

Data XRF analysis (Röntgenfluorescentiespectrometrisch onderzoek)

Object :	569 (KN&V) transparant area	
Artist :	Anonymous	
Title and date :	Kom - à la façon de Venise	1600-1700
Conservator :	Mandy Slager - Restauratieatelier	
Analist :	Luc Megens - sr. Researcher RCE	
Date analysis :	20-03-2024 16:13:00	

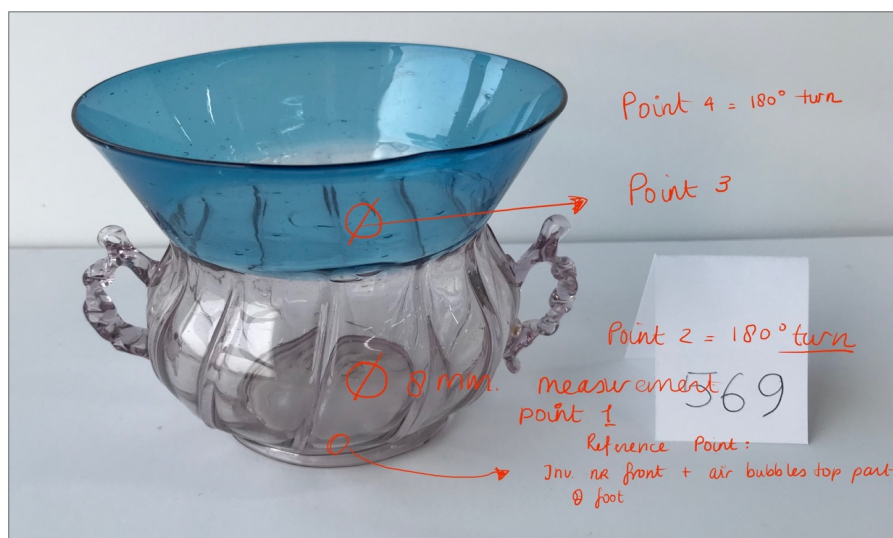


Visual examination - microscopy	Ion - Chromatography	pXRF - analysis (relative to objects in the project)
Date: 20-03-2024	Date: 16-09-2020	Date: 20-03-2024
Very poor	Likely unstable	Further research needed

Specific research question
What type of glass? Which elements are detected by pXRF that might be related to the stability of the glass? Does comparison of all the objects analysed in this project reveal particular information? Can we identify any correlations between the pXRF results and the visual and IC results relating to the condition of an object? Would it be possible to carefully work towards condition categorization based on non destructive pXRF analysis of composition in combination with visual and IC analysis? Additional analysis needed: what are the options? Does the current information give impetus to further (non destructive) research to support the monitoring of condition?

Examination and analysis	
Instrument	Bruker Tracer 5g
Instrument settings	MudRock Application in ambient air, 8 mm collimator 1: 50 kV, 14.7 µA, filter Ti 25 µm/Al 300 µm, 45 s 2: 15 kV, 19.9 µA, no filter, 60 s
Additional information	Setting 1 for heavier elements and setting 2 to better detect the lighter elements (from Mg). Automatic quantification, average of 2 measurements. DualMudrock Air version 730.0646.01 .

Location mapping of compositional analysis and measurement of approximate glass thickness



Practical limitations	Surface morphology might have had an influence on the accuracy of the measurements
App. thickness in mm.	1,17 (not at location of measurement: thickness of blue area: but likely similar to transparant area)

Spectra

EDS spectrum of the sample. The x-axis represents X-ray energy in keV, ranging from 0 to 10. The y-axis represents intensity. The spectrum shows several characteristic peaks for the elements present in the sample:

- Si** (Silicon): The most intense peak, located at approximately 1.74 keV.
- Mg** (Magnesium): A small peak at approximately 1.30 keV.
- Al** (Aluminum): A small peak at approximately 1.55 keV.
- Pb** (Lead): A small peak at approximately 2.42 keV.
- K** (Potassium): A small peak at approximately 3.96 keV.
- Ca** (Calcium): A small peak at approximately 2.98 keV.
- Ti** (Titanium): Two small peaks at approximately 4.51 keV and 4.94 keV.
- Mn** (Manganese): A small peak at approximately 5.89 keV.
- Fe** (Iron): Two small peaks at approximately 6.40 keV and 7.11 keV.
- Cu** (Copper): A small peak at approximately 8.93 keV.

filter: Ti, 25 / Al, 300

EDS spectrum showing peaks for various elements. The x-axis is X-ray energy (keV) from 0 to 40. The y-axis is intensity. Peaks are labeled with element symbols: Si, Cl, Ca, K, Mn, Fe, Cu, Ni, Zr, Pb, Br, Rb, Sr, and a large peak for Fe. A filter of Ti, 25 / Al, 300 is indicated.

Results

Limitations	Na2O can not be accurately quantified (underestimated). Sn and Sb are not quantified with the MudRock application. As isn't quantified properly in Pb glass. Quantitative results give an indication of composition, but not exact concentrations.											
Main elements in wt%	Na2O	MgO	Al2O3	SiO2	P2O5	SO3	K2O	CaO	ZnO	BaO	PbO	UO2
	1,55	0,98	< LOD	45,04	0,17	0,68	1,1	1,97	0,01	0	0,09	< LOD
Trace elements in ppm	Cl	Ti	V	Cr	Mn	Fe	Co	Ni	Cu	As	Rb/Sr	Zr/Mo
	3777	127	21	< LOD	3714	1918	< LOD	32	303	215	84/207	14/< LOD
Type of glass I	Potassium - Lime											
Type of glass II	Mixed alkali											

Additional information	
------------------------	--

Both colours (blue part and colourless part) likely a potassium-lime glass, but the concentration potassium-calcium is higher in the blue than in the colourless glass. The blue part is coloured with copper and contains more iron than the colourless part. It is therefore possible that two different batches were used (and not that just colouring agents such as copper were added to the same batch). Together with the lead likely some lead came into the batch as well. But it is also possible that recycled lead-containing glass was used.

The quantitative data were obtained by applying the DualMudrock_Air quantification method (version 730.0646.01) in the Bruker Tracer 5g. This quantification is based on rock standards and is therefore not entirely accurate for glass. This is especially the case when the concentration of an element lies far outside the range of concentrations of that element in the standards, as the matrix effect is then not accounted for in the extrapolation. Furthermore, antimony is not quantified in this method, even though it is present in many of the analyzed glasses. For this reason, the concentration was estimated based on a comparison with the most closely matching glass standard from the Corning Museum of Glass (Adlington, 2017). Sodium is quantified very unreliably because the measurement was conducted in air (which strongly absorbs the fluorescence radiation of Na), and this element is detected only within a maximum of the uppermost micrometer of the sample due to self-absorption. The depth up to which elements with high-energy fluorescence radiation are detected (such as Pb, Rb, Sr, Zr, Sn, Sb) can exceed the thickness of the glass. In that case, these concentrations are underestimated because a portion of air behind the glass is measured. This must also be taken into account when comparing the values for these elements (see the glass thickness measurement above).

	Gewichts%								
ID	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	ppm Cl	K ₂ O	CaO
kleurloos	1,5	1,0		57,5	0,2	0,7	4840	1,3	2,3
blauw	1,4	1,0	0,7	74,9	0,3	0,8	3051	2,9	3,3

Discussion of results
1. In absolute and relative comparison a larger spreading of the K₂O and PbO measures can be detected. This is mostly due to the fact that the energy the fluorescent radiation of K emits is lower than of Pb. De subsequent percentage calculations derive from this. With greater distance of the detector to the surface the radiation of K is absorbed much stronger by the air than is the case with Pb. 2. Not yet possible to draw conclusions on sensitivity of glass to instability by means of pXRF. But in combination with the visual and IC results it might be possible to group objects or determine correlations between the diagnostic data. 3. Quantitative XRF results on composition are not the same as the components of a recipe. 4. Presence of Mn might be related to sensitivity to degradation 5. <LOD = Below Limit of Detection - element might be present but in a smaller amount than can be detected by means of pXRF 6. Composition blue part: potassium-lime or mixed alkali with little lead, more than in the colourless glass. 7. Blue part: copper gives the blue colour, contains more iron than colourless part. 8. In blue part: also a little Sn and Sb present (more than in the colourless glass) 9. Mixed alkali (type 2) with quite low alkali and lime or potassium lime? Corning B. 10. Final conclusion: potassium-lime glass.

Object in relation to other objects in this project
See also: RCE Report: Megens, L., 2025, Verkenning van de samenstelling van verschillende types glas in Museum Boijmans van Beuningen in relatie tot het risico op degradatie, RCE projectnummer 2023-099, Amsterdam, Rijksdienst voor het Cultureel Erfgoed, Rijks erfgoedlaboratorium which can be found in Conservation Studio under <i>verwante media</i>.

Suggestions for further research
1. Further pXRF analysis on comparable objects 2. Search for (archival) information on manufacturing process or recipes 3. Correlations to be found by comparing data to reflectance spectral images with Optical Coherence Tomography? U of Nottingham 4. Correlations to be found by comparing current data with info acquired by means of Terahertz (THz) examination? U of Eindhoven 5. Further research by means of Raman Spectroscopie or Inductively Coupled Plasma Mass Spectrometrie (ICP-MS) or Scanning Electron Microscopy (SEM) - possible in situations where sample fragments are available. Many more analytical examination can then be explored, but usually analysis on museum objects is limited due to non invasive requirements and practical complications when having to move objects to lab facilities.

Literature
1. Shelby, J.E., Introduction to Glass Science and Technology, Cambridge 2005, p.30. 2. Megens, L., 2021, Verkenning van de samenstelling van verschillende types glas in het Nationaal Glasmuseum Leerdam in relatie tot het risico op degradatie, RCE projectnummer 2020-110, Amsterdam, Rijksdienst voor het Cultureel Erfgoed, Rijks erfgoedlaboratorium. 3. Burghout, F. en M. Slager, Cloudy Patches and Misty Glass: Early Signs of Glass Disease? In: Roemich H. and K. van Lookeren Campagne (eds.) Recent Advances in Glass, Stained-Glass and Ceramics Conservation, Amsterdam, the Netherlands, 7-10 October 2013, pp. 327-329. 4. Adlington, L.W., 2017. The Corning Archaeological Reference Glasses: New Values for "Old" Compositions. Pap. Inst. Archaeol. 27, 1-8. https://doi.org/10.5334/pia-515