Performance of the cooled pyrolytic graphite analyser bank on the OSIRIS spectrometer at ISIS

Preliminary results

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ABSTRACT

The pyrolytic graphite (PG) analyser bank on the OSIRIS high-resolution quasi-elastic/low energy inelastic spectrometer at ISIS has been upgraded. The graphite is now cooled close to liquid helium temperature to improve the sensitivity of the spectrometer. In this report we ascertain the performance of the newly upgraded instrument via resolution and intensity measurements collected before and after the modifications. In addition, OSIRIS resolution measurements are compared to resolution data collected from the cooled pyrolytic graphite analyser bank on the IRIS spectrometer.

CONTENTS

I. INTRODUCTION

Development of the OSIRIS neutron instrument at ISIS is a 3-stage process, namely the sequential realization of high-resolution diffraction, low energy spectroscopy and full polarisation analysis capabilities on the instrument.

The first stage, implementation of high-resolution diffraction, was completed in 1998. Indeed, up until August 2002 OSIRIS operated predominantly, yet very successfully, as a high-resolution back-scattering powder and single crystal diffractometer.

However, November 2003 saw the completion of Stage 2. As a result, the instrument now *also* affords the ISIS user the option of high-resolution quasi-elastic and low-energy inelastic spectroscopy – OSIRIS operating as a time of flight (t.o.f) inverted-geometry crystal analyser spectrometer. For this purpose a large pyrolytic analyser bank oriented close to back-scattering geometry (Figure 1) was installed in August 2002. Using non-backscattering geometry avoids the loss in intensity caused by a beam modulation chopper when exact back scattering is employed.

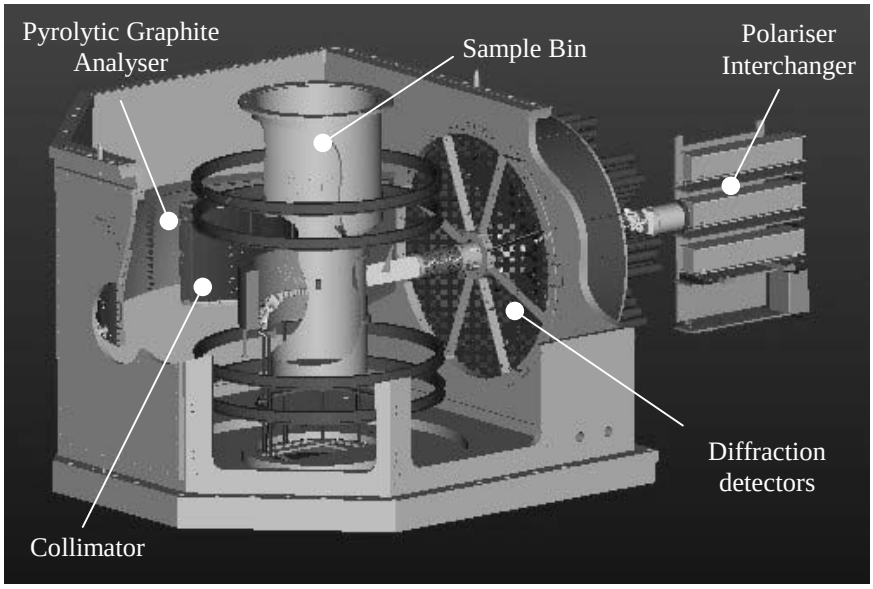


Figure 1. *The OSIRIS spectrometer at the ISIS Facility, Rutherford Appleton Laboratory, UK*

The pyrolytic graphite (PG) analyser bank on OSIRIS is set approximately 0.9 meters from the sample position in the horizontal scattering plane and covers scattering angles from 11^{o} to 155^{o}. The analysed beam is back scattered through approximately 170^{o}, slightly below

the horizontal scattering plane, and detected using a multi-detector composed of 42 ^{3}He 'half inch' detector tubes located approximately 0.7 meters from the analyser (the total secondary flight path is equal to 1.582m). The analyser itself is comprised of a 40 row by 226 column array of pyrolytic graphite crystals (10mm x 10mm x 1 mm thick) with a mosaic spread (η) of 0.8^{o} mounted on a spherically machined aluminium backing plate. Indeed, the OSIRIS graphite analyser intercepts approximately 9% of the total scattered beam. The use of pyrolytic graphite affords the possibility of two analysing reflections, 002 and 004, with analysing energies of 1.845meV and 7.375meV providing resolutions of 24.5μeV and 95μeV respectively. Prior to November 2003 the analyser operated at room temperature.

In contrast, graphite analyser on the neighbouring backscattering spectrometer, IRIS [1], consists of 4212 crystals (1cm^2, 1mm thick, 18 rows by 234 columns) and affords the user an energy resolution of 17.5μeV or 55μeV for the 002 and 004 analysing reflections respectively. However, as is demonstrated by the current operation of the instrument [3], the sensitivity of IRIS has been greatly improved by simply cooling the graphite crystals close to liquid helium temperature [2].

II. MODIFICATIONS

Provision has now been made to cool the graphite crystals on the OSIRIS analyser from room temperature to close to liquid helium temperature in a manner similar to that employed on IRIS. Cooling the graphite close to 4.2K reduces background contributions arising from Thermal Diffuse Scattering (TDS) thus significantly improving the sensitivity of the spectrometer [2]. To achieve such a low temperature it has been necessary to address the method by which the graphite crystals are cooled. Full details of the cooling circuit will be presented elsewhere. However, in brief, a uniform sub-10K temperature is achieved across the analyser bank using three 1.5 Watt Sumitomo Closed Cycle Refrigerators. While the 'first stage' of each CCR is attached to the radiation shield surrounding the analyser (see Appendix I, Figures 11 and 12) to lessen the radiative load on the anlayser crystals themselves, the 'second stage' is bolted directly onto the back surface of the machined aluminium backing plate. As a result, the analyser is seen to cool from room temperature to 8K in approximately 48 hours (Figure 2). The positions of the 16 silicon and 4 platinum sensors mounted on the CCRs, analyser and radiation shield are illustrated in Appendix II

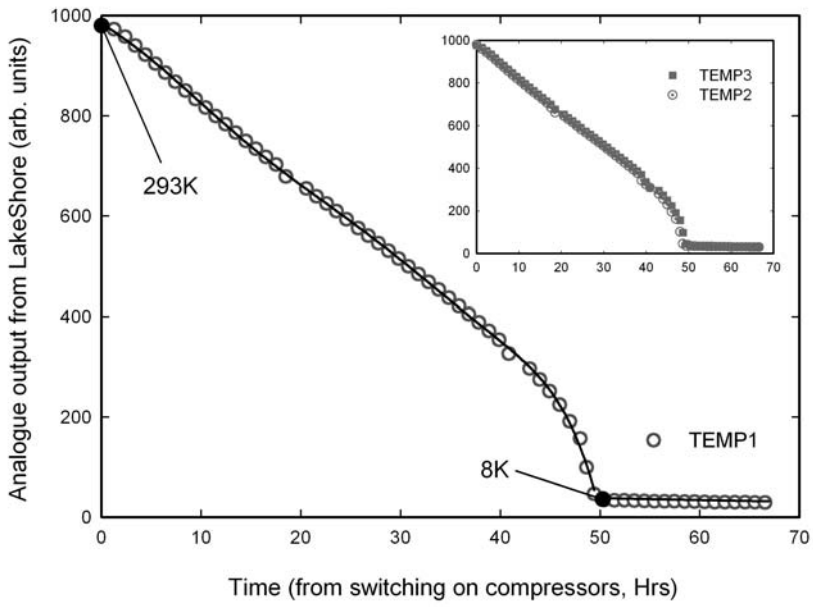


Figure 2. *Cooling profile of the graphite analyser bank. Temp1, 2 and 3 refer to three different sensors attached to the aluminium backing plate. The 'base' radiation shield temperature is approx.160K*

III. RESULTS

Performance of the newly cooled OSIRS pyrolytic graphite analyser was ascertained by comparing the performance of the newly upgraded instrument to resolution and intensity measurements collected prior to the modifications. In addition, such data was also compared to resolution measurements collected from the cooled (12K) pyrolytic graphite analyser bank on the IRIS spectrometer. Unless otherwise stated, data was collected from both instruments with their respective radial collimators situated between sample and analyser. The results are shown in Figures 3-9 and tabulated in Table 1. The findings are also likened to the predicted energy resolution of the complete upgraded OSIRIS spectrometer obtained from Monte Carlo PG002 and PG004 simulations of the complete instrument [see for example reference 4]. In addition, photographs showing the installation of the new analyser bank are presented in Appendix 1.

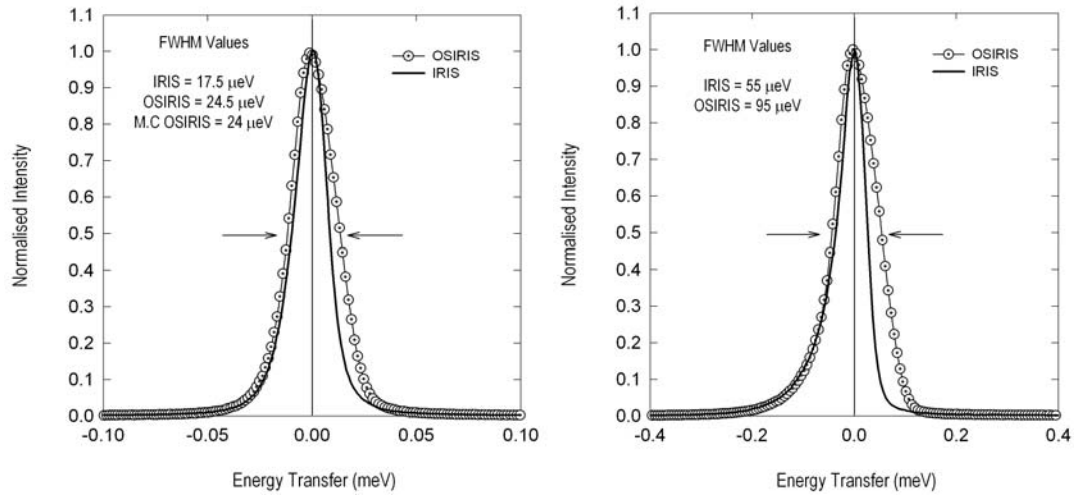


Figure 3. *Normalised intensity vs. energy transfer for PG002 and PG004 respectively. All data was collected from the cylindrical vanadium standard.*

In brief, the OSIRIS spectra presented in this report were generated by collating the data collected in each of the 42 pyrolytic graphite bank detectors, each detector having been first corrected for efficiency. A similar treatment was applied to the 51 spectra collected on IRIS. All resolution measurements, irrespective of instrument, were collected using the same cylindrical vanadium standard; the spectrometer configured to use either the 002 or 004 analysing reflection afforded by pyrolytic graphite. From here on in, these two analysing reflections will be referred to as PG002 and PG004 respectively.

The marked increase in vertical extension of the OSIRIS analyser (40 cm high) out of the horizontal scattering plane, compared to the IRIS analyser (18 cm high), accounts for the observed, yet unavoidable, degradation of energy resolution for both the PG002 and PG004 configurations (Figure 3). For PG002, comparison of resolution measurements collected from OSIRIS and IRIS highlights a 8μeV increase in the breadth of the elastic line at full width half maximum (f.w.h.m), i.e. from 17.5μeV (IRIS) to 24.5μeV (OSIRIS). Similarly, for PG004, an increase in f.w.h.m of 40μeV, from 55μeV (IRIS) to 95μeV (OSIRIS), is observed.

As Table 1 illustrates, these f.w.h.m results are in good agreement with those values predicted from Monte Carlo PG002 and PG004 energy resolution simulations of the complete upgraded OSIRIS spectrometer.

	Monte Carlo Prediction	OSIRIS (Analyser at 8K)	IRIS (Analyser at 12K)
FWHM of Elastic Line PG002 PG004	 24.2 (34) μeV 97.23 (23) μeV	 24.5 μeV 95 μeV	 17.5 μeV 55 μeV
Signal to Noise Ratio	-	3350:1	3250:1

Table 1. *Performance of the newly cooled pyrolytic graphite analyser on the OSIRIS spectrometer*

However, comparison of the number of detected neutrons in the elastic channel ($\Delta E=0$) from each spectrometer does highlight a 4.5-fold increase in neutrons count rate per microsecond on OSIRIS. Such an increase is the multiplicative consequence of i) the approximate 2-fold increase in the number of 6.6 Angstrom neutrons transported from moderator to sample position on OSIRIS and ii) the approximate 2-fold increase in vertical elongation of the OSIRIS (40cm high) analyser compared to that installed on IRIS (18cm high).

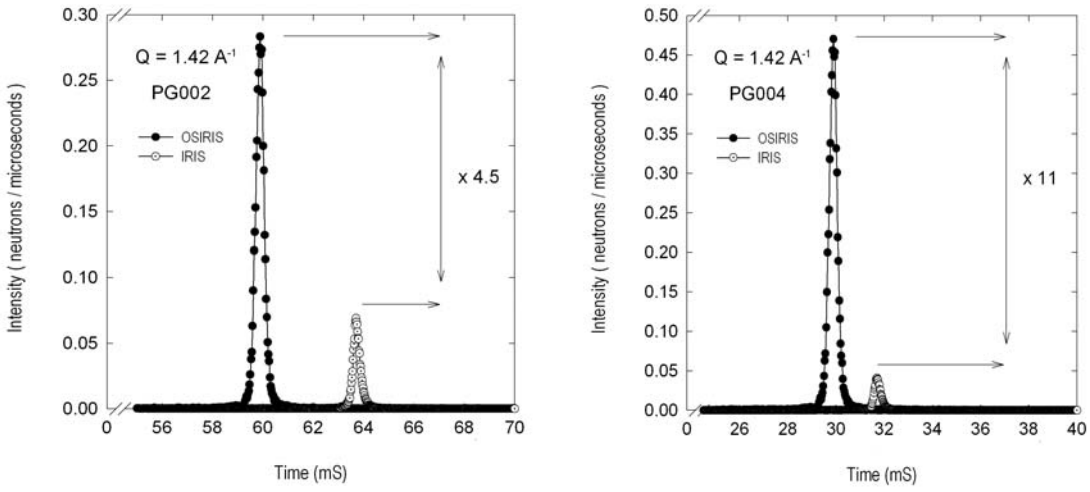


Figure 4(a) *Increase in number of detected neutrons per μS in the elastic channel for PG002 on OSIRIS compared to IRIS* **(b)** *similarly for PG004*

Finally, cooling the graphite crystals close to liquid helium temperature is seen to significantly improve the sensitivity of the spectrometer, as is illustrated in figure 5. While the OSIRIS spectrometer exhibits a signal to noise ratio (SNR) of approximately 500:1 at

room temperature, a SNR value of 3350:1 is achieved by 8 Kelvin. For comparison, IRIS exhibits a SNR of 3250:1 with its analyser cooled to 12K.

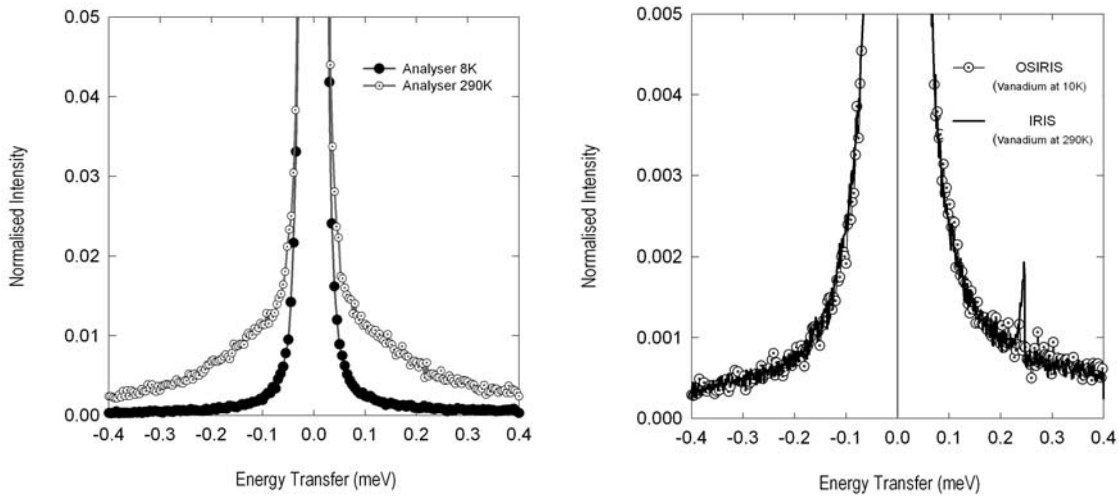


Figure 5(a). *Suppression of Thermal Diffuse Scattering (TDS) by cooling the analyser crystals [2]* ***(b)*** *comparison of vanadium resolution measurements from OSIRIS (vanadium cooled to10K) and IRIS (vanadium at 290K) NOTE: the feature at approx. 0.22meV is a consequence of the ISIS proton pulse extraction*

However, it should be noted that on OSIRIS a SNR ratio of 3350:1 is obtained only when the cylindrical vanadium standard is cooled close to liquid helium temperature. With the vanadium at room temperature an instrumental SNR of approximately 1200:1 is observed. Such degradation at elevated temperatures is due to the temperature dependant background feature illustrated in figure 6. This feature is also observed in data collected from an empty OSIRIS aluminium flat plate sample can and, it has been recently noted, to a greater or lesser extent during scheduled experiments.

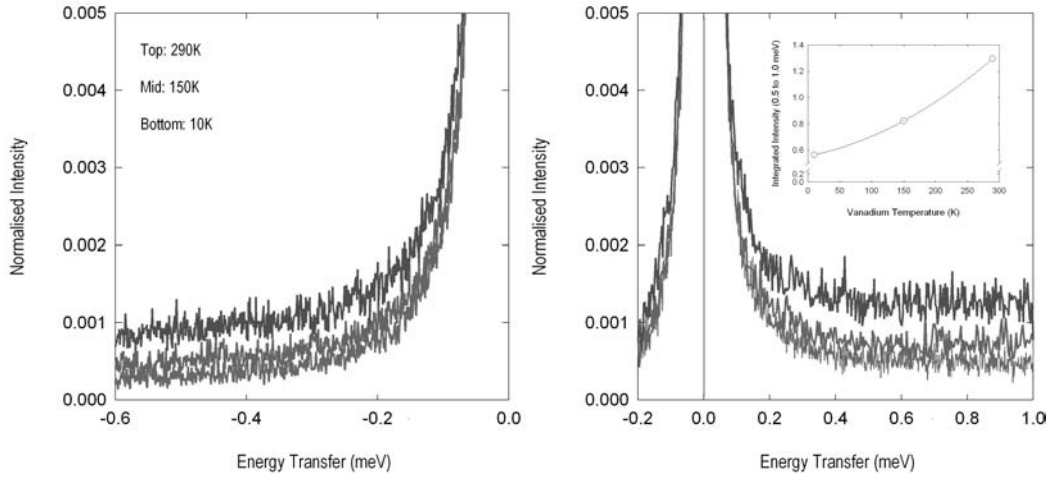


Figure 6. *Observed temperature dependant background from the cylindrical vanadium standard. Inset: Integrated background level (from 0.5meV to 1meV) as a function of temperature*

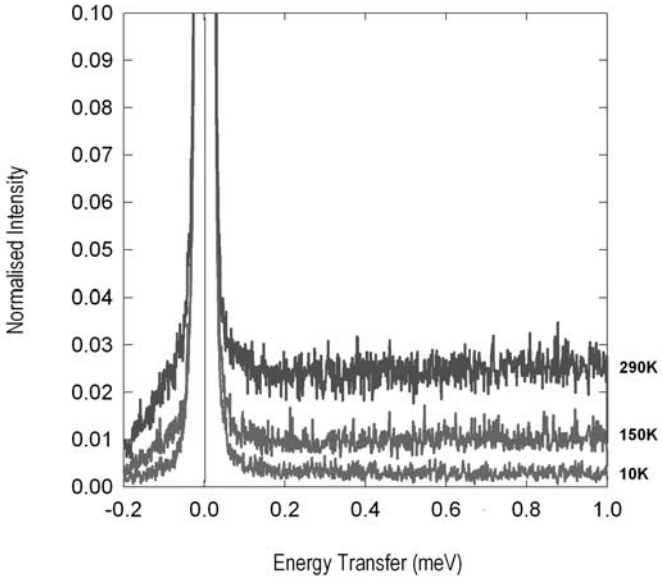


Figure 7. *Temperature dependant background observed from an empty OSIRIS flat plate sample can*

The background feature also appears to be momentum transfer (Q) dependent at elevated temperatures, as illustrated in Figure 8

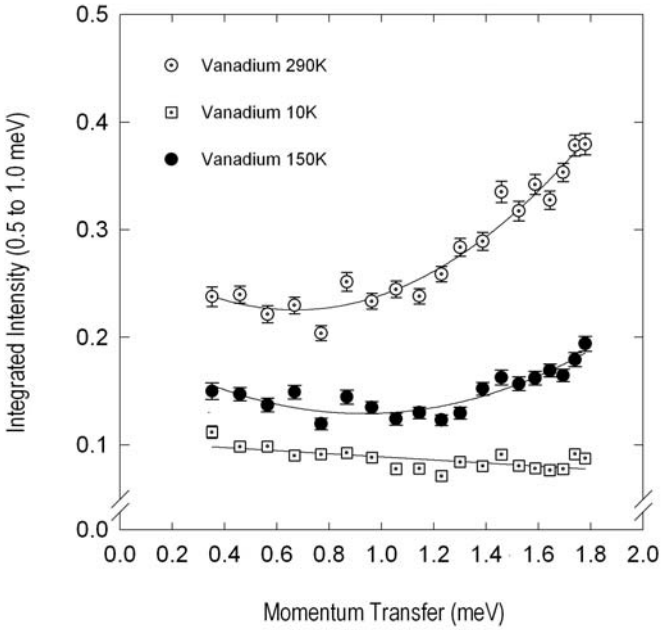


Figure 8. *Observed variation in intensity of the background feature as a function of sample (vanadium cylinder) temperature and momentum transfer. The intensity values plotted were determined by integrating the vanadium spectra from 0.5 to 1.0 meV. The solid lines are a guide to the eye.*

At present, this potentially problematic background feature is believed to be a consequence of contamination of the PG002 signal from the PG004 analysing reflection. On IRIS, in theory, such contamination can be removed when using the PG002 setting by simply inserting a cooled (25K) Beryllium filter between the sample position and the analyser – the cooled beryllium suppressing transmission of neutrons with energies greater than 5meV. However, it has been noted that IRIS cylindrical vanadium standard resolution measurements taken with and without the beryllium filter in position show little contrast (Figure 9). It appears that the temperature dependant effect observed on OSIRIS is suppressed on the neighbouring spectrometer. Further measurements are needed to ascertain why this should be the case.

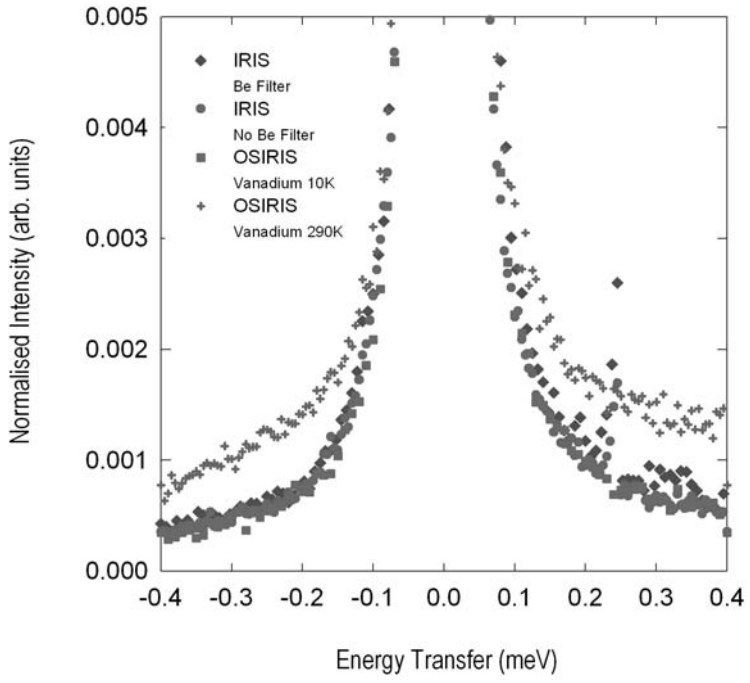


Figure 9. *Vanadium standard measurements (room temperature) collected on IRIS (analyser at 12K) with and without the cooled Beryllium filter between the sample and analyser. For comparison, vanadium standard measurements (10K and 290K) collected on OSIRIS (analyser at 8K) are also plotted. NOTE: the feature at approx. 0.22meV is a consequence of the ISIS pulse extraction*

IV. Discussion

The pyrolytic graphite (PG) analyser bank on the OSIRIS high-resolution quasi-elastic/low energy inelastic spectrometer at ISIS has been upgraded. The graphite crystals are now cooled close to liquid helium temperature to improve the sensitivity of the spectrometer. The performance of the newly cooled analyser bank has been ascertained by comparing resolution and intensity measurements collected before and after the instrument upgrade. In addition, OSIRIS data has been compared to resolution measurements colleted using the cooled pyrolytic graphite analyser bank on the IRIS spectrometer.

Analysis shows that the instrument upgrade project has resulted in a marked improvement in the performance of the complete spectrometer. While energy resolution measurements for both PG002 and PG004 configurations are in good agreement with predictions made for the performance of the complete upgraded instrument using Monte Carlo simulations, cooling the analyser crystals close to liquid helium temperature greatly improves the signal to noise ratio, and hence sensitivity, of the spectrometer. Furthermore, when compared to the current operation of the IRIS spectrometer, OSIRIS shows a 4.5-fold and 11-fold increase in number of detected neutrons per microsecond for PG002 and PG004 respectively. Such improvements will greatly aid and enhance not only the study of condensed matter systems but, with regard the improved SNR and ability to use sub-1K and magnetic field sample environment apparatus on OSIRIS, low energy magnetic excitations.

However, detailed analysis does highlight a potentially problematic PG002 background response that appears to be sample temperature dependant. Further investigation into the origin of this feature is necessary. However, preliminary tests suggest the feature to be contamination of the PG002 signal from the PG004 analysing reflection. Should this hypothesis prove correct then a cooled beryllium filter will be commissioned, designed and installed to suppress PG004 contamination.

V. References

1. C.J.Carlile *et al*, Physica B, 182 (1992) 431-440

2. C.J.Carlile *et al*, Nuclear Instruments and Methods In Physics Research A, 338 (1994) 78-82

3. M.T.F.Telling *et al*, Applied Phys A 74 (2002) [Suppl1], s61-s63

4. *The optimisation of analyser geometry for a near backscattering spectrometer – IRIS on the ISIS pulsed source*, M.T.F.Telling and S. I. Campbell, Technical report RAL-TR-1999-044 (*http//www-dienst.rl.ac.uk/library/1999/tr/raltr-1999044.pdf)*

Appendix I. Installation Photographs

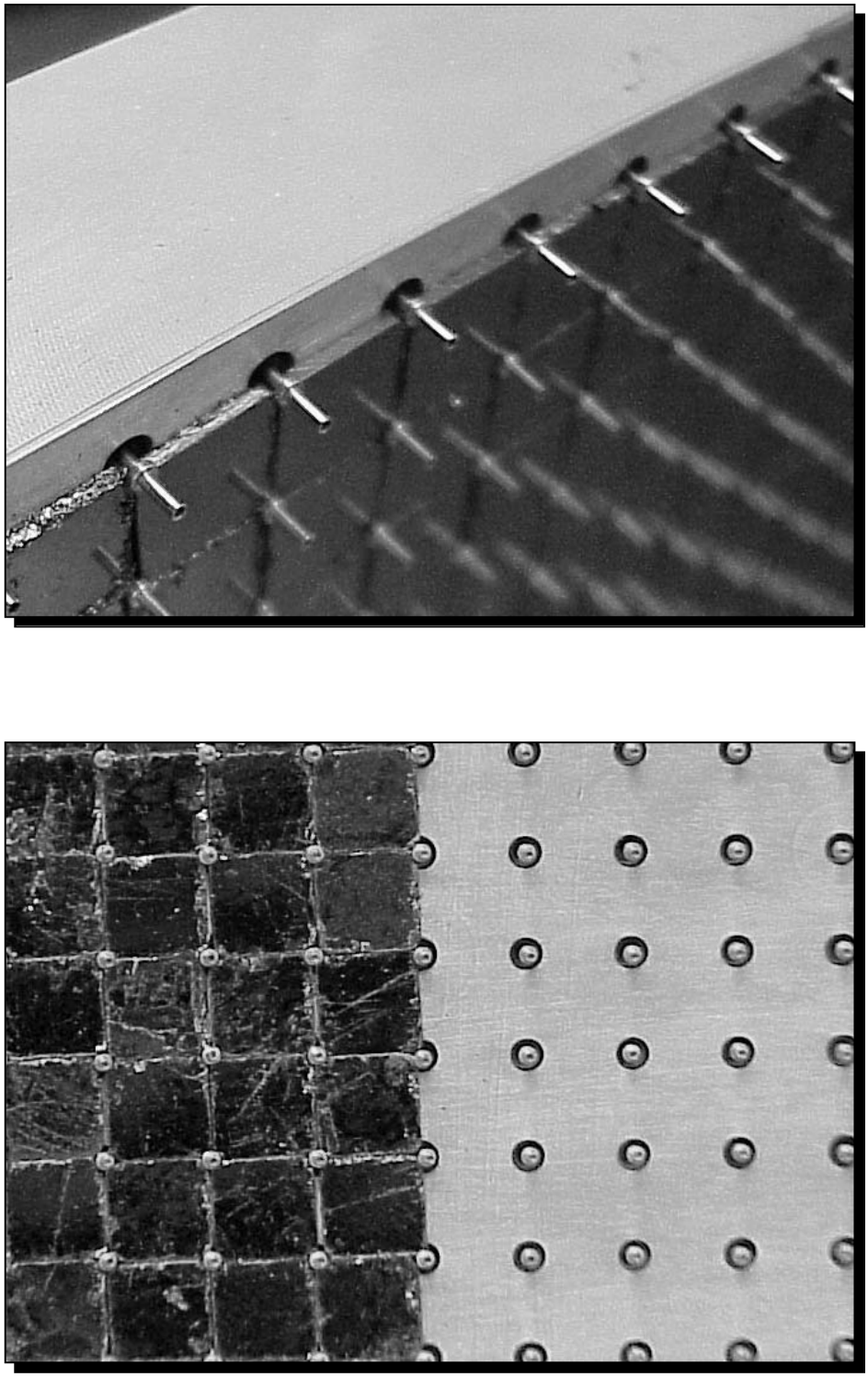


Figure 10. *The method of 'pinning' the pyrolytic graphite crystals to the cadmium sheet lining the face of the aluminium backing plate*

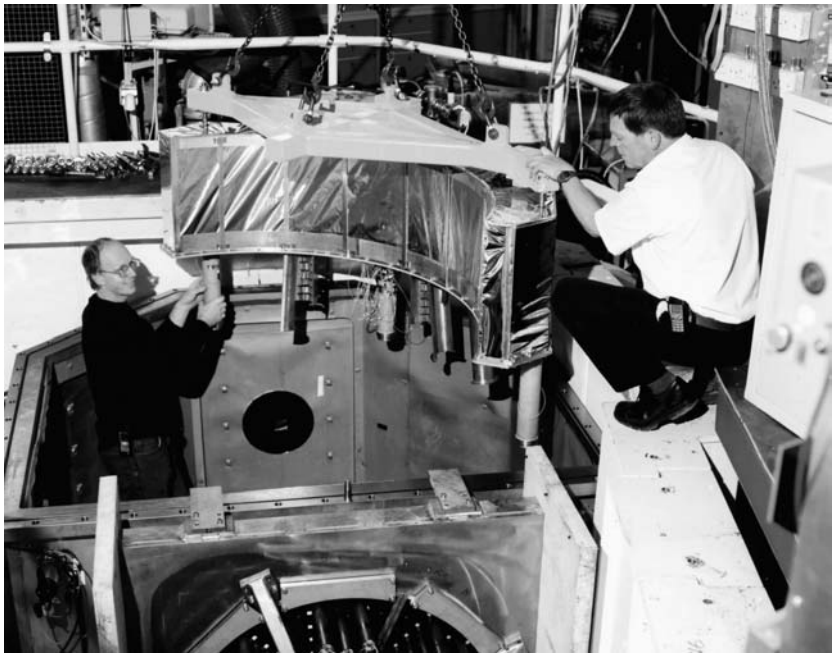


Figure 11. *The graphite analyser bank being lowered into position. The entire analyser assembly, including the mounted graphite crystals, is surrounded by a radiation shield and fronted by a thin aluminium window.*

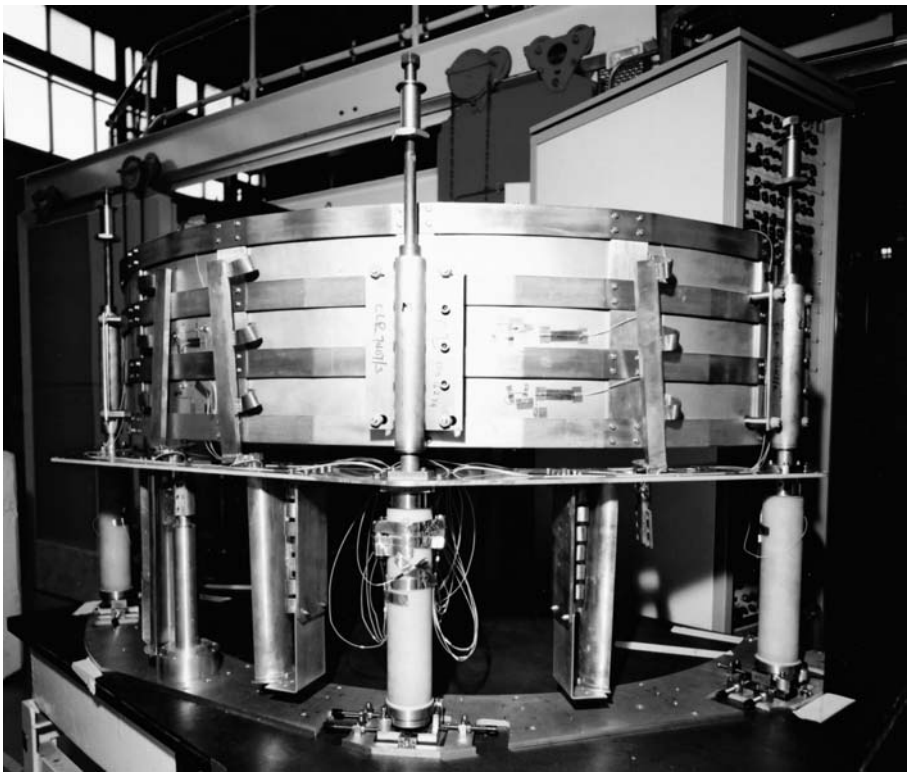
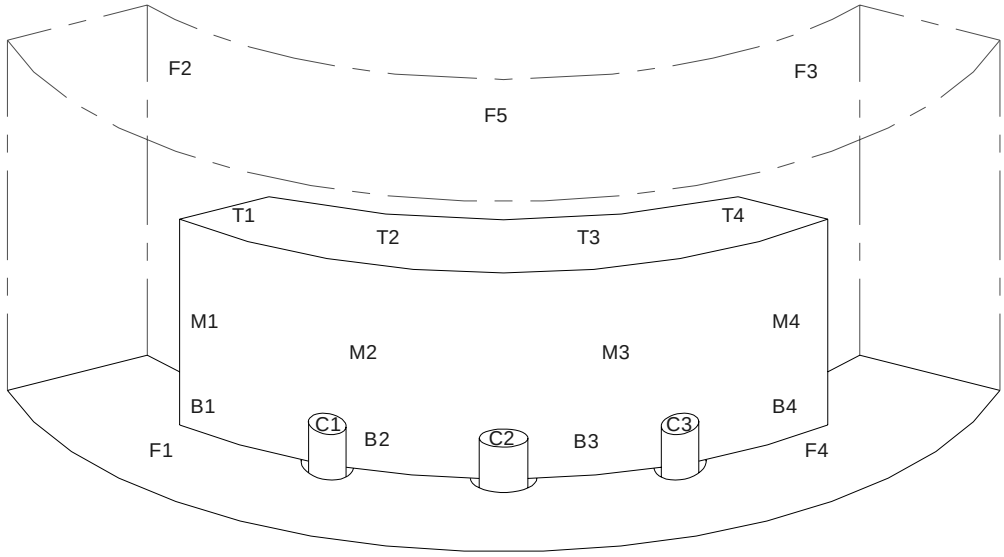


Figure 12. *The copper straps attached to the back of the aluminium backing plate used to ensure a uniform temperature profile across the graphite crystals*

Appendix II. Sensor Positions



1. TOP1	5. MID1
2. TOP2	6. MID2
3. TOP3	7. MID3
4. TOP4	8. MID4

1. BOT1	5. 1ST1
2. BOT2	6. 1ST2
3. BOT3	7. 1ST3
4. BOT4	8. 1ST4

AUX
$\overline{\text{CCR1}}$
CCR2
CCR3
1ST5

Appendix III. Cryomagnet Test Results

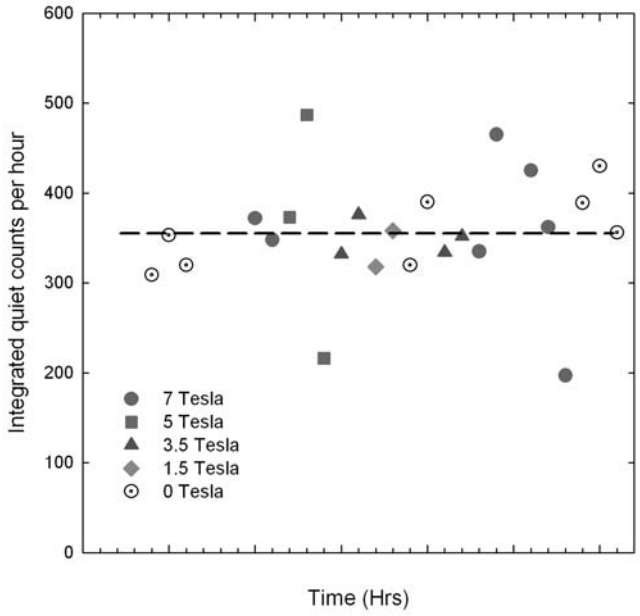


Figure 13. *Quiet counts as a function of magnetic field. Data collected during ISIS shut down period.*

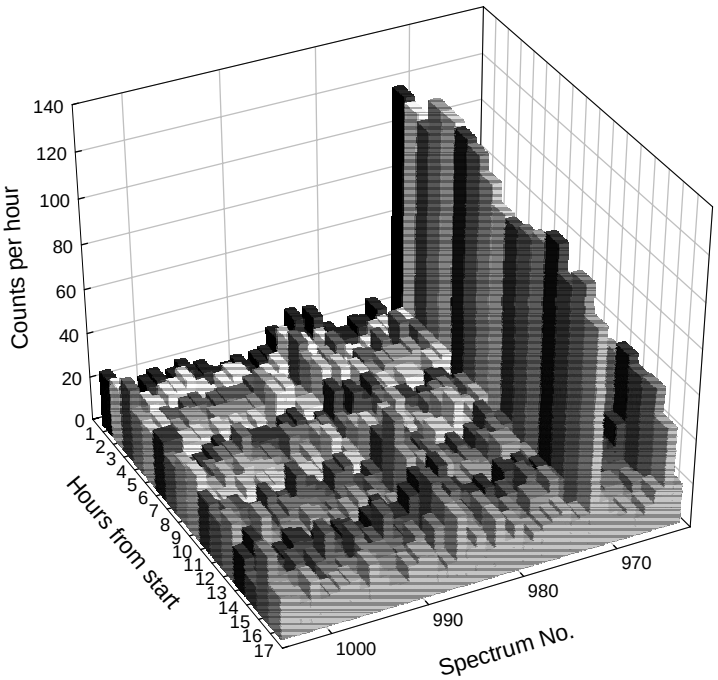


Figure 14. *Operational 'quiet counts'. Data collected after cryomagnet tests had finished and DEGAUSS.COM had been run. ISIS running at MS but OSIRIS intermediate shutter closed. NOTE: prompt pulse counts add to overall increase in observed count rate per channel*

Appendix IV. OpenGenie procedures

1. Procedure ‘SNR’ (Signal to Noise Ratio) integrates an OSIRIS I(Q,ω) spectrum, read from an .IPG file, over two well-defined regions i.e. the elastic line and the background. The two values returned are divided to give a SNR value for the spectrum.

```
PROCEDURE snr

GLOBAL run frunno lrunno stringno s1 w1 max bck
GLOBAL a i w f l h num back peak peakint ratio diff value output index

frunno = inquire("enter first run number")
lrunno = inquire("enter last run number")

diff=lrunno-frunno
value=dimensions(diff)
back=dimensions(diff)
peak=dimensions(diff)
peakint=dimensions(diff)
i=0

LOOP run FROM frunno TO lrunno STEP 1

i=i+1

# first read in new file

stringno=as_string(run)
s1="scratch$disk:[osiris.mt]osi"+stringno+".ipg"
printn s1
w1=get(1,s1)
d w1

# next first find x position of max(y) and shift spectra so x=0

a=max(w1.y)
printn a
l=bracket(w1.x,max(w1.x))
printn l

LOOP i FROM 1 TO l-1 STEP 1
num=w1.y[i]
IF num = a ; index=i; ENDIF
ENDLOOP

printn w1.x[index] w1.y[index]

# now modify x value so that max(y) falls at x=0

w1.x=w1.x-w1.x[index]
d w1

# now integrate elastic line and background to determine ratio

max=integrate(w1,-0.005,0.005)
bck=integrate(w1,-0.40,-0.39)
ratio=max.sum/bck.sum
printn ratio "_" max.sum "_" bck.sum
value[i]=ratio
back[i]=bck.sum
```

```
peak[i]=max(w1.y)
peakint[i]=max.sum
ENDLOOP

h=asciifile:open:new("ratio.dat")

LOOP m FROM 1 TO diff STEP 1
run=frunno-1
run=run+m
output=value[m]
asciifile/writefree h _ _ run  output peak[m] back[m]
ENDLOOP

asciifile/close h

ENDPROCEDURE
```

2. Procedure ‘HEDET’ takes an OSIRIS raw file and integrates quiet count data in s963 to s1004. The 42 integral values returned are then written to a file ('run_number'.int). The procedure also keeps a running total of the sum of integrated counts in each individual spectrum. This running total is written to 'total.int'. The idea is that if all 3He tubes are operating free of intermittent noise then the spectrum observed when total.int is plotted will show a constant level.

```
PROCEDURE hedet

GLOBAL firstrunno lastrunno increment
GLOBAL w1 r i s1 stringno a inc m s2 openfile

firstrunno = inquire("enter first run number")
lastrunno = inquire("enter last run number")

r=dimensions(42)
fill r 963 1

LOOP i FROM firstrunno TO lastrunno

assign i

w1=integrate(s(963:1004))
printn w1
stringno=as_string(i)
s1=stringno+".int"
a=asciifile:open:new(s1)
r=r*1.0
asciifile/writefree a " " 1 r w1.sum w1.error
#total.sum=total.sum+w1.sum
#total.error=total.error+w1.error
asciifile/close a

ENDLOOP

#a=asciifile:open:new("total.int")
#asciifile/writefree a " " 1 r total.sum total.error
#asciifile/close a

ENDPROCEDURE
```