D2.3 Protocols for Early Assessment of Individual Objects

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GLOSSARY

D	Deliverable
DoA	Description of the Action
DRIFTS	Diffuse Reflectance Infrared Fourier Transform Spectroscopy
ECOS	Environmental Control of Salts
FTIR	Fourier Transform Infrared
IPR	Intellectual Property Rights
KIK-IRPA	Koninklijk Instituut voor het Kunstapatrimonium (Dutch) Institut royal du Patrimoine artistique (French) Royal Institute for Cultural Heritage (English)
PREDICT project	Phase Transitions of Salts Under Changing Climatic Conditions (KIK-IRPA)
PTFE	Polytetrafluoroethylene
RH	Relative Humidity
WP	Work Package
WPL	Work Package Leader

TABLE OF CONTENTS

Executive summary

Preventive conservation is presently applied considering material groups, assuming they react identically. There is often very large difference in reactivity across a material group. For example, some glasses are stable to high RH values whilst others rapidly deteriorate at even 50%. This document provides the analytical methods for stone, ceramic and glass that can be used to identify and classify materials into stable and less stable groups, following extensive investigations.

For stone materials (section 2.1), the first method describes FTIR analysis to identify the swelling clays that may be present in the objects. The subsequent methods provide the steps for the identification of soluble salts using a poultice technique, and two possible analytical techniques (ion chromatography and specific ion meters). The poulticing technique has also been adapted and described for ceramic objects (section 2.2). The methodologies to assess early stages of glass degradation (section 2.3) using a swabbing method, ion chromatography and specific ion meters is provided in the subsequent section.

The final section (section 2.4) contains the instructions to correct the ion chromatography data for gypsum to allow for input into ECOS Runsalt Software v1.9. ECOS was the European project that developed the thermodynamic modelling. Runsalt is the program that allows users to input their salt data. Three possible methods are described for accessibility. The ECOS Runsalt software allows users to plot the data at different temperatures and relative humidities. This information can be used to predict the regions of risk for each salt present in the material. The final instructions provide detailed information on how to create a graph in Microsoft Excel that displays the regions of risk for salts and the environmental data from a site. This allows users to determine whether additional environmental controls are necessary due to the amount of time the environmental data falls within the regions of risk.

As this document presents methodologies only, there are no conclusions to present. The dissemination and subsequent use of these methods contributes to the development of green preventive conservation practices by providing conservators with information on the vulnerability of objects to different environmental conditions and the degree to which environmental controls are required.

List of contributors

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1. Introduction

This document outlines the different methodologies to assess material vulnerability for stone, ceramic and glass objects. There are also further instructions for creating graphs for data interpretation using certain software. Soluble salts make ceramic and stone objects more sensitive and vulnerable to fluctuations in temperature and relative humidity (Bradley and Middleton, 1988). Identifying and quantifying the salts present in these object types allows for the identification of stone and ceramic objects more at risk (Thickett, 2023; Thickett et al., 2023b). About 80-90% of glass objects react minimally to ambient environments and need little environmental control. A small proportion undergo rapid decay under ambient conditions and need to be in stable, low RH environments to survive (Thickett et al., 2022; Thickett and Ling, 2022). Glasses generate sodium and potassium ions on their surface during the decay process. Quantification of these ions gives a measure of the deterioration rate of the glass (Verhaar et al., 2020).

The methods to assess individual objects stability are designed for use by conservators, as well as scientists. The stone and ceramics methods detect species present, whilst the glass method estimates the deterioration rate. Several compromises (quality of water, extraction processes, blanks, replicates, error calculations)have been made in the methods from those in analytical laboratories. However, the accuracy is thought to be sufficient for the determinations. The methodologies are represented in the schematic below.

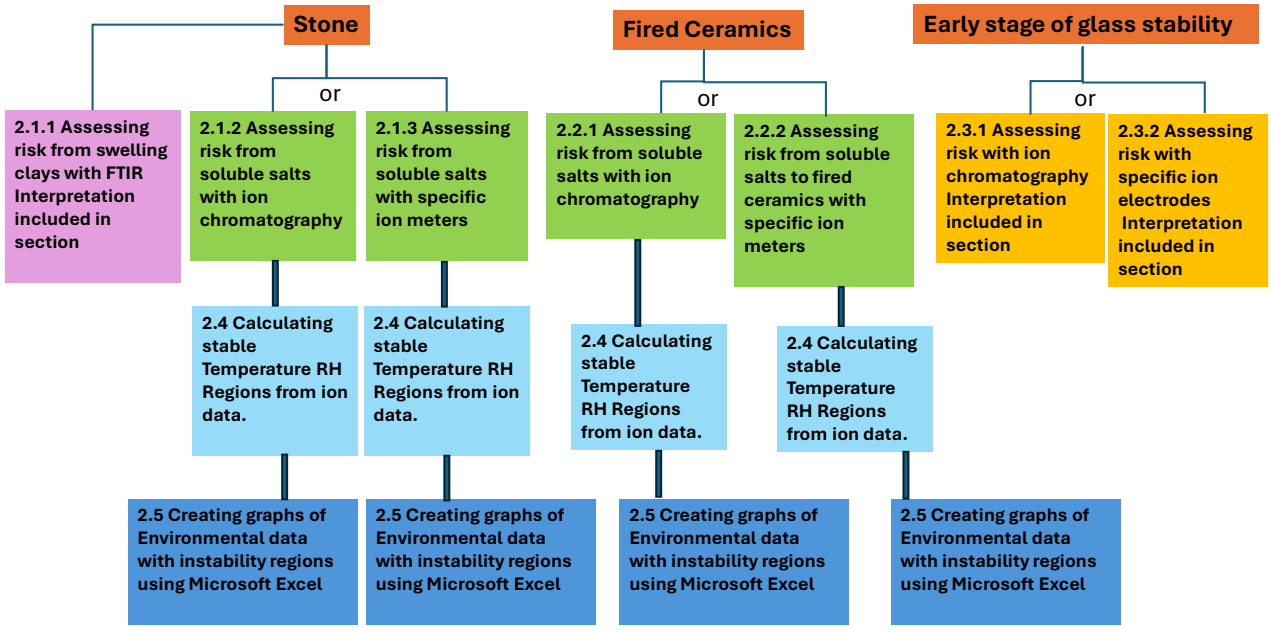

Figure 1. Schematic representation of work flow for different materials and methodologies.

The identification of more vulnerable objects allows for more tailored preventive conservation measures to be developed for those objects, rather than applying general environmental controls for large numbers of objects (Thickett et al., 2023a, 2023b). This approach allows for less energy-intensive and sustainable applications of environmental control. The major limitation on this approach is the remaining uncertainty about what exact environmental conditions are required, especially for glass and clays in stone and disagreement between authors.

2. Main body of report

2.1 Stone

2.1.1 Assessing risk from swelling clays in stone with FTIR

Before poulticing the stone to assess risk from soluble salts or to assess environmental risk, it is useful to assess if swelling clays are present. Some FTIR instruments allow for external analysis and are portable. For example, FTIR instruments that detect in a non-invasive way on the object are the Bruker Alpha with external reflection or Agilent 1400 FTIR with DRIFTS. Calibrations with physical samples have been developed for some clay types on Bruker Alpha external reflection (Thickett 2023, Thickett et al 2023a).

The common swelling clays problematic in stones are kaolinites, illites, muscovites, sepiolite and palygorskites (Środoń, 2006). The characteristic peaks are shown in the Table **1** below:

Clay	**Indicative characteristic peaks (wavenumbers)**				**Main peak**	**Factor (see page after next for description)**	**Indicative baseline start**	**Indicative baseline end**
Kaolinite	3619	3650	3675	3685	3685	0.967	3650	3710
Illite	3620	3699			3620	ND		
Muscovite	3264	3435			3624	0.030	3510	3720
Sepiolite	3550	3441	3687		3550	0.854	3450	3700
Palygorskite	3550	3416	3614		3416	0.654	2800	3800

Table 1. Clay characteristic peaks and characteristics. Factor is the multiplication factor for the peak height to determine the amount of clay present.

A larger analytical area helps with gaining representative spectra, but smaller areas cope better with stone curvature. The distribution of clay in limestones and sandstones can be very variable. In the absence of other analytical techniques, three analyses are recommended for the Bruker Alpha, and nine analyses are recommended for the Agilent 1400 system. The area with the highest clay content i.e. the spectrum with the highest swelling clay peaks, is the maximum risk. Any area can cause damage.

Collect a spectrum from the surface. A minimum of 16 scans should be used, covering the range 4000-1000 cm^{-1} wavenumbers.

Zoom up the 3000 to 4000 cm^{-1} wavenumber region and inspect for the characteristic peaks. If the peak shape is normal (see below), then the calibrations can be used.

FTIR peaks are usually symmetrical in shape, resembling a normal bell curve, with a peak maximum and equal wings on each side. Deviations in band symmetry, such as a slight shoulder or an unusual tail, often indicate the presence of an overlapping band.

Derivative peaks are not symmetrical and usually show a negative dip on the high wavenumber side of the peak. The apparent peak maximum will be at a higher wavenumber value than the unaffected peak.

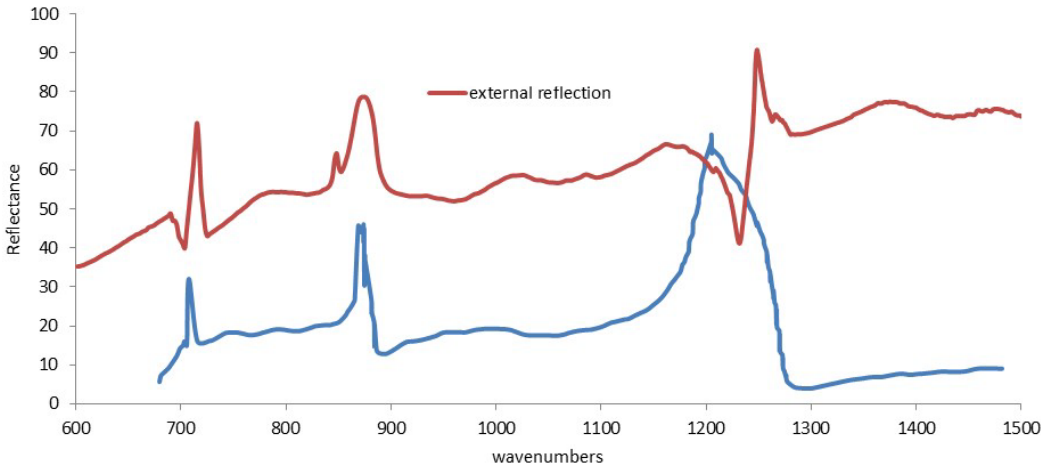

Figure 2. FTIR spectra of clay. Red by Bruker Alpha with external reflection, blue for Agilent 1400 with DRIFTs
Blue spectrum shows normal peaks, but those at 1200 and 870cm^{-1} are distorted. The red spectrum has derivative peaks at the same positions.

If the peak shape is considered normal, quantification can be attempted. Set the baseline start and end. The table start and end values are only a guide and may need to be moved to values with minimum absorbance. The correct placement of baselines is illustrated below.

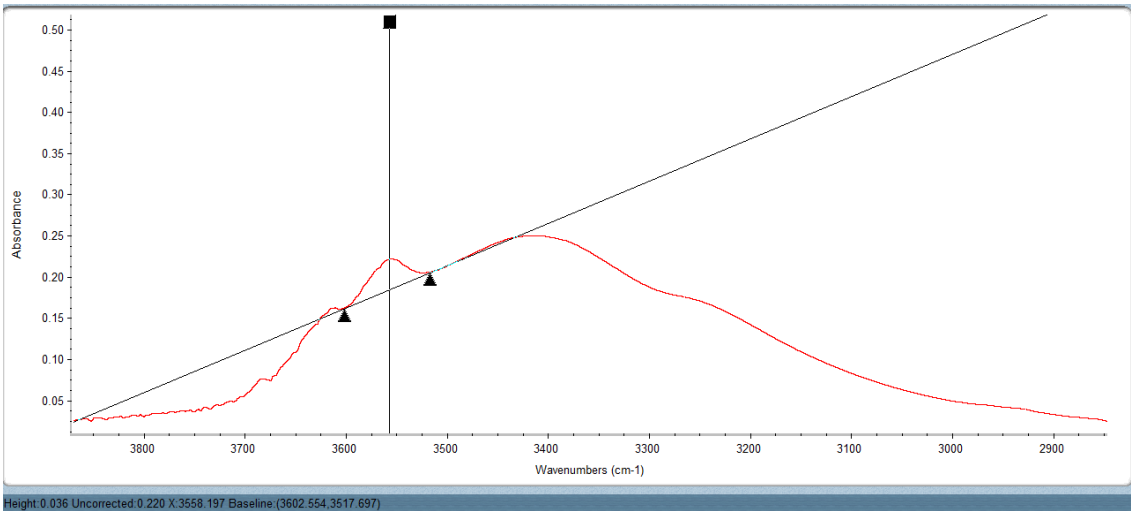

Figure 3. Expanded region of clay FTIR spectrum showing main peak and baseline.

Absorbed water complicates the analysis, causing the large, broad peak at 3430cm^{-1}. If the water content is too high, the clay peaks can be obscured. For sepiolite in limestone, a water content of 6% has been found to be the tolerable maximum for this analysis.

Read the corrected height of the peak from the spectrum. The approximate amount of clay can be calculated by:

Approximate amount (% by weight) = corrected peak height x factor (for clay type)

The presence of any swelling clay indicates a risk. It has been found empirically that clay contents above 5% by weight contribute to stone deterioration.

2.1.2 Assessing risk from soluble salts to stone objects with ion chromatography

This method is not suitable for pigmented objects or those with friable surfaces. A conservator should assess the objects to be tested for water tolerance.

There is a wide range of ion chromatography equipment, columns and conditions available. The below methods should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted.

1. Conservator assessment of the object to ensure it can withstand poulticing.

2. A poultice should be formed of three layers of Whatman's number 1 filter paper. The paper should be cut into 5x5cm pieces and soaked with 5ml of deionised or distilled water. Using a larger area poultice will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this area will be adequate.

3. The poultice is placed on the stone surface. If there is doubt about water tolerance, this should be in a discrete area.

4. Allow the poultice to dry for several hours depending on room conditions. See the Table **2** below:

Relative humidity	**50%**	**60%**	**70%**	**80%**
Temperature				
10°C	5.5	6.8	9.3	15.8
20°C	4.7	6.0	7.0	12.8
30°C	2.7	3.1	3.3	4.8

Table 2. Drying time (in hours) of poultice at different temperatures and humidities.

Important notice on Table **2**: Do not allow to dry beyond twice these times.

5. Carefully remove the dried poultice. Place poultice in an oven at 110°C for 4 hours.

6. Extract the salts from the poultice in 5ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this volume will be adequate.

7. Filter through a 0.45µm PTFE filter.

8. Submit the filtrate for ion chromatography analysis for sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ion concentrations.

9. Calculate the ion concentration in micrograms per centimeter squared:

> *If sodium concentration is 5.2 parts per million. The amount of sodium in the extraction water is 5.2x5 micrograms. The concentration per area is 26 / (5x5) = 1.04 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient.*

10. Use the Runsalt program as detailed in section 2.4 to calculate the high and low risk temperature, RH regions with the ion concentration calculated.

2.1.3 Assessing risk from soluble salts to stone objects with specific ion meters

This method is not suitable for pigmented objects or objects with friable surfaces. A conservator should assess the objects to be tested for water tolerance.

There is a wide range of specific ion electrodes available. Sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ions should be analysed. The below methods should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted.

1. Conservator assessment of the object to ensure can withstand poulticing.

2. A poultice should be formed of three layers of Whatman's number 1 filter paper. The paper should be cut into 10x10cm pieces and soaked with 20ml of deionised or distilled water. Using a larger area poultice will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this area will be adequate.

3. The poultice is placed on the stone surface. If there is doubt about water tolerance, this should be in a discrete area, and the surface should be frequently tested for any softening.

4. Allow the poultice to dry for several hours depending on room conditions. See the table below:

Relative humidity	**50%**	**60%**	**70%**	**80%**
Temperature				
10°C	5.5	6.8	9.3	15.8
20°C	4.7	6.0	7.0	12.8
30°C	2.7	3.1	3.3	4.8

Table 2. Drying time (in hours) of poultice at different temperatures and humidities.

Important notice on Table **2**: Do not allow to dry beyond twice these times.

5. Carefully remove the dried poultice. Place poultice in an oven at 110°C for 4 hours.

6. Extract the salts from the poultice in 70ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this volume will be adequate.

7. Separate the solution into seven sets of 10ml. Add ion adjustors as per manufacturer's instructions. Analyse the extracts for sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ion concentrations.

8. Calculate the ion concentration in micrograms per centimeter squared:

> *If sodium concentration is 5.2 parts per million. The amount of sodium in the extraction water is 5.2x70 micrograms. The concentration per area is 364 / (10x10) = 3.64 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient.*

9. Use the Runsalt program as detailed in section 2.4 to calculate the high and low risk temperature, RH regions with the ion concentration calculated.

2.2 Ceramic

2.2.1 Assessing risk from soluble salts to fired ceramics with ion chromatography

This method is not suitable for: pigmented ceramics, ceramics fired to low temperatures, ceramics with friable surfaces.

Many earlier ceramics have only been fired to low temperatures and will be adversely affected by water. A conservator should assess the objects to be tested for water tolerance.

There is a wide range of ion chromatography equipment, columns and conditions available. The below methods should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted.

1. Conservator assessment of the object to ensure can withstand poulticing.

2. A poultice should be formed of three layers of Whatman's number 1 filter paper. The paper should be cut into 5 by 5cm pieces and soaked with 5ml of deionised or distilled water. Using a larger area poultice will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this area will be adequate.

3. Place the poultice on the ceramic surface. If there is doubt about water tolerance, this should be in a discrete area.

4. Allow the poultice to dry for several hours depending on room conditions. See the table below:

Relative humidity	50%	60%	70%	80%
Temperature				
10°C	5.5	6.8	9.3	15.8
20°C	4.7	6.0	7.0	12.8
30°C	2.7	3.1	3.3	4.8

Table 2. Drying time (in hours) of poultice at different temperatures and humidities.

Important notice on Table **2**: Do not allow to dry beyond twice these times.

5. Carefully remove the dried poultice. Place poultice in an oven at 110°C for 4 hours.

6. Extract salts from poultice in 5ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, if needed for instrument sensitivity. For most instruments this volume will be adequate.

7. Filter through a 0.45µm PTFE filter.

8. Submit the filtrate for ion chromatography analysis for sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ion concentrations.

9. Calculate the ion concentration in micrograms per centimeter squared:

> *If sodium concentration is 5.2 parts per million: the amount of sodium in the extraction water is 5.2x5 microgram. The concentration per area is 26/(5x5) = 1.04 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient.*

10. Use the Runsalt program as detailed in section 2.4 to calculate the high and low risk temperature, RH regions with the ion concentration calculated.

2.2.2 Assessing risk from soluble salts to fired ceramics with specific ion meters

This method is not suitable for: pigmented ceramics, ceramics fired to low temperatures, ceramics with friable surfaces.

Many earlier ceramics have only been fired to low temperatures and will be adversely affected by water. A conservator should assess the objects to be tested for water tolerance.

There is a wide range of specific ion electrodes available. Sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ions should be analysed. The below methods should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in

most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted.

1. Conservator assessment of the object to ensure it can withstand poulticing.

2. A poultice should be formed of three layers of Whatman's number 1 filter paper. The paper should be cut into 10x10cm pieces and soaked with 40ml of deionised or distilled water. Using a larger area poultice will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this area will be adequate.

3. The poultice is placed on the ceramic surface. If there is doubt about water tolerance, this should be in a discrete area, and the surface should be frequently tested for any softening.

4. Allow the poultice to dry for several hours depending on room conditions. See the table below:

Relative humidity	**50%**	**60%**	**70%**	**80%**
Temperature				
10°C	5.5	6.8	9.3	15.8
20°C	4.7	6.0	7.0	12.8
30°C	2.7	3.1	3.3	4.8

Table 2. Drying time (in hours) of poultice at different temperatures and humidities.

Important notice on Table **2**: Do not allow to dry beyond twice these times.

5. Carefully remove the dried poultice. Place the poultice in an oven at 110°C for 4 hours.

6. Extract the salts from the poultice in 70ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, if needed for the instrument sensitivity. For most instruments this volume will be adequate.

7. Separate the solution into seven sets of 10ml. Add ion adjustors as per manufacturer's instructions. Analyse the extracts for sodium, potassium, magnesium, calcium, chloride, nitrate and sulphate ion concentrations.

8. Calculate the ion concentration in micrograms per centimeter squared:

> *If sodium concentration is 5.2 parts per million. The amount of sodium in the extraction water is 5.2x70 micrograms. The concentration per area is 364 / (10x10 = 3.64 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient.*

9. Use the Runsalt program as detailed in section 2.4 to calculate the high and low risk temperature, RH regions with the ion concentration calculated.

2.3 Glass

2.3.1 Assessing the early stages of glass stability with swabbing and ion chromatography

There is a wide range of ion chromatography equipment, columns and conditions available. The below methods should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted. Analytical ion chromatography normally uses very high purity polished water (18.2MΩ cm^{-1}) to limit the background ion concentrations present. Most conservation labs do not have this purity of water. However, with the ratios in this method deionised or doubly distilled water will allow sufficient accuracy with subtraction of blank samples.

1. The objects should be assessed by a glass conservator before any swabbing takes place.

2. An 8x8 cm^2 area should be swabbed with a polyester swab (Texwipe Alpha® TX761), wetted on one side with 50 microL of deionised water. Samples of the water and an unused swab should be analysed as well.

3. Place the used swab in an oven at 110°C for 1 hour.

4. Extract the swab in 1.5 ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, which may be needed for certain instrument sensitivities. For most instruments this volume will be adequate.

5. Filter the extracted water through a 0.45micron PTFE filter.

6. Sodium and potassium ions can be deposited in dust particles. This can be accounted for by placing a glass microscope slide horizontally or vertically in the showcase or store close to the glass object. Ideally this should be left for 12 months and then swabbed and analysed as per the glass.

7. Submit the filtrate for ion chromatography for sodium and potassium. The presence of other ions can give indications of the causes of decay to the glass.

8. Calculate the sodium and potassium ion concentrations in micrograms per centimeter squared:

> *If sodium concentration is 5.2 parts per million. The amount of sodium in the extraction water is 5.2x1.5 micrograms. The concentration per area is 7.8/8 = 0.975 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient. The glass slide value is subtracted along with the sodium and potassium concentration extracted from the blank swab.*

Ideally the calculated values are divided by the number of years since the last cleaning of the glass surface (which should remove all the ions). A combined sodium and potassium ion production rate of 1.5microgram per square centimeter per year indicates an unstable glass.

If the period since cleaning is unknown, it may be possible to estimate a minimum period from existing records or staff memories. Otherwise, the system devised by Verhaar (2018) is probably the best available at present and is described below.

9. Convert the ion chromatography solution values from parts per million to micromole per liter. Divide the sodium value by 23 and the potassium by 39 (Verhaar, 2018). Add the two values. The table below indicates stability:

Micromole/L	
Less than 20	Likely stable
20 to 50	Potentially unstable
Greater than 50	Likely unstable

Table 3. Solution value stability conversion chart.

Unfortunately, the literature is somewhat unclear on the exact RH range that is best to maintain for such glasses. The consensus is that low RH is required, but values vary. Some authors give 40-42%RH, others 35-40%. Flowing air is recommended to reduce the reaction rate.

2.3.2 Assessing the early stages of glass stability with swabbing and specific ion electrodes

There is a wide range of specific ion electrodes available. Sodium and potassium electrodes are required for this analysis. This method should produce water concentrations suitable for most combinations. Some larger museums may have the equipment. Analysis can be purchased in most countries. Amounts may need to be varied to match the equipment and protocols. Suppliers should be consulted.

1. The objects should be assessed by a glass conservator before any swabbing.

2. An 8x8 cm^2 area should be swabbed with a polyester swab (Texwipe Alpha® TX761), wetted on one side with 50µL of deionised water. Samples of the water and an unused swab should be analysed as well. Retain an unused swab as a blank.

3. Place the used swab in an oven at 110°C for 1 hour.

4. Extract the swab (and blank) in 10 ml of deionised or distilled water for 2 hours. Decreasing the extraction volume will increase the ion concentrations, which may be needed for instrument sensitivity. For most instruments this volume will be adequate.

5. Filter the extracted water through a 0.45-micron PTFE filter.

6. Sodium and potassium ions can be deposited in dust particles. This can be accounted for by placing a glass microscope slide horizontally or vertically in the showcase or store close to the glass object. Ideally this should be left for 12 months and then swabbed and analysed as per the glass.

7. Submit the filtrate (and blank filtrate) for specific ion electrode analysis for sodium and potassium. The presence of other ions can give indications of the causes of decay to the glass. A single ionic strength adjustor should be selected (not containing sodium or potassium).

Subtract

8. Calculate the sodium and potassium ion concentrations in micrograms per centimeter squared (with eth blank subtracted from the value):

> *If sodium concentration is 5.2 parts per million. The amount of sodium in the extraction water is 5.2x10 micrograms. The concentration per area is 52/8 = 6.5 micrograms per centimeter squared. Figures should be calculated to at least two decimal places. Many instruments and protocols can produce better precision, but this is sufficient. The glass slide value is subtracted along with the sodium and potassium concentration extracted from the blank swab.*

Ideally the calculated values are divided by the number of years since the last cleaning of the glass surface (which should remove all the ions). A combined sodium and potassium production rate of 1.5microgram per square centimeter per year indicates an unstable glass.

If the period since cleaning is unknown, it may be possible to estimate a minimum period from existing records or staff memories. Otherwise, the system devised by Verhaar (2018) is probably the best available at present.

9. Convert the ion chromatography solution values from parts per million to micromole per liter. Divide the sodium value by 23 and the potassium by 39 (Verhaar, 2018). Add the two values. The table below indicates stability:

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Less than 20	Likely stable
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Unfortunately, the literature is somewhat unclear on the exact RH range that is best to maintain for such glasses. The consensus is that low RH is required, but values vary. Some authors give 40-42%RH, others 35-40%. Flowing air is recommended to reduce the reaction rate (Thickett and Ling, 2022).

2.4 Calculating stable Temperature RH Regions from ion data

2.4.1 Runsalt and SaltsR

The ion data will first need to be corrected for gypsum content using the salt content calculator developed by the KIK-IRPA as part of the PREDICT project. This must be done in order to input the data into the ECOS Runsalt software. ECOS was the project that developed the method, Runsalt is the program used for eth calculations.

1. Salt ion correction: Gypsum correction.

Before running raw ion chromatography data in Runsalt, the raw ion chromatography data is recommended to be corrected for gypsum content. There are three possible methods of doing this. The first (a) and most recommended, uses the salt content calculator provided by the KIK-IRPA (part of the PREDICT project). The second (b) uses a web app currently under development. The R code (c) is also shown below.

a. Salt ion correction: KIK-IRPA Salt Content Calculator (recommended): https://predict.kikirpa.be/index.php/tools/moisture-and-salt-sample-data-analysis-tool/. Follow the online instructions.

Ion chromatography data required to be input:

- Dry sample mass (g).
- Amount of water added (mL).
- Cation concentrations (mg/L): Sodium (Na^+), Potassium (K^+), Calcium (Ca^{2+}), Magnesium (Mg^{2+}).
- Anion concentrations (mg/L): Chloride (Cl^-), Nitrate (NO_3^-), Sulphate (SO_3^{2-}).

If warnings are issued, record them.

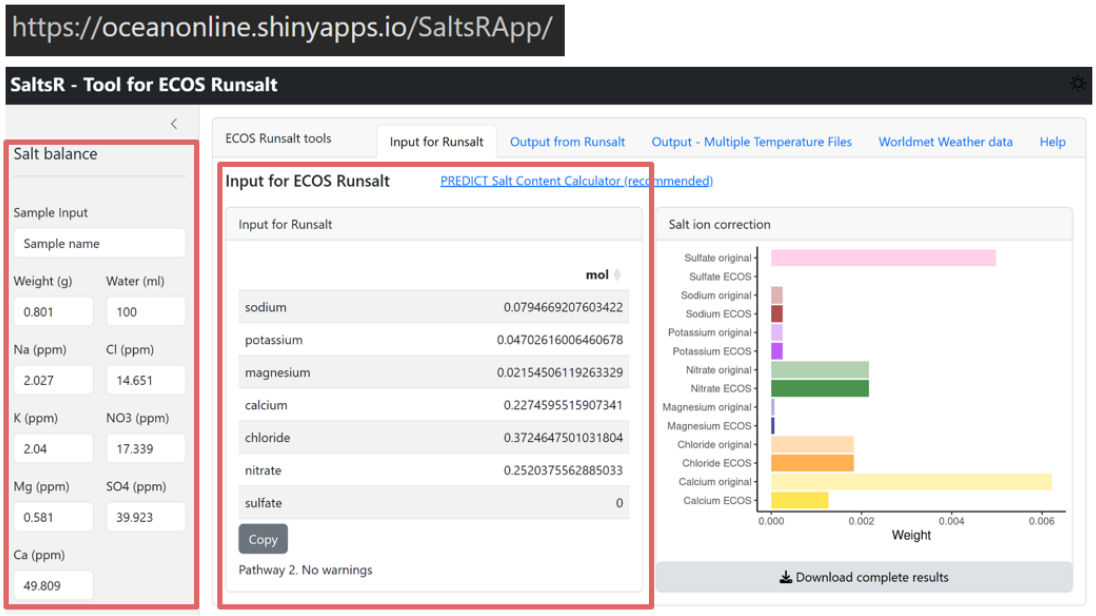
The output (see Figure below) gives corrected salt ion concentrations, suitable for use in ECOS Runsalt at the bottom of the page.

Ion	Amount [mol/kg]	Mole Fraction
Cl^-	0.051592365	0.372470676
NO_3^-	0.034911260	0.252041566
SO_4^{2-}	0.000000000	0.000000000
Na^+	0.011003041	0.079436366
K^+	0.006513880	0.047026908
Mg^{2+}	0.002984338	0.021545404
Ca^{2+}	0.031509014	0.227479080

b. Alternatively, online: Salt ion correction.
Go to the website: https://oceanonline.shinyapps.io/SaltsRApp/.

Note: This is version 0.1, the most current correction algorithm will always be available on the PREDICT website[1]. Discrepancies could exist between tools.

[1] The full link: https://predict.kikirpa.be/index.php/tools/moisture-and-salt-sample-data-analysis-tool/.



c. Alternatively, using R: Salt ion correction.

Note: The most current correction algorithm is always available on the PREDICT website. Discrepancies could exist between tools.

In R, load the Shiny application and add the corrected ion chromatography data:

```
> install.packages("pak")
> pak::pak("BhavShah01/SaltsR")
> library(SaltsR)
> runSaltsR_app()
```

You can also run the “fun_salt_balance” function in R. This will return a full list of calculations:

```
> fun_salt_balance(sample_name = "Pathway 2",
                   dry_g = 0.801,
                   water_ml = 100,
                   chloride_ppm = 14.651,
                   nitrate_ppm = 17.339,
                   sulfate_ppm = 39.923,
                   sodium_ppm = 2.027,
                   potassium_ppm = 2.04,
                   calcium_ppm = 49.809,
                   magnesium_ppm = 0.581) |> glimpse()
```

The output will return the following for input into ECOS (mol):

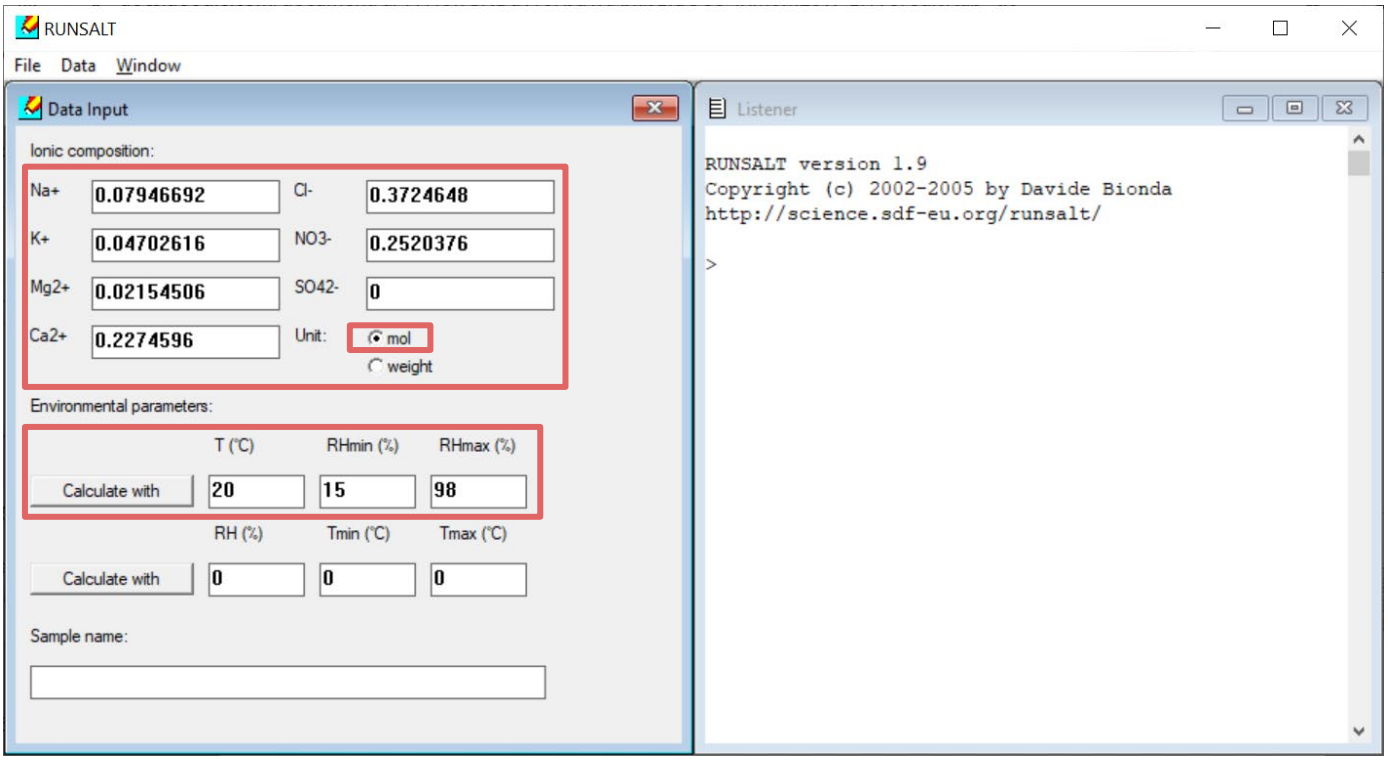
```
$ sodium_ECOS_mol          <dbl> 0.07946692
$ potassium_ECOS_mol       <dbl> 0.04702616
$ magnesium_ECOS_mol       <dbl> 0.02154506
$ calcium_ECOS_mol         <dbl> 0.2274596
$ chloride_ECOS_mol        <dbl> 0.3724648
$ nitrate_ECOS_mol         <dbl> 0.2520376
$ sulfate_ECOS_mol         <dbl> 0
```

2. ECOS Runsalt.

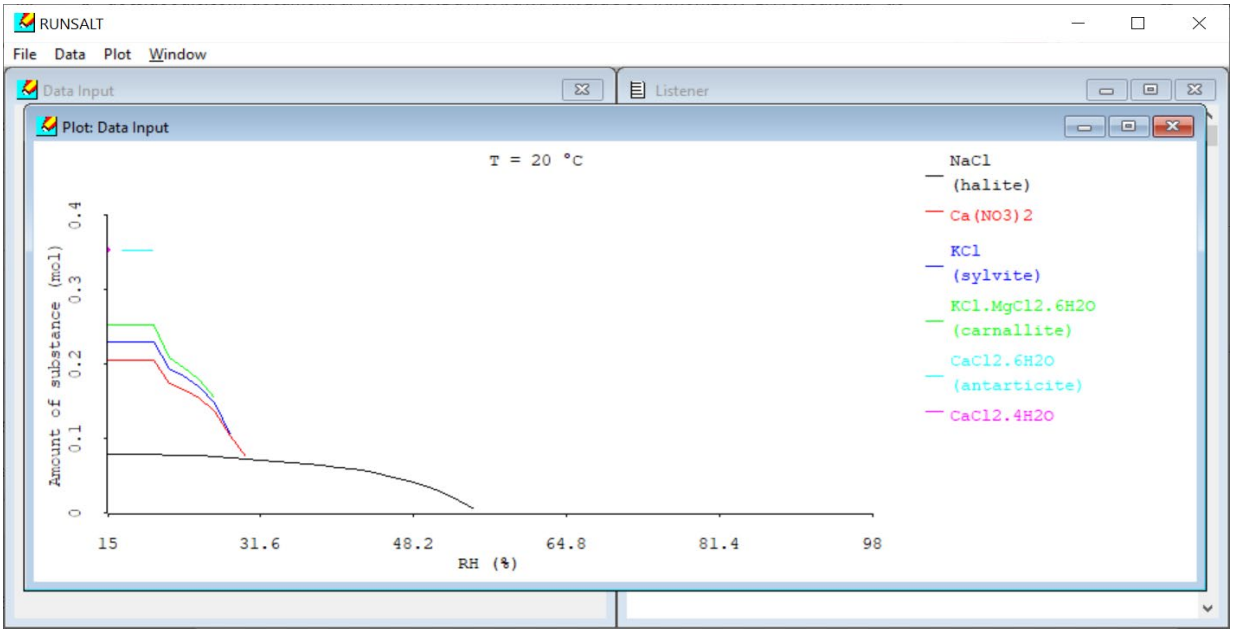
Once the data has been corrected for gypsum, the data can be inputted into the ECOS Runsalt software:

Runsalt v1.9 software: http://science.sdf-eu.org/runsalt/
Saltwiki: https://www.saltwiki.net/index.php/Software

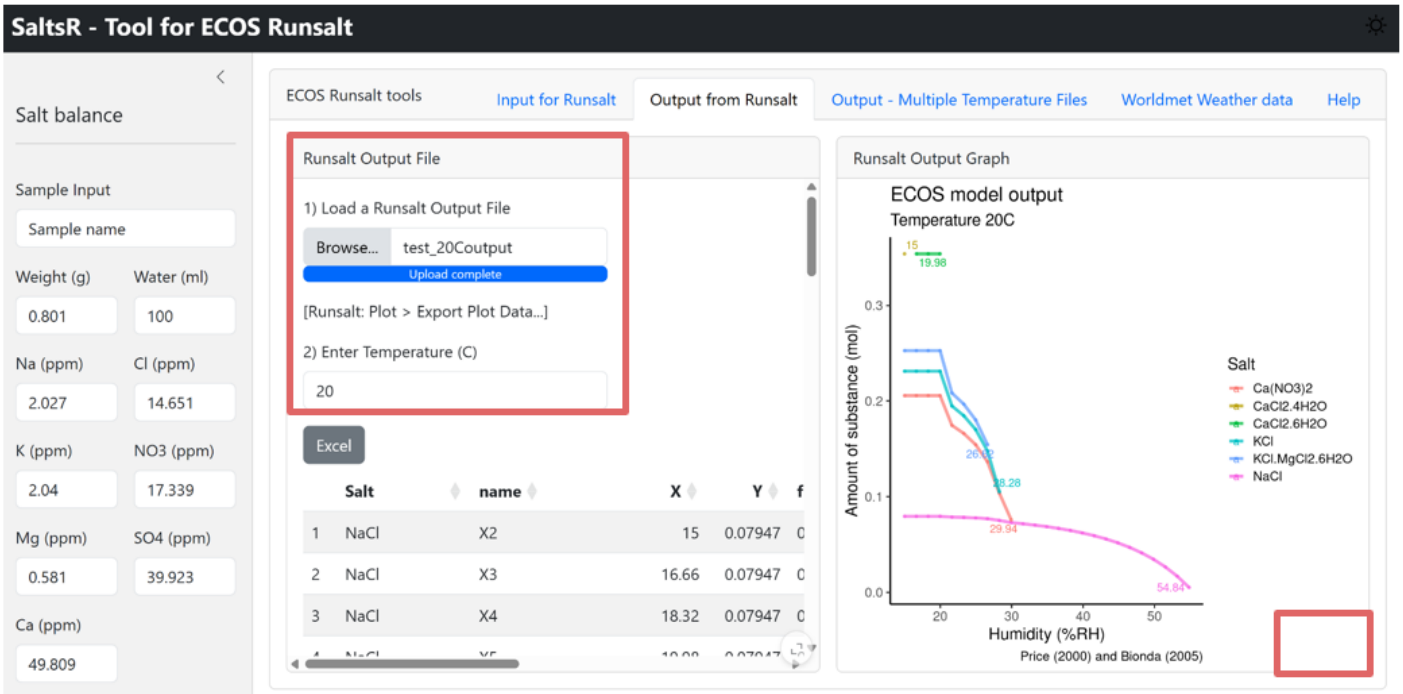
a. Data Input:
   i. Enter the corrected salt ion data into the software's input fields.
   ii. Set humidity range, can be run between 15–98%RH.
   iii. Set the temperature at one value. Can increase by 5°C increments across the required range.
   iv. Save the file to re-run the calculations later, for example: TestECOSinput'.



b. Data Output:
   i. After running the simulation, export results by navigating to: *Plot > Export Plot Data…*
   ii. Save the output file with a filename (e.g., `Test_ECOSOutput_20C`).
   iii. Re-run if other temperature values are required (e.g. in 5°C increments).
   iv. Save the plot output data: Plot > Export Plot Data...



c. Graphing the results:
   i. Go to the website: https://oceanonline.shinyapps.io/SaltsRApp/.
   ii. Upload the Runsalt output file. [Plot > Export Plot Data…].
   iii. Enter the temperature.
   iv. Expand graph to right-click and save.
   v. The table can return the results with crystallisation points and RH equilibrium (2nd differential).



d. Issues:
The SaltsR R package: https://bhavshah01.github.io/SaltsR/ is still being developed. Some mixed concentrations produce errors. Details are given at https://github.com/BhavShah01/SaltsR/issues)

2.5 Creating graphs of Environmental data with instability regions using Microsoft Excel

Input environmental data into Excel spreadsheet (making sure temperature is the first column).

To create the instability region graph, it is easiest to create three different charts with the following method, then overlaying them. Instructions will be laid out below, but should you wish for a narrated walk through, one can be found here: https://www.youtube.com/watch?v=2nAnNEATUZU.

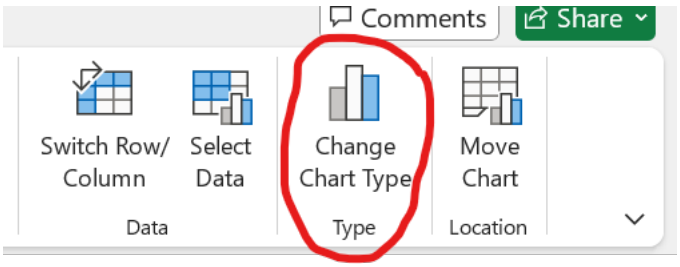
a. To create the instability regions, you will need the minimum (Bottom RH) and maximum (Top RH) RH values for each salt you would like to graph at each temperature (this is the risk region for a given salt present). The ECOS Runsalt and SaltsR softwares can provide this information.

	10	**15**	**20**	**25**	**30**
Top RH	90	85	80	80	80
Bottom RH	70	70	70	70	70
Base	90	85	80	80	80
Diff	-20	-15	-10	-10	-10

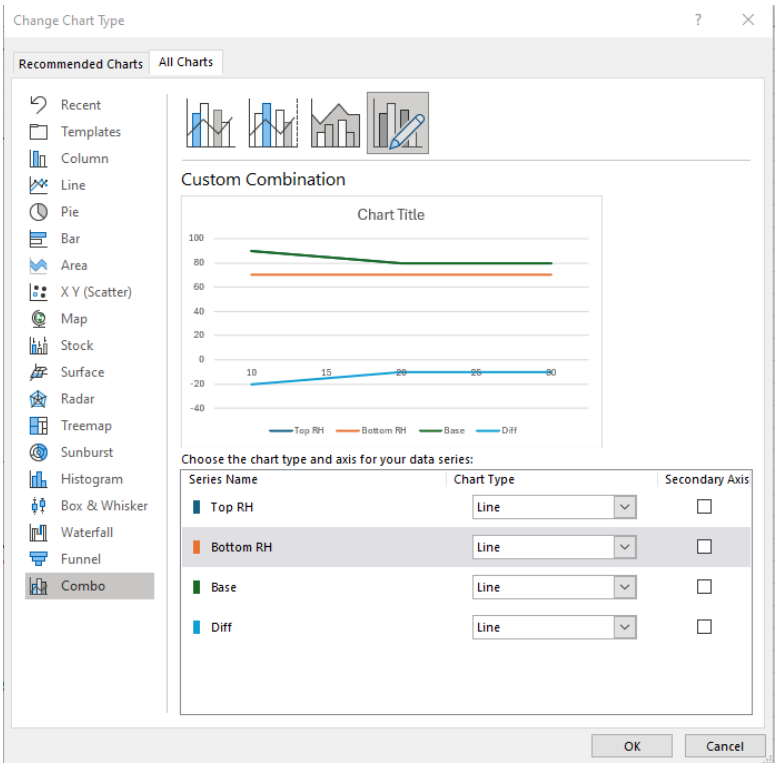
The top row is the temperature. The upper and lower boundaries of the region (top and bottom RH in the table) correspond to the minimum and maximum values at each temperature for the salt.

The base is the same as the top row, and the difference (Diff in the table) is the bottom RH value minus the top RH value. You must include the base, otherwise this will not work!

b. Insert a line chart for the table. The temperature should be along the X-axis, and you should have a line for each row, with the RH values as the Y-axis.
c. Once the chart has been created, open the Chart Design tab along the top ribbon and click Change Chart Type.

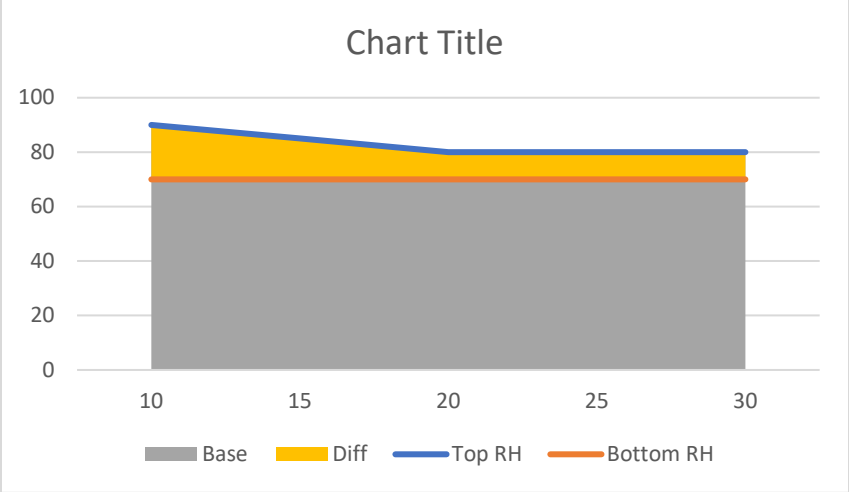


d. Select the Combo option, then Custom Combination. You should have a dialogue box like the following:



e. Change the chart type for Base and Diff to stacked area. Make sure the Top and Bottom RH Chart Types are set to Line. Click OK.

f. You will end up with something like this:

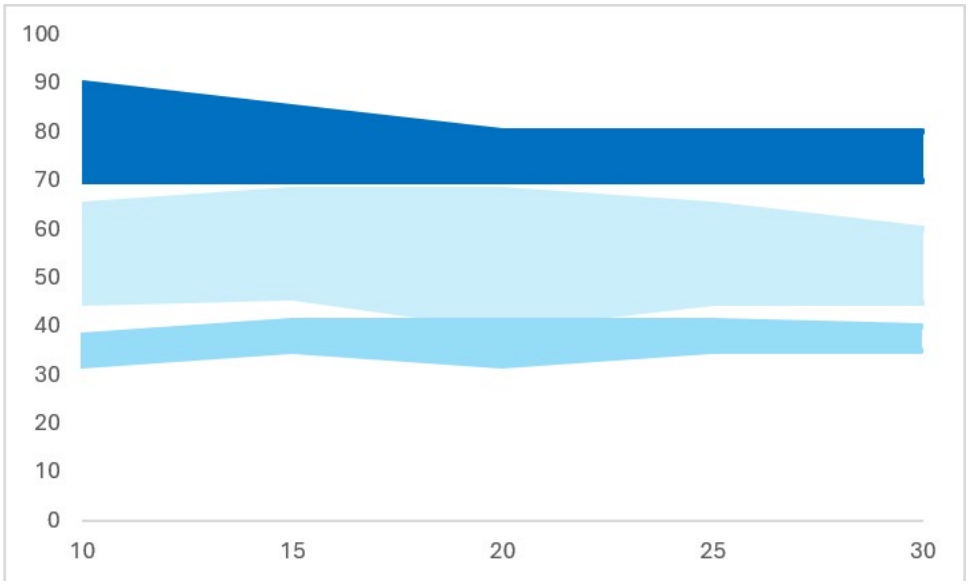


g. Double click on the base area (anywhere in the green area) to select it. The format data series panel should appear (usually on the left).

h. Under the Fill & Line tab (bucket symbol), select no fill. You should then end up with the instability region highlighted.

i. You can adjust the axes to suit, but an option (under Axis Options tab) is to set the X-axis position to be on tick marks, which will then set the chart to begin and end at the edges of the region.

j. You can then customize the chart as you wish (i.e. colours for the region and lines). If you select the same colours for each, it will appear as a complete, solid region, but this is down to personal preference. You can also change the transparency of the area to suit (which may be worthwhile if you are graphing overlapping instability regions). It is recommended that you remove the legend, chart title and gridlines.
k. Repeat steps A-J for each region you would like to include in the chart (you will have separate charts for each region).
l. Once you have made your charts, pick one to be the bottommost one (your base chart).
m. On the chart you have selected for the second layer, set the axes to full transparency (do not remove them, as they help with lining up).
n. Select the whole chart and under the Chart Options and Fill & Line tabs, select No Fill. This will remove the background and allow you to overlay this layer onto the base chart.
o. Repeat steps M & N until you have all the required regions.
p. Under the Format tab in the ribbon, click Selection Pane.
q. Using the Selection Pane to hide/reveal each chart, select all charts (Shift or Ctrl click), then Group the charts together using the Group function in the ribbon.
r. You should then have your completed instability region graph:



s. Should you wish to have axis titles or other chart features, add them to the *base chart only*. A legend might be tricky, and it might be easier to create one not linked to the charts.

To create the scatterplot element, create a scatterplot of your environmental data, making sure the temperature is along the X-axis and the Relative Humidity is along the Y-axis.

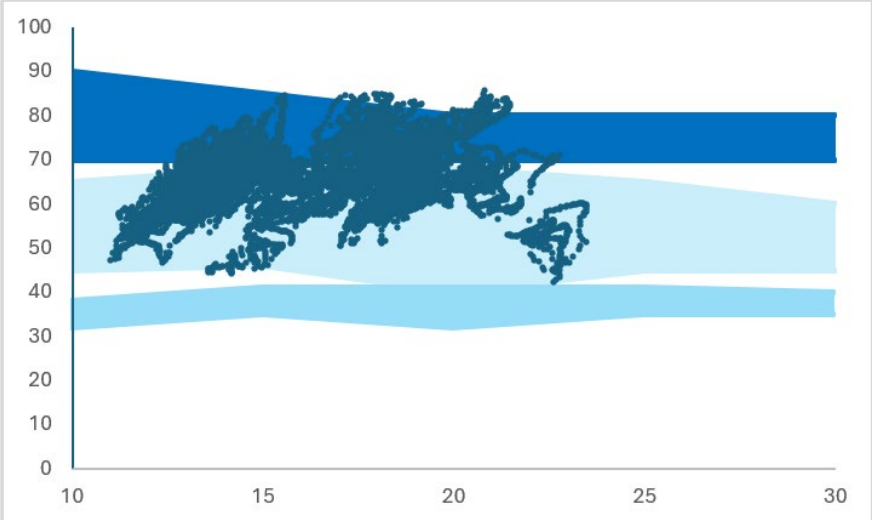
t. Adjust the X and Y axes to match those of the instability region graph.

Remove the legend, chart title and gridlines from the scatterplot.

u. You may also want to adjust the marker size at this stage (under Series Options and Marker Options).

Adjust the axes and make them transparent (see steps I, J and M).

Remove the background of the chart (see step N).



Overlay the scatterplot onto the instability regions graph. You can add the scatterplot to the existing group of charts or create a new one with it.

You should now have your completed graph!

3. Conclusions

The methodologies for the materials listed in the project proposal have been developed. Significant work has been undertaken to verify the environmental conditions required for unstable objects (Thickett et al 2023, 2023a, Thicket and Ling 2022, Thickett et al 2023b). Only a few materials have sufficient knowledge to determine their stability. The methods produced in this work compliment, noninvasive oxygen depletion for archaeological iron and copper alloys and the microinvasive shrinkage temperature measurement for parchment and micro-fading methods already developed. The methods have been adapted to allow easy access, avoiding more complex preparation equipment and particularly assessing the water quality needed. Thorough instructions have been provided to implement the ECOS Runsalt software and interpret the results.

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