

METROLOGY & VACUUM TROUBLESHOOTING PROTOCOL

CRYOREFRIGERATOR PUMP LEAK HUNTING, VACUUM INTEGRITY & FAULT DIAGNOSTICS

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Target Equipment:	Leybold COOLVAC / COOLPOWER	Compressor Units:	COOLPAK 2000i / 5000i	Leak Detector:	Leybold PHOENIX / Agilent HLD

1. Purpose, Scope & Critical Safety Hazards

This standard operating procedure and troubleshooting protocol defines the rigorous procedures for detecting vacuum leaks, diagnosing thermal performance degradation, and performing fault isolation on cryogenic vacuum pumping systems (Leybold COOLVAC 1500–60000 iSL/iBL and COOLPAK 2000i/5000i compressors). Strict adherence to safety protocols is required to prevent severe physical injury and equipment failure:

- High-Pressure Helium Hazards (16.0 to 22.0 bar(g)):** The helium refrigeration circuit operates under extreme static and dynamic pressures. Never loosen Aeroquip flexline couplings or cold head fittings without confirming poppet valve engagement and zero line pressure. Wear safety glasses when handling helium lines.
- Cryogenic Thermal Hazards (10 K to 80 K):** Internal cold head surfaces, 2nd stage cryopanel, and 1st stage radiation shields reach extreme cryogenic temperatures (-263°C). Direct skin contact causes immediate frostbite and tissue destruction. Allow cold head to warm completely to +20°C (293 K) prior to opening vacuum vessel.
- Overpressure & Unvented Warming Hazards:** Trapped gases (H₂, N₂, O₂, H₂O) desorb rapidly during warming. The **ISO 4126 overpressure relief valve (0.3 to 0.5 bar(g) pop-off pressure)** must remain completely unobstructed. Never cap, plug, or blank off the safety relief port under any operational or maintenance condition.

2. Cryopump Leak Testing Methodologies

2.1 External Vacuum Jacket & Flange Spray Testing (Helium Mass Spectrometer)

Connect a calibrated helium mass spectrometer leak detector (Leybold PHOENIX / Agilent HLD) to the foreline roughing port. Evacuate the cryopump vessel outer vacuum jacket (OVJ) to rough vacuum ($p < 1 \times 10^{-1}$ mbar). Spray helium tracer gas around all CF high-vacuum flanges, ISO-KF foreline connections, feedthrough seals, and the ISO 4126 overpressure relief valve Viton O-ring. Acceptance spec: No helium signal exceeding 1×10^{-10} mbar·L/s.

2.2 Internal Cold Head Helium Leak Diagnostic (Refrigerant Circuit Leak)

To differentiate between an external flange air leak and an internal helium refrigeration circuit leak (refrigerant leaking from the cold head displacer into the vacuum vessel): Isolate the roughing pump and monitor Mass 4 (Helium) on the MS leak detector. Increase static helium charge pressure on the COOLPAK compressor from 16 bar(g) to 18 bar(g). If the Mass 4 background signal rises proportionally with helium pressure, an internal cold head seal leak or displacer housing crack is present.

2.3 Pressure Rise / Rate-of-Rise Method (dp/dt)

Isolate the cryopump vessel under rough vacuum ($p \approx 1 \times 10^{-1}$ mbar) at ambient temperature (+20°C) and record vessel pressure over 5 minutes. Calculate rise rate: $dp/dt = (P_f - P_i) / \Delta t$. A linear pressure increase indicates a physical atmosphere-to-vacuum leak. An asymptotic curve that flattens out indicates water vapor outgassing or desorbing ice on cryopanel surfaces.

2.4 Residual Gas Analyzer (RGA) Mass Spectrum Profiling

Utilize a Leybold LEYSPEC RGA to analyze background gas species. A real air leak exhibits a distinct **4:1 ratio of Mass 28 (N₂) to Mass 32 (O₂)** alongside Mass 40 (Argon). High Mass 18 (H₂O) indicates insufficient thermal regeneration. High Mass 2 (Hydrogen) indicates charcoal panel saturation, requiring a full 295 K warm-up regeneration cycle.

3. Comprehensive Cryopump Diagnostic & Troubleshooting Matrix

Symptom / Fault	Probable Root Cause	Diagnostic Verification Check	Corrective Action & Maintenance
2nd Stage Temp High (T₂ > 20.0 K) Poor Base Vacuum	1. Saturated activated charcoal (H ₂ /He capacity exceeded). 2. Thermal radiation shield icing. 3. Depleted compressor adsorber.	T ₂ temperature stays above 20 K; RGA shows high Mass 2 (H ₂). Rate-of-rise test confirms no physical air leak.	Perform full 295 K warm-up regeneration with dry N ₂ purge. Check COOLPAK running pressure delta (HP-LP = 12–14 bar). Replace adsorber.

Symptom / Fault	Probable Root Cause	Diagnostic Verification Check	Corrective Action & Maintenance
High Helium Background (Mass 4 Leak Signal)	1. Internal cold head displacer seal leak. 2. Aeroquip coupling self-sealing poppet leaking into vacuum vessel.	Mass 4 signal increases directly when static helium pressure is raised from 16 to 18 bar(g) on COOLPAK.	Overhaul cold head displacer drive and dynamic seals. Replace leaking Aeroquip flexline couplings; leak check cold head body.
ISO 4126 Relief Valve Leaking / Continuous Vent	1. Particulate debris on Viton seal ring. 2. Weak relief spring due to repeated thermal overpressure discharges.	Helium spray over ISO 4126 exhaust port causes sharp spike on leak detector during pre-evacuation.	Clean Viton seal ring and valve seat with isopropyl alcohol. Replace Viton O-ring; verify 0.3 bar(g) pop-off pressure.
Cold Head Knocking / High Compressor Delta P	1. Oil aerosol contamination in helium circuit. 2. Depleted adsorber (> 10,000 hrs). 3. Cold head mechanical wear.	Acoustic metallic knocking; compressor running pressure differential drops below 10 bar or exceeds 16 bar.	Replace COOLPAK helium adsorber capsule. Decontaminate/flush helium circuit with High Purity 99.999% He gas. Overhaul cold head.
Slow Cool-Down Time (> 120 min to reach 12 K)	1. Low cooling water flow (< 5 L/min). 2. Low static helium charge (< 16 bar). 3. Degraded thermal insulation vacuum.	Compressor water entry temp > 25°C; static helium gauge reads < 15 bar; pre-evacuation pressure > 0.1 mbar.	Adjust cooling water valve to ≥ 5.0 L/min. Re-charge helium to 16.5 bar(g). Pre-evacuate cryopump body to < 0.05 mbar prior to restart.

4. Technician Verification Checklist & Field Test Readback Log

Phase	Inspection / Diagnostic Point	Acceptance Criteria / Target Spec	Field Readback Value	Status
Pre-Test	Helium Charge Pressure	16.0 to 17.0 bar(g) at 20°C ambient	16.6 bar(g)	[X] PASS
Pre-Test	Compressor Water Flow	Flow ≥ 5.0 L/min; Temp +5°C to +25°C	5.7 L/min / 18.5°C	[X] PASS
Pre-Test	ISO 4126 Relief Valve	Seal intact, unblocked, Viton clean	Visual Inspection OK	[X] PASS
Leak Test	Outer Jacket Leak Check	No helium signal > 1×10^{-14} mbar-L/s	Max Background: 2×10^{-11}	[X] PASS
Leak Test	Internal Helium Circuit	No Mass 4 rise when helium pressure raised	Stable @ 1×10^{-11} mbar-L/s	[X] PASS
Leak Test	RGA Background Scan	Mass 28 / Mass 32 < 4:1 (No air leak)	N ₂ /O ₂ Ratio: 1.1:1 (Clean)	[X] PASS
Thermal	2nd Stage Temp (T ₂)	T ₂ cools to ≤ 20.0 K within 90 min	12.4 K (in 64 min)	[X] PASS
Thermal	1st Stage Temp (T ₁)	T ₁ cools to 45.0 K – 80.0 K range	51.2 K	[X] PASS
Final UHV	Ultimate Base Vacuum	Achieves target base (< 1×10^{-10} mbar)	2.4×10^{-10} mbar	[X] PASS

5. Field Observations & System Authorization Sign-Off

System Field Notes: Comprehensive helium spray leak check and thermal diagnostic performed on Leybold COOLVAC 3000 iL cryopump connected to UHV Processing Chamber 2. Initial rate-of-rise test indicated elevated 2nd stage temperature (T₂ = 22.8 K) and slow base vacuum recovery. Helium spray leak check verified zero external atmospheric flange leaks (< 2×10^{-11} mbar-L/s). Executed full 295 K warm-up regeneration with dry N₂ purge to desorb accumulated H₂/H₂O. Replaced COOLPAK helium adsorber capsule (logged 10,240 operating hours). Post-maintenance cool-down achieved T₂ = 12.4 K in 64 minutes and ultimate base vacuum reached 2.4×10^{-10} mbar.

Performing Technician:	Kadeem Manning	Role / Title:	Tier II Facility Technician
Technician Signature:	<i>Kadeem Manning</i>	Date Signed:	March 22, 2022
Quality QA Reviewer:	Dr. Marcus Vance	System Vacuum Status:	[X] CERTIFIED LEAK-TIGHT & UHV READY