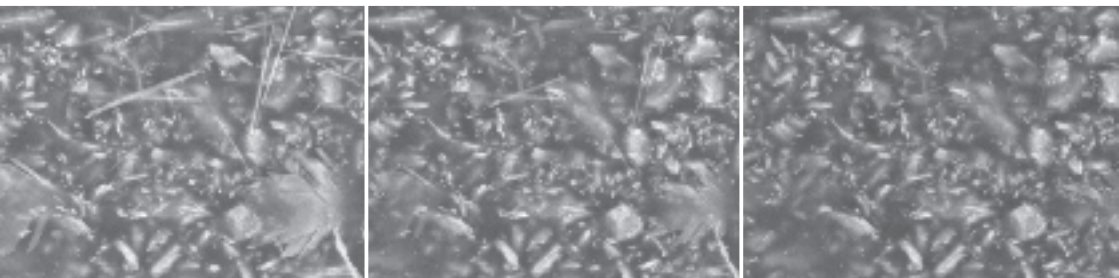


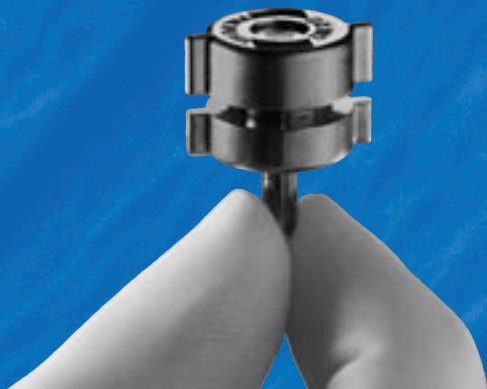
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QUANTOMiX™



QX-202C

User Manual



QUANTOMiX™

QX-202C

User Manual

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The information provided in this User Manual is believed to be accurate. It is the user's responsibility to confirm the technical aspects and the suitability of the technology for any particular application.

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Table of Contents

List of Figures	4
List of Tables	4
Cited Registered Trademarks	4
Chapter 1: Safety	5
Chapter 2: Introduction	6
2.1 Manual Scope and Contents.....	6
2.2 References.....	6
Chapter 3: Technology and Components	7
3.1 The QX-202C Capsule.....	7
3.2 The Capsule Plate (MP-12).....	10
3.3 The Applicator.....	11
3.4 Imaging Buffer (IB-64).....	11
3.5 Calibration Capsule (RT-56).....	12
Chapter 4: Using the QX-202C Capsules	13
4.1 Getting Started.....	13
4.2 Sample Preparation.....	14
4.3 Inserting Samples into the QX-202C Capsule.....	15
4.4 Storing Specimens in the QX-202C Capsule.....	16
Chapter 5: Imaging	17
5.1 Optimal Imaging Conditions.....	17
5.2 Recommendations for SEM Imaging with QX-202C Capsules.....	19
5.3 Recommendations for EDS Analysis with QX-202C Capsules.....	20
Chapter 6: Appendices	23
6.1 Appendix A: Glossary.....	23
6.2 Appendix B: Troubleshooting.....	24
6.3 Appendix C: Ordering Information.....	26
6.4 Appendix D: Legal Notices.....	27

List of Figures

Figure 1: The QX-202C capsule	8
Figure 2: Sealed capsule - dimensions	8
Figure 3: Positioning the QX-202C capsule in the capsule plate	9
Figure 4: Correct positioning of the rubber seal.....	9
Figure 5: The capsule plate.....	10
Figure 6: Opening the QX-202C capsule.....	13

List of Tables

Table 1: Recommendations for SEM Imaging with QX-202C capsules..	19
Table 2: Troubleshooting	24

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Microman® (Gilson® S.A.S.)

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Chapter 1: Safety



Cautions

Please observe the following cautions while using the QuantomiX QX-202C capsules:

1. Correct sealing of the capsule is essential for its proper functioning. Capsule sealing is achieved when the wings of the sample dish and the sealing stub are aligned.
2. The top surface of the sample dish must be kept clean in order to ensure proper sealing.
3. The QX-202C sealing stub includes a rubber seal. If the rubber seal accidentally detaches from the sealing stub, it should be re-positioned with the narrow cylinder towards the sample dish.
4. Use powder-free gloves to maintain cleanness of the QX-202C capsules. Powdered gloves should be avoided.
5. Do not place the sample dish or the capsule with the capsule membrane facing down, except in the capsule plate. To prevent rupture, avoid touching the capsule membrane at all stages.
6. Capsule plate must be kept clean.
7. Extreme care must be exercised when using the Microman® M25 supplied with the QX-202C Starter Kit: when applying materials into the capsule, the piston protrudes from the capillary and may potentially damage the capsule membrane. Keep the Microman® tip and piston at a safe distance from the membrane.

Chapter 2: Introduction

Imaging the micro-dynamics of hydration processes in their early stages has been a long-needed capability. The WETSEM™ technology developed by QuantomiX combines the resolution that characterizes Scanning Electron Microscopes (SEM) with the ability to image water-containing mixtures.

The technology is based on a thin, electron-transparent membrane that completely isolates the sample from the vacuum in the microscope chamber. Samples are simply placed in the capsule. The capsule is sealed, placed in the SEM and imaged.

2.1 Manual Scope and Contents

This manual provides a detailed description of the components required for using the QX-202C capsules. It also provides guidelines for applying samples into the capsules and for SEM imaging.

2.2 References

2.2.1 Sites

<http://www.quantomix.com>

2.2.2 Technical Support

For technical support please contact your local distributor or tech@quantomix.com.

Chapter 3: Technology and Components

The WETSEM™ technology is a proprietary technology developed by QuantomiX Ltd., which enables direct imaging of fully hydrated samples in scanning electron microscopes (SEM). The patented technology is based on a thin, electron-transparent membrane, which seals the sample from the vacuum in the microscope chamber. No freezing or drying of the sample is required, enabling the easy visualization of the dynamics of hydration processes.

The QX-202C capsules can be used for imaging the dynamics of cement, gypsum or other materials' hydration processes.

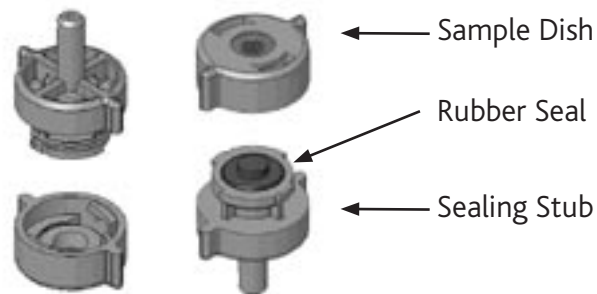
The QuantomiX components required for using the technology are listed below.

- QX-202C capsules
- Capsule plate (MP-12)
- Applicator (Microman® M25 and tips)
- Calibration Capsule and Imaging Buffer

3.1 The QX-202C Capsule

The QX-202C capsule shown in Figure 1 is a single use specimen enclosure consisting of a sample dish and a sealing stub. The base of the capsule is the sample dish. It is designed as a small dish in which samples can be placed. The top of the capsule (the sealing stub) is designed to seal the sample.

Figure 1.
The QX-202C capsule



3.1.1 Technical Data

Storage The capsules should be stored in a dry, dark environment at room temperature.

Shelf life 18 months from specified production date (printed on the box).

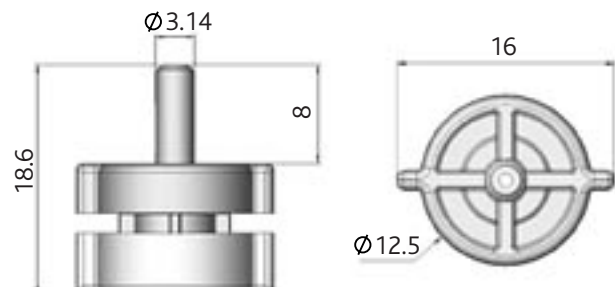
Application The capsules are intended for single use and are not reusable. They are intended for research purposes only. The QX-202C capsule is suitable for imaging a variety of dynamic reactions.

Dimensions Sample dish - diameter 3mm
Working volume - 6.5-7.5µl
Sealed capsule - see Figure 2. In cases where the QX-202C capsule does not fit your SEM stage, inquire about available adaptors.

Operation

Temperature 4° to 40° C

Figure 2.
Sealed capsule - dimensions



3.1.2 Use of the QX-202C Capsule

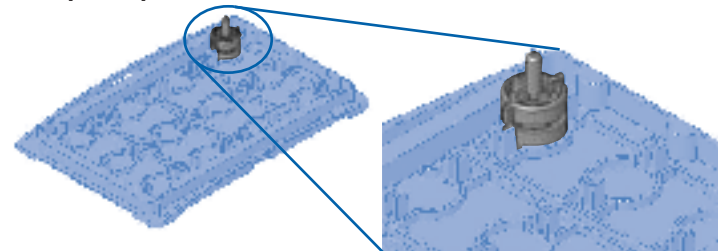
Detailed instructions on the use of the QX-202C capsule are given in Chapter 4.



Cautions

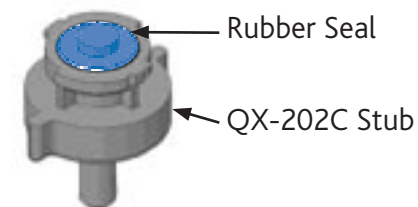
- a. The capsule should be placed in the capsule plate (as seen in Figure 3).

Figure 3. Positioning of the QX-202C capsule in the capsule plate



- b. Correct sealing of the capsule is essential for its proper functioning. Capsule sealing is achieved when the top surface of the sample dish is clean and the wings of the sample dish and the sealing stub are aligned.
- c. Prior to sealing the capsule, ensure the rubber seal is properly positioned in the sealing stub (as seen in Figure 4)

Figure 4.
Position of the rubber seal

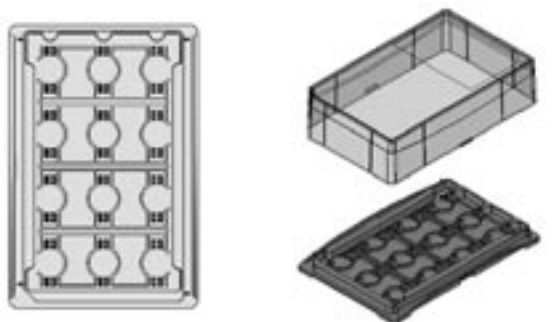


- d. Do not use sharp objects, such as sharp-ended forceps, to hold the rubber seal, in order to prevent damage to the sealing.

3.2 The Capsule Plate (MP-12)

The QX capsule plate is a clear plastic holder for the capsules (see Figure 5). It is designed to hold the capsules during specimen preparation, ensuring the capsule membrane is not damaged, and is also used for storage of capsules.

Figure 5.
The Capsule Plate



Capsule plate, as seen from above

Capsule plate and lid

3.2.1 Technical Data

Storage	Should be stored in a dry, dark environment at room temperature.
Shelf Life	Three years from specified production date.
Application	The plates should be used for all stages during which capsules are being handled.
Dimensions	85 x 128 x 33 mm
Operation Temperature	4° to 40°C

3.3 The Applicator

Supplied with the QX-202C Starter Kit is a Gilson® Microman® pipette which is specially designed to accurately displace small quantities of viscous solutions and mixtures. Also supplied is a box of single-use capillaries and pistons which are used in conjunction with the Microman.



Note

Enclosed in the Microman box is a warranty registration card. To ensure registration of warranty, please complete and return this card, by mail or fax, to QuantomiX.

For technical details and operating instructions please refer to the enclosed Microman datasheet.

3.4 Imaging Buffer (IB-64)

QX Imaging Buffer (Cat. No. IB-64) is a solution required for the imaging of the Calibration Capsule

3.4.1 Technical Data

Storage	The lyophilized product is stored at room temperature. After reconstitution, store in the dark at 4°C. The reconstituted solution is stable for 1 month at 4°C.
Shelf Life	See expiry date printed on the bottle.

3.4.2 Use of the Imaging Buffer

Use the Microman to add 15 µl of Imaging Buffer to the Calibration Capsule prior to SEM imaging.

3.5 Calibration Capsule (RT-56)

The Calibration Capsule (Cat. No. RT-56) is designed for finding the optimal imaging conditions for wet samples with WETSEM™ technology.

It is strongly recommended that new users initially use the Calibration Capsule to find the optimal working conditions of the SEM. Sample imaging with the QX-202C capsule in an SEM differs from standard SEM imaging in some aspects. The factors that affect imaging vary depending on the application and differ from one SEM model to another. The Calibration Capsule contains nanoparticles (40 and 500 nm in size) firmly attached to the capsule membrane. The particles are easily imaged in an SEM and provide a convenient means to calibrate parameters for optimal wet imaging conditions.

3.5.1 Technical Data

Storage Store in dry, dark conditions until opened.
After adding Imaging Buffer, store at 4°

Shelf Life See expiry date printed on the container.

3.5.2 Use of the Calibration Capsule

For use and calibration details, refer to the product information sheet accompanying the product and to the supplied "QuickGuide for Imaging with WETSEM™".



Note

Prior to using the Calibration Capsule add 15µl of reconstituted Imaging Buffer to the capsule.

Chapter 4: Using the QX-202C Capsules

The following procedures are general and appropriate for many different QX-202C applications.

4.1 Getting Started

The QX-202C capsules and the QX capsule plates (MP-12) are supplied in a sealed container.

- Open the QX-202C capsule container by peeling back the paper cover.
- Position the required capsules in the MP-12 capsule plate.
- Open the positioned capsules by turning the sealing stub counter-clockwise (see Figure 6).

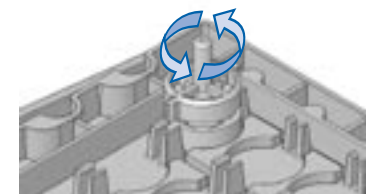


Figure 6. Opening the QX-202C Capsule



Note

For ease of application open the capsules prior to sample preparation.

- Cover the MP-12 capsule plate with the lid to protect the sample dish.



Caution

To prevent rupture, never touch the membrane. Keep the sample dish properly positioned in the capsule plate at all times.

- Put aside the sealing stub until the sample is ready for imaging. It is recommended to use the original QX-202C capsule package for storing the sealing stubs.
- To prepare a sample for SEM imaging, follow the steps described in the following section, or refer to the QX-202C Graphic User Guide supplied with the Starter Kit.

4.2 Sample Preparation

4.2.1 Cementitious/Gypsum Samples

- Place a measured quantity of powder in a dish.
- Add water according to the ratio required for the experiment.



Note

Amount of water (ml) = Quantity of cement (gr) X Required ratio (w/c)

- Mix thoroughly and quickly until mixture is homogeneous.
- Transfer a sample into the sample dish (see section 4.3) as soon as possible, in order to be able to observe the early stages of the reaction.



Note

Any water:cement ratio can be used with the QX-202C capsule. In very dilute reaction mixtures, large particles may drift away from the membrane. The image in such cases will show mostly the smaller particles. Reaction mixtures which have a paste consistency are most common for WETSEM™ imaging.

4.2.2 Other Samples

Prepare the reaction mixture according to the experimental design and transfer a sample into the sample dish as soon as possible (see section 4.3).

4.3 Inserting Samples into the QX-202C Capsule

4.3.1 Placing the sample



Caution

The sample dish contains the membrane that enables viewing of the samples with an SEM. The membrane is very delicate and must not be touched at any time. A damaged membrane is not suitable for use in an SEM.

- Set the Microman® to 6.5-7.5µl.
- Using the Microman, aspirate a sample from the reaction mixture.
- Carefully dispense the sample into the sample dish.
- Gently tap the capsule plate, with the filled liquid dishes in it, onto the bench-top. This ensures that the sample homogeneously covers the capsule membrane.



Caution

As the push-button of the Microman is pressed down, the piston will eject the sample out of the capillary and in itself will be slightly ejected. **Extreme care must be exercised to prevent the ejected piston from coming close to the capsule membrane.**

With highly diluted reaction mixtures (with Portland cement, approximately w/c>0.7), it may be difficult to image larger particles. Placing only 1-3µl onto the capsule membrane often solves this difficulty. This method, if used, requires further caution since inserting a small sample places the piston very close to the capsule membrane.

4.3.2 Sealing the capsule

Once the sample is placed in the sample dish, the capsule may be sealed. While keeping the specimen dish in the capsule plate, place the sealing stub on it. Turn the sealing stub clockwise until the "wings" are aligned. At this point, the capsule is tightly closed and ready for imaging in the SEM (see Chapter 5). To better protect the membrane leave the capsule in the capsule plate until it is placed in the SEM. For further protection, the capsule plate itself should be covered.

4.4 Storing Specimens in the QX-202C Capsule

Samples can be stored in sealed capsules for a limited period. Sample quality will deteriorate over time. The rate of deterioration is a function of the nature of the sample and the storage conditions.

Capsules should be left in the SEM vacuum chamber until the reaction mixture is partially cured and hardened. Subsequently samples can be removed from the vacuum chamber and re-imaged at different intervals through the duration of the reaction.



Note

The sealed capsules can be conveniently stored in the capsule plate.

Chapter 5: Imaging

The factors that affect imaging vary with different applications and different SEMs. This chapter provides guidelines for achieving the best imaging results for wet samples with WETSEM™ technology.

The imaging conditions are best optimized using the Calibration Capsule. The Calibration Capsule contains nanoparticles, which are easily visualized in the SEM and provide a convenient means to calibrate the parameters for optimal imaging conditions. The Calibration Capsule is provided with a reference image which enables the user to evaluate the imaging result after optimization.

5.1 Optimal Imaging Conditions

Follow the guidelines below to find the optimal imaging conditions.



Note

Recommendations for SEM parameters are summarized in Table 1. Optimize the conditions first with the Calibration Capsule, and then move on to imaging your sample.



Caution

The Calibration Capsule and the QX-202C capsule are taller than the average specimen. Lower the stage before placing the capsule in the SEM.

1. Place the closed QX-202C capsule on the microscope stage as a conventional 'stub' with the capsule membrane facing up. In case the QX-202C capsule does not fit your SEM stage, inquire about available adaptors.

2. The QX-202C capsules are suitable for use with high vacuum or low vacuum modes.
3. Ensure sufficient working distance. Note that the QX-202C capsules may be taller than conventional SEM stubs. Adjust the height of the stage as necessary.
4. For best results, the QX-202C capsules should be used in conjunction with a backscattered electron detector (BSED). If a BSED is not available, secondary electron (SE) imaging can also be performed. Please contact tech@quantomix.com to inquire about the use of secondary electron detectors with the QX-202C capsule. The capsule also enables qualitative X-ray microanalysis using suitable detectors.
5. For best imaging, adjust the working distance (WD) to maximize the sample BSE signal. See Table 1 for the recommended WD range.
6. To obtain best imaging conditions, start with an acceleration voltage of 30 kV and a low-range probe current/spot size.
7. Increase the contrast until the desired signal from the sample is obtained. It is recommended to start imaging with a slow scan speed (a few seconds per frame). Note that since the signal from the sample is generally weaker and of lower contrast than the signal from the support grid, the optimization of contrast and brightness should be carried out with respect to the sample and not to the grid.
8. Focus on the sample. If you have difficulties in focusing on your sample at this stage, focus first on the supporting grid of the capsule using SE (secondary electron) detector, and then switch back to BSE detection mode.
9. The recommended acceleration voltage range is 15 kV to 30 kV. Different acceleration voltages correspond to different penetration depths.
10. Probe current/spot size is determined empirically by the optimal signal obtained. A higher probe current generates a larger signal. A higher probe current may also damage the sample. The optimal probe current/spot size is determined by optimizing the signal and minimizing damage to the sample. For maximal allowed probe current see Table 1.
11. Scan speed should be adjusted according to the signal from the sample. For low contrast samples, it is recommended to work with lower scan speeds.
12. For samples sensitive to beam damage, it is recommended to optimize the working parameters first and then obtain images from different locations.

**Note**

Once the sample has been imaged in the SEM during the initial curing period, it can be either left in the chamber under vacuum for up to 24 hours or removed for intermittent imaging. Removing the sample before initial curing may result in reduced imaging quality.

5.2 Recommendations for SEM Imaging with QX-202C Capsules

Table 1: Recommendations for SEM Imaging with QX-202C Capsules

Parameter Recommended	Range	Comments
Acceleration voltage	15-30 kV	Not lower than 10 kV
Probe current (based on source type)		
Tungsten filament	0.4-1.0 nA	Not higher than 1.0 nA
FEG	0.1-0.5 nA	Not higher than 0.5 nA
Working distance (based on detector type)		
Semiconductor (BSE)	6-10 mm	Acceptable: 5-15 mm
Robinson (BSE)	10-20 mm	Better efficiency at higher kV
Scintillator (BSE)	6-10 mm	Acceptable: 6-10 mm
Everhart- Thornley (SE)	8-12 mm	Acceptable: 6-15 mm
In-lens / Through the lens (all detectors)	2-4 mm	Manufacturer dependent

5.3 Recommendations for EDS Analysis with QX- 202C Capsules

Qualitative EDS analysis using WETSEM™ technology is similar to standard EDS analysis.

The optimal conditions for EDS analysis vary amongst specimens and different SEM configurations. When analyzing wet specimens with WETSEM™ technology, several factors should be considered.

5.3.1 Working Distance

Optimal working distance for WETSEM™ EDS will usually be the standard working distance for dry EDS measurements (10-12mm). This distance is typically larger than the recommended working distance for imaging. If both a sharp image and an EDS spectrum are required, it is recommended to obtain an image at the optimal working distance for this purpose, then lower the stage to the optimal working distance for EDS work.

5.3.2 Accelerating Voltage

When doing EDS with WETSEM™ technology the accelerating voltage will typically be in the range of 20-30 kV. To prevent damage to the QX Capsule membrane, it is recommended to start with 30 kV and a spot size which assures the integrity of the sample and membrane (probe current corresponding to no more than 0.75 nA). Reduce the accelerating voltage if needed and if possible. A voltage of 20 kV is typically a suitable compromise between the need for sufficient over-voltage and the need to minimize absorption in the sample.

5.3.3 Selected Area and Spot Analysis

When analyzing very small areas or a single spot, sensitive samples may cause failure of the membrane, resulting in a leak into the SEM chamber. To prevent such an occurrence, the smaller the scanned area - the shorter the exposure time and spot size should be.

Initially, measurement time should be preset to 20 seconds. If an insufficient signal is obtained, exposure time may gradually be increased. Sensitivity to measurement time will differ from one sample to another.

5.3.4 The Membrane Contribution to the EDS Spectrum

The QX capsule membrane is only a few hundred nanometers thick, while the X-ray signal comes from a much thicker layer. Hence the membrane does not interfere with the EDS measurement. However, when carrying out EDS measurements using the QX capsule, the user should take into account the contribution of the membrane's X-ray signal which is manifested by a small carbon peak.

Chapter 6: Appendices

6.1 Appendix A: Glossary

BSE	Back-scattered electrons
BSED	Back-scattered electrons detector
Calibration Capsule	A QX-capsule containing a reference sample used for optimization of imaging conditions
EDS	Energy Dispersive Spectroscopy, a method enabling elemental analysis of a sample
MP-12	Capsule plate; a holder for parallel handling of up to 12 individual QX-202C capsules, serving to hold and store the capsules
QX-202C	Capsule used for SEM-imaging of various dynamic processes
QX Imaging Buffer	Buffer required for imaging the QX Calibration Capsule in a SEM
SE	Secondary electrons
SED	Secondary electron detector
SEM	Scanning electron microscope
Sealing Stub	Part of the QX-202C capsule used for sealing the capsule and for holding the capsule in the SEM
Sample Dish	QX-202C capsule base designed as a dish for placing specimens

6.2 Appendix B: Troubleshooting

Table 2: Troubleshooting

Problem	Possible Cause	Solution
Sample is spilling out of the sample dish	The amount of sample placed in the sample dish is too large	Place only 6.5-7.5µl of sample in the sample dish.
Sample is leaking through the bottom of the sample dish	The membrane was ruptured during sample insertion	A new sample must be prepared. Samples with a torn membrane should not be placed in the SEM.
Capsule plate underneath the capsule is dirty	The membrane was ruptured during sample insertion, allowing some of the sample to penetrate it and reach the capsule plate.	A new sample must be prepared. Samples with a torn membrane should not be placed in the SEM.
No sample is observed	The sample is not in contact with the membrane	The amount of sample placed in the sample dish is too small. A total of 6.5-7.5µl of the reaction mixture should be placed in the sample dish.
Only small particles are observed	The sample is not in full contact with the membrane	The amount of sample placed in the sample dish is too small. A total of 6.5-7.5µl of the reaction mixture should be placed in the sample dish.
	The sample is very dilute and large particles drift away from the capsule membrane.	Prepare a reaction mixture with a lower water content and a paste texture. Otherwise, place a very small quantity (appx. 1µl) of the reaction mixture on the capsule membrane.

Problem	Possible Cause	Solution
Sample is not stable while imaging	Interaction with the electron beam, especially at high magnifications, can cause local artifacts.	Reduce the spot size to the minimum at which a clear image can be obtained. An increase in the acceleration voltage may also reduce artifacts. Minimize sample exposure time to the beam.
Reaction stops earlier than expected	The sample dried out due to damage caused to the membrane	A new sample must be prepared. Samples with a torn membrane should not be placed in the SEM.
	The sample dried out due to improper sealing.	Make sure that the top surface of the sample dish and the circumference of the rubber seal are clean prior to sealing the capsule.
Microman does not aspirate the sample	The Microman's capillary and piston are designed for a single use only.	Replace the capillary and piston and try aspirating the sample again.
	Sample is too thick for the Microman	Use a more dilute reaction mixture or carefully place the sample in the sample dish with the aid of a spatula.
The reaction mixture does not seem to be dynamic and no progress is observed	The reaction studied is a slow reaction	Follow the reaction by imaging at longer time intervals
	The reaction had been completed and all the water had been consumed.	No further change can be expected and a new sample must be prepared.
	The reaction mixture dried out due to improper sealing or a damaged membrane.	No further change can be expected and a new sample must be prepared.

6.3 Appendix C: Ordering Information

Please see www.quantomix.com for your local distributor or QuantomiX Sales Representative or contact sales@quantomix.com.

6.4 Appendix D: Legal Notices

6.4.1 Product Warranty, Liability and License for Use

1. QuantomiX guarantees the performance of all Products in the manner described in our product literature. The Purchaser must determine the suitability of the product for its particular use or application. Should any product fail to perform satisfactorily, within a period of twelve months from receipt, or within the shelf life expiry date of the product, whichever is shorter, due to any reason other than misuse or unsuitable application, QuantomiX will replace it free of charge or refund the purchase price. Please contact tech@QuantomiX.com or your local distributor or sales representative.
2. QuantomiX QX capsules have been validated for single-use in Scanning Electron Microscopes of the major manufacturers. Reuse of capsules is prohibited, is deemed misuse of the Products and releases QuantomiX from all warranty obligations.
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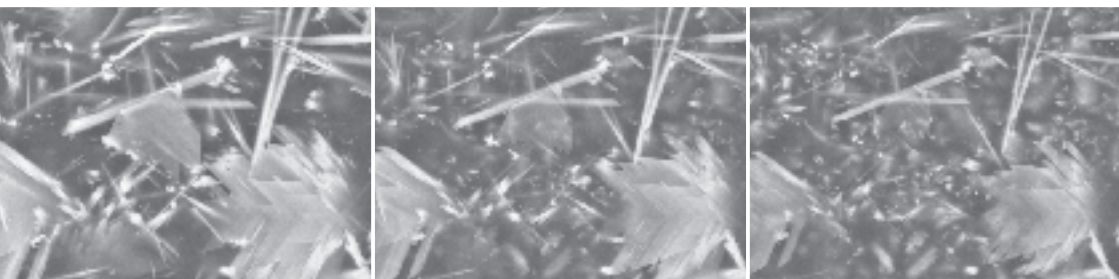
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