

After the RISEnergy Transnational Access, Users are required to submit a User Report. This should be done within 4 weeks after the Access is completed unless otherwise agreed. The User Report will be given to the User(s) by the WP2 leader. The report contains sections related to the work performed, the main results and observations that were achieved.

This document should be completed, signed, and sent by e-mail to [risenergy@for.kit.edu](mailto:risenergy@for.kit.edu).

Summary questionnaire for Users who have been granted Transnational Access (TA) under the RISEnergy project Horizon Europe TA scheme. More information on RISEnergy TA can be found in "General Rules" and in "Access Policy" which can be found on the RISEnergy webpage.

Please complete, sign, and send this form, together with the Cost claim by e-mail to [risenergy@for.kit.edu](mailto:risenergy@for.kit.edu) with title: *RISEnergy APPXXX - reports*.

<b>General information about the project</b>	
Project title (as used in Application)	Advanced Electrolyte Materials for High-Performance Fuel Cells
Project number (APPXXX) and acronym (max 15 characters)	APP163 E-Fuel
RISEnergy RI(s) accessed	Laboratory Center of Competence HITMOBIL
Keywords (up to five, free text)	Proton conducting electrolytes, Ceramic Electrolytes, Polymer and Inorganic Composites, Electrochemical Impedance Spectroscopy
Arrival date (in town where RI is located)	18/07/2025 (1 <sup>st</sup> access) and 02/10/2025 (2 <sup>nd</sup> access)
Departure date (from town where RI is located)	31/07/2025 (1 <sup>st</sup> access) and 16/10/2025 (2 <sup>nd</sup> access)
Starting date of Access (first day at RI)	19/07/2025 (1 <sup>st</sup> access) and 03/10/2025 (2 <sup>nd</sup> access)
Finishing date of Access (last day at RI)	30/07/2025 (1 <sup>st</sup> access) and 15/10/2025 (2 <sup>nd</sup> access)
Number of days not using the RI (during the above period)	4 on 1 <sup>st</sup> access and 4 on 2 <sup>nd</sup> access
Reason for not using RI those days (describe)	The access for this RI was split into 2 access sessions. The first access to the RI was made from the 18 <sup>th</sup> of July to the 31 <sup>st</sup> of July of 2025. During this period, the RI was not used on the arrival and departure days, due to the flight's hours of arrival and departure, as well as on Sundays (20 <sup>th</sup> and 27 <sup>th</sup> of July). During the 2 <sup>nd</sup> access session, the RI was not used on the arrival and departure days, due to the flight's

	hours of arrival and departure, as well as on Sundays (5 <sup>th</sup> and 12 <sup>th</sup> of October).
Number of days using the RI	10 (1 <sup>st</sup> access) + 11 (2 <sup>nd</sup> access) = 21
Number of Users granted Access (group size)	2 persons (1 <sup>st</sup> access) and 1 person (2 <sup>nd</sup> access)
Comments	
<b>User</b>	
<b>User group leader or sole applicant (user group member 1)</b>	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
User travelling to RI?	
Comments	
<b>User group member 2</b>	
First name	
Last name	
Affiliation / Employer	
Country of Employer	
E-mail	
User travelling to RI?	
Comments	
<b>Access Summary Report - work performed and initial results</b>	
Brief description of the objectives of your project (up to 200 words)	
<p><i>The project aimed to investigate advanced proton-conducting electrolytes for intermediate-temperature fuel cells (ITFCs) through the development and characterization of ceramic-polymer composite materials. The main goal was to compare the structural and electrochemical behavior of traditional proton-conducting ceramic electrolytes (<math>BaZr_{0.8}Y_{0.2}O_{3-\delta}</math>) with innovative cold-sintered composite membranes incorporating cesium hydrogen phosphate (<math>CsH_5(PO_4)_2</math>) and polybenzimidazole (PBI). This approach sought to overcome the limitations of high-temperature ceramic processing by applying cold sintering, a low-energy densification method that enables co-processing of inorganic and polymeric phases.</i></p> <p><i>Through microstructural and electrochemical analysis, the project aimed to assess the feasibility of using these materials as efficient proton-conducting membranes under moderate temperatures (&lt;180 °C). The study contributes to the broader objective of</i></p>	

developing sustainable, low-cost electrolyte materials suitable for next-generation fuel cell technologies.

#### Activities performed (up to 600 words)

The access at HITMOBIL was divided into two periods: 18–31 July 2025 and 2–16 October 2025.

Prior to the accesses, ceramic  $\text{BaZr}_{0.8}\text{Y}_{0.2}\text{O}_{3.6}$  (BZY) powders were synthesized and structurally characterized at the University of Aveiro using X-ray diffraction (XRD). These materials served as the traditional proton-conducting electrolyte, being characterized by Electrochemical Impedance Spectroscopy (EIS) at HITMOBIL. During the first visit, preliminary EIS results were obtained for a sintered BZY sample with NiO, as sintering additive, at a temperature of 1500 °C. After analyzing these results, and with the intent of improving proton conductivity, another sample was produced for the 2<sup>nd</sup> access, with a higher sintering temperature of 1600 °C. EIS was also performed on this sample. Regarding the testing conditions, both samples were tested in  $\text{H}_2$  atmospheres, dry and wet, from the temperature of 400 to 800 °C.

Regarding the composite samples, they were prepared at the University of Aveiro. The process began with the dissolution of polybenzimidazole (PBI) in dimethylformamide (DMF) to form a homogeneous polymer matrix. Cesium hydrogen phosphate ( $\text{CsH}_5(\text{PO}_4)_2$ ) powder was added to the polymer solution, and the resulting mixture was then pressed into pellets (13 mm diameter) using a uniaxial press applying 4 tons of force. The cold sintering process was conducted at 180 °C for 1 hour, exploiting the partial plasticity of the composite to achieve densification at temperatures well below those required for conventional ceramics. For the first visit, the following samples were prepared: 0 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , 25 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , 50 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , 75 % V.  $\text{CsH}_5(\text{PO}_4)_2$  and 100 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , with the remaining volume percentage comprising of the polymer (PBI). During the first visit, preliminary EIS were obtained for all the samples from 100 to 180 °C, the maximum operational limit of the HITMOBIL Baltic Fuel Cells setup, in multiple gas conditions (e.g. dry  $\text{H}_2$ , wet  $\text{H}_2$ , dry  $\text{N}_2$  etc.) to evaluate proton conductivity. X-ray diffraction (XRD) was performed on these samples to assess crystallinity and phase composition after processing. After analyzing the obtained EIS results, more compositions were synthesized by the same method: 85 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , 90 % V.  $\text{CsH}_5(\text{PO}_4)_2$  and 95 % V.  $\text{CsH}_5(\text{PO}_4)_2$ , with the remaining volume percentage comprising of the polymer (PBI). These 3 samples were characterized in the HITMOBIL infrastructure using EIS, as well as secondary electron microscopy (SEM). I-V curves were performed on the best performing sample, in order to access its stability to current loads. Since the HITMOBIL Baltic Fuel Cells setup was limited to 180 °C, a furnace was used to access thermal stability of the 95 % V.  $\text{CsH}_5(\text{PO}_4)_2$  sample, up to 240 °C, in order to verify if it was possible to use another equipment to perform EIS, up to 240 °C. Since the sample did not degrade visually, it was tested using another EIS test rig, that could withstand the higher temperature. The sample was tested between room temperature and 240 °C, in both wet and dry  $\text{H}_2$  atmospheres.

Data collection and characterization was supported by the HITMOBIL staff. All measurements were systematically documented, and the resulting raw and processed datasets were stored following the agreed data management plan.

#### Scientific results (up to 800 words)

Regarding the BZY samples, the XRD characterization made displayed that pure crystalline phases of the material were obtained in both cases. Regarding EIS

characterization, in both dry and wet atmospheres, the resistance of the bulk and grain boundaries decreased with the increase in temperature. For the 1500 °C, comparing the results of EIS at 800 °C, for wet and dry H<sub>2</sub> atmospheres, it is visible that in wet atmospheres the resistance of the sample is lower. Nevertheless, in both cases, a conductivity around 10<sup>-2</sup> S·cm<sup>-1</sup> was achieved at this temperature. On the 1600 °C sample, the conductivities seem to be lower (10<sup>-3</sup> S·cm<sup>-1</sup>), when compared with the latter. In terms of results in wet and dry atmospheres, no really noticeable differences were observed in the tests.

The cold-sintered composites exhibited satisfactory structural integrity and homogeneity, demonstrating the feasibility of low-temperature densification via the cold sintering route. XRD analysis confirmed that the CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> phase was present in all of the samples, although CsH<sub>2</sub>PO<sub>4</sub> was detected as a secondary phase. The PBI XRD presented amorphous peaks. Regarding the composite samples, this amorphousness related with PBI was only visible in the 25 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> sample, as the peaks related with the crystalline phases presented much higher intensities.

Scanning Electron Microscopy (SEM) was carried out on the 85 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>, 90 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> and 95 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>, where it was possible to clearly distinguish the organic PBI phase from the ceramic CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>, as well as grain boundaries between each grain. For all of these samples, grains are on a micrometer scale.

EIS measurements were carried out between 100 and 180 °C in the Baltic Fuel Cells Setup. Regarding the PBI sample, the results obtained only depicted noise, probably due to its high resistance. It was not possible to measure the 25 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> sample, as its thickness was higher than the thickness permitted for the Baltic Cell to close properly. The obtained bulk conductivity values, for an H<sub>2</sub> atmosphere, were in the range of 10<sup>-7</sup>–10<sup>-6</sup> S·cm<sup>-1</sup> (at 180°C), increasing steadily with temperature, for the samples 50 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>, 75 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>, 85 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> and 90 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>. It was not possible to determine the grain boundary conductivity of the samples, due to the temperature and frequency range of the Gamry Potentiostat. Regarding the 95 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> and 100 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> samples, their total conductivity was possible to be determined and was around 10<sup>-4</sup> S·cm<sup>-1</sup>. Although these conductivities are moderate compared to the superprotonic regime of related phosphates, they clearly indicate protonic conduction pathways within the composite structure. The relatively low conductivity can be attributed to the temperature limitation of the setup. The 100 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> sample ended up melting during its measurement at 180 °C, as the melting point of CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> is around 150 °C. Since the 95 % V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> sample held up to the temperature of 180 °C, in the Baltic Fuel Cells setup, and had the highest conductivity of the stable samples, more tests were warranted to verify its conductivity at higher temperatures. Taking this into mind, the samples' thermal stability was first tested in a furnace, at the temperature of 240 °C. The sample visually maintained its structure. So, it was tested in the a higher temperature EIS setup. The highest conductivity for this sample was in the order of magnitude of 10<sup>-3</sup> S·cm<sup>-1</sup>, achieved at 225 °C, in dry H<sub>2</sub>, which, interestingly, was not the highest temperature of testing (~240 °C). This sample was additionally tested in the Baltic Fuel Cells Setup by performing I-V curves, in order to verify its stability under current loads. Under H<sub>2</sub> atmospheres, after 5 cycles, the samples proved to be stable, by always outputting similar currents. In the assays made in N<sub>2</sub> atmospheres, this behavior was not observed, with the sample showing instability in the current created. Nevertheless, the results confirmed that cold sintering enables stable composite fabrication at only 180 °C, well below conventional ceramic sintering temperatures (>1000 °C). This validates the technique as a low-energy and environmentally friendly densification route. Measured protonic behavior supports the material's suitability for intermediate-temperature electrochemical devices, with potential for optimization through adjustments in polymer content, sintering pressure, and additive selection.

### Interpretation of the results (up to 400 words)

Regarding the BZY 1500 °C sample, the fact that the resistance decreased with the temperature in H<sub>2</sub> atmospheres, showing conductivities up to 10<sup>-2</sup> S·cm<sup>-1</sup> is a possible signal of the presence of proton conductivity, as desired. The increase in conductivity in the wet atmosphere, might be related with the proton conduction mechanisms in BZY, which further demonstrates the existence of proton conduction. The fact that the BZY 1600 °C sample showed lower conductivities could be related with the higher temperature of sintering, which could lead to Barium evaporation, tempering with the stoichiometry in the perovskite, worsening the conductivity.

The experimental findings demonstrated that CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>-PBI composites processed by cold sintering exhibit stable structures, effective polymer integration, and reproducible proton conductivity.

The SEM results show the ceramic and polymer phases, with little porosity, which might be an indicator of the densification of the electrolyte samples, under the currently employed cold sintering conditions. Although the conductivity of the best sample (~10<sup>-4</sup> S·cm<sup>-1</sup> at 180 °C) falls below typical target values for ITFC electrolytes, it confirms that proton transport occurs even under the low-temperature conditions imposed by the experimental setup.

The thermal stability of the sample was evaluated up to 240 °C, being possible to extrapolate that the use of PBI proved to be valuable, as it prevented the sample from deforming, even at temperatures higher than the melting point of the Cesium Phosphate phase, which is essential for its use as a proton conducting electrolyte. In comparison, the 100% V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> sample, although presenting a comparable conductivity, melted and consequently deformed, after being tested up to 180 °C. When tested up to 240 °C, the 95% V. CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub> achieved a conductivity of 10<sup>-3</sup> S·cm<sup>-1</sup> at 225 °C, which seems to prove the existence of proton conductivity in the sample. The decrease in conductivity for higher temperatures could be related with some degradation in the sample.

The study thus validates the concept of using cold sintering as an energy-efficient route to fabricate composite electrolytes. Future work will explore higher-temperature setups and optimized composite ratios to further enhance proton mobility. Overall, the results confirm that cold sintering is a promising processing strategy to develop mechanically stable, low-energy, and potentially scalable composite electrolytes for sustainable fuel cell technologies.

### Main achievements during the TA related work (up to 250 words)

*During the access periods at HITMOBIL, key achievements were obtained in the processing and characterization of both traditional and composite proton-conducting electrolytes for intermediate-temperature fuel cells.*

*For the BaZr<sub>0.8</sub>Y<sub>0.2</sub>O<sub>3-δ</sub> (BZY) ceramics, XRD confirmed single-phase perovskite structures, while EIS revealed protonic conduction in both dry and wet H<sub>2</sub> atmospheres. The sample sintered at 1500 °C displayed the highest conductivity (~10<sup>-2</sup> S·cm<sup>-1</sup> at 800 °C), whereas the 1600 °C sample exhibited slightly lower values (~10<sup>-3</sup> S·cm<sup>-1</sup>), likely due to barium volatilization.*

*At HITMOBIL, cold-sintered CsH<sub>5</sub>(PO<sub>4</sub>)<sub>2</sub>-PBI composites, fabricated at 180 °C under 4 tons of pressure, were successfully characterized. Structural (XRD), morphological (SEM), and electrochemical (EIS) analyses confirmed phase stability, dense microstructures, and*

protonic conductivity ranging from  $10^{-7}$  to  $10^{-4} \text{ S}\cdot\text{cm}^{-1}$  at 180 °C. The 95 vol.%  $\text{CsH}_5(\text{PO}_4)_2$  sample combined high conductivity with thermal stability up to 240 °C, achieving  $\sim 10^{-3} \text{ S}\cdot\text{cm}^{-1}$  at 225 °C, without melting.

The results validate cold sintering as a low-energy densification route capable of producing structurally stable, proton-conducting composites compatible with polymer phases.

Future work will optimize polymer ratios, mixing, as well as sintering parameters to enhance conductivity and mechanical strength. These findings establish a foundation for sustainable electrolyte design and foster potential collaborations and publications, strengthening Europe's progress in hydrogen and fuel cell technologies.

#### Data Management

All raw data (XRD diffractograms, EIS Nyquist plots, SEM micrographs) were stored in cloud repositories (OneDrive) during the project, and will be kept. Copies were also kept by the RI. Processed and curated data, including phase analyses and conductivity calculations, will also be archived in OneDrive, and an attempt will be made to archive it in the University of Aveiro's DUnAs Dataverse repository, ensuring long-term accessibility and adherence to FAIR principles. The person responsible for data stewardship is João Pedro Ferreira Carvalho (jpfc@ua.pt)

#### Difficulties during the TA related work (up to 250 words)

[List problems and issues, you had, completing out your research project: Did you get access to all the necessary equipment, facilities, databases, etc.? If not, please specify the problems that occurred and list equipment that was not working or accessible.]

The main limitation encountered was the **temperature ceiling of 180 °C** in the Baltic Fuel Cells setup, which was surpassed by using the high temperature setup, for evaluation of the composite's superprotonic phase. No major equipment failures or safety issues were reported.

These constraints primarily affected conductivity optimization but did not compromise the overall project objectives.

#### Intended publications

[Explain where and how you expect to publish the outcomes of your project work. Include also anything already published (What and where?)]

It is intended to publish a paper about the use of Cold-Sintered  $\text{CsH}_5(\text{PO}_4)_2$ -PBI Composites for Intermediate-Temperature Proton Conducting Electrolytes in one journal from this subject.

#### Expected impact

[The impact the expected results will have on current and future research or practice, public safety, European standardization, competitiveness, integration and cohesion and on sustainable growth. any follow on proposals, projects, collaborations, commercialisation]

The results demonstrate a viable pathway for **low-temperature processing of proton-conducting composites**, supporting the EU's sustainability and clean energy goals. By reducing sintering energy consumption and integrating polymeric flexibility with ceramic conductivity, this approach may **lower manufacturing costs** and **expand material compatibility** for ITFCs and other hydrogen-based systems. The work strengthens European cooperation in hydrogen technologies and lays the groundwork for future proposals within Horizon Europe on **cold-sintered electrolyte optimization**.

### Conclusions / additional comments

[Provide any other comments you might have on your work]

*The access at HITMOBIL provided a valuable opportunity to validate the cold sintering approach for hybrid electrolytes. The experiments confirmed material stability, hybridization, and measurable proton conduction. The collaboration fostered knowledge exchange and technical training in advanced electrochemical characterization, representing an important milestone in the development of **sustainable, low-energy electrolyte materials**.*

Did you complete the European Commission User questionnaire  
<https://ec.europa.eu/eusurvey/runner/RIsurveyUSERS?>

Yes    No

### Feedback - HSE, Ethics and Satisfaction

Please rate on a scale from 1 (excellent) to 5 (poor). Feel free to provide additional comments

Practical information on how to apply for Transnational Access and the overall application process

1 (excellent)	2	3 (neutral)	4	5 (poor)
<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Comment

Information provided, once your project was accepted, on how to proceed

1 (excellent)	2	3 (neutral)	4	5 (poor)
<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

Comment

Support received at the site(s) regarding technical/scientific matters and logistics

Have you got sufficient support from the RI staff during the project? If not, please, specify the problems.  Yes    No

Please specify any problems

RI extension / upgrades required

In your opinion, is the RI needed to be upgraded? If yes, please give an explanation.

	<input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please specify</i>	
Problems with local regulations	Have you had any problems with regulations of the visited RI owner (HSE, lab working hours, etc.)? If yes, please, specify <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please specify</i>	
Health and safety issues	Did you encounter any health or safety issue during your research? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	
<b>Environment &amp; Ethics</b>	Did your research involve the use of elements that may cause harm to the environment, to animals or plants? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	
Environment & Ethics	Did your research deal with endangered fauna and/or flora and/or protected areas? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	
Environment & Ethics	Did your research involve the use of elements that may cause harm to humans, including research staff? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	
Environment & Ethics - Dual use	Does your research have the potential for military applications? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	
Environment & Ethics - Misuse	Does your research have the potential for malevolent /criminal/terrorist abuse? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No
<i>Please provide details</i>	

Environmental issues	Were any potentially dangerous substances (materials / gases etc.) released into the environment (atmosphere, water, or land)? Please provide details. <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No				
<i>Please provide details</i>					
Ethics issues	Are there any other ethics issues that should be taken into consideration? Please specify <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No				
<i>Please provide details</i>					
Overall impression of communication and interaction after finishing your TA and related work	1 (excellent)	2	3 (neutral)	4	5 (poor)
	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
Comment					
Suggestions for facilities not included in RISEnergy which you would use for your research					
[Please provide suggestions for specific type of facilities missing (RI gaps) or measurement / experiments you would like to perform which can not be done on current RISEnergy facilities.]					
Suggestions how RISEnergy can improve future TA programme, how to make the TA more impactful and how to enable the achievement of high TRL levels					
[Your suggestions]					

## Feedback - Pro-active Innovation Support

Awareness	Did you know about the pro-active innovation support of RISEnergy? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No				
<i>[Please specify how you learned about the pro-active innovation support]</i>					
Personal experience	Have you taken advantage of or benefited from the pro-active innovation support? <input type="checkbox"/> Yes <input checked="" type="checkbox"/> No				
<i>[Please provide details]</i>					
Information/service provided by the pro-active innovation support?	1 (excellent)	2	3 (neutral)	4	5 (poor)

*[Please provide details]*

I declare that the above provided information and especially that information on the number of days visited the RI is correct.

*I have read the [RISEenergy privacy policy](#) for participation in the RISEenergy TA and consent to participation and the associated data processing.*

Your full name: !

Your signature: