ROBL-CRG	**Experiment title:** **Experiments to test the SPLICE program using real EXAFS spectra**	**Experiment number**: 20_01_20
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Names and affiliations of applicants (* indicates experimentalists):

Harald Funke[1]*, Horst Böttger[2], Tobias Reich[1]*, Christoph Hennig[1]*, Andrè Rossberg[1]*
Forschungszentrum Rossendorf e.V., 01314 Dresden, Postfach 510119, Germany
[1]Institute of Radiochemistry
[2]Department Communication and Data Processing

Report:

During the first commissioning experiments with the cryostat in autumn 1999, sometimes the technical problem arose that it was impossible to get one continuous EXAFS scan over a wide k range. In spite of the fact that this situation was overcome, the idea of connecting two different parts of an EXAFS scan in a unique way was developed. So, the computer program SPLICE was written to merge two different EXAFS spectra of the same sample in an overlapping energy region. The word “splice” was chosen because of the analogy to the old sailing trade “splicing”. The SPLICE program may be a useful tool to overcome difficulties due to insufficient time during an EXAFS experiment. Different situations may cause such time problems, e.g.:

- Any interruption or perturbation of the synchrotron beam,
- The refill times are too frequent for the experiment,
- Only the noisy part (in general the rear part of the spectrum) needs some repetitions for an amendment of the statistics of the EXAFS scan.

Two raw EXAFS scans of the same sample, F_1 and F_2, are given. Both sets are recorded at different energy regions with an overlapping region of about 50 data points. The intention of the program is to find an optimal translation for F_2 (concerning x and y) and a re-calibration factor for "splicing" both scans together to handle them like **one continuous scan**. Experiments were performed to test the SPLICE program using real EXAFS spectra up to a k of 22 $Å^{-1}$ at the U L_{III} edge of a uranyl arsenate sample ($H_2[UO_2AsO_4]_2 \cdot 8H_2O$).

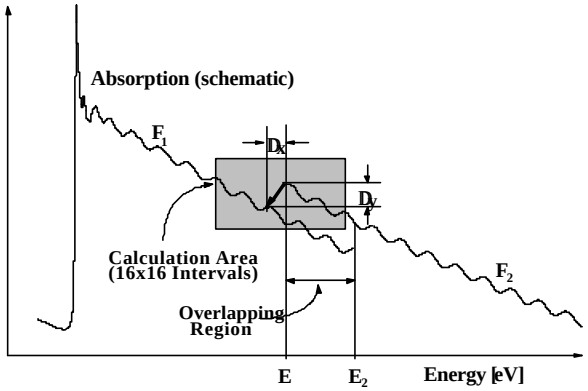

Figure 1. Schematic illustration of the task of the program SPLICE

The final range of the parameters found is usually:
$|\Delta x| < 1$ eV,
$|\Delta y| < 0.005$,
$|\lambda - 1| < 0.001$

To reduce thermal vibrations, the sample was cooled to 15 K using a closed-cycle He cryostat.
Three different EXAFS spectra, their Fourier transforms and fit-results were compared. The first two spectra consist of two different scans connected by the program SPLICE, and the third spectrum consists of one uninterrupted continuous scan.

- Splice A: Two absorption spectra, both scanned with an equal step in k-space of $\Delta k = 0.05$ Å^{-1} are spliced at k =15 Å^{-1} to give an EXAFS-spectrum with a length of k = 22 Å^{-1}.
- Splice B: Two absorption spectra, the first scanned with an equal step in k-space of $\Delta k = 0.05$ Å^{-1} and the second with an equal step of $\Delta k = 0.02$ Å^{-1} are spliced at k =15 Å^{-1} to give an EXAFS spectrum with a length of k = 22 Å^{-1} as demonstration of the ability of the SPLICE program to connect two scans recorded with different steps in k-space.
- Without splicing C: For comparison with the spliced spectra, one entire spectrum was recorded over the whole range of k = 22 Å^{-1} with an equal step of $\Delta k = 0.05$ Å^{-1}.

Table 1. As example: fitted co-ordination numbers and distances of the three test spectra (see text) for the five relevant co-ordination shells.

Coord. shell	A	B	C	A	B	C
	N			R [Å]		
Oax	1.94(0.11)	2.05(0.10)	1.82(0.10)	1.789(0.003)	1.791(0.002)	1.790(0.002)
Oeq	4.65(0.26)	4.31(0.22)	4.76(0.25)	2.296(0.003)	2.306(0.002)	2.302(0.003)
As	3.99(0.17)	4.15(0.15)	3.90(0.15)	3.702(0.001)	3.700(0.001)	3.702(0.001)
U1	5.88(0.67)	5.16(0.49)	5.32(0.57)	5.402(0.003)	5.400(0.002)	5.400(0.003)
U2	4.61(1.16)	5.88(1.17)	5.92(1.32)	7.191(0.006)	7.181(0.004)	7.180(0.005)

Significant differences are not visible between the graphs of the three spectra. For comparison, table 1 shows the numerical values of the fitted data including the standard deviations. The differences between the fit results have the same order as the usual experimental uncertainties of EXAFS measurements. Therefore, the possibility to connect two different absorption spectra of the same sample with an overlapping energy region using the program SPLICE is demonstrated by examples.