Archaeological block lifting with volatile binding media:
exploring alternatives to cyclododecane

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The goal: improving on an imperfect best practice

Cyclododecane (CDD) is the only volatile solid commonly used in archaeological conservation. It is considered the best consolidant for block lifting, a technique for stabilizing and reinforcing blocks of soil and fragile artefacts until they can be fully excavated in a lab setting (Cronyn 1990; Watters 2007). Traditional consolidants (water-soluble adhesives, acrylic resins, etc.) require solvents and mechanical action for removal and cannot be entirely extracted from the artefact (Watters 2007). CDD sublimes completely away. However, its slow sublimation rate sometimes prevents conservators from accessing the blocks within a suitable time frame, thus endangering the artefacts. In an effort to find an alternative to CDD, certain volatile binding media were compared to determine their potential usefulness for archaeological block lifting.

Figure 1. Application of CDD at Amarna, Egypt.

Criteria for new best option:

- T_m is under 65°C
- Immiscible with water
- Soluble in commonly used organic solvents
- Low toxicity
- Low impact on environment
  – Hangleiter, Jägers, and Jägers (1995)
- Commercially available to conservators
- Faster sublimation rate than CDD
- Physical characteristics suitable for consolidation

Block consolidation mock-ups for sublimation in field lab conditions

Materials test 1: sublimation from slides

- Each compound was melted and applied onto glass microscope slides at certain thicknesses. Mass Measurements were taken to track the rate of change in sublimation. Visual observations revealed behavioural phenomena of the different compounds. Residue analysis was conducted after sublimation.
- Samples of each VBM were prepared using a draw plate to 0.5 mm of VBM, 1.0 mm of VBM and a third sample set at 0.5 mm of VBM + two-ply gauze + additional VBM to seal in the gauze.

Figure 2. K. Langdon tracking mass changes.

Materials test 2: sublimation from jars

- The compounds were melted and poured into small, straight-walled jars to promote constant surface area during sublimation. Mass measurements and visual observations were made.
- Samples were made for each VBM with two methods of preparation. First, the VBM was allowed to cool completely without disruption. In the second set, the VBM was **agitated** (stirred with pins) during cooling to disrupt crystallization.

Simulated archaeological field lab (a very warm, ventilated space)

- The samples were left to sublime at an ambient temperature of 26-30 °C in a fume hood. Most trials ran for approximately one month.

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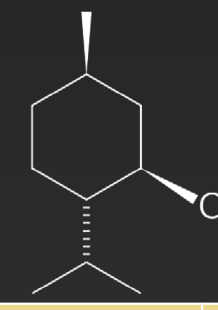
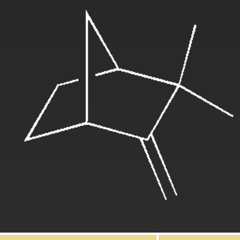
Cyclododecane (CDD)



L-menthol



Camphene

Source: Wikimedia Commons

Characteristics	CDD	L-Menthol	Camphene	65:35 mix CDD/menthol	90:10 mix CDD/menthol	95:5 mix CDD/menthol
Published T_m	61° C	42-45° C	48-52° C			
Published sublimation rate	0.03 mm/24 h	0.04 mm/24 h	0.4 mm/24 h			
Source for testing	Kremer Pigmente	Acros Organics, Fisher Scientific	Sigma-Aldrich			
Tested on slides	✓	✓	✓	✓		1 sample to confirm behaviour
Tested in jars	✓	✓			✓	✓
Notable behaviour & characteristics	Small, hard crystals, some long and thin. Shiny surface. Sublimation occurred fastest at grain boundaries with frequent small pits.	Large, dome-shaped crystals in both sets of tests. Matte surface. Sublimation occurred primarily at grain boundaries as few, large pits.	Sticky paste, never rigid. Sublimed rapidly, completely evolved within 5 days.	Melted at ambient temperature, ran off slide. As menthol evolved CDD formed dendritic crystals.	Similar in appearance and behaviour to CDD.	Similar in appearance and behaviour to CDD.
Residue description	Slides slightly clouded, barely enough to sample.	Slides slightly clouded.	Significant slick of sticky, clear residue.	 **Figure 3:** CDD dendrites from the 63:35 mixture.		
Residue identification with transmission FTIR microscopy	Wax: Most similar to candelilla wax (85% match).	Likely a natural gum. (Closest match 60%)	Natural resin: Most similar to dammar (86% match).			
Appropriate for consolidation?	✓	✓	✗	✗	✓	✓

Trends observed from sublimation tests

- On slides, where the VBM froze more rapidly and crystal formation was disrupted by the drawplate, the menthol predictably evaporated faster than CDD. The opposite was true in the jar tests, where **the crystal structure appeared to have a significant effect on sublimation**.
- In the jar tests, **constant surface area was not achieved because the grain structure largely determined the loci of fastest sublimation**. CDD (small, thin crystals) had pitting on the surface but also visible up to 1 cm below surface, as seen through the transparent glass walls of the jars. Menthol (large, dense crystals) had few large pits forming at exposed grain boundaries.
- In all cases the **VBM with embedded gauze took much longer to evolve** completely than those without gauze.
- Agitated samples (two CDD, two menthol, one of each mixture (95:5 and 90:10)) in jars were compared in a brief, 12-day trial. The data was insufficient to draw firm conclusions, but **a promising trend was observed**: the mixtures both sublimed at twice the rate of the CDD and the menthol, which had similar sublimation rates in this test.

References

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Conclusions

- **L-menthol fits the criteria but has low heat tolerance**, as shown by a field test by L. Skinner (author) in Egypt, where the menthol did not set firmly during consolidation. It is not suitable for use in warm climates but may be ideal in cooler locations.
- **Camphene is too soft and fast-subliming** for use in block lifting, and currently **cannot be purchased pure enough** for other conservation work.
- **CDD/menthol mixtures with a low amount of menthol may be effective** for increasing sublimation rate.

- **Residues** of even "conservation grade" compounds (CDD) are present. Therefore, VBM should be used only as necessary, but compared with the residue from traditional consolidants this has a negligible impact and **should not be a deterrent from using CDD in block lifting**. A standard for "conservation grade" menthol should be determined, at which purity it can be used in the same manner as CDD.
- In case of residues, **if radiocarbon dating** of the find is necessary, a **sample** should should be collected prior to consolidation with VBM derived from plant extracts (which currently include both menthol and camphene).
- **Gauze and porous objects** (such as a block of soil or organic artefacts) may significantly **prolong the sublimation period** (Han, Huang, and Luo 2013). This should be taken into account when predicting treatment schedules.
- **Sublimation time may be improved mechanically** during application of the VBM **by limiting crystal size and orderliness**. Crystal size can be minimised during solidification by keeping the block and VBM as cool as practically possible: the excavation should be shaded, and the molten VBM kept to a minimum workable temperature, close to its melting point. Application by brush rather than pour will disrupt crystals and create small air pockets. This may accelerate sublimation rates over time by exposing substantially more surface area and grain boundaries as sublimation progresses.

Future work

- Conduct further testing on CDD/menthol mixtures
- Determine whether solvent-based application of VBM can provide enough strength while decreasing grain size and increasing sublimation rates
- Search scientific literature for more volatile materials to bring to conservation
- Compare properties of DL-menthol and L-menthol
- Conduct hardness and adhesive strength tests to compare promising VBM and application methods
- Identify suitable chemical products available to conservators
  - Create list of suppliers and products
  - Identify sources for bulk purchases

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