EUROPEAN **S**YNCHROTRON **R**ADIATION **F**ACILITY

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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

fill in a separate form for each project or series of measurements.

type your report, in English.

include the reference number of the proposal to which the report refers.

make sure that the text, tables and figures fit into the space available.

if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

	Experiment title: Anomalous scattering from polyelectrolyte gels during volume transitions	Experiment number: SC 808
Beamline: BM02	**Date of experiment**: from: 18/04/01 to: 23/04/01	**Date of report**: 01/09/01
Shifts: 15	**Local contact(s)**: Erik Geissler	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

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Report:

Neutralised polyelectrolyte networks swollen in aqueous solutions with monovalent cations undergo a volume transition when a small amount of bivalent cations are introduced into the diluent. The aim of the SC 808 experiments was to obtain information about the cation distribution in PAA gels around this transition, in swollen, intermediate and collapsed gels.

A variation of contrast was performed (anomalous SAXS) by changing the X ray wavelength just below the absorption edge of the cation in order to obtain this information. Three samples were prepared, PAASr50, PAASr100 and PAASr200 as well as electrolyte solutions having Sr2+ concentrations corresponding to the solvent included in gels. Similar gels and solutions were prepared with calcium, for which the wavelength changes do not create contrast variation. SAXS measurements in the range $9.10^{-4} < q(A^{-1}) < 0.44$ were performed on each sample, after having previously determined that the maximum X-ray exposure time before significant radiation damage occurs is 50 sec. By scanning in energy, the concentration in Sr2+ in each sample was determined from the absorption level.

The scattering curves obtained for the gels reveal two main components : a Debye-Bueche law at the smallest q values and an Ornstein-Zernike behaviour at larger q, or a plateau followed by a power law in q^{-2}. For example, figure 1 shows the SAXS response of PAASr200 at 15.8 keV.

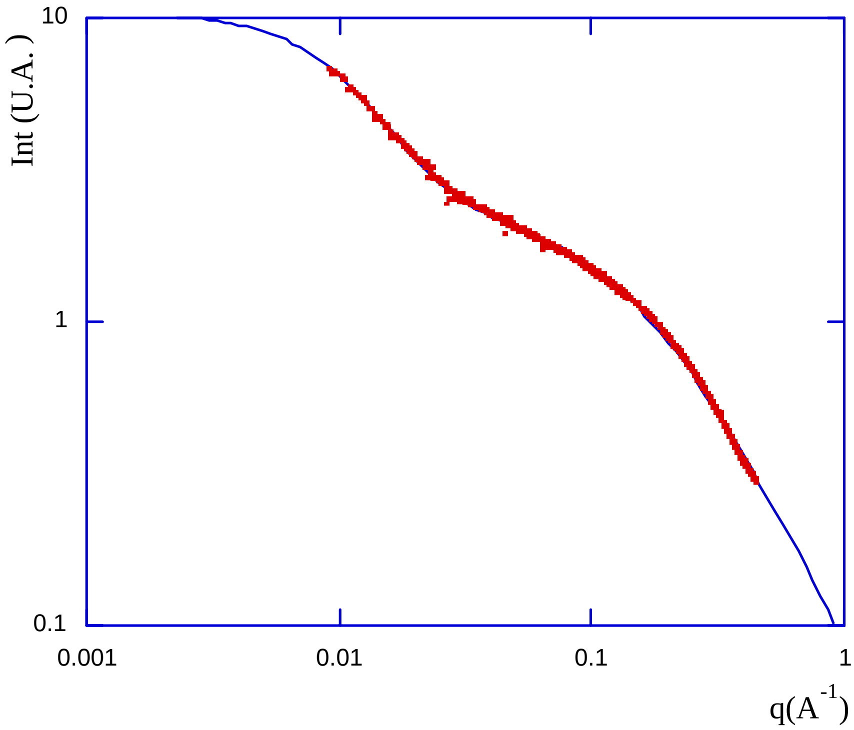

Fig. 1 : Scattering of the PAASr200 at E=15.8KeV. The line is the fit to the sum of a OZ and a DB equations.

These results are closely similar to those we obtained from SANS measurements, which reveal the structure of the polymeric gel only. The first (OZ) term represents the thermal concentration fluctuations and the second longer range static variations due to the permanent elastic constraints inside the gel. Table 1 shows the values of the thermal correlation length ξ for the 3 PAASr gels obtained from SAXS as well as those obtained previously by SANS (Ref. 1). ξ displays a maximum at the transition as expected (Ref. 2).

	ξ SANS (Angt)	ξ SAXS (Angt)
PAASr50	12	14.5
PAASr100	19.7	23.4
PAASr200	6.2	6.7

Qualitative observation of the curves obtained for a same sample at different wavelengths seems to indicate that counterions are positioned close to the chains. Effectively, changing the contrast creates a small shift along the vertical axis rather than changing the shape of the curve. The quantitative interpretation of the data is however still in progress. It is in particular necessary to determine the contrast factors for each experimental conditions in order to extract precisely the counterion contribution to the total scattering.

Ref. 1 : F. Horkay, P. Basser, A.-M. Hecht, E. Geissler, *Macromolecules* **2000**, *33*, 8329.
Ref. 2 : P.-G. de Gennes, Scaling concepts in polymer physics; Cornell : Ithaca, NY 1979.