

STANDARD OPERATING PROCEDURE: RESIDUAL GAS ANALYZER (RGA) HYDROCARBON SPECTRUM VERIFICATION & BACKSTREAMING DETECTION

HIGH-VACUUM MASS SPECTROMETRY SPECTRUM ANALYSIS & OIL CONTAMINATION DIAGNOSTICS

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1.0 PURPOSE & SCOPE

This Standard Operating Procedure (SOP) defines the technical requirements for operating a Quadrupole Residual Gas Analyzer (RGA) to perform partial pressure spectrum analysis, detect hydrocarbon backstreaming from oil-sealed roughing pumps, and verify vacuum chamber cleanliness prior to thin-film deposition or UHV processing. It applies to all high-vacuum processing systems equipped with 1–100 amu or 1–200 amu quadrupole mass spectrometers.

2.0 SAFETY & OPERATIONAL INTERLOCKS

- Maximum Operating Pressure Interlock:** Never energize the RGA filament (hot cathode) or Electron Multiplier (EM) at total chamber pressures exceeding 1.0×10^{-4} Torr (1.33×10^{-4} mbar). Operating above this threshold causes rapid filament oxidation and multiplier degradation.
- High Voltage Safety:** Electron Multiplier detectors operate at bias voltages up to -1500V to -2500V DC. Ensure proper electrical grounding before handling RGA control electronics.
- Nitrile Glove Policy:** Mandatory 6 mil powder-free nitrile gloves during sensor head handling to prevent skin oil transfer.

3.0 EQUIPMENT SETUP & MASS CALIBRATION

- Hardware Required:** KJLC / Pfeiffer / SRS 100/200 amu Quadrupole RGA with Faraday Cup and Secondary Electron Multiplier (SEM) detectors.
- Mass Scale Calibration:** Perform 2-point mass calibration using residual water ($m/z = 18.0$) and atmospheric nitrogen ($m/z = 28.0$) or argon ($m/z = 39.9$) peaks. Peak centers must align within ± 0.1 amu.
- Emission Current & Electron Energy:** Set electron ionization energy to 70 eV and emission current to 0.5–1.0 mA for standardized electron impact cracking patterns.

4.0 HYDROCARBON BACKSTREAMING SPECTRUM ANALYSIS

Hydrocarbon backstreaming from mechanical roughing pumps (rotary vane) occurs when oil vapors travel counter to gas flow into the vacuum chamber under molecular flow conditions ($< 10^{-4}$ Torr). RGA mass spectra reveal distinct fragmentation cracking patterns characteristic of aliphatic hydrocarbons:

Mass-to-Charge (m/z)	Ionic Species / Fragment Formula	Source Indication	Action Limit / Threshold
m/z = 18	H ₂ O ⁺ (Water Vapor)	Residual moisture outgassing from chamber walls.	Normal dominant peak in non-baked chamber.
m/z = 28	N ₂ ⁺ / CO ⁺ (Nitrogen / Carbon Monoxide)	Atmospheric air leak if N ₂ :O ₂ (m/z 28:32) is 4:1.	Investigate if m/z 28 > 10× water peak.
m/z = 32	O ₂ ⁺ (Oxygen)	Real atmosphere leak through flange or weld seal.	If present with m/z 28 at 1:4 ratio = leak check.
m/z = 41	C ₃ H ₅ ⁺ (Propenyl fragment)	Aliphatic hydrocarbon oil breakdown.	> 1.0×10^{-4} Torr = Backstreaming Flag
m/z = 43	C ₃ H ₇ ⁺ (Propyl fragment)	Hydrocarbon vacuum oil cracking peak.	> 1.0×10^{-4} Torr = Backstreaming Flag
m/z = 55	C ₄ H ₇ ⁺ (Butenyl fragment)	Refined hydrocarbon oil fraction.	> 5.0×10^{-5} Torr = Backstreaming Flag
m/z = 57	C ₄ H ₉ ⁺ (Butyl fragment)	Heavy hydrocarbon pump fluid vapor.	> 5.0×10^{-5} Torr = Backstreaming Flag

5.0 MITIGATION & CORRECTIVE ACTIONS

When RGA spectrum analysis reveals high intensity at $m/z = 41, 43, 55, 57$ exceeding the 1.0×10^{-10} Torr action limit, execute the following corrective protocol:

1. **Gas Ballast Sweeping:** Open roughing pump gas ballast valve to sweep accumulated hydrocarbon vapors out of the pump housing oil.
2. **Anti-Suckback Valve Test:** Verify fast-acting (<20 ms) electro-pneumatic anti-suckback valve closure upon power isolation to prevent oil climbing foreline walls.
3. **Foreline Cold Trap Servicing:** Replenish Liquid Nitrogen (LN₂) in inline foreline cold traps or regenerate molecular sieve traps at 200°C under dry N₂ flow.
4. **Chamber Solvent Wipe & Bake-Out:** Isolate pump, wipe chamber interior with lint-free wipes dampened with LC-MS grade Isopropanol, and run 150°C chamber bake-out for 24 hours.
5. **Dry Scroll Pump Conversion:** If hydrocarbon peaks persist, convert roughing line from oil-sealed rotary vane to oil-free dry scroll backing pump.

6.0 QA/QC SPECTRUM LOGGING & DOCUMENTATION

Export RGA trend logs and mass scan ASCII files (`.csv`) for every production run. Save baseline spectrum plots to the CMMS database under the asset file. Record total chamber pressure, partial pressure of m/z 18, 28, 32, 43, and 57, and document technician attribution.

AUTHORIZATION & QUALITY ASSURANCE SIGN-OFF

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