

SERVICE REPORT

Job Number:	SE0765	Quote:	Sage:
Customer:	Uni of Lisbon, Portugal		
Contact:	Analiza Mónica Silva		
Instrument:	20-20, Hydra, Cetac autosampler with heated block for 12ml exetainer		
Date of Visit:	26 th – 28 th August 2026	Days:	3
Engineer:	David Harris		
Reason for Visit:			
Service of instrument. Investigation into CO ₂ (¹⁸ O) equilibration sampling issues.			
Service Contract?	Yes / No	Expires:	30 JUNE 2026
Warranty?	Yes / No	Expires:	N/A
Tick as Applicable:	Call Out / Routine Service / Warranty / Emergency / Contract Visit		
Details of Visit:			
Arrived on site and inspected instrument. System is in working order. Customer reports that HD equilibration method is working successfully but ¹⁸O equilibration analysis is not functioning. No CO₂ present during sampling. Trouble shooting had taken place prior to visit via email, but cause not found. CO₂ regulator replaced for new prior to engineer's visit. The instruments have not been serviced for approx. 5 years. No filament changes, or rotary pump oil changes have been performed during this time. Rotary pump oil viewing windows shows no oil. This is below the required amount to safely operate. Viewing window look blackened from burnt oil residue. Pump also covered in soot and has not been maintained. No visible leaks present. Emissions Ok. Approx 1800µA with Trap @ 600µ. Turbo pump speeds 90k RPM (Source & Analyser side). Vac pressure good holding at 5^{e-08} mBar with all isolation valves closed. System initially set to HD mode. Tested system using HD ref gas. Results good (see below), however stepping noted during stability checks. Due to time restrains on eMag drift settle time, CO₂ not checked for precision or linearity. CO₂ Peak Shape present via Cylinder gas. No MS related reasons for ¹⁸O application to not function noted. CO₂ Stability and peak shape scans taken with eMag temp not stabilised. Precision test; n=5, 5 second ref gas pulses. Ref = 5 sec pulse Linearity test; n=5, 1-7 ref gas pulses. Ref = 5 sec pulse.			

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HD Peak Shape = OK

Ref Gas HD Precision = 0.398% S.D.

HD 2/1 Stability = Stepping observed

CO₂ Peak Shape = OK

CO₂ 2/1 Stability = Stepping observed

Proceeded to carry out PM Service.

Vented instrument – All OK.

Rotary pump oil viewing windows shows no oil. This is below the required amount to safely operate. Viewing window look blackened from burnt oil residue. Pump also covered in soot and has not been maintained. No visible leaks present

Drained old oil from rotary pump. Only a small amount of oil in pump. Oil is dark from over use.

Advise users that pump may have become damaged. Advise user to purchase and install new clean rotary pump for continued usage.

Advise user that oil replacement should take place at least once per year for continued use of instrument.

Stainless steel flexi tubing from rotary pump to turbo pumps are filled with oil residue. Rear Analyser pump in especially bad condition. Removed flexi tubing and attempted to clean.

Added new Edwards 15 Ultra grade oil to rotary pump. Refitted all parts & pump.

Removed Source, removed old filament. Fitted new filament. No issues with Ion Source assembly noted.

Fitted new copper gasket and PTFE ferrule to inlet. – All OK.

Activated pumps. Turbo pump speeds 90k RPM (Source & Analyser side).

Edwards AIM gauge fail. Both Source and analyser side vac gauges do not respond. Broken ceramic material observed withing red pod unit.

Tested readback with dummy head. SysCon shows readback as expected with test box (Cables and SysCon have no fault). Issue must be present within AIM gauges.

Possible contamination from oil suspected. Possible failure due to age of gauges. On day 3 gauges were removed for inspection. Oily substance observed within Analyser gauge. No contamination noted on Source side gauge. Engineer attempted to clean both gauges with ethanol. Gauges swapped in orientation when refitted. Upon re-pump down part failure persists.

Advise user to purchase and install new AIM gauge onto system. Model on unit now obsolete, Edwards have a new type of gauge which can be fitted. New gauge model type will require a software update for software to readback.

Unable to see vac pressure without operating AIM gauge. Engineer waited overnight to attempt filament conditioning. Vacuum should have had enough time to fully form in this timer period but Engineer notes that it is unknow what vac pressure is from this point forward during service visit.

Conditioned filament - All OK. Emissions approx. 150µA (with Trap set to 50µA in standby).

System set to high mass mode for CO₂ analysis. Emaget allowed to settle overnight – All OK.

Retuned instrument as required.

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Checked N₂ background levels (with isolation valves closed) to monitor if leak present on flight tube (vacuumed) areas. N₂ background levels relatively low and dropping over time. This indicates that there is no leak present on system. Exact vac pressure unknown due to AIM gauge failure. Possible that vac pressure still higher than desired level.

Checked tuning, peak shapes and stabilities for CO₂ Ref Gas - All OK.

¹³C & ¹⁸O results good for precision and linearity testing.

Precision test; n=5, 5 second ref gas pulses. Ref = 5 sec pulse

Linearity test; n=5, 1-7 ref gas pulses. Ref = 5 sec pulse.

Ran post service Ref Gas specs.

CO₂ 2/1 Peak Shape = OK

Ref Gas ¹³C Precision = 0.046‰ S.D.

CO₂ 2/1 Stability = OK

Ref Gas ¹³C Linearity = 0.172‰ S.D.

CO₂ 3/1 Peak Shape = OK

Ref Gas ¹⁸O Precision = 0.037‰ S.D.

CO₂ 3/1 Stability = OK

Ref Gas ¹⁸O Linearity = 0.098‰ S.D.

Investigated issue with ¹⁸O equilibration method.

No comms issues noted with prep unit.

All valves respond OK.

All events correct. No method related issues noted.

CETAC autosampler responds with no issues. All coordinates OK for sampling positions – All OK.

Slight kink in peek tubing on needle exit. Removed fitting, cut down line, refitted connection.

Gas flow measured at exit flush vent when flowing via injected exetainer (approx. 40ml/min).

Upon inspection for type of gas present; CO₂ gas line closed. H₂ gas line open. Switched to CO₂ gas flow.

User unaware that CO₂ required manually opening prior to ¹⁸O testing. Exetainers were being flushed with H₂ gas instead of CO₂ during testing. No CO₂ was present for analysis. Customer notes that last time analysis was undertaken the instrument had a lab tech. Lab tech has since departed. New users unaware of plumbing situation. Advised users on plumbing of equilibration gas.

CO₂ flush flow low via exetainer. Adjusted flow crimp and regulator pressures are required. Flow for both CO₂ & H₂ now approx. 40-50ml/min through exetainer.

Test samples yield CO₂ peaks as expected. Ran test batch with dry vials (no equilibration, 100% CO₂ cylinder gas present in exetainer). Results good. Large volume of sample present. (n=5).

HYDRA ¹³C Precision = 0.032‰ S.D.

HYDRA ¹⁸O Precision = 0.039‰ S.D.

N.B. ~ Customer ran own sample set over night to test response. Customer reports that results are good and are in expected range.

Method made to switch system into HD mode following customer sample batch. Multi batch function used. System set to HD mode over night successfully. Trained users in use of Multi batch setup up and advised how

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this can be useful with equilibration application. (Flush exetainers with equilibration, set delay time, automatically run sample batch).

HD tuning displays unstable trap current with required high IR setting (+30 to 50V) with trap settings above 300 μ A.

Unstable Trap current is more likely with HD tuning due to higher voltages used vs CO₂ / N₂ tunings with current emag setup. Issue not observed in previous CO₂ / N₂ tunings. Issue could also be vac pressure related, however vac pressure unknown at time of analysis.

Vented instrument to inspect Ion Source. No obvious signs of problem.

Filament condition looks good. Possible crack on ceramic ring to trap. Part replaced for new. Opened up ionisation chamber to inspect IR plate. Minor adjustments made. Source assembly rebuilt and part refitted.

AIMs gauges removed for inspection (see note above for details). Gauges refitted. Still no response during pump down.

System pumped down for retesting. Re-fired filament after several hours wait. Filament fires okay.

HD tuning now stable. Issue resolved.

HD peak shape not optimal. Adjusted eMag position as required. Retuned HD as required.

Peak shape & 2/1 stability good. Tested with H₂ Ref gas – Results good (see below for details).

Precision test; n=5, 5 second ref gas pulses. Ref = 5 sec pulse

Tested HD from 12ml exetainers (dry) batch. Test samples yield HD peaks as expected. Ran test batch with dry vials (no equilibration, 100% H₂ cylinder gas present in exetainer). Results good. (n=5). (see below for details).

S AIM gauge began reading incorrect value (3.0^{e-03}mBar). This locked out HT & Source capabilities. Swapped red pods over between gauges, incorrect readback followed red pod to A readback. Red pod deemed to be damaged. Part removed. S side gauge now displays non-readback. This allows the system to still be used.

System changed over to high mass mode for CO₂ re-testing. Time required to allow eMag temp to settle. Tuning and analysis took place after hours via dial in session due to time constraints on site.

During dial in session S AIM gauge fired and began readback. Read back of correct values. Vac pressure of 5.8^{e-08} mBar observed with all isolation valves closed. Vac pressure good.

Engineer still recommends purchase of AIM gauge as backup part in case current AIM gauge fails again.

Engineer re-ran CO₂ Ref gas specification testing. Checked tuning, peak shapes and stabilities for CO₂ Ref Gas - All OK.

¹³C & ¹⁸O results good for precision and linearity testing.

Precision test; n=5, 5 second ref gas pulses. Ref = 5 sec pulse

Linearity test; n=5, 1-7 ref gas pulses. Ref = 5 sec pulse.

Ran test batch with dry vials (no equilibration, 100% CO₂ cylinder gas present in exetainer). Results good. Large volume of sample present. (n=5).

See below for final results of testing...

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HD 2/1 Peak Shape = OK

HD 2/1 Stability = OK

Ref Gas ^{HD} Precision =0.279‰ S.D.

CO₂ 2/1 Peak Shape = OK

CO₂ 2/1 Stability = OK

CO₂ 3/1 Peak Shape = OK

CO₂ 3/1 Stability = OK

Ref Gas ¹³C Precision =0.023‰ S.D.

Ref Gas ¹³C Linearity =0.114‰ S.D.

Ref Gas ¹⁸O Precision =0.014‰ S.D.

Ref Gas ¹⁸O Linearity =0.082‰ S.D.

HYDRA HD Precision =0.14‰ S.D.

HYDRA ¹³C Precision =0.007‰ S.D.

HYDRA ¹⁸O Precision =0.027‰ S.D.

Service complete instrument handed back to customer for normal usage.

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Recommendations:

- Advise users that pump may have become damaged. Advise user to purchase and install new clean rotary pump for continued usage.
- Advise user that oil replacement should take place at least once per year for continued use of instrument.
- Advise user to purchase and install new AIM gauge (X2) onto system. Model on unit now obsolete, Edwards have a new type of gauge which can be fitted. New gauge model type will require a software update for software readback function.
- [After current S AIM gauge began reading back] Engineer still recommends purchase of AIM gauge as backup part in case current AIM gauge fails again.

Follow Up:

N/A

Parts Used:	Stock Number:	Quantity:	Charge:
Service Kit + Edwards 15 Oil	SC9001	1	Included in service
Total:			N/A

Engineer: David Harris

Date: 31st August 2026

Signed:



Customer:

Date:

Signed:

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NOTES:

N/A