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FT-IR study of stable surface species derived from $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$

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Abstract

The thermal behaviour of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ adsorbed on partly dehydroxylated alumina and silica surfaces has been followed by FT-IR spectroscopy in the temperature range of 303–723 K. The main products of surface reactions were the very stable tetrameric $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ and the less stable monomeric $(\eta\text{-}C_5H_5)Fe(CO)_2(\text{–O–})$ species. On alumina traces of 1:1 and 1:2 adducts of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ with surface Lewis acidic sites, while on silica traces of the 1:2 hydrogen bonded complex and oxidized monomeric cation $[(\eta\text{-}C_5H_5)Fe(CO)_3]^+$ were also detected.

Introduction

We recently reported an FT-IR study of the interaction of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ with hydroxylated alumina and silica surfaces [1]. The experimental conditions used in the above investigations were quite special: the supported $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ samples were wetted immediately after impregnation with paraffin oil (Nujol mull), which slowed down the surface reactions [2] and, therefore, we were able to get information about the earliest steps of the cluster–support interaction. At this stage of interaction we identified simultaneously a whole series of different surface complexes of the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecule: (i) hydrogen bonded species of one or two bridging carbonyls with surface hydroxyles; (ii) adduct type complexes with surface Lewis acidic sites; (iii) protonated species; and (iv) monomeric cyclopentadienyliron carbonyls. These experiments gave no information about the stability ranges of these species.

This paper examines the thermal stability of the cyclopentadienyliron carbonylic species. It will be shown, that the lifetime of some complexes formed in the early steps of the surface–cluster interaction (in the absence of paraffin oil) is so short that one cannot characterize them at room temperature. In addition, under conditions used in the present work, further new and stable surface species have been detected.

The thermal stability of $(\eta\text{-}C_5H_5)Fe(CO)_2I$ has also been studied, because a monomer moiety of the type $(\eta\text{-}C_5H_5)Fe(CO)_2(\text{–O–})$ may form from the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ on the surface.

Experimental

Commercial (Ventron) $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ and $(\eta\text{-}C_5H_5)FeICO)_2I$ were used as received and deposited from pentane solution on the alumina (Degussa, Alon C) and silica (Cabot Corporation, Cab-O-Sil HS5), respectively, to an extent of 1% metal. Prior to impregnation both supports were partially dehydroxylated (573 K, 16 h, in vacuum). The supported complexes were dried under vacuum and pressed into wafers (10 mg/cm^2) and then placed in a heatable vacuum IR cell.

Infrared spectra were recorded under vacuum at temperatures ranging from 303–723 K using a Digilab FTS-20C spectrometer. For all spectra reported a 200-scan data accumulation was carried out at a resolution of 2 cm^{-1}. As references we used spectra of partially dehydroxylated alumina and silica recorded at the same temperatures.

Difference spectra were obtained using a subtraction procedure between two spectra taken at two different temperatures.

In some cases, the angles between the carbonyls were estimated using the integrated intensities of the CO stretching bands.

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Results and discussion

The spectra of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2/Al_2O_3$ and $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2/SiO_2$ as a function of temperature are shown in Figs. 1(a) and 2(a), respectively. The respective difference spectra are shown in Figs. 1(b) and 2(b). The spectra in Figs. 1(c) and 2(c) are characteristic expanded difference spectra in the bridging carbonyl stretching region.

In rows 1–8 of Table 1 is a summary of the characteristic frequencies of the different complexes (**I–VIII**) identified on oxide surfaces in the first steps of surface–cluster interaction [1]. Using the original—first of all the difference—spectra of ref. 1, we reviewed and partly corrected these former assignments. In initial steps, the dominating surface complexes involve the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecule (**I**), forming hydrogen bonded or adduct type linkages with active surface sites **II–V**.

In non-polar solvents two conformers of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ (both containing two bridging and two terminal carbonyls) exist: a centrosymmetric *trans* and a *cis* form (C_{2v}). The *trans* form has two CO stretching bands around 2000 (terminal CO) and 1790 (bridging CO) cm^{-1}. For the *cis* form four bands are expected around 2000 (a_1), 1820 (a_1), 1790 (b_1) and 1960 (b_2) cm^{-1}. The band at 1960 cm^{-1} is very weak, while that at 1820 cm^{-1} has practically zero intensity.

Stable cyclopentadienyliron surface species on alumina support

The spectra recorded at different temperatures (Fig. 1(a)) show two main features: a doublet at around 2062 and 2015 cm^{-1} that is stable under 393 K, and a broad system between 1740–1400 cm^{-1} that disappears at 723 K. These two features suggest that two main species occur on the surface. However, several weak bands in the expanded difference spectra of Fig. 1(c) indicate the presence of other surface

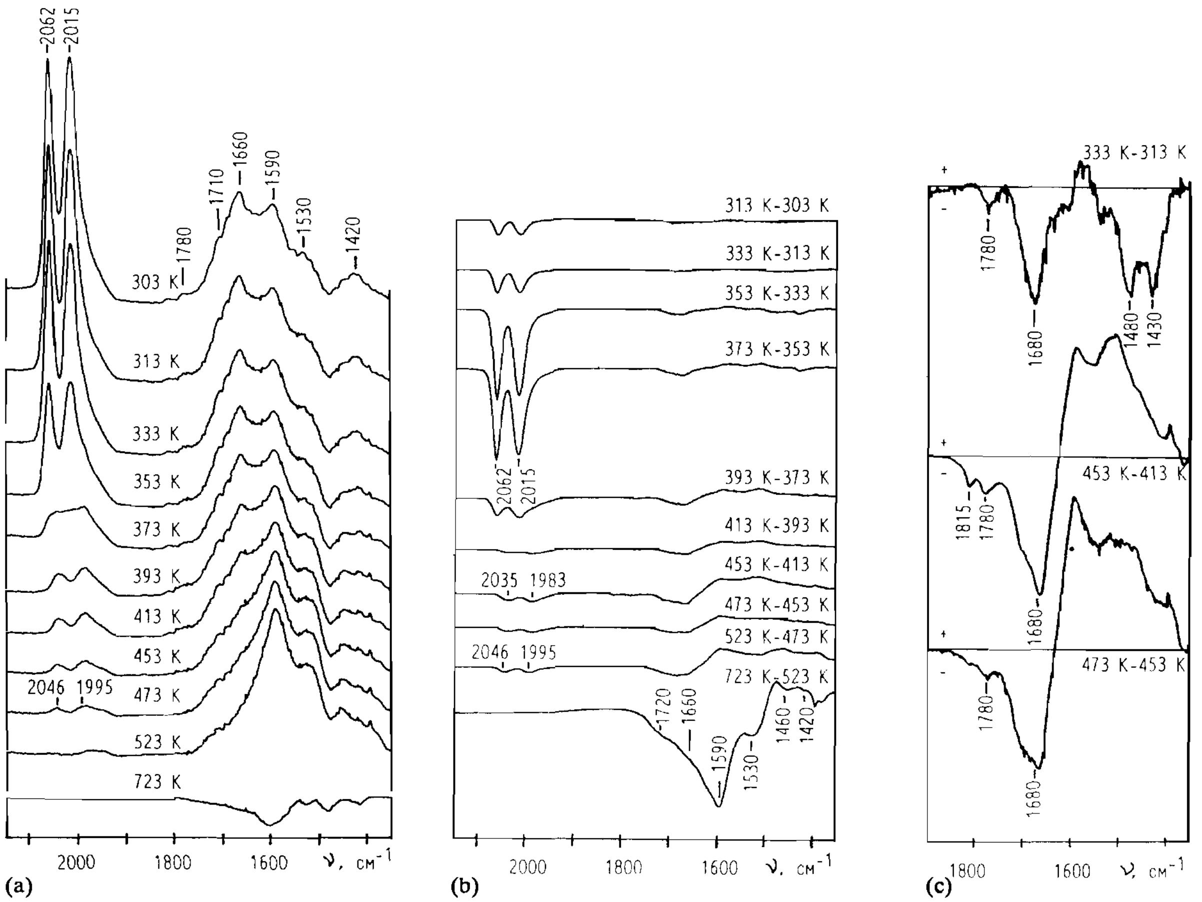


Fig. 1. Infrared spectra (a), difference spectra (b) and extended difference spectra (c) of the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2/Al_2O_3$ system during thermal treatment in vacuum.

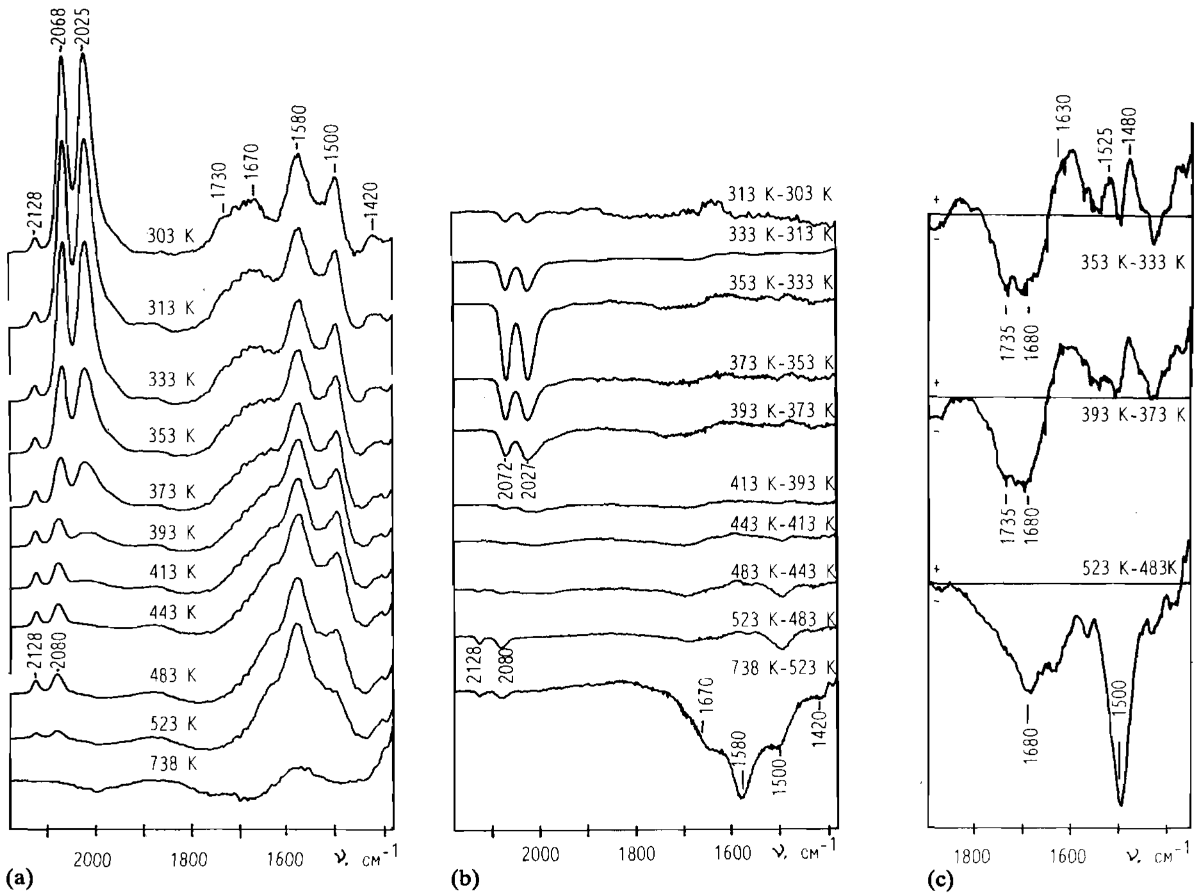


Fig. 2. Infrared spectra (a), difference spectra (b) and extended difference spectra (c) of the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2/SiO_2$ system during thermal treatment in vacuum.

species in low concentrations and with different thermal stabilities.

The very weak band around 1780 cm^{-1} indicates the presence of unperturbed $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecules (I) on the alumina surface, but in a very low amount. The band around 1780 cm^{-1}, and the lack of its a_1 pair around 1820 cm^{-1}, is characteristic of free $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$. The thermal behaviour of free *cis*-$[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ can be monitored by the expanded difference spectra of Fig. 1(c). Free $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ starts disappearing at 313–333 K, and the decomposition is complete by 453–473 K.

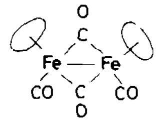
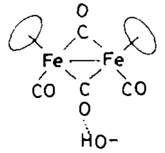
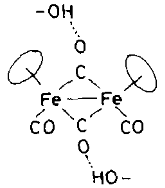
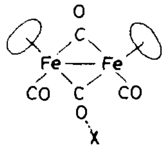
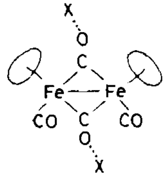
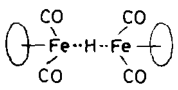
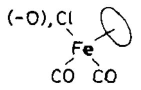
When $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ forms II or IV type 1:1 complexes, the bridging carbonyl band around 1780–90 cm^{-1} shifts to lower frequencies (1740–1400 cm^{-1}), while the inactive bridging CO band around 1820 cm^{-1} becomes active. Thus, the presence of the very weak band at 1815 cm^{-1} (together with that at 1680 cm^{-1}) could be attributed to traces of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecules linked to the active surface sites as 1:1 adducts (IV). The thermal behaviour of these bands is characteristic of the stability of the 1:1 adduct that decomposes in the temperature range of 413–453 K (Fig. 1(b) and (c)).

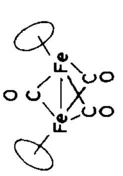
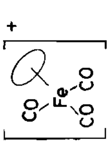
If no band around 1820 cm^{-1} appears, the components of the broad band system ranging from 1740 to 1400 cm^{-1} might be assigned to complexes involving both bridging carbonyls (structures III and V). In the first approximation, the two intensive bands at about 2017–1990 and 2062–2020 cm^{-1} also seem to belong to 1:2 complexes [1]. However, in the bridging CO stretching region of the expanded difference spectra rather weak bands at 1680, 1480 and 1420 cm^{-1} are detected. These can be assigned to small amounts of 1:2 adducts (V), which are stable up to 523 K.

The lack of any separate difference band around 1740–30 cm^{-1} indicates an absence of 1:1 or 1:2 hydrogen bonded complexes below 413 K.

More sophisticated examination of terminal and bridging CO stretching regions in Fig. 1(a) and (b)

TABLE 1. Spectral data of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ complexes

	Structure	Frequencies (cm^{-1})						Conditions	Reference	Stability (K)	Remarks
I		2004	1960		1776			in cyclohexane	3		*cis* + *trans*
		2000	1960		1788			Al_2O_3/Nujol, room T	[a]		medium concentration
		2000	1960		1780			Al_2O_3/Vacuum, T	this work	< 473	traces
II		1997	1958	1807	1733			in m-cresol	4		1:1 H-bonded complexes
		2015	1973	1817	1733			in liquid xenon + $(CF_3)_3COH$	5		
		2015	1970	1814	1742			Al_2O_3/Nujol, room T	[a]		low concentration
III		1997	1958		1733			in m-cresol	4		1:2 H-bonded complexes
		2022	1983		1733			in liquid xenon + $(CF_3)_3COH$	5		
		2020	1970		1733			Al_2O_3/Nujol, room T	[a]		medium concentration
					1735			SiO_2/Vacuum, T	this work	< 393	traces
IV		2026	1985	1828	1682			AlR_3	6, 7		1:1 adducts with Lewis acids
		2038	2020	1845	1463			BCl_3	6		
		2035	2000	1814	1700			Al_2O_3/Nujol, room T	[a]		low concentration
		2035	1983	1815	1680	1480	1430	Al_2O_3/Vacuum, T	this work	< 453	traces
V		2042	2004		1682			$2AlR_3$	6, 7		1:2 adducts with Lewis acids
		2035	2000		1700			Al_2O_3/Nujol, room T	[a]		medium concentration
		2046	1995		1680	1480	1430	Al_2O_3/Vacuum, T	this work	< 523	low concentration
					1680			SiO_2/Vacuum, T	this work	< 393	traces
VI		2069	2033					in liquid xenon + $(CF_3)_3COH$	5		
		2062	2015					Al_2O_3/Nujol, room T	[a]		not confirmed
VII		2060	2012					CCl_4, UV	8		
		2050	2004					$CpFe(CO)_2I/Al_2O_3$/Vacuum, T	this work	< 393	
		2062	2017					$CpFe(CO)_2I/SiO_2$/Vacuum, T	this work	< 373	
		2055	2015					Al_2O_3/Nujol, room T	[a]		high concentration
		2062	2015					Al_2O_3/Vacuum, T	this work	< 393	high concentration
		2068	2025					SiO_2/Vacuum, T	this work	< 393	high concentration

VIII				1812				pvc matrix, UV	9		
				1815				Al_2O_3/Nujol, room T	a		low concentration
IX		2125	2077					oxid. acids	10		
		2125	2080					SiO_2/Vacuum, T	this work	<473	medium concentration
X	$[(\eta\text{-}C_5H_5Fe(\mu Co)]_4$			1640[b]	1620[c]			UV	6[b], 11[c]		
				1650	1630			Al_2O_3/Nujol, room T	a		medium concentration
				1660	1590	1530	1420	Al_2O_3/Vacuum, T	this work	<533	high concentration
				1670	1580	1500	1420	SiO_2/Vacuum,T	this work	<533	high concentration

X stands for strong Lewis acid; Cp stands for η-C_5H_5. [a]Reviewed assignments of the spectra of ref. 1. [b]Dichloromethane solution. [c]KBr pellet.

suggests that in addition to the above complexes, there are several other stable species of higher concentration on the surface.

It would be misleading to assign the totality of the triad of bands around 2062, 2015 and 1740–1400 cm^{-1} to 1:1 (IV) and 1:2 type (V) adducts. As is seen in the spectra of Fig. 1(a) and (b), the terminal CO band pair at 2062 and 2015 rapidly decreases with increasing temperature and at about 393 K the decomposition is practically complete. These strong components are to be assigned to a surface species containing terminal carbonyls, only. The remaining two pairs of band components of lower intensities and of lower frequencies are the bands belonging to the adducts. In the difference spectra of Fig. 1(b) and (c) between 413–453 K the decrease of the band components at 2035 and 1983 cm^{-1} parallels the thermal behaviour of the band at 1815 cm^{-1}, and so these components belong to the 1:1 adduct. In a similar way, the bands at 2046 and 1995 cm^{-1}, together with that at 1680 cm^{-1}, could be assigned to the 1:2 adduct.

The most intense components of the terminal CO stretching bands at 2062 and 2015 cm^{-1}, which disappear between 333–393 K without being accompanied with notable changes in the bridging CO region, can be assigned to the surface bonded monomer (η-C_5H_5)Fe$(CO)_2$(–O–) (VII). In order to support this assignment we also measured the thermal behaviour of (η-C_5H_5)Fe$(CO)_2$I on silica and alumina supports (Fig. 3(a) and (b)). This compound produced CO bands at similar frequencies (2050 and 2004 cm^{-1}), and its thermal behaviour paralleled that of surface bonded (η-C_5H_5)Fe$(CO)_2$(–O–). The angles between both carbonyls (calculated from the integrated band intensities) are 98.9° for the surface bonded (η-C_5H_5)Fe$(CO)_2$(–O–) (at 303 K) and 95.4° for (η-C_5H_5)Fe$(CO)_2$I (in cyclohexane solution). These results indicate a similar CO arrangement in both cases. The first step of the surface reaction leading to this species might be the protonation of the $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecules by protons of acidic surface hydroxo groups.

Recall the very intensive components of the broad band system of the bridging carbonyls. These disappear above 523 K and belong to a molecule that has no terminal carbonyls. Such a surface molecule was already discovered to form on alumina surfaces in the Nujol mull experiments [1]. This surface molecule has structure VIII, and it is a known decomposition product of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ [9]. It should be, however, regarded as an unstable intermediate in surface reactions. The intensive, broad band at much lower (1740–1400 cm^{-1}) frequency in Fig. 1(a) indicates

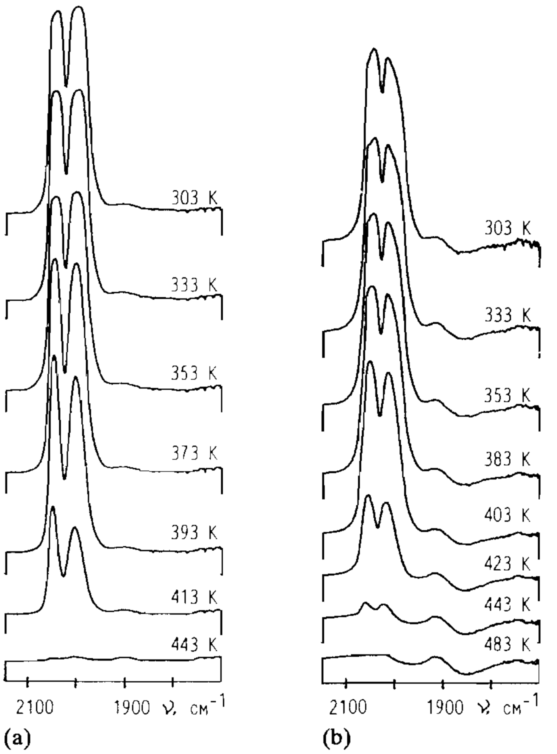


Fig. 3. Infrared spectra of (η-C_5H_5)Fe(CO)$_2$I/Al_2O_3 (a) and (η-C_5H_5)Fe(CO)$_2$I/SiO_2 (b) systems during thermal treatment in vacuum.

bridging carbonyls have a markedly higher basicity.

It is known that refluxing of [(η-C_5H_5)Fe(CO)$_2$]$_2$ in xylene results in the [(η-C_5H_5)Fe(μ-CO)]$_4$ tetramer compound [11]. The reaction can be accelerated by photolysis [12] or by exchanging one of the terminal CO ligands with triphenylphosphine [13]. The tetramer compound has been the subject of studies concerned with the catalytic properties of the neat complex [14]. The high stability of [(η-C_5H_5)Fe(μ-CO)]$_4$ makes it an efficient catalyst for selective hydrogenation of alkynes to alkenes. We believe that active sites on the alumina surface are favorable to the [(η-C_5H_5)Fe(CO)$_2$]$_2$ → [(η-C_5H_5)Fe(μ-CO)]$_4$ reaction. The very stable compound producing the above intensive bridging carbonyl stretching band system should be assigned to [(η-C_5H_5)Fe(μ-CO)]$_4$ (structure **X** in Table 1).

The natural [(η-C_5H_5)Fe(μ-CO)]$_4$ molecule shows a single strong carbonyl band in the infrared spectrum at 1620 cm^{-1} in KBr pellet [11] and at 1640 cm^{-1} in dichloromethane solution [6]. The rather large difference between the frequencies measured in KBr pellet and that in solution indicates the bridging carbonyls are very sensitive to the environment of the molecule. In the structure of the tetramer the iron atoms are located at the corners of a regular tetrahedron. Each iron atom is bonded directly to the other three, and each carbonyl group is bonded symmetrically to three iron atoms on each face of the tetrahedron, also forming a tetrahedral array [15]. These special type of bridging carbonyls show a very high basicity. This is manifested by their ability to form donor–acceptor linkages with electrophiles [6]. Such linkages certainly also occur on oxide surfaces, and result in the splitting of the single CO band. This expectation is in accordance with the main features of the bridging carbonyl stretching band system, as seen in Fig. 1(a) and (b). The difference spectra in this region are dominated by three main ranges: the first one contains two components around 1720 and 1660 cm^{-1}, the second shows a very stable and a very intensive band at 1590 cm^{-1} and the third one is a group of lower frequency components of medium intensity at 1530 and 1460–1420 cm^{-1}. In the difference spectra, the lowest frequency broad band is essentially a doublet.

These bands can be assigned to [(η-C_5H_5)Fe(μ-CO)]$_4$ molecules (**X**) bonded by weaker forces (physisorption) to the surface and to [(η-C_5H_5)Fe(μ-CO)]$_4$ molecules in adduct type linkages with Lewis acidic surface sites. By heating the system, the sum of the amount of the differently bonded forms does not change below 523 K. As seen in Fig. 1(c), below 523 K the band around 1590 cm^{-1} increases at the expense of that at 1665 cm^{-1} suggesting one of the possible structures is more stable. At present our data are not sufficient to say which type of structure is preferred.

Stable cyclopentadienyliron surface species on silica support

It is interesting to note that even traces of free [(η-C_5H_5)Fe(CO)$_2$]$_2$ molecules do not occur in this system (no band around 1780 cm^{-1}). In accordance with the lack of any bands around 1815 cm^{-1}, the same holds for 1:1 complexes of [(η-C_5H_5)Fe(CO)$_2$]$_2$. The weak negative difference band in Fig. 2(c) around 1735 cm^{-1} (in the temperature range of 333–393 K) indicates a 1:2 type hydrogen bonded complex of structure **III**, but in low concentration. The traces of structure **V** are not excluded (see the very weak shoulder at 1680 cm^{-1} in Fig. 2(c)). Therefore, the presence of the original [(η-C_5H_5)Fe(CO)$_2$]$_2$ skeleton on silica support is only characteristic of the initial steps of cluster–support interaction, as was shown in the Nujol mull experiments reported in ref. 1.

The dominating doublet of the terminal CO stretching modes around 2068 and 2025 cm^{-1} can be undoubtedly assigned to the (η-C_5H_5)Fe(CO)$_2$(–O–) monomer cyclopentadienyliron, and it is stable up to 393 K. The CO stretching bands of (η-

$C_5H_5)Fe(CO)_2I$ adsorbed on silica appear at higher frequencies (2062 and 2017 cm^{-1}) than those on alumina (2050 and 2004 cm^{-1}), and decompose in the temperature range of 393–443 K. The angle between both carbonyls (calculated from the integrated intensities) is 98.3°.

In the terminal CO stretching region a new doublet appears at 2125 and 2080 cm^{-1}, which belongs to a rather stable (decomposition temperature 483–573 K!) surface species. An analogous complex was not detected on alumina support. The very high frequencies of these bands suggest that they should be assigned to the oxidized monomeric carbonyl $[(\eta\text{-}C_5H_5)Fe(CO)_3]^+$ (structure **IX**) and/or $[(\eta\text{-}C_5H_5)Fe(CO)_2]^+$ [10, 12]. $[(\eta\text{-}C_5H_5)Fe(CO)_3]^+$ shows bands at 2125 and 2077 cm^{-1} in acidic solutions [12]. Using integrated intensities of both bands and supposing a tricarbonylic structure the calculated angle between the carbonyls is 94.0°, which is more realistic than the value of 115.2° calculated using a dicarbonylic model.

The region of bridging CO stretching modes is also dominated by the bands of differently bonded $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$, with the difference that the frequencies and relative intensities of the band components are different (main features are at 1670, 1580, 1500 and 1420 cm^{-1}). In the expanded difference spectra of Fig. 2(c) it is seen that at higher temperatures the band around 1580–1620 cm^{-1} is increasing with temperature at the expense of the high frequency component around 1700 cm^{-1}. (For this region, like that of the terminal carbonyls, it is also characteristic that the respective bands appear at somewhat higher frequencies.) It is very interesting that at low temperature (293–313 K) weak positive and at higher temperatures negative difference bands at 1630, 1525 and 1480 cm^{-1} appear indicating further transitions between differently bonded $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ molecules.

From $(\eta\text{-}C_5H_5)Fe(CO)_2I$ on alumina (Fig. 3(a)) only a negligible amount, and on silica (Fig. 3(b)) no $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ forms, which means that once the Fe–Fe bond splits in $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$, the surface reaction does not follow the pathway that concludes in the formation of the $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ tetramer.

Ready formation of $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ from $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$in high concentration on both alumina and silica suggests a practical method for the preparation of the $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ catalyst, directly on the surface of the oxide supports. Previous methods of preparation [11–13] of $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ are very slow (several days) and of low yields (14–56%).

Conclusions

After the initial interaction [1] of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ with hydroxylated alumina and silica at room temperature, a complex system of carbonylic surface species has been detected.

(i) Traces of the original unperturbed $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ molecules (**I**) are detectable only on alumina. Hydrogen bonded and adduct type 1:1 and 1:2 complexes of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ (structures **II–V**) occur also in low concentration on alumina only, except the 1:2 hydrogen bonded complex (**III**) which was detected on silica as well.

(ii) Both alumina and silica surfaces contain $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ (**X**), a molecule that has only bridging carbonyls of very high basicity. The other main surface species is the surface bonded monomeric cyclopentadienyliron carbonyl $(\eta\text{-}C_5H_5)Fe(CO)_2\text{-}(\text{–O–})$ (**VII**). On silica an oxidized monomeric species $[(\eta\text{-}C_5H_5)Fe(CO)_3]^+$ (**IX**) has also been detected in medium concentration.

(iii) $[(\eta\text{-}C_5H_5)Fe(\mu\text{-}CO)]_4$ which forms directly from $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ can be regarded as the endproduct of the surface reactions. Although the original frame of $[(\eta\text{-}C_5H_5)Fe(CO)_2]_2$ on hydroxylated oxide surfaces is only partly preserved, the presence of the cyclopentadienyl ligand prevents iron atoms from losing carbonyls, in a very striking contrast to pure carbonylic iron clusters [2].

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