Electrochemistry and Microfluidic Environments for the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS

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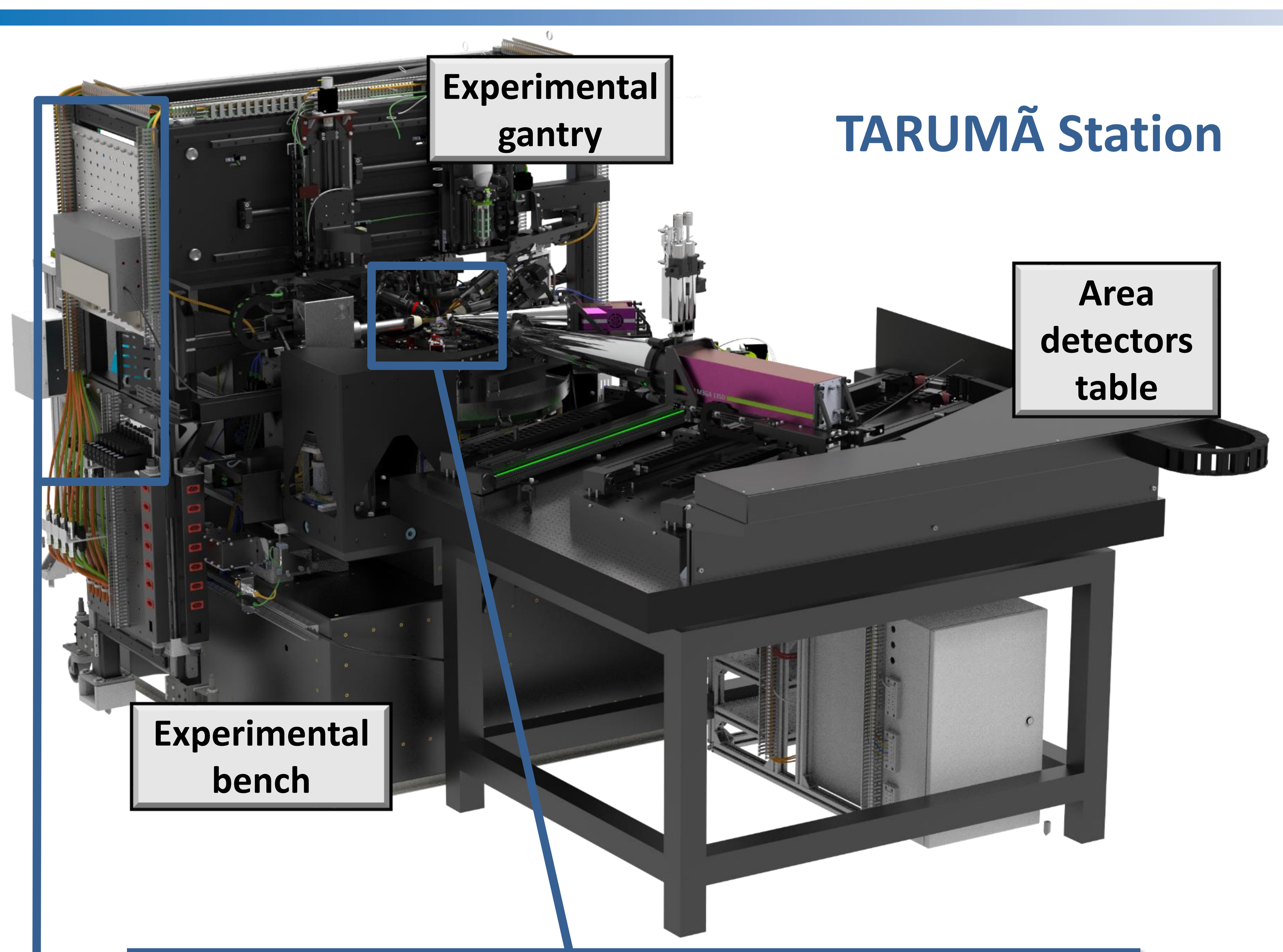
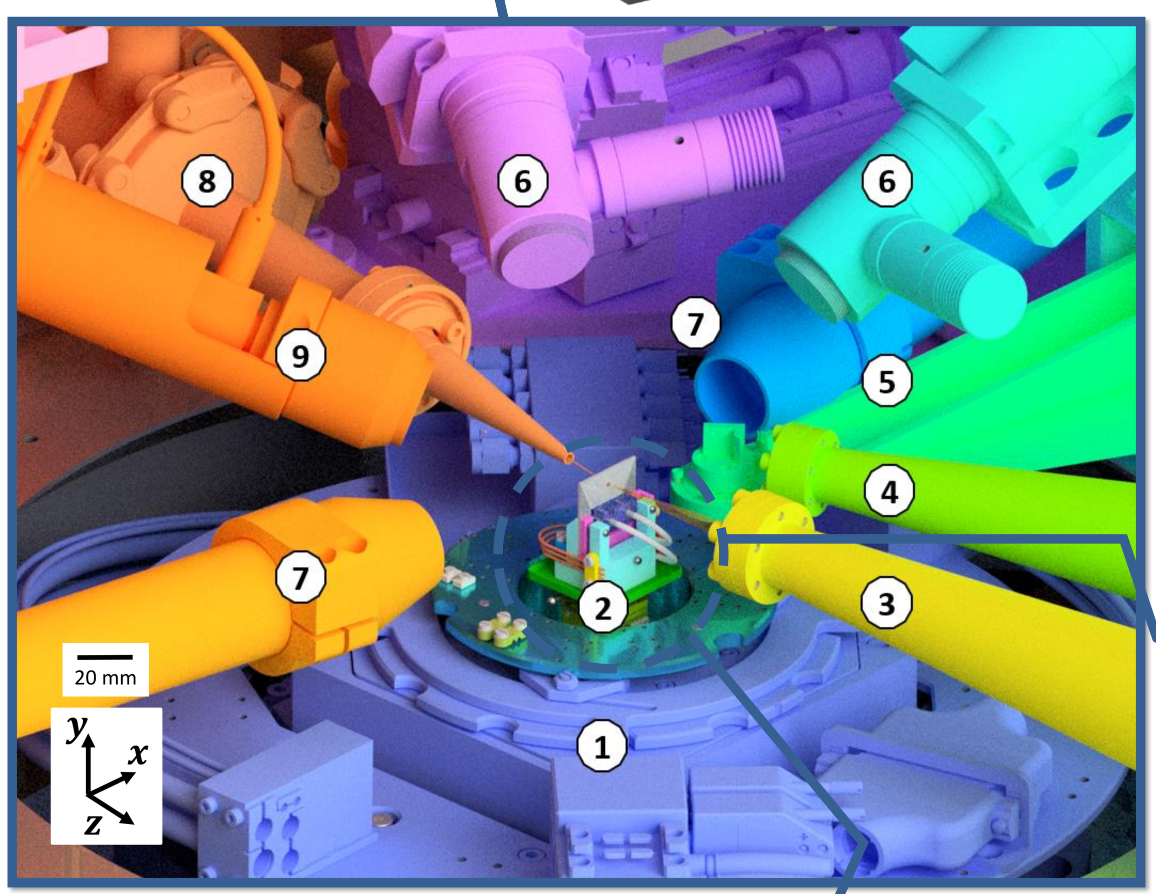
CNPEM Brazilian Center for Research in Energy and Materials

TARUMÃ Station





1. Sample positioning stages;
2. Sample environment setups;
3. Transmission area detector;
4. Diffraction area detector;
5. Crystal analyzer spectrometer;
6. Optical microscopes;
7. Fluorescence detectors;
8. KB vessel;
9. Luminescence detector (XEOL optics).

Abstract

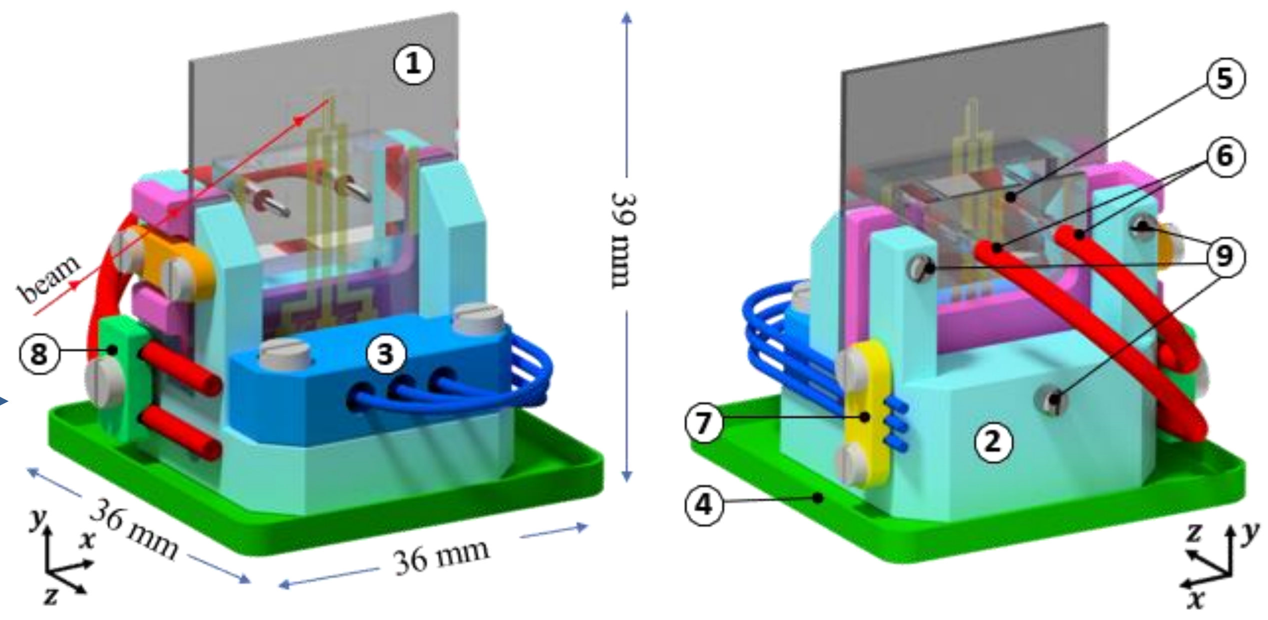
TARUMÃ [1] is a multi-technique sub-microprobe experimental station of the CARNAÚBA [2] (Coherent X-Ray Nanoprobe Beamline) beamline at Sirius [3], the 4th-generation Synchrotron Light Source at the Brazilian Synchrotron Light Laboratory (LNLS). This work describes two related setups that have been developed in-house for TARUMÃ: a small-volume electrochemical cell, and another multifunctional environment that can be used both as a microfluidic device and as an electrochemical cell that allows for fluid control over electrodes. The mechanical design of the devices, as well as the architecture for the fluid and electrical supply systems are described in detail.

Optics Overview:
- ❑ Undulator source;
- ❑ 2.05 to 15 keV;
- ❑ Four-bounce monochromator;
- ❑ All-achromatic optics;
- ❑ Flux up to 1e11 ph/s/100 mA;
- ❑ KB focusing: 550 to 120 nm;
- ❑ Large working distance: 440 mm.

Features:
- ❑ Simultaneous multi-analytical X-ray techniques;
- ❑ Macro and micro sample holders: from centimeter range samples to microscopy standards;
- ❑ Special sample setups: cryogenic, Rhizomicrocosm, electrochemistry, electrocatalysis, batteries, etc;
- ❑ Sub-millisecond hardware integration;
- ❑ High-speed flyscan mapping;

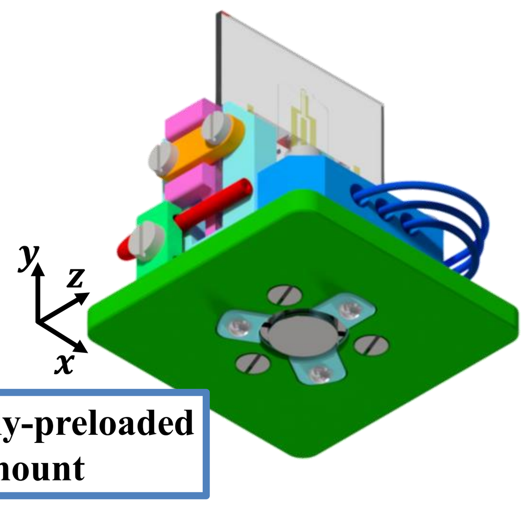
Techniques:
- ❑ XRD
- ❑ XAS
- ❑ XRF
- ❑ XEOL
- ❑ Ptycho-CDI
- ❑ Ptycho-Bragg-CDI
- ❑ Tomography

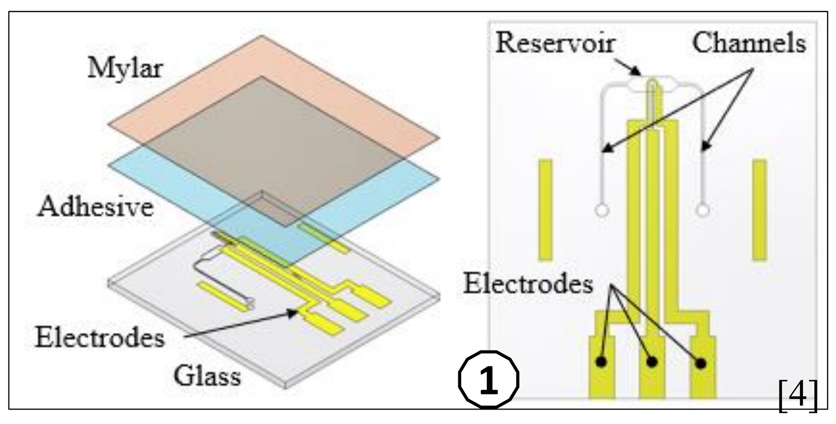
Microfluidic Setup



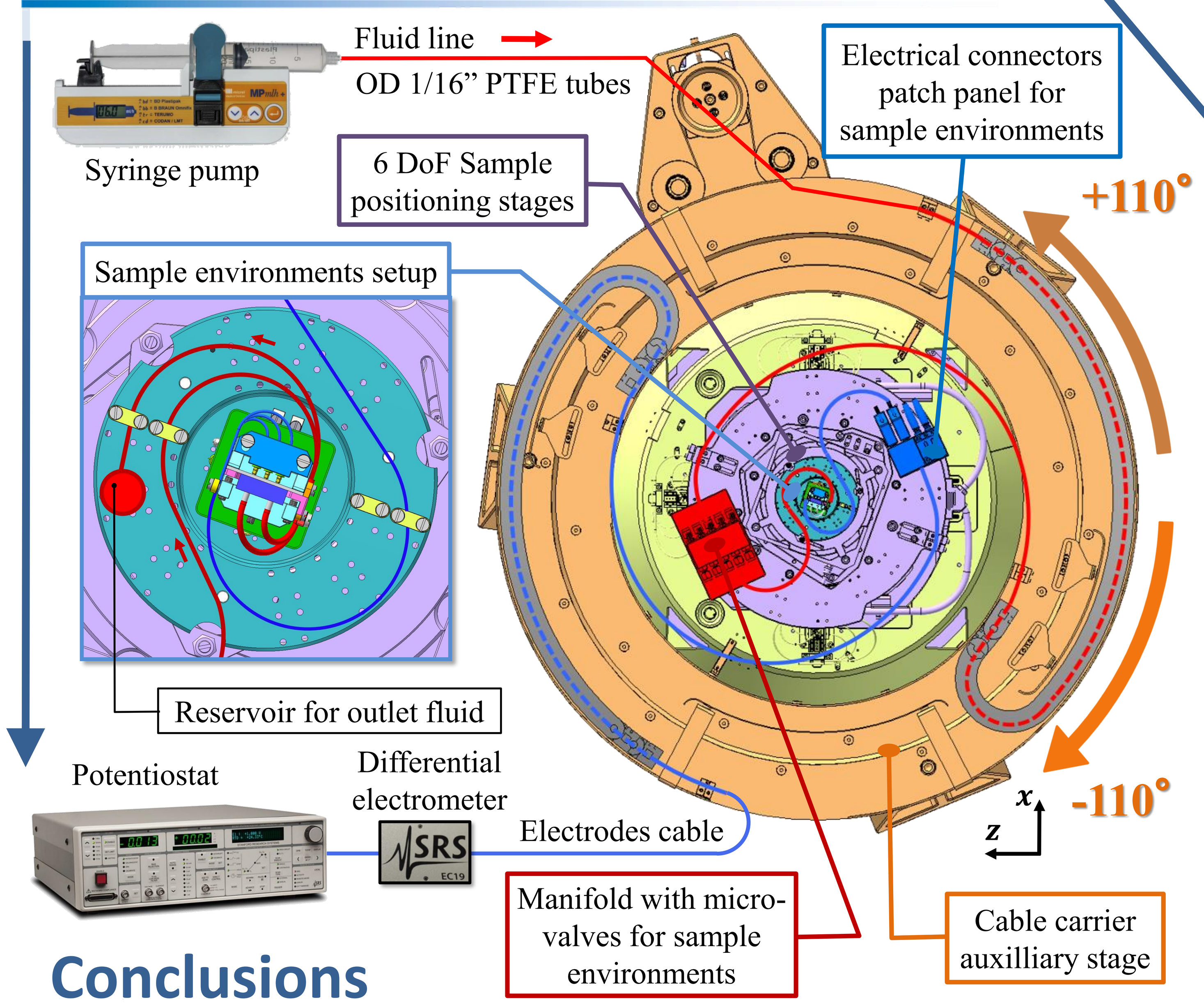
- ❑ Reduced-size assembly;
- ❑ Lightweight (~ 35 g) design oriented to high dynamics for flyscan mapping;
- ❑ Magnetically-preloaded kinematic mount for determinism and easy exchangeability;
- ❑ Microliter reservoir with controlled flow;
- ❑ Electrodes for electrochemistry/catalysis;
- ❑ Cable management;
- ❑ Leak containment;

1. Microfluidic device (glass + Mylar);
2. Main frame (Al);
3. Electrical connector (POM + contact);
4. Containment tray (Al);
5. PDMS closing structure;
6. Inlet/outlet tubes (Silicone);
7. Cable clamp
8. Tube clamp
9. Fastening screws

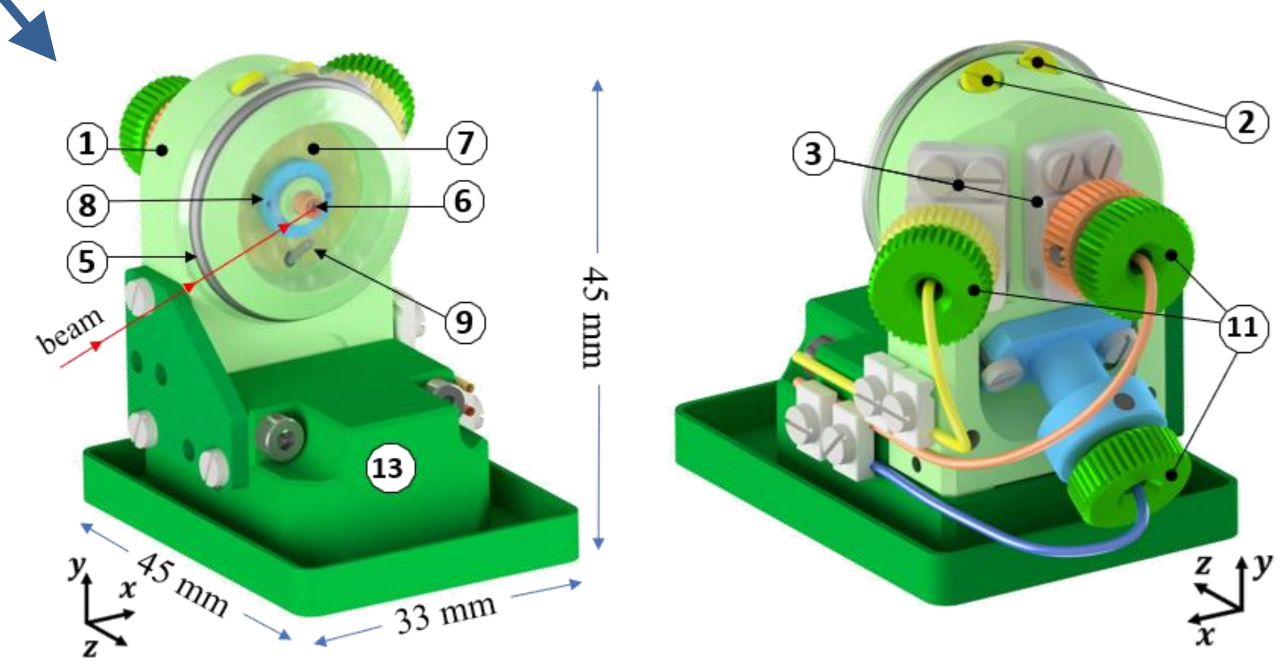




Supply System

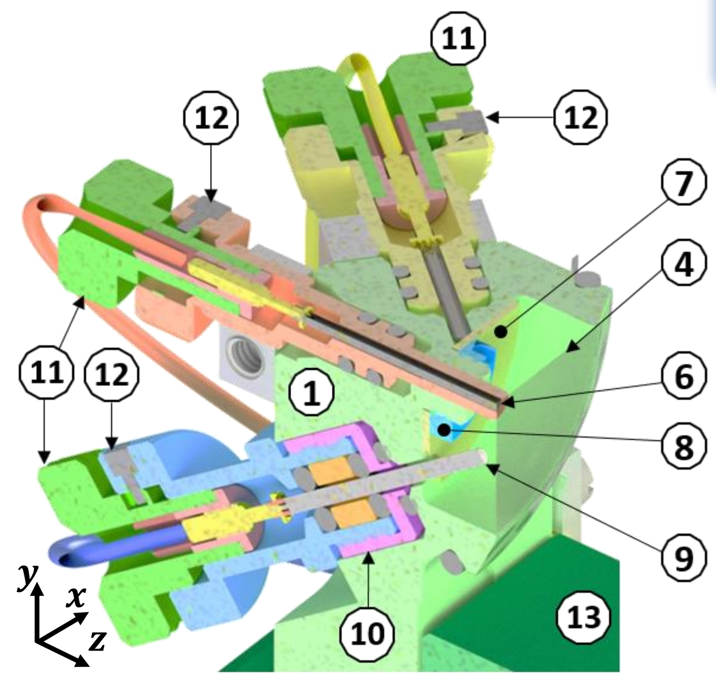


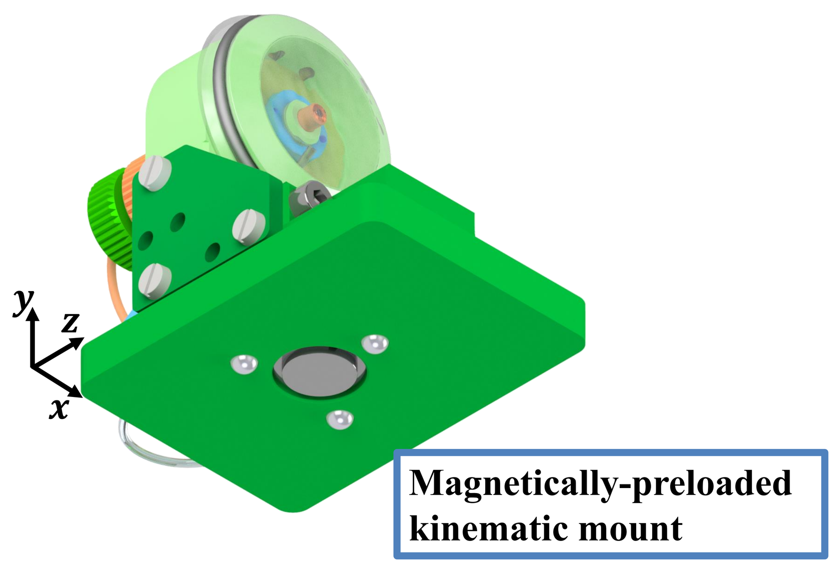
Electrochemical Setup



- ❑ Reduced-size assembly;
- ❑ Lightweight (~ 40 g) design oriented to high dynamics for flyscan mapping;
- ❑ Magnetically-preloaded kinematic mount for determinism and easy exchangeability;
- ❑ Container for 1 ml static electrolyte;
- ❑ 3 exchangeable electrodes;
- ❑ Fast-lock electrical connectors;
- ❑ Cable management
- ❑ Leak containment

1. Main frame (PEEK);
2. Auxiliary access points;
3. Nuts of electrode housings;
4. Container sealing film;
5. Container sealing ring;
6. Working electrode;
7. Counter electrode;
8. Nut of counter electrode;
9. Reference electrode;
10. Nut of reference electrode;
11. Electrical connectors;
12. Connector lock pin;
13. Interface base (POM).





Conclusions

Two sample environments for in situ experiments in electrochemistry and microfluidic have been designed in-house for compatibility with the multiple X-rays techniques at the TARUMÃ station. They are based on previous experience in the field and follow precision engineering concepts, not only for stability with respect to the nanometric beam and but also for flyscan compatibility. After validating assembling and sealing concepts via preliminary prototypes, the microfluidics cell has just been manufactured and the electrochemical cell is in procurement. They are expected to be finalized and fully commissioned soon, becoming part of the station experiments portfolio in the second half of 2021.

Acknowledgements

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References

[1] Geraldes, R. R., et al. "Design and Comissioning of the TARUMÃ Station at the CARNAÚBA Beamline at Sirius/LNLS," presented at MEDSI 2020, Chicago, USA, 2021, this conference.
[2] Tolentino, H.C.N., et al., "CARNAÚBA: The Coherent X-Ray Nanoprobe Beamline for the Brazilian Synchrotron SIRIUS/LNLS," J. Physics: Conf. Series 849 (1), 012057, 2017.
[3] Sirius Project, https://www.lnls.cnpem.br/sirius-en/ (17 July 2021).
[4] Neckel, I. T., et al., "Dispositivo microfluídico selado com película de poliester", September 30th, 2020, Patent App. BR 10 2020 020040 2.

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