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Note

Chiral recognition of the Schiff bases by NMR spectroscopy in the presence of a chiral dirhodium complex. Deuterium isotope effect on ^{13}C chemical shift of the optically active Schiff bases and their dirhodium adducts

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The dirhodium method has been successfully applied in chiral recognition of the optically active Schiff bases, derivatives of *ortho*-hydroxyaldehydes existing in the NH-form. or at tautomeric equilibrium. The position of the equilibrium of Schiff bases as well as their adducts has been established on the basis of measurements of deuterium isotope effects on ^{13}C chemical shifts. The presence of the proton transfer equilibrium or NH-tautomer has promoted the adduct formation. At the equilibrium state, formation of the adducts has shifted the proton transfer equilibrium towards the NH-form. The binding site was the oxygen atom of the proton donor group. Copyright © 2007 John Wiley & Sons, Ltd.

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KEYWORDS: Schiff bases; proton transfer equilibrium; deuterium isotope effect; ^{13}C NMR; chiral recognition; dirhodium method

INTRODUCTION

Schiff bases, derivatives of *ortho*-hydroxyaldehydes and various amines have been widely studied because of their biological activity and application as ligands for complexes used in enatioselective catalysis or as model compounds in investigation of enzymatic reactions.[1–11] The interesting features of these compounds are usually connected with the presence of the intramolecular hydrogen bond. Investigation of such intramolecular hydrogen bonds has been very successful with measurements of the deuterium isotope effects on chemical shifts. In some systems this method detects the presence of proton transfer equilibrium and allows determination of the mole fraction of tautomers. Measurements of the deuterium isotope effect can be easily performed on a standard NMR spectrometer as one-tube experiments for partially deuterated samples containing deuterated and non-deuterated species.[12,13] In studies of biologically active compounds and enantioselective reactions, the determination of enantiomeric ratios is of great importance. The dirhodium method, developed by Duddeck, has been widely used in chiral recognition of various groups of compounds like olefins, selenides and nitriles, for which the classical method of chiral lanthanide shift reagent (CLSR) usually fails.[14–19] Depending on the molar ratios of the components and the binding site in the ligand typically, dirhodium complex forms with ligands kinetically labile 1 : 1 or 1 : 2 adducts (Fig. 1). From NMR spectra, two parameters can be extracted: a signal shift ($\Delta\delta$) due to a change in the inductive and/or mesomeric influence of the complexing atom/group in the ligand, and signal dispersion ($\Delta\nu$) due to the presence of diastereomers.

The aim of this study was to apply the dirhodium method for chiral recognition of Schiff bases existing in different tautomeric forms. To the best of the author's knowledge, Schiff bases have not been studied by this method yet. The position of the proton transfer equilibrium in Schiff bases and their adducts has been estimated by means of deuterium isotope effect on the ^{13}C chemical shift.

EXPERIMENTAL

The Schiff bases studied (Fig. 2) were prepared by condensation of appropriate aromatic *ortho*-hydroxyaldehydes with

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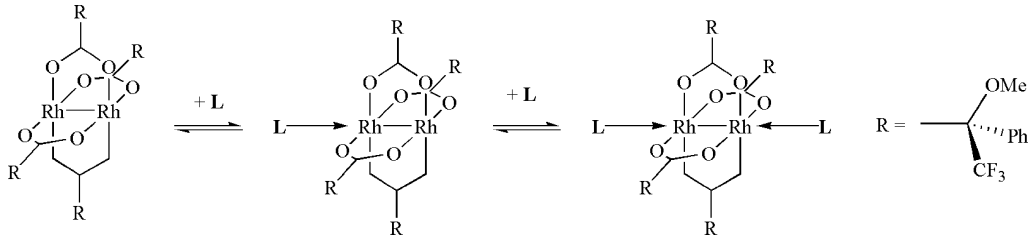
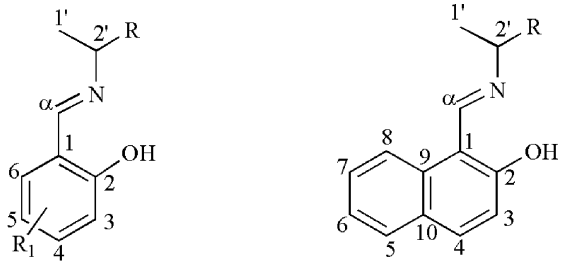


Figure 1. Structure of dirhodium tetrakis[(*R*)-α-methoxy-α-(trifluoromethyl)-phenylacetate] and adduct formation with one or two ligands **L**.



(**1**) R = CH_2CH_3 R_1 = H
(**2**) R = CH_2CH_3 R_1 = 3,5-di$C(CH_3)_3$
(**3**) R = CH_2CH_3 R_1 = 5-NO_2
(**4**) R = C_6H_5 R_1 = 5-NO_2
(**5**) R = CH_2CH_3 R_1 = 3,5-diNO_2
(**6**) R = C_6H_5 R_1 = 3,5-diNO_2
(**7**) R = CH_2CH_3
(**8**) R = C_6H_5

Figure 2. Schiff bases studied (**1**–**8**).

racemic 2-aminobutane or α-methyl-benzylamine according to the procedure described elsewhere.[20,21] The synthesis of dirhodium tetrakis[(*R*)-α-methoxy-α-(trifluoromethyl)-phenylacetate] has been reported earlier.[14]

The ^{1}H and ^{13}C NMR spectra in $CDCl_3$ were recorded on a BRUKER DPX-400 spectrometer operating at 400.13 MHz (^{1}H) and 100.62 MHz (^{13}C). Typical spectral parameters used for ^{1}H NMR were as follows: spectral width 8 kHz, number of data points 131 K, 0.12 Hz per point digital resolution, acquisition time 8.19 s, relaxation delay 1 s, pulse width 7.8 μs, number of scans 16; for ^{13}C NMR: spectral width 24 kHz, number of data points 131 K, 0.37 Hz per point digital resolution, acquisition time 2.72 s, relaxation delay 1 s, pulse width 9.2 μs, number of scans 1000–8000. The chemical shifts were referred to TMS as internal standard. Typical concentration of the samples was 0.1 M. The temperature was maintained and measured with Eurotherm BV-T 2000 to an accuracy of 1 K. The deuterium isotope effects were measured as the differences between the ^{13}C signals in the spectra of non-deuterated and deuterated species: $^n\Delta C(D) = \delta C(H) - \delta C(D)$. Deuteration of the compounds was achieved by dissolving the sample in CH_3OD followed by evaporation under reduced pressure. All measurements of deuterium isotope effects on ^{13}C chemical shift were performed as one-tube experiments of the partially deuterated samples (yield of deuteration 30–75%). In chiral recognition experiments, equimolar amounts of the Schiff bases studied (deuterated and/or non-deuterated) and dirhodium complex were dissolved in 0.6 ml of $CDCl_3$. One drop of acetone-d_6 (*ca* 4 μl) was added to each the sample to increase the solubility of a given complex.

RESULTS AND DISCUSSION

The selected ^{1}H and ^{13}C chemical shifts, deuterium isotope effects on the ^{13}C chemical shift without and with dirhodium complex (Rh*) as well as signal shift ($\delta\Delta$) and signal dispersion ($\Delta\nu$) of the compounds studied are collected in Table 1. For the sake of clarity of the data, all signals are available as Supplementary Material (Table S1, S2 and S3). Overlapping of the signals caused by Mosher acid residues or the signal's complex structure made it difficult to determine the signal shift ($\Delta\delta$) for some hydrogen and carbon atoms. In these cases, the $\delta\Delta$ values are not included in Tables S1 and S2. Assignments of the signals were made on the basis of the substituent effects and NMR correlation 2D experiments.

The optically active Schiff bases studied **1**–**8** may exist in different tautomeric forms (Fig. 3). In the studies of proton transfer equilibrium, the measurements of deuterium isotope effects on ^{13}C chemical shift are very useful. The value of deuterium isotope effect measured for the carbon linked to the phenolic group $^n\Delta C$-2(D) (the most sensitive to the position of the proton in the intramolecular hydrogen bond) detects the presence of the proton transfer equilibrium and allows determination of the mole fraction of tautomers.[12,20,22] For compounds **1**–**2** the $^n\Delta C$-2(D) values were in range of ~400 to ~500 ppb and only slightly temperature sensitive (*ca* 40–50 ppb). These features and small changes of the C-2 chemical shift (*ca* 0.25–0.50 ppm) with changing temperature confirmed the absence of proton transfer equilibrium in Schiff bases **1**–**2** and indicated that the proton is localized at the oxygen atom.[22] It was confirmed also by the lack of ^3J(NH,H) coupling constants on the signal of the azomethine proton. Introduction of the electron-withdrawing substituents (**3**–**6**) into the aromatic ring or expansion of the aromatic system (**7**–**8**) promoted the proton transfer equilibrium.[20,21] For compounds **3**–**4** and **7**–**8** large positive (*ca* 206–541 ppb) and negative (−175–546 ppb) $^n\Delta C$-2(D) values were measured. With decreasing temperature, these deuterium isotope effects changed; the largest changes in $^n\Delta C$-2(D) were found for compounds **3** (from −175 up to 336 ppb) and **8** (from −237 up to −546 ppb). Great changes in C-2 chemical shifts of *ca* 2.5–4.5 ppm, temperature sensitive $^n\Delta C$-2(D) values, change in their sign with lowering temperature and the values of ^3J(NH,H) coupling constants (~2–12 Hz) indicated the presence of the proton transfer equilibrium in compounds **3**–**4** and **7**–**8**.[20,21,22] The position of the equilibrium was established on the basis of the S-shape dependence of the $^n\Delta C$-2(D) value on the mole fraction of the proton transferred NH-form (χ) (Fig. 4). In compounds **3** and **4** at room temperature, the equilibrium was shifted

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Table 1. Selected ^{1}H and ^{13}C chemical shifts (ppm), signal shift (Δ, ppm), signal dispersion ($\Delta\nu$, Hz), deuterium isotope effects (ppb) and $^{3}J_{(NH,H)}$ coupling constants (Hz) of studied Schiff bases and their adducts with dirhodium complex (Rh*) in $CDCl_3$

		Position 2						Position α								^{3}J(NH,H)	
Compound	T (K)	δH (ppm)	$\Delta\delta$ (ppm)	δC (ppm)	$\Delta\delta$ (ppm)	$^{n}\Delta C$(D) (ppb)	Rh* (ppb)	δH (ppm)	$\Delta\delta$ (ppm)	$\Delta\nu$ (Hz)	δC (ppm)	$\Delta\delta$ (ppm)	$\Delta\nu$ (Hz)	$^{n}\Delta C$(D) (ppb)	Rh* (ppb)	(Hz)	Rh* (Hz)
(**1**)	295	13.56	1.09	161.06	9.3	~381	br.	8.41	−0.30		163.43	0.37		~94	br.	n.o.	n.o.
	270	13.68	1.36	160.85	br.	424	br.	8.43	−0.34		163.37	0.42		131	br.	n.o.	n.o.
	250	13.79	1.38	160.69	br.	425	br.	8.45	−0.37		163.33	0.45		140	br.	n.o.	n.o.
	230	13.91	1.45	160.52	br.	435	br.	8.48	−0.39		163.30	0.45		137	br.	n.o.	n.o.
(**2**)	295	13.96	0	158.20	−0.23	482	br.	8.33	0		163.64	−0.06		n.o.	br.	n.o.	n.o.
	270	14.10	0	158.08	br.	495	br.	8.35	0		163.57	−0.05		134	br.	n.o.	n.o.
	250	14.23	0	158.01	0.04	507	br.	8.36	0		163.52	−0.8		154	br.	n.o.	n.o.
	230	14.40	0	157.95	0.04	525	524	8.38	0		163.46	0.05		164	164	n.o.	n.o.
(**3**)	295	15.01	−0.12	171.45	9.90	336	−214	8.33	0		162.16	1.98		254	n.o.	n.o.	12.4
	270	15.12	−0.26	172.63	9.28	206	−198	8.32	0.03		162.39	1.95/1.79	8	243	160 167	4.4	13.2
	250	15.16	−0.34	174.06	8.08	29	−180	8.32	0.06		162.70	1.75/1.59	16.1	223	160	6.8	13.4
	230	15.10	−0.25 −0.48	175.97	6.49	−175	br.	~8.31	0.10		163.13	1.29		190	br.	8.8 (ov.)	br. ~13 .
(**4**)	295	14.90	0.44	168.77	12.0	541	br.	~8.40	−0.30/−0.34	18	162.39	2.2		541	br.	n.o.	ov. ~12.4
	270	15.04	0.34	169.19	12.3	530	−230	8.41	−0.29/−0.35	24	162.40	2.2		530	−230	n.o.	13.4
	250	15.17	0.22	169.71	12.2	499	br.	8.41	−0.27/-0.34	28	162.45	2.2		499	br.	2.4	13.1
	230	15.30	~0 −0.10	170.48	~12	423	br.	8.42	−0.24/−0.34	40	162.55	~2		423	br.	3.6	br.~13
(**5**)	295	14.75	−0.07	171.03	0.45	n.o	−107	8.35	−0.04		164.41	0		n.o.	150	12.8	13.1
	270	14.66	−0.05	171.14	0.48	−21	−93	8.40	−0.06		164.51	−0.01/−0.03	2	n.o.	167 168	13.2	13.4
	250	14.59	−0.03	171.20	0.47	−46	−87	8.44	−0.05		164.61	0.03/0.07	4.7	149	157 172	13.2	13.4
	230	14.51	0	171.26	0.39	−31	br.	8.52	−0.09		164.73	−0.07		149	132	13.4	~13
(**6**)	295	15.36	−0.06	170.65	0.46	n.o	−175	8.21	−0.03		164.30	0.11		n.o.	143	br.	~12.4
	270	15.28	−0.06	170.93	0.43	n.o	−146	8.24	−0.03		164.38	0.11/0.07	4.4	n.o.	160 160	12.4	12.8
	250	15.19	−0.03	171.08	0.41	−80	−170	8.27	−0.03		164.44	0.11/0.04	6.5	163	175 160	12.8	13.6
	230	15.14	−0.04	171.18	0.4	−63	br.	8.32	−0.03		164.48	0.04		166	182	13.2	13.8

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Table 1. (*Continued*)

Compound	T (K)	Position 2						Position α						^3J(NH,H)	
		δH (ppm)	$\Delta\delta$ (ppm)	δC (ppm)	$\Delta\delta$ (ppm)	$^n\Delta C$(D) (ppb)	Rh* (ppb)	δH (ppm)	$\Delta\delta$ (ppm)	$\Delta\nu$ (Hz)	δC (ppm)	$\Delta\delta$ (ppm)	$\Delta\nu$ (Hz)	$^n\Delta C$(D) (ppb)	Rh* (ppb)		Rh* (Hz)
(**7**)	295	14.44	−0.36	176.96	5.54	~−393	−212	8.71	0.08		155.91	0.12		425	n.o.	n.o.	~11.2
	270	14.36	−0.27	178.01	4.11	−497	−207	8.69	0.1		155.81	0.35/0.22	13.3	484	283	n.o.	12.8
															289		
	250	14.25	−0.15	178.83	3.50	−497	−197	8.68	0.12		155.77	0.43/0.27	16	469	br.	9.6	13.6
	230	14.14	−0.14	179.53	2.71	−468	−234	8.69	~0.12		155.79	0.28		446	br.	11.6	br.
			0.16														
(**8**)	295	14.98	0.07	174.19	8.50	−237	n.o.	8.84	−0.17/−0.20	10	156.88	−0.36		455	n.o.	n.o.	11
	270	14.98	−0.39	175.35	7.37	−372	−255	8.81	−0.13/−0.17	14	156.64	−0.18		495	289	7.6	14
	250	14.93	−0.31	176.49	6.23	−469	−234	8.78	−0.08/−0.12	18	156.44	−0.02		511	327	8.9	~11
	230	14.87	−0.09	177.69	5.02	−546	−235	8.75	−0.01/−0.09	28	156.27	0.07		522	321	9.9	13.2
			−0.36														

br. broad; n.o. not observed; *italic* 2 : 1 adduct.

towards the OH-form ($\chi = 0.25$ and 0.1, respectively, Fig. 4) and with decreasing temperature it moved towards the NH-form. At 230 K the mole fractions of the NH-form were ~0.7 for **3** and ~0.4 for **4**. In compounds **7** and **8** at room temperature, the equilibrium strongly shifted towards the NH form ($\chi = 0.8$–0.9). For these compounds, a decrease in temperature caused further increase in the NH form of mole fraction. The change in the position of the equilibrium was confirmed by an increase in the ^3J(NH,H) coupling constant values. Similar behaviour of the deuterium isotope effects was observed for Schiff bases, derivatives of 5-nitrosalicylaldehyde or 2-hydroxynaphthaldehyde and benzylamine and *n*-butylamine.[20,21]

In Schiff bases **5**–**6**, derivatives of 3,5-dinitrosalicylalde hyde, the values of ^3J(NH,H) higher than 13 Hz[23,24] indicated that these compounds are in the pure NH-form. The $^n\Delta C$-2(D) values measured for **5**–**6** were lower (in range from −20 up to −80 ppb) than those measured for other Schiff bases existing also exclusively in the NH-form, e.g. derivatives of gossypol and benzylamine or tetrabutylammonium salts of amino acid Schiff bases (*ca* −200 ppb).[24,25] This exceptional behaviour is difficult to explain and may be related to different electronic structure of the NH-form. Judging from the δ C-2 value of *ca* 171 ppm, it suggests that the NH-form with transferred proton was more zwitterionic than quinoidic. In the quinoidic form, the C-2 chemical shift was close to *ca* 180 ppm.[20]

In the next step, the adduct formation of different tautomers of Schiff bases with dirhodium complex was studied. The dirhodium method has been successfully used in chiral

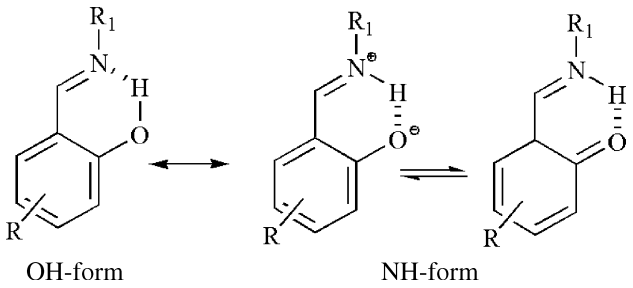

Figure 3. Proton transfer equilibrium in Schiff bases.

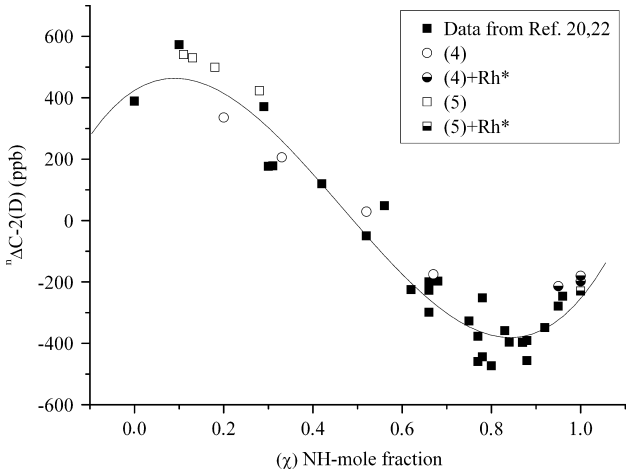

Figure 4. Plot of $^n\Delta C$-2(D) vs χ (mole fraction of NH-form).

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recognition of different classes of compounds.[14–19] This method does not require a series of measurements with increasing molar ratios of the ligand to dirhodium complex in contrast to the CLSR method. In this work the dirhodium method was applied for chiral recognition of Schiff bases for the first time.

Compounds **1** and **2**, existing exclusively in the OH-form, showed different behaviour from that of the other Schiff bases studied. The most affected by complexation was the hydrogen in the proton donor group. The $\Delta\delta$ values of the signal were in range of 0–1.5 ppm (Table 1). The disappearance of some of the carbon signals in the baseline (e.g. C-2), their broadening at temperatures below 295 K and a lack of signal dispersion in the whole range of temperatures were observed. This indicated that the adduct formation equilibrium is shifted towards the free ligand (Fig. 1) and that the presence of dirhodium complex had no influence on the position of the proton in the intramolecular hydrogen bond. The lack of signal shifts for compound **2** suggested that large *tert*-butyl substituents prevent the adduct formation between the Schiff base and the dirhodium complex. For compound **1** it was not possible to measure deuterium isotope effects in the presence of Rh* because the signals were very broad and disappearing in the baseline. For adduct of compound **2**, only the measurement at 230 K gave sufficient results and the found $^{n}\Delta C$(D) values were similar to those obtained for the samples without the dirhodium complex (Table 1).

The other Schiff bases, being in the NH-form (**5**–**6**) or tautomeric equilibrium (**3**–**4** and **7**–**8**), formed adducts with dirhodium complex. The $\Delta\delta$ values of the signal of the hydrogen in the proton donor group varied from −0.48 ppm for the adduct of **3** up to 0.44 ppm for **4**. At 230 K, the majority of the signals in the ^{1}H NMR spectra were very broad and of low intensity. The splitting of the proton donor group signal at 230 K for compounds **3**, **4**, **7**, **8** was connected with the presence of two adducts: 2 : 1 and 1 : 1. On the basis of the experiments with 2 : 1 molar ratio of compound **7** to Rh* both signals were assigned: the one at the lower field to the 2 : 1 adduct and the one at the higher field to the 1 : 1 adduct. An increase in temperature caused a shift of the adduct formation equilibrium towards the 1 : 1 adduct. The highest values of signal dispersion $\Delta\nu$ were observed for the adducts of compounds **3**–**8** in the aliphatic side-chain of the molecules (up to 30 Hz for **6** at 250 K) or methine proton (up to 40 Hz for **4** at 230 K) close to the anisotropic groups in the chiral Mosher acids residues (Table S1 and S2).[18] The signal dispersion ($\Delta\nu$) in range of 2.4–14 Hz was also observed for the aromatic protons. Dispersion $\Delta\nu$ was observed at least for two protons in compound **5** and up to 5 protons in compounds **3** and **7** (Table S1).

In ^{13}C NMR spectra at 230 and 250 K, some signals of the adducts were broadened and of low intensity while some disappeared in the baseline. The major changes in the chemical shifts observed for the C-2 signals (deshielding) indicated that the complexation site in the Schiff bases studied was the oxygen atom of the proton donor group (Table 1).[18] For compounds **5** and **6** existing in the NH-form, the $\Delta\delta$ values were close to 0.5 ppm, while for the compounds in which the proton transfer equilibrium takes place, **3**–**4** and **7**–**8**, they were in the range from ~3 up to 12 ppm. These major changes in the chemical shift observed for this position can be associated with changes in the position of the proton transfer equilibrium due to the presence of the dirhodium complex. The change in the position of the equilibrium was confirmed by changes in the values of deuterium isotope effects (e.g. $^{n}\Delta C$-2(D) for **4** at 270 K from +530 ppb to −230 ppb) and as well as in the values of ^{3}J(NH,H) coupling constants (Table 1). For adducts of **3**–**4** and **6**–**8** the $^{n}\Delta C$-2(D) values were negative and changed only slightly (*ca* 0–40 ppb) with lowering temperature (Table 1). This indicated the absence of the proton transfer equilibrium and the existence of the adducts exclusively in the NH-form (Fig. 3). Also, the almost temperature insensitive, high ^{3}J(NH,H) values in range of ~11–14 Hz confirmed the presence of the pure NH-form. A similar change in the position of the equilibrium due to the presence of the dirhodium complex was also observed for secondary phosphane oxides, in which the equilibrium was shifted towards the hydroxyphosphane form.[18] Smaller changes in the values of deuterium isotope effect were observed for the adduct of **5**.

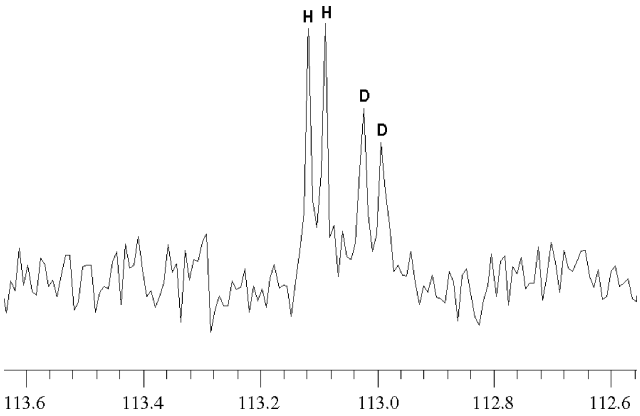

Figure 5. $^{n}\Delta C$-1(D) observed for adduct of **3** at 270 K.

The dispersion of the signals due to the presence of two diastereomers was observed for all compounds studied except **1** and **2**. The $\Delta\nu$ values in a range of 2.2–56 Hz were measured for the carbons of the aliphatic chain. The signals of the other carbons showed much smaller values of dispersion $\Delta\nu$; up to 16 Hz for C-α for compound **7** at 250 K. Dispersion $\Delta\nu$ was observed for a minimum of two carbons in compound **5** and up to nine carbons in compound **7** (Table S2).

The adducts of all compounds, except **1** and **2**, have shown interesting feature. Because of the presence of four species (deuterated and non-deuterated diastereomers of Schiff bases), two sets of deuterium isotope effects for some of the carbon atoms were observed in the presence of the dirhodium complex (Fig. 5). This feature was also recorded for the carbons remote from the chirality centre (Table S3 and 1).

The values of deuterium isotope effects were equal or very close to each other for both diastereomers. The small differences may be related to the conditions of the measurements, resolution of the spectra and accuracy of measurements of the deuterium isotope effects.

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CONCLUSIONS

The usefulness of the dirhodium method in chiral recognition of optically active Schiff bases existing in tautomeric equilibrium (**3**–**4** and **7**–**8**) or proton transferred form (**5**–**6**) has been demonstrated. The presence of the dirhodium complex shifted the proton transfer equilibrium towards the NH-form for the Schiff bases at equilibrium (**3**–**4** and **7**–**8**). The NH-form promoted the adduct formation and the binding site was the oxygen atom of the proton donor group. The signal shift due to the presence of dirhodium complex was small, *ca* 0.5 ppm, for the compounds in the NH-form (**5**–**6**) and larger for the compounds in which proton transfer takes place (**3**–**4** and **7**–**8**). The signal dispersion was observed not only at the proximity of the chiral centre. Schiff bases existing exclusively in the OH-form (**1**–**2**) favour the free ligand in adduct formation equilibrium. Combination of the dirhodium method with the measurements of deuterium isotope effects allowed discrimination between the signal shift due to the presence of Rh^* and the change in the chemical shift due to changes in the position of the proton transfer equilibrium.

Supplementary material

Supplementary electronic material for this paper is available in Wiley InterScience at: http://www.interscience.wiley.com/jpages/0749-1581/suppmat/

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