Sample environment issues relevant to the acquisition of inelastic neutron scattering measurements of heterogeneous catalyst samples

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Abstract. Refinements to established sample handling issues connected with the acquisition of inelastic neutron scattering (INS) spectra of heterogeneous catalyst samples are described. This involves modification of a reactor and improvements to gas handling apparatus. The latter includes provision of laboratory space and ventilation facilities that are suited to the increasingly wide range of catalytic samples being examined. INS measurements for CO hydrogenation over an iron-based Fischer-Tropsch catalyst recorded using the MARI spectrometer are presented to illustrate the applicability of the new arrangements.

1. Introduction
The technique of inelastic neutron scattering (INS) is a useful method for obtaining the vibrational spectrum of ‘black’ samples that might contain significant quantities of carbon and/or metal, as these materials present significant challenges when conventional optical techniques, such as infrared (IR) spectroscopy, are used [1]. Further, materials such as metal oxides may also exhibit restricted spectral range in IR measurements due to strong absorption by the lattice modes and their overtones and combinations. For example, IR measurements of alumina are typically restricted to the range 1100 – 4000 cm⁻¹ [2], whereas this range may be extended to 50-4000 cm⁻¹ when INS is used [3]. Against this background, the recent realisation that direct geometry neutron spectrometers, such as MARI and MAPS, provide some advantages in terms of sensitivity, spectral range, and resolution with respect to indirect geometry spectrometers, such as TOSCA, is enabling INS to be applied to a wider range of technically relevant samples than was previously the case [4]. One such class of sample is heterogeneous catalysts.

For many years IR spectroscopy has been used to examine heterogeneous catalysts and chemisorbed systems [5], as it provides a combination of high sensitivity and excellent spectral resolution. Moreover, modern commercial IR spectrometers may be coupled to conventional catalyst test apparatus, facilitating a platform for operand spectroscopy [6]. INS spectroscopy supplements that valuable dataset by being able to examine technical grade catalysts post-reaction, something that, for the reasons mentioned above, is not so easily achievable with optical techniques. However, INS is relatively insensitive; therefore, for studies concentrating on the surface chemistry of catalysts or on the form of chemisorbed species, sample masses typically ≥10 g are required, as opposed to the 20-50 mg typically used in IR measurements [7]. This restriction forces INS measurements away from the...
micro-reactor configuration commonly encountered in laboratory catalyst testing and requires a significant degree of scale-up for INS sample handling apparatus. Presently, the majority of INS studies on catalysts have tended to feature chemistry at the gas/solid interface [1].

A recent collaboration between the universities of Glasgow and Keele and the ISIS Facility of the STFC Rutherford Appleton Laboratory has used INS to investigate a series of alumina-supported nickel catalysts active for the reforming of methane in to synthesis gas (CO + H$_2$). Initial studies using CO$_2$ as the oxidant, i.e. ‘dry’ reforming, utilised an indirect geometry neutron spectrometer (TOSCA) and produced weak INS spectra; demonstrating that these catalysts are very efficient at cycling hydrogen [8]. Subsequent studies were performed on a direct geometry chopper instrument (MAPS), where improved sensitivity yielded better-defined spectra [8-10]. The investigations were extended to examine the more industrially relevant steam reforming reaction of methane; with that work providing further challenges in relation to sample handling capability [11]. These studies forced the development and commissioning of a new catalyst handling apparatus that was constructed at ISIS and is described elsewhere [12]. The adoption of such sample handling procedures is an essential part of being able to successfully apply INS to probe the surface chemistry of high surface area catalytic systems.

Whilst the INS studies of the nickel based catalysts provided new insight into how hydrogen partitions within the catalyst matrix for the economically relevant methane reforming reactions, it also exposed vulnerabilities in the apparatus used to prepare the samples. Namely, the equipment was quite bulky and, consequently, in a busy Central Facility with high demand for laboratory space, it had to be assembled for each experimental run and then disassembled and stored afterwards. The equipment worked well but, when assembled, it took up two fume cupboards in addition to a section of laboratory floor space required to accommodate the various gas cylinders connected with each experiment. With an increasing number of applications for neutron beam time to study catalytic systems at ISIS, this arrangement became untenable. To overcome these issues, a more compact and more permanent arrangement was proposed. This communication describes the resulting improvements in availability of hardware and outlines the associated methodology. Firstly, an improved gas handling system and associated laboratory infrastructure is outlined. Secondly, a new stainless steel reactor is described that is designed to undertake reactions under more modest conditions of temperature and pressure than required in the aforementioned experiments on nickel based methane reforming catalysts. Further, in order to demonstrate the usefulness of this upgrade in capability, the acquisition of INS spectra for CO hydrogenation over an iron-based Fisher-Tropsch (F-T) catalyst is presented.

### 2. Experimental

#### 2.1 Gas handling facility

Figure 1 shows a schematic drawing of the new gas handling apparatus. In concept, the layout is similar to that successfully adopted in the initial layout [12]; however, the design has been modified to significantly improve the compactness of the whole apparatus, so that the gas handling section can all be accommodated on a single panel. The panel is portable but resides within a walk-in fume cupboard (Kotterman, type 2-451-JAND, average duct velocity = 0.54 ms$^{-1}$) within a sample preparation area off the main experimental hall of the ISIS Facility. A gas cylinder compound has been constructed outside the laboratory and four gas lines (presently configured for helium, carbon monoxide, hydrogen and carbon dioxide) are piped in to the fume cupboard via individual regulators and isolation valves. Components are connected using 1/4 in. o.d. stainless steel tubing and Swagelok tube fittings. The laboratory is fitted with a gas detection system (Gas Measurements Instruments Ltd., Active-8 alarm system) in order to warn the operator of any release of hazardous gases. Specifically, two CO and two hydrogen gas detectors are located alongside the gas line regulators and adjacent to the fume cupboard. A manually activated electrical switch mounted on the side of the fume cupboard can be used to trigger in-line isolation valves mounted above the gas cylinder compound to immediately
isolate the gas supply if required. This combination of features allows higher gas flow rates to be permissible than was previously the case with the temporary arrangement when the gas cylinders were contained within the laboratory. Higher permissible gas flow rates mean that reactions can be studied at gas hourly space velocities and residence times that now more closely resemble values accessible in micro-reactor studies. Gas flow to the reactor is controlled by four Hastings model HFC302 mass flow controllers (MFC) attached to a Teledyne THPS-400 controller. These controllers have been calibrated for nitrogen flow but the use of gas correction factors allows other gases to be accurately metered. One controller has a maximum flow rate of 2000 standard cubic centimeters per minute (sccm) whilst the other three are specified at 200 sccm (all N₂).

Figure 1. A schematic representation of the gas handling apparatus. The mass flow controllers are indicated with the rectangular boxes with the maximum flow rate (sccm) contained within. Other abbreviations include; P – pressure sensor, R – regulator, PRV – pressure relief valve, MV – mixing volume, BPR – back pressure regulator and MS – mass spectrometer. The dashed box schematically represents the enclosed fume cupboard.

A saturator is accessible via the inlet line, which means that reactions involving vapourised reagents, e.g. steam [11] or methanol [2], can be passed over the catalyst sample. Heated lines connect the gas manifold to the reactor, which is housed within a bucket furnace (Instron SPL, model no: TF105/3/12/F) that is controlled by a PID module (Eurotherm 3508). The furnace is mounted on a heavy duty lab-jack, so that the furnace can be raised to enclose the reactor during reaction and lowered to facilitate disconnection when the cell requires demounting from the gas line. The pressure of the gas line is controlled by a back pressure regulator (Parker Veriflo ABP-1ST-43-PP-X4), with check relief valves placed at several points within the gas manifold. The apparatus is variably rated
(see later) up to a maximum operating pressure of 20 bar. Pressure gauges (Druck DPI 280 Series) are located upstream and downstream of the reactor. Pressure readings from these gauges provide information on the presence of any pressure drop in the reactor zone. The reactor exit line can be passed through a ‘catch-pot’, which will extract condensable products if required. The reactor exit gases are then analysed by mass spectrometry. Initially, this involved a removable quadrupole mass spectrometer (MKS Spectra Microvision Plus) that sampled the eluting gas stream via a fine control valve. That arrangement has now been replaced with a permanently dedicated mass spectrometer (Hiden Analytical, HPR20 QMS Sampling System) that samples the gas stream via a differentially pumped continuous leak. The new arrangement simplifies calibration of the product and reaction streams. The exit lines are all vented within the fume cupboard.

Figure 2 presents a photograph of the gas panel mounted within the fume cupboard. The MFC control unit is located at the front of the blue gas panel. A two-section glass sash at the front of the fume cupboard means that the mass flow controllers can be adjusted by the operator without compromising the negative pressure within the fume cupboard. The furnace is visible in Figure 2 to the left hand side of the gas panel, with the reactor visible above the furnace. The heated lines to and from the reactor are terminated by high temperature valves (Parker HNV54A3) that are capable of operating at temperatures up to 811 K.

Figure 2. A photograph of the gas handling apparatus (blue panel), that is housed within a walk-in fume cupboard. The furnace is located to the left hand side of the gas panel and is mounted on a heavy-duty lab-jack. Above the furnace the reactor can be seen, which is connected to the gas panel by heating lines that terminate in high temperature valves, which may be used to isolate the cell. During operation, the furnace is raised so as to enclose the reaction cell.

2.2 Reactor
The Inconel reactor described previously is capable of operation at temperatures up to 873 K and pressures up to 20 bar [12]. It has been used adopting a ‘quench’ format [13-14] where, post-reaction, the reactor is isolated, transferred to the neutron spectrometer and the spectrum acquired. The
specification of the reactor enables high temperature reactions such as methane reforming to be studied in this way [10-11]. However, the Inconel body of the reactor has a vibrational signature that occludes a significant part of the low frequency region of the spectrum [12]; an attribute that constrains the use of the Inconel reactor for spectral acquisition. Furthermore, its significant mass means that it takes long periods of time to cool from reaction temperature to ambient temperature for disconnection from the gas line, and then subsequent cooling to the cryogenic temperatures, ca. 20-30 K, typically used for the measurement of the INS spectrum. To address these matters, a new reactor has been constructed from stainless steel. Figure 3 presents a photograph of this reactor, which is intended for lower temperature reactions (≤ 673 K) than obtainable with the Inconel reactor. As with the earlier reactor design [12], the flanges at the bottom and top of the cell are based on Conflat fittings sealed by copper gaskets but now the flanges have been reduced from a thickness of 12 mm to 6 mm, which reduces mass and enables the reactor to be cooled quicker. The lighter reactor makes gravimetric measurements easier and, if desired, it is simpler to mount on a conventional INS sample ‘stick’ that positions the sample in the cryostat during the INS measurements [15]. The reduction of flange width restricts the operational pressure range of the cell to a maximum operating pressure of 5 bar. Thus, the new reactor is intended for modest temperature and pressure reactions, such as hydrogenations, rather than the more demanding conditions such as encountered with methane reforming. Different check relief valves are fitted to the gas lines depending on the type of reactor being used (Inconel reactor: 20 bar; stainless steel reactor: 5 bar).

Figure 3. Photograph of the stainless steel reaction cell for INS measurements.
Figure 4. INS spectra of empty Inconel (red), stainless steel (blue) and aluminium (olive) cells recorded on MAPS with incident energies of: (a) 5050 cm$^{-1}$ (626 meV) at 600 Hz, (b) 2016 cm$^{-1}$ (250 meV) at 400 Hz and (c) 484 cm$^{-1}$ (60 meV) at 200 Hz. The right-hand part of each figure shows an ordinate expanded portion of the spectrum: (a) ×100, (b) ×100 and (c) ×30. All data were recorded for 150 μA hr (approx. one hour measurement) using the ‘S’ Fermi chopper package at the rotation speed given. The different rotation rates largely account for the difference in elastic line intensity between (a), (b) and (c).
In order to compare the relative contributions to the INS spectrum of the two types of reactor, the spectra of an empty Inconel reactor and a stainless steel reactor were recorded at three different incident energies (5050, 2016 and 484 cm\(^{-1}\)) on the MAPS spectrometer using the S chopper package; the results are presented in Figure 3. Although the stainless steel reactor does present a reduced signal over regions of the spectrum, this difference is minimal. Previous work on the Inconel reactor using the MERLIN spectrometer did demonstrate that, despite a significant background contribution, the reactor was amenable to background subtraction strategies that enabled the INS spectrum of small populations of chemisorbed entities present at a catalyst surface to be obtained [12]. However, from inspection of Figure 3, it is evident that neither reactor is particularly well suited to spectral acquisition purposes. This was not the anticipated outcome envisaged when the stainless steel reactor was commissioned.

To maximize the scattering intensity of surface species present at catalyst surfaces an alternative approach was adopted. Namely, following a procedure used in earlier catalyst studies that utilized a quartz tubular reactor [8-9, 16], the catalyst sample post-reaction was transferred to a conventional aluminum INS sample cell [9] and the INS spectrum recorded. The INS spectrum of an empty aluminum sample holder is also presented in Figure 4 (olive line), where a dramatic contrast to that observed for the denser metal cells is apparent: the aluminum cell exhibits a flat background from 4000 cm\(^{-1}\) down to the tail of the elastic peak. So, although aluminum is an unsuitable material for construction of a catalytic reactor due to its relatively low melting point and possible reactivity at elevated temperatures, it is an excellent choice of material for use as an INS sample holder. This scenario reinforces the idea of a separate reactor and INS measurement cell that necessitates the provision of a high specification glove box to facilitate the transfer of sample between the two containers. Future studies will examine the possibility of avoiding the sample transfer from reactor to a separate INS cell. Nevertheless, at present, for the reaction systems examined here, INS inspection of heterogeneously catalysed reactions constitutes a form of \textit{ex situ} analysis.

The fixed bed arrangement of these reactors can potentially lead to problems with inhomogeneous reaction profiles existing across the length of the reactor bed. For instance, it is possible that a discrete reaction front could slowly progress along the length of the bed (65 mm, Figure 3), so that catalyst at the top of the reactor may experience different chemistry to catalytic material located at the bottom of the bed. One way around this issue is to disperse the catalyst with diluent, such as silicone carbide or alumina, which can also reduce the presence of ‘hot spots’ in the reactor. However, due to the insensitivity of the INS technique, invariably all of the catalyst charge is used in the INS measurements and, hence, the INS spectra represent an average of all of the material within the reactor. If greater sensitivity could be attained, then it would be possible to use INS to analyse different fractions of the bed and, hence, to build up a composite description of the catalyst throughout the whole length of the bed. Within the constraints of the specifications of the present suite of INS spectrometers at ISIS [4], that desirable option is not available at present. (We note in passing that upgrades to MAPS and TOSCA currently in progress, coupled with the proposed ISIS TS1 upgrade, may make this option feasible in the near future.) Reactions that inherently exhibit low conversions are less prone to these problems. For example, XRD and Raman analysis of an iron Fischer-Tropsch catalyst that had experienced 6 h reaction with syngas at 623 K and atmospheric pressure, with samples taken from different regions of a tubular reactor, showed identical diffraction patterns and Raman spectra; indicating a uniformity of catalyst composition and form throughout the whole length of the reactor [16]. In that case, CO conversion was low (ca. 1%). In cases where high feedstock conversions are evident, operators are encouraged to be aware of possible problems with sample inhomogeneity. One way round these difficulties is to run the reactions at reduced temperatures but for longer times. However, the relevance of the operating conditions that favour lower conversions need evaluating in separate micro-reactor experiments. Presently, the analysis of heterogeneous catalyst samples remains a non-trivial process.
3. Results and discussion

3.1 Reaction testing: CO hydrogenation over an iron-based Fischer-Tropsch catalyst

To illustrate the effectiveness of the new apparatus, a preliminary analysis of an iron-based Fischer-Tropsch catalyst that is active for the hydrogenation of CO is presented in this communication. Iron-based Fischer-Tropsch catalysts typically originate as hematite (α-Fe₂O₃) but under steady state reaction conditions the nature and form of the catalyst is changed so that the catalyst consists of a combination of magnetite (Fe₃O₄), iron carbides, and amorphous carbon [17-19]. Recent work from this group has also proposed a role for a hydrocarbonaceous overlayer [16, 20].

A sample of α-Fe₂O₃ pre-catalyst was prepared using the precipitation technique in a similar manner to that described previously [16]. However, on this occasion, a Mettler-Toledo chemical preparation system (LabMax batch reactor) was used to ensure careful control of pH during the precipitation process. The resulting α-Fe₂O₃ exhibited a BET surface area of 70.8 m² g⁻¹, which exceeds the value of a previously reported catalyst [16]; with the higher surface area thought to reflect a more controlled precipitation in this case. The catalyst was ground and sieved to a size fraction of 250 - 500 µm. Given the fixed bed reactor adopted for these experiments, it is important to avoid the presence of fine catalyst particles which could aggregate on reaction, ‘plug’ the reactor and give rise to a significant pressure drop across the catalyst bed. Two samples were prepared for INS analysis; a sample exposed to dehydration conditions to serve as a background and subsequently reacted under the CO hydrogenation reaction conditions. This procedure is intended to provide information on how the surface of the catalyst may have changed as a consequence of a test reaction (CO hydrogenation at ambient pressure and elevated temperature) that produces gaseous products without inducing actual Fischer-Tropsch activity [16]. The latter option is avoided as it would generate high molecular weight products, which would complicate analysis of the INS spectrum. CO hydrogenation over iron based catalysts has previously been shown to provide useful information on aspects of Fe/CO/H₂ surface chemistry that is inherently relevant to F-T catalysis [16, 20-21].

The background sample was prepared as follows. A stainless steel reactor was charged with 17.2 g of the α-Fe₂O₃ pre-catalyst, which was located between two sections of quartz wool. This mass of catalyst is smaller than used on previous occasions [16] but is sufficient to fill an aluminium INS cell and provide a spectrum with reasonable signal-to-noise ratio with ca. 8 h measurement time on the MAPS spectrometer. Further, the lower mass of catalyst coupled with the superior gas handling facilities described in Section 2.1 means that weight hourly space velocities closer to those encountered in a micro-reactor are possible. The catalyst was purged by passing helium at a flow rate of 1000 ml min⁻¹ whilst heating to 373 K at a rate of 5 K min⁻¹, then maintaining that temperature for 12 h. This procedure desorbs any water that may be absorbed within the pore structure of the sample. After this treatment, the heating was stopped, the cell isolated and transferred to an argon-filled glove box (MBraun UniLab MB-20-G, [H₂O] < 1 ppm, [O₂] < 2 ppm), where the dried catalyst was transferred to a standard aluminium INS cell [15], which was sealed via an indium wire gasket. The sample could then be transported to the INS spectrometer.

The α-Fe₂O₃ sample selected for CO hydrogenation was exposed to a feedstream comprising CO and H₂ in a 1:2 ratio (CO flow rate = 75 ml min⁻¹, H₂ flow rate = 150 ml min⁻¹) in the presence of a helium diluent (600 ml min⁻¹) at ambient pressure as the temperature was ramped up to 623 K at 5 K min⁻¹ then held at 623 K for 6 h. These conditions have previously been shown to support CO hydrogenation [16]. Figure 5 shows the (uncalibrated) mass spectrometer response as a function of time-on-stream with respect to the temperature programme. The profile for reagents and products broadly follows that recently reported for a comparably specified iron-based F-T catalyst [16]. An
initial consumption of CO at ca. 600 K is associated with the conversion of hematite ($\alpha$-Fe$_2$O$_3$) to magnetite (Fe$_3$O$_4$). A second more gradual consumption of CO is seen once the operating temperature of 623 K is reached, which then progressively diminishes and approaches a steady state value at about 7 h. Relatively minor inflections in the dihydrogen signal up to $t = 1.5$ h indicate that hematite reduction is primarily facilitated using CO rather than H$_2$ as the reductant. From $t = 1.5$ h onwards a small consumption of dihydrogen in the feedstream is connected with the CO profile over this period and is associated with methane formation. A gradual decline in methane production is associated with a slow deactivation channel.

![Figure 5](image)

**Figure 5** Mass spectrometer response (uncalibrated) defining the reaction profile for the CO hydrogenation reaction at 623 K over the $\alpha$-Fe$_2$O$_3$ sample which is located in the INS sample cell (Figure 3). The mass traces are labelled on the right hand side of the profile in descending order. T defines the temperature profile over the seven hour period.

The production of CO$_2$ and H$_2$O during the temperature ramp could be the result of the stepwise reduction of the $\alpha$-Fe$_2$O$_3$ starting material towards Fe$^0$ via Fe$_3$O$_4$ and FeO by CO and H$_2$ resulting in the evolution of CO$_2$ and H$_2$O respectively [22]. Several contributing pathways could be responsible for the second CO$_2$ feature at ca. 623 K: CO disproportionation via the Boudouard reaction [23] and/or CO oxidation with surface oxygen. The increase in H$_2$O signal at 623 K could be attributed to the hydrogenation of surface oxygen after CO dissociation. The water gas shift reaction [24] is also thought to be active in consuming CO at 623 K to form CO$_2$ and H$_2$. A more comprehensive discussion of reaction profiles associated with CO hydrogenation over iron based F-T catalysts is available elsewhere [16].

After 6 h on stream, the reaction was quenched by terminating the syngas flow and allowing the sample to cool to room temperature in flowing helium. As with the background sample, the cell was isolated and placed within the argon-filled glove box. The reacted catalyst sample was transferred to a standard aluminium INS cell and then transported to the INS spectrometer for spectral acquisition.
3.2 Inelastic neutron scattering

The INS spectra of the dehydrated and reacted samples were recorded using the MARI spectrometer, which is located at the ISIS Facility of the STFC Rutherford Appleton Laboratory. A description of the operational characteristics of the MARI spectrometer is presented elsewhere [2,25]. Spectra were recorded at 20 K and at an incident neutron energy of 4840 cm\(^{-1}\) using the A-chopper package. An acquisition period of 8 h was used for both the background and reacted spectra.

The spectrum (2000-4000 cm\(^{-1}\)) of the dehydrated \(\alpha\)-Fe\(_2\)O\(_3\) before reaction is presented in Figure 6(a), whereas the catalyst post-reaction is presented in Figure 6(b). The unreacted \(\alpha\)-Fe\(_2\)O\(_3\) spectrum exhibits no features in the 2000-4000 cm\(^{-1}\) region; in direct contrast to a recently reported INS spectrum of an iron based F-T catalyst that was prepared in-house, where a distinct band at 3442 cm\(^{-1}\), assigned to the O-H stretch of hydroxyl groups present as surface terminations of the hematite precursor compound, is observed [16]. The absence of a \(\nu\)(O-H) band in Figure 6(a) indicates that the \(\alpha\)-Fe\(_2\)O\(_3\) examined here differs from the previously studied in-house catalyst; possibly as a consequence of the tighter control of the precipitation stage used in the catalyst preparative part of this work that has prevented generation of hydroxyl groups at the catalyst surface. Often hydroxyl groups are present at metal oxide surfaces as a means of minimizing the chemical potential in the surface region, as the bulk metal oxide structure terminates. Direct geometry INS spectrometers are particularly good at characterizing hydroxyl groups associated with high surface area metal oxides [7] and, as hydroxyl groups can play a major role in influencing surface chemical reactions [7], this is an area where INS is expected to find increasing application.

The INS spectrum of the reacted catalyst (Figure 6(b)) is characterized by two distinct features. Firstly, a sharp band is observed at 3061 cm\(^{-1}\), which is assigned to a C-H stretch of olefinic or aromatic C-H bonds (sp\(^2\) hybridised C atoms) present at the catalyst surface. Secondly, a low frequency shoulder is evident on the main peak at 2942 cm\(^{-1}\), which is assigned to an aliphatic \(\nu\)(C-H) mode (sp\(^3\) hybridised C atoms). Thus, Figure 6(b) indicates a population of predominantly sp\(^2\)-C–H species has formed at the catalyst surface upon reaction, which is supplemented by a lower density of aliphatic C-H species. Following recent INS investigations of iron based F-T catalysts [16, 20], Figure 6 is interpreted as indicating the presence of a hydrocarbonaceous overlayer. Traditionally, iron based F-T catalysts are typically analysed by techniques such as X-ray diffraction, Mössbauer spectroscopy, electron microscopy, etc. and, consequently, described in terms of solid-state chemistry (hematite, magnetite, iron carbides, etc.) [17-19], whilst techniques such as temperature-programmed oxidation and Raman scattering highlight the relevance of carbonaceous materials [26-27]. This new application of INS offers the exciting potential of being able to determine how hydrogen atoms are being partitioned within the F-T catalyst matrix. In order to better understand the surface chemistry connected with the lucrative associated process chemistry, a role for hydrogenous species needs to be more precisely defined. This combination of INS and an analysis of CO hydrogenation reactions over iron based F-T catalysts, representative of industrial grade catalysts [16, 20] is providing new knowledge that complements information accessible via the more traditional analytical methods.
4. Conclusions
Recent improvements to sampling environment methodologies connected with the acquisition of INS spectra of heterogeneous catalysis samples at the ISIS Facility are described. This includes the re-configuration and up-grading of a sample preparation laboratory, the commissioning of a new gas handling apparatus and the construction of new reactors. Together, these facilities are able to support a broad programme of investigations of heterogeneous catalysts featuring chemistry at the gas/solid interface. As an illustration of the type of work presently utilizing these enhanced sample-handling facilities, the INS spectrum of an iron based F-T catalyst is presented before and after sustained CO hydrogenation at 623 K. The resulting spectra, recorded using the MARI spectrometer, provide further evidence for a role for a hydrocarbonaceous overlayer in F-T related chemistry.

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