Scientific report of the international workshop "Pushing the size limits of radiocarbon analysis"

The workshop was sponsored by the ESF-funded Research Networking Programme MOLTER (Molecular structures as drivers and tracers of terrestrial C fluxes), together with the Swiss National Science Foundation.

Summary
The workshop was renamed 'International workshop on small scale radiocarbon analysis', and took place from Tuesday September 13, 17h, until Friday September 16, 16h, on the campus of ETH Zürich, Switzerland. Around sixty people attended the workshop, including the organizers. The workshop program consisted of a first part meant for knowledge exchange, which transitioned into a second part set up for discussions. The workshop was clearly a success, and the goals defined at the beginning were reached:
1. Serve as a forum to share existing knowledge and perspectives from various laboratories and scientific fields involved in small-scale radiocarbon analysis
2. Discuss the state of the art, including ongoing and future developments
3. Set first steps towards the organization of an international working group / steering committee, which takes the lead in defining a set of specific standards for small scale ^{14}C analysis, and an inter-laboratory ring trial using these standards.

Description of the scientific content and discussion at the event (up to 4 pages)
The topic of the workshop was small-scale (<50 microgram carbon) and compound-specific radiocarbon analysis. Radiocarbon (^{14}C) is one of the most important dating tools in archaeology and for the dating of sedimentary records up to 50.000 years old, but is also increasingly being used in carbon cycle studies, environmental sciences and pollution studies. New developments in accelerator mass spectrometry and sample introduction methods have allowed the downscaling of sample sizes required for analysis as small as a few microgram of pure carbon. With newly developed sample preparation techniques it is now also possible to separate individual organic compounds for radiocarbon measurement. These developments have created new opportunities for the use of ^{14}C in scientific fields where its application had previously been limited by sample size. However, this also presents new challenges with respect to sample preparation and assessment of uncertainties caused by contamination and instrumental background.
The primary goal of the workshop was to serve as a forum to share existing knowledge and perspectives from various laboratories and scientific fields, in order to benefit the larger community that uses or plans to use small-scale / compound-specific radiocarbon analysis ('small ^{14}C'). A second goal was to discuss state of the art and future analytical developments and applications of this powerful technique. A third goal was to set up an international effort to define new standards representing different matrices suitable for small-scale / single compound applications, and that complement the limited suite of existing radiocarbon standards. Such a wider range of reference standards is crucial for establishing quality control across the various preparation procedures. This reference standard set would then also form the basis of an international laboratory comparison study. Finally, the formation of an international steering committee on small-scale radiocarbon analysis was foreseen.

The workshop opened with two introductory lectures that gave an overview over the field. The first half of the workshop was focused around knowledge sharing. This was done by lectures given by a part of the participants, with ample time for discussion. Others presented their work during a poster session that lasted a whole afternoon, also giving ample time for discussion and interaction between the participants. All participants had handed in an abstract of their presentation prior to the workshop, which was made available a week before the workshop. The program and abstract book are provided as annexes to this report.
The second half of the workshop was dedicated to discussion of problem connected to small ^{14}C analysis. This started with a brainstorming session that aimed to narrow down the broadly defined theme of the workshop toward more specific topics that would then be discussed in the following sessions. These topics were:

- Requirements of accuracy and precision, depending on the specific application of small ^{14}C.
- The most limiting factors in small ^{14}C
- Assessment of background and contamination
- Evaluation of uncertainties in the final data, and how to reduce these.
- Reporting of data
- Effect of matrices
- Differences between types of instrumentation (primarily graphite versus gas measurements) and types of handling (towards 'continuous flow')
- Protocol sharing
- Inter-comparison and inter-calibration of small ^{14}C - preparation and measurement. Besides using primary and secondary standards that are already available to the community, generation of standards specifically geared towards small ^{14}C.
- Diversification of sample introduction into AMS, including hyphenation to 'sample prep' systems (chromatography).
- Generation of a list of recommendations for best practices in sample preparation, handling and measurement; sharing of protocols for sample preparation.
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Once these themes were selected, they were discussed during the following discussion sessions, which had following outcomes:

- No clear preference graphitization or direct gas-ionization-AMS was indicated by users.
- Instrumentation applied to measure small ^{14}C is not the limiting factor in most cases, but it is the pre-processing and handling part, where contamination is a prominent issues. Therefore, the following recommendations can be made (note: this list is not fully exhaustive):

- Assess your extraneous C introduced during sample processing by monitoring contamination of a modern and a ^{14}C-free standard if at all possible.
- Assess the purity of a sample also by other independent means, for example in case of individual compounds, via stable carbon isotope (^{13}C) measurement or NMR.
- Perform purity checks and analyze multiple standards and blanks to constrain and assess the uncertainty related to small-scale ^{14}C measurements. Continue to do so because conditions may change through time.
- Know where you samples come from, especially when collaborators may be less aware of ^{14}C contamination issues.

- Be aware of contaminations from the surrounding of the laboratories, for instance from pharmaceutical industries where 14C-labeling experiments are performed.
- Ideally, build your own instruments and equipment optimized for 'cleanest' results with lowest background noise. In particular, one has control over all possible contamination sources.
- The smaller the samples, the more critical this issue becomes

- A clear reporting of uncertainty is needed, including what reported errors / uncertainties refer to (e.g. only the AMS measurement error, or the uncertainty of the whole procedure). Reporting should be done using the commonly accepted notation 'Fraction ^{14}C', as defined by Reimer et al. (Radiocarbon vol.46, p.1299, 2004). This value is normalized to 1950 AD and is corrected for $\delta^{13}C$ of the measured sample.
- with correction for isotope fractionation using measured ^{13}C contents, and without correction for the year of sampling.
- Hyphenated chromatography - AMS systems are desirable. However, because of its complex nature, this will not likely become a main development goal for AMS laboratories in the near future.
- Importance of collaboration: The ‘user’ needs to work close together with the AMS lab get to the best sampling and measurement strategy in order to optimize the quality of the data. One of the key issues in this collaboration is the opportunity to address the issue of amount of tests and blanks, and related measurement costs.
- It turned out to be too complex to already come up with a specific wish list for small-scale ^{14}C standards. It was decided that this would be further developed by a committee, who would start with a questionnaire to the worldwide community to gather an overview of needs and ideas.
- To get to a comprehensive list of recommendations (as just above), it makes sense to use the power of the Internet for sharing knowledge, instead of having a small team doing a lot of work. A 'wiki' is seen as the best option to do this. Such a platform is especially geared towards collecting and condensing knowledge and information from a wide variety of sources, and making this available to the wider public. It was decided that this 'wiki' would be set up in the coming half year.

Assessment of the result and impact of the event on future directions of the field

The workshop was clearly a success. The 'instrumentalists' and the 'users' could give each other many insights and feedbacks on the needs, wishes, possibilities, and applications of small ^{14}C (goals one and two listed above).

More specific results, beyond exchange of knowledge, were the following:

1. A 'wiki' / internet-based community platform is being set up (persons in charge: S. Shah and L. Ziolkowski) that serves as a virtual meeting place for everyone who does small ^{14}C. Here, a set of recommendations and guidelines for small ^{14}C will be published, which will be continuously updated following progress in the field.
2. Generation of a set of standards for small ^{14}C, in addition to 'large ^{14}C' standards currently available. This will be spearheaded by the following team: R.H. Smittenberg, Ö. Gustafsson, L. Wacker, T. Eglinton, A. McNichol, C. McIntyre. To make sure that the set will serve the broadest community possible, it will be based on the answers to a questionnaire sent to the wider community, i.e. also those that did not attend the workshop.

3. An inter-laboratory comparison study of these standards, to be processed by as many different laboratories as possible (the 'users'). The different AMS laboratories ('instrumentalists') who were represented at the workshop will be involved in this as well, and offered measurements free of charge.
4. Goal 3 above was reached with the establishment of a committee for small ^{14}C standards (see above) that is to be used in an inter-laboratory comparison study. This is then, together with the people involved in the 'wiki', a de facto steering committee on small ^{14}C, and consists of both young and senior researchers.
5. A number of informal plans for exchange research visits of students and future collaborations has sprung up, for instance: a) Potential exchange visit of M. Gierga (ETH Zurich) to the laboratories in Japan (N. Ohkouchi); b) Improving the small ^{14}C capacity (funding required) within Sweden, based on knowledge of, amongst others, the ETH laboratory; c) Future workshops on small ^{14}C will be organized either as sessions within, or connected to, upcoming international meetings, for instance the 'Radiocarbon' meeting next year in Paris. In other words, the one presently organized may have been the first one in a series.
6. The workshop will result in some publications. First, a workshop report has been submitted for publication to the newsletter 'EOS' of the American Geophysical Union (AGU). This will reach the approximately 61000 members of AGU and increase awareness of the present-day utility and future potential of small-scale and compound-specific radiocarbon analysis among the broader scientific community. On the longer term, results and discussion of an inter-laboratory comparison will be published in a peer-reviewed journal or book chapter. This manuscript will also contain the recommendations distilled from the 'wiki'.
7. If there is one thing that has become clear from the workshop, it is that the main limiting factors of the continuing development are manpower and funding, as there are enough ideas and applications. Hence, the workshop did not so much result in new directions, but functioned mainly as a first step to assemble and consolidate all the knowledge available in this emerging field.
8. The goal of the workshop was not only to bring together knowledge, but also to pass this on to scientists who are considering or just starting to perform small ^{14}C analysis. Around one third of the attendants fell into this category, and the workshop functioned as a great source of first-hand information for them. The upcoming 'wiki' and foreseen future workshops will serve this purpose in the future.

Annexes:
1. Programme of the meeting
2. Full list of speakers and participants

Eidgenössische Technische Hochschule Zürich
Swiss Federal Institute of Technology Zurich

UNIVERSITÄT BERN

International Workshop on Small Scale Radiocarbon Analysis

ETH Zurich, Switzerland

13 – 16 September 2011

Program and Abstracts

Supported by

Swiss National Science Foundation

MolTER

European Science Foundation

Program

Tuesday 13 September - Department of Earth Sciences, ETH

17:00	*20:00*	*Registration; welcome drinks & buffet*
		Session 1 - Introductory lectures
20:00	21:00	Tim Eglinton and Hans-Arno Synal

Wednesday 14 September - ETH Hönggerberg

		Session 2 - Technical overview
8:30	9:15	Peter Steier - AMS systems, ion sources, sample input - technical limitations and future possibilities
9:15	10:00	Guaciara Dos Santos - The good and the bad of small-scale ^{14}C-AMS sample preparation and analysis
10:00	*10:30*	*Coffee*
		Session 3 - Solid and gas measurements
10:30	10:55	Cameron McIntyre - CSRA by Gas Chromatography and Continuous-Flow AMS
10:55	11:20	Lukas Wacker - Routine radiocarbon analyses of small samples with a gas ion source
11:20	11:45	Yusuke Yokoyama - Small size samples radiocarbon dating at the University of Tokyo
11:45	12:10	Ann McNichol - Improved Preparation of Volatile and Semi-Volatile Compounds for CSRA //OR// Suni Shah: Compound-specific sample preparation by elemental analyzer and small-volume graphitization reactors
12:10	*13:30*	*Lunch*
		Session 4 - Sample preparation techniques
13:30	13:55	Brad Rosenheim - A (Dirt) Burning Desire: Running smaller samples from ramped pyrolysis radiocarbon preparation
13:55	14:20	Lori Ziolkowski - Quantifying extraneous carbon added during GC preparation of CSRA samples
14:20	14:45	Kristina Stenström - Small scale radiocarbon analysis – the threat of anthropogenic ^{14}C contamination
14:45	15:05	Örjan Gustafsson - CSRA and other small-sample ^{14}C studies of air, seawater and sediments at Stockholm University: Focus on technical considerations and observations
15:05	*15:30*	*Coffee*
		Discussion 1
15:30	17:30	**Brainstorming on needs and wishes.** Focused on needs, wishes and possibilities of systems, including sample introduction and ionization, to bring together ‘users' and ‘developers’.
18:00	*22:00*	*Workshop Dinner in Restaurant 'Die Waid' - nearby campus*

Thursday 15 September - ETH Hönggerberg

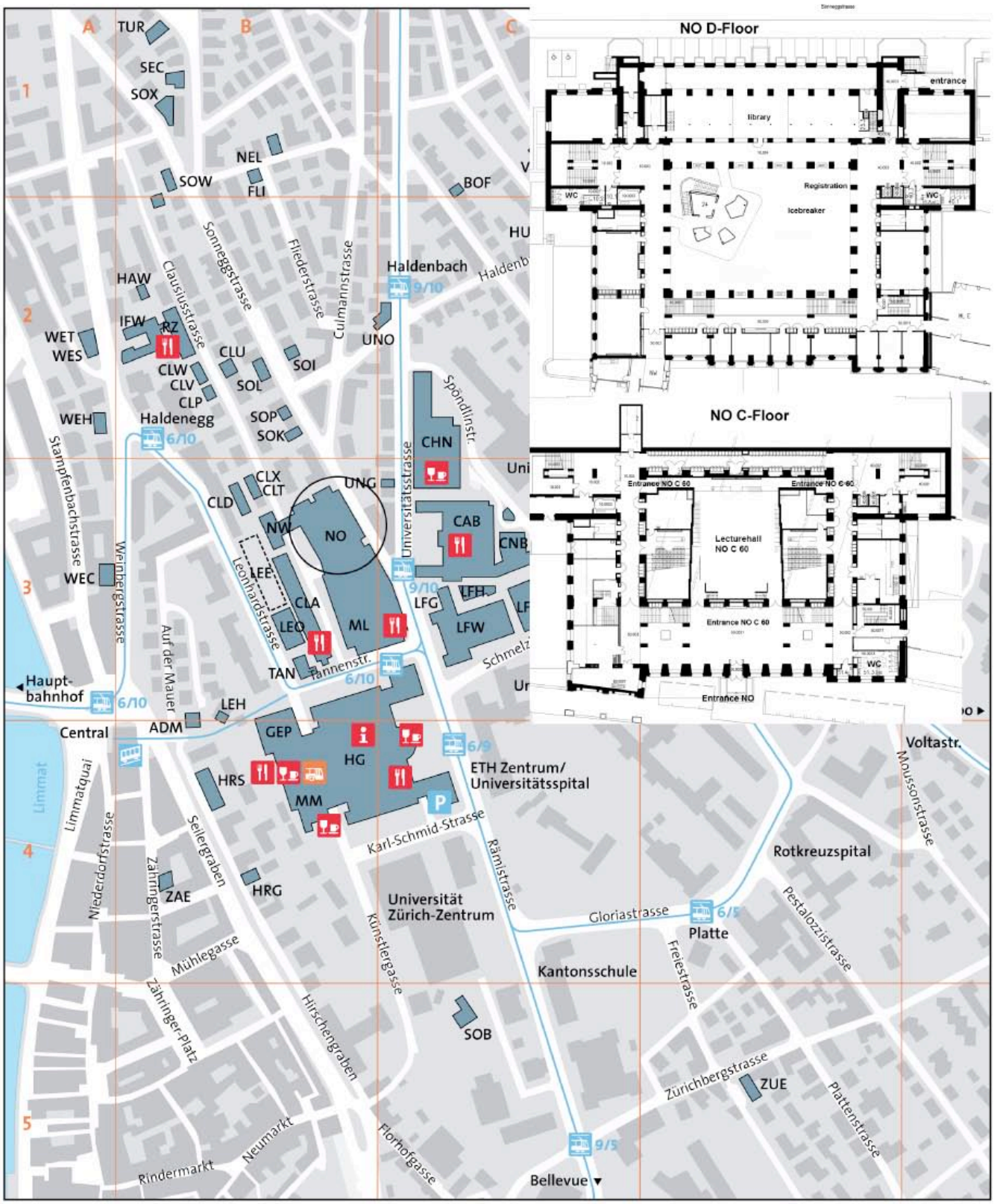
		Session 5 - CSRA and cell studies
8:30	9:15	Nao Ohkouchi - Preparation, Purification, Sample Handling and Laboratory Practices for Compound-Specific Radiocarbon Dating
9:15	10:00	Kirsty Spalding - Radiocarbon Analyses of Cell Turnover in the Adult Human Brain and Body
10:00	*10:30*	*Coffee*
		Session 6 - Carbon cycle and dating
10:30	10:55	Stefanie Kusch - Culturing chlorophyll a under manipulated seawater DIC 14C concentrations for precise blank C determinations
10:55	11:20	Erin Ellis - The age of plant-derived organic carbon transported by the Mekong River, Cambodia: A comparison between lignin-derived phenols and bulk organic matter
11:20	11:55	Irka Hajdas - From 14C content to calibrated ages: methods and considerations
11:55	13:30	*Lunch*
13:30	17:00	**Poster session and visit Radiocarbon laboratory ETH**

Friday 16 September - ETH Hönggerberg

		Discussion 2
8:30	10:00	**Best practices in sample preparation / purification**
10:00	*10:30*	*Coffee*
		Discussion 3
10:30	12:00	**Best practices in sample handling, introduction into AMS, measurement strategies**
12:00	*13:00*	*Lunch*
		Discussion 4 - Ring trials
13:00	14:30	Wish list regarding ^{14}C standards and ring trial of small-scale / compound-specific radiocarbon analysis
14:30	*15:00*	*Coffee*
		Conclusion - Workshop proceedings / Steering committee
15:00	16:00	Set up an outline for workshop proceedings, selection of a steering committee on small-scale radiocarbon analysis.

Location of EVENTS:
Registration, Icebreaker, introductory lecture: NO Building, Sonneggstr. 5, 8092 Zürich

ETH Zürich – Standort Zentrum



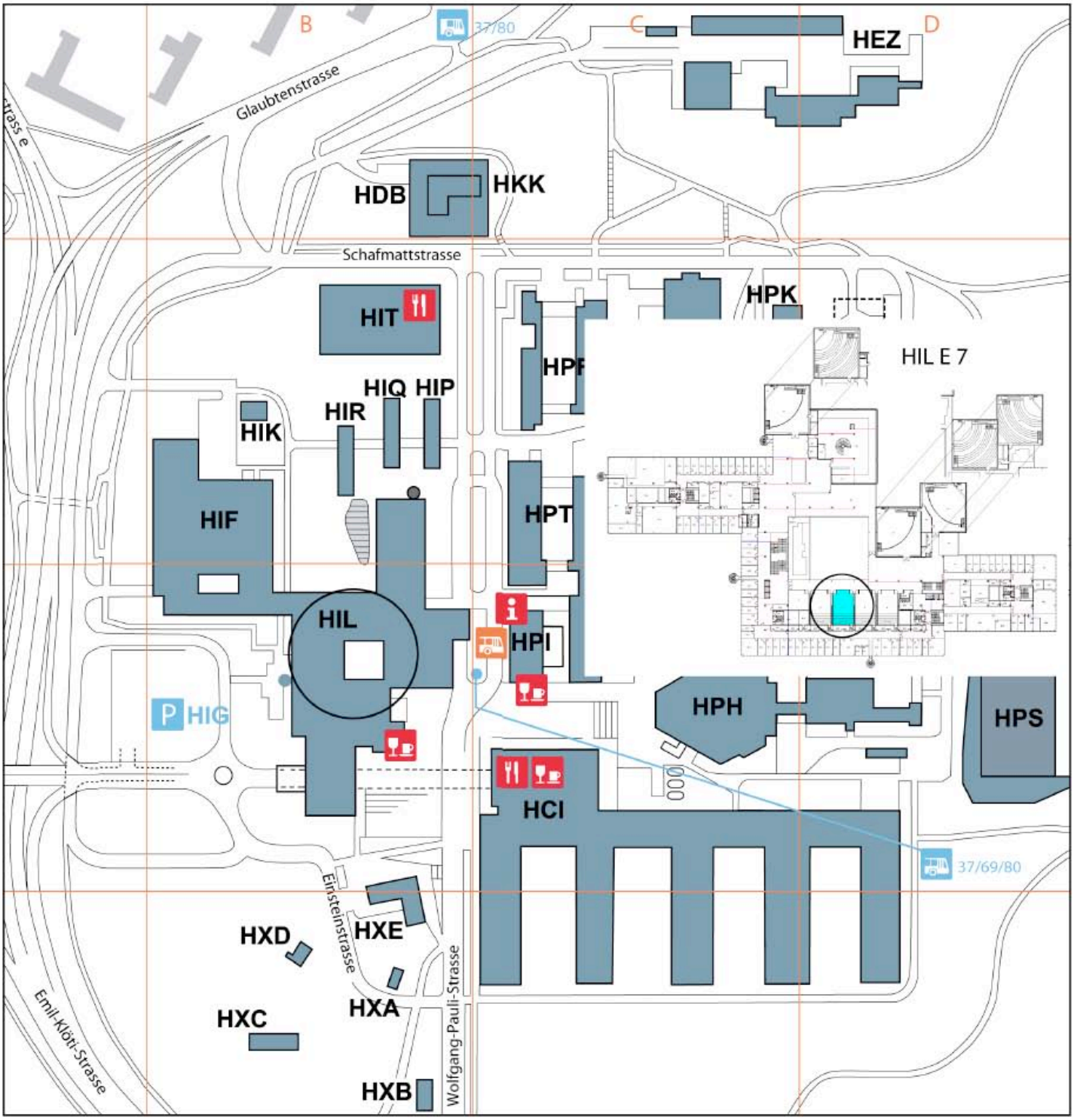
Getting there:

From Zurich main station:
Tram no. 6 (direction Zoo) to tram stop "ETH/Universitätsspital". Journey time: about 6 minutes
From tram stop "Bahnhofplatz/HB": Tram no. 10 (direction Flughafen or Bahnhof Oerlikon) to tram stop "ETH/Universitätsspital" or tram no. 3 (direction Klusplatz) to tram stop "Central" (first stop), from "Central" by Polybahn (departing every 3 minutes) to the Polyterrasse. Journey time: about 8 minutes

From Zurich airport, tram station Zürich Flughafen Bahnhof
Tram no. 10 (direction Bahnhofplatz/HB) to tram stop "ETH/Universitätsspital". The tram operates daily from 6 a.m to 11 p.m. with trams departing every 7 to 15 minutes. Journey time: 30 minutes
If you wish to travel from the airport to the city centre (main station), you should travel by S-Bahn or train. The trains leave from the station "Zürich Flughafen". Journey time: about 10 minutes
You need a valid ticket for 3 zones.
Depending on your needs, you can buy a day ticket valid for 24 hours for all trams and buses.

Workshop Sessions:
ETH HHönggerberg, HIL Building, Wolfgang-Pauli-Strasse 15, 8093 Zürich

ETH Zürich – Standort Hönggerberg (Campus Science City)



Bushaltestelle
ETH-Pendelbus «Science City Link»
Mensa
Cafeteria
Science City Welcome Desk (Telefon +041 44 633 64 44)

Alle Gebäude und Parkgaragen sind rollstuhlgängig. Weitere Informationen am Science City Welcome Desk .

Getting there:

From Zurich main station
The shuttle bus ETH Science City Link offers the fastest non-stop transfer.
From Monday to Friday: Departure at 7.34 and 7.54 a.m. from Zurich main station (bus stop "Bahnhofplatz/HB", in front of Hotel Schweizerhof), transfer to Science City with a stop at tram stop "Haldenegg". Journey time: about 15 minutes.

Regular public transport connections from main station to Hönggerberg (campus Science City)
S-Bahn (lines 2, 5, 6, 7, 8, 14, 16) to Bahnhof Oerlikon; bus no. 80 (direction Triemlispital) from Bahnhof Oerlikon Nord to bus stop "ETH Hönggerberg". Journey time: about 25 minutes
Tram no. 11 (direction Auzelg) to "Bucheggplatz", from there by bus no. 69 (direction ETH Hönggerberg) to the terminal stop. Journey time: about 25 minutes
Tram no. 14 (direction Seebach) to "Milchbuck", from there by bus no. 69 (direction ETH Hönggerberg) to the terminal stop. Journey time: about 25 minutes
You need a valid ticket for zone no. 10 ("Stadt Zürich").

From ETH Zentrum
The shuttle bus ETH Science City Link offers the fastest non-stop transfer.
From Monday to Friday: From 8.14 a.m. every 20 minutes at .14, .34 and .54 to Science City, stopping at tram stop "Haldenegg". The last service is at 6.14 p.m. Buses leave from the subway under the 'Polyterrasse' (floor C). Journey time: about 15 minutes.

Regular public transport connections from ETH Zentrum to Hönggerberg (campus Science City)
Tram no. 9 (direction Hirzenbach) or Tram no. 10 (direction Zürich Flughafen) to "Milchbuck", from there by bus no. 69 (direction ETH Hönggerberg) to the terminal stop. Journey time: about 25 minutes
You need a valid ticket for zone no. 10 ("Stadt Zürich").

From railway station Altstetten
Bus No. 80 (direction Bahnhof Oerlikon Nord) to the bus stop "ETH Hönggerberg". Journey time: about 10 minutes
You need a valid ticket for zone no. 10 ("Stadt Zürich").

From railway station Oerlikon
From Bahnhof Oerlikon Nord by bus no. 80 (direction Triemlispital) to the bus stop "ETH Hönggerberg". Journey time: about 10 minutes
You need a valid ticket for zone no. 10 ("Stadt Zürich").

From railway station Zurich-Affoltern
Bus no. 37 (direction ETH Hönggerberg) to the terminal stop. Journey time: about 10 minutes
You need a valid ticket for zone no. 10 ("Stadt Zürich").

From Zurich airport
S-Bahn no. 2 (direction Ziegelbrücke) or no. 16 (direction Herrliberg/Feldmeilen) to Bahnhof Oerlikon, from Bahnhof Oerlikon Nord by bus no. 80 (direction Triemlispital) to the bus stop "ETH Hönggerberg". Journey time: about 20 minutes
You need a valid ticket for 3 zones. Depending on your needs, you can buy a day ticket valid for 24 hours for all trams and buses.

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