Materials testing: Prior to conservation treatment of Stele 045 from Takeo, several formulations of bulked ethyl silicate grouts were prepared and evaluated for their working properties and performance by Kasey Hamilton (UCLA/Getty). Three different ethyl silicate consolidants were tested (Remmers KSE 510, KSE 300 (E?), and KSE 500 STE), with the addition of bulking agents (micronized silica, diatomaceous earth, fine stone powder, fine sand powder, glass and ceramic microballoons).

Remmers KSE 510 Trial I (11:30AM July 3 2018)

KSE 510 is a "classic" stone strengthener with ~42% gel deposit rate, free of additional solvents.

Volumes were measured w/ syringe, mixed with wooden tongue depressor

Volume KSE 510 (mL)	Filler	Working properties	After 24 hours
4	None (control)	Very fluid, easily injectable	No cracking, flowed around edges of plastic container
4	Silica – 1 scoop	Too viscous. More like a gel. Quicker to set.	Severe cracking, shrinkage
4	Silica – ½ scoop	Still v. fluid	*
4	Silica – ¾ scoop	Max amount dry silica to be added while still remaining flowable/injectable	Moderate cracking (three large radial cracks) Cracks run through to bottom of the film. Broken when removed from cup.
4	Silica – ¼ scoop Fine stone powder – ¼ scoop	Stone powder settles at the bottom. Not fine enough. May be possible to sieve to make finer. 2 runs completed – one where stone was added first, another siica added first. Seems to work better to mix in stone first, then silica.	Glossy top layer, bottom layer matte and more concentrated with the stone powder.
4	Silica – ¼ scoop Fine sand – ¼ scoop	Sand settles instantly. Does not mix well with consolidant.	Glossy top layer, bottom layer matte and more concentrated with the sand powder.

* additional silica added to reach ¾ scoop, drying behavior not observed

General note: silica is somewhat clumpy, likely already has high moisture content. Causes clumps of more quickly reacted ethyl silicate. Would be more ideal to use dry silica.

After 48 hours, all samples showed significant cracking. The control is the most intact – but crizzling and shrinkage/warping is observed.

Remmers KSE "300 STE" Trial I

300 STE does not exist – the product was undoubtedly mis-labeled. This is either 300, 300 E, or 500 STE, but cannot be confirmed at this time.

Volume KSE 300 (mL)	Filler	Working properties	After 24 hours
2	None (control)	Very fluid, easily injectable	Severe cracking
2	Silica – ½ scoop	Mixed well, still fluid (~1/4 added and mixed, followed by remainder)	Severe cracking
6	None – to test thickness	Very fluid	Still very severe cracking, very brittle.

Cracking and poor performance may be related to fast hydrolysis, or thickness of film (too thin)

Remmers technical sheet advises use of 300 for only areas up to 10 microns thick. For anything larger, an elasticized product must be used.

<u>Remmers KSE 500 (STE)</u> Trial I

The product is labeled "500" – which again, does not exist. The product is either 500 E or 500 STE. The product was obtained from GACP. According to Hans Leisen (email correspondence), GACP uses only 500 STE for repair mortars, they do not carry 500 E. Therefore, this product is likely 500 STE.

500 STE is an elasticized stone strengthener with a gel deposit rate of ~50%. It contains suspended mineral particles/fillers.

Volume KSE 500 (mL)	Filler	Working properties	Curing behavior
4	None (control)	Very fluid, easily injectable	No cracking. Still a little soft – not completely set (after 24 hrs).
4	Silica – ½ scoop	Mixed well, still fluid (~1/4 added and mixed, followed by remainder)	No cracking. Still a little soft. (after 24 hrs)
6	Silica – ¾ scoop (same rough proportions as other)	Mixed well, still fluid (~1/4 added and mixed, followed by remainder)	No cracking. Still a little soft (after 24 hrs).
4	Diatomaceous earth – ½ scoop	Appears to mix well at first but settles on bottom.	Mostly cohesive, but two large cracks (after the weekend).
4	Diatomaceous earth – ¼ scoop Silica – ¼ scoop	Appears to mix well at first but DE settles on bottom.	Completely cohesive (after 72 hrs) some shrinkage noted around edges
4	Stone powder – ¼ scoop Silica – ½ scoop	Stone powder settles on bottom.	Some cracking and shrinkage. Stone powder settles at bottom.
6	Silica – approx. ¼ scoop Ceramic microballoons - approx. ¼ scoop	Microballooons added first. Appears to mix well, despite silica still being clumpy. Grey color overall, suitable for stone applications	No cracking or shrinkage observed after 24 hours
6	Silica – approx. ¼ scoop Glass microballoons - approx. ¼ scoop	Microballooons added first. Appears to mix well, despite silica still being clumpy. Cloudy	Still weak. Some cracking observed. Opaque white in color.

		white overall, color could be improved with addition of dry pigments.	
8-10	3:1:1 Sandstone powder:sand:lime	Color very dark Lime not necessary for this formulation	Very weak, significant cracking and shrinkage
3	2:1 sandstone powder : sand (1/2 scoop sandstone powder, ¼ sand)	More gray in color Some settling, still fluid	Good results – very little cracking
3	4:2:1 sandstone powder : sand : ceramic microballoons*	Sandstone powder and sand settle at bottom when left for too long, while microballoons float due to their low density – need to mix continually or use in small volumes	Slightly lighter in color More lightweight

Much more cohesive – silica also seemed to mix better. A thick (5 mm) sample was made to compare. Most likely KSE 500 STE.

From Remmer's technical sheet: "Classic" stone strengtheners (e.g. non-elasticized formulations) are brittle in character – medium sized silica gel sheets ~10 microns max. are formed. Not suitable for hollow spaces larger than 10 microns.

From Wheeler, Alkoxysilanes in Conservation – dab surface with paper towels, cloth rags to removed excess consolidant. Application of volatile solvents (acetone/EtOH) assists removal.

4:2:1 sandstone : sand : CMB was selected for treatment.

*Ceramic microballoons -- From Fillite technical sheet:
"Fillite is a glass hard, inert, hollow silicate sphere. Fillite is primarily used to reduce the weight of plastics, rubbers, resins, cement, etc., but also imparts further benefits in many situations. Many of the advantages from the use of Fillite, including increased filler loading and improved rheology, are directly attributed to the spherical nature of the material."

Shell composition: 27-33% Al_2O_3, 55-65% SiO_2, <4% Fe_2O_3
Average particle density: 0.85-0.95 g/cc.
Particle size: between 5-500 microns (see technical sheet for further details on distribution of particle size).

<u>Remmers KSE 500 (STE)</u> Trial II – Slow setting times

In this trial, setting times were controlled by lightly covering the sample cups with a plastic lid. This was done due to suspicion of fast hydrolysis occurring in the above trials (which had mostly hardened in 1 day), weakening the gel formed.

Trials were conducted both within plastic sample cups, as well as directly on the surface of a piece of sandstone. A small amount of consolidant/fill material was deposited into naturally occurring depressions in the stone, and loosely covered with a plastic lid.

In cups

Volume KSE 500 (mL)	Filler	Working properties	After setting
~10	Ceramic microballoons + fine stone powder (~1:1, 1-2 scoops each)	Microballoons mix in well, tend to float if not mixed, but appear to be well distributed once the ethyl silicate begins to set. Sand powder was obtained from freshly drilled hole in pedestal of Rama. Still injectable, good consistency.	Much slower to set (likely due to volume/thickness) – beads of liquid on top. Good cohesion, minimal cracking, very little shrinkage.
6	Silica – ½ scoop	Still a bit clumpy. still fluid (~1/4 added and mixed, followed by remainder)	No cracking. Still a little soft. Becomes more opaque and brittle as it continues to harden – eventually some cracking observed. Inconsistent color – some areas appear to be more concentrated in silica than others (related to the silica not mixing in well)
3	2:1 sandstone powder : sand (1/2 scoop sandstone powder, ¼ sand)	Some settling, still quite fluid	Dark grey in color. A bit of warping/curling. Minor cracking but otherwise appears pretty cohesive. Definitely a difference between appearance in bottom & top layers related to settling, but may not be a problem in practice (liquid phase absorbed by stone)
3	1:1:1 sandstone powder : sand : ceramic microballoons (1/3 scoop each)	Some settling, a little bit too thick (better to have less than 1/3 scoop of each). Somewhere between ¾ -1 scoop of dry material seems to be the ideal	Lighter sandy grey in color than the above trial. Remains flat in cup – no significant warping or shrinkage observed. Moderate cracking observed (more than above formulation). May be related to too much filler in the formulation.

On stone – KSE 500 STE + fill material

Filler	Working properties	After setting
Ceramic microballoons	Flowability good Significant staining / darkening around the area treated	Cracking, poor adhesion to stone
Ceramic microballoons + silica	Flowability good Still some staining / darkening, but not as severe as above	Cracking, poor adhesion to stone
Sandstone powder + sand	Very fluid, significant staining/darkening	No cracking observed after several days, very thin layer over stone, darkening remains
Sandstone powder + sand + ceramic microballoons	Flowability good, light beige/grey in color, some staining of surrounding stone	Hard, cohesive, good adhesion to stone, no cracking observed