

JSC-622: HTE vial for O2 measurements

Date: 2024-03-13
Tags: O2 JSC Calibration HTE
Status: Done
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Reagents

Name	CAS Number / Experiment Number	Amount [mmol]	Equivalents	Mass _{theo} [mg]	Mass _{exp} [mg]	Molar mass [g/mol]	Volume [ml]
Milli-Q Water	/	/	/	/	/	/	ca. 10 ml

Procedure/observations

All steps (unless stated otherwise) were carried out under argon using either standard [Protocol - Schlenk Technique](#) or in an argon filled glovebox (MBRAUN).

Date	Time	Step	Observations
12.03.2024	17:00	A PyroScience trace range oxygen sensor spot as well as a PyroScience optical temperature sensor spot were glued into a HTE glas vial provided by Michael Ringleb (silicone glue from PyroScience)	Green O2 sensor spot and white temperature sensor spot
		Two 3D printed adapter to hold the optical fiber cables (printed by Michael Ringlet, dimensions: width 12 mm, height 8 mm, diameter of inner hole 2.55 mm, ca. 15 adapters were printed, four were selected that fit the optical fiber cables well) were glued on the outside of the vial (silicone glue from PyroScience)	White optical fiber cable adapters IMG_8721.jpeg
		The silicone glue was allowed to cure over night	
13.03.2024	16:00	The optical fibers were connected to the adapters and a Therma Pt100 was placed inside the vial on the same height as the sensor spots.	
	16:10	The vial was filled with. ca. 10 ml of water	
	16:30	A 1-point calibration of the temperature sensor spot was performed using the Therma Pt100 as reference	

	16:50	A 2-point calibration of the oxygen sensor spot was performed. For this, the Thermo Pt100 was removed from the vial and a slit septum cap was screwed onto the HTE vial. Through the septum cap two needles were inserted (one in the liquid phase, one in the gas phase) and through the liquid phase needle, air was bubbled through the water phase with a balloon. Once a steady state was reached, the upper calibration point was set.	
	17:00	The balloon was removed and the liquid phase needle was connected to argon, bubbling argon through the water phase	
	17:20	Once a steady state was reached, the lower calibration point was set	
	17:30	Both needles were removed from the vial and a log was started (JSC-622)	2024-03-13_170705_JSC-622.txt Graph01.png O2 leakage of ca. 0.5 umol(O2)/l within 10 min
	17:40	The log was stopped.	
	17:50	The instrument configuration was saved.	240313_Instrument_Settings_HTE_Vial_Liquid_Phase.ini

Analysis

Date	Time	Sample name	Analysis method	Analytical device	Solvent	Raw Data	Processed Data	Interpretation
14.03.2024	17:30	JSC_622	Liquid phase optical O2 measurement	FireStingO2	H2O	2024-03-13_170705_JSC-622.txt	Graph01.png	O2 leakage of ca. 0.5 umol(O2)/l within 10 min for a slit septum cap that was punctured ca. 5 - 7 times

Attached files

IMG_8721.jpeg

sha256: 2eff0707512f0f40b7edd2b7e52306f66f2bd0c02c794b5a5c0fac4da12e0b68



240313_Instrument_Settings_HTE_Vial_Liquid_Phase.ini

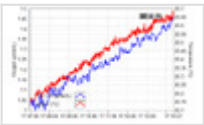
sha256: 21873b82b576912e5cbd662fc747c6d2b2fdcf5acb66e507838ccb642d296a2c

Firesting-O2-2-Channels_-Ch.1_Oxygen.txt

sha256: 4635281cb9495c2fe1116527defcdc71502a0c1d941aa1bf642ad33df5c36170

Graph01.png

sha256: c74611111af9c5321b5de2d8ab82381719e9d0cdf6445bd9e2f22829e6c7513b



2024-03-13_170705_JSC-622.txt

sha256: e3373091029d9dafd04f8dea703ec3aeefda9fe9474a49c9c0b3de22bbc5e0c0



Unique eLabID: 20240313-460f266ea8a58ad83130a0a59f2d6e0de9c37603

Link: <https://elab.water-splitting.org/experiments.php?mode=view&id=888>