

THE OPERA INSTRUMENT: AN ADVANCED CURATION DEVELOPMENT FOR MARS SAMPLE RETURN ORGANIC CONTAMINATION MONITORING. M.D. Fries¹, W.D. Fries, F.M. McCubbin¹, and R.A. Zeigler¹, ¹Astromaterials Acquisition and Curation Office, Johnson Space Center, Houston, TX 77058. Email: marc.d.fries@nasa.gov

Introduction: Mars Sample Return (MSR) requires strict organic contamination control (CC) and contamination knowledge (CK)[1] as outlined by the Mars 2020 Organic Contamination Panel (OCP)[2]. This includes a need to monitor surficial organic contamination to a ng/cm² sensitivity level. Archiving and maintaining this degree of surface cleanliness may be difficult but has been achieved [3,4]. MSR's CK effort will be very important because all returned samples will be studied thoroughly and in minute detail. Consequently, accurate CK must be collected and characterized to best interpret scientific results from the returned samples. The CK data are not only required to make accurate measurements and interpretations for carbon-depleted martian samples, but also to strengthen the validity of science investigations performed on the samples. The Opera instrument prototype is intended to fulfill a CC/CK role in the assembly, cleaning, and overall contamination history of hardware used in the MSR effort, from initial hardware assembly through post-flight sample curation. Opera is intended to monitor particulate and organic contamination using quartz crystal microbalances (QCMs), in a self-contained portable package that is cleanroom-compliant. The Opera prototype is in initial development capable of ~100 ng/cm² organic contamination sensitivity, with additional development planned to achieve 1 ng/cm². The Opera prototype was funded by the 2017 NASA Johnson Space Center Innovation Charge Account (ICA), which provides funding for small, short-term projects.

Quartz Crystal Microbalances (QCMs): QCMs are not a new technology, and in fact they feature significant heritage to include use as spaceflight instruments [5,6]. A QCM is composed of a polished quartz disk cut from a single crystal of α -quartz along a specific crystallographic orientation, chosen based on desired resonant waveform. Electrodes are deposited onto both sides of the crystal, and an AC voltage is applied to the electrodes. The piezoelectric quartz disk then oscillates at a resonant frequency, which is measured by QCM electronics. Very small changes in the mass of this system will change the resonant frequency, such that QCMs can be capable of sensing ng/cm² (e.g. monolayer) changes in mass deposited on the crystal surface.

The Opera Concept: Organic contamination monitoring can be performed by exposing witness plates, followed by analysis with techniques sensitive to very small amounts of surficial carbon such as X-ray photoelectron spectroscopy (XPS). This technique works, but

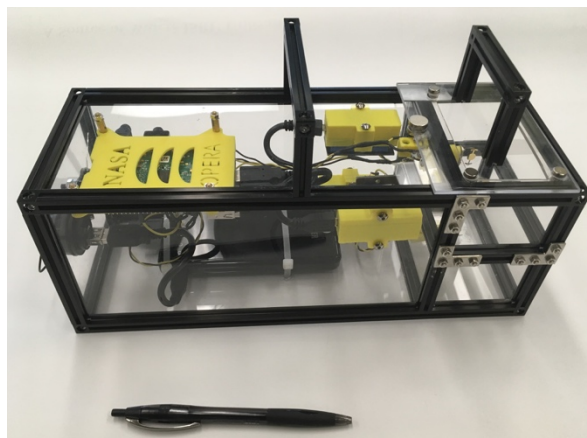


Figure 1: The Opera prototype. Data is collected by removing the panel at right, which is held in place by magnets. Data collection ends when the panel is replaced.

its limits include moderate cost and a whole-exposure measurement, as opposed to a methodology capable of detecting changes in organic contamination over the course of the measurement period. Detecting contamination changes is useful for assessing when a contamination event occurs, such as while performing a specific task during sample processing or flight hardware assembly. Opera is intended to improve contamination monitoring in several ways:

- 1) Active monitoring over a period of time with time-stamped data.
- 2) Detection of organic contamination and particulate contamination concurrently.
- 3) Detection of the chemical composition of organic contaminants, via post-test analysis of the QCM wafers via GC-MS, DART-MS, etc.
- 4) Near-real-time data availability.

In actual use, the Opera instrument will be utilized in a range of situations where contamination monitoring is required. These include fabrication and cleaning of sample collection hardware, assembly test and launch operations (ATLO), recovery and processing of the sample return capsule (SRC), and sample processing in a glove-box or cleanroom environment.

QCMs measure mass accumulation on their surfaces, but cannot distinguish between particulate and organic contamination. Opera is designed to overcome this limitation by using a trio of QCMs. One is completely sealed and serves as a reference. Two QCMs are exposed when the lid is removed (see Figure 1). One QCM is oriented with the quartz wafer horizontally and the

second is vertically mounted. Particulates and organic contaminants accrue on the horizontal QCM, but the vertically mounted QCM should strongly prefer accumulation of organic contamination with minimal particulate adherence. Organic contamination is measured as a difference between the two QCMs, as compared to the reference QCM. Particulate contamination can be further analyzed by examining the QCM wafer via optical or scanning electron microscopy.

Functions of the Opera prototype include:

- 1) Test the triple-QCM concept and validate the functionality and lower limit of detection
- 2) Test the geometry of the exposure cell; removable lid versus removable corner section
- 3) Measure the maximum measurement time length

The Opera prototype uses off-the-shelf QCM hardware as an inexpensive proof of concept. The three QCMs use OpenQCM (www.openqcm.com) Arduino shields operated by one Arduino Micro apiece. Communication and power for all three are handled by USB connection to a Raspberry Pi 3 board. A reed switch signals the removal of the measurement cell lid, at which point custom Python code on the Raspberry Pi initiates data collection from all three QCMs into a common data file. Data collection ceases when the lid is replaced. Future prototypes will transfer data files via Bluetooth and/or Wi-Fi to a laptop for data processing. The QCMs operate at a resonant frequency of 6 MHz.

Preliminary Results and Future Direction: Testing of the Opera prototype is currently underway. Preliminary measurements show that the Opera prototype successfully differentiates between particulate and organic contamination, at the ~ 100 ng/cm² level, and additional measurements will constrain this value to include optical measurements of dust adherence on the vertical QCM wafer. Battery life for the prototype allows for 22 continuous hours of data collection.

Additional tests include validation against bare, exposed witness plates to test the geometry of the QCM cell. The prototype housing, assembled using MakerBeam (www.makerbeam.com) rail hardware, will be reconfigured for comparative testing against a second geometry whereby the entire front corner of the prototype is removed to conduct a measurement. This geometry will render the QCMs more exposed to air currents, but may better sample the contamination environment. Data analysis using only the measurement cell QCMs might also obviate the need for the reference QCM, but this hypothesis must be quantitatively challenged using the prototype.

The 6MHz resonant frequency of the prototype's QCM wafers restrict the lower limit of detection of around 100 ng/cm², but have allowed an inexpensive proof of concept trial. A future Opera prototype v.2 will

substitute the OpenQCM 6 MHz wafers for oscillation and readout electronics, and wafers with a higher resonant frequency such as 27 GHz. QCM detection sensitivity scales with resonance frequency [7], and 1 ng/cm² is achievable. Temperature sensors built into the OpenQCM Arduino shields will be moved to the QCM wafer location in the v.2 prototype. Beyond the v.2 prototype, a final Opera version will feature a cleanroom-compliant housing with any switches, plugs, or other fittings concealed in a compartment. This version of Opera will be validated for cleanroom use and multiple copies will be manufactured as part of NASA Curation's MSR CK effort.

References: [1] Harrington A., Calaway M., Regberg A., Mitchell J., Fries M., Zeigler R., McCubbin F., see abstract at this meeting (2018). [2] Summons, R.E., Sessions, A.L., Allwood, A.C., Barton, H.A., Beaty, D.W., Blakkolb, B., Canham, J., Clark, B.C., Dworkin, J.P., Lin, Y. and Mathies, R., 2014. *Astrobiology*, 14(12), pp.969-1027. [3] Steininger, H., Goesmann, F., Raulin, F., Brinckerhoff, W.B., Mahaffy, P.R. and Szopa, C., 2016. *LPI Contributions*, 1980. [4] Flory, D.A., Oró, J. and Fennessey, P.V., 1974. In *Cosmochemical Evolution and the Origins of Life* (pp. 443-455). Springer Netherlands.[5] Wood, B.E., Hall, D.F., Lesho, J.C., Dyer, J.S., Uy, O.M. and Bertrand, W.T., 1996, November. In *Optical System Contamination V, and Stray Light and System Optimization* (Vol. 2864, pp. 187-195). Int'l Soc. Opt. Phot. [6] ASTM E2311-04 "Standard Practice for QCM Measurement of Spacecraft Molecular Contamination in Space". [7] Stockbridge, C. D., *Vacuum Microbalance Techniques*, edited by K.H. Behrndt, Vol 5, Plenum, NY, 1966, p. 179.