

Detection of insect infestation in museum objects by carbon dioxide measurement using FTIR

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Abstract

Detecting the presence of active insects in various stages of their lifecycle within an art object is often difficult in the absence of direct visualization of the insect. It usually relies on one's ability to assess the evidence of their activity around, on, or in the object. To augment this "eyeball" method, a Fourier transform infrared spectroscopy system was used to determine the presence of the insect through its respired gas, carbon dioxide. The application of the procedure to assist in control of insect infestation within art objects is described. Two instruments are discussed in this paper. The prototype instrument could detect a change in carbon dioxide that was 0.3–0.4 ppm/h and required days to reliably determine this level. The second instrument could detect changes as low as 0.09 ppm/h and could provide results in only 4 h. Carbon dioxide measurements were collected from representative insect groups that have caused problems in art objects; these were worker termites (*Reticulitermes* sp.), carpet beetle adult and larvae (*Anthrenus* sp.), an odd beetle larva (*Thylocladius* sp.) and a mature silverfish (*Lepisma* sp.), as well as selected museum art objects composed of wood, fabric, and/or feathers suspected of harboring insects. © 2001 Elsevier Science Ltd. All rights reserved.

1. Introduction

Insect infestation is a major threat to the preservation of art objects. Unchecked they can irrevocably damage objects and decimate a collection. Museums expend significant efforts to minimize the damage and prevent the spread of insect infestations within their collections. Many art objects, because of their material composition and construction, present difficulties in visually detecting whether insects are hidden within, as does the type of insect, its lifecycle stage, and its habits. Recognizing the presence of an active insect infestation requires trained eyes and experience. It is rare to find adult insects or larvae, usually objects are suspected of insect activity when remnants such as larval or pupal casings, frass, or recent exit holes are found. (In wooden objects, these are generally lighter around the edge of the hole because the adult insect had to chew its way out of the interior, and in doing so exposes new edges of the wood to view.) Without a history of the object's condition, it is often difficult to interpret the remnants as indicating an active infestation.

Any scientific approach used to assess for the presence of an infestation and to test the effectiveness of a treatment is constrained by the necessity of ensuring that no harm is done to the art object. The art object cannot be treated as a normal laboratory sample and "opened up" to determine if it is in fact infested by insects or if indeed a treatment had been successful. Instead, an indirect approach has to be employed to answer these questions.

Once an active infestation is suspected, the usual course of action includes prompt isolation of the infested object(s), observation, and insect eradication treatment. The technique that has been in use at the Metropolitan Museum of Art, and many other museums worldwide, since 1992 is the use of a low oxygen atmosphere generated with argon gas to suffocate insect infestations. Unlike chemical fumigation this treatment, when performed properly, has no known side effects and is believed to be safe for objects, the environment and personnel (Koestler, 1996). The anoxic treatment method is replacing the use of chemical treatments in collections worldwide (see Zycherman and Schrock, 1988; or Mallis and Moreland, 1997, for a discussion of chemical treatment for pest control in museums).

While we have confidence in the use of this treatment, there has been up to now no accurate method to evaluate

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Fig. 1. The prototype Bomem–Michelson FTIR with ambient temperature 25-cm single-pass cell with a fixed path length optimized to measure CO₂, vacuum pump, a computer for data reduction and analysis and control of the valving system.

the effectiveness of the treatment when insects are harbored within the object or even to determine whether the object had in fact been initially infested as suspected. The current practice to determine if an infestation is active (before or after treatment) is to isolate the treated object and to periodically examine it for signs of any new insect activity. This is often impractical to carry out because of time constraints from exhibition schedules, and the unavailability of trained personnel. Other possible solutions, tried in the agricultural field, include the use of sound, X-rays, chemical reagents, microwaves, nuclear magnetic resonance or Fourier transform infrared spectroscopy (FTIR). All but FTIR are currently impractical or unsafe for museum objects (Koestler, 1993).

2. Materials and methods

Two different FTIR systems were tested for their ability to measure the change in insect respired CO₂ levels over time. The first system used a Bomem–Michelson FTIR with ambient temperature 25-cm single-pass cell with a fixed path length optimized to measure CO₂, and a computer for data reduction, analysis, and control of a vacuum pump and valving system; this system was reported in Koestler (1993) and is seen in Fig. 1. The design of this system permitted a dual-bag approach — one bag contained the control gas, and the other the art object to be measured. The vacuum pump would, at set intervals (usually 2 h intervals), draw air from the control bag into the cell to generate a background reading, then at other predetermined intervals, that ranged from 30 min to 24 h, draw air from the bag containing the art object into the cell. For each measurement, the vacuum pump system extracted 250 ml of air from the bag. Difference measurements were calculated at the 2330 cm⁻¹ region for CO₂.

The second system used a Bomem MB 100, combined with a more sensitive InSb liquid-N₂-cooled (LN₂) detector for mid-IR ranges (Fig. 2). This was coupled with a capillary pump air recirculating system to move the air into the cell from the measurement bag and back. With this system the air was re-circulated through a single bag containing the object

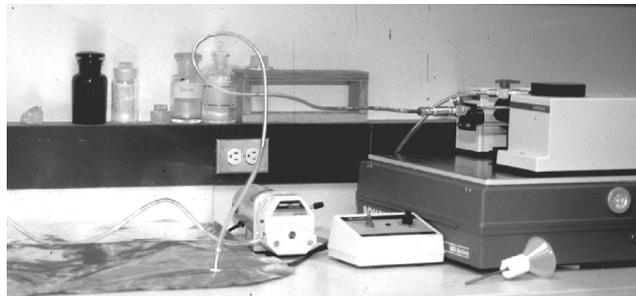


Fig. 2. The Bomem MB 100, combined with an InSb liquid N₂-cooled detector for mid-IR ranges and capillary-pumped system.

to be measured and the sensor. The flow rate and duration of the pumping were controlled to experimentally determined values. Valves were inserted at opposite corners of the bag to promote more thorough mixing of the re-circulated air. Tygon tubing was used on the line connecting the capillary pump with the cell and bag; stainless-steel tubing was used for most of the return line. Since Tygon tubing adsorbs CO₂, steel tubing was used where possible to reduce this error. At periodic intervals, the pump was stopped, turbulence was allowed to subside in the FTIR cell for 1–2 min, and triplicate readings were collected by the FTIR in the 2330 cm⁻¹ region for CO₂. Quantitative results were generated by comparison of the experimental counts with a calibration line for CO₂ (generated from gases with 10, 10.5, 53.7, 105, 517, 1000, 1538 and 3038 ppm of CO₂ in air). The air within each bag generally started at ambient room CO₂ levels, usually more than 300 ppm, unless flushed with HC-free air, or a calibration gas.

Objects to be measured were encapsulated in heat-sealable aluminized plastic (Marvalseal 360, Ludlow Corp., USA). The aluminized-plastic prohibits the commingling of respired gases with outside air. The volumes of the bags that have been tested ranged from 1 to 10 l.

In this study, the change in CO₂ over time was more important than the absolute value. Since the initial measurement varies depending on the level of CO₂ in the air surrounding the bagged object, results were normalized to 0.0 ppm for the initial data point on each graph. Results of insect-CO₂ measurements are averages of three separate runs, except for the adult carpet beetle that represents only one measurement cycle. Regression lines were generated from the normalized data (95% confidence intervals (CI) lines are indicated for the means of the regression line for data collected with the second system).

3. Results and discussion

3.1. The air-path FTIR

The air-path FTIR system tested using 20 subterranean worker termites (*Reticulitermes* sp., in wood fragments) is shown in Fig. 3. The instrument variability is compared with

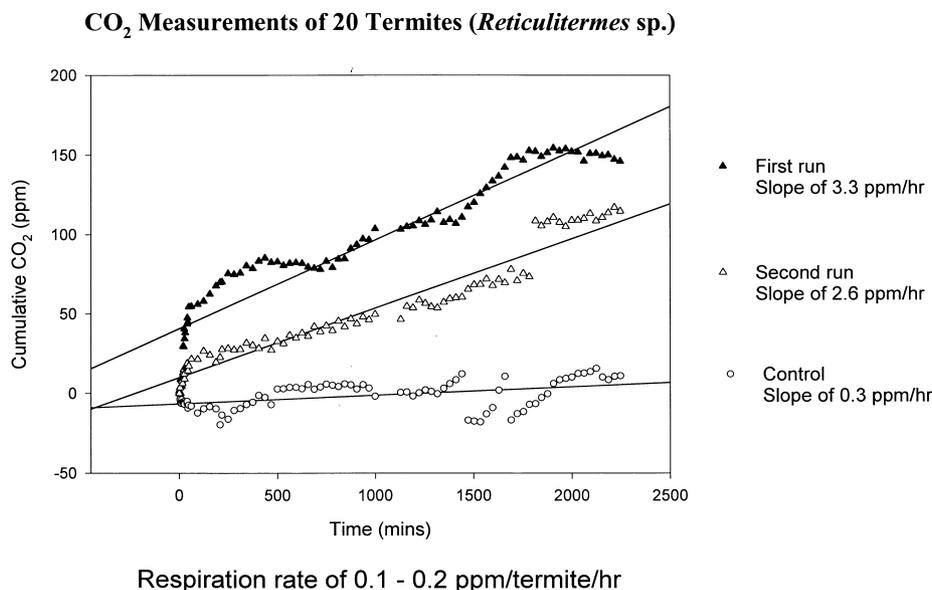


Fig. 3. CO₂ respiration measurements using the prototype air-path cell of 20 common ground worker termites (*Reticulitermes* sp.). Respiration rate of 0.1–0.2 ppm/termite/h.

the CO₂ output of the 20 termites over a period of 40 h. The regression line of the control test runs had a slope of 0.3 ppm/h. It should be noted that each data point could vary by ± 32 ppm, therefore, the accuracy of this slope could only be achieved after many hours or even days of data collection. Below that value, the variability in sample collection and the instrumentation masked any increase in CO₂. Measurements were taken over several weeks, in separate 2–3 days runs. In each case a steady increase in CO₂ level was detected over the course of each of the tests. The diurnal cycle of the termites can be seen on the first run (represented by the upper line) — it illustrates a higher activity level during the day and a lower activity level at night. The respiration rate is calculated to be 0.1–0.2 ppm/termite/h. The second run (represented by the middle line) shows less of a slope with a more even-paced respiration probably because by then the termites were already dying out and under more stress.

The measurements obtained using the FTIR air-path system indicated that worker *Reticulitermes* sp. termites produced about 0.1–0.2 ppm/termite/h in a 1 l volume. This respiration rate compares favorably with the results of O₂ consumption and CO₂ production by other wood boring insects (Dr. Frank Pohleven, University of Ljubljana, Slovenia, personal communication). Bruce and Street (1974), using a continuous-flow FTIR system, reported that they detected a 1 ppm/min change of CO₂ from 1 grain beetle living in 1 grain of cereal in about a 1 l volume. It would seem unlikely that the grain beetle respiration rate would be orders of magnitude greater than the larger insects reported in the present paper (see Table 1).

After establishing the instrument variability and detection sensitivity with known insects, tests were conducted on objects suspected of being infested. Fig. 4 shows mea-

Table 1
Respiration rate of selected insects

Ground worker termites <i>Reticulitermes</i> sp.	0.1–0.2 ppm/termite/h
Carpet beetle adult <i>Anthrenus</i> sp.	0.09 ppm/adult carpet beetle/h
Carpet beetle larva <i>Anthrenus</i> sp.	0.65 ppm/larva/h
Odd beetle larva <i>Thylocharis</i> sp.	0.35 ppm/larva/h
Silverfish adult <i>Lepisma</i> sp.	0.07 ppm/adult silverfish/h (below instrument detection limits)

surements from a wood frame of a Degas drawing that was infested with dry wood termites (*Cryptotermes* sp.). The insects produced 3.1 ppm/h of CO₂. Fig. 5 shows before and after treatment measurements of a gilt wood frame from a Flemish painting. A before treatment measurement, over 4 days, showed a steady increase in CO₂ with a slope of 52.6 ppm/h. The experimental bag was then flushed with humidified argon until the oxygen level was below 0.1% (1000 ppm) and new CO₂ measurements taken. No CO₂ was produced under the anoxic environment. After 16 days the argon was flushed out and the bag filled with ambient laboratory air. Carbon dioxide measurements were taken over the next 3 days to assess for insect recovery. No significant increase in CO₂ was found — 0.4 ppm/h — an indication that the insects were dead or had not recovered.

Measurements from the two frames demonstrated that the air-path FTIR was able not only to detect the presence of an insect infestation in an art object but also permitted an estimate of the level of the infestation. A low level of

CO₂ Measurements from a Frame of a Degas Drawing

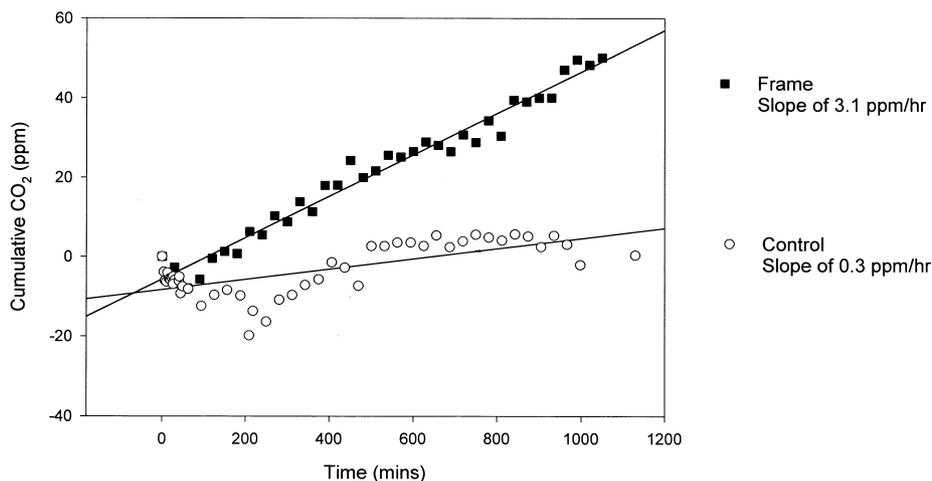


Fig. 4. CO₂ respiration measurements using air-path cell system of an infested wood frame from a Degas drawing. Respiration rate of 3.1 ppm/h. for *Cryptotermes* sp. infestation.

CO₂ Measurements from a Flemish Frame (before and after treatment)

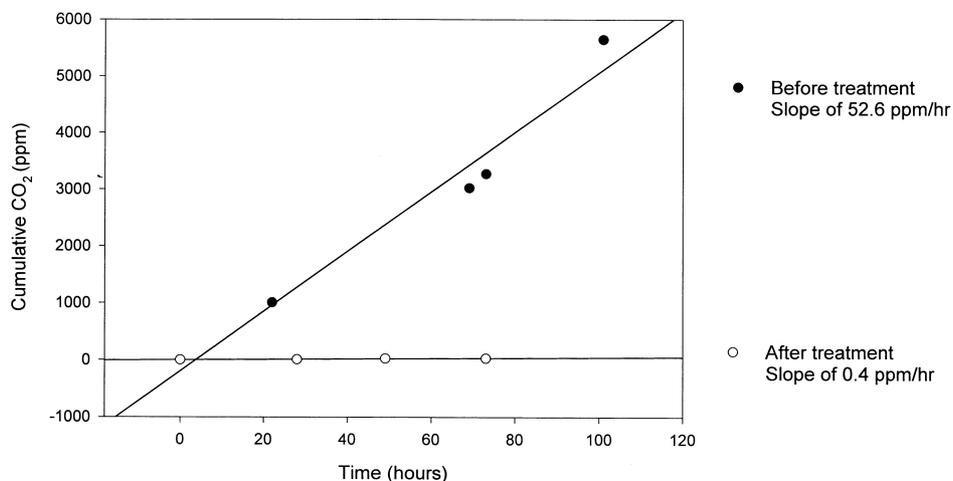


Fig. 5. CO₂ respiration measurements using the prototype air-path cell from an infested wood frame of a Flemish painting — before and after treatment. Before treatment slope is 52.6 ppm/h and after treatment 0.4 ppm/h.

infestation for this system was about 0.5 ppm/h range. The 3.1 ppm/h respiration rate from the Degas frame was significantly above the 0.3–0.4 background levels for the air-path system and was considered to be a modest infestation. The respiration rate of the Flemish painting, 52.6 ppm/h, indicated a much greater infestation.

The air-path FTIR system has advantages and disadvantages. Some advantages are that it is relatively inexpensive to purchase and maintain and it can be set to measure respiration over many days, or weeks. Some disadvantages are that it requires 1–2 weeks of testing to reliably assess for

most levels of infestations; for low levels of infestation, more testing time is necessary. In a museum context, where the concern of infestation is most often raised when objects are in preparation for an exhibition or a loan to another museum, one week, or more, is often not available to test objects, let alone treat them for infestation. Another drawback of this air-path system is the vacuum pump system; it reduces the volume (by about 250 ml) each time it takes a measurement thus inflating the true measure (this by itself is not a problem here) and thereby limits the number of measurements that could be taken before a vacuum is pulled on the object.

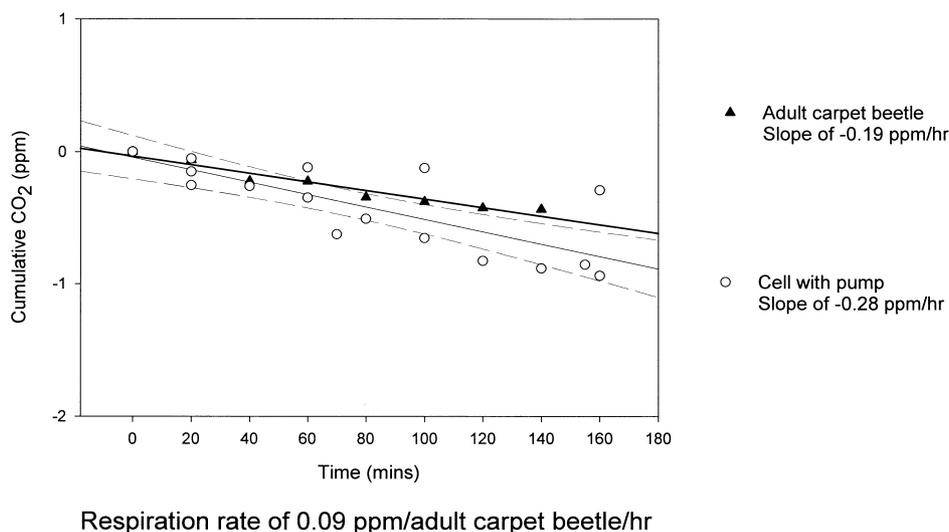
CO₂ Measurements of 1 Adult Carpet Beetle (*Anthrenus* sp.)

Fig. 6. CO₂ measurements using the LN₂-cooled-InSb system of a single adult carpet beetle (*Anthrenus* sp.). Slope is just above the 95% CI for the control line of the means. Respiration rate is 0.09 ppm/h.

3.2. The LN₂-cooled InSb FTIR

This system is more expensive and complicated to acquire good data with than the air-path system. The precision of any measurement for the LN₂-cooled-InSb detector is ± 0.006 ppm. A single measurement by itself does not permit an accurate assessment of the state of insect infestation, rather a series over 4 h, with 2 or 3 different runs plotted with regression lines proved to be the most reliable method. Background measurements of the cell by itself and the cell with the re-circulating capillary pump indicated the greater variability of the pumped system. The pumping system, adsorption of CO₂ to the Tygon tubing, and/or variable mixing of CO₂ within the bag, combined with the detector variability reduced the sensitivity of the system. The regression line for the pump background was used as the control and is indicated as such in Figs. 6–11. Measurements of one adult carpet beetle, in a 1 l bag are illustrated in Fig. 6. The pump regression mean and 95% confidence interval (CI) for the mean of the regression lines are drawn on the graph. The 95% CI indicates the region where 95% of the time the regression line of the pump control will fall; any single measurement may not. The slope of the cell with the pump is -0.28 ppm/h while the slope of a single adult carpet beetle is -0.19 ppm/h. The slope of one adult carpet beetle, -0.19 ppm/h, is just outside the 95% CI for the background. This means that one adult carpet beetle produced about 0.09 ppm/h of CO₂. This value is at or near the detection limit of the system.

Data from other insects measured with the LN₂-cooled system are seen in Figs. 7–9 and are summarized in Table 1. Three carpet beetle larvae (*Anthrenus* sp.) produced a slope

of 1.8 ppm/h, for a total change of 2.08 ppm/h. Therefore, the average respiration rate of each larva is 0.69 ppm/h (Fig. 7). These three larvae were clearly detectable within 20 min. Carbon dioxide production of one odd beetle larva (*Thyrodrias* sp.) took longer, about 40 min, to clearly detect the presence of the insect (Fig. 8). The respiration rate of one odd beetle larva is calculated to be about 0.35 ppm/h. One adult silverfish (*Lepisma* sp.), on the other hand, produced a slope of -0.21 ppm/h, for a respiration rate of 0.07 ppm/h. This is not distinguishable from the background at the 95% CI. An interesting point is that if the CI is lowered to 1 standard deviation, so that more false positives are accepted, then the single silverfish would have been detected.

The lower limits of detection of the LN₂-cooled system are encountered when measuring one adult beetle (Fig. 6) or one adult silverfish (Fig. 9). Considering biological variability it is likely that one adult insect in a 1-l bag would sometimes not be detected. In practice, this is not really a serious drawback for many reasons: it is rare to find just one insect infesting an object, for most of the insects that are museum problems it is the larval stage that causes the damage and this stage seems to be easily detectable by CO₂, and adult insects that cause problems either are easily seen, like silverfish that scurry away when disturbed, or live in colonies, like dry wood termites and thus would be easily detected by CO₂. The egg stage might be difficult to detect, but this generally represents about a 2 week period in the lifecycle for most collection pests.

Measurements performed with the LN₂-cooled system of art objects suspected of being infested are illustrated in Figs. 10 and 11. Fig. 10 shows measurements from an American

CO₂ Measurements of 3 Carpet Beetle Larvae (*Anthrenus* sp.)

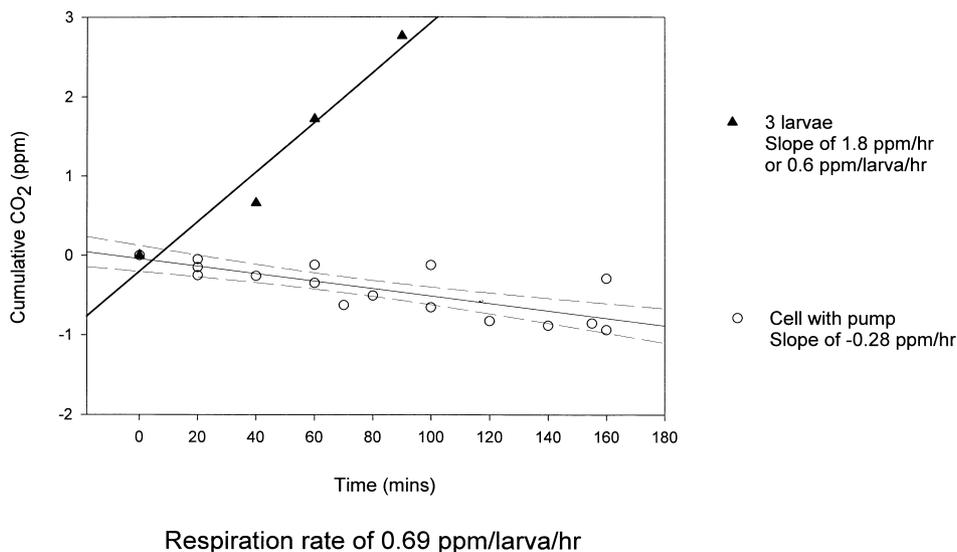


Fig. 7. CO₂ measurements of three carpet beetle larvae (*Anthrenus* sp.) using the LN₂-cooled InSb with capillary pump system. Background slope is -0.28 ppm/h. The 95% CI for control line of the means is also graphed. The larvae are clearly detected within 20 min. The respiration rate is 0.69 ppm/larva/h.

CO₂ Measurements of 1 Odd Beetle Larva (*Thylophorus* sp.)

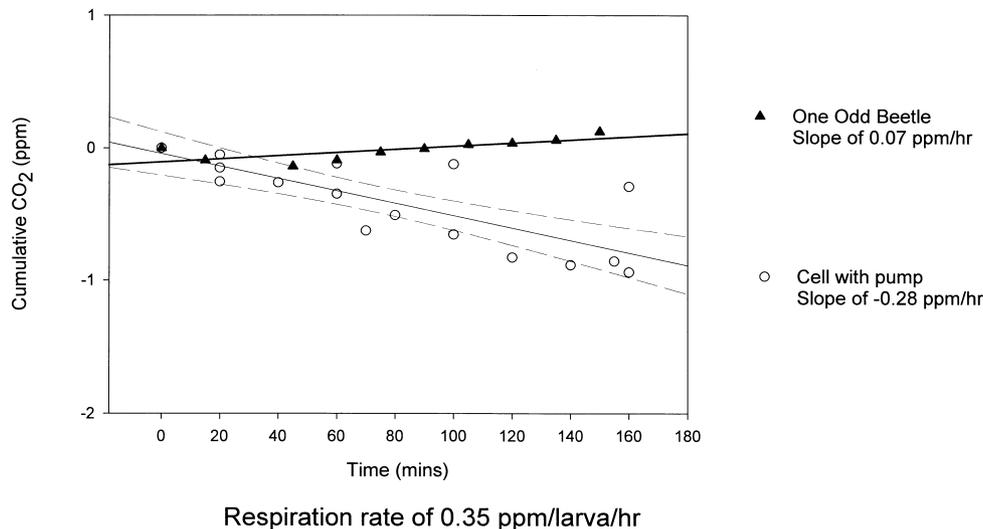
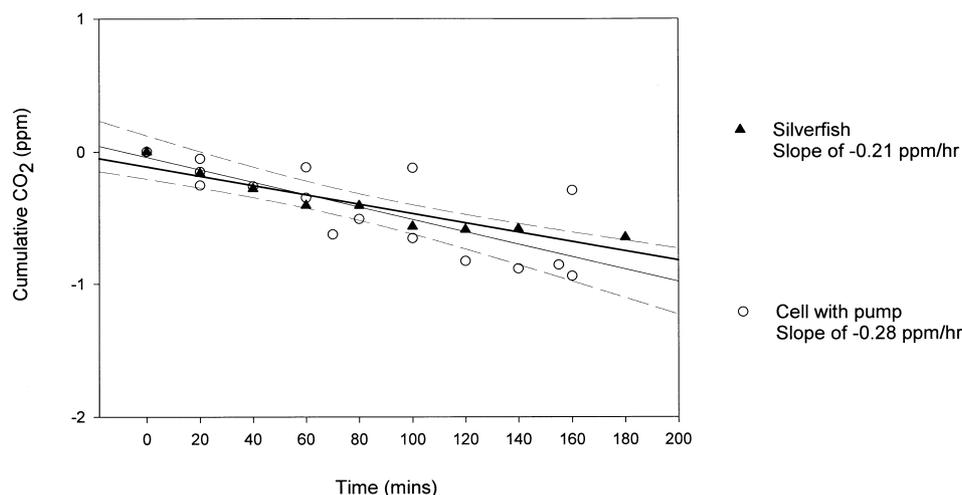


Fig. 8. CO₂ measurements using the LN₂-cooled-InSb system of one odd beetle larva (*Thylophorus* sp.). The respiration rate of one odd beetle larva is calculated to be 0.35 ppm/h. The larva is detectable within 40 min.

Indian bandoleer and 2 bow-cases composed of leather, fur, and textile; the respiration rate was 0.55 ppm/h. The insect attack on these objects was consistent with that caused by carpet beetle larvae. Fig. 11 shows measurements taken from a 20th Century wood frame. This frame had been monitored in the laboratory for several years for signs of activity. Observations had indicated that insect holes and frass were the result of past infestations that were no longer active. The

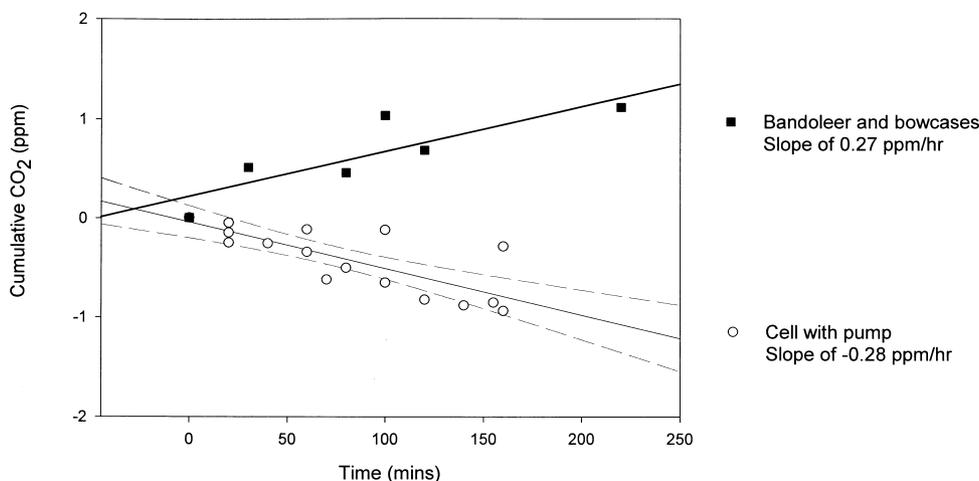
slope from the measurements of the frame was well within the 95% CI for the control means of the regression line, confirming the visual assessment.

Results gathered from the LN₂-cooled InSb FTIR system demonstrated clearly the ability of the system to reliably determine the presence and absence of an infestation within a few hours. This is significantly faster than the 40 or more hours necessary with the air-path system. In addition, the

CO₂ Measurements of an Adult Silverfish (*Lepisma* sp.)

Respiration rate of 0.07 ppm/adult silverfish/hr

Fig. 9. CO₂ using the LN₂-cooled-InSb system of a single adult silverfish (*Lepisma* sp.) falls within the 95% CI for the control line of the means.

CO₂ Measurements from a Bandoleer and Bowcases

Respiration rate of 0.55 ppm/hr

Fig. 10. CO₂ measurements using the LN₂-cooled-InSb system from a bandoleer and bow cases. The respiration rate is 0.55 ppm/h.

lower limit of detection of the LN₂-cooled system was 0.09 ppm/h vs. 0.3–0.4 for the air-path system.

The LN₂-cooled InSb FTIR system has become an important tool at the MMA for insect control efforts. While this system is much faster and more sensitive than the air-path system, in practice, it is necessary to test an object over 2 or 3 days to ensure reliability of the results. Further studies will concentrate on measuring respiration of insect eggs, testing larger bag volumes to determine the pump rate and collimator settings necessary to achieve satisfactory results, and testing the efficacy of an MCT detector in the FTIR to

collect methane data as a second method to measure for respiration activity and perhaps to aid in differentiating insect from microbial infestations.

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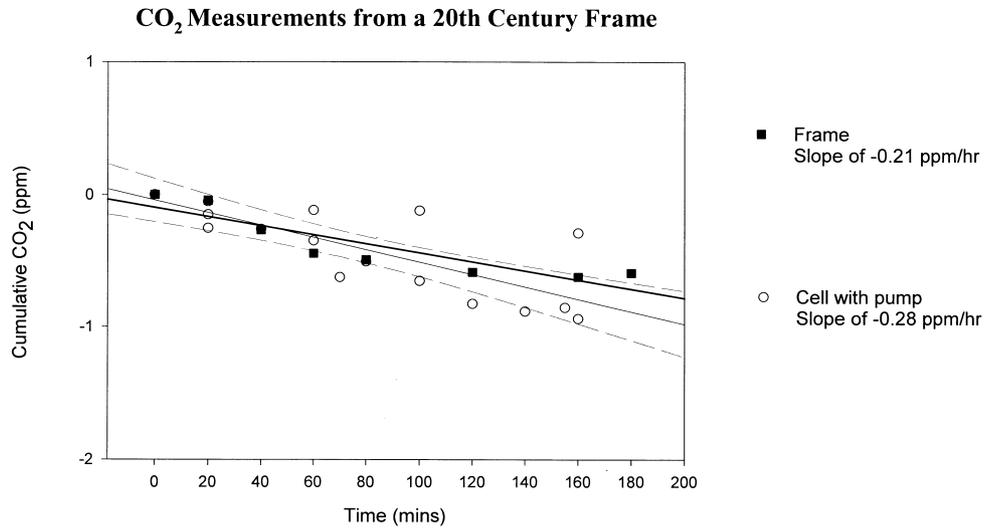


Fig. 11. CO₂ measurements using the LN₂-cooled-InSb system from a 20th Century wood frame. The slope for this object is well within the 95% CI for the control line of the means.

discussions concerning this project and for reading over the paper.

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