

## Ultra-High Vacuum Evolved Gas Analysis (UHV-EGA) Application Note 1

### Introduction

Ultra-High Vacuum Evolved Gas Analyzer (UHV-EGA), model 510, is a mass spectrometer system that has been developed by Oneida Research Services, Inc. to identify gases evolved from any type of solid or semi-solid sample (1) under ultra-high vacuum conditions and (2) over a temperature range. This method is particularly valuable in the microelectronics industry where hermetically sealed packages are manufactured using variety of components including adhesives, metal and/or ceramic substrates and lids. Prior to sealing these components in hermetic packages, baking of these components at a specific temperature for a fixed time is critical. Improper baking will result in outgassing that could cause the presence of unwanted gases inside the package and lead to moisture failures per MIL-STD 883, TM 1018 - also known as internal gas analysis (IGA) testing.

### Principle of Operation

UHV-EGA involves heating a sample in an ultra-high vacuum environment (< 5E-8 Torr) to temperatures up to 1200°C. The gases released during this process are dynamically identified and characterized by a quadrupole mass spectrometer with a mass range of 1-300 amu. The specially designed ultra-high vacuum heater can achieve ramp rates from 0.1°C to 15°C per minute.

### EVACS™ : Superior Operation and Data Processing

The UHV-EGA system runs on the dedicated EVACS™ software platform that provides user friendly procedure set up and operation, to conduct a seamless analysis. Developed by highly experienced programmers at ORS, EVACS™ facilitates precise control of the heating protocols along with mass spectrometer tuning and vacuum parameters, ensuring accurate and reproducible results. EVACS™ also provides multiple data processing methods to understand the acquired results. It features robust data processing capabilities, including real-time monitoring, automated peak identification, and comprehensive data analysis tools including matching unknown mass spectra with NIST database. The user-friendly interface streamlines the operation, making it accessible for researchers and engineers to achieve high-quality data with ease.

### Applications

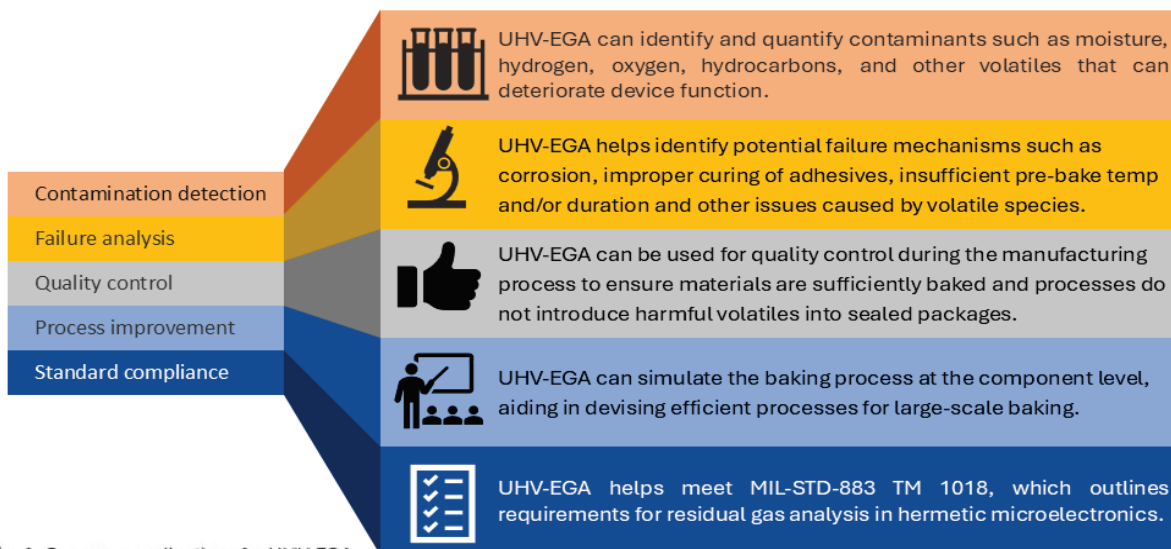
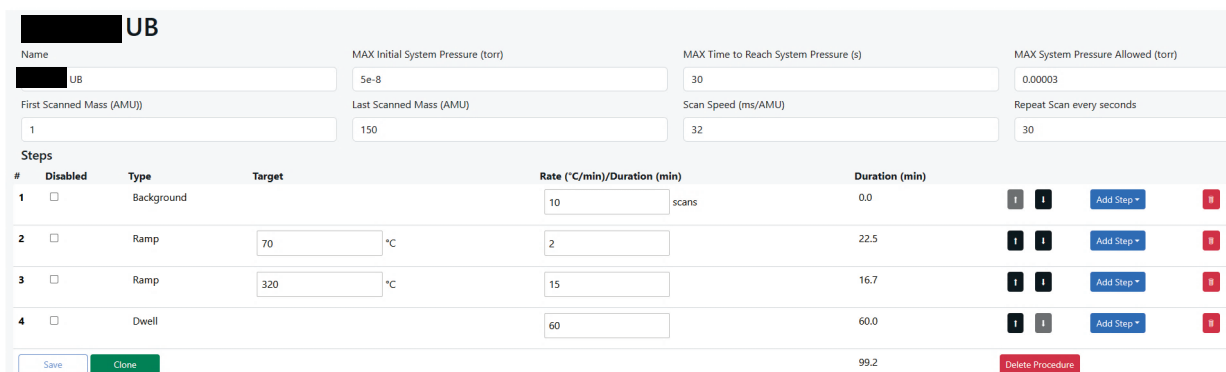


Fig. 1: Common applications for UHV-EGA

## Case Study

UB lids must undergo chemical cleaning before they are used for hermetic packaging. To ensure the package meets MIL-STD 883, TM 1018 requirements, it is crucial to verify the cleaning effectiveness and confirm that no residues remain. This case study examines two types of UB package components: metal and ceramic lids. For the comparative study, two groups of each type of lid were selected: one group was left uncleaned, and the other underwent chemical cleaning.

### Heating protocol:



The screenshot shows the 'Steps Table' in the EVACS™ software. The table is titled 'UB' and contains the following data:

#	Disabled	Type	Target	Rate (°C/min)/Duration (min)	Duration (min)	Buttons
1	<input type="checkbox"/>	Background		10 scans	0.0	[I] [I] [Add Step] [X]
2	<input type="checkbox"/>	Ramp	70 °C	2	22.5	[I] [I] [Add Step] [X]
3	<input type="checkbox"/>	Ramp	320 °C	15	16.7	[I] [I] [Add Step] [X]
4	<input type="checkbox"/>	Dwell		60	60.0	[I] [I] [Add Step] [X]
					99.2	[Delete Procedure]

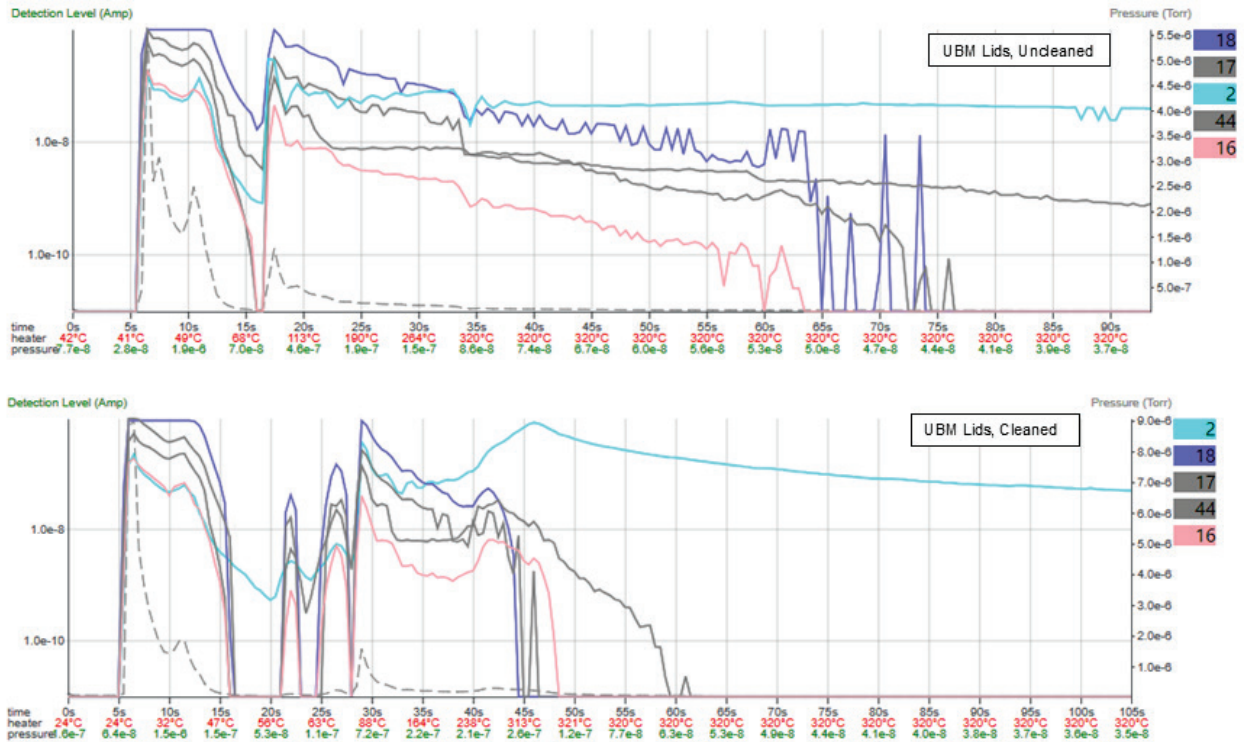
Additional parameters shown in the UI include: Name: UB, MAX Initial System Pressure (torr): 5e-8, MAX Time to Reach System Pressure (s): 30, MAX System Pressure Allowed (torr): 0.00003, First Scanned Mass (AMU): 1, Last Scanned Mass (AMU): 150, Scan Speed (ms/AMU): 32, Repeat Scan every seconds: 30. Buttons for 'Save', 'Clone', and 'Delete Procedure' are visible at the bottom.

Fig. 2: A screen capture of the Steps Table UI in EVACS™

The EVACS™ Step Table is used to define heating protocol for the UB lid samples. MAX initial system pressure (Torr) defines the maximum value for system pressure required to start the analysis. This means pressure has to be below 5E-8 torr to initiate the analysis. During analysis the max system pressure allowed defines the value over which MS will shut off the filament and stop the analysis. This limit is essential to protect the ion source filament from high gas pressure. First and last scanned mass gives the mass range over which the analysis will be performed. For example, 1 to 150 amu scan were taken for UB lid samples. Scan speed is scan time per amu. For example, when a scan is done at 32 ms/amu speed, to complete the scan over 1-150 amu, the total scan time will be  $32 \text{ ms} \times 150 = 4,800 \text{ ms} = 4.8 \text{ s}$ . This scan was repeated every 30 seconds.

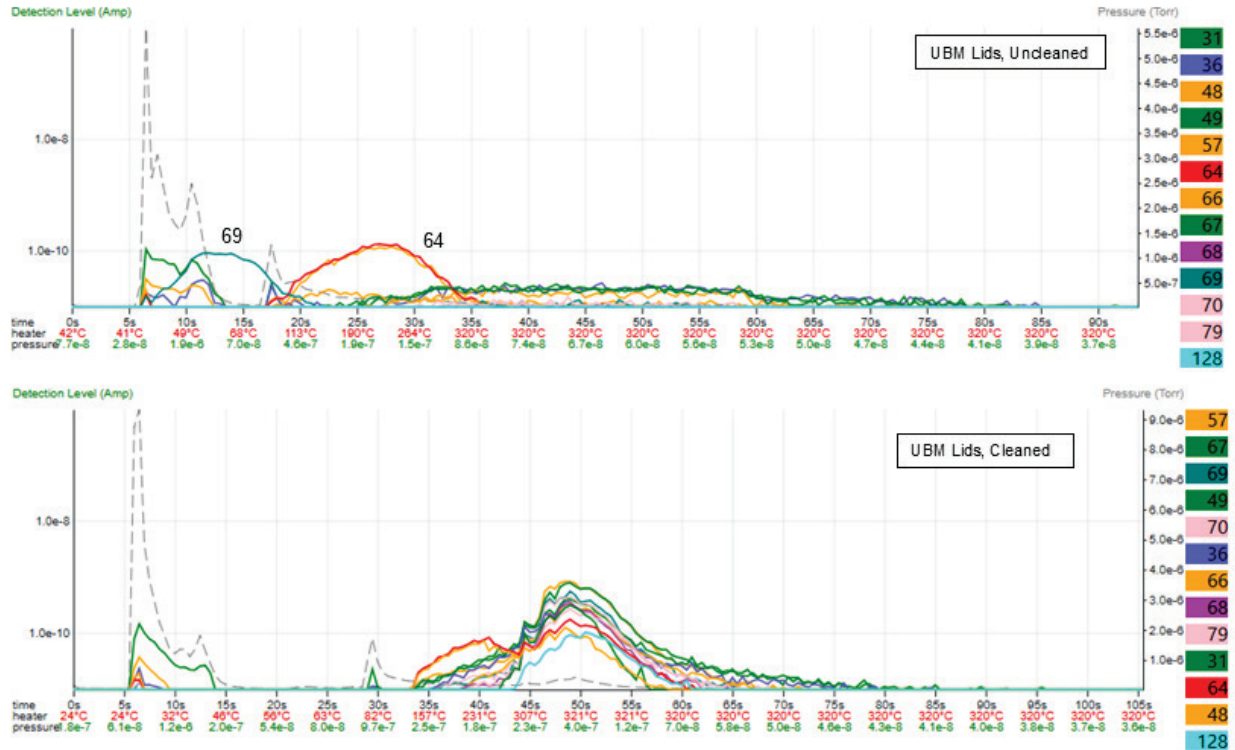
Under the Steps Table, users can define different types of steps. For example, the first step is for collecting 10 background scans without any change in the temperature. Second step is a ramp type step, where heating is started from current heater value to 70°C, at the rate of 2°C/min. “Duration” lists the time estimated to achieve this step. The next step is set with higher heating rate (i.e., 15°C/min) to heat up to 320°C. The fourth step is dwell type, where the temperature of 320°C will be maintained for 60 mins. The analysis will be stopped after completing all the four steps, which is estimated to be completed in 99.2 mins.

**Results:** The evolved gas analysis for UB lid samples shows the presence of trace amounts of hydrocarbons along with other non-organic gas species. UB metal lid groups of uncleaned and cleaned samples show clear differences. As shown in Fig. 3, uncleaned UB metal lids have the presence of mass 44 (likely to be CO<sub>2</sub>) that remains detectable during the entire course of analysis. For cleaned UB metal lids, mass 44 is present during the ramping steps however reduces to background level during dwelling at 320°C. This clearly shows that while cleaning process does not completely remove CO<sub>2</sub>, it facilitates easy removal of CO<sub>2</sub> during the dwell time (i.e., baking of the lids at 320°C). Similar observation is made for masses 18, 17 and 16 (likely to be moisture (H<sub>2</sub>O) and its fragments (OH ion and O ion)).



**Fig. 3:** Comparison between uncleaned and cleaned UB metal lids clearly shows the difference in release pattern of mass 44 (likely to be CO<sub>2</sub>). Release of mass 2 (likely to be hydrogen) is observed for both the samples and the release pattern is very similar. It should also be noted that release of other masses (like 18, 17 and 16) from uncleaned UB metal lids takes longer to reach the background level.

For the same samples, upon investigating other masses with lower outgassing rates, a similar observation was made further confirming the effect of cleaning on these UB metal lids. As shown in Fig. 4, unclean UB metal lids show release of various masses spread over the temperature range (from 40°C to 320°C), whereas for cleaned UB metal lids it is observed that masses are released synchronously at 320°C. This can be explained by how cleaning process is resetting residues on the sample surface which outgasses synchronously at the same time and temperature. In the case of uncleaned UB metal lids, these residues release as per the origin of contamination. This observation confirms that cleaning process can enable effective removal of contaminants from the sample surfaces.



**Fig. 4:** Comparison between uncleaned and cleaned UB metal lids clearly shows the difference in release pattern for masses likely to be released from long chain hydrocarbon species. For uncleaned UB metal lids, the release of these masses are over the temperature range while for cleaned UB metal lids, the masses are released synchronously at a sharper temperature range and time.

## Conclusion

The case study carried out for UB metal lids show that UHV-EGA and the EVACS™ software is a powerful tool for the microelectronics industry, providing critical insights into the presence of contaminants, failure mechanisms, and overall quality of components used in hermetically sealed packages. Its ability to simulate baking processes makes it unique in its ability to evaluate the efficiency of baking process which includes heating temperature and duration. UHV-EGA can identify and characterize evolved gases under ultra-high vacuum conditions and elevated desired temperature, makes it indispensable for ensuring the reliability and longevity of microelectronic devices.

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