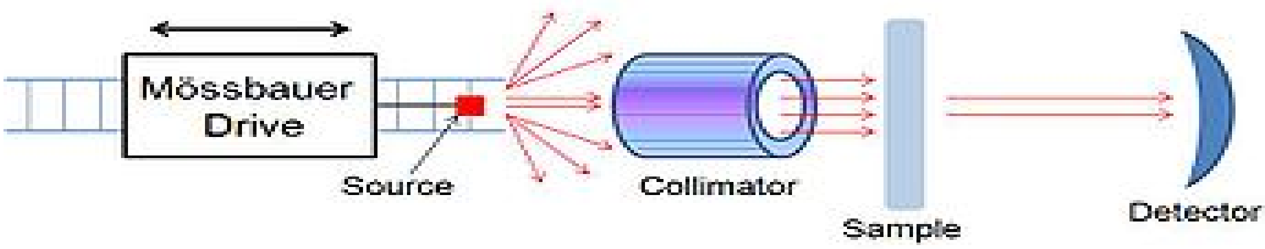
MOSSBAUER SPECTROSCOPY IN CATALYSIS



MöSSBAUER SPECTROMETER

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MOSSBAUER SPECTROMETER

MOSSBAUER SPECTROSCOPY IN CATALYSIS

INTRODUCTION:

Mossbauer spectroscopy is one of the techniques that is relatively little used in catalysis. Nevertheless, it has yielded very useful information on a number of important catalysts, such as the iron catalyst for Fischer-Tropsch and ammonia synthesis, and the cobalt molybdenum catalyst for hydrodesulfurization reactions. The technique is limited to those elements that exhibit the Mossbauer Effect. Iron, tin, iridium, ruthenium, antimony, platinum and gold are the ones relevant for catalysis. Through the Mossbauer Effect in iron, one can also obtain information on the state of cobalt. Mossbauer spectroscopy provides valuable information on oxidation states, magnetic fields, lattice symmetry and lattice vibrations. Several books on Mossbauer spectroscopy and reviews on the application of the technique on catalysts are available.

Mossbauer spectroscopy is a nuclear technique. The nuclear technique is useful for the study of catalysts because the nucleus, being at the heart of the atom, feels precisely what the state of the atom is. Mossbauer spectroscopy analyzes the energy levels of the nucleus with extremely high accuracy and in this way it reveals for example what the oxidation state of the atom is, or how large the magnetic field is at the nucleus. In this way one determines straightforwardly what the compound is to which the atom belongs. The great advantage of Mossbauer spectroscopy for catalyst research is that it uses γ-radiation of high penetrating power such that the technique can be applied `in-`situ. An economic advantage is that the technique is relatively incxpensivc. The price is about a factor of ten less than equipment for electron microscopy or photoelectron spectroscopy. The Mossbauer Effect can only be observed in the solid state and in a limited number of elements. The so called hyperfine interactions between the nucleus and its environment, makes the technique so informative.

THE MOSSBAUER EFFECT:

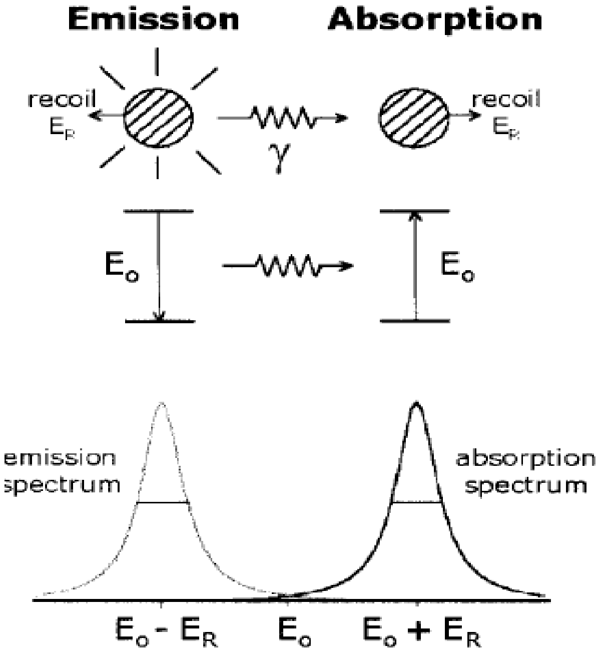


Figure 5.1: Resonant absorption of γ-radiation by a nucleus can only take place in the solid state because of recoil effects. The excited nucleus of a free atom emits a γ-photon with an energy E_0-E_R, whereas the nucleus in the ground state of a free atom can only absorb a photon if it has an energy equal to E_0+E_R. As the linewidth of nuclear transitions is extremely narrow, the emission spectrum does not overlap with the absorption spectrum. In a solid, a considerable fraction of events occurs recoil free (E_R=0), and here the emission spectrum overlaps completely with the absorption spectrum (provided sourcc and absorber have the same chemical environment).

Let us consider the following experiment (fig 5.1).We have two identical atoms, one with a nucleus in the excited state and the other in the ground state. The excited nucleus decays to the ground state by emitting a photon with energy of typically some 10-100 keV. This photon falls on the nucleus of the second atom, which is in the ground state. The question is, whether the second nucleus absorb the photon to get into the excited state? The answer is no, because recoil energy is involved. The experiment is analyzed in terms of an energy balance. Suppose the energy difference between the excited state and the ground state of the nucleus is Eo. When the excited nucleus decays and emits a photon, it will recoil, just as a gun from which a bullet is fired. This recoil energy, E_R, can easily be calculated from mechanics, and equals $E_0/2mc^2$ where **m** is the mass of the nucleus and c the velocity of light. Thus the energy of the emitted photon, $E\gamma$ equals (E_o–E_R). Similarly, if the photon hits the second nucleus, it will recoil also with an energy E_R, and the energy available to excite the nucleus is only E_0-$2E_R$, whereas an energy equal to Eo would be needed. For illustration, if the nuclei are those of iron, E_O = 14.4 keV and E_R is on the order of a few meV. The natural line width of the transition, however, is much smaller, in the case of iron as small as $4.6*10^{-9}$eV. Hence, the experiment to absorb the emitted γ-photon fails, due to recoil effects. In a solid lattice, the atom cannot recoil as if it were free. Here the recoil energy is taken up by vibrations of the lattice as a whole. Lattice vibrations are quantized, just as the vibrations or rotations of a molecule are; the quantum is called a phonon. If the recoil energy due to emission of the γ-quantum is larger than the phonon energy, the lattice will simply take up the recoil energy in portions equal to the phonon energy and the Mossbauer experiment fails again. If, and this is the situation of interest, the recoil energy is smaller than the energy of the lattice a vibration, a situation arises which can only occur in quantum mechanics: **A** number of emission or absorption events takes place without exchange of recoil energy. The fraction of recoil-free events follows from the correspondence principle, which relates quantum mechanics to everyday life: The average value of the recoil energy over a large number of events must be equal to E_R. The important thing to realize is that the recoil energy is taken up in portions which are larger than E_R, and thus there must also be emission and absorption events for which the recoil energy is zero. The conclusion of all this is the following. If we place the two atoms in a lattice and do the experiment under conditions where recoil energy of the photon emission and absorption are significantly smaller than the energy of the lattice vibrations, a fraction of the photons emitted by the source nucleus will be absorbed by the nucleus in the absorber. This is the Mossbauer Effect, named after Rudolf L. Mossbauer, who discovered it in 1957 and received the Nobel Prize in 1961.The intensity of the

Mossbauer Effect is determined by the recoil-free fraction, or f-factor, which can be considered as a kind of efficiency. It is determined by the lattice vibrations of the solid to which the nucleus belongs, the mass of the nucleus and the photon energy, ***E,*** and is given by:

$$f = e^{-k_\gamma^2 <x^2>}$$

which, if we express $<x^2>$ in the Debye model, becomes:

$$f = \exp\left[-\frac{3}{2}\frac{E_R}{k\theta_D}\left(1 + 4\frac{T^2}{\theta_D^2}\right)\int_0^{\theta_D/T} \frac{x\mathrm{d}x}{e^x - 1}\right] \qquad \textbf{(5-2)}$$

in which

f	is the recoil-free fraction
k_γ	is the wave number of the γ-radiation, equal to $2\pi/\lambda$
$<x^2>$	is the mean squared displacement of atoms from their average position due to lattice vibrations
E_R	is the recoil energy of the nucleus upon emission of a γ-quantum
k	is Boltzmann's constant
θ_D	is the Debye temperature; $k\theta_D$ corresponds to the maximum energy of the phonons
T	is the temperature.

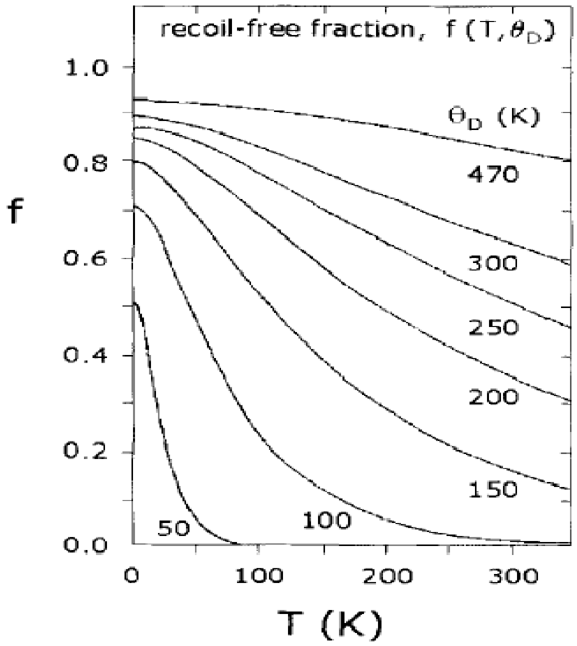


Figure 5.2: The recoil-free fraction, f, of iron as a function of temperature for different values of the Debye temperature, θ_D. Bulk iron compounds have Debye temperatures on the order of 450–500 K; surface phases, however, have significantly lower Debye temperatures, implying that measurements may have to be carried out at lower temperatures

The above equation expresses the fact that the recoil-free fraction is larger for low energy (i.e. low k_γ) transitions as in Fe and Sn than for high energy transitions as in Ir, Pt and Au. Both expressions indicate that the recoilless fraction is higher for a rigid lattice ($<x^2>$ small, Θ_D large) than for a structure with soft vibrational modes ($<x^2>$large, Θ_D low). The above figure shows plots for the Mossbauer Effect in iron; the Debye temperature of bulk α-Fe is 470 K that of Fe_20_3 and Fe_30_4, is of the order of 500 K. Surface atoms are coordinatively unsaturated and have larger freedom to vibrate. As a result, the surface Debye temperature of well crystallized solids is generally between 1/3 and 2/3 of the bulk value. However, Debye temperatures as low as 50 K have been reported in passivation layers on top of small iron catalyst particles.

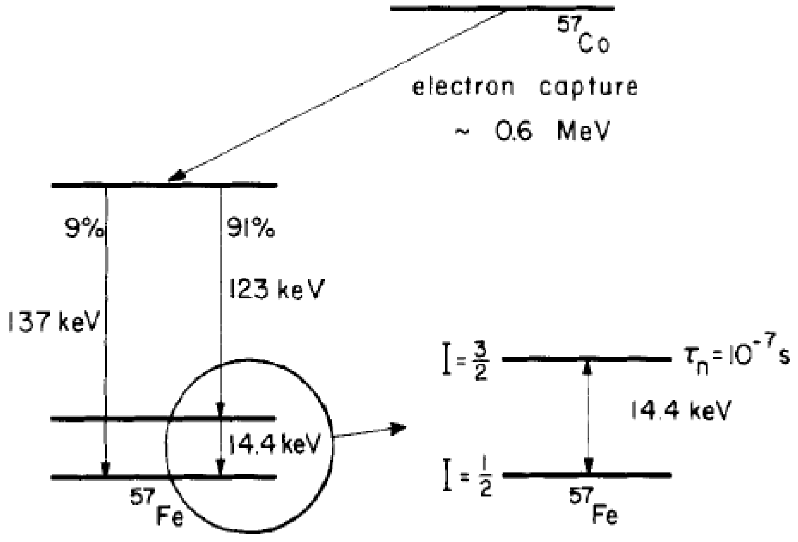


Figure 5.3: The decay of ^{57}Co to ^{57}Fe. The encircled part is the transition commonly used for Mössbauer spectroscopy of iron-containing samples.

There is a second condition that must be satisfied in order to observe the Mossbauer Effect: one needs nuclei in the excited state as a source of the γ-photons. Figure 5.3 shows how this is achieved in the case of the most frequently used nucleus for Mossbauer spectroscopy, the 57-Fe isotope. In this case, Co is used as a source. This isotope is made in a nuclear accelerator, and decays with a half life of 270 days to an excited level of 57-Fe, which in its turn decays rapidly (half life 10^{-8}s) to the excited level we want. The last step in the decay process generates the γ-quantum of approximately 14.4 keV used in Mossbauer spectroscopy. The parallel decay process that leads to the emission of a 137 keV photon can also be used, but the corresponding recoilless fractions are much smaller, as (equation 5-1) predicts. Thus a necessary condition for an observable Mossbauer Effect is that one has a source which decays to the excited state of the nucleus that is to be studied with a sufficiently long lifetime such that experiments are practical. The actual transition used for the Mossbauer Effect should follow instantaneously. Only a limited number of elements satisfy this condition. For catalysis, iron, tin, antimony, ruthenium, iridium and platinum are the most important.

MOSSBAUER SPECTROSCOPY:

Suppose that the atom of the absorbing nucleus has a different chemical environment from that of the emitting atom in the source. For example, the source is metallic iron and the absorber is an iron oxide. Because the nucleus is coupled to its environment through hyperfine interactions, the nuclear levels in the absorber have slightly different energies than those in the emitter. Again, the Mossbauer Effect will not be observed because the energy of the emitted γ-quantum does not match the energy difference between the levels in the absorber. Hence, we need to vary the energy of the photons. This can be done by using the Doppler Effect: if we move the emitter towards the absorber at a velocity `v,` the energy of the photon becomes:

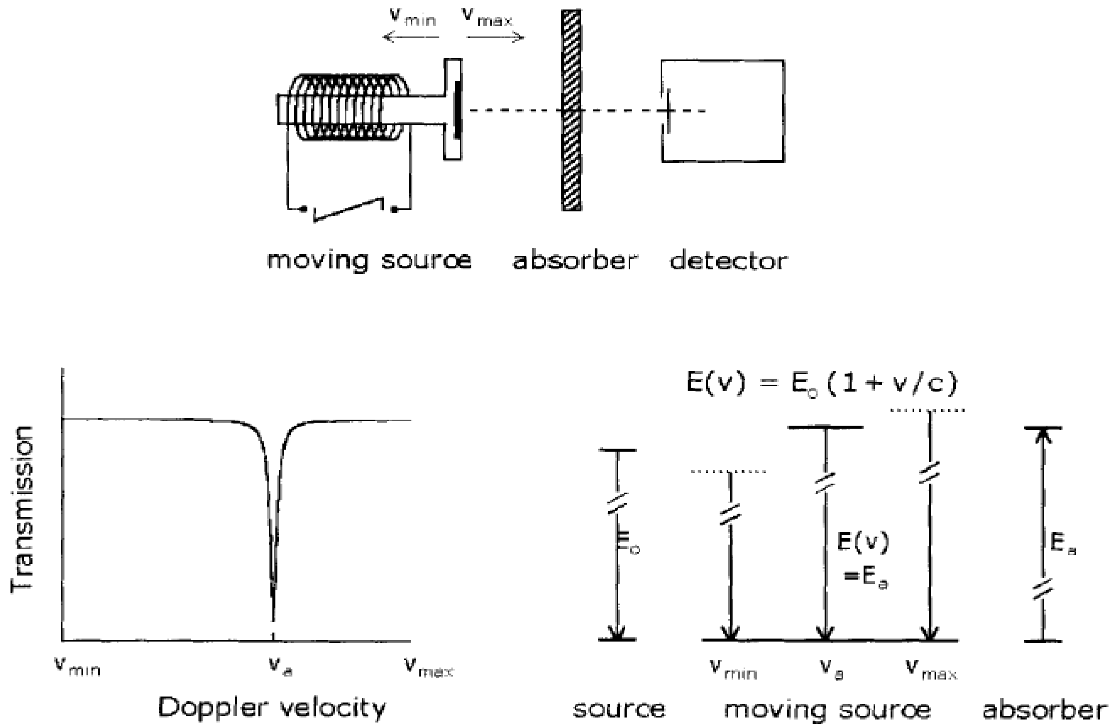


Figure 5.4: In order to cover all possible transitions in the absorbing nucleus, the energy of the source radiation is modulated by using the Doppler effect. For ^{57}Fe the required velocities fall in the range between −1 and +1 cm/s. In Mössbauer emission spectroscopy, the sample under investigation is the source, and a single line absorber is used to scan the emission spectrum.

$$E(v) = E_0\left(1 + \frac{v}{c}\right) \qquad \textbf{(5-3)}$$

in which

$E(v)$ is the energy of the γ-quantum emitted by the source

v is the velocity of the source

E_0 is the energy difference between the excited state and the ground state of the nucleus

c is the velocity of light.

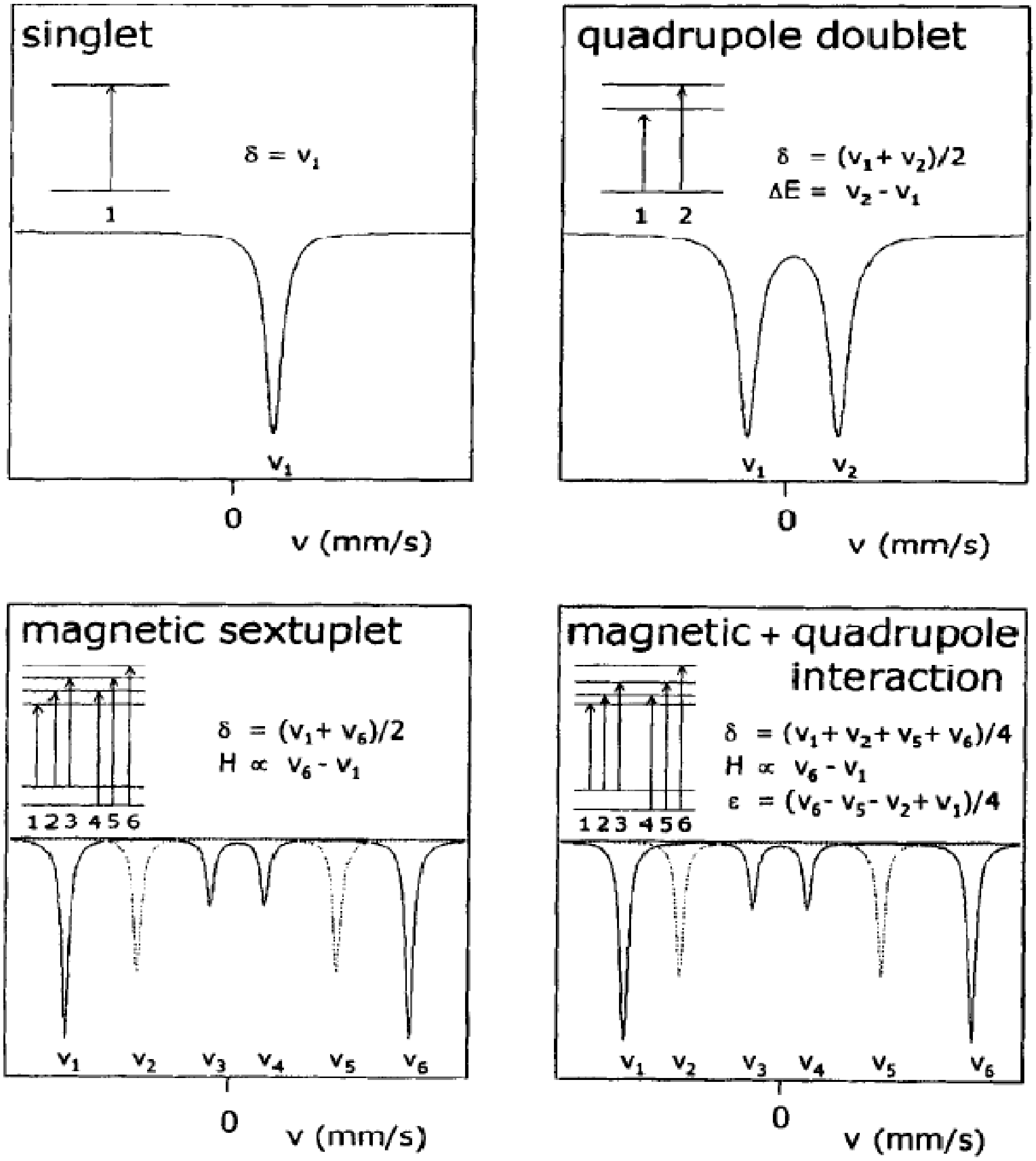


Figure 5.5: The four most common types of Mössbauer spectra observed in iron-containing catalysts along with the corresponding nuclear transitions. Also indicated is how the Mössbauer parameters are derived from the spectra.

We ignore higher order contributions such as $v^2/2c^2$ in the above equation. In order to detect shifts and splitting in the nuclear levels due to hyperfine interactions in iron, one needs an energy range of at most $5*10^{-8}$ eV around E_0, which is achieved with Doppler velocities in the range of -10 to +10 mm/s. Figure 5.4 gives a schematic picture of a Mossbauer experiment in transmission mode with a moving single-line source and the absorbing sample in fixed position. A Mossbauer spectrum is a plot of the γ-ray intensity transmitted by the sample against the velocity v of the source. The latter is related to the actual energy by the above expression. This is the common mode of operation, called Mossbauer absorption spectroscopy, sometimes abbreviated as MAS. It is also possible to fix the ^{57}Co-containing source and move the single-line ^{57}Fe absorber in order to investigate cobalt-containing catalysts. This technique is called Mossbauer emission spectroscopy (MES). Hyperfine interactions couple the nucleus to its surroundings and make it a sensitive probe for the state of the absorber. Three interactions play a role; they are shown in the above figure.

MOSSBAUER SPECTROSCOPY IN CATALYST CHARACTERIZATION:

The application of Mossbauer spectroscopy to the investigation of catalysts began around 1970. By 1990, over 600 scientific papers had been published. Most applications of Mossbauer spectroscopy to catalysts fall in one of the following categories:

1) Identification of phases
2) Determination of oxidation states
3) Structure information
4) Determination of particle size
5) Kinetics of bulk transformations.

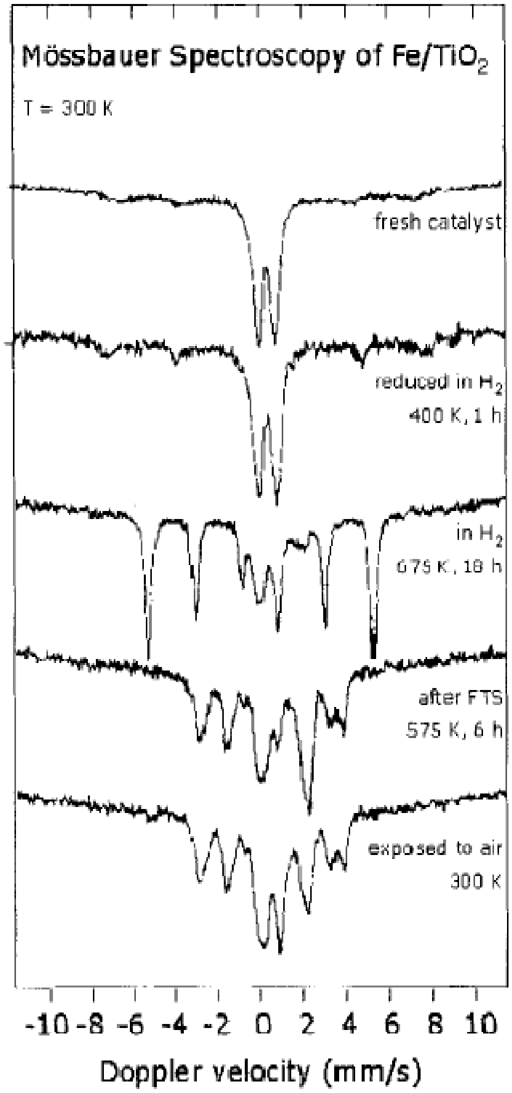


Figure 5.8: Mössbauer spectra at room temperature give detailed information on the state of iron in a TiO_2-supported iron catalyst after different treatments. Abbreviations such as H_2, 675 K, 18 h stand for 18 h of reduction in H_2 at 675 K, FTS for Fischer-Tropsch synthesis (from van der Kraan *et al.* [19]).

A typical example of how Mossbauer spectroscopy is used in the identification of oxidic, metallic and carbidic phases is provided by a study on titania-supported iron, prepared by impregnating the $Ti0_2$ support with a solution of iron nitrate (Fig.5.8). The top spectrum is that of a freshly impregnated and dried $Fe/Ti0_2$ catalyst. It shows a doublet with an isomer shift of 0.37 mm/s and a quadrupole splitting of 0.82 mm/s. It reveals that the iron is a high-spin Fe^{3+} species. It is difficult to draw conclusions on the type of compound that is present. Reference compounds of iron oxide (Fe_20_3, or Fe_30_4) possess magnetically split Mossbauer spectra. The relatively high value of the quadrupole splitting points to a highly asymmetric environment, as surface atoms would have. Chemical intuition - for what it is worth - suggests that we should expect a well-dispersed layer of iron oxide or oxy hydroxide. This would be consistent with the spectrum. After reduction in H_2 at 675 K the catalyst consists mainly of metallic iron, as evidenced by the sextet ($\delta =$ 0.00 mm/s , H = 331 kOe), along with some unreduced iron, which gives rise to two doublet contributions of Fe^{2+} and Fe^{3+} in the center.

The overall degree of iron reduction, as reflected by the relative area under the bcc-ion sextet, is high. One should not consider the relative spectral contributions as concentrations, however, because the three types of iron species may have different recoilless fractions. When reduced Fe/TiO_2 is used as a catalyst for the reaction between CO and H_2 to form hydrocarbons (the Fischer-Tropsch synthesis) the spectrum changes entirely. All metallic iron has been converted into a new phase. The spectrum is that of crystallographically well-defined iron carbide, namely the Hagg carbide. Apparently the strongly reducing atmosphere has affected the unreduced iron as well: all ions are now present as Fe^{2+} .The bottom spectrum has been recorded after exposing the used catalyst to the air at room temperature. The spectrum clearly has changed.

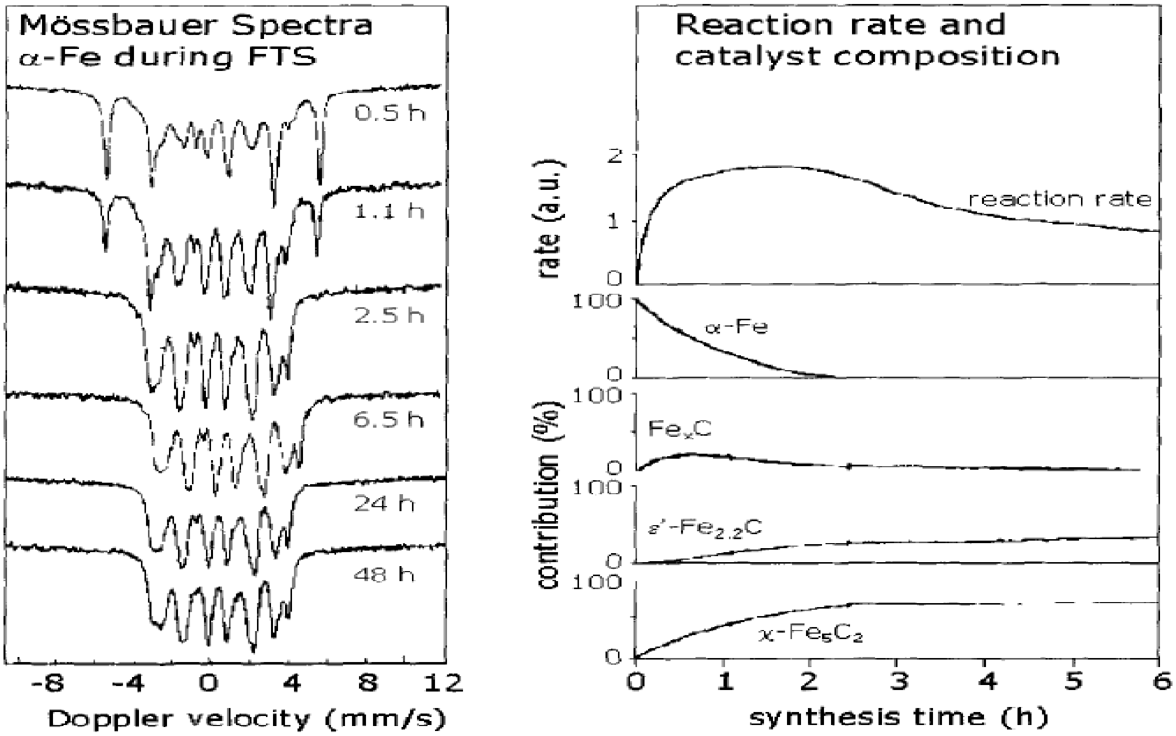


Figure 5.9: *Left*: Mössbauer spectra of a metallic iron catalyst after different periods of Fischer-Tropsch synthesis in CO+H_2 at 240°C, showing the conversion of metallic iron (visible by the outer two lines in the upper two spectra) into iron carbides; all spectra were recorded at room temperature. *Right*: reaction rate of the Fischer-Tropsch synthesis (*upper curve*) and the relative contributions of metallic iron and various carbides to the Mössbauer spectra (from [22]).

Although most of the carbide phase is still present, some of the ferrous iron has been oxidized to ferric iron. Hence, it is essential that the catalyst be studied under **`in`** situ conditions. The conversion of iron catalysts into iron carbide under Fischer-Tropsch conditions is well known and has been the subject of several studies. A fundamentally intriguing question is why the active iron Fischer-Tropsch catalyst consists of iron carbide, while cobalt, nickel and ruthenium are active as a metal. (Fig 5.9) shows how metallic iron particles convert to carbides in a mixture of CO and H2 at 515 K. After 0.5 and 1.1 h of reaction, the sharp six-line pattern of metallic iron is still clearly visible in addition to the complicated carbide spectra, but after 2.5 h the metallic iron has disappeared. At short reaction times, a rather broad spectral component appears - better visible in carburization experiments at lower temperatures - indicated as Fe_xC. The eventually remaining pattern can be understood as the combination of two different carbides: ε'-$Fe_{2.2}C$ and χ-Fe_5C_2. The relationship between carbide composition and catalytic activity is given by the above Fig 5.9. The catalytic activity starts low, increases rapidly to a maximum and decreases slowly thereafter. The increase inactivity occurs simultaneously with the carburization, but, without any direct correlation, the maximum in rate occurs when there is still metallic iron present. The interpretation given to the results in Fig. 5.9 is the following:

CO dissociates readily on iron. The carbon atoms have three possibilities to react:

$C + \alpha\text{-}Fe \rightarrow$ carbides
$C + H_2 \rightarrow$ hydrocarbons
$C \rightarrow$ inactive carbon

1st reaction is fast: carbon diffusion in metallic iron has an activation energy on the order of 15 kcal/mol only. However, as soon as the iron lattice becomes significantly disturbed, the activation energy increases and the diffusion becomes comparable to that of carbon in cobalt and nickel. Hence, in the early stage of the Fischer-Tropsch synthesis, 1st step consumes most of the carbon until the rate of carbon diffusion into the bulk of the catalyst slows down. Gradually, 2nd reaction, the actual synthesis of hydrocarbons, takes over. When the catalyst becomes saturated with carbon, more and more carbon stays at the surface, where it can either be hydrogenated or form inactive carbon (3rd reaction), a reaction which is responsible for the deactivation of the hydrocarbon formation. Thus, the time-dependent behavior of iron in the Fischer-Tropsch synthesis is initially governed by a competition between bulk carburization and hydrocarbon formation. For cobalt and nickel, the activation energy of carbon diffusion through the lattice is more than twice as high as that for iron, and consequently 1st reaction has hardly any influence.

These two examples illustrate how Mossbauer spectroscopy reveals the identity of iron phases in a catalyst after different treatments. The examples are typical for many applications of the technique in catalysis. A catalyst is reduced, carburized, sulfide, or passivated, and, after cooling down, its Mossbauer spectrum is taken at room temperature. However, a complete characterization of phases in a catalyst sometimes requires that spectra are measured at cryogenic temperatures, in particular when the catalysts are highly dispersed.

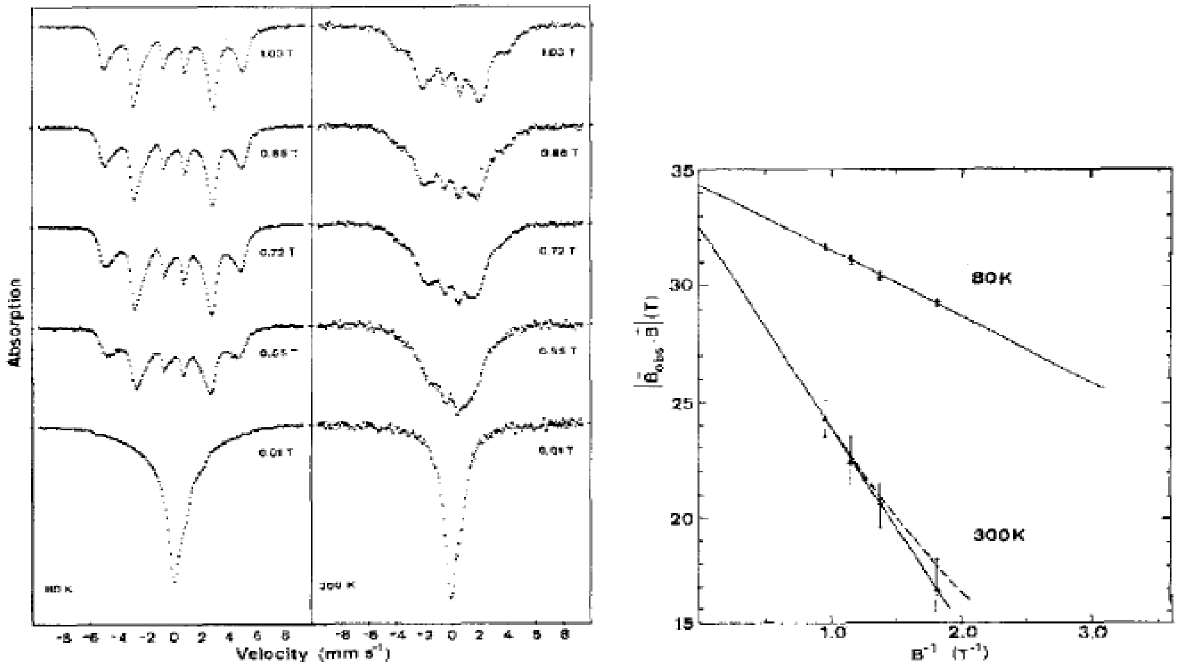
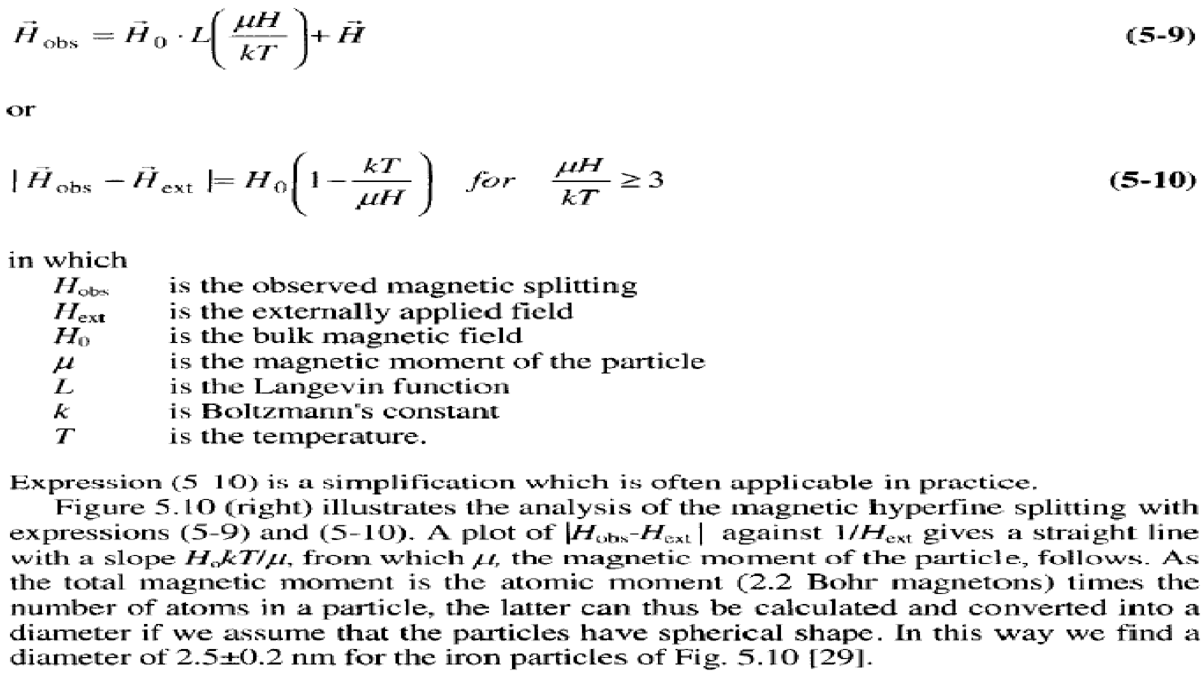


Figure 5.10: *Left*: Mössbauer spectra of a reduced carbon-supported iron catalyst at 80 and 300 K obtained in different applied magnetic fields. The spectra at the bottom, measured without external field, consist mainly of a singlet due to superparamagnetic metallic iron. The application of magnetic fields induces magnetic splitting. *Right*: Langevin plots according to Expression (5-10) for the spectra on the *left*. The lines extrapolate for $1/H \rightarrow 0$ to the magnetic splitting expected for single domain metallic iron particles; the slopes correspond to a particle diameter of 2.5±0.2 nm. The *dashed line* is a plot according to Expression (5-9) and confirms that the use of the high field approximation is justified (from Christensen *et al.* [29]).

PARTICLE SIZE DETERMINATION:

The occurrence of super paramagnetism allows one to determine the particle size if an external magnetic field is applied. (Fig 5.10) illustrates how it works on an Fe/C catalyst .The spectra without external field shows the single peak of super paramagnetic iron. As soon as the magnetic field is applied, magnetic splitting sets in. Its magnitude increases with increasing field strength. The external field orients the magnetization vector of the particles, but thermal excitations let the magnetization vector fluctuate around the direction of the applied field. One measures an average magnetic splitting, given by the Langevin equation:



$$\vec{H}_{\mathrm{obs}} = \vec{H}_0 \cdot L\left(\frac{\mu H}{kT}\right) + \vec{H} \qquad \textbf{(5-9)}$$

or

$$|\,\vec{H}_{\mathrm{obs}} - \vec{H}_{\mathrm{ext}}\,| = H_0\left(1 - \frac{kT}{\mu H}\right) \quad \mathit{for} \quad \frac{\mu H}{kT} \geq 3 \qquad \textbf{(5-10)}$$

in which

H_{obs}	is the observed magnetic splitting
H_{ext}	is the externally applied field
H_0	is the bulk magnetic field
μ	is the magnetic moment of the particle
L	is the Langevin function
k	is Boltzmann's constant
T	is the temperature.

Expression (5 10) is a simplification which is often applicable in practice.

Figure 5.10 (right) illustrates the analysis of the magnetic hyperfine splitting with expressions (5-9) and (5-10). A plot of $|H_{\mathrm{obs}}-H_{\mathrm{ext}}|$ against $1/H_{\mathrm{ext}}$ gives a straight line with a slope $H_o kT/\mu$, from which μ, the magnetic moment of the particle, follows. As the total magnetic moment is the atomic moment (2.2 Bohr magnetons) times the number of atoms in a particle, the latter can thus be calculated and converted into a diameter if we assume that the particles have spherical shape. In this way we find a diameter of 2.5±0.2 nm for the iron particles of Fig. 5.10 [29].

KINETICS OF SOLID STATE REACTIONS FROM SINGLE VELOCITY EXPERIMENTS:

Recording the complete Mossbauer spectrum of an iron catalyst takes typically several hours, which is too slow for following reactions in real time.

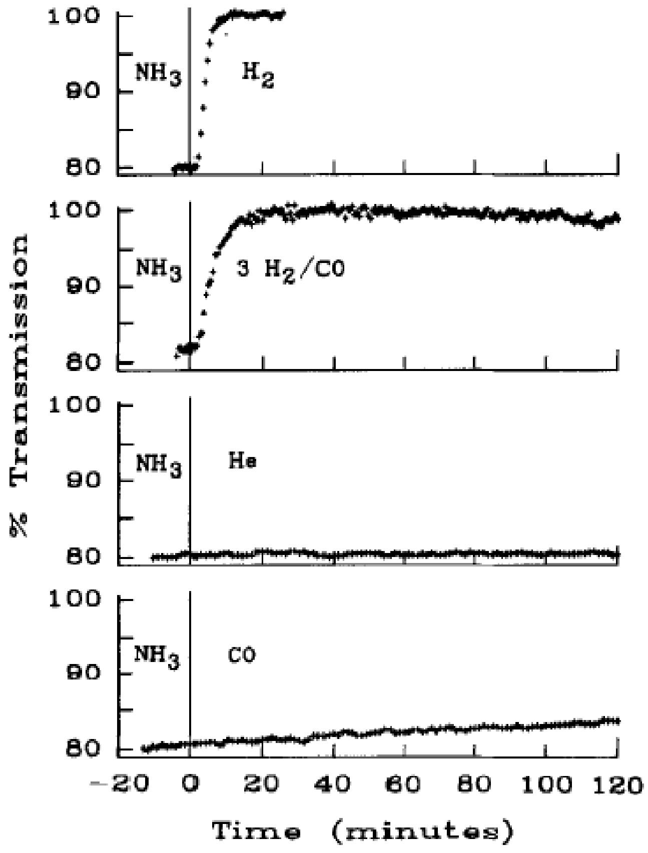


Figure 5.11: A constant velocity Mössbauer experiment reveals the kinetics of the denitridation of an iron nitride in different gases at 525 K. The negative part of the time scale gives the transmission of the most intense peak of the nitride; at time zero the gas atmosphere is changed to the desired gas. Denitridation occurs relatively fast in H_2, but is retarded by CO, whereas the nitride is stable in an inert gas such as helium (from Hummel *et al.* [33]).

Nevertheless, measuring the intensity of a characteristic peak at constant velocity can monitor processes occurring on the time scale of minutes to hours, such as the reduction of oxides. An elegant example has been reported by Hummel et al., who studied the stability of the nitride ζ-Fe_2N in different gases. Complete Mossbauer spectra show that denitridation of Fe_2N in H_2 yields metallic iron. In CO/H_2, however, a carbonitride forms. The constant velocity spectra of Fig. 5.11 reflect the rate at which the nitride is converted and reveal the kinetics of these processes. Other examples of constant velocity experiments have been given by Raupp and Delgass, who studied the conversion of iron into carbides with this technique. Alternative ways to study the kinetics of bulk transformations would be to monitor changes in weight or in magnetization. Such methods, however, are less specific about the initial and final state of the catalyst than Mossbauer spectroscopy.

IN-SITU MOSSBAUER SPECTRA UNDER REACTION CONDITIONS:

In situ characterization becomes an absolute necessity in cases where catalysts change their structure during the start-up of the catalytic reaction. (Fig.5.12) below shows the example of a bimetallic Fe-Ir catalyst during synthesis of methanol from CO and H_2. Noble metals such as platinum and iridium are poor CO hydrogenation catalysts, producing mainly methane. Addition of iron, however, increases the activity and shifts the product distribution towards methanol and higher oxygenates, particularly at pressures above 10 bars. At steady state, methanol selectivities of up to 97% have been observed, though at activity levels below those of the commercially applied Cu/ZnO systems. However, a catalyst containing equal amounts of iron and iridium on silica starts to convert CO and H_2 almost exclusively into methane. it typically takes 10-50 h for the product distribution to change gradually from methane to methanol. Mossbauer spectra taken **in** situ under high pressure reaction conditions (Fig.5.12) show that the initially reduced Fe-Ir catalyst, consisting of Fe-Ir particles and some unreduced iron believed to be in intimate contact with the support, has changed significantly when the catalyst has reached steady state. The bottom trace in Fig. 5.12 represents the difference between the working and the initial catalyst. It is characteristic of iron carbide in the super paramagnetic state. Also, the total absorption area of the spectrum increased, indicating loss of overall dispersion of the iron. Further characterization studies by Mossbauer spectroscopy and EXAFS after cooling down to ambient and cryogenic temperatures confirmed the interpretation and added much detail to it concerning the composition of the surface. In brief, during high pressure CO hydrogenation, the active part of the Fe-lr catalyst restructures from Fe-Ir alloy particles with a suface enriched in iron to an iridium-rich alloy accompanied by a, probably largely inactive, iron carbide phase. During the reconstruction, the chemical properties of the catalyst surface change significantly as well.

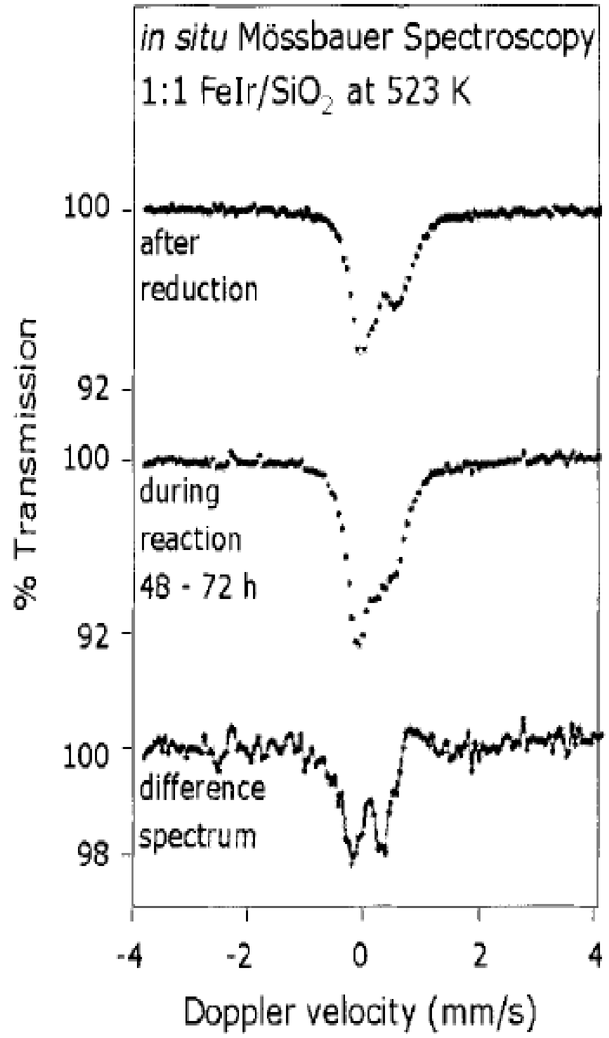


Figure 5.12: *In situ* Mössbauer spectra of a reduced FeIr/SiO$_2$ catalyst at a reaction temperature of 525 K and during CO hydrogenation when the catalyst is in its steady state (methanol-producing state). The bottom spectrum represents the difference between the two upper spectra; it is characteristic of an iron carbide in superparamagnetic state (courtesy of Hyung Woo, adapted from [8]).

The interesting notion of the work is that the Fe-Ir catalyst restructures itself by letting excess iron segregate into carbide particles, leading to an Fe-Ir alloy that is well tuned towards methanol formation. This process of self assembly, which has been encountered in other catalyst systems as well, has successfully been monitored by in situ Mossbauer spectroscopy.

CONCLUSION:

Although the majority of Mossbauer studies on catalysts deal with iron, other elements exhibiting the Mossbauer Effect have been used as well. Bussiere and coworkers used the Mossbauer Effect to study the state of tin in supported Pt-Sn and Ir-Sn reforming catalysts and of tin and antimony in mixed Sb-Sn oxides for the selective oxidation of propylene. Well known are the investigations by Clausen and Good on supported ruthenium catalysts by means of the difficult ^{99}Ru isotope. Wagner and coworkers used **Ir** Mossbauer spectroscopy to study supported **Ir** and **Pt-Ir** catalysts, used in the catalytic reforming of naphtha to hydrocarbon mixtures of higher octane numbers, and Au Mossbauer spectroscopy to investigate Au/MgO oxidation catalysts. In conclusion, Mossbauer spectroscopy has matured into one of the classical techniques for catalyst characterization, although its application is limited to a relatively small number of elements which exhibit the Mossbauer Effect. The technique is used to identify phases, determine oxidation states, and to follow the kinetics of bulk reactions. Mossbauer spectra of super-paramagnetic iron particles in applied magnetic fields can be used to determine particle sizes. In favorable cases the technique gives information on the structure of catalysts. The great advantage of Mossbauer spectroscopy is that its high energy photons see inside reactors to reveal information on catalysts under `in` situ conditions.

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Firstly, I like to thank our Head Of The Department, PROF.B.VISWANATHAN for giving us the assignment on –'application of any spectroscopic methods in catalysis', to work on it, to think on it and ultimately to gain some knowledge on this topic. His teachings during our class session helped me a lot and finally paved the way for the completion of the assignment. The materials have been collected from different references and informations given in the internet. So I am indebted to the authors of the books from where the informations have been extracted. The references are given below. Lastly I like to thank my pals for their continuous support and help.

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THANK YOU