What Little I Remember*
The Birth of D3

Introduction

My assigned task is to explain how the polarised beam diffractometer, D3, which has been associated with Francis throughout his career at the ILL, emerged from a primordial sea of experience and prejudice.

When, on 1st January 1973, the UK Science Research Council succeeded in negotiating its late entry into membership of the ILL, a part of the bargain was that it should be responsible for supplying a small number of neutron beam instruments. D3 was amongst the chosen and I had previously attended meetings at the ILL to hammer out its specifications. Scientific members of the ILL Inquisition Team were Francis Tasset and Jacques Schweizer! D3 would be installed on the thermal beam H5 in the main reactor hall in 1973. It's low number in the D series resulted from the rapid demise of an earlier instrument intended to ………………..

The Origin of the Specie

Polarised beam diffractometers for flipping ratio measurements on magnetised single crystals were already in use at a number of reactors. Starting with the original measurements by Cliff Shull and his team in Cambridge, Mass, instruments were built at Brookhaven, Argonne and Oak Ridge in the US. In France, the Saclay and CENG laboratories and in England the AERE Harwell followed suite, as did other European States.

The Design of D3

To better appreciate the form the instrument initially took, I shall outline my experiences and the prejudices acquired before its inception.

While we were still in the Cavendish Laboratory, Jane Brown and I built a digitally controlled, single crystal X-ray diffractometer, MAXIM, (Machine for Automatic X-ray Intensity Measurement). MAXIM was a two-circle instrument with a <u>lifting detector</u> for normal-beam, zero and higher layer measurements (we could not afford a (φ, χ) assembly - an early form of χ circle appears in Figure 1).

The ω and γ circles were driven by DC motors through worm and wheel links and set incrementally to 0.01 degree. The increments to both circles needed to pass from one reflection to the next were computed on the EDSAC computer located in the nearby Mathematical Laboratory and punched on 5-hole paper tape. The control console contained an unholy mixture of uniselectors, hard valves, solid state diodes, decatrons and trochotrons. The integrated intensities measured at slow angular speed were punched out to paper tape, which was then processed on EDSAC. An *astuce,* which pleased us greatly, was that each positioning increment on the setting tape was followed by the absolute position which should result. MAXIM kept count of the incremental pulses and, should agreement not be found with the absolute position supplied, would attempt to recover by winding the circle back to zero and then, using the absolute position as a single increment, achieve error correction. We demonstrated MAXIM at the Physical Society's Annual Exhibition in 1959.

Jane spent 1961-63 at Brookhaven where, amongst a number of more conventional neutron studies, she made flipping ratio measurements on Mn_2Sb with Stan Pickart and also confounded the resident pundits by demonstrating that a large flipping ratio could be observed from a single crystal of an *antiferromagnet*, specifically MnF_2 .

I joined AERE Harwell in 1961 to help with the commissioning of a polarised neutron diffractometer on the 7H1L beam tube of the 14 MW heavy water moderated Pluto reactor.

Bill Gardner, Bob Ferrier and Roger Calder were already in residence, but were in thrall to the Harwell engineers and Martin Wood for the design, construction and installation of a Helmholtz pair of water-cooled, strip-copper coils to produce a 2T vertical field in a 50mm clear bore which would allow access for a cryostat and the sample. Two motor-generator sets (ex film studio) were housed in a special building outside the reactor shell. The larger was rated at 300 KW.

Meanwhile a diffractometer, PN1, based on a brass (non-magnetic) version of the ω and 2θ circles of an AERE Mk 5 instrument was about to be operational and a second beam from the same hole was promised to allow a second monochromator to be installed further from the face of the reactor to service a second diffractometer, PN2. The circles were set by hand.

It was not long before I had added a lifting detector to PN1 and a cam system to provide automated ω offset for background measurements. I begged a slice of $Fe_{92}Co_8$ polarising monochromator crystal from Brookhaven, via Jane, for PN2. An iron-cored electromagnet with asymmetric Helmholtz coils to allow from –5° to +25° access in the vertical plane was built to my design by the Newport Instrument Co. The poles were bored with a 51mm diameter hole, which could be filled, either with more iron tapering at their tips to some 12 mm for experiments at room temperature, or with one of two cryostats for work at liquid nitrogen or helium temperatures. The result was that a vertical field of some 1.7T could be applied to the sample.

Amongst other studies, these developments allowed Jane and me to determine the spatial distribution at 4.2 K of the Dzyoshinski weak ferromagnetism in $MnCO_3$ and Alan Wedgwood to study the aligned paramagnetism in the elpasolite $Na_2K_2CrF_6$.

Following her return to Cambridge in 1964 until her departure for the ILL in 1974, Jane and many of her PhD students continued to make X ray diffractometer measurements in Cambridge on MAXIM and collect polarised neutron data in AERE Harwell. At Harwell, we made the first attempt to optimise the counting statistics in flipping ratio measurements. This took the form of a logic box containing NOR transistor gates, two 7-position rotary switches to select the ratio of up-to-down counting times in peak and background and a bipolar switch to select whether the majority counting time was for neutrons 'up' or neutrons 'down'. PN1 and PN2 remained without computer control.

Before leaving Harwell in 1971, I participated with other physicists in the design of the Mk6 diffractometer, Ron Dyer providing engineers as usual. Some features of the new design were directly due to the physicists:

Shafts to be driven by DC motors and positioned by absolute encoding (in spite of the success of MAXIM). The ω and 2θ circles should be separated by a stationary sleeve to avoid any mechanical interaction. The weight of the detector and its shielding should be supported below its centre of gravity by being mounted on a table with wheels running on steel tracks mounted on a massive cast-iron base.

Detailed design and manufacture were undertaken by Grubb Parsons. A (φ, χ) assembly and a lifting detector module in non-magnetic materials were included. Moore-Read multi-turn brush encoders were geared directly to the shafts such that one bit signified 0.01°. Positive and negative motions from zero could exceed ±180°, but were subject to limit switches.

I also had had some practice in programming DEC PDP8's to control Ferranti-Hilger-Watts neutron diffractometers. The PDP8 was built with 4K fields of 12 bit word memory. A single interrupt register was available. With two fields (24K bytes), no floating point hardware and a hand-crafted operating system by Johnny Hall, we nevertheless were able to compute the setting angles for two diffractometers (using a crystal package designed by Prior and Ellis at Lucas Heights), issue the control instructions, acquire the data and allow one user to evaluate his data on line with the aid of a graphical displayof the reflection profile.

I left Harwell for the Neutron Beam Research Unit in the Rutherford Laboratory in 1971 and, almost immediately, was given the job of delivering D3. Harold Wroe controlled the budget, Peter Hay the engineering support, Kate Knight the electronics and Jeff Penfold help with the programming.

Construction of D3

Mk6 modules, including the lifting detector arm, were used for the basic diffractometer. Polarising crystals of $Fe_{92}Co_8$ and Cu_2MnAl (grown in Grenoble) in their permanent magnets were provided in two interchangeable drums some 300 mm in diameter. A set of interlocking Jabroc wedges with copper tips could be interchanged to change the position of a larger wedge containing the magnetic collimator for the monochromatic beam. In this way, a stepped change in wavelength was available.

The more massive monochrometer shielding and a carriage and rail system to allow the diffractometer to be moved to match the chosen 2θ of the monochromator was provided by the ILL and was under the control of M. Gobert.

Samples were to be magnetised using an electromagnet of the type I had developed at Harwell and fixed temperature liquid nitrogen and helium cryostats with 50 mm diameter tails containing additional iron were part of the consignment. The use of a cryostat was facilitated by second ω table mounted on top of the electromagnet, just below the body of the cryostat.

The ILL had installed two medium-sized computers to control and receive data from the diffractometers (CARINE) and inelastic instruments (NICOLE).
Throughout the specification stage, I had insisted that D3 should have its own dedicated control computer. Prior to this, only the Hedgehog (Igel) diffractometer conceived by B Klar had succeeded in avoiding the somewhat suspect charms of CARINE. As I saw it, the rather specialised nature of D3 set it apart from other diffractometers and I was not in favour of being dependent on CARINE not developing a headache, thereby paralysing half the neutron instruments, irrespective of the state of the reactor.

Consequently, Jeff Penfold and I found ouselves up to our necks in PDP11 assembler language (MACY-11) and the late Philippe Ledept's brilliant operating system, with only 2 K resident in the 16 K memory (an already too generous purchase in the view of ILL's Director Rudolf Mőssbauer).

The PDP11 provided for vectored interrupts. The interface between the PDP11 and the instrument used the CAMAC standard and Kate Knight designed a novel module to provide Flipper Control. Using an internal clock, measurement time was chopped into the requested ratio with incident 'spins' up compared to 'down', detected neutrons being switched between the relevant scalers. Short intervals between up and down periods allowed the flipper to stabilize. The flipper itself used the, by then traditional method, of spin Larmor precession when the neutron beam passed through a solenoid in a fixed vertical magnetic field. The solenoid was fed with RF at a frequency to suit the vertical field and at a power dependent on the neutron wavelength.

<u>Installation and Commissioning</u>

D3 was delivered to the ILL in 1973 and my wife and I lived in Grenoble during 73/74 for the installation and commissioning, including much more program development.

Control instructions were introduced by a three letter nemonic, chosen to indicate the function, preceded by a / or a * and followed by numerical data if required. '/' orders just stored the data, whereas '*' orders would act on already stored data or, if followed by more data, store that and then act on it. All instructions appeared on the output teleprinter and on DECtape together with the numbers of neutrons detected in response to a measuring instruction *MES. The heirarchical nature of the instructions made for an ultimate *CPB – collect Bragg peaks!

On arrival at the ILL, Jane joined in the fun adding software for counting statistics optimisation and automatic half-shutter peak location routines that are still in use today. Francis suggested that provision be made such that groups of orders could be treated as a subroutine to be entered one or more times.

The mainframe computer at the ILL was a DEC10 on which the output DEC tapes were read and the data processed by programs using the CCSL subroutine library conceived by Jane and Judy Matthewman.

Early Developments

It soon became clear that the Larmor Precession module was not very reliable and it was replaced by one from the LETI.

Funds became available from the ILL for a superconducting magnet to increase the available flux at the sample. In close collaboration with Oxford Instuments, a specification for a 4.6 T Nb/Zr magnet specifically designed for diffraction and D3 with its lifting detector was arrived at. Each of four solid separators between the two asymmetric halves of the Helmholtz coil occupied somewhat less than 45º, so that all angles of diffraction could be reached using one of two alternative rotational positions for the magnet. What an excellent use for the basic ω table!

These manoeuvres were transparent to the user, since the control program divided the desired ω angle for the sample between the upper and lower ω tables as appropriate for the requested γ angle. The superconducting magnet was delivered in 197**?**, and it is still working well. A light-weight ω table mounted on top of the cryostat drove a centre-stick with the sample at its lower end as can be seen in Figure?

Tribute

It has been a privilege to be asked to give this talk, during which I have attempted to name many of the usual suspects. Since my good friend Francis assumed complete responsibility for D3, I believe that he has proved to be an exceptional parent who has continued to invent major improvements to its performance and capabilities, about which we shall hear from the following speakers.

*With acknowledgements to the late Professor O R Frisch (1904-79). Author of 'What Little I Remember' Cambridge University Press 1979.

J B Forsyth
6th March 2009

MAXIM

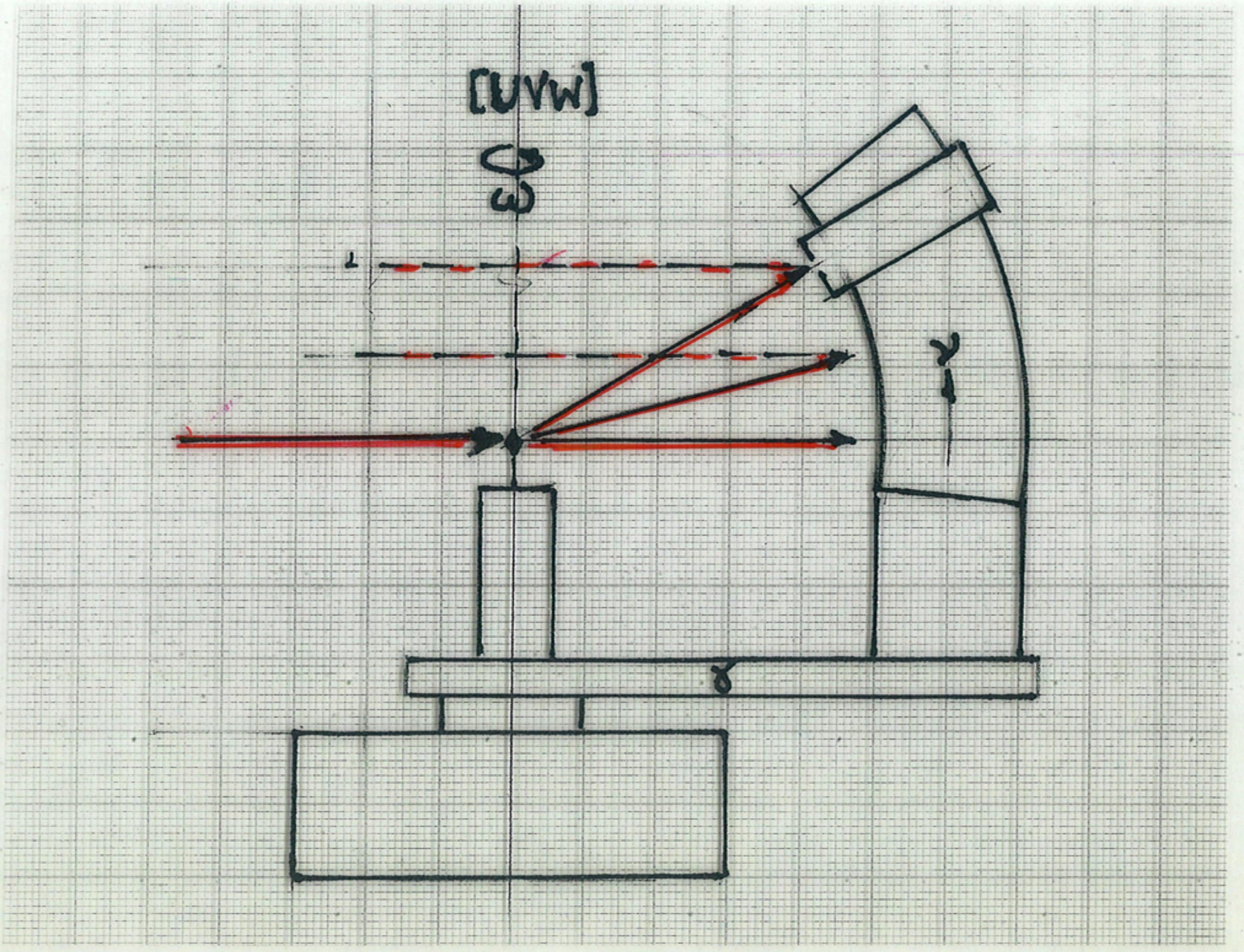
Worm drives to two axes fitted with home-made incremental encoders to provide a pulse every 0.01degree on the crystal rotation, ω, and the detector arm.

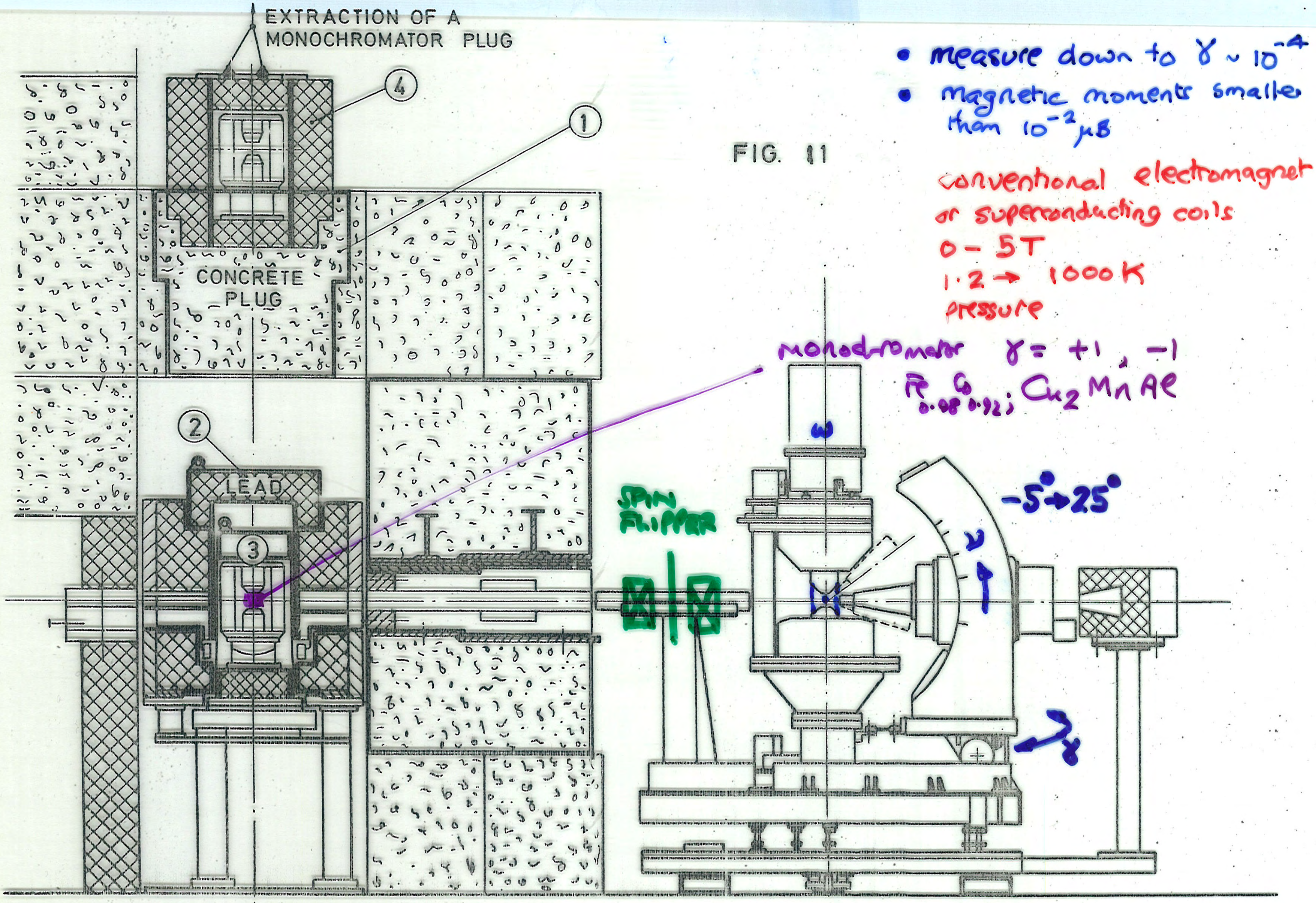
Limited three dimensional data obtained using normal-beam, zero and higher layer geometry, the detector being tilted out of the horizontal and hand set to receive the layer of reflections to be measured. The φ,χ modules commonly used in 4-circle diffractometers had yet to be developed.

Reflection settings, both incremental from the previous reflection and absolute with reference to the axes zeros, calculated on the EDSAC computer some 100m distant. Transmission via punched paper tape.

Error checking and recovery. Measurement data to EDSAC, again via punched paper tape.

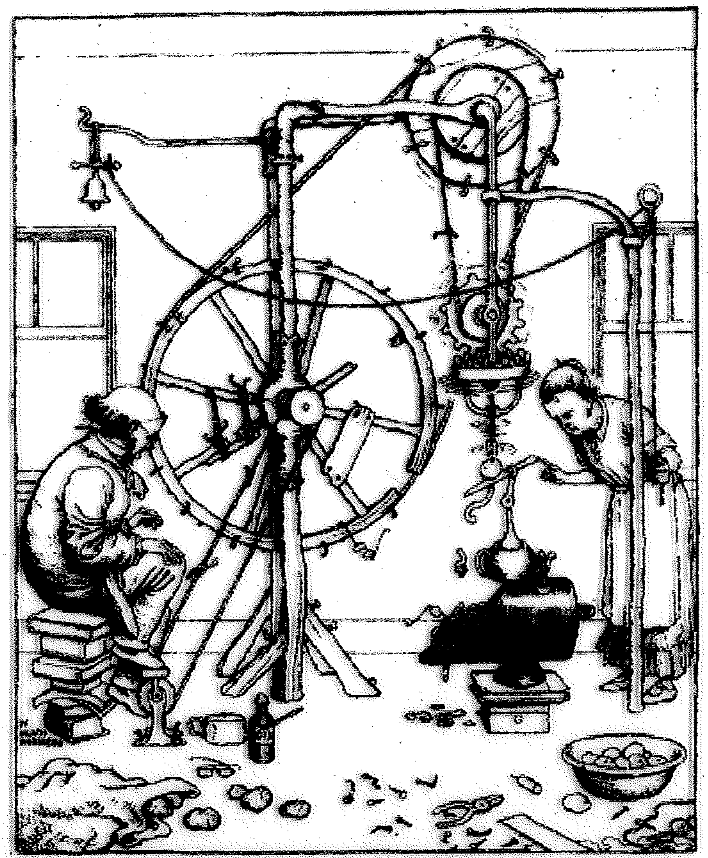
Lesson learnt: absolute encoding of shafts would be better when encoders became available.



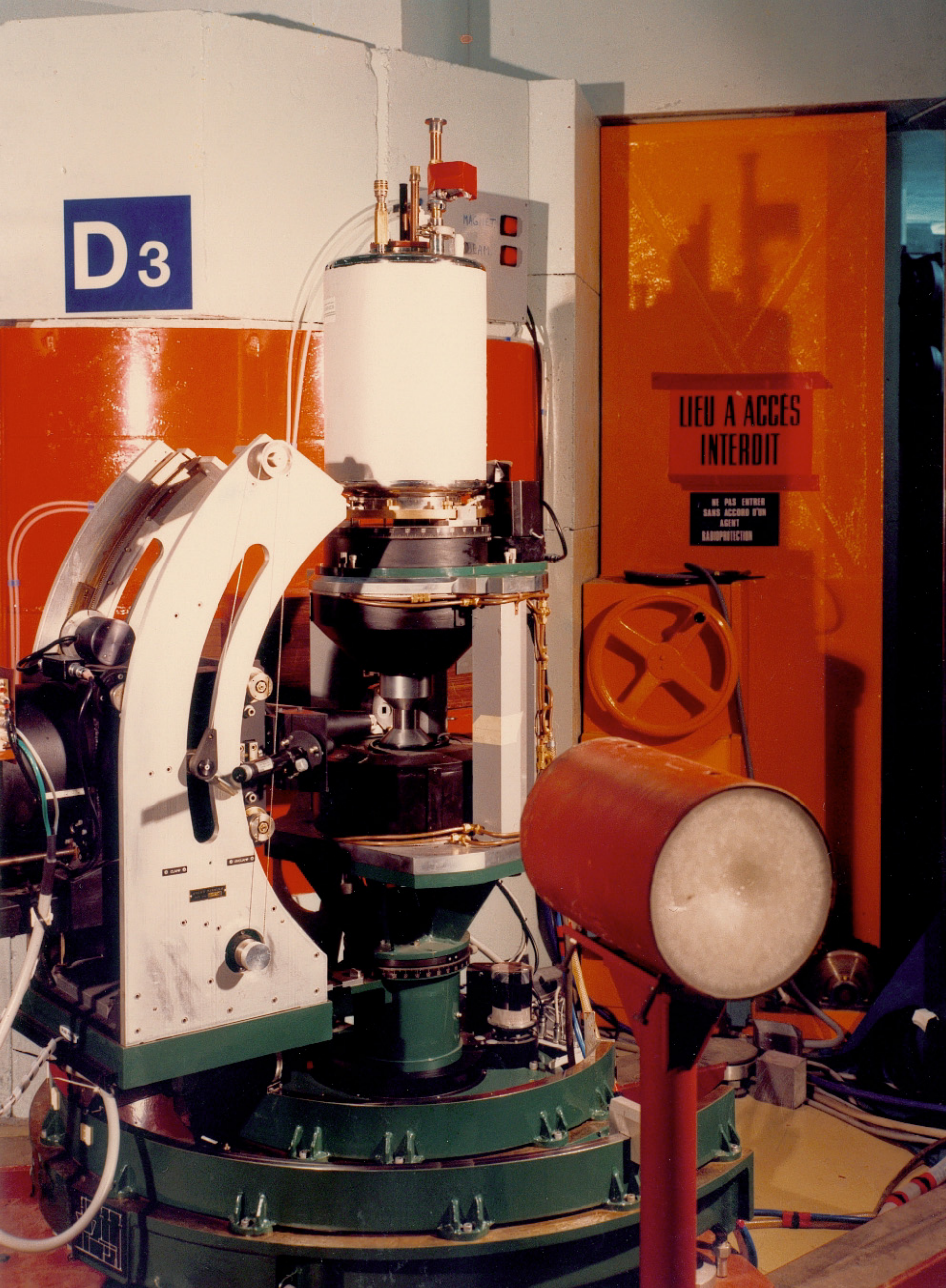


EXTRACTION OF A MONOCHROMATOR PLUG
4
1
CONCRETE PLUG
2
LEAD
3
• measure down to γ ~ 10^-4
• magnetic moments smaller than 10^-2 μB
conventional electromagnet or superconducting coils
0 – 5T
1·2 → 1000K
pressure
monochromator γ = +1, -1
Fe0.08 Co0.92 ; Cu2 Mn Al
SPIN FLIPPER
ω
-5° → 25°
ν
γ

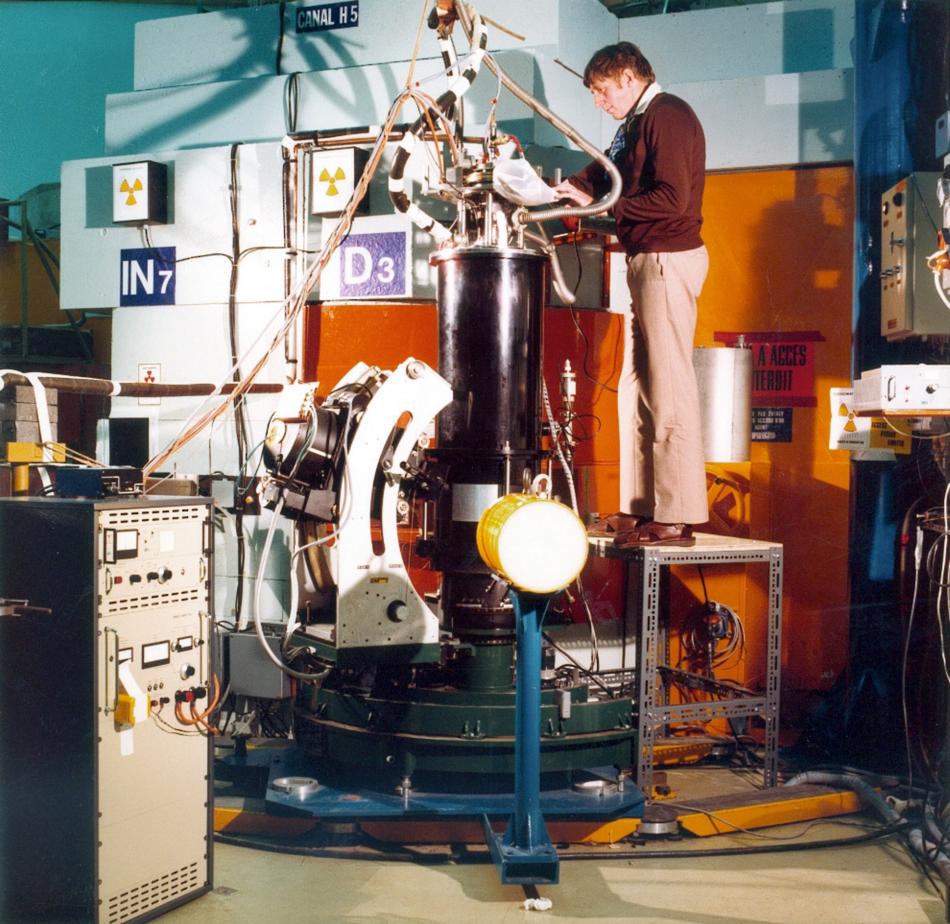
FIG. 11



The Professor's invention for peeling potatoes.


D3
LIEU A ACCES INTERDIT
NE PAS ENTRER SANS ACCORD D'UN AGENT RADIOPROTECTION


digital
pdp11
digital equipment corporation maynard massachusetts
PDP 11/07
DECTAPE 173120
DISK 173220
pdp11/40
decwriter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