



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: Structural characterisation of zeolite ITQ-28 from high resolution powder diffraction data.	Experiment number:
Beamline: ID31	Date of experiment: from: 13-Sept-2004 to: 14-Sept-2004	Date of report: 27-July-2005
Shifts: 3	Local contact(s): Dr. Irene Margiolaki	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Fernando Rey Instituto de Tecnología Química. (CSIC-UPV). Avda. de los Naranjos s/n. 46022-Valencia, Spain Jordi Rius and Xavier Torrelles Institut de Ciència de Materials de Barcelona (CSIC). 08193-Bellaterra, Catalunya, Spain.		

Report:

The powder X-Ray diffraction measurement of zeolite ITQ-28 in the calcined and in the as-prepared forms were carried out as planned. However, due to the extremely large volume and low symmetry of the unit cell a strong peak overlapping was observed even at very low 2θ angles. Attempts to overcome this problem were performed by carrying out the measurement of the X-Ray diffraction patterns at different temperatures with the aim of solve some X-Ray diffractions if there is any anisotropy in the unit cell expansion upon heating. Additionally, our local contact, Dr.a Irene Margiolaki, has collected X-Ray diffraction data at higher wavelength trying to improve the resolution of the X-ray peaks. At the moment even after all this work, all the attempts of indexing the X-Ray pattern of ITQ-28 zeolite have failed. At the moment, we are working in the indexation of the different patterns collected in this beam allocation and also, we are attempting to synthesise larger and better crystals of ITQ-28 that could allow its measurement by single-crystal microdiffraction technique.

Additionally to the scheduled work on ITQ-28, we carried out some experiments (which were completed during experiment CH-1859) on a different zeolite, named ITQ-32.

Two patterns were collected, one for the as-prepared solid and the second for the calcined material. With these two patterns and by applying a new methodology for solving complex crystal structures by direct methods, the structure was successfully solved. Two papers have been submitted. A paper entitled 'Solving centrosymetrical zeolites from powder diffraction data by combining the direct methods origin-free modulus sum function with the

isomorphous replacement technique.X.” in which our local contact is one of the authors has been sent to Journal of Applied Crystallography.

A second paper entitled ‘Synthesis and Structure of the Bidimensional Zeolite ITQ-32 with Small and Large Pores’ has been accepted for publication in Journal American Chemical Society.

The abstract of the accepted paper is as follows:

‘A new bidimensional zeolite containing 8R and 12R pores, denoted as ITQ-32, has been synthesized and its structure solved from powder X-Ray diffraction data. This zeolite presents a relatively large pore volume ($0.16 \text{ cm}^3/\text{g}$) and pore apertures of $3.5 \times 4.3 \text{ \AA}$ and can be prepared as nearly pure silica zeolite and as aluminosilicate. In the latest case, acidic properties are developed.’

A copy of this publication will be submitted to the ESRF when published in the Journal.

