



## DUBBLE – EXPERIMENT REPORT



We kindly request you to answer the questions (max 2 pages) and return the form to NWO **within 2 months of the completion of the experiment** to [dubble@nwo.nl](mailto:dubble@nwo.nl)

<b>Beam time number:</b> 26-02-885		<b>File number:</b> 195.068.1146
<b>Beamline:</b> BM26B	<b>Date(s) of experiment:</b> 7.11.2018-9.11.2018	<b>Date of report:</b> 12.12.2018
<b>Shifts:</b> 6	<b>Local contact(s):</b> Daniel Hermida Merino Michela Brunelli	

### 1. Who took part in the experiments? (Please indicate names and affiliations)

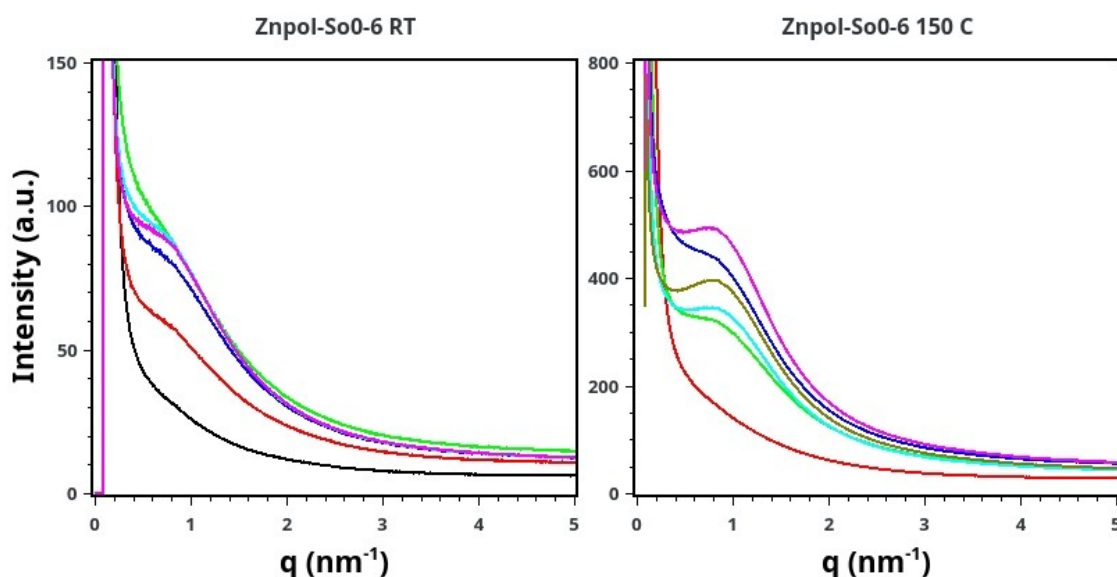
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### 2. Were you able to execute the planned experiments?

Yes. In these experiments we aimed to investigate the SAXS signal in linseed oil based ionomers which are models for aged binding medium. It was shown previously that these systems contain a so-called "ionomer peak" in SAXS which is characteristic for the scattering of ionic clusters. In this case the scattering particles are clusters of zinc carboxylates. The set of samples was expanded to include a larger set with different zinc neutralization levels and different absolute zinc concentrations at 100% neutralization. Because we obtained detailed information about chemical changes in the zinc carboxylate coordination from IR spectroscopy upon heating, it was investigated how the behaviour of these polymers is at high temperature. Figure 1 shows that there is a strong intensity drop and a change in the shape of the peak. Fitting the Yarusso-Cooper model indicated that the change in shape can be interpreted as a slight increase of the scattering clusters and an increase in the concentration per scattering unit. It is at present unclear if we can clearly attribute that to a structural change rather than a temperature effect. This will be investigated in the near future.



**Figure 1:** Zinc ionomers with increasing neutralisation at room temperature (RT) and 150 C. A strong intensity drop is visible at high temperature.

**3. Did you encounter experimental problems?**

Minor issues with a cable connecting the Linkam stage that broke down. Some small software issues where BUBBLE would not automatically reduce the data after measuring. This was easily fixed later.

**4. Was the local support adequate?**

The beamline staff were very helpful in with switching between the grid and the Linkam (for heating). Especially the email support and explanations about the use of the software afterwards was very good. For example a new version of BUBBLE was released to fix a bug that did not allow us to process average EDF images.

**5. Are the obtained results at this stage in line with the expected results as mentioned on the project proposal?**

Yes, but the proposal also mentions humidity effects of the SAXS signal. We chose to measure temperature effects first because these are expected to cause a similar change. Humidity effects remain to be studied.

**6. Are you planning follow-up experiments at DUBBLE for this project?**

No, because of the 1 year shutdown.

**7. Are you planning experiments at other synchrotrons in the near future?**

Yes, a proposal will be submitted.

**8. Do you expect any scientific output from this experimental session (publication, patent, ..)**

Yes, I expect a publication somewhere early 2019

**9. Additional remarks**

None further.



## DUBBLE - CLAIM FORM FOR COSTS OF TRAVEL/SUBSISTENCE

Dutch users of beam time at DUBBLE can use this form to claim full/partial reimbursement of the associated costs of travel and subsistence. The form must be returned to NWO **within 2 months of the completion of the experiment** to [dubble@nwo.nl](mailto:dubble@nwo.nl)

### **Reimbursement rules (costs are reimbursed to the Main Proposer)**

#### **Travel costs**

€ 400 p.p. for max. 3 persons.

#### **Subsistence costs**

Subsistence costs are reimbursed for max. 3 persons @ € 60 p.p. per day (incl. 1 day before the experiment).

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**Applicant (Main Proposer)** : Lambert Baij\_\_\_\_\_

Beam time number : **26-02-885**\_\_\_\_\_

Experiment dates : 7.11.2018-9.11.2018\_\_\_\_\_

#### **Participants (max 3 persons):**

Name : Lambert Baij\_\_\_\_\_

Name : Joen J. Hermans\_\_\_\_\_

Name : Sanne Berbers\_\_\_\_\_

#### **Payment details**

Bank name: Triodos Bank Nederland N.V.  
Bank address: 3704 EC Zeist, The Netherlands  
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IBAN: NL74TRIO0786886374  
Bank swift: TRIONL2U