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Lamain, B.; van Bommel, M.; Verhaar, G.; Tennent, N.H.

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# The Development of an Ion Chromatography Protocol for Detecting the Early Stages of Glass Degradation

Bodill Lamain, Maarten van Bommel, Guus Verhaar, Norman H. Tennent\*

\*Corresponding author

## Keywords:

Rijksmuseum; unstable glass; glass sickness; condition survey; ion chromatography

## Abstract

*In conjunction with the re-display of the collections of the Rijksmuseum, Amsterdam, a condition survey of the vessel glass holdings revealed that approximately 10% displayed changes of appearance, ranging from surface haze to advanced crizzling. Ion chromatography analysis of samples taken from the glass surface by moist cotton swabs has the potential to pinpoint unstable glasses. Thus, difficulties in identifying objects that were indubitably in the early stages of glass deterioration led to investigation of ion chromatography to quantify surface residues of sodium, potassium, calcium, and magnesium ions, symptomatic of only those items undergoing breakdown of the glass matrix. Results are reported for the development and optimisation of a straightforward analytical protocol. Good recovery and reproducibility were achieved for extraction of all four cations from cotton swabs in a laboratory validation, prior to full implementation for the examination of historic glass.*

## Introduction

'Glass sickness' is the evocative term coined, and still commonly in use, to describe the propensity of vessel glass with unstable composition to undergo surface changes primarily referred to as 'weeping' and 'crizzling'. Most studies of glass sickness have been devoted to understanding its causes and to controlling the environment in order to prevent the onset of glass weeping and crizzling in museum collections (see, for example: Organ 1957; Brill 1972; Brill 1975; Hogg and others 1999; Römich 1999; Koob 2006; Kunincki-Goldfinger 2008). Less attention has been focused on identifying the early stages in the deterioration process and, in particular, the difficulty of distinguishing the first signs of true glass sickness from other surface effects. This paper concerns the visual categorisation of a range of surface changes displayed by vessel glass in the Rijksmuseum, Amsterdam, and the initial phase of the development of a straightforward analytical approach to pinpoint those glasses actually undergoing breakdown of the glass matrix.

This research was motivated by a condition survey of vessel glass in the Rijksmuseum. The conclusions had urgency

because the survey was being carried out in preparation for re-display of the collection for the re-opening of the museum, in 2013, after several years of refurbishment. Priorities had to be set for conservation treatments, and recommendations were required for optimal parameters for the display and storage environment. A clear-cut specification of vulnerable glasses with unstable compositions was necessary in order to identify items where special measures should be put in place to minimise deterioration of the glass. Prior to initiating the survey, discussions took place with conservators in other museums. Account was also taken of the methodology and conclusions of similar assessments, notably in the United Kingdom (Cobo del Arco 1999; Oakley 1999). The stages of glass deterioration proposed by Koob for specifying the advancement of crizzling (Koob 2006) provided a very useful framework to categorise unstable glass, but it soon became apparent that many Rijksmuseum items could not be classified with certainty as unstable. Particularly problematic were those glasses where the surface changes were minor; were those effects due to glass deterioration or less worrisome causes that could broadly be described as soiling?

This led to the development of five descriptive categories covering the full range of deterioration (table 1) but focusing on the numerous examples of minor surface effects (denoted by terms such as ‘loss of brilliance’, ‘surface haze’, ‘greasy appearance’, etc). For many glasses, it was realised that, without relevant scientific analysis, no conclusions could be made about whether they were exhibiting the initial stages of glass sickness or what the prognosis for these items would be, on display or in storage, without special environmental control. However, as a support for the visual analysis, no suitable analytical technique and protocol had been reported by other museums confronted by this question.

The ability of instrumental means of analysis to determine glass stability is clearly of benefit to those charged with the conservation of ancient and historic glass. Fourier transform infrared spectroscopy (FTIR) and Raman spectroscopy have been used to categorise glass stability (Earl 1999; Robinet and others 2006). While the potential of these spectroscopic non-invasive techniques has been demonstrated, as yet they are unable to provide conservators with a sufficiently clear-cut assessment of glass stability. Accordingly, the research described in this paper was initiated to explore the scope of ion chromatography, a technique that has the potential to detect minute quantities of glass breakdown products on the surface of objects and thus pinpoint the onset of glass sickness.

### Rijksmuseum Glass Categories

The objects in the Rijksmuseum glass collection were divided into the categories fully described in table 1. Examples are depicted in figures 1–3. Category A consists of items that are in perfect condition and no treatment is necessary. Category B glasses show very minor surface changes, possibly indicative of degradation, such as loss of brilliance or mild haziness. Category C consists of objects that show very probable signs of glass degradation denoted by, for example, a distinctly ‘oily’ surface (figure 1). Category D contains objects that show undisputed symptoms of degradation such as clear crizzling or the formation of small droplets on the surface (figures 2 and 3). Finally, Category E contains objects that are beyond restoration; these objects are no longer fit for display. This paper is primarily concerned with the need to categorise more precisely the dubious effects represented in Categories B and C. It is anticipated that the positioning of items within these categories may well be revised once more information becomes available.



Fig. 1. (top) Ornate Dutch glass, height 25.6 cm, ca 1550–1600 (Rijksmuseum Amsterdam, Reg. No. BK-NM-776). (bottom) Detail showing surface deterioration, assigned to category C (Photo: © Rijksmuseum).



Fig. 2. (top) Dutch engraved glass flask, height 17.6 cm, ca. 1675–1692 (Rijksmuseum Amsterdam, Reg. No. BK-NM-754). (bottom) Detail showing surface deterioration, assigned to category D (Photo: © Rijksmuseum).



Fig. 3. (top) Venetian clear glass vase, decorated with blue dots, height 14.1 cm, ca. 1900–1910 (Rijksmuseum Amsterdam, Reg. No. BK-1978-111). (bottom) Detail showing surface deterioration, assigned to category D (Photo: © Rijksmuseum).

## **Ion Chromatography**

Ion chromatography is a form of high performance liquid chromatography for quantification of low concentrations of cations or anions in aqueous solutions. In this technique, ion exchange mechanisms are used to separate ionic species in a solution that is pumped at high pressure through a column containing small spherical beads with ion-exchange sites. Various procedures, but most commonly conductivity detection (as used in the experiments described below), can be used to monitor the elution of the separated ions from the column. The first commercial instrument was produced by Dionex almost 40 years ago following pioneering work by Small, Stevens, and Bauman (1975). Since then, ion chromatography has become an important technique in analytical chemistry. Several textbooks are available; that by Fritz, Gjerde, and Pohlandt (1982) covers the subject well. A description of the technique and some applications to the examination of surface changes occurring on historical artefacts has been reported (Tennent and others 1992). Of importance to the present study is the power of ion chromatography to detect the presence of 'incipient efflorescence'; it was demonstrated that ionic species could be detected on the surface of artefacts, even when invisible to the naked eye (Tennent and others 1992).

The research reported in the present paper explored the potential of ion chromatography to detect cations that are liberated from the glass structure by ion exchange (Römich 1999) and easily removable from the glass surface by swabbing with moist cotton buds. The research, the first stage of a continuing project, focused on the identification and quantification of sodium, potassium, calcium, and magnesium, the principal glass components whose presence on the surface, associated with barely discernible visual changes, is likely to signify breakdown of the glass. Consequently, this approach offers the potential for a direct and clear-cut categorisation of unstable glass. Changes in the glass itself as represented by modification of the Si–O bonding arrangements can be probed by FTIR and Raman spectroscopy (Earl 1999; Robinet and others 2006; Robinet and others 2008); however, as yet, neither has the ability to discern, unambiguously, the early stages of glass sickness.

Experiments were undertaken to develop a protocol to quantify cations on the glass surface, indicative of the inherent instability of the glass composition and the susceptibility of these glasses to progressive decay. As with all new applications of analytical techniques, it was necessary to establish a straightforward methodology and to verify that it produces

reliable results. The validation of this protocol, prior to implementation for the examination of historic glasses, is the focus of the research reported below.

## **Experimental**

### **Instrumental Set-up**

The set-up used in this research is illustrated schematically in figure 4.

Sample injection was performed using a Waters 717 Autosampler that enabled multiple samples to be analysed consecutively from a carousel with 1 mL vials. The eluent used in this study was 20 mM methane sulfonic acid, filtered using a 0.22  $\mu\text{m}$  filter. A Waters 616 Pump was used with a flow rate of 0.5 mL  $\text{min}^{-1}$ . A Waters 6005 Controller gave connection between the pump, autosampler, and conductivity detector. A Dionex IonPac CG12A-5  $\mu\text{m}$  3 x 30 mm Guard Column and 3 x 150 mm Analytical Column, designed specifically for the analysis of alkali and alkaline earth metals, was used in conjunction with a Dionex CSRS300 2 mm self-regenerating suppressor and a Dionex CD20 conductivity detector in combination with a Dionex DS3 detection stabiliser. A Waters CHM column heater was set at 30°C to give maximum peak resolution. Waters Empower chromatography software controlled the analytical set-up features.

A typical cation chromatogram is shown in figure 5. The peak surface area is a measure of the concentration of a species in solution.

### **Extraction Experiments**

This paragraph describes the experimental set-up and the protocols developed for the extraction experiments. The first experiments focused on extracting given amounts of cations from cotton swabs as simply as possible. As the research proceeded, more complex methods of extraction were investigated in order to achieve consistent and reliable results. The goal of these experiments was to improve the extraction of ions from the swabs into solution with good recovery and reproducibility.

#### *Basic Extraction Method*

An amount between 50 and 100  $\mu\text{L}$  of a stock solution containing the four cations under investigation (hereafter referred to as the test solution) was applied to a cotton swab

using a micro-pipette. After application of the test solution to the swabs, they were left to dry until all the water evaporated; this was done to simulate a real-life sampling procedure (namely, when a conservator takes a sample from a glass surface and lets the sample dry before sending it for scientific analysis). After drying, the swab was placed in 1 mL of de-ionised water and left for 90 minutes. After extraction,

the swab was removed from the tube, and the extraction fluid was centrifuged for 10 minutes, in order to avoid possible contamination by particles or fibres released from the swab. 800  $\mu$ L of the extraction fluid was removed from the tube using a pipette and inserted in a vial. The solutions obtained were analysed to assess recovery and reproducibility of the extraction method.

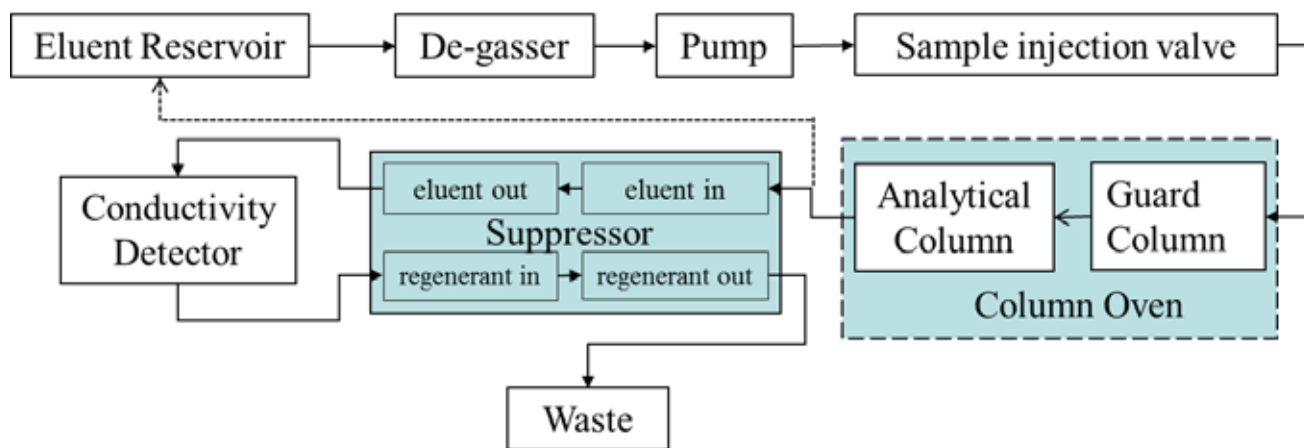


Fig. 4. Schematic overview of the set-up of the ion chromatography system used for the experiments.

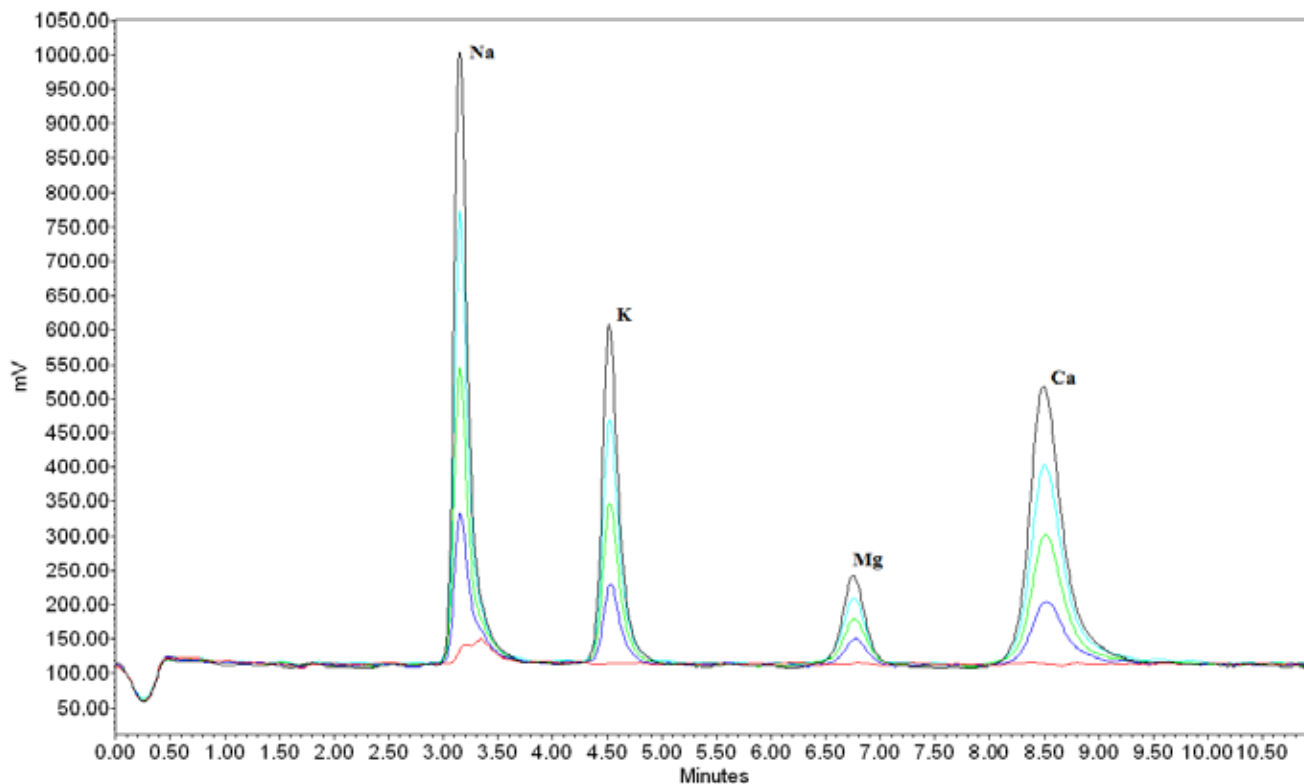


Fig. 5. A typical chromatogram for sodium, potassium, magnesium, and calcium; ion calibration solutions with analyte concentrations of 100 ppm (black), 75 ppm (light blue), 50 ppm (green) and 25 ppm (dark blue) and a blank sample (red).

## Reference Samples

In parallel to the extraction experiments, reference samples were also analysed. These samples were prepared by making a solution similar to that of the extraction solutions. The same volume as applied to the cotton swabs was pipetted directly into a vial and diluted to a total volume of 1 mL. Ideally, this would yield a solution with the same concentration of ions as the extracted solution. In this way the analytical results of the extracted solutions can be compared with the reference samples, and the recovery – the degree to which the ions applied on the cotton swabs are brought into solution during extraction – can be determined.

## Variants of the Extraction Procedure

### *Extraction in an Ultrasonic Bath*

This experiment was performed to investigate if extraction in an ultrasonic bath provided more reproducible analytical results and increased recovery. The application of the test solution to the swab, drying procedures, centrifugation, and sample analysis were the same as during basic extraction. During extraction, however, the samples were placed in a Styrofoam tube holder and extracted for 90 minutes in an ultrasonic bath.

### *Extraction With and Without Drying*

Drying samples is time-consuming and can be avoided if the conservator places a sample directly into a tube filled with de-ionised water after sampling. In order to assess the influence of drying the cotton buds, identical volumes of concentrated solutions were applied to a series of cotton swabs. One set of swabs was left to dry overnight in a fume hood, the other set was placed immediately into a tube for extraction. The dried swabs were placed in a tube for extraction the following morning when the water had evaporated. For both sets of swabs, the extraction time was 90 minutes. Since the volume applied to the swab also contributes to the extraction volume if the swabs are not left to dry, the volume of de-ionised water in the extraction tube was adjusted so that the total extraction volume added up to 1 mL.

### *Extraction in Eluent*

The water used for extraction of the swabs has an influence on the chromatogram obtained. The water causes a change in conductivity compared to the eluent and is, therefore, a possible cause of irreproducible results. Thus, extraction was performed with the eluent as the extraction fluid.

Application of the test solution to the swab was followed by extraction in the eluent for 90 minutes. The sample was centrifuged and the extraction fluid was transferred into a vial.

### *Swab Removed from its Plastic Support*

The manner in which a cotton swab is wrapped around the support can be a cause of decreased recovery during extraction, especially if swabs produced industrially are wrapped tightly around the support. In order to decrease their compactness, the cotton swabs were removed from the plastic holder by cutting them open. After removal of the swabs, the test solution was applied and the swabs were then placed in a tube for extraction.

### *Tissue Extraction*

Tissue paper was used as an alternative carrier for the test solution. The advantage of using tissue paper for extraction is that the surface is larger and the material is thinner and more homogeneous than a cotton bud. A disadvantage of using tissue paper for sampling is that the handling of a piece of tissue paper is less convenient than handling a cotton swab on a stick. To perform the tissue-extraction experiment, a sheet of tissue paper was cut into as small a square as possible, so that the paper could just hold the volume of test solution applied without it being saturated. The test solution was applied by holding the small piece of tissue paper in a pair of tweezers.

### *Double Extraction*

The possibility was investigated that, by repeating the extraction step, more ions could be brought into solution than with a single extraction. After application of the test solution to the swab, the swab was placed in a plastic tube for a 90-minute period of extraction. Thereafter, the swab was removed from the first tube and placed in a second tube for a further 90-minute extraction.

### *Extraction from Melinex as a Simulation of Ions on a Glass Surface*

Extraction from the Melinex surface was done by applying the same amount of test solution to a small piece of Melinex and letting it dry overnight, so that salts were present on the surface. After drying, the small pieces of Melinex were placed in an extraction tube containing 1 mL of de-ionised water for a period of 90 minutes. The centrifugation and analysis steps were the same as the basic extraction experiment.

### *Sampling of Surface Material from Vessel Glass Using a Swab*

Several objects showing various surface effects were selected for sampling. Sampling was done by swabbing 1 cm<sup>2</sup> area of the glass surface ten times back and forth. The samples were stored in a tube, and extraction was subsequently performed in the laboratory using de-ionised water. The extraction volume was 1.1 mL, so that the solution could be filtered; the filters absorbed approximately 0.1 mL of the solution that is being filtered. Prior to filtering, the cotton swabs were removed from the tubes, and the extraction fluid was centrifuged for 10 minutes.

## **Results and Discussion**

The results achieved in this study represent the conclusion of the first phase of the development and implementation of a regime for chemical analysis as a tool to aid conservators in identifying unstable glasses. A protocol has been developed whereby ion chromatography can reliably quantify low concentrations of ionic species absorbed by cotton swabs.

Validation of this protocol was necessary prior to the second phase of the project, namely ion chromatography of the ionic species extracted from swabs used to sample the surface of historic glasses displaying a range of stability. Unexpectedly, much experimentation was required to ensure that this protocol gave good recovery and reliable reproducibility.

Initial experiments focused on the ion chromatography analytical errors. Relative standard deviations for analysis of 50 ppm standard solutions of Na<sup>+</sup> and K<sup>+</sup> were 3% in both cases. For analysis of pipetted samples of 50 µL of 1000 ppm Na<sup>+</sup> solutions added to two 475 µL pipetted aliquots of water, a relative standard deviation (in this case somewhat less than 3%) indicated that the error in sample preparation can be neglected compared to the error in the determination of peak surface area from chromatograms.

Less straightforward was the extraction and quantification of ions applied to cotton swabs, the key factor that had to be optimised prior to the use of cotton swabs for sampling from the surface of historic glasses. The basic extraction of a standard solution of the four analyte ions applied to a commercially prepared cotton swab gave poor total recovery and too wide a spread of results, i.e. the reproducibility of the extraction step was inadequate. In order to give superior results, extraction was carried out in an ultrasonic bath in an attempt to free the ions from the cotton swabs; however, this experiment did not increase recovery or reproducibility. The effect of drying

the samples before extraction was evaluated by performing experiments in which the extracts of dried samples were compared to those where the swabs were placed directly in the extraction fluid after application of the test solution. This demonstrated that drying the samples had no effect on the analytical results. Thus, for sampling by conservators, the swab may be either dried or placed directly in a vial with extraction liquid prior to analysis. For simplicity, the drying step is now omitted from our sampling protocol. Experiments to investigate extraction in the acidic eluent, as opposed to distilled water, increased the recovery somewhat but did not improve the reproducibility – the recovery was generally below 70%, and the relative standard deviation was still above 10%.

Finally, satisfactory results for both recovery and reproducibility were obtained by removal of the cotton swab from its plastic holder prior to application of the test solution and extraction. This achieved recovery of more than 90%, with a reproducibility comparable to the internal error of the ion chromatography system. It is concluded that the compactness of the commercially prepared cotton swabs was such that extraction of the applied salts was inhibited. Loosening of the swab from its plastic holder enabled satisfactory extraction. The same results were obtained for analysis with tissue paper extraction. As a further refinement, a double extraction was also performed. This showed that, during a second extraction, more ions were extracted from the swab; however, because of the complexity of determining ion concentrations in the second extraction, combined with the high level of recovery in a single extraction, this does not form part of the recommended sampling protocol.

With this thorough validation in place, implementation of the protocol for historic glasses has become possible. The preliminary results, first for a simulation with salts on the surface of Melinex and then using the swab protocol for sampling a small set of unstable historic glasses, confirm the usefulness of this approach. These results will be reported in full at a later date.

## **Conclusions**

This investigation had its impetus in the glass condition survey carried out at the Rijksmuseum in preparation for its re-opening after 10 years of refurbishment. The survey resulted in the establishment of five overall categories of deterioration, representing approximately 10% of the museum's glass collection, in order to assist in prioritisation of items for display and storage.



It transpired that the numerous items where the visual signs of possible glass deterioration were minor were most problematic for the conservation decision-making process. It was concluded, therefore, that non-invasive analysis of the glass was necessary to supplement conservators' visual observations in order to identify which glasses were actually of unstable composition. Since no appropriate methods had been reported to do this, ion chromatography was identified as the most promising analytical method. The development of a protocol to extract sodium, potassium, calcium, and magnesium ions from cotton swabs was the essential first step in an overall strategy to sample surface deposits from vessel glass as indicators of glass deterioration. The successful validation of this protocol is the focus of the results presented above.

Accordingly, the research can now enter the implementation phase, already trialled with a small number of items in this initial phase, involving optimisation of the procedure for sam-

pling and analysis of surface deposits from a range of historic glass displaying different degrees of degradation.

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	Description
<b>Category A</b>	<i>Glasses in this category are in a perfect condition</i> No conservation treatment is necessary.
<b>Category B</b>	<i>Symptoms B1–B4 may concern glass degradation</i> This category contains glasses showing very slight surface changes. It is desirable to clean the glass – after cleaning most of the glasses should have the same appearance as those in category A. Glasses in this category should be monitored after cleaning to see if new surface changes have occurred.
<b>B1</b>	Very slight 'oily' appearance which may be an early stage of glass degradation, but can also be unrelated to degradation (grease, etc.)
<b>B2</b>	Very light haze, clearly visible in raking light.
<b>B3</b>	Fingerprints are visible on the surface.
<b>B4</b>	Combination of B1–B3.
<b>Category C</b>	<i>Symptoms C1–C9 may concern glass degradation</i> This category contains glasses that show probable early stages of glass degradation. Glasses that were treated previously for glass degradation and now appear stable are placed in this category. After cleaning, most glasses (except C7 and C10) should have the same appearance as those in category A. Monitoring is essential in order to detect new surface changes or other symptoms of degradation.
<b>C1</b>	Slight or incipient crizzling; tiny localised cracks on the surface.
<b>C2</b>	Slight discolouration of the glass; suspicion that a colour change has occurred over the years.
<b>C3</b>	Oily surface which may also be localised, manifesting itself as a spotted appearance. This can be an early stage of glass degradation or the presence of external deposits, such as grease.
<b>C4</b>	Harder deposits or haze (which can be scratched away using a finger nail). They may also be localised, giving a spotted appearance.
<b>C6</b>	A cavity in the glass (from the manufacturing process) in which a fine powder or light haze is visible.
<b>C7</b>	Archaeological glass that shows iridescence.

<b>C8</b>	A combination of symptoms C1–C6.
<b>C9</b>	A combination of symptoms C1–C6 and symptoms from category B.
<b>C10</b>	Results from earlier treatments and restoration such as glued cracks or fractures and fills.
<b>Category D</b>	<i>Symptoms D1–D6 concern glass degradation</i> This category includes glasses with clear symptoms of degradation. Glasses with very unstable or severely aged restoration(s) are also included in this group. It is desirable to clean the glasses (but not D5 and D8). Monitoring is essential in order to detect new surface changes or other degradation symptoms.
<b>D1</b>	Clear crizzling; tiny cracks on large areas of the glass surface.
<b>D2</b>	Clear discolouration of the glass; especially a shift towards purple or pink.
<b>D3</b>	Clear symptoms of weeping glass; small droplets forming on the surface which may appear oily.
<b>D4</b>	Very hard deposits or haze which are difficult to remove.
<b>D5</b>	Archaeological glass with pronounced iridescence.
<b>D6</b>	A combination of the symptoms in category D.
<b>D7</b>	A combination of the symptoms in categories in B, C, and D.
<b>D8</b>	Unstable or severely aged restorations.
<b>Category E</b>	<i>Symptoms E1–E3 concern glass degradation</i> This category contains glasses with crizzling that is so advanced that restoration is hardly possible. Glasses in this category can be kept only in storage – the current state is too fragile for display.
<b>E1</b>	Severe crizzling; cracks on large areas of the surface so that the structure of the object is severely affected. Possible flaking.
<b>E2</b>	A combination of different factors; crizzling, discolouration, weeping glass, possibly combined with a surface haze (irremovable) and/or an oily surface appearance.
<b>E3</b>	Severe deterioration such as fractures, complex cracks, and/or missing parts.

Table 1. Description of glass degradation categories developed prior to re-display of the Rijksmuseum vessel glass collection.

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## Authors

Norman H. Tennent

Universiteit van Amsterdam, Ateliergebouw, Hobbemastraat 22, Amsterdam 1071 ZC, the Netherlands  
normantennent@yahoo.co.uk

Bodill Lamain

Rijksmuseum, Ateliergebouw, Hobbemastraat 22, Amsterdam 1071 ZC, the Netherlands

Maarten van Bommel

Rijksdienst voor het Cultureel Erfgoed, Ateliergebouw, Hobbemastraat 22, Amsterdam 1071 ZC, the Netherlands

Guus Verhaar

Universiteit van Amsterdam, Ateliergebouw, Hobbemastraat 22, Amsterdam 1071 ZC, the Netherlands

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