

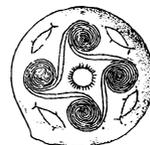
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# **Vitreous Materials in the Late Bronze Age Aegean**

Edited by

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SHEFFIELD STUDIES IN  
AEGEAN ARCHAEOLOGY



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# Crushed Rock and Molten Salt? Some Aspects of the Primary Glass Production at Qantir/Pi-Ramesse

*Thilo Rehren and Edgar B. Pusch*

## Introduction

Egyptian glass has fascinated art historians, archaeologists and scientists for well over one hundred years, mostly because of its classical elegance and often stunning preservation of polychrome patterns. The investigation of the practical production of these objects, in contrast, has been a minority interest with only a few researchers specializing in this subject (*e.g.* Stern and Schlick-Nolte 1994). Most scientific research on ancient Egyptian glass has focused on its chemical composition and the identification of the raw materials used; both are now relatively well understood. The issue of Late Bronze Age (LBA) glass production, however, has found much less scholarly attention, not least due to the absence of direct archaeological evidence for it other than the prolific presence of finished glass objects, the glass for which obviously must have been made somewhere, somehow. Notable exceptions to this lack of technical research into LBA glass production are the pioneering studies by Turner (1954; 1956), investigating material excavated in 1892 and 1893 by Sir Flinders Petrie at Amarna; by Brill and co-workers (Lilyquist and Brill 1993; Oppenheim *et al.* 1970); and recently the attempts by Nicholson (1995a; 1995b; 1996; 1997; 1998; Nicholson and Jackson 1998) to demonstrate a potential link between glass-making and two circular furnaces excavated in the early and mid 1990s in Amarna. Shortland's and Tite's analyses of relevant material from Amarna (*e.g.* Shortland 2000; Shortland and Tite 2000; Tite and Shortland 2003) over the last ten years or so have added considerably to our understanding of the relationship between glass and the other vitreous industries, in particular the production of faience and Egyptian blue. They have also identified some interesting intermediate products such as cobalt-blue frits, and the potential use of mineral natron for glass-making in the New Kingdom.

The current consensus regarding the bulk of Egyptian LBA glass production is that it is made from two main raw materials, namely specific plant ashes to provide a soda-rich flux, and a very clean silica source such as crushed quartz pebbles. These two raw materials are sufficient to explain the occurrence and concentrations of the seven main (or base) oxides present in LBA glasses as defined by Lilyquist and Brill (1993): silica, alumina and iron oxide; soda and potash; and lime and magnesia. It is also well understood that LBA glass was almost exclusively strongly coloured and mostly opaque, rather than the colourless, transparent material we associate with glass today; colouration and opacification were achieved through the addition of specific, often exotic, metal oxides.

It has been argued earlier (Rehren 1997) that the colouration of the glass was done after the initial raw glass-making and went together with the production of glass ingots, which were then passed on to other workshops for re-melting and shaping into objects. Both textual and archaeological evidence demonstrate that this transfer of glass ingots regularly took place over large distances, involving the major political powers of the time. This further underlines that the final working into objects took place at workshops or 'glass studios' geographically often separate from the primary glass-making sites, as it required rather different sets of skills and, therefore, probably different craftsmen. We define primary glass production as beginning with the procurement and preparation of the raw materials, followed by their melting to form glass, and ending with the addition of colourants and the production of monochrome glass ingots. The excavation of the Ramesside capital of Pi-Ramesse, beneath the present-day village of Qantir in the eastern Nile Delta, currently offers the most comprehensive assemblage for the study of this primary production.

## The Quest for Primary Glass-Making

Archaeological evidence demonstrates that glass first appeared in Mesopotamia, possibly half a century earlier than in Egypt (Nicholson 1993); furthermore, the stylistic criteria of the earliest Egyptian glass and related texts point to a Mesopotamian origin (Lilyquist and Brill 1993). Requests by Egyptian rulers to their counterparts in the East for glass supplies continue well into the New Kingdom, as documented in the *Amarna Letters* (Moran 1992), further corroborating the impression of a Mesopotamian-dominated glass industry, even at a time when there is ample stylistic and archaeological evidence for local Egyptian glass vessel production from Malkata, Amarna and Lisht, among others (Nicholson and Henderson 2000).

A number of essential issues, however, remain unresolved. Most fundamentally, it was until recently unknown whether Egypt was completely dependent on glass imports from 'countries to the east', or whether it was capable of producing at least some of its own glass. The latter is indicated by the identifica-

tion of Egyptian cobalt minerals as the most likely source of this dark blue colourant (Kaczmarczyk 1983), but it is not clear how these minerals were processed for their use as a glass colourant, nor does an Egyptian origin for the colourant necessarily imply Egypt-based glass production (Rehren 2001; Shortland and Tite 2000). However, some of the glass-related, frit-like materials from Amarna are consistent with local glass-making (Tite and Shortland 2003).

Unfortunately, archaeological evidence for LBA primary glass production is also completely lacking for the 'eastern countries' stretching from southern Mesopotamia to the Caucasus that are routinely implicated in this activity. This partly reflects the difficulties in identifying with modern geographical terms the place names of the historical Egyptian references to glass sources, and the possibility that the texts refer more to trade origins than to the real physical origins. However, a rather pessimistic comment in this context by Petrie may also still be valid:

... a good deal more knowledge of the history of glass might become available when Asia was really opened up for proper investigation and study, but for this to happen it might be necessary to wait for another cycle of civilization. ... and then – and probably not until then – one might really get to know, for the first time, what was the actual origin of glass-making (Petrie 1926: 233).

As a result, and with the Mesopotamian Iron Age cuneiform texts being ambiguous in their relevance for LBA glass-making practice on a number of counts (Oppenheim *et al.* 1970), we have no firm idea about either the geographical location or the technical practicalities of LBA glass-making, despite the indisputable significance of glass as a high-status and widely-distributed artificial material, possibly deeply imbued with social and religious meaning.

Over the last ten years, we have published our developing ideas of primary glass production in Ramesside Egypt's capital Pi-Ramesse, modern-day Qantir (Pusch 1999; Rehren 1997; 2000a; 2000b; Rehren and Pusch 1997; 1999; Rehren *et al.* 2001), up to the most recent, necessarily limited, discussion in Rehren and Pusch (2005). Here, we now aim to present and discuss some of the evidence for primary glass production in more detail, even though this is work in progress and any interpretation or reconstruction is therefore only tentative.

## **The Archaeological Context**

The excavation of Qantir/Pi-Ramesse is situated in Faqus Province, eastern Nile Delta, and forms the northern part of a joint Austrian-German mission, with Tell el Dab'a/Avaris in the south. The main excavation sites are placed within an area of large architectural structures, partly visible on the ground, partly revealed by large-scale geomagnetic prospection (Pusch *et al.* 1999). The material of interest here originates from three main sites within Qantir: Q I, Q IV and Q V. Pending the final stratigraphic interpretation, it appears that the glass-bearing layers in all

three sites date to the same early Ramesside period of activity in the largest high-temperature workshops yet known from anywhere in the Near and Middle East, and comprise evidence for the simultaneous production and processing of bronze, Egyptian blue, faience and glass (Rehren *et al.* 1998). They predate the re-development of this industrial complex to a predominantly military complex with extensive stables, exercise grounds for chariots, associated workshops and garrisons.

The difficulties in dealing with such a mixed assemblage of technical waste material have been discussed earlier elsewhere (Rehren *et al.* 2001), as has the identification of cylindrical crucibles as diagnostic vessels for coloured glass ingot production (Rehren 1997). Here, we focus on the hitherto unpublished evidence that we associate with the primary production of glass from its raw materials, most of which was excavated in the 1980s and again in 2000 and 2002. However, it was only fully identified during two study seasons in 2004 and 2005. A full account of the relevant finds, their archaeological context and technical interpretation has been published recently (Pusch and Rehren 2007), and related experimental work is ongoing (Merkel and Rehren 2007; Tanimoto 2007).

### *Material and Terminology*

In studying the high-temperature material from Qantir, we use a number of terms for glass-making-related materials previously unknown in Egyptian archaeology; these include reaction vessels (in contrast and addition to the cylindrical crucibles), grey parting layers (in addition to the white parting layers), semi-finished glass, and funnel-shaped rim extensions.

Reaction vessels – these occur as mostly small fragments of ovoid storage vessel, such as funnel-neck jars and beer jars (Colour Plate 2.1), coated on their interior with a white or grey parting layer and often showing signs of moderate heating and discolouration through chemical reactions. The presence of this internal coating is the defining criterion to separate reaction vessel fragments from ordinary jar fragments; in addition, quite a few fragments have the remains of semi-finished glass on top of the parting layer (Colour Plate 2.2). Analyses of the semi-finished glass are in progress and some are reported in Tanimoto (2007). Here, it suffices to say that it is a low-lime-soda glass containing abundant silica grains, in keeping with the assumption of a low melting temperature in the reaction vessels (Rehren and Pusch 2005). Due to its low lime content, the initial glass phase is particularly strongly weathered. Beneath the parting layer, the ceramic shows signs of chemical alteration, ranging from a slight discolouration, to purple, to the formation of a thin layer of dark-green interface glass. Wall fragments dominate by far; few base or rim fragments occur. The vast majority of finds are from closely-defined areas within sites Q IV and Q V.

The reaction vessels differ not only in shape from the cylindrical crucibles. They were systematically used at a much lower temperature than the crucibles,

which frequently show signs of firing right up to their thermal limit, at around 1150–1200°C (Turner 1954), although the majority were used at more moderate temperatures. Both vessel types have whitish parting layers; grey parting layers are restricted to reaction vessels. Remains of coloured glass are only known from the crucibles, where they outnumber the evidence for semi-finished glass, while the reaction vessels are almost exclusively dominated by semi-finished glass. Interestingly, the distribution of crucible and reaction vessel fragments, respectively, differs across Qantir; large crucible fragments with more than 50% of the base preserved were predominantly excavated at Q I (Colour Plate 2.3), which is otherwise characterized by huge bronze-casting installations (Pusch 1990; 1994) and where reaction vessels are virtually absent. Smaller fragments of both crucibles and reaction vessels occur more or less equally at Q IV and Q V, while the remains of semi-finished glass were predominantly found at Q V. This distribution pattern probably reflects different activity areas within the ‘industrial estate’ of Pi-Ramesse, a hypothesis which is further explored in Pusch and Rehren (2007).

Grey parting layers – these are restricted to the inside of reaction vessels. They are typically about 1mm thick, very smooth and regular in appearance, and comprise predominantly calcium carbonate with varying amounts of silica and rather lower levels of alumina, magnesia and iron oxide. They occur both on their own and overlain by semi-finished glass; in both cases they show a thin reaction zone towards the ceramic body (Colour Plate 2.4). The grey colour of the layer is probably due to the presence of charcoal dust, but this can not be demonstrated.

The white parting layers of the crucibles are of essentially the same make-up as the grey ones, but appear to have gone through higher firing temperatures, resulting in the development of much stronger reaction zones towards the ceramic (Colour Plate 2.5), more interaction with the crucible charge, and an overall more ‘worn’ and warped appearance (see below).

Semi-finished glass – this is characterized by the presence of significant quantities of quartz grains in a glassy matrix (Colour Plate 2.6), of an estimated 2–5% by volume, and locally reaching 10%. These particles are typically rather fine and often show rounded edges from their absorption into the surrounding glass melt. They are very distinct from the relatively much coarser and more spherical quartz grains of typical sand; the latter occur infrequently together with crushed quartz dust, probably as contamination rather than by intent. The relative proportion of glass matrix and quartz particles varies widely between different specimens, as does the degree of preservation of the glass matrix. Areas rich in glass appear colourless-transparent when preserved, or milky-cream white and very friable when corroded. Several specimens are rich in platy crystals, possibly wollastonite, in addition to the ubiquitous quartz dust. Notable is the almost complete absence of any colouring components in the glass, in stark contrast to the prevalence of copper in the finished quartz-free glass found both adhering to crucibles or as irregular lumps or fragments of artefacts.

Funnel-shaped rim extensions are a recently-discovered accessory to the well-known glass-colouring crucibles (Rehren 1997; Turner 1954). They are wheel-thrown collars of Nile silt with a roughly triangular cross-section. The body forms a right-angled triangle placed with its shortest side on the rim of the crucible and its hypotenuse sloping towards the interior (Colour Plate 2.7). The crucible rim is pushed securely into the soft clay. The inside or hypotenuse of the rim extension is covered with a thin layer of vitrification, often containing traces of green copper corrosion (Colour Plate 2.8).

The remainder of this paper focuses on two aspects of LBA glass production as represented in the material from Qantir. Firstly, we will discuss how the presence of residual quartz dust in the semi-finished glass enables us to characterize more closely the silica source of the glass. Secondly, the reaction temperatures and reagents active during the glass-forming process will be discussed, based on the changes which the parting layers and ceramic material of the reaction vessels have undergone.

## The Silica Source

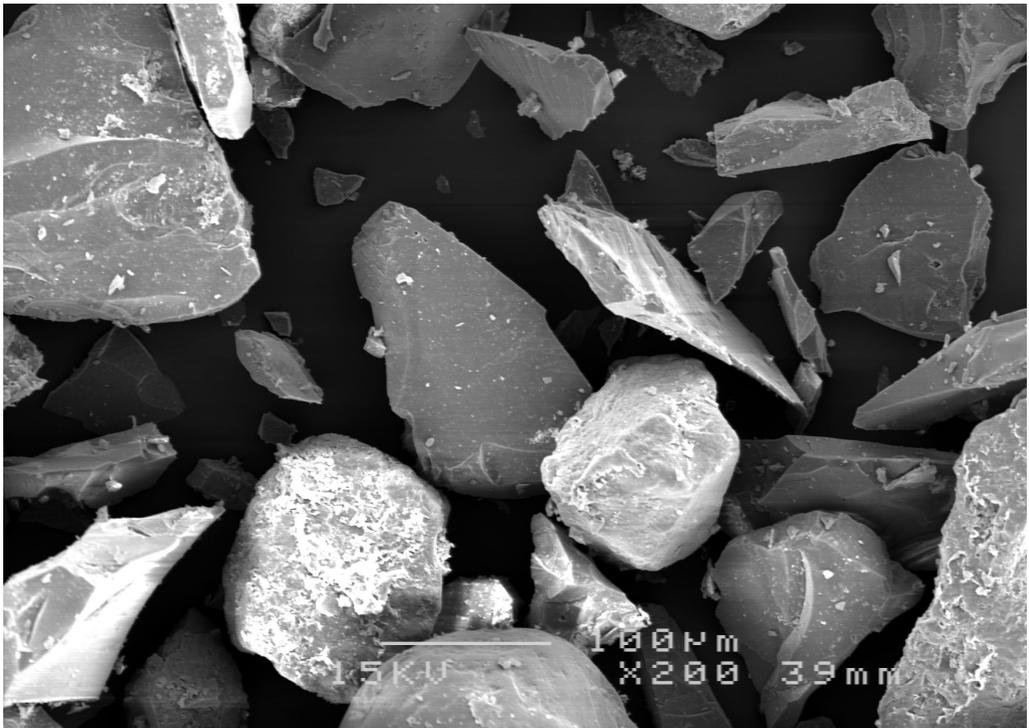
LBA glass is a chemically complex material beyond the nominal soda-lime-silica composition, typically comprising around 10 to 12 different compounds at concentrations above 0.1wt%, irrespective of colourants, and even more at levels above 50ppm. This complexity, however, is based on an apparently simple two-component formula, comprising quartz and plant ash. The ash is thought to provide both soda and lime, together with all the minor components, while quartz provides pure silica (Shortland 2005). In addition, the quartz contributes small amounts of titania and zirconia from mineral inclusions, and possibly some alumina and iron oxide, *etc.*, from the grinding stones used to produce the flour-like quartz dust (Rehren and Pusch 2007).

Ongoing work by Andrew Shortland concentrates on the characterization of the plant ash components in the glass. However, the environmental conditions in the Nile Delta are not conducive for the preservation of glass, and much of the material excavated at Qantir is largely or completely weathered due to the high and fluctuating humidity and alkalinity of the soil. This weathering affects primarily the glass phase, while any crystalline inclusions remain intact. As a result, most of the finished glass has disintegrated to hydrated silica gel and leaves little to analyze, while the presence of residual quartz grains identifies it as weathered semi-finished glass.

One of the most intriguing finds from Qantir is the well-preserved crucible 00/0344 with its content of heavily-consolidated quartz dust (Colour Plate 2.9). The crucible ceramic is dark and shows signs of strong heating, indicating use. The rim is missing, almost as if it has been deliberately knocked off, and the content in some parts reaches higher than the broken-off rim. We believe that this indicates that the content was present within the crucible, in an already

consolidated form, when the rim was removed. Thus, it is unlikely that this is a used crucible that was re-used for storage of crushed quartz. Removing a sample of the content for analysis required sustained attack with a hard, pointed metal tool and strong force. Dissolving some in hydrochloric acid resulted in some fizzing and finally left a residue of two fractions: amorphous silica gel floating in the acid and a sediment of very fine-grained, flour-like, irregular quartz particles, with only a few round sand grains in between (Figure 2.1). Semi-quantitative bulk analyses by XRF of a sample of the powder removed from the crucible totalled about 90wt%, including approximately 65–70wt% silica, just under 20wt% calcium oxide, slightly more than 1% alumina, and about 0.5% iron oxide. Trace elements included 800ppm potash, some 500–600ppm titanium oxide and about 400ppm strontium oxide. Unfortunately, the method used operates in air and is therefore unable to detect sodium and magnesium, so no information is currently available for these two important elements.

We are tempted to interpret the content of this crucible as heavily corroded semi-finished glass, from a melt probably interrupted at a very early stage of



*Figure 2.1. Micrograph of some of the content from crucible FZN 00/0344, showing finely-crushed quartz powder. The powder has been washed with HCl to remove the silica gel of the weathered glass matrix.*

formation. The amorphous silica gel could be the remains of a heavily weathered glass phase, and the sediment semi- or unreacted quartz dust. Thus, we believe that the charge was fired only for a short time or at a very low temperature, just enough to start the reaction and fuse the quartz particles together, forming only a small amount of bonded glass. The lime content is too high for a typical glass, and could be one of the reasons why this particular batch failed to react. We do, however, acknowledge that the quartz grains do not show clear signs of resorption or erosion, as one would expect if they were indeed beginning to react with the plant ash flux. Residual quartz grains in other samples of semi-finished glass are much more rounded and show typical shattering (Figure 2.2); both criteria are apparently missing here. Clearly, the large amount of quartz present in this vessel, and the sharp angularity of the quartz grains, point to a very incomplete reaction. Further work is in progress to characterize the material from this vessel more fully, by comparison with both local faience body material and the residual quartz in other semi-finished glass samples.

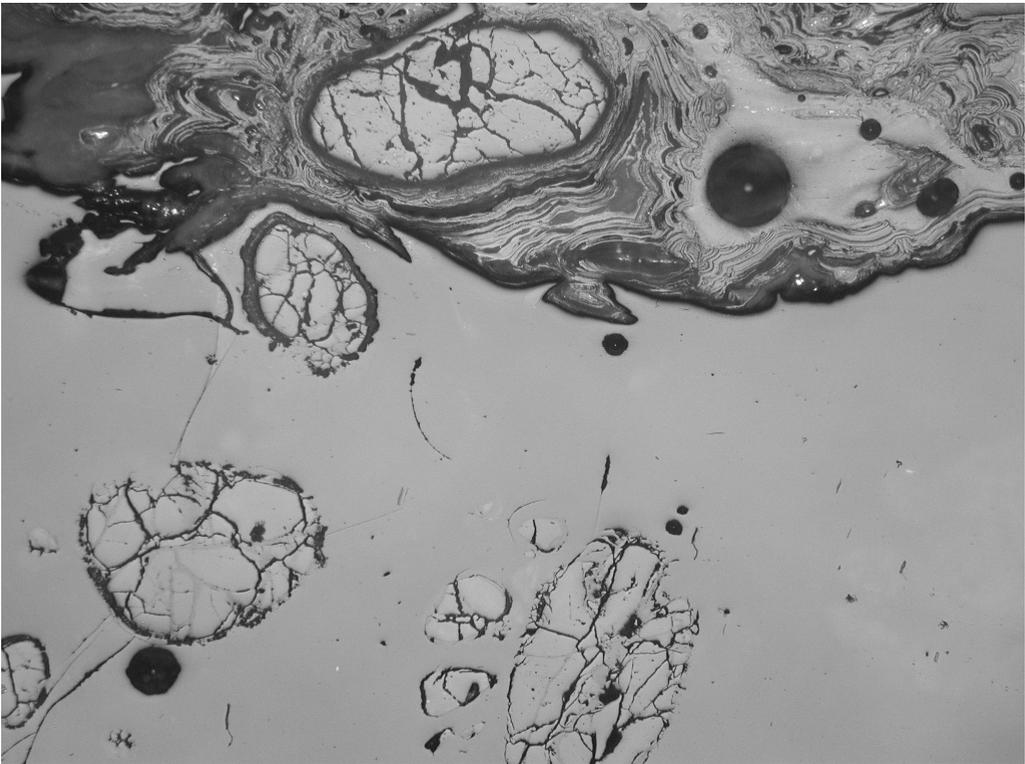


Figure 2.2. Micrograph of semi-finished glass (FZN 88/0517), showing abundant residual quartz crystals (rounded, shattered). The glass phase is partly corroded (top). Reflected light; width of image: c. 1.8mm.

A small amount of quartz powder of essentially the same characteristics as the quartz sediment above, but neither fizzing nor releasing silica gel when treated with hydrochloric acid, is preserved on another fragment, vessel 02/0329 (Colour Plate 2.10). In contrast to the crucible, this is a fragment of an ovoid storage vessel with neither a parting layer nor any signs of heating. Furthermore, the content is a very soft powder, easily removed with a soft brush. It is most likely that the content of this vessel represents the crushed quartz powder that formed part of the charge in the reaction vessels.

Further support for the assumption that these finds are related to glass-making comes from their archaeological association. Frequent finds across all excavation sites and layers in Qantir are quartz pebbles and agate nodules. They are typically egg-sized, irregularly shaped and of yellow-brown to dark brown-red colour. Some appear to have been used as polishing stones and the agate nodules in particular as raw material for bead-making. Among this overall assemblage of quartz pebbles stand out hundreds of white or almost white pebbles that occur specifically in close connection with tools such as hammer stones and grinding plates, and together with fragments of reaction vessels, crucibles and raw glass at sites Q IV and Q V (Pusch and Rehren 2007). This indicates that these white pebbles were indeed crushed locally for glass-making purposes, as first suggested by Petrie more than 100 years ago.

A specific case for this assertion can be made based on the detailed archaeological evidence (Pusch and Rehren 2007). Crucible 00/0344, excavated at site Q V, was found placed on a white-washed floor in the corner of Room 02. The floor was covered with splinters and finely-crushed fragments of limestone and charcoal dust. Next to it were other crucible fragments and several stone tools, including whetstones, hammer stones, grinding stones, *etc.*, of a range of rock types, including limestone, sandstone, granite and diorite. A flat diorite lump of about 20cm diameter had a patch of semi-finished glass attached to it, as if it had dripped onto it while hot. Finally, quantities of white quartz pebbles were found scattered across the room. These and similar findings in the immediately adjacent room give the impression of a 'cold' workshop, where limestone fragments and quartz pebbles were crushed and ground to powder. Fragments of alabaster inlays were also found among the waste, indicating that these were being made here as part of the faience factory. Alternatively, they may have been used as a clean raw material for the lime powder employed in the parting layer (see below). The situation is quite different in the area immediately to the east of these two rooms; here, the finds are predominantly 'hot', with numerous crucible fragments (with and without glass attached), various types of glass slag, and burned and baked clay and mudbrick fragments. Again, crushed limestone particles are frequent finds here. Unfortunately, later deep-cutting disturbance during the redevelopment of the area has destroyed many of the *in situ* remains of this workshop, and no identifiable furnace remains were found.

Another possible source of the quartz dust used in the glass-making process

is indicated by the occurrence of gold prills in some of the red glass from Qantir (authors' own unpublished data). They spin a tantalisingly thin thread to the contemporary revival of gold-mining activity in the Wadi Hamamat and elsewhere in the Eastern Desert, where huge quantities of gold-bearing quartz were finely ground to release the gold flakes; the possibility exists that some of the tailings of finely-crushed quartz dust from the gold mines were later put to good use in the glassworks at Pi-Ramesse. After all, both Ramesses II and his father Sethos I not only set up the new capital of Pi-Ramesse in the Nile Delta, but were also heavily engaged in developing Egypt's gold-mining activities (Klemm *et al.* 2001).

## Glass-Making Evidence

We have previously stated that the plant ash component is typically not preserved in the heavily-corroded Qantir glasses, due to the environmental conditions in the Delta. However, some indirect evidence from the reaction vessels and crucibles gives a strong indication that a soda-rich and very reactive component was indeed present during the use of these vessels. The evidence for this is preserved in the parting layer in the crucibles and reaction vessels, and particularly in those samples that have developed the underlying dark-green interface glass. A detailed analysis of the various zones or layers in the ceramic towards the inner of the vessels is ongoing (Merkel and Rehren 2007; Schoer and Rehren 2007); the results so far already give us some technically relevant information concerning their function and origin.

### *Parting Layers and Interface Glass*

Previous work had already identified the parting layers on the inside of crucibles from both Amarna (Turner 1954) and Qantir (Rehren 1997) as a thin, lime-based coat. Its main function was most likely to separate the glass melt from the ceramic of the crucibles to prevent discolouration of the glass by the ferruginous ceramic, and to facilitate removal of the glass ingots from the crucibles. The identification of the parting layer in the reaction vessels from Qantir raises questions as to whether the same function applies here too and whether, therefore, the reaction vessels and the crucibles can indeed be seen as representing two steps in a sequential operation as postulated in Rehren and Pusch (2005), and as indicated by their joint occurrence and the strong similarities in the parting layer and use traces.

To determine the relationship between the crucibles and the reaction vessels, and to understand better the grey variety of parting layer that is restricted in occurrence to the reaction vessels, we first characterized more closely both the grey and white parting layers (Table 2.1). We chose two different fragments (98/0785<sub>,0006</sub> and <sub>,0008</sub>; see Colour Plate 2.4) with a smooth grey parting layer; they

were visibly low-fired and did not show a layer of interface glass. In contrast, two other samples (96/0706<sub>,0001</sub> and <sub>,0002</sub>; see Colour Plate 2.5) had both a parting layer comprising two slightly different-coloured sub-layers (labelled white and brown in Table 2.1, in exaggeration of their real colour), and a film of interface glass between the layer and the ceramic.

As can be seen in Table 2.1, silica and lime are the dominant constituents in the white and grey layers analyzed, although the compositions are quite variable, even within the individual layers. The given values are averages of multiple area analyses within each distinct layer, and the individual area analyses within each sample are very variable in their silica and lime proportions (see below; Table 2.5). Magnesia is consistently present as a minor component. Alumina and iron oxide occur in the low percentage range, possibly due to contamination with clay or ceramic in the raw material used for the preparation of the parting layers.

Comparison between the grey and white layers reveals no material differences; they are virtually identical except for their colour. We therefore assume that the grey colour is due to fine charcoal dust, which probably simply burned out in the more highly-fired layers, turning them white.

The brown layers, in contrast, are much richer in alumina, iron oxide, titania, soda and phosphate; they resemble typical ceramic compositions. The average compositions of the ceramic of the reaction vessels are given in the final row of Table 2.1. They are ferruginous, mildly calcareous ceramics with relatively low alumina content. The levels of soda and potash are similar, around 3wt% each, and concentrations of phosphate and titania are relatively high. The composition of the brown sub-layers in the two samples 96/0706<sub>,0001</sub> and <sub>,0002</sub> is best explained as a mixture of the underlying ceramic and the white parting layer material, but

<i>RV Parting Layer</i>	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
98/0785 <sub>,0006</sub> grey	41.2	-	3.4	2.5	47.7	3.9	0.5	0.1	0.2
98/0785 <sub>,0008</sub> grey	24.1	-	1.5	1.3	68.1	3.0	0.1	0.1	0.7
96/0706 <sub>,0001</sub> white	30.8	-	2.1	1.3	57.8	5.4	0.2	0.3	0.8
96/0706 <sub>,0001</sub> brown	36.7	0.7	10.8	8.6	33.7	3.0	0.6	2.9	2.2
96/0706 <sub>,0002</sub> white	44.4	-	1.7	1.0	45.8	5.4	0.2	-	0.6
96/0706 <sub>,0002</sub> brown	46.9	1.9	11.3	9.2	22.4	3.8	0.8	1.9	1.0
<b><i>Ceramic Average</i></b>	<b>58.4</b>	<b>1.7</b>	<b>14.8</b>	<b>11.1</b>	<b>4.6</b>	<b>2.1</b>	<b>2.9</b>	<b>2.5</b>	<b>1.8</b>

Table 2.1. Average compositions of parting layers in some reaction vessels from Qantir (first six data rows), and average ceramic composition of these reaction vessels (final data row). SEM-EDS data, normalised to 100wt% to account for porosity; some minor components have been omitted from the table. (All analyses after Schoer 2004.)

with depleted potash content. Research is continuing to understand how this apparent mixing of ceramic matrix and parting layer material is to be explained, and how this brown layer typical of the reaction vessels relates to the interface glass of the crucibles and some reaction vessels.

The layer of dark-green or interface glass between the parting layer and the ceramic was noted early on in our investigation (Rehren 1997), and its degree of development taken as a measure of intensity of heating (Rehren and Pusch 2005). However, no detailed analysis of it was then undertaken. Our field hypothesis was that the interface glass was the product of *in situ* reaction between the ceramic and the lime-based parting layer; after all, these are the two materials sandwiching the interface glass, and lime is a fluxing agent for ferruginous ceramics. In this context, it has to be stressed that the interface glass is typically more strongly developed in the crucibles than the reaction vessels. To test further our hypothesis of a direct technological link between the crucibles and the reaction vessels, in the sense of one being a precursor to the other in a sequential operation, we analyzed interface glasses from both vessel types. The results are given in Table 2.2, with separate averages for crucibles and reaction vessels, as well as the average ceramic composition for comparison, as in Table 2.1.

Two important observations emerge from these data. First, the interface glass is substantially the same for crucibles and reaction vessels, even if it is less well developed in the latter vessel type. Therefore, we may assume that the two vessel

		SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
Crucibles	96/0076 <sub>,0001</sub>	55.4	1.3	11.8	7.8	5.6	2.2	3.4	10.7	1.1
	83/0660c <sub>,0001</sub>	57.3	1.4	11.4	7.4	3.9	3.2	3.1	11.2	0.3
	98/0785 <sub>,0005</sub>	54.3	1.5	11.7	9.9	3.4	2.4	2.6	12.4	1.7
	84/1373b	55.4	1.2	12.6	6.3	4.9	2.7	3.8	11.0	1.1
	<i>Average</i>	<b>55.6</b>	<b>1.4</b>	<b>11.9</b>	<b>7.9</b>	<b>4.5</b>	<b>2.6</b>	<b>3.2</b>	<b>11.3</b>	<b>1.1</b>
Reaction Vessels	96/0706 <sub>,0001</sub>	54.2	1.4	10.6	8.4	5.0	1.9	3.7	13.1	0.0
	96/0706 <sub>,0002</sub>	58.8	0.9	15.9	3.5	4.2	2.3	2.7	10.9	0.0
	<i>Average</i>	<b>56.5</b>	<b>1.2</b>	<b>13.3</b>	<b>6.0</b>	<b>4.6</b>	<b>2.1</b>	<b>3.2</b>	<b>12.0</b>	<b>0.0</b>
	<i>Ceramic (RV) Average</i>	<b>58.4</b>	<b>1.7</b>	<b>14.8</b>	<b>11.1</b>	<b>4.6</b>	<b>2.1</b>	<b>2.9</b>	<b>2.5</b>	<b>1.8</b>

Table 2.2. Composition of the interface glass from crucibles and reaction vessels. SEM-EDS data, normalised to 100wt% to account for porosity; some minor components have been omitted from the table. (All analyses after Schoer 2004.)

types were indeed used for similar technical operations, although at different intensity. Second, looking at the lime content of the interface glass immediately eliminates the assumption of it being the result of fusion of parting layer and ceramic material. The level of lime in the interface glass is almost identical to the lime level in the ceramic and is, therefore, far too low, at slightly above 4wt%, to be significantly influenced by the parting layer, which has well over 50wt% CaO. The major chemical aspect of the interface glass distinguishing it from the ceramic compositions is its soda content, which indicates a strong contribution to its formation from the glass charge in these vessels.

Thus, if the interface glass is not the result of a reaction between the ceramic and the parting layer, could it represent mixing or fusing of the ceramic with the glass itself? This is indicated by the intermediate soda content and the glassy nature of the interface glass. To explore this possibility we compared the composition of the various materials. Table 2.3 juxtaposes the average compositions of the crucible fabrics and the related interface glasses, the absolute differences between these values, and the average base glass compositions of all analyzed Qantir glasses.

It is obvious that the interface layer is not the result of a simple fusion of roughly equal amounts of ceramic and glass, despite the suggestive intermediate soda concentration of the interface glass. If this had been the case, the concentrations of all the other oxides should also be about halfway between their concentrations in the ceramic and the base glass, respectively. This is clearly not the case. Rather, the alkali and earth alkali oxides are all more or less enriched in the interface glass compared to the ceramic, while the other oxides are all lower, as if diluted by this addition. Probably significantly, the interface glass is most depleted in iron oxide, relative to the original ceramic composition.

	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
<b>Ceramic (Crucible)</b>	59.5	1.5	14.2	10.5	4.3	2.2	2.7	3.2	1.8
<b>Interface Glass</b>	55.6	1.4	11.9	7.9	4.5	2.6	3.2	11.3	1.1
Difference	-3.9	-0.1	-2.3	-2.6	+0.2	+0.4	+0.5	+8.1	-0.7
<b>Base Glass</b>	66.9	-	1.2	0.6	7.5	4.1	1.8	17.9	0.2

Table 2.3. Average compositions of ceramic and interface glass in the analysed crucibles, their difference in absolute terms, and the average base glass compositions of the analysed glasses from Qantir. Based on SEM-EDS and EPMA data, normalised to 100wt% to account for porosity; some minor components have been omitted from the table. (All analyses after Schoer 2004.)

### *A Fluid Soda Phase?*

Comparison of the composition of parting layers, interface glass, ceramic bodies and glass charge indicates that the interface glass is based on the underlying ceramic, but is neither mixed directly with the parting layer nor the glass. Instead, a very soda-rich phase appears to be the main component reacting with the ceramic to form the interface glass.

We interpret the interface glass composition as indicating that the crucible charge oozed a very fluid phase rich in alkali oxides which penetrated the chemically inert but very porous parting layer into the ceramic. On contact, this phase reacted with the ceramic to form the interface glass. Most remarkable in this respect is the increase in potash in the interface glass compared to the ceramic composition, given that the potash concentrations in the glass charge are even lower than those of the ceramic. If this increase is indeed due to the existence of a fluid alkali-rich phase, then this phase would have to draw potash preferentially from the glass charge and deposit it in the ceramic. We do not believe that finished soda-lime-silica glass exudes such alkali-rich vapours upon simple melting at *c.* 1000°C, and assume instead that this enrichment indicates the presence in the crucibles of raw plant ash. This is known to contain varying but generally significant amounts of chlorides and sulphates (Brill 1999) which form a highly fluid melt separate from the more viscous silicate glass. This aspect of the glass-making process clearly requires detailed experimental research before a technical interpretation can be given. Such work has recently been undertaken (Merkel and Rehren 2007; Tanimoto 2007) and the results so far already show that the salt phase is enriched in potash relative to a co-existing soda-lime-silicate melt, passes easily through the parting layer, and reacts with the underlying ceramic in a way very similar to that seen in the archaeological material. Melting the same glass batch without sodium chloride being present, in contrast, results in virtually no change in the appearance of the ceramic beneath the parting layer (Merkel and Rehren 2007).

A series of stratified SEM-EDS analyses, across the profile of the two samples without fully developed interface glass, from the main ceramic body into the grey parting layer, allows us to trace the movement of this alkali phase further. In Table 2.4, data from both series are arranged with the parting layer (separated into lime-rich and silica-rich areas) shown in the first two data rows, followed by the sequence of layers identified visually through to the main ceramic body (bottom row).

Several observations stand out. Beginning with the different areas in the parting layers, it seems that the magnesia content is not coupled to the lime component, but linked to the silica-rich phase. Iron oxide and alumina are also richer in these areas than in the lime-rich parts. The latter two oxides could indicate the presence (by contamination) of a minor ceramic component in these areas; the magnesia level, however, is much higher than in any other part of the vessels. Virtually no alkali oxides were found in any of the parting layer phases.

98/0785 <sub>,0006</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
Ca-rich areas	4.4	-	0.1	0.3	94.2	1.0	-	-	-
Si-rich areas	61.6	-	5.3	3.7	21.9	5.5	0.8	0.2	0.4
pale yellow band	52.5	1.7	17.0	5.6	11.2	2.0	1.8	7.1	-
thin dark band	54.8	1.7	18.2	7.1	4.6	1.7	2.7	9.1	-
reaction rim	59.0	1.3	14.4	9.5	3.5	2.3	3.7	4.2	1.8
ceramic	60.2	1.5	16.0	10.1	3.6	2.2	3.2	2.5	0.7

98/0785 <sub>,0008</sub>	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>
Ca-rich areas	1.0	-	-	-	97.7	1.3	-	-	-
Si-rich areas	51.8	-	3.4	2.3	32.6	5.1	0.3	-	1.5
thin dark band	48.7	0.3	9.6	4.4	22.3	1.7	1.9	2.3	7.1
reaction rim	61.7	1.5	13.5	9.0	3.2	1.8	4.0	4.2	1.2
ceramic	59.1	1.6	14.9	13.0	3.5	1.9	2.9	2.0	1.0

Table 2.4. Individual compositions of different areas and sub-layers in two reaction vessels from Quantir. SEM-EDS data, normalised to 100wt% to account for porosity; some minor components have been omitted from the table. See text for details and Table 2.1 for average composition of the grey parting layer. (All analyses after Schoer 2004.)

The transition to the ceramic, indicated chemically by the appearance of titania and visible microscopically from the appearance of quartz grains, goes together with an apparent penetration of lime (but not magnesia) from the parting layer into the ceramic. The main feature, however, is the gradual increase in iron oxide across the intermediate layers, from rather low levels, in contrast to the abrupt emergence of silica and alumina at the levels present in the unchanged ceramic. Under the optical microscope (Colour Plates 2.11a, b), the transition is marked by the very porous appearance of the iron-depleted layer (centre, with sand grains clearly visible), and the fully vitrified area beneath. The main ceramic body of this sample (not shown here) is far less vitrified, although the vessel was clearly heated from the outside and the temperature, therefore, must have been the same across the full profile. Hence, differences in degree of vitrification are certain to reflect different chemical compositions, in this case increased presence of a flux such as soda. The upper sequence in Table 2.4 appears to be slightly more developed than the lower one; it shows an embryonic soda-rich layer in the thin dark band, which we interpret as a precursor of the true interface glass.

One possible mechanism to explain such a sequence includes the permeation of liquid sodium chloride through the porous and chemically inert parting layer; on contact with the ceramic, the sodium chloride reacts with the iron oxide to form volatile iron chloride and sodium oxide. The iron chloride migrates outwards, resulting in an iron-depleted zone (pale yellow band) followed by purple discolouration of the inner part of the ceramic body, while the soda reacts *in situ* with the remaining ceramic material to form a low-melting soda-alumina-silica glass. This in turn absorbs more of the surrounding ceramic, including any iron oxide still present, forming the dark-green glass we see in the fully-developed interface glass. In this scenario, the driving agent is not the sodium carbonate component of the glass, but a sodium chloride melt that is fluid enough to penetrate through the parting layer, while the glass melt is far too viscous to do so.

These investigations may appear rather technical and of little significance to the archaeological interpretation of the reaction vessels and crucibles from Qantir. However, we argue that sodium chloride is more likely to be present in primary glass-making batches, but mostly absent from finished glass. Indeed, typical analyses of LBA glasses consistently give chlorine concentrations of only around or below 1wt%, and experimental work by Gerth and co-workers (Gerth *et al.* 1998) has shown that evaporation of chlorine from such glasses is negligible at temperatures below *c.* 1400°C. We, therefore, wondered whether the proposed activity of a soda-rich volatile phase could be shown to be restricted to those vessels in which glass was actually made from its raw materials, and possibly also those in which the semi-finished glass was re-melted to form coloured ingots. A series of experimental melts of glass-making batches with and without sodium chloride was undertaken using modern, locally-made Nile silt vessels and controlled raw materials (Merkel and Rehren 2007). This showed that the forma-

tion of the interface glass is restricted to glass-making as opposed to glass-melting. Its presence in crucibles and reaction vessels is, therefore, a powerful diagnostic criterion for the positive identification of ancient glass-making from the analysis of crucible fragments, even if the direct evidence, such as semi-finished glass or raw materials, has been lost through recycling in antiquity, or weathering. The interface glass, by virtue of its extremely high alumina content, appears to be almost indestructible even in the humid and aggressive conditions of burial in the Nile Delta and, in combination with diagnostic vessel fragments, is potentially a prime witness of past glass-making.

## Conclusion

Investigation of semi-finished glass has confirmed crushed quartz as the main source of silica for the New Kingdom glass-making workshops at Pi-Ramesse, as opposed to the use of sand as previously thought (most recently Nicholson and Henderson 2000). The archaeological association of much of the semi-finished glass and the reaction vessels with clusters of white quartz pebbles and tools for smashing and grinding (Pusch and Rehren 2007) indicates that Petrie's (1926) original assumption, that such quartz pebbles probably were the main silica source, is still valid. An alternative source for finely-ground quartz is the large-scale gold mining and beneficiation activity of the New Kingdom, and the occurrence of tiny gold prills in some red glasses from Qantir points to this potential origin.

The question of how to identify and localize LBA primary glass production in the archaeological record, the main focus of this paper, seems to be convincingly answered with the widespread and systematic occurrence of semi-finished glass in the reaction vessels. Further indirect evidence for primary glass-making as opposed to glass-working comes from the presence of chemical and colour changes in the ceramic immediately beneath the parting layers of both reaction vessels and cylindrical crucibles. We interpret these as the result of the activity of a salt phase (galle), as is known to exist in most plant ashes (Brill 1999; Busz and Sengele 1999), but which is not likely to be part of the finished glass. The exact mechanism which leads from the initial discolouration of near-contact parts of the ceramic to the eventual formation of a solid layer of dark green interface glass is still only partly understood, and appears to comprise a complex sequence of reactions. The exact details of these may seem of only limited significance for archaeological science; eventually, however, they may possibly lead to the development of diagnostic criteria for the identification of LBA glass-making activity in Egypt, Mesopotamia and beyond. A tailored programme of experiments is underway to explore this further.

## Acknowledgements

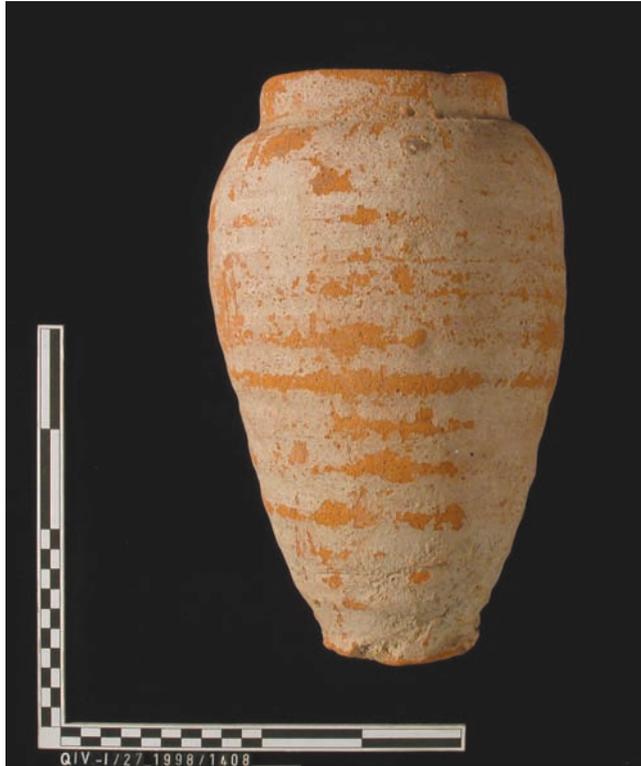
We are very grateful for years of support from the SCA and their representatives at the excavation in Qantir. Without them, we would not have been able to conduct the necessary analyses, including sectioning, chemical testing and microscopic analysis. The unfailing contributions of generations of Egyptian and European team members in all their various roles now enable us to link finds from the early 1980s to their technical counterparts excavated in 2002, finally bringing some answers to the many interesting questions arising from such a complex project. Birgit Schoer is thanked for her ongoing contributions to the analysis of the glass-making material, and her detailed documentation of the parting layers and reaction zones forms the basis of much of our interpretation here. The referees' comments on an earlier text provided helpful criticism, further stimulating our own thoughts. The long-term support for the excavations at Qantir, from the Deutsche Forschungsgemeinschaft since 1980 and though a small grant from the British Academy towards some of the glass-related work in 2002, is gratefully acknowledged.

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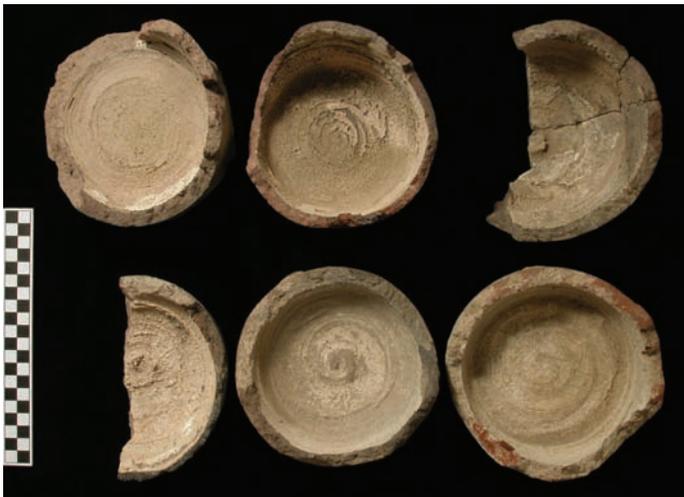
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Colour Plate 2.1. Example of a beer jar from Qantir. Vessels of this type were adapted for use as reaction vessels in glass-making.



Colour Plate 2.3. Collection of various well-preserved crucible fragments from Qantir site Q I.



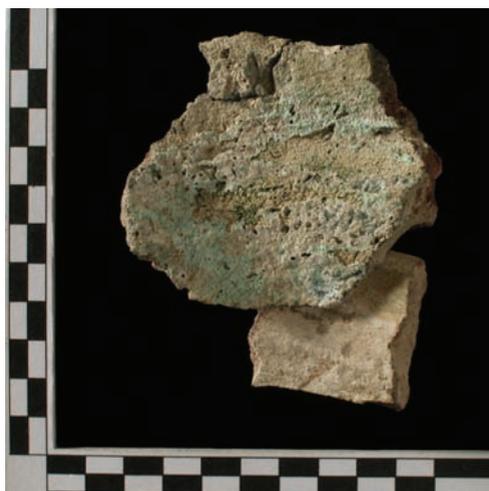
Colour Plate 2.2. Close-up of semi-finished glass on a reaction vessel fragment (FZN 98/0785). Width of image: c. 1cm.



Colour Plate 2.4. Polished cross-section through a reaction vessel fragment with a grey parting layer (FZN 98/0785<sub>,0006</sub>).



Colour Plate 2.5. Polished cross-section through a reaction vessel fragment with a white parting layer and well-developed interface glass (FZN 96/0706<sub>,0002</sub>).



Colour Plate 2.8. Front view of a funnel-shaped rim extension (FZN 00/0166<sub>,0001</sub>) mounted on a crucible rim.



Colour Plate 2.6. Close-up of a residual quartz grain in semi-finished glass (FZN 98/0785).



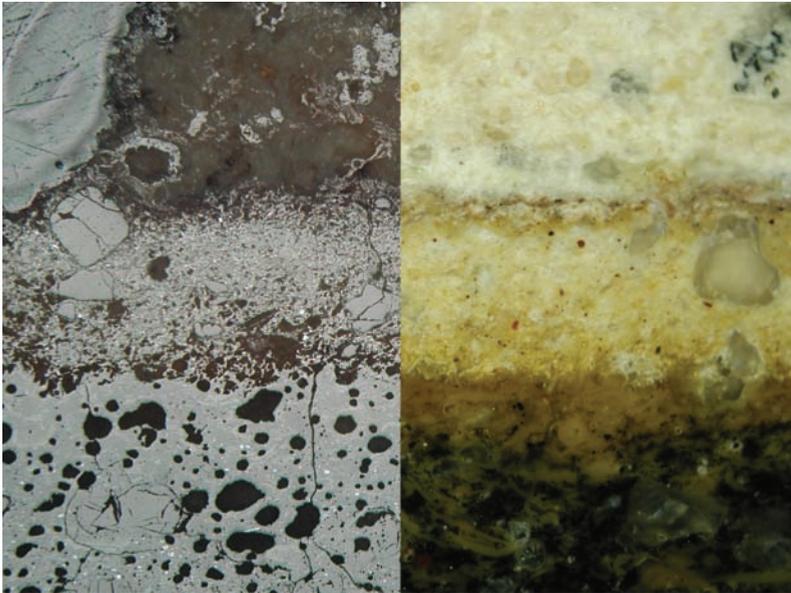
Colour Plate 2.7. Side view of a funnel-shaped rim extension (FZN 00/0166<sub>,0001</sub>) mounted on a crucible rim.



Colour Plate 2.9. Crucible FZN 00/0344 with its preserved filling of heavily-corroded semi-finished glass. The quarter segment to the rear has been removed to test the internal structure of the filling.



Colour Plate 2.10. Fragment of a transport or storage jar containing finely-ground quartz dust, but without any silica gel or other traces of weathered glass, and without a parting layer on the inside of the vessel (FZN 02/0339<sub>,0025</sub>).



Colour Plate 2.11. Composite micrograph of a typical cross-section through a reaction vessel fragment. There are three distinct layers visible, namely the parting layer (top), followed by an iron-depleted zone (centre), and an enrichment zone with elevated soda levels (below). The left half is photographed with plain polarized reflected light, the right half with crossed polarizers to show the internal reflection colours of the ceramic material. Note the highly porous (left) and white (right) appearance of the parting layer, in contrast to the generally much finer porous structure and yellower colour of the central iron-depleted zone. The underlying vitrified ceramic has a dense glassy matrix and is much richer in iron oxide. The original surface of the ceramic body is indicated by the sand grains in the depletion zone (far right and left, and centre) but not in the parting layer. Width of image: c. 3.5mm.