the water bottle, the RH in the third bottle fell to 57%, and the RH in the chamber leveled at about 50%. The chamber is typically operated with the RH at 45-55%. Flushing the system over three eight-hour shifts with 99.995% nitrogen brought the oxygen concentration to 600-800 ppm. The chamber allows 200 ppm oxygen to leak in over twenty-four hours, so sealing it at this level means that the oxygen level will drift up to under 3000 ppm, or 0.3 %, over ten days. This is within the allowable increase in oxygen concentration for an effective anoxia treatment.

The Vacudyne Company has been testing a prototype of another kind of rigid-chamber anoxia system, offering the possibility that a turnkey unit will be made available commercially. The system uses a conventional 50 gallon (190 liter) steel drum for the anoxia chamber and comes with a three-jar humidification system as well as the necessary gas and utility lines. The unit has been evaluated by Mark Roosa, chief preservation officer for the Huntington Library Art Collections and Botanical Gardens in San Marino, California. The system will purge down to an oxygen concentration of 2000 ppm in 6-8 hours using nitrogen containing 20 ppm oxygen. Treatment is generally run at 2000 ppm oxygen, 20 °C, and 45-50% RH for up to 7 days, depending on the nature of the infestation. At the Huntington Library, treatment is done only where infestation is seen or highly suspected. Pests have included silverfish, lice, beetles, and termites, which have attacked books, masks, papier-mache objects, photographs, and other archival material. This is a usable and relatively inexpensive system that can be transported to sites as needed. The main limitation may be one of capacity.

# Chapter 8

# Fumigation with Carbon Dioxide

Although cultural institutions use anoxia more widely than they do carbon dioxide fumigation to control infestations, the quantity of material treated by carbon dioxide fumigation is larger. When big reusable chambers are employed to treat large batches of material, carbon dioxide is the gas of choice because gas compositions effective for killing insects are more readily and cheaply achieved with it than with nitrogen. It is faster and easier to replace 60-80% of the air in a 30 m³ bubble with carbon dioxide than it is to purge the chamber to an oxygen content of 500 ppm with high-purity nitrogen and hold it at that level. Carbon dioxide is preferred over nitrogen because it requires less stringent sealing conditions and because fluctuations in concentration have much less influence on performance. Carbon dioxide is not used with pouches and small containers, where argon and nitrogen work well. Many institutions use both carbon dioxide fumigation in large reusable bubbles and nitrogen treatments in bags to deal with infestations. Other reasons for favoring carbon dioxide for large-volume operations are its much lower cost than nitrogen on an equal volume basis and the lesser need for auxiliary humidification. A nitrogen purge sweeps out all ambient moisture from a treatment container, but at a 60% carbon dioxide level, 40% of the original water vapor remains. In addition, large chambers are often filled with objects in wooden and cardboard boxes packed with paper, which provides a considerable amount of moisture buffering. There is some concern about the formation of carbonic acid when carbon dioxide encounters water during treatment as pointed out by Reichmuth (1987). The carbonic acid provides a pH of 4, which might damage sensitive materials such as metal filaments, zinc objects, and organic pigments. These could be harmed by the acidity, and conservators should be aware of this factor when planning treatment. However, no problems of this type were noted in the many reports on carbon dioxide fumigation reviewed for this chapter.

## Effectiveness of Carbon Dioxide versus Nitrogen

To the extent that a direct experimental comparison can be made, nitrogen containing very low concentrations of oxygen appears to be the more lethal medium. In a study comparing nitrogen, argon, and carbon dioxide, Valentín (1993) observed that there was very little mortality among *Hylotrupes bajulus,* especially old larvae and pupae, held in an atmosphere of 60% carbon dioxide at 30 °C for 3 weeks. Under the same conditions with nitrogen containing 300 ppm oxygen, total mortality of all life stages of the borer is achieved in 10 days. Valentín found that for most beetle pests found in museums, exposure to 60% carbon dioxide for 10-25 days at 30-35 °C was needed, while nitrogen containing 300 ppm oxygen required only 4-6 days. She concluded that when comparing nitrogen or argon with carbon dioxide under typical fumigation conditions, the carbon dioxide atmosphere was ineffective for controlling populations of Cerambycidae (boring beetles), and elimination of species in the Anobiidae and Lyctidae families took more time than with nitrogen. Banks and Annis (1990) attempted to make a comparison based on the limited available literature and concluded that none of the gases has a marked advantage. However, the two studies they cited (Jay 1984; Reichmuth 1987) both describe the use of nitrogen containing 10,000 ppm (1%) oxygen, which is essentially ineffective for anoxia. Therefore, conclusions that nitrogen is no more effective than carbon dioxide were erroneously based on studies made with high oxygen concentrations.

## Carbon Dioxide Fumigation Requirements

In the current practice of conducting carbon dioxide fumigations in large bubbles, operators make up for lower effectiveness of this gas by running treatments under more severe conditions, that is, over longer times and at higher temperatures, but normally staying at 60% carbon dioxide. Treatments generally last 2-5 weeks at temperatures above 25 °C. The use of carbon dioxide at 60%

concentration derives from earlier studies involving this gas in treating stored grains. Davis and Jay (1983) describe a number of successful field studies in silos, going back to 1973, where the concentration of the carbon dioxide was generally 60%. Banks and Annis discuss recommended dose rates in some detail. Again, 60% is favored, but if more resistant species are present, such as cigarette or khapra beetles or rice weevils, up to 80% carbon dioxide may be required to control all life stages within a reasonable exposure period. Smith and Newton (1991), after surveying the literature up to 1991, state that 60% is the concentration generally considered to be most practical for control of stored product insects. Higher concentrations do not confer any great advantage and are more difficult to maintain. They confirmed this in their laboratory with studies at 60%, 80%, and 100% concentrations. Recently published case studies find that fumigations typically are carried out at 60% carbon dioxide levels. Cipera and Segal (1996) normally use this dosage but increase the concentration to 80% when dealing with wood-boring insects. Warren (1996), who conducts fumigations in a custom-built 50 m$^3$ bubble, starts treatment with the carbon dioxide concentration at 80%. This is allowed to drop to 60% over a 10-day period and then is maintained at this level. The state-of-the-art unit now marketed by Rentokil automatically releases carbon dioxide until a 60% concentration is reached.

Duration of exposure is highly dependent on temperature. In 1984 Jay recommended the following time-temperature profile for fumigation with 60% carbon dioxide: 21-28 days at 16 °C; 10-14 days at 21 °C; and 4-7 days at 27 °C. In 1987 Annis claimed that the time required for the control of most grain-infesting species at 60% carbon dioxide and 20-29 °C is about 11 days. Annis chose this wide temperature range so that he could review data from a collection of trials carried out under diverse conditions. However, Jeremy Jacobs (1996), working with natural history collections at the Smithsonian Institution, found that somewhat more control of temperature was required for effective treatment of the species that were plaguing his collection, particularly odd beetles *(Thylodrias contractus)*. Jacobs was using carbon dioxide for fumigation in a large Rentokil bubble. His initial program was designed to operate with 60% carbon dioxide for 15 days at 30 °C. Fifteen runs were made over a two-year period. The carbon dioxide level fluctuated above and below 60%, but never lower than 55%, while the temperature varied in a range lower than the desired 30 °C, generally between 19 and 27 °C. When either parameter fell too low, Jacobs attempted to compensate by running the treatment for 15 more days or longer. Success was determined by returning items to their storage cases, which had been given a pyrethrum-silica gel treatment while the objects were in the bubble, and monitoring very closely for any sign of reappearance of insects over the ensuing twenty-four months. A reemergence of insects occurred in 40% of the runs. Jacobs examined his operating parameters against these results and noted that getting 100% mortality was not related to carbon dioxide level nor to length of treatment. However, a complete kill was obtained in every run where the temperature was 25 °C or higher. Getting to a suitable temperature can be a problem for installations housed in poorly heated quarters during cold seasons. Both Jacobs and John Burke, who operates a similar system at the Oakland Museum of California, do not carry out treatments in the winter.

Smith and Newton (1991) described studies designed to provide minimum assured kill times for carbon dioxide fumigation with the same goal that Rust and Kennedy had for nitrogen anoxia. Table 8.1 provides the minimum number of days of exposure to a 60% carbon dioxide atmosphere required to obtain 100% mortality of cultures containing all life stages of fourteen species of insects or mites. Experiments were carried out for 2 and 4 weeks at 15 °C, while at 23 °C and 35 °C they were generally run for 1, 2,4, 8, and 14 days in order to

**Table 8.1**

Minimum time for assured 100% kill of all stages of property-destroying insects and mites by use of 60% carbon dioxide at 75% RH.

| Species | Temperature (°C) | Time (days) |
|---|---|---|
| Mold mite | 23 | 24 |
| | 35 | 14 |
| Grain mite | 15 | 14 |
| | 23 | 14 |
| | 35 | 1 |
| American cockroach | 15 | 14 |
| | 23 | 2 |
| | 35 | 4 |
| Khapra beetle | 35 | 14 |
| Rice weevil | 15 | 28 |
| | 25 | 14 |
| | 35 | 4 |
| Varied carpet beetle | 15 | 28 |
| | 25 | 14 |
| | 35 | 2 |
| Merchant grain beetle | 15 | 14 |
| | 23 | 4 |
| | 35 | 1 |
| Red flour beetle | 15 | 14 |
| | 23 | 4 |
| | 35 | 4 |
| Cigarette beetle | 15 | 14 |
| | 23 | 4 |
| | 35 | 1 |
| Hide beetle | 15 | 14 |
| | 23 | 4 |
| Australian spider beetle | 15 | 14 |
| | 23 | 1 |
| | 35 | 4 |
| Tropical warehouse moth | 15 | 14 |
| | 23 | 4 |
| | 35 | 2 |
| Book louse | 23 | 8 |
| Furniture beetle larvae | 15 | 14 |
| | 23 | 1 |
| | 35 | 14 |

find the minimum time needed for complete kill. Cultures containing insect eggs, pupae, adults, and young and mature larvae were obtained by placing samples in 100 g of standard rearing medium in half-liter jars and incubating under standard conditions. Mite cultures were inoculated during the week before treatment with 5 ml of live culture medium. The jars were stacked in a high-density polyethylene container with a 35 l capacity. After closing, it was purged with commercial carbon dioxide until the concentration reached 60%. Carbon dioxide was added as needed to maintain this level. The RH was kept at 75% to promote the survival of mites and book lice. One culture of each species was exposed to each combination of temperature and treatment duration. Each culture was

examined two days after treatment and again after a holding period long enough to permit the development of visible life stages. Complete mortality was declared only if there were no signs of life at both inspections. Mite and book louse cultures were examined with a low-power microscope.

The teams of Smith and Newton and Rust and Kennedy determined minimum times for assured complete kill with two different fumigants. Both studies showed the same relative susceptibility of species to dehydration, but the anomalies in the work by Smith and Newton indicate that it was not done with the same rigor as the research by Rust and Kennedy.

Rentokil, Inc., is an international corporation in the United Kingdom that deals with many aspects of pest management. Through its Project Development Unit in West Sussex, under the direction of general manager Colin Smith, the company has in recent years focused on designing and promoting large reusable plastic chambers, called bubbles, for insect disinfestation. These were initially used with conventional fumigants like methyl bromide but more recently with controlled atmospheres, particularly carbon dioxide. John Newton (1990) has been heavily involved in Rentokil's research programs. The Rentokil bubbles are manufactured by Power-Plastics Limited in Thirsk, North Yorkshire, England, a company best known for its production of tarpaulins and tents. Fully equipped and automated units are sold and serviced in North America by Maheu & Maheu, Inc., of Quebec, Canada, and elsewhere by Power-Plastics Limited.

Carbon dioxide fumigation has a long history of use for killing insects infesting stored grains. One of the earliest demonstrations of its use for treating museum items was work with costume collections in the Museum of St. John in London (Child 1988). Textile pests were successfully eradicated with 60% carbon dioxide in a standard Rentokil bubble. Since then, carbon dioxide fumigation has become popular among museums in the northeastern United States and in Canada. There are several reasons for this. Institutions in this geographical area have a need for large-volume fumigations; Canada has favorable regulations on the use of carbon dioxide for treating insect infestations; and Maheu & Maheu, supplier of the Rentokil bubble, backs up its sales efforts with strong technical support. The Canadian government does not classify carbon dioxide as a fumigant but as an inert gas. Therefore, no special license is required to use it. In contrast, the British government now lists carbon dioxide as a pesticide. As a consequence, this gas is not likely to find much use by conservators in the United Kingdom. In the United States, there appears to be no federal policy on the handling of carbon dioxide, and each institution is required to check with state regulatory authorities.

## Treatment Experience in the United States

In the late 1980s, when the most important toxic fumigants—such as ethylene oxide—were being banned in many areas, several institutions in the northeastern United States began looking at carbon dioxide as a safe fumigation substitute that could disinfest large quantities of material. The Winterthur Museum in Delaware, the Society for the Preservation of New England Antiquities (SPNEA), and Old Sturbridge Village in Massachusetts each have very large holdings of decorative arts, historic furniture, and objects of paper, wood, and textile that are prone to attack and infestation, generally by powderpost and carpet beetles, silverfish, and clothes moths. At that time, SPNEA had forty-four houses filled with rooms of furniture under its control, while Old Sturbridge Village owned forty-two historic buildings holding thirty-eight thousand books and eighty thousand objects.

## Old Sturbridge Village

Old Sturbridge Village was the first of these institutions to act. The nature and size of its holdings create a substantial need for dealing with insect infestations. Webbing clothes moths and carpet beetles are the most common problems. In contrast to almost everyone else who is using carbon dioxide fumigation in a plastic bubble, Old Sturbridge Village carries out its treatments in a rigid metal chamber originally purchased in the early 1980s for operation with ethylene oxide. In 1989, with a few modest changes in the external plumbing, it was converted to use with carbon dioxide. The chamber, with a 2 $m^3$ capacity, can handle relatively small loads, but the conservators at Old Sturbridge Village have been able to turn to their colleagues at SPNEA for help with larger objects, such as sofas. The rigid chamber makes it possible to draw a slight vacuum, which speeds the purging process when a new run is started. The chamber is filled with carbon dioxide by two displacements. Instead of humidifying the gas stream, objects are conditioned to 45% RH before being put into the chamber. An infrared safety monitor outside the unit trips an alarm if the outside carbon dioxide concentration reaches 1000 ppm.

An efficient heating-evaporation device, placed between the gas cylinder and the chamber, is an important part of any carbon dioxide fumigation system. The carbon dioxide coming from the cylinder must evaporate and expand rapidly; in so doing, its temperature drops sharply. To avoid ice formation and other problems and to bring the carbon dioxide stream into the chamber at close to the desired run temperature, heat must be supplied at the point where the evaporation occurs. To do this, the Sturbridge system uses an in-line electric heater, E99-E-Heater 320, which is supplied by the Air Products and Chemicals Company. It is thermostatically controlled to operate between 24 and 29 °C (75 and 85 °F).

## The Winterthur Museum

In 1988 Rentokil representatives brought a small plastic chamber to the Winterthur Museum and successfully demonstrated the ability of carbon dioxide fumigation to rid trial objects of living insects. As a result, a standard 30 $m^3$ Rentokil bubble was purchased and installed in 1990 in an isolated area of the museum that had previously been dedicated to the use of toxic fumigants. Winterthur adopted a procedure that requires 14 days of treatment with 60-70% carbon dioxide at ambient temperature,, which is normally 20-25 °C. The carbon dioxide is not humidified. Although this has been a concern, resulting in close inspections of treated objects, no damage due to not adding water vapor has been found. Rugs and upholstered furniture have been the most routinely treated items. Fumigations have been done primarily as a precaution rather than as a response to observed infestations. Objects, as they are received or sent out, are routinely examined for the possibility of infestation and the need for processing. After several years of operation, the bubble has been needed less frequently and treatment schedules have been erratic. The unit was not used at all in 1995.

## Society for the Preservation of New England Antiquities

In 1991 SPNEA had a burgeoning infestation problem. One of the historic houses under its management had become infested with webbing clothes moths. In addition, the house accidentally received an internal covering of soot from a malfunctioning furnace. Cleaning away the soot, exposure to a cold New England winter, and some contact spraying with insecticide eliminated about 95% of the infestation. However, rolls of textiles in the society's storage facility were also infested, and a standard Rentokil bubble was purchased and

immediately put into service in 1992. The stored textiles and other remaining problems were completely rid of their insect pests in the chamber, using carbon dioxide at 20 °C. Typically, the carbon dioxide level is maintained between 65% and 75%. This is monitored by measuring the oxygen level, which is kept between 4.9% and 8.4%, a spread that corresponds to a carbon dioxide range of 75-60%. Humidity is provided by a bubbler system that takes the unit to 40% RH in the summer but only 30% in the winter.

Only two weeks of treatment are required to eradicate powderpost beetles and clothes moths in textiles, even when these are wrapped in plastic film. However, on three occasions adult carpet beetles survived two weeks of fumigation, and the standard run time is now three weeks when this species is found. Very heavily infested objects may be subjected to two treatments. Longer run times and repeated treatments might have been avoided by operating the system at 25-30 °C. About four to six T-cylinders of gas are used per run. The carbon dioxide goes through a system of two gauges and a heat exchanger, which was obtained from the gas supplier. The carbon dioxide concentration in the room outside the bubble is monitored by an infrared analyzer, which is set to trigger an alarm and turn on a powerful ventilation system when safe levels are exceeded. The ventilation system is also used to exhaust carbon dioxide from the bubble and draw in air at the end of a run. Over a four-year period leading into 1996, thirty-five treatments, averaging about one hundred objects each, had been put through the three-week process without any apparent failure. SPNEA also does work for other institutions. Perhaps the most unusual assignment was the treatment of bales of hay used to make a sculpture for display in the Portland Museum, in Maine.

## The Oakland Museum

A grant from the Institute of Museum Services enabled John Burke to purchase a standard Rentokil bubble in 1991 and install it in the conservation facility of the Oakland Museum, in California (Fig. 8.1). The unit obtained by Burke has a ceiling adjustable to a height of 2.44 m (8 ft). One side contains a plastic window, and a gas-transfer manifold is in place along the bottom of an adjacent side. Burke had intended to use nitrogen anoxia as his method of treatment. After

**Figure 8.1**
Room-size carbon dioxide bubble. (Photo courtesy of John Burke, Oakland Museum.)

Figure 8.2
Collections in open trays. (Photo courtesy of John Burke, Oakland Museum.)
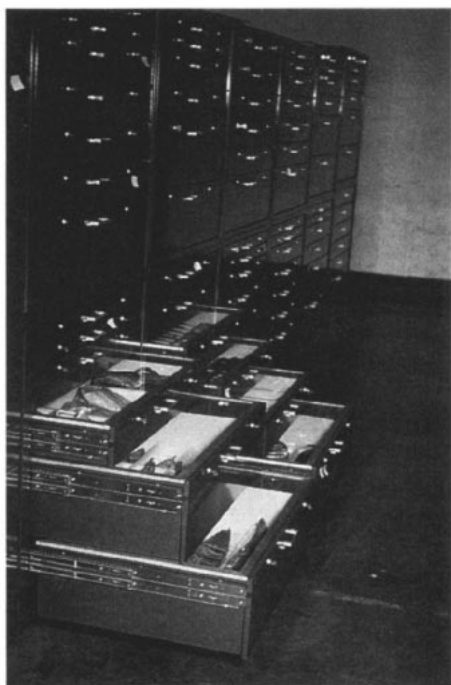


Figure 8.3
Collections in steel cabinets. (Photo courtesy of John Burke, Oakland Museum.)

several months of manipulating the bubble, however, he concluded that a nitrogen atmosphere with less than 1% oxygen could not be maintained in the system because of high leakage rates. Accordingly, the system was altered for use with carbon dioxide. Treatment is carried out by displacing enough air with pure carbon dioxide to establish and hold the concentration of this gas at 65-70% for relatively long periods of time. The system was first used to disinfest the entire ethnographic collection of the museum over six months using 4-week exposure sessions. Subsequently, large batches were treated for 5 weeks for both in-house projects and for other clients. Because of his work, Burke introduced the practice of both nitrogen anoxia and carbon dioxide fumigation to a large number of conservators in the San Francisco Bay Area very early in the development of this technology. Large batches of material are now cleared of invading insects in his chamber five or six times a year. These include ethnographic collections, bibliographic materials, textiles, sculpture, and furniture and other large pieces that are placed in the chamber in a loose arrangement. Smaller objects are packed in boxes, and collections are stored in drawers and cabinets (Figs. 8.2 and 8.3). When objects are treated, the materials are placed in the chamber, and the ceiling is lowered to eliminate as much free volume as possible. First, a slight vacuum is pulled, and then carbon dioxide is flushed into the chamber through a heat exchanger. The cycle is repeated several times until a carbon dioxide level near 70% has been achieved. The concentration is determined with an oxygen sensor connected to a digital readout, which is calibrated to show increasing carbon dioxide levels as the oxygen level falls. Because there is leakage in the system, the carbon dioxide level is logged on a daily basis and brought up to 70% as needed. Sensors placed inside closed cardboard boxes have shown that carbon dioxide concentrations inside these containers usually rise to free-space levels in less than two hours. No attempt is made to add water or otherwise control humidity.

On two occasions initial treatment did not provide 100% kill, and this was remedied with another run through the process. Burke has found that warm, dry conditions, normal at his location, are most effective for achieving a rapid kill, while cold, damp weather inhibits the process. Thus, he tends to avoid making runs during the winter months. The five-week treatment period might seem excessive, but the success achieved with this process over four years has created a body of data showing that 35 days of 65-70% carbon dioxide exposure is effective.

### The Smithsonian Institution

The Smithsonian Institution began treating parts of its holdings with carbon dioxide in 1993. Other than the fact that emphasis has been on natural history collections rather than on fine arts or decorative arts, the issues behind its acquisition of a fumigation bubble are not unlike those faced by other institutions. The Smithsonian has over 580,000 mammal specimens, the largest collection in the world, housed in several large and separate rooms. Odd beetles and redlegged ham beetles were known to have infested the collection, but these were believed to have been held in check by fumigation based on carbon tetrachloride done in the 1980s. No integrated pest management was employed, and it was not known how effective the fumigation program was. The use of toxic chlorocarbons in the museum was finally halted in November 1990, and an integrated pest management program was started. In 1992 the Smithsonian purchased a Rentokil bubble, measuring 2.7 m square and 2.4 m tall, that was custom-made to fit into available building space. Following a period of installation and trial, treatments began in September 1993. After fifteen runs, made over a two-year period by museum specialist Jeremy Jacobs, an optimum

procedure was developed requiring fumigation with 60% carbon dioxide at 30 °C for 2 weeks.

## Treatment Experience in Canada

### The National Museum of Science and Technology

The National Museum of Science and Technology in Ottawa has an outstanding collection of historic vehicles comprising railcars, automobiles, and horse-drawn carriages and sleighs, all very large museum objects. Some are on display, but most are kept in warehouse storage. The fabrics of these vehicles are prone to infestation by casemaking clothes moths and, occasionally, by carpet beetles. The presence of moths in the collection had been noted in the early 1980s, when a number of inadequate control steps were initiated. By 1989 it was clear to Sue Warren, conservator at the museum, that the infestation had spread through the warehouse. She turned to Thomas Strang of the Canadian Conservation Institute for help. He was an important early advocate of carbon dioxide fumigation and promoted its use particularly with museums in Canada. Strang provided support and guidance to conservators and responded effectively to concerns about the use of carbon dioxide, such as the formation of carbonic acid.

About the time that Warren contacted Strang, he was looking for a location to test his ideas and recommended carbon dioxide fumigation to the National Museum. A large, custom-built bubble would be needed to handle the museum's sizable vehicles, but before the museum made this commitment, it leased a standard 30 m³ Rentokil chamber from Maheu & Maheu to evaluate the system and its effectiveness. Two fumigations were carried out according to the procedures described below. Four small, moth-infested sleighs and control groups of ten live adult moths and ten larvae were placed in the bubble for each run. A complete kill was obtained for both control groups, and the sleighs appear totally free of infestation after five years. With proof in hand, the museum purchased a custom-built bubble measuring 6.6 × 2.44 × 2.74 m. This bubble will hold the museum's largest horse-drawn carriage, two medium-size vehicles, or four small sleighs.

In addition to the bubble, the system contains a vacuum pump, an ultrasonic humidifier, a small fan, a data logger for recording temperature and humidity, and a carbon dioxide monitor with remote sensor. Warren decided to include a humidifier when she noted that unless moisture were added, small cracks or splits in old wooden paneling would widen over a 2-week fumigation, although this was not observed to make much of a difference on relatively large splits. Movement around loose paint or joints where there were fills was also very damaging. The museum made several additions to the chamber. Wiring is brought into the bubble through an acrylonitrile-butadiene-styrene (ABS) plumbing cap, which is sealed with silicone caulking. Small metal pulleys replace the plastic loops used to raise the top of the bubble from the base. A sturdy tarpaulin covers the bubble's ground sheet to minimize wear on the bubble floor. A ramp over the zipper is used to facilitate rolling vehicles into the unit and to protect the seal. With constant use over five years, the bubble, including the zipper, has held up well. The seal is maintained in good working condition by keeping the zipper clean and treating it with a silicone spray every three or four cycles. Two tears, both caused by nails protruding from vehicles, were easily patched.

For a treatment, the chamber is first flushed to a level of 80% carbon dioxide in six to seven hours. This is done by initially removing a portion of the air with the vacuum pump and then refilling the bubble by flowing carbon dioxide into it through two entry ports at a rate of 2 m³ (60 ft³) per hour. The concentration decreases by 2% per day but is not allowed to fall below 60%. Temperatures

range from 15 to 25 °C in spring and 20 to 24 °C in summer. The temperature in the warehouse in winter is as low as 12 °C, and as is the practice of a number of institutions, fumigations with carbon dioxide are not done during the cold season. A typical cycle lasts 10-14 days. These conditions seem to be relatively mild, but clothing moths are quite susceptible to modified atmospheres.

After five years the list of treated objects includes ninety-two carriages and sleighs, five automobiles, one piano, and a large assortment of smaller objects. Following fumigation, the vehicles are thoroughly vacuumed and enclosed in a sealed polyethylene bag to prevent reinfestation. Insect sticky traps are placed with objects that had been more heavily infested. All stored items are examined in the spring and in the fall when moths are most likely to be active. After all of this, only three findings of dead moths suggest that reinfestation may have taken place. The conservators at the National Museum of Science and Technology have eliminated clothes moths from a large portion of their collection and feel that their carbon dioxide fumigation program is successful.

## The Canadian Museum of Civilization

The Canadian Museum of Civilization in Hull, Quebec, has an ongoing need to fumigate large quantities of material, including traveling exhibits and new acquisitions as well as parts of its permanent collection suspected of infestation. In 1991, soon after contracting for US$40,000 to eradicate a major infestation of clothing moths by freezing, the museum switched to carbon dioxide fumigation for pest control. It purchased a standard 30 m$^3$ Rentokil bubble from Maheu & Maheu and has found the procedure to be much cheaper than freezing. In the first two years, the bubble was used forty times at a total cost of US$12,000. This sum includes US$8,000 for the bubble, US$2,500 for an Armstrong (AM 6-1011/1012) two-channel carbon dioxide monitor, and US$35 per run for carbon dioxide. In addition to the economic advantage of carbon dioxide fumigation, the conservators responsible for the work, Luci Cipera and Martha Segal, feel that the in-house operation provides better control of the process. There is less handling and movement of material and, therefore, less chance of damage to the objects. Another reason for choosing this approach is the versatility in the types, quantity, and size of artifacts that could be treated. Large objects that have been easily accommodated in the bubble include 4.27 m (14 ft) wooden house posts in crates, entire displays of mannequins and props, and tree stumps and large branches used in large scenic representations.

All of the museum conservators are trained to use the system, and personnel are scheduled on a rotating basis. The most effective and safe operation is achieved by using two people during each run. Depending on the frequency of fumigation, which is limited to twice a month, up to six to eight days of personnel time are required per month. This includes time spent on getting artifacts from storage, loading them into the bubble, filling the bubble with carbon dioxide, deflating it after the run, and unloading and returning the artifacts. Typically, articles are placed directly on the floor of the bubble or, for smaller pieces, on shelves. After the top is lowered and the plastic zipper is sealed, the bubble is inflated with air and left overnight as a leak check. The air is then pumped out and replaced with an atmosphere of 60% carbon dioxide. Normally, the bubble is kept at that concentration and at room temperature for 2 weeks. As would be expected, wood-boring insects have been found harder to exterminate, and objects containing them are treated with 80% carbon dioxide for 3 weeks. A recording hygrothermograph is used to monitor RH and temperature, but these parameters change little during a run. Auxiliary humidification is not used, and the RH is generally unchanged by the partial replacement of air with carbon dioxide. Occasionally, the introduction of the dry carbon dioxide will cause the RH to fall at the beginning of a run. In a short time, however, humidity buffering

by the contents of the bubble brings the RH back to a level close to the initial value. Safety is a major concern. The bubble is normally placed in an isolated room originally designed to house a chamber employing toxic fumigants. The room has negative pressure, a ventilation system separate from the other systems in the building, and a strong fresh-air exchange. The bubble is portable and can be used elsewhere, preferably in an isolated, outdoor location where no one else is working. To follow carbon dioxide levels at critical points, an Armstrong two-channel carbon dioxide monitor is used. One sensor monitors carbon dioxide inside the bubble, and the other is calibrated to measure carbon dioxide in parts per million in the room. This will set off an alarm when the level of carbon dioxide there exceeds 1000 ppm. Other safety features include warning signs on each entrance to the room and the limiting of access to personnel operating the bubble.

Carbon dioxide fumigation at the Canadian Museum of Civilization has been highly successful. In May 1996, museum conservators reported that there had been no reinfestations occurring in the collections treated with this gas. The museum's success with carbon dioxide fumigation has also been helped with the strong support and advice from sister institutions in Canada—the Canadian Conservation Institute and the National Museum of Science and Technology. The awareness of the seriousness of insect infestation has increased at the Canadian Museum of Civilization, and this has led to a greater involvement on the part of other divisions in a total, integrated program of pest management throughout the institution.

## Treatment Experience in Germany

Perhaps the most extensive and creative application of carbon dioxide for pest control has been done in Germany by professional exterminators like Gerhard Binker. Binker is experienced in the use of the conventional toxic fumigants but has also embraced nitrogen and carbon dioxide for a variety of applications. He has designed a series of procedures to meet needs on both small and large scales, from treating movable objects to fumigating sections of buildings where containment involves using portions of the structure itself as container walls. He has even led the way in developing a technology for treating entire buildings with carbon dioxide (Binker 1993a). Binker has developed an expandable gastight chamber, sold commercially as the "Altarion," to deal with movable loads and objects of varying size. The capacity of this unit can be increased by adding chamber elements as needed. Binker claims that the system is able to control and record temperature, humidity, and gas composition for either nitrogen or carbon dioxide treatment of museum objects and archival materials. The system can be operated in locations where other activities are taking place if external gas sensors are carefully placed and monitored.

Binker treats infested objects that are very large or cannot be moved—for example, high altars, pulpits, and church pews—by first covering them with impermeable sheeting that is sealed against floor or wall sections and made as gastight as possible. Fumigation with carbon dioxide is then done in a conventional way. Binker found wood-boring beetles to be the major culprits attacking the churches and historic buildings he has treated. Before doing a partial-building fumigation for these pests, it is vital to determine that the rest of the structure is not infested. This is done with a complete and careful examination using established pest management procedures before fumigation is undertaken. It makes no sense to kill off these insects in one part of a building if adjacent areas are also infested.

An excellent example of carbon dioxide fumigation of an entire large building is the Catholic church in Schaeftlarn, Germany, that Binker treated during the

Figure 8.4
Tonnage delivery of carbon dioxide. (Photo courtesy of Gerhard Binker.)
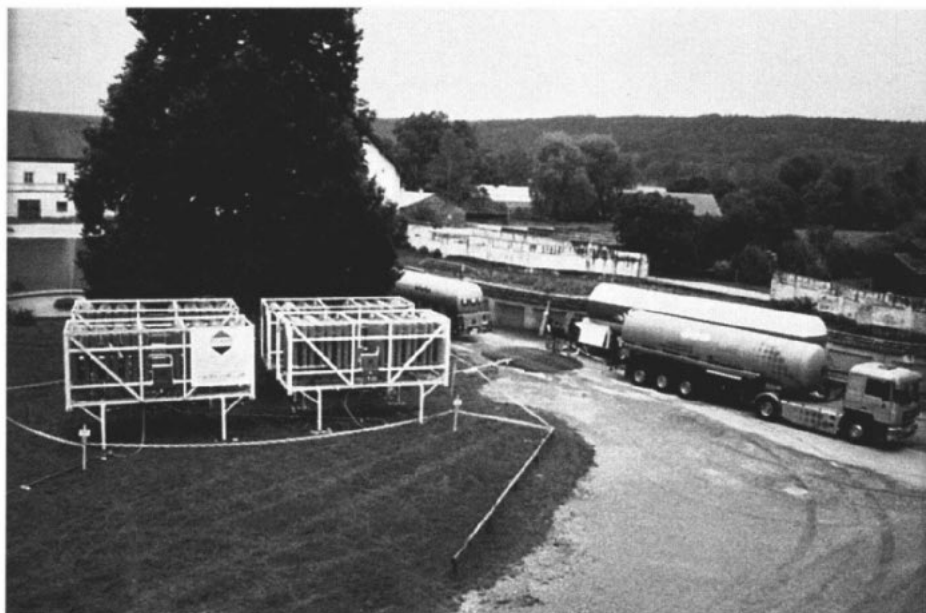


Figure 8.5
*Salmdorf Pietà* undergoing treatment. (Photo courtesy of Gerhard Binker.)

summer of 1995. The volume of space that had to be filled with gas, originally $2.31 \times 10^5$ m³, was decreased with air-inflated balloons. This reduced the amount of fumigant that was needed. Still, an enormous quantity of carbon dioxide had to be maintained at a concentration of 70% over the six weeks of treatment. To meet this need, carbon dioxide in liquid form was periodically delivered by tanker and pumped into a large supply tank. It was automatically withdrawn from the tank and passed through a battery of heat exchangers to convert it to room-temperature gas. A monitoring and control device measured the concentration of carbon dioxide in the church and automatically made up for losses incurred by leakage during the extended treatment period. An unexpected source of leakage was through the floor and out the basement. Overall, a thousand tons of carbon dioxide were needed to complete the project (Binker 1996). Figure 8.4 is a photograph showing the magnitude of the carbon dioxide delivery operation. The tanker on the right is unloading carbon dioxide into the main storage tank while a second tanker behind it waits its turn. Two banks of heat exchangers are visible on the left. Throughout the treatment period, the RH ranged from 58% to 70% and the temperature from 19 to 29 °C.

A second example of Binker's work on a large scale is the fumigation of the Catholic church in Salmdorf, near Munich, where an important wooden religious sculpture, the *Salmdorf Pietà,* is housed (Fig. 8.5). The church was infested with wood-boring beetles, particularly furniture beetles, which had gotten into the altars, pews, pulpit, and organ, as well as into the religious sculpture. After the building was covered with large sheets of poly(vinyl chloride) film, sealed with tape, and made as gastight as possible, an additional stormproof tent was constructed over the church, including the steeple (Fig. 8.6). This was done to minimize gas loss and to help retain humidity required for the protection of the wooden artifacts. A temperature and moisture sensor was placed on the Pietà to ensure that dehydration was not occurring. There were heavy rains during the sealing of the church, boosting the RH. Still, additional humidification was required to maintain a moisture level that the operators felt would protect the statues from becoming too dry and cracking. Over the treatment period, the RH ranged from 58% to 70% and the temperature from 19 to 29 °C.

In addition to worries about dehydration, there was concern that the carbon dioxide might change the polychrome of the Pietà. A pigment panel was placed near the statue during fumigation and later compared to a reference panel. Neither color change nor cracking of the statue were detected. The effectiveness of

Figure 8.6
Church tented for carbon dioxide fumigation.
(Photo courtesy of Gerhard Binker.)

the treatment was determined by sealing larvae of the furniture beetle and the old house borer inside blocks of wood and placing them in the church during fumigation. Subsequent examination found that all larvae were killed. Catholic churches in Kranzberg, Unterschwillach, Peiting, and Gstadt/Chiemsee were also successfully treated with this approach.

Binker has also demonstrated a very rapid procedure that uses carbon dioxide under pressure for dispatching insects. Objects containing infestations are placed in a pressure chamber and brought to a pressure of 300-600 psig. After two to three hours, the system is depressurized, and 100% mortality is realized. This procedure kills by a mechanism other than dehydration. The cells of insects readily absorb carbon dioxide under pressure; with rapid depressurization, the cell walls burst irreversibly and the insects die. Inert cellulosic material is not damaged by this treatment since it cannot absorb large amounts of carbon dioxide. Binker speculates that this procedure may, in the same manner, also kill fungal growth (Binker 1993b).

# Professional Contacts

## Conservators

The following conservators and conservation scientists have substantial experience using methods described in this book for the practice of insect control in museums. In many cases, they have also done important research and conducted workshops in this field. All have expressed a willingness—indeed, even an eagerness—to share their experience with other conservators and have dared to respond to requests for help.

John Burke, Chief of Conservation
Oakland Museum
1000 Oak Street
Oakland, CA 94607
Tel: (510) 238-3806
Fax:(510) 238-6123

Steve Colton, Objects Conservator
755 Luton Drive
Glendale, CA 91206
Tel: (818) 247-2398
Fax:(818) 548-6286

Vinod Daniel, Scientific Officer
Materials Conservation Division
Australian Museum
6 College Street
Sydney, NSW 2000
Australia
Tel: (61) 29320 6115
Fax: (61) 2 9320 6070
E-mail: vinodd@amsg.ausmus.gov.au

Mark Gilberg, Research Coordinator
National Center for Preservation
Technology and Training
NSU Box 5682
Natchitoches, LA 71497
Tel: (318) 357-6464
Fax: (318) 357-6421

Gordon Hanlon, Associate
Conservator
Decorative Arts and Sculpture
Conservation
The J. Paul Getty Museum
Suite 1000
1200 Getty Center Drive
Los Angeles, CA 90049-1684
Tel: (310) 440-7178
Fax: (310) 440-7745
E-mail: ghanlon@getty.edu

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Smithsonian Institution
10th and Constitution Avenue, N.W.
Washington, DC 20560
Tel: (202) 786-2550
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Alan Johnston, Principal Museums
Officer
Project Management and Collections
Care
Hampshire County Council Museums
Service
Chilcomb House
Chilcomb Lane
Bar End
Winchester SO23 8RD
United Kingdom
Tel: (44) 962 846304
Fax: (44) 962 869836

Dale Paul Kronkright, Senior
Conservator
Museum of New Mexico
P.O. Box 2087
Santa Fe, NM 87504-2087
Tel: (505) 827-6358, ext. 634
Fax: (505) 827-6349
E-mail: dpkobjscon@aol.com

Shin Maekawa, Senior Scientist
The Getty Conservation Institute
1200 Getty Center Drive
Los Angeles, CA 90049-1684
Tel: (310) 440-6813
Fax: (310) 440-7711
E-mail: smaekawa@getty.edu

Steven Pine, Decorative Arts
Conservator
Museum of Fine Arts, Houston
P.O. Box 6826
Houston, TX 77265
Tel: (713) 639-7730
Fax: (713) 639-7740

Gary Rattigan, Preparator
Society for the Preservation of
New England Antiquities
141 Cambridge Street
Boston, MA 02114
Tel: (508) 521-4788
Fax: (508) 541-4789

Dr. Thomas Strang
Canadian Conservation Institute
1030 Innes Road
Ottawa, Ontario K1A OMB
Canada
Tel: (819) 776-8417
Fax: (819) 776-8300

Dr. Nieves Valentín, Director
Ministerio de Cultura
Institute del Patrimonio Histórico
Español
Greco 4
28040 Madrid
Spain
Tel: (34) 1 544 7894
Fax: (34) 1 544 7778

Sue Warren, Conservator
National Museum of Science and
Technology
P.O. Box 8724, Station T
Ottawa, Ontario K1G-5A3
Canada
Tel: (613) 991-3061
E-mail: conserv@istar.ca

## Professional Fumigators

The following individuals are professional fumigators experienced in the use of nontoxic gases for insect control. They have worked with museum collections and are sensitive to the frailties and needs of fine art.

Werner Biebel
Biebl und Söhne, Hygiene GmbH
Bergestrasse 8
82024 Taufkürchen
Germany
Tel: (49) 89 612 00 00
Fax: (49) 89 612 00 037

Gerhard Binker
Binker Materialschutz GmbH
Postfach 4
D90571 Schwaig
Germany
Tel: (49) 911 5075011
Fax: (49) 911 5075782

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710 Bouvier Street, Suite 195
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Fax: (44) 1342 326229
E-mail: ficsman@rentokil-initial.com

Appendix B

# Materials and Suppliers

The following list of materials used to make the anoxia systems described herein is not exhaustive. Other sources for most of the items include general hardware stores, as well as laboratory and conservation supply houses.

**Anoxia Systems (Custom-Made)**
**Alan Johnston System**

***Suppliers***

**Tank**
Solent Plastics
136 Shirley Road
Southhampton
United Kingdom

**Tubes/Valves/Fittings**
Air and Hydraulic Hose Services
Unit 29E Parnham Drive
Eastleigh, Hants
United Kingdom

**Oxygen Analyzer**
Teledyne Brown Engineering
Harlequin Centre
Southall Lane
Southall, London
United Kingdom

**Perspex Sheet**
Alda Plastics
21A High Street
Lyndhurst, Hants
United Kingdom

**Neoprene Seal O-Ring**
Blue Diamond Bearings
1 Bishopstoke Road
Eastleigh, Hants
United Kingdom

**Nitrogen and Regulators**
British Oxygen Company

**Anoxia Systems (Custom-Made)**
**Steven Pine System**

**Parts**
Swagelok brass fittings: Union Tee B-600-3, Port Connector B-601-PC, Union Elbow B-600-9, Bulkhead Union B-600-61, Ferrule Set B-600-Set, Nupro Valve B-6JN

Dow Corning silicone caulk, 732, as gasket material at the couplings that penetrate the containment walls. Neoprene gaskets and Teflon tape can also be used.

Nalgone polyethylene bottles (half-gallon, Fisher 11-815-11A)

Gasketed PVC sewer pipe, 15 in. (38.1 cm) diameter by 20 ft (6.1 m) long

Gasketed PVC sewer pipe caps, 15 in. (38.1 cm) diameter, ½ in. (1.27 cm) thick

Silicone gaskets, 0.5 in. O-rings—round cross section for the Hardigg case and square cross section for the pipe. From Amerflex Rubber and Gasket Co. and many other sources.

Teledyne oxygen monitor cap—TBE/A1 part no. 1406A51

Retroseal PVC and ABS cleaner and PVC solvent cement

Cole-Parmer E-37500-06 panel-mounted digital thermohygrometer

Sears 16 in. oscillating fan

Sears Duracraft Delux, 2.5 gal humidifier

**Anoxia Tent Supplies**
**(for Custom-Made Systems)**

***Supplier***

McMaster-Carr Supply Co.
9630 Norwalk Blvd.
Santa Fe Springs, CA 90670
Tel: (310) 692-5911

Most materials can be obtained from McMaster-Carr, including ¼ in. acrylic sheets for the tent base; self-adhesive rubber grommet strips (Reg. 8 #93085-K12); centimeter-thick acrylic plates; weighting bags filled with lead shot or sand; high-vacuum grease (100 g tube, #1326K59); and parts for the frame and lifting device, i.e., aluminum pipe and pipe fittings (or use PVC pipe).

## Barrier Films

**Filmpak 1193 (oxygen transmission 0.1 cm$^3$ m$^{-2}$ day$^{-1}$, 0.12 mm thickness)**

### Manufacturer

Ludlow Laminating & Coating Division
4058 Highway 79
Homer, LA 71040
Tel: (318) 927-2531

### Suppliers

CalTex Plastics
2380 East 51st Street
Vernon, CA 90056
Tel: (213) 583-4140

Edco Supply
327 36th Street
Brooklyn, NY 11232
Tel: (718) 788-8108

Sealpak Flexible Packaging
13826 S. Prairie Avenue
Hawthorne, CA 90250
Tel: (310) 973-1321

Shannon Packaging Co.
575 E. Edna Place
Covina, CA 91723
Tel: (818) 967-7329

**Aclar Composites (Aclar oxygen transmission 51.15 cm$^3$ m$^{-2}$ day$^{-1}$ calculated value, 0.11 mm thickness)**

### Manufacturers

Aclar
Allied-Signal, Inc.
P.O. Box 233R
Morristown, NJ 07960
Tel: (201) 455-2000

Film-O-Rap 7750 (FR 7750)
Bell Fibre Products
P.O. Box 1158
Columbus, GA 31993
Tel: (706) 323-7316

Filmpak 1177
Ludlow Laminating & Coating Division
Homer, LA 71040
Tel: (318) 927-2531

### Suppliers

Keepsafe Systems, Inc.
59 Glenmount Park Road
Toronto, ON M4E 2N1
Canada
Tel: (416) 691-8854

Sealpak Flexible Packaging
13826 S. Prairie Avenue
Hawthorne, CA 90250
Tel: (310) 973-1321

Shannon Packaging Co.
575 E. Edna Place
Covina, CA 91723
Tel: (818) 967-7329

George B. Woodcock & Co.
21900 Marilla Street
P.O. Box 3009
Chatsworth, CA 91313
Tel: (818) 998-3774

**Marvelseal 360 aluminized polymer film (oxygen transmission 0.01 cm$^3$ m$^{-2}$ day$^{-1}$, 0.13 mm thickness)**

### Manufacturer

Ludlow Laminating & Coating Division
4058 Highway 79
Homer, LA 71040
Tel: (318) 927-2531

### Suppliers

CalTex Plastics
2380 East 51st Street
Vernon, CA 90056
Tel: (213) 583-4140

Shannon Packaging Co.
575 E. Edna Place
Covina, CA 91723
Tel: (818) 967-7329

George B. Woodcock & Co.
21900 Marilla Street
P.O. Box 3009
Chatsworth, CA 91313
Tel: (818) 998-3774

Preservation Equipment, Ltd.
Shelfanger, Diss
Norfolk IP22 2DG
England
Tel: (44) 1379 651 527

Conservation by Design, Ltd.
Timecare Works
60 Park Road West
Bedford MK41 7SL
United Kingdom
Tel: (44) 1234 217258

Multipak B.V.
Industrieweg 14
3881 LB Putten
Holland
Tel: (31) 341 357474

**BDF 200 (oxygen transmission 4 cm$^3$ m$^{-2}$ day$^{-1}$, 0.025 mm thickness)**

### *Supplier*

Conservation by Design, Ltd.
Timecare Works
60 Park Road West
Bedford MK41 7SL
United Kingdom
Tel: (44) 1234 217258

## Carbon Dioxide Detection Systems

### *Suppliers*

Bruel and Kjaer
Div. of Spectris Technologies, Inc.
2364 Park Central Blvd.
Decatur, GA 30035
Tel: (800) 332-2040

Campbell Scientific Inc.
815 West 1800 North
Logan, UT 84321
Tel: (801) 753-2342

CEA Instruments, Inc.
16 Chestnut Street
Emerson, NJ 07630
Tel: (201) 967-5660

Gas Tech, Inc.
8407 Central Avenue
Newark, CA 94560
Tel: (510) 745-8700

Teledyne Analytical Instruments
16830 Chestnut Street
City of Industry, CA 91740
Tel: (818) 961-9221

## Electrical Feed-Through, Hermetically Sealed Connector

### *Suppliers*

Douglas Engineering Co.
14 Beach Street
Rockaway, NJ 07866
Tel: (201) 627-8230

Omega Engineering
1 Omega Drive
Stamford, CT 06907
Tel: (203) 359-1660

Pave Technology Co., Inc.
2751 Thunderhawk Court
Dayton, OH 45414
Tel: (513) 890-1100

Foiltec GmbH
Steinacker 3
28717 Bremen
Germany
Tel: (49) 421 69351-0
(This company supplies valve that can be converted.)

## Heat Sealers

### *Suppliers*

Tacking Iron (Sealector II)
Conservation Materials
P.O. Box 2884
Sparks, NV 89432
Tel: (702) 331-0582

McMaster-Carr Supply Co.
9630 Norwalk Blvd.
Santa Fe Springs, CA 90670
Tel: (310) 692-5911

Spatula Tacking Iron (Tacking Tool 74535AZ)
McMaster-Carr Supply Co.
9630 Norwalk Blvd.
Santa Fe Springs, CA 90670
Tel: (310) 692-5911

Spade-Tip Tacking Iron (Adem C6)
Conservation Materials
P.O. Box 2884
Sparks, NV 89432
Tel: (702) 331-0582

Pouch Sealing Machine
Preservation Equipment, Ltd.
Shelfanger, Diss
Norfolk 1P22 2DG
England
Tel: (44) 1379 651527

Automatic Impulse Sealer
(#92-96348)
National Bag Co.
2233 Old Mill Road
Hudson, OH 44236
Tel: (216) 425-2600

## Humidification Systems
## (Commercial)

### *Suppliers*

Edge Tech Moisture and Humidity
Systems
455 Fortune Blvd.
Milford, MA 01757
Tel: (800) 276-3729
Fax:(800) 634-3010

General Eastern
20 Commerce Way
Woburn, MA 01801
Tel: (617) 938-7070

Maheu & Maheu, Inc.
710 Rue Bouvier, Suite 195
Quebec, QC G2J 1C2
Canada
Tel: (418) 623 8000

Vacudyne Inc.
375 Joe Orr Road
Chicago Heights, IL 60411
Tel: (708) 757-5200

Kojima Seisakujo Inc.
6F, Popura Building
Ohdenma-cho, Nihonbashi, Chuo-ku
Tokyo 103
Japan

Power-Plastics, Ltd.
Station Road
Thirsk
North Yorkshire YO7 1PZ
England
Tel: (44) 1845 525503

## Humidification Systems
## (Parts for Custom Systems)

### *Suppliers*

**Heavy-Duty Polypropylene Bottle**
(4 l volume, #02-923-5)
Fisher Scientific (Headquarters)
711 Forbes Avenue
Pittsburgh, PA 15219
Tel: (412) 562-8300

**Flowmeter**
Cole-Parmer Instrument Co.
7425 North Oak Park Avenue
Niles, IL 60714
Tel: (800) 323-4340

**Nylon Tubing**
Cole-Parmer Instrument Co.
7425 North Oak Park Avenue
Niles, IL 60714
Tel: (800) 323-4340

**Swagelok Tube Fitting and Valves**
Angeles Valve & Fitting Co.
427 S. Victory Blvd.
Burbank, CA 91502
Tel: (213) 849-6257

B.E.S.T. Ventil & Fitting
GmbH Frankfurt
Robert-Bosch-Str. 20
63477 Maintal-Doernigheim
Germany
Tel: (49) 6181 46041

Birmingham Valve & Fitting Co., Ltd.
Claymore, Tame Valley Industrial
Estate
Tamworth
Staffordshire B77 5DQ
England
Tel: (44) 827 281118

## Humidity Monitor

Testo 610 Thermohygrometer

### *Supplier*

Conservation Support Systems
EM-12105
P.O. Box 91746
Santa Barbara, CA 93190-1746
Tel: (805) 682-9843

### *Manufacturers*

ACR Systems, Inc.
Surry
British Columbia, Canada
Tel: (604) 591-1128

Cole-Parmer Instrument Co.
Vernon Hills, IL
Tel: (847) 549-7600

General Eastern Instruments
Woburn, MA
Tel: (617) 938-7070

Hy-Cal Engineering
El Monte, CA
Tel: (818) 444-4000

Hygrometrix, Inc.
Alpine, CA
Tel: (619) 659-9292

Kahn Instruments, Inc.
Wethersfield, CT
Tel: (860) 529-8643

Metrosonics, Inc.
Rochester, NY
Tel: (716) 334-7300

Omega Engineering
Stamford, CT
Tel: (203) 359-1660

Rotronic Instrument Corp.
Huntington, NY
Tel: (516) 427-3898

Teledyne Analytical Instruments
City of Industry, CA
Tel: (818) 961-9221

TSI Inc.
St. Paul, MN
Tel: (612) 483-0900

Vaisala, Inc., Sensors Systems Div.
Woburn, MA
Tel: (617) 933-4500

## Leak Detectors

### *Manufacturers*

Dresser Industries, Inc.
Newton, CT
Tel: (203) 426-3115

Foxboro ICT
San Jose, CA
Tel: (408) 432-1860

Gas Tech, Inc.
Newark, CA
Tel: (510) 745-8700

Matheson Gas Products
Montgomeryville, PA
Tel: (215) 641-2700

Neotronics Scientific, Inc.
Flowery Branch, GA
Tel: (770) 967-2196

Scott Specialty Gases Inc.
Plumsteadville, PA
Tel: (215) 766-8861

TIF Instruments, Inc.
9101 NW 7th Avenue
Miami, FL 33150
Tel: (800) 327-5060
Tel: (305) 757-8811
Fax: (305) 757-3105

TSI Inc.
St. Paul, MN
Tel: (612) 483-0900

Universal Sensors and Devices, Inc.
Chatsworth, CA
Tel: (818) 998-7121

Weed Instruments
Round Rock, TX
Tel: (512) 434-2900

## Nitrogen
## (High-Pressure Cylinders)

In addition to the primary suppliers below, others may be listed in local telephone directories. Request guaranteed analyses and price quotations for high-purity nitrogen or argon.

### *Suppliers*

Gilmore Cryogenics
9503 East Rush Street
South El Monte, CA 91733
Tel: (213) 283-4721

Union Carbide Industrial Gases Inc.
Linde Division
200 Cottontail Lane
Sommerset, NJ 08875
Tel: (201) 271-2600

Air Products
United Kingdom
Tel: (44) 01 256 7062

Linde AG
Seitner Str. 70
82049 Hoelriegelskreuth
Germany
Tel: (49) 89 7446 1360

## Nitrogen (Liquid-Nitrogen Tanks) (160 liters of liquid nitrogen, 99,000 liters of gas, 99.998% purity)

### *Suppliers*

Gilmore Cryogenics
9503 East Rush Street
South El Monte, CA 91733
Tel: (213) 283-4721

Union Carbide Industrial Gases Inc.
Linde Division
200 Cottontail Lane
Sommerset, NJ 08875
Tel: (201) 271-2600

Air Products
United Kingdom
Tel: (44) 01 256 7062

Linde AG
Seitner Str. 70
82049 Hoelriegelskreuth
Germany
Tel: (49) 89 7446 1360

## Nitrogen Generator

**Permea Prism Model 1300 (performance: 41 m$^3$ day$^{-1}$ of 99.99% nitrogen)**

### *Suppliers*

Air Products and Chemicals, Inc.
7201 Hamilton Blvd.
Allentown, PA 18195
Tel: (610) 481-4911

Kojima Seisakujo Inc.
6F, Popura Building
Ohdenma-cho, Nihonbashi, Chuo-ku
Tokyo 103
Japan

## Nitrogen Regulators (for Cylinders and Liquid-Nitrogen Tanks)

### *Suppliers*

Gilmore Cryogenics
9503 East Rush Street
South El Monte, CA 91733
Tel: (213) 283-4721

Union Carbide Industrial Gases Inc.
Linde Division
200 Cottontail Lane
Sommerset, NJ 08875
Tel: (201) 271-2600

Air Products
United Kingdom
Tel: (44) 01 256 7062

Linde AG
Seitner Str. 70
82049 Hoelriegelskreuth
Germany
Tel: (49) 89 7446 1360

Power-Plastics, Ltd.
Station Road
Thirsk
North Yorkshire YO7 1PZ
England
Tel: (44) 1845 525503

Oxygen Absorbers

Ageless Z

**Manufacturers**

Mitsubishi Gas Chemical America, Inc.
520 Madison Avenue
25th Floor
New York, NY 10022
Tel: (212) 752-4620

Mitsubishi Gas Chemical Europe
GmbH
Immermannstrasse 45
4000 Düsseldorf 1
Germany
Tel: (49) 211 363080

**Suppliers**

Conservation Support Systems
P.O. Box 91746
Santa Barbara, CA 93190-1746
Tel: (805) 682-9843

Cryovac Division
W. R. Grace & Co.
16201 Commerce Way
Cerritos, CA 90701
Tel: (562) 926-0424

Cryovac Division
W. R. Grace & Co.
P.O. Box 464
Duncan, SC 29334
Tel: (803) 433-3121

Keepsafe Systems, Inc.
59 Glenmount Park Road
Toronto, ON M4E 2N1
Canada
Tel: (416) 691-8854

Conservation by Design, Ltd.
Timecare Works
60 Park Road West
Bedford MK41 7SL
United Kingdom
Tel: (44) 1234 217258

Cryovac
Grace GmbH
Erlengang 31
22841 Norderstedt
Germany
Tel: (49) 40 52601-0

Atco

**Manufacturer**

Standa Industrie
184 Rue Maréchal-Gallieni
14050 Caen
France
Tel: (33) 31745489

*Supplier*

Preservation Equipment, Ltd.
Shelfanger, Diss
Norfolk IP22 2DG
England
Tel: (44) 1379651527

Oxygen Analyzer
(Specification Unknown)

**Supplier**

Power-Plastics, Ltd.
Station Road
Thirsk
North Yorkshire YO7 1PZ
England
Tel: (44) 1845 525503

Oxygen Indicators (Passive)

Ageless-Eye

**Manufacturers**

Mitsubishi Gas Chemical America, Inc.
520 Madison Avenue
25th Floor
New York, NY 10022
Tel: (212) 752-4620

Mitsubishi Gas Chemical Europe
GmbH
Immermannstrasse 45
4000 Düsseldorf 1
Germany
Tel: (49) 211 363080

**Suppliers**

Conservation by Design, Ltd.
Timecare Works
60 Park Road West
Bedford MK41 7SL
United Kingdom
Tel: (44) 1234 217258

Cryovac
Grace GmbH
Erlengang 31
22841 Norderstedt
Germany
Tel: 49 (0) 40 52601-0

Power-Plastics, Ltd.
Station Road
Thirsk
North Yorkshire YO7 1PZ
England
Tel: (44) 1845 525503

## Oxygen Indicator Eye

### *Supplier*

Preservation Equipment, Ltd.
Shelfanger, Diss
Norfolk IP22 2DG
England
Tel: (44) 1379 651 527

## Oxygen Sensors (Electronic)

### *Manufacturers*

Ametek, Inc., U.S. Gauge Div.
Feasterville, PA
Tel: (215) 355-6900

Control Instruments Corp.
Westbury, NY
Tel: (516) 876-8400

Delta F. Corp.
Woburn, WA
Tel: (617) 935-4600

Gas Tech, Inc.
Newark, CA
Tel: (510) 745-8700

Matheson Gas Products
Montgomeryville, PA
Tel: (214) 641-2700

Neutronics Inc.
Exton, PA
Tel: (610) 524-8800

Panametrics Inc.
Process Control Instrument Division
Tel: (800) 833-9438

BühlerMess- u. Regeltechnik
GmbH & Co. KG
40880 Ratingen
Germany
Tel: (49) 2102 49890

Teledyne, Ltd.
Southhall La
London VB2 5NH
England
Tel: (44) 181 5719596

## Temperature Control Systems

### *Suppliers*

Honeywell Industrial
Automation & Control
16404 N. Black Canyon Highway
Phoenix, AZ 85023
Tel: (602) 863-5000

Johnson Controls Inc.
Milwaukee, Wl
Tel: (800) 333-2222

Omega Engineering
1 Omega Drive
Stamford, CT 06907
Tel: (203) 359-1660

Yokogawa Corporation of America
Test & Measurement Division
2 Dart Road
Newnan, GA 30265
Tel: (800) 258-2552

## Temperature Monitors

### *Manufacturers*

ACR Systems, Inc.
Surry, British Columbia, Canada
Tel: (604) 591-1128

Cole-Parmer Instrument, Co.
Vernon Hills, IL
Tel: (847) 549-7600

Everest Interscience, Inc.
Tucson, AZ
Tel: (520) 797-0927

Omega Engineering
Stamford, CT
Tel: (203) 359-1660

R. M. Young Co.
Traverse City, Ml
Tel: (616) 946-3980

Raytek, Inc.
Santa Cruz, CA
Tel: (408) 458-1175

Vaisala, Inc., Sensors Systems Div.
Woburn, WA
Tel: (617) 933-4500

Weed Instrument
Round Rock, TX
Tel: (512) 434-2900

Young Environments Systems
Richmond, British Columbia, Canada
Tel: (604) 276-9923

YSI Inc.
Yellow Springs, OH
Tel: (513) 767-7241

## Treatment Containers

### Rigid Enclosures

#### *Suppliers*

Bemco Inc.
Environmental & Space Simulation
Systems
2255 Union Place
Simi Valley, CA 93065
Tel: (805) 583-4970

Harris Environmental Systems, Inc.
11 Connector Road
Andover, MA 01810
Tel: (508) 475-0104

Thermatron Industries
291 Kollen Park Drive
Holland, Ml 49423
Tel: (616) 393-4580

Vacudyne Inc.
375 Joe Orr Road
Chicago Heights, IL 60411
Tel: (708) 757-5200
(Company supplies conversion kit
for chambers previously used for
ethylene oxide fumigation.)

### Soft Enclosures
### (Rentokil Bubble)

#### *Suppliers*

Maheu & Maheu, Inc.
710 Rue Bouvier, Suite 195
Quebec, QC G2J1C2
Canada

Power-Plastics, Ltd.
Station Road
Thirsk
North Yorkshire YO7 1PZ
England
Tel: (44) 1845 525503

# Appendix C

# Common and Scientific Names of Insect Species

| Common Name | Scientific Name |
| --- | --- |
| American cockroach | *Periplaneta americana* |
| Australian spider beetle | *Ptinus ocellus* |
| Black carpet beetle | *Attagenus piceus* |
| Book beetle | *Nicobeum casteneum* |
| Book louse | *Liposcelis corrodens* |
| Brownbanded cockroach | *Supella longipalpa* |
| Cabinet beetle | *Trogoderma inclusum* |
| Casemaking clothes moth | *Tinea pellionella* |
| Cigarette beetle | *Lasioderma serricorne* |
| Common clothes moth | *Tineola bisselliella* |
| Confused flour beetle | *Tribolium confusion* |
| Deathwatch beetle | *Xestobium rufovillosum* |
| Drugstore beetle | *Stegobium paniceum* |
| Drywood termite | *Cryptotermes brevis* |
| Firebrat | *Thermobia domestica* |
| Fruit fly | *Drisopholus melanogaster* |
| Fur beetle | *Attagenus fasciatus* |
| Furniture beetle | *Anobium punctatum* |
| Furniture carpet beetle | *Anthrenus flavipes* |
| German cockroach | *Blattella germanica* |
| Grain mite | *Acarus siro* |
| Hide beetle | *Dermestes maculatus* |
| Khapra beetle | *Trogoderma granarium* |
| Larder beetle | *Dermistes lardarius* |
| Longhorn borer beetle[a] | *Hylotrupes bajulus* |
| Merchant grain beetle | *Oryzaephilus mercator* |
| Mold mite | *Tyrophagus putrescentia* |
| Odd beetle | *Thylodrias contractus* |
| Old house borer[a] | *Hylotrupes bajulus* |
| Powderpost beetle | *Lyctus* spp. |
| Red flour beetle | *Tribolium castaneum* |
| Redlegged ham beetle | *Necrobia rufipes* |
| Rice weevil | *Sitophilus oryzae* |
| Silverfish | *Lepisma saccharina* |
| Varied carpet beetle | *Anthrenus verbasci* |
| Webbing clothes moth | *Tineola bisselliella* |

[a] One of several common names for *Hylotrupes bajulus.*

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# Index

*Note: The index designations* f *and* t *refer, respectively, to figures and tables.*
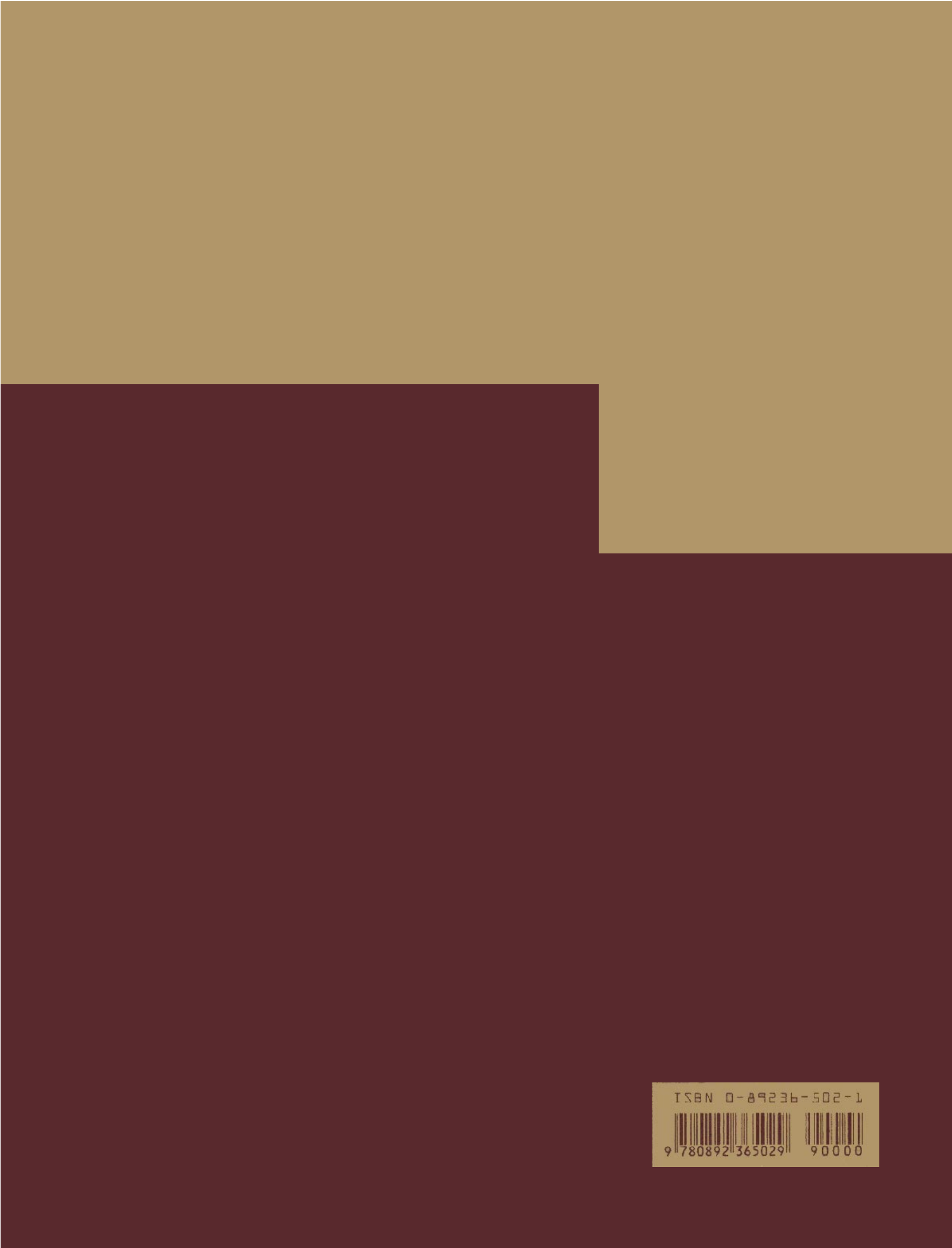
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