Application Note

easyLab

Innovative Solutions for Materials Characterisation

easyLab Technologies Limited
Royal Holloway Enterprise Centre
Egham, Surrey, TW20 0EX, UK
Tel: +44 (0) 1784 497 337
Fax: +44 (0) 1784 497 338
Email: info@easy-Lab.co.uk
www.easy-Lab.co.uk

easyLab Pcell 15 / easyLab Pcell 30 – 15 & 30 kbar hydrostatic high-pressure cell modules for Quantum Design PPMS measurement platform. (ACT option)

Introduction

This application note introduces the new high-pressure cell modules from easyLab, Pcell 15 & Pcell 30[1], which have been specifically engineered to generate 15 & 30 kbar respectively, enabling end-users to perform AC electro-transport measurements under high-pressures and at low temperatures down to below 2 K.

In particular, it is explained how the pressure cell is assembled and interfaced with the PPMS. Pressure loading results are also presented. Finally, measurements performed on a single crystal of $CeRh_2Si_2$ are presented and compared with published data.

[1] In the rest of the text, otherwise clearly stated, the term 'easyLab Pcell 15/30' refers respectively to the 15 kbar and 30 kbar variants of the easyLab Pcell module.

easyLab Pcell 15/30 module contents

The high-pressure cell module Pcell 15/30 represents a complete package providing the end-users with all the necessary primary components of the pressure cell and the interfacing components to connect to the PPMS.

Very importantly, the easyLab Pcell 15/30 module is also supplied with all the necessary tooling and required consumables to enable up to twenty measurement campaigns.

Components:

- Pcell external body (1) and locknuts (2)
- Tungsten carbide pistons (2) and steel pad (1)
- Liquid transmitting medium (40ml)
- Vials for pressure-transmitting medium (2) and mixing vial (1)[2]
- Syringe & dyspenser (1)
- PTFE caps (20)
- Pre-wired feedthroughs fitted with 8 twisted pairs copper wires, sample platform, Sn and Manganin manometers fitted (3)
- Anti-extrusion rings (20)
- Anti-extrusion disks (20)
- Mounting platform (1)
- Extrusion pistons (2)
- Locking-nut extraction tool (1)
- Special PPMS compatible ACT puck (1)
- Puck mounting sock (1)
- Interfacing collar fully compatible with PPMS extraction tool (1)
- Sn manometer (10 cm)
- Manganin manometer (10 cm)
- Locking spanners (2) and adjustable spanner (1)
- Locking-nut steel opening wrench (1)
- User guide (1)
- Storage case (1)

[2] The mixing vial is dependant on the nature of the pressure-transmitting medium. easyLab Technologies offers three different types of medium.

Figure 1: easyLab Pcell 15/30 storing case content.

easyLab Pcell 15/30 features and benefits

The Pcell 15/30 modules are cylindrical hydrostatic pressure cells based on a single and double walled-design, respectively. The materials composing these cells have been carefully selected and tested to give the lowest magnetic background possible and yet maintaining high mechanical strength in order to withstand thermal and pressure cycles. The overall dimensions of the pressure cell have been optimised to give the maximum of sample space whilst still fitting in the sample space of the PPMS of Quantum Design. The concept of module has been developed to provide the novice as well as the advanced user with all the required tools to easily set-up, apply and release the pressure of the pressure cell.

Notably, the module comes as standard with three pre-wired and pre-tested electrical feedthroughs. This component, notoriously important for the good working of this type of cell, is coming fitted with all the required manometers and one sample platform with pre-soldered connections. This gives the benefit of greatly reducing the set-up time by just having to solder your sample contacts.

easyLab Pcell 15/30 technical specifications

The Pcell 15/30 modules can achieve, at room temperature, maximum pressures of 15 kbar and 30 kbar, respectively. With a careful choice of the pressure-transmitting medium similar pressures can be achieved also at low temperatures. The overall length of the pressure cell is 126 mm with an outer diameter of 24 mm. The available sample space is typically 2.9 mm diameter and 10 mm maximum long. The recommended size is of 1.5 x 1.5 x 5 mm.

The total weight of the pressure cell assembled and ready to be mounted on the PPMS is approximatively 300 g.

easyLab Pcell 15/30 structure and main components

The figure below represents a 3D solid modelling exploded view and description of the Pcell 15.

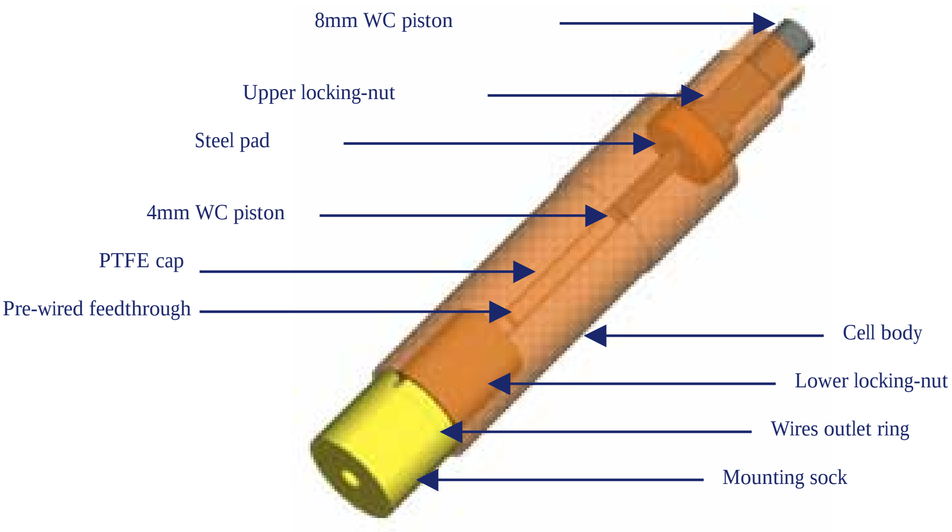


Figure 2: 3D modelling view of the Pcell 15

Pcell 15/30 setting – up

In order to set up the pressure cell and mount it on the PPMS, we have engineered a range of tools and accessories to facilitate these various steps. The setting up time is typically of around 60 minutes including sample mounting, pressure cell setting up, pressurisation and mounting on the PPMS. The pictures below represent these various steps.

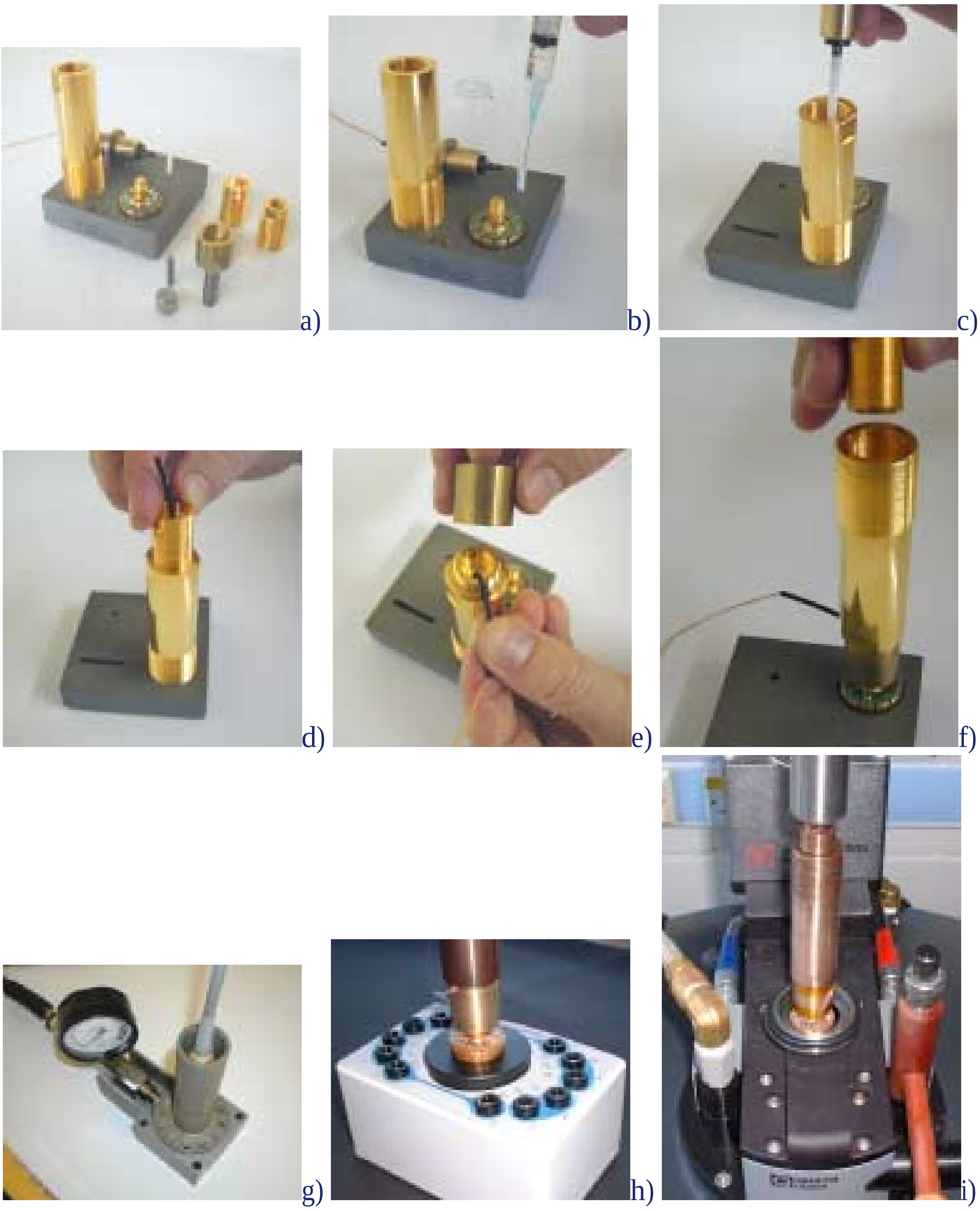


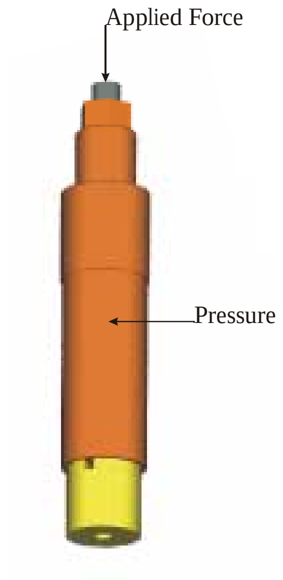
Figure 3: Key setting up stages of the Pcell 15/30 on the MPMS

easyLab Pcell 15/30 Pressurisation

In order to apply pressure, a hydraulic press ram is required. Although any hydraulic press ram capable of generating at least 5 ton of force can be used, easyLab Technologies strongly recommends the use of its miniature desktop ram: the easyLab Ppress. Although this is available as an option to the Pcell 15/30, its use greatly facilitates the loading and, very importantly, the unloading of the Pcell 15/30. Moreover the easyLab Ppress purposely engineered to the easyLab Pcell 15/30 will ensure the safety of the user and of the pressure cell. Indeed the easyLab Ppress includes key components ensuring the alignment of the cell during the pressurisation whilst maintaining a high degree of lateral safety protection for the end user.

The way the cell is pressurised is by the application a known force on a series of tungsten carbide pistons. In turn, the PTFE cap, where the sample is sitting surrounded by the transmitting medium, has its volume reduced and hence the pressure increases.

The figure below shows a typical pressurisation curve at room temperature.



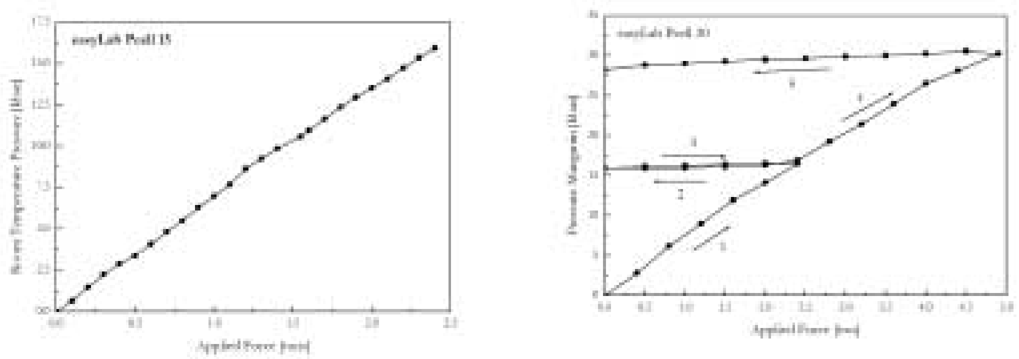


Figure 4: Typical loading curves of the Pcell 15/30 at room temperature

Low temperature pressure measurements

In order to know the pressure at low temperatures, a tin manometer sample is electrically connected on the underside of the sample platform. The value of the superconducting transition temperature, T_c [P], enables the estimate of the in-situ pressure as shown in the figures below.

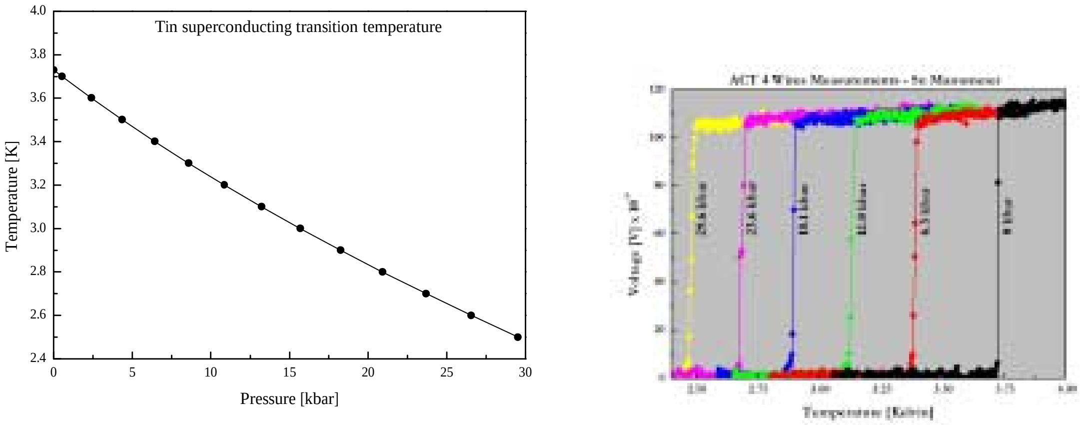


Figure 4: Pressure calibration based on the shift of the tin's superconducting transition temperature

The superconducting transitions presented on the right hand side panel have been measured on a PPMS-9T. The figure below represents a typical screenshot of MultiVu of such transition in a set-up where Channel 1 is measuring the tin manometer mounted outside the easyLab Pcell 30 (zero-pressure – black points) whereas Channel 2 is measuring the tin manometer mounted inside the Pcell 30 (30 kbar – red points).

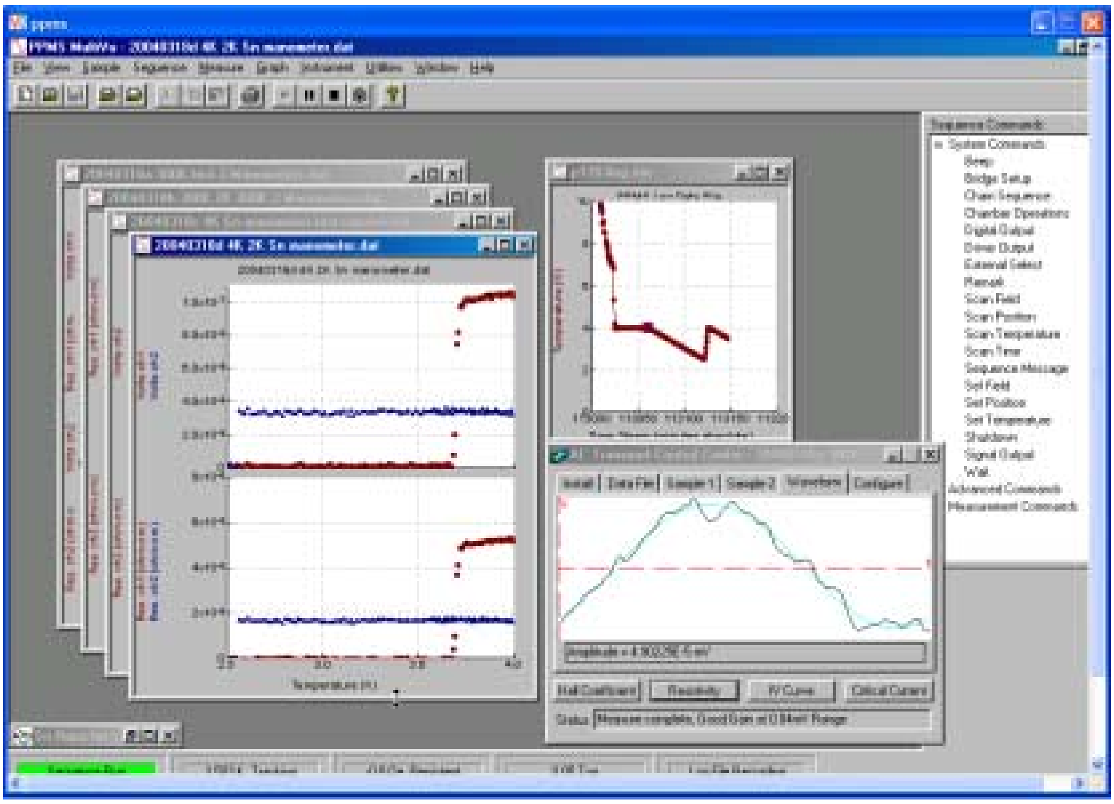


Figure 5: Screenshot of the tin transition – The right-hand side of the screen illustrates the shift of the tin manometer superconducting transition under applied pressure (0 and 30 kbar for black and red points, respectively)

Based on the previous results, one can determine the calibration of the easyLab Pcell 15/30 (Note: these curves depend on the pressure-transmitting medium used): low temperature pressure versus room temperature applied pressure is shown in the figures below.

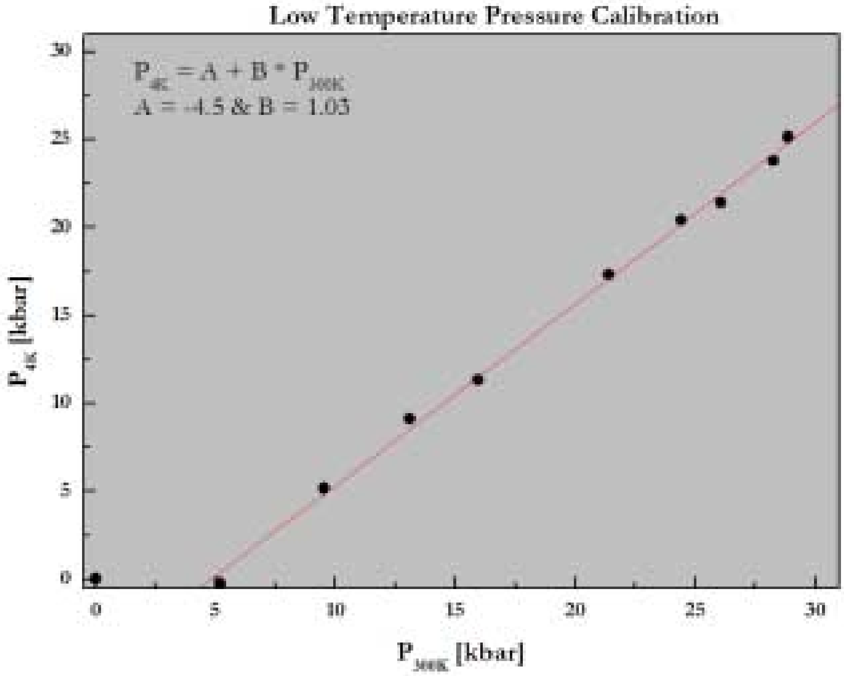


Figure 6: 4 K pressure versus room temperature applied pressure of the easyLab Pcell 30 (note: this curve is dependent of the nature of the pressure-transmitting medium used)

Examples of measurements performed using the easyLab Pcell 15 with a PPMS-9T

This work has been performed using a PPMS/ACT option equipped with a 9 T superconducting magnet. The single crystal of $CeRh_2Si_2$ has been kindly provided and set-up by Dr M. Grosche from the Physics Department, Royal Holloway & Bedford College, University of London (United Kingdom).

The picture below shows the single crystal of the heavy fermion, $CeRh_2Si_2$, mounted on the polyimide platform of the Pcell 15 pre-wired feedthroughs.

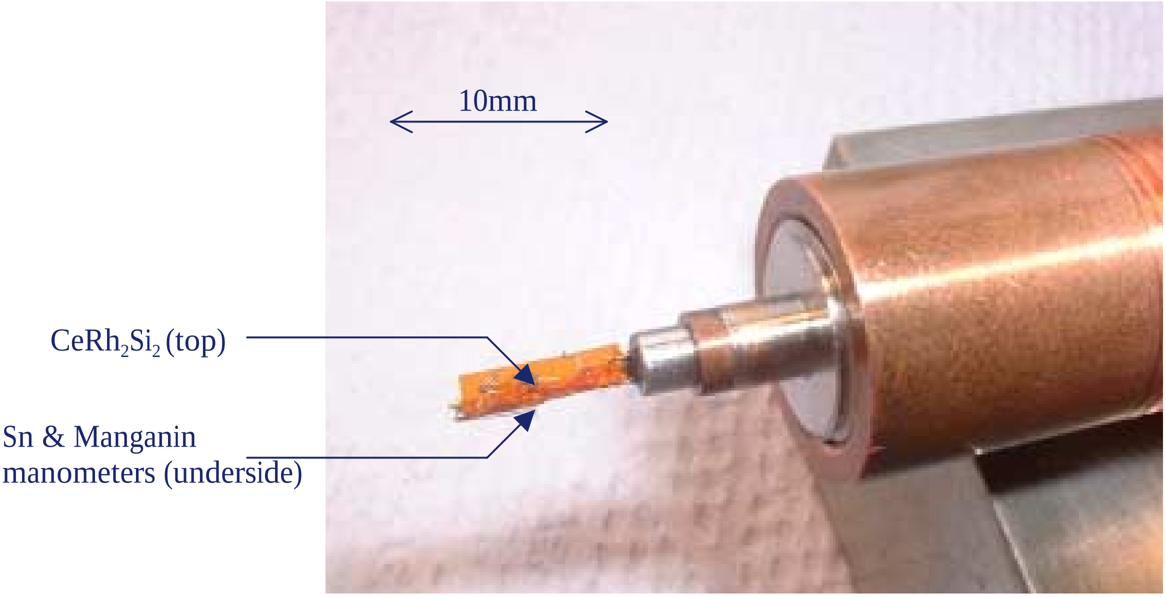


Figure 7: $CeRh_2Si_2$ sample and manometers (tin & manganin) set-up on wired feedthrough.

The data of $CeRh_2Si_2$ have been obtained by cooling down the easyLab Pcell 15 module at rate of approximately 0.3 K/min using the ACT option. The applied pressure was increased up the maximum applied force of 2.5 tons by steps of around 0.5 tons, then by releasing the force by similar steps of 0.5 tons. The various loadings are presented in the figure below (the numbers 1-10 indicate the sequential order of loadings and unloadings).

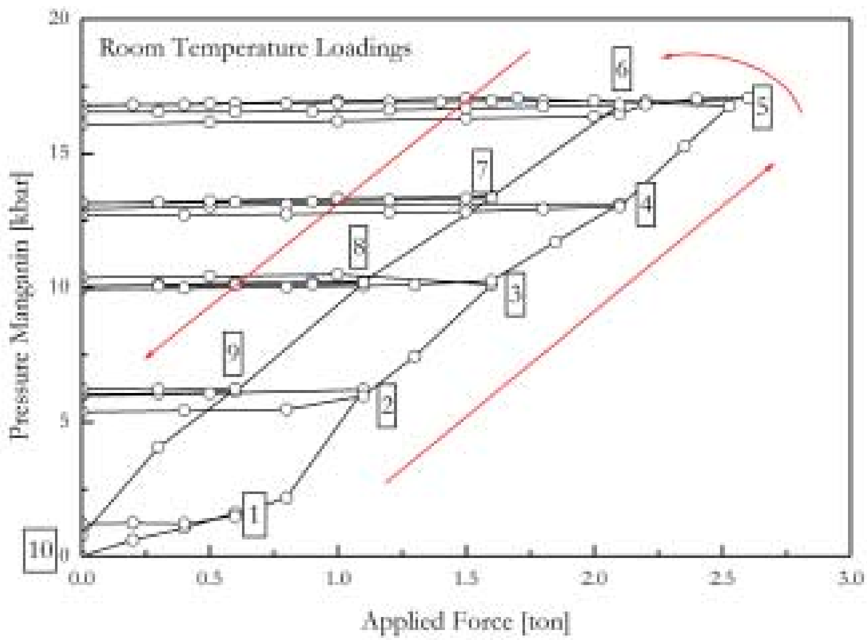
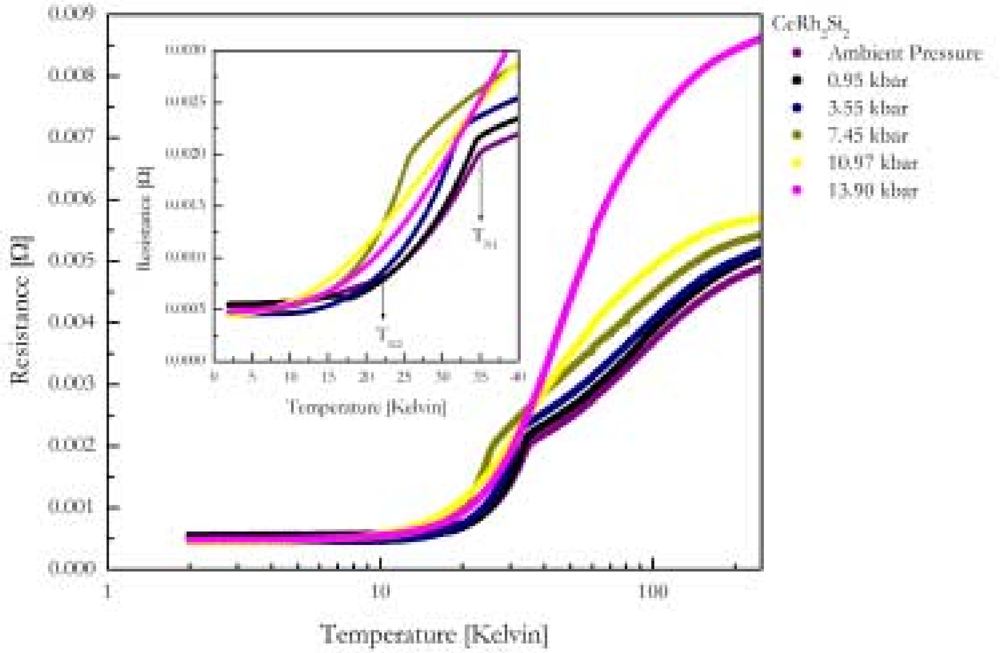


Figure 8: Successive room temperature pressure loading of the easyLab Pcell 15 (300 K)

The resistance data of $CeRh_2Si_2$ are presented in the figure below. At ambient pressure $CeRh_2Si_2$ is an antiferromagnetic material with two successive phase transitions, T_{N1} & T_{N2}. The effect of an applied pressure is quite drastic on these two transition temperatures as they both disappear at respectively 10 kbar and 5 kbar respectively.



Based on these results, the magnetic phase diagram as function of pressure of $CeRh_2Si_2$ can be compared with published results as shown in the figure below. The black and red solid points represent the results obtained with the easyLab Pcell 15, whereas the black and red circles are previous results published by M. Ohashi *et al.*, Physica B 312-313, (2002), 443-444.

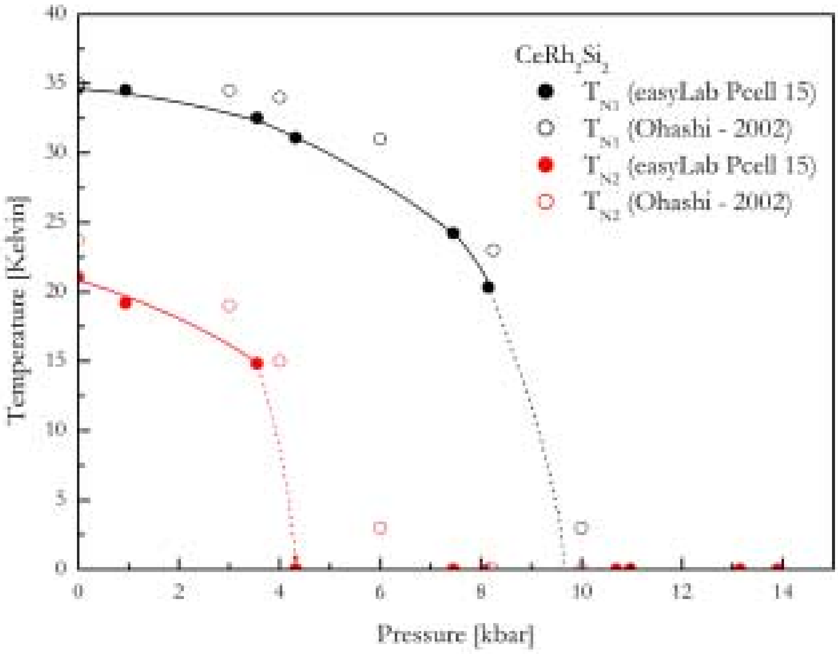


Figure 9: Magnetic phase diagram of CeRh2Si2 as function of applied pressure