



Article: Dilemmas in transporting unstable ceramics: A look at cyclododecane

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DILEMMAS IN TRANSPORTING UNSTABLE CERAMICS: A LOOK AT CYCLODODECANE

Sara Caspi and Emily Kaplan

Introduction

The National Museum of the American Indian (NMAI) is in the process of transporting its collection of over 800,000 artifacts from the museum's Research Branch (RB) in the Bronx, NY, to the new Cultural Resources Center (CRC) in Suitland, MD. The move began in mid-1999 and is projected to take five years in all. This article focuses on research into the transport of a group of unstable ceramics, using cyclododecane as a temporary consolidant.

Background

In 1997, NMAI conservation staff began a collection-wide survey to assess conservation needs prior to the move. The conservators created a database that listed the condition of the objects in the collection with keywords, along with an indication of treatment priorities. Priority #1 was assigned to objects in need of stabilization before transport; #2 designated an object that was stable but needed re-evaluation after the move; and #3 was assigned to stable objects that could be cleaned and packed without further treatment. For ceramics, keywords included "efflorescence," "powdery paste," and "spalling surface." These conditions were suspected to be associated with a soluble salt problem, and these vessels were flagged as #1's - in need of stabilization before transport. As stabilizing these vessels was anticipated to be a time-consuming and complex process, the project was earmarked for Sara Caspi and Joanne Boyer, Andrew W. Mellon Fellows in Conservation who would be stationed at the RB starting in 1998.

The Project

All of the ceramic objects listed in the survey as having soluble salts and/or ceramic body instabilities were re-examined. In order for an object to be included in the project, it needed to satisfy two criteria: the presence of loose fragments that would be disturbed or lost in transit, and a problem with soluble salts. Objects that had salt efflorescence but no active ceramic body instabilities were not included in the project because they could be safely transported to the CRC without consolidation. Objects with loose areas that did not have an apparent soluble salt problem were not included because they could be stabilized by consolidation with a standard conservation material like Acryloid B72 and then safely transported to the CRC. Ceramics that met the two criteria but could be transported by special packing were also not included in the project.

The ceramics in the project

The final group of ceramics selected for the project included about thirty, out of the thousands in the museum's collection. Of this group, about a third were archaeological vessels, mostly from South America. The treatment approach towards the archeological vessels was relatively straightforward, based on accepted conservation methods of consolidation with Acryloid B72 followed by desalination; hence they could be made stable for transport without further investigation.

The 21 remaining ceramics were low-fired, hand-built vessels made by potters from the Pima, Maricopa, and Tohono O'odham tribes in Arizona; from the Cochiti, Santo Domingo, San Ildefonso, and San Juan pueblos in New Mexico; and from the Kawia tribe in California. While potters from these tribes use some similar clay preparation and manufacture techniques, there are significant variations, determined by local traditions, and local clay and temper resources.

All of the vessels are fragile and suffering from some combination of salt-related conditions including delamination, flaking, powdering, and subsequent loss, and sometimes deformation in the form of "blistering". All of the vessels test positive with micro-chemical tests to one or more soluble salts: chlorides, nitrates, and/or sulfates.

Sources of Soluble Salts

While the presence of soluble salts in the archaeological pots was most likely an artifact of burial and could be acceptably treated, the presence of soluble salts in the ethnographic pots presented different questions. The salts could have been introduced during fabrication or use and thus might constitute important information about the object and its makers. Alternatively, soluble salts could have been introduced during a previous treatment.

For example, soluble salts may be naturally present in the clay, and might not be removed during dry processing. Some potters do process using wet methods, which might help remove salt impurities (Blair and Blair 1986). Soluble salts may also be introduced as part of the temper, which can be volcanic ash, ground-up sherds, stone, pulverized bone, shell, and sand (Peterson 1997; Peterson 1977). Salts may have come from use: some vessels are used for water gathering and storage, and the water in the Southwest can be highly alkaline. Other vessels are used for cooking, and some, used to store grain and liquid, may be partially buried in the ground (Peterson 1997; Fontana 1962). In addition, some ceramic vessels are used ceremonially, which might introduce soluble salts.

Because the source and significance of the soluble salts were in question, we met with Dr. Bruce Bernstein, NMAI Assistant Director for Cultural Resources, who is a scholar of ethnographic ceramics from the North American southwest. He felt that desalination across the board for the vessels would be inappropriate. He suggested in-depth culture-by-culture field research into the

source of the salts, focusing on use and technology of manufacture.

As it turned out, this kind of research was not possible during the course of this project: time constraints of the move prohibited such a comprehensive study. And as a new museum, NMAI is still in the process of creating policy and procedures for consultations with Native Americans regarding conservation and research. We expect that there will be better possibilities of consultations with potters from these various communities of origin in the future. Therefore, we used secondary sources for background research and focused on finding a way to securely transport the objects without desalination. Specifically, we considered the feasibility of cyclododecane as a temporary consolidant to stabilize sensitive areas prior to travel.

Cyclododecane in Conservation

Cyclododecane has been attractive to conservators because of its ability to sublime: due to its high vapor pressure, cyclododecane goes directly from solid to vapor in the typical laboratory environment. In theory, the treatment leaves the substrate unchanged, and thus promises to be a self-reversing consolidant or barrier film that should not affect future treatment or analysis (Hangleiter et al. 1995; Jägers and Jägers 1999). Cyclododecane is a solid wax-like volatile cyclic alkane ($C_{12}H_{24}$) with a melting point between 58-61° C. It is applied in liquid form as a melt or in solution, and hardens upon cooling or upon evaporation of the solvent.

Hangleiter (1995) and Jägers first developed and tested the material, introducing it to the conservation field in 1995. The primary uses in conservation are stabilization during transport (Hangleiter et al. 1995; Hiby 1997; McGowan 2001); and local surface protection during various treatments (Hiby 1997; Hangleiter 1998a; Bruckle et al. 1999; Bandow 1999). It has been used on several substrates, including paper (Bandow 1999; Bruckle et al. 1999; Keynan and Eyb-Green 2000); textiles (Hiby 1997; Jägers 1996); tempera (Hiby 1997) and oil paintings (Hangleiter 1998b); and unbound earth sculptures (McGowan 2001). It has been used on architectural elements, including stone (Bruckle et al. 1999; Stein et al. 2000); terracotta (Bruckle et al. 1999); mortar (Hiby 1999); plaster (Hangleiter 1998b; Riedl and Hilbert 1998); wall paintings (Hangleiter 1998a; Hangleiter 1998b).

Readers are referred to the excellent recent articles (in English) by Bruckle et al. (1999) and Stein et al. (2000) in which the results of comprehensive literature searches are usefully summarized. The authors discuss the properties of cyclododecane, its various uses in conservation, and application methods. Cyclododecane does not work on all media and in all treatments, mandating careful testing before use. These articles present the results of several tests to assess the suitability of cyclododecane as a conservation material for several treatments. Bruckle et al. report test results of cyclododecane for two uses: first, as a temporary protective fixative for water-sensitive media on paper during aqueous treatments; and second, as a barrier film for taking molds from objects easily stained by silicone mold-making materials. Stein et al., in investigating cyclododecane as a temporary consolidant for stone, address aspects of

penetration into various substrates; sublimation rates; and purity.

Regarding application techniques, we agree with the findings of most of these writers. The melt produces a more homogenous and cohesive layer, while the cyclododecane film produced from solution is more crystalline (Bruckle et al. 1999; Hiby 1997; Stein et al. 2000). The substrate to which the melt is applied may be heated to increase the penetration of the cyclododecane, and deep penetration of the cyclododecane results in a longer sublimation time (Riedl and Hilbert 1998). This of course would not be appropriate for our ceramic vessels.

As our goal was to use cyclododecane to protect the fragile surfaces of the ceramics for the relatively short time it would take to pack, transport, unpack, rehouse, and shelve the vessels, there would be no advantage to deep penetration into the ceramic substrate nor to the resulting long sublimation time. We found that applying molten cyclododecane with a glass pipette was a gentle way to apply the consolidant locally to the most fragile areas without disturbing the powdering and flaking surfaces (Fig. 3).

Tests on mock-ups

We conducted several tests on mock-ups to evaluate application and packing methods. For one series of tests we applied cyclododecane onto fragmentary and powdered ceramic material. The tests were successful: there were no losses in transit and there was no apparent change after sublimation. Based on the success of these tests, we determined that cyclododecane would work as a temporary consolidant to allow for the transport of the rest of the ceramics in our project from the RB to the CRC.

When we saw that the mock-up tests succeeded, we consolidated, packed, and trucked one artifact from the RB to the CRC. The vessel was placed in a fume hood for the sublimation process at the CRC. After about one month the cyclododecane appeared to have completely sublimed. Fourteen months later, the vessel is, at least visually, unchanged (Figs. 4-6).

Residue

Product literature and most of the conservation literature indicate that cyclododecane leaves no residue behind after sublimation. For example, Stein et al. (2000) tested a batch for residues and reported that there were none. Hiby (1997), cautions that it is important to use only a very pure cyclododecane in order to avoid residues. Jägers (1999) notes that “a fundamental precondition for the application of these materials [volatile binding media] is their complete and absolutely residue-free evaporation. A complete evaporation is guaranteed only when the materials are free from impurities. For this reason only products of high purity have to be used.”

We received our first shipment of cyclododecane from Kremer Pigmente in May 2000, and

started experimenting with it to learn about its working properties and rate of sublimation. We dripped melted cyclododecane with a pipette onto glass microscope slides to assess determine the sublimation rate. Stein et al. (2000) used a similar procedure. The slides were left unenclosed in ambient conditions in the laboratory. In the meantime, however, our tests on mock-ups appeared successful (Figs. 1-2).

To our surprise and disappointment, approximately five months later we noticed a minute amount of material, visible only in raking light, remaining on the glass slides after what had appeared to be complete sublimation. Furthermore, we had begun to test a second group of mock-ups at about this time. These were salty brick fragments covered with small mounds of powdery dirt from outdoors. These tests were not as successful as the first group. While the dirt and bricks arrived safely with no losses during transit, a white crystalline material was clearly visible after sublimation on some of the samples. Tide lines from the cyclododecane were also evident. This material is removable from the surface of the ceramic (Figs. 7-9).

We then contacted distributors and manufacturers of cyclododecane to ask about possible residues. Georg Kremer (2001), of Kremer Pigments, the primary United States cyclododecane distributor for conservators, responded that residue had never been reported. On further discussion, he said that while cyclododecane itself leaves no residue, there might be by-products from synthesis. Hans Hangleiter (2001), the German conservator who has been working with cyclododecane since before 1995, told us that he is familiar with an impurity and a residue but that he did not think it would have any measurable effect on treated materials. Our attempts to obtain information from manufacturers were unsuccessful.

Analysis of the Residue

We put the transport of the unstable ceramics on hold while we had the residue analyzed. Samples of cyclododecane were analyzed independently with similar results. Samples analyzed included neat cyclododecane taken directly from three different 500 gram cans from Kremer; one sample from a batch of cyclododecane from Chem Service, and samples from the white crystalline loose layer left on the dirt-covered bricks. It is important to note that no impurities could be detected when solid cyclododecane crystals were analyzed. Other compounds could be detected only when the cyclododecane in the samples was heated to force sublimation.

We were able to have analyses done at three different laboratories. Jamie Martin at Orion Analytical in Williamstown, Massachusetts performed Fourier transform infrared micro spectrometry (FTIR); David Erhardt performed Gas Chromatography-Mass Spectrometry (GC/MS) and Walter Hopwood FTIR at the Smithsonian Center for Materials Research and Education; and Richard Newman at the Boston Museum of Fine Arts carried out GC/MS.

Results of GC/MS analyses done by Erhardt indicate the presence of very small amounts of residue consisting of compounds chemically close to cyclododecane but about twice its molecular weight:

dodecacyclododecane and hydroxydodecacyclododecane. In addition, traces of hydrocarbons such as cyclododecene, cyclododecane, cyclododecanone, and perhaps cyclododecanol were found. [1]

Richard Newman's GC/MS findings confirm those of Erhardt, with a few minor differences. Newman detected most of the same major compounds: cyclododecene, residual cyclododecane, cyclodecanone (probably), cyclododecanol, and a family of higher molecular weight compounds, including one that is probably bimolecular derivatives of cyclododecane. This compound was by far the major component in the residue found on the test bricks (Figs. 10-12). [2]

We have not yet quantified the amount of residual material: it is so small that our balances are not adequate. Erhardt, Hopwood, and Newman all thought it unlikely that the tiny amounts of this residue would be harmful to the ceramics. Erhardt and Hopwood (2001) concluded "Except for its persistence, residual material does not present a threat to substrates treated with the consolidant. For the most part, the residue consists of compounds chemically close to cyclododecane, but about twice its molecular weight, that are probably byproducts of the synthesis of cyclododecane. The percentage of residue is quite small."

The Industrial Literature

A search of the industrial and chemical literature indicates the possibility, perhaps the probability, of impurities. When cyclododecane is produced in industry, it is expected that by-products will also be present unless a purification process is used. Production can be fine-tuned to get the highest yield of pure cyclododecane.

For example, several patents and articles report various processes for the synthesis and purification of cyclododecane. The majority of the literature (Lee 1970; Levine 1972; Morikawa et al. 1972; Hanika 1983; Okamoto 1990) reviewed that included detailed information on the production of cyclododecane described a general process of trimerization of 1,3-butadiene to 1,5,9 cyclododecatriene followed by hydrogenation of 1,5,9 cyclododecatriene to produce cyclododecane. Catalysts, reagents, and processes used vary, as does the reported purity of the resulting cyclododecane. The identification of high boiling point impurities and low molecular weight impurities in cyclododecane are reported by others (Manfred 1971; Usova et al. 1975).

For example, Manfred (1971) specifically mentions cyclododecanone, cyclododecanol, their mixtures, 1,12-n dodecandi-acid, and cyclododecene as by-products of cyclododecane production from butadiene. A method of removing these impurities is discussed. Morikawa et al. (1972) describe a process of producing cyclododecane from butadiene. Part of the process involves the removal of low boiling point by-products such as dimers of butadiene and high boiling point by-products. The resulting cyclododecane is expected to have 99.6% purity.

Cyclododecanone and cyclododecanol are compounds known to be produced from cyclododecane through oxidation (Furusaki and Mitamoto 1993; Zeng and Cui 1993). The impurities seen during the production of cyclododecane are unreacted cyclododecadiene and its incomplete hydrogenated

products (cyclododecene and cyclododecadiene isomers) as well as cyclododecanone and cyclododecanol. These ketone and alcohol high-boiling impurities could be formed either from the autooxidation of cyclododecane or from the hydrogenation of cyclododecadiene that contains impurities. (Usova et al. 1975).

The production methods for the cyclododecane we purchased are proprietary and were therefore not divulged to us, but it is reasonable to conclude from the industrial literature that the cyclododecanol, cyclododecanone, and cyclododecene impurities found in the GC/MS analysis could be by-products from synthesis. The other compounds found in the GC/MS analysis are not specifically mentioned in the literature; however, it seems likely that they are also by-products of synthesis.

Conclusions

There is a real possibility of very small quantities of impurities in cyclododecane. However, we believe that for these ceramics, the quantities are too small to interfere with any future analysis or treatments such as desalination or consolidation. Cyclododecane has been an acceptable and useful product for this project, but we emphasize that of course it may not be suitable for other media.

As the chemists we consulted pointed out, how many of the materials that we have in our conservation laboratories are completely pure and residue-free? Other conservators have tested and used cyclododecane without having apparent impurities and subsequent residue problems. It seems likely that there are batch variations.

The white material that remains visible on some of the dirt-covered brick mock-ups needs further investigation. The residue is easily removed manually, or with solvents such as Xylene. This phenomenon has not occurred on any of the other mock-ups or any of the treated objects.

Over a period of approximately six months we treated 21 ceramics with cyclododecane. We packed and trucked the pots to Suitland, and without exception all arrived safely with no losses. The presence of cyclododecane on the fragile surfaces allowed us to handle the vessels much more easily in the process of unpacking, rehousing, and shelving in our compactor storage ranges. We are examining the objects periodically as the cyclododecane sublimates, and there are no signs of darkening or tide-lines. The pots are all rehoused now, so they can be handled in their storage mounts should they need to be moved again.

The vessels have been moved safely, but now remain in their respective original conditions, for example, one vessel exhibits ongoing loss of ceramic powder as the cyclododecane sublimates in storage. We expected this loss, as it had been occurring over the years prior to the temporary consolidation with cyclododecane.

Based on our findings, we would recommend that anyone interested in using cyclododecane should purchase it in advance, apply a thick layer onto a glass slide and wait for it to sublime. Of course, if access and resources allow, GC/MS analysis would be more precise. In addition, while we may not receive answers, efforts should be made to inquire of the suppliers and manufacturers about the production method and the likelihood of any possible impurities. And of course any information about residues and production is important to include in the treatment documentation.

Given the condition of our ceramics, the time constraints of our move, and our questions about the source of the soluble salts, we believe that using cyclododecane as a temporary consolidant was a good solution to our transport dilemma.

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Endnotes

1. From Erhardt and Hopwood (2001):

“A few flakes of one of the residues [from two brick samples and from Kremer can] prepared by E.K. were dissolved in methylene dichloride. Approximately two microliters of the resulting concentrated solution was injected into a gas chromatograph. The instrument used was a Hewlett-Packard 5890 with a 5972 mass selective detector. With injector temperature 250 C, DB-1 30 m X 0.25 mm column. Temperature was ramped from 50 to 340 C at 10 C deg/min. The interface was set at 340 C.

“In the residue, traces of cyclododecene, cyclododecane, cyclododecanone, and, perhaps, cyclododecanol were detected. About 83 percent of the area under the peaks of the total ion chromatogram (TIC) was attributed to dodecacyclododecane. A significant but lesser amount of a slightly higher boiling constituent, perhaps hydroxydodecacyclododecane, was detected. The hydroxy group on the side chain must be near the ring.

“Finally, trace amounts of three dodecacyclododecanols were detected. Our unconfirmed identifications are 1-dodecacyclododecan-1-ol, 1-dodecacyclododecan-2-ol, and hydroxydodecacyclododecan-2-ol (hydroxy near the ring).”

2. From Newman (2001):

“All three Kremer samples gave similar results (see the chromatograms). In the chromatograms, please note that there are various peaks, including a very large one at about 17.3 minutes, that are from contamination in the instrument. These do not interfere with any of the residual compounds. I have not labeled any of these contamination peaks. Major compounds detected: cyclododecene, residual cyclododecane, cyclodecanone (probably), cyclododecanol, and a family of higher molecular compounds (retention times ~ 17.5-18.5 minutes). Most of these higher MW compounds cannot be specifically identified, but one of the major ones (c. 18 minutes) has a mass spectrum that closely matches that of 'cyclododecane, bicyclo compound' in the Wiley MS library. This compound essentially consists of two cyclododecane molecules bonded together. The pattern of peaks in the 17.5-18.5 minute region is quite similar, but not absolutely identical, for all three Kremer samples. All of the Kremer samples were prepared for analysis by placing a fairly large crystal or cluster of crystals in a cleaned vial, heating the vial to just above the melting point of cyclododecane and leaving the vial in the heating block until virtually all of the visible liquid had evaporated. The residual material was then dissolved in methylene chloride for GC/MS analysis. If a lot of cyclododecane remained, the solution was evaporated to dryness again, left on the block for a while, and redissolved.

“The three brick residue samples gave fairly similar results to one another (see the chromatograms). The major compound by far was the compound identified in the Kremer raw material samples as 'cyclododecane, bicyclo compound.' The brick residues lack the family of other higher MW compounds that the Kremer residues contained (compare the detail chromatograms of the 18-20 minute region). The other compounds (lower MW) found by Erhardt and Hopwood in the brick residues from 2 and 3 seem to be present at lower levels (relative to this high MW compound) in the samples I analyzed than they were in the ones they analyzed. We used somewhat different GC/MS procedures, which could account for some of the differences. The compound that made up about 83% of the total peak area in the brick 3 residue analyzed by Erhardt and Hopwood is clearly the same compound that I found to be the major component in all three of brick residues (although the peak area it accounted for in my analyses would have been much higher than 83%). This is the compound that they identified as dodecacyclododecane. This compound would have the exact same composition as 'cyclododecane, bicyclo compound'. I am only relying on the MS library for the identification.

“The conclusion from my analyses is that the Kremer material contains only very small amounts of compounds other than cyclododecane. Some of this associated material is clearly higher MW material, probably essentially 'dimers'. Apparently quite a range of specific compounds is present in this higher MW group. The remainder of the associated material consists of 'monomers' of the types noted by Erhardt and Hopwood. For some reason, the brick residues are enriched in a single 'dimer' component. I don't know whether the evaporation procedure I used, which involved an elevated temperature, could

have somehow affected the composition (the heating temperature, however, was not that high -- between 60-70 C).

“One final note on the Kremer materials: one can also contained some fatty acids and hydrocarbons, in quite small amounts. I don't believe these are due to any contamination in my instrument, solvents, or sample vials.

“Re. the Chem Service cyclododecane. It gave a quite different result. It contained some of the same high MW compound (the 'dimer') and some of the same residual lower MW compounds, but the major compound detected in it fairly closely matches the reference mass spectrum for erucylamide, C₂₂H₄₃NO (MW 337). This particular compound is not present in any of the Kremer residues.” [Only one sample from Chem Service cyclododecane was analyzed.]

Suppliers

Kremer Pigments, Inc
228 Elizabeth St.
New York, NY 10012

Lancaster Synthesis, Inc.
P.O. Box 1000
Windham, NH 03087
603-889-3302

Chem Service, Inc.
600 Tower Lane
PO Box 599
West Chester, PA 19381-0599

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Figure 1. Cyclododecane applied by pipette in the melt on glass microscope slides.

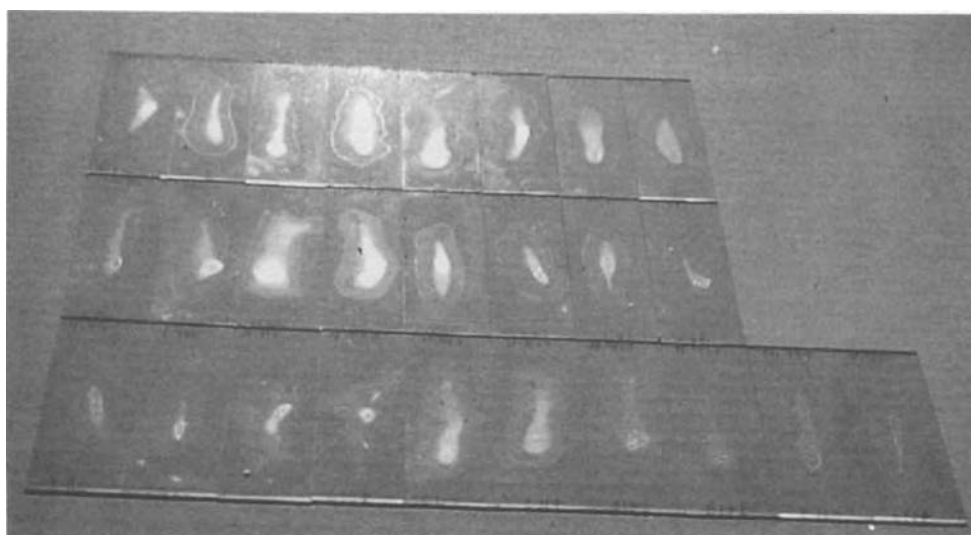


Figure 2. Cyclododecane residue on glass microscope slides, visible in raking light.

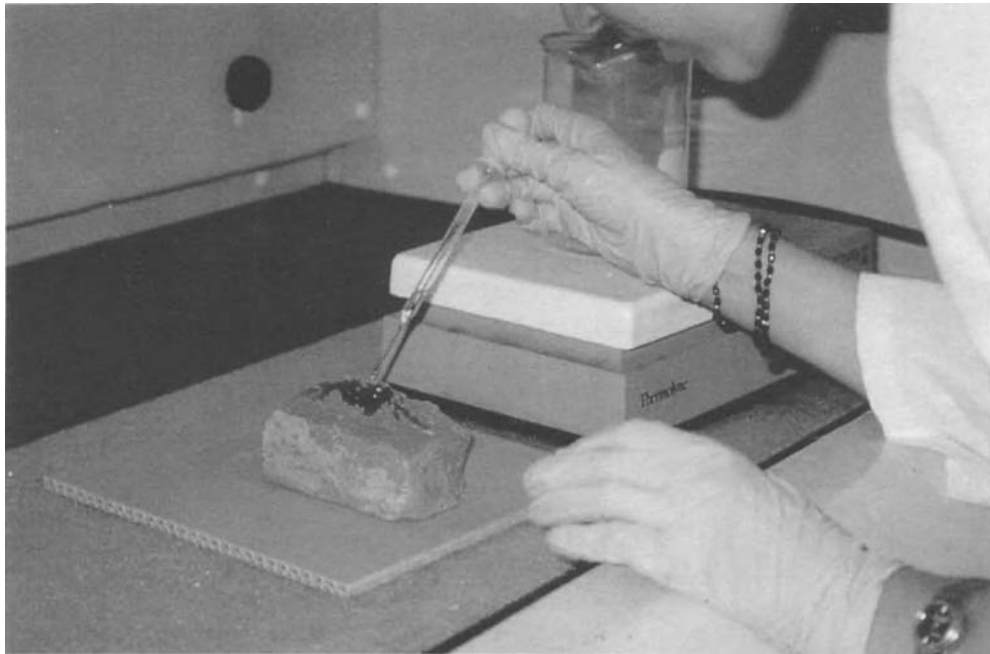


Figure 3. Applying molten cyclododecane by pipette to test brick sample.



Figure 4. Santo Domingo Pueblo pitcher (NMAI 050833.000). Before treatment.



Figure 5. Santo Domingo Pueblo pitcher (NMAI 050833.000). During treatment, cyclododecane partially sublimed.



Figure 6. Santo Domingo Pueblo pitcher (NMAI 050833.000). After treatment. The cyclododecane has sublimed completely.



Figure 7. Test Brick #4, cyclododecane newly applied.



Figure 8. Test Brick #4, after six months, cyclododecane partly sublimed.



Figure 9. Test Brick #4, after nine months, Residue from cyclododecane is visible.

Chromatograms from GC/MS analyses
of residues from Kremer cyclododecane

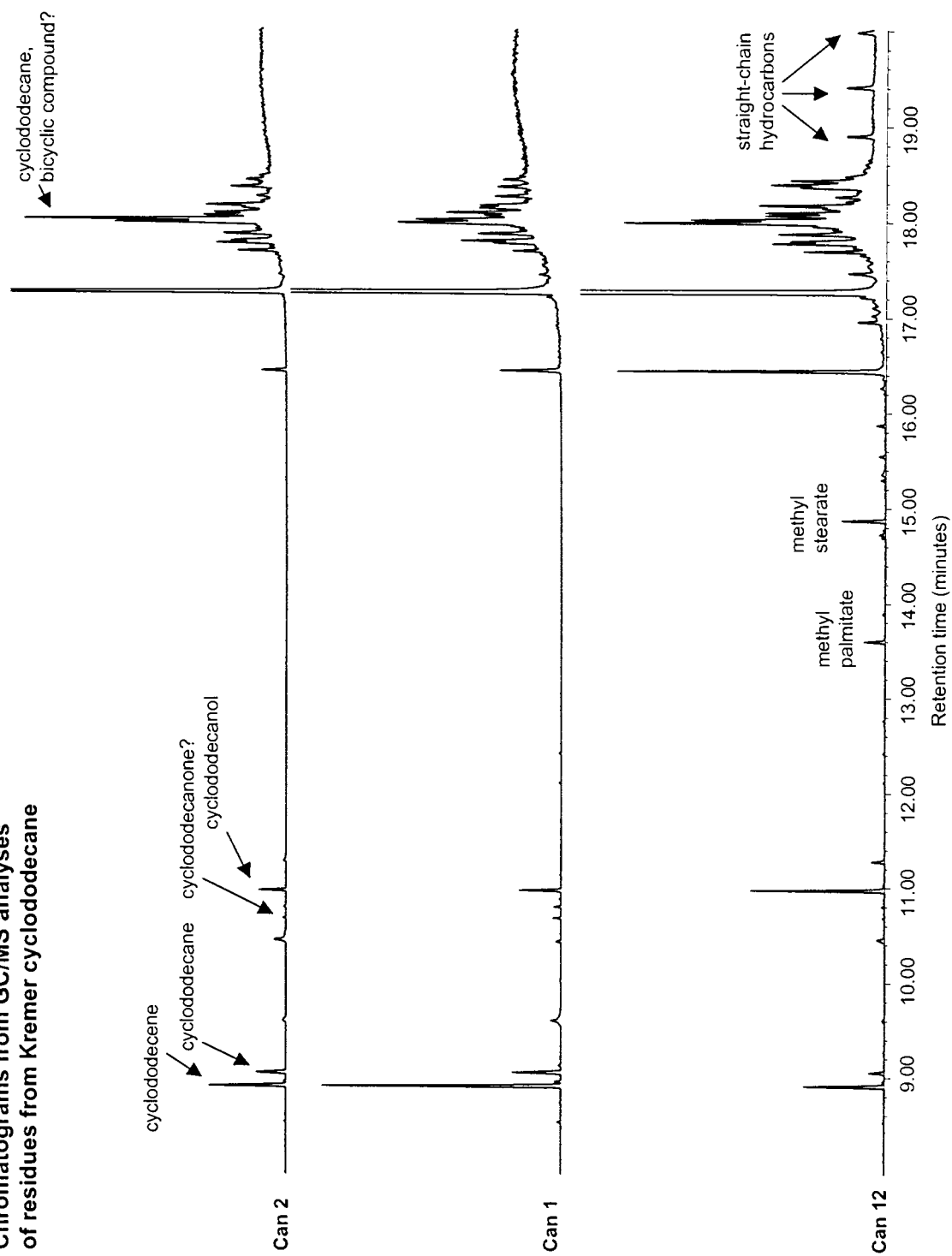


Figure 10. Chromatograms from GC/MS analyses of residues from Kremer cyclododecane (Newman 2001).

Chromatograms from GC/MS analyses
of residues from bricks treated with cyclododecane

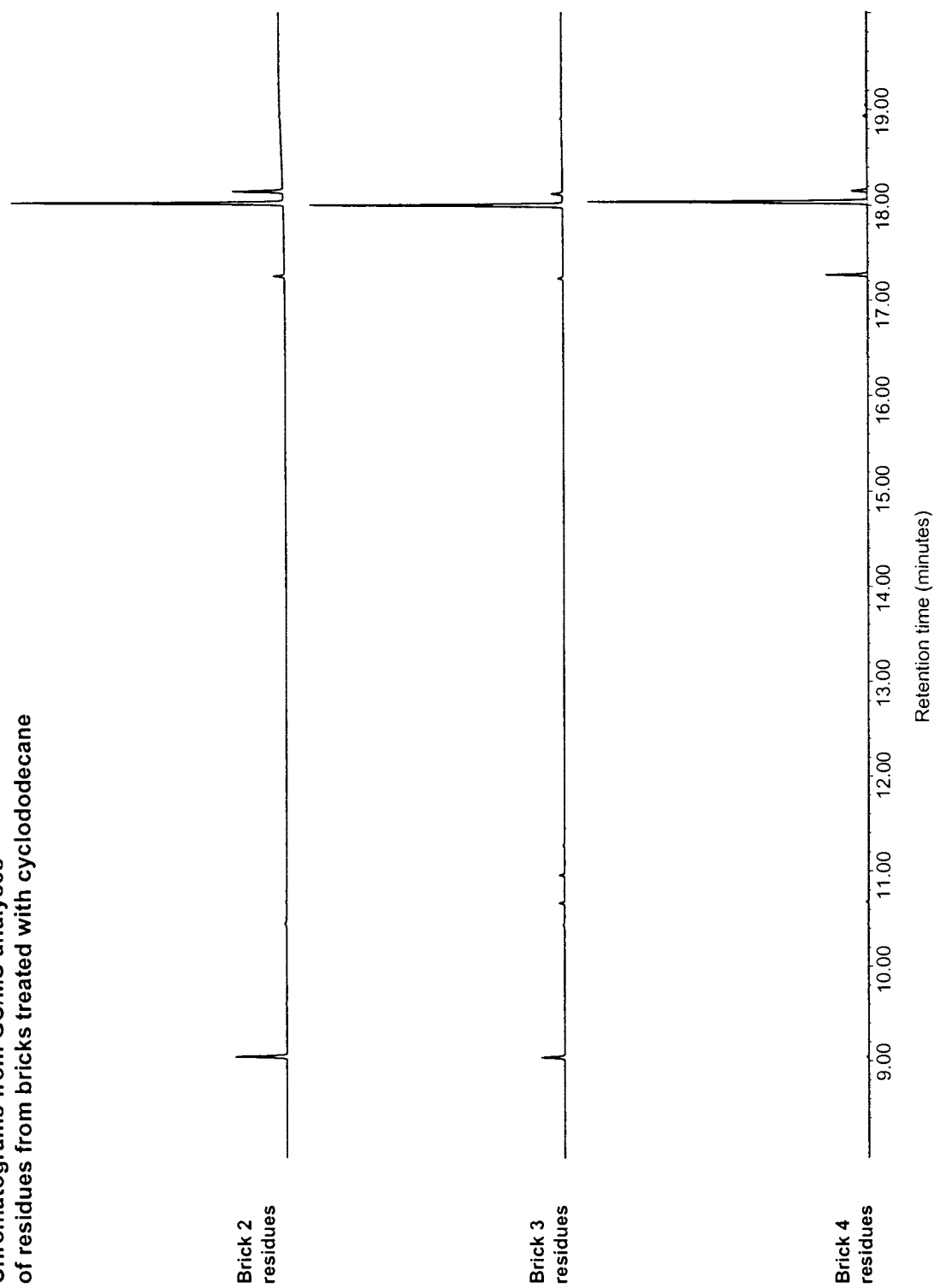


Figure 11. Chromatograms from GC/MS analyses of residues from test bricks #2, 3 and 4 (Newman 2001).

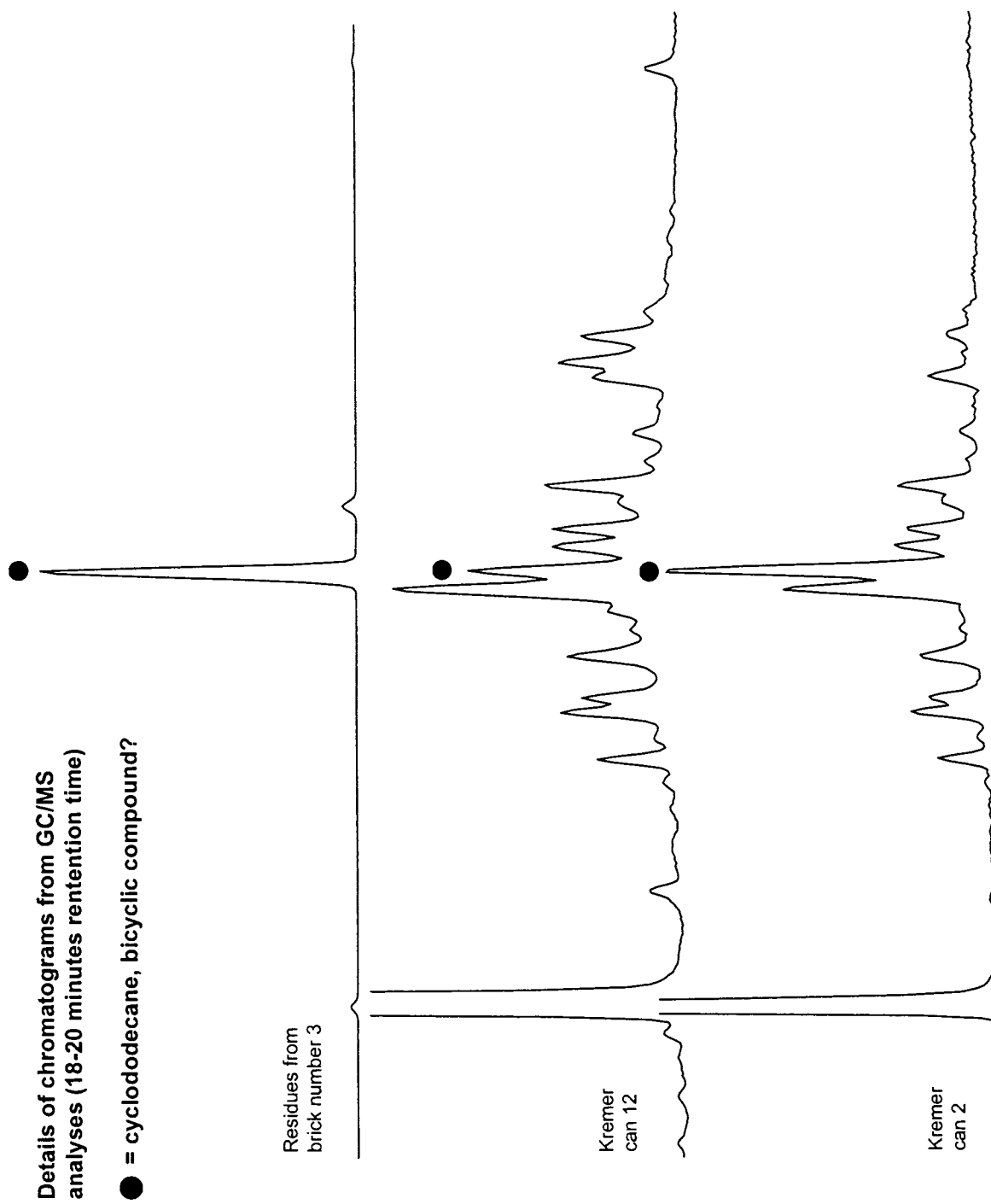


Figure 12. Details of chromatograms from GC/MS analysis (Newman 2001).