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Stereoselective synthesis, conformation and complexing behaviour of 1,2,3-trisubstituted chiral ferrocenes

Norbert Deus, Gerd Hübener, and Rudolf Herrmann *

Organisch-Chemisches Institut der Technischen Universität München, Lichtenbergstr. 4, D-8046 Garching (F.R.G)

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Abstract

Sulfur and phosphorus substituents have been introduced into the 2 and 5 positions of the ferrocene nucleus in chiral 1-(*N,N*-dimethylamino)ethylferrocene by stereoselective lithiation and reaction with electrophiles. The conformations of the diastereoisomeric trisubstituted ferrocenes obtained have been determined by NMR methods. The compounds behave as bidentate or monodentate ligands for transition metals, leaving one or two coordination sites for a further metal. NMR experiments suggest different site selectivity in the formation of nickel(II) and rhodium(I) complexes.

Introduction

The stereochemistry of the metallocenes, and especially of ferrocene derivatives, has been a topic of considerable interest for many years [1–3]. The reasons lie not only in the interesting chemistry involved, e.g. the extraordinary configurational stability of α-ferrocenyl alkyl carbocations [4–5] and the "planar" chirality of homoannular disubstituted metallocenes [6–7], but also in the use of the ferrocene derivatives as chiral ligands in asymmetric catalytic processes [8], such as hydrogenation [9], or Grignard cross coupling reactions [10]. Many modifications of Kumada's original ligand, the 2-(*N,N*-dimethylamino-1-ethyl)-1-diphenylphosphinoferrocene (**2**), in order to introduce more donating groups, have been reported, but this additional group has always been in the second cyclopentadienyl ring, or in the ethyl side chain [11]. Homoannular trisubstituted ferrocenes with a definite substitution pattern were prepared first by a metalation/alkylation sequence from 2-methyl-chloroferrocene, using the *ortho*-directing effect of the chloro substituent [12], but chiral compounds could not be obtained by this technique. For the synthesis of the diastereoisomer of Kumada's ligand, which is not formed by the normal metalation/phosphorylation sequence, a trimethylsilyl substituent was introduced first in the α-position, and by a second metalation, the diphenylphosphino

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substituent was placed in the α'-position. Finally, the silyl substituent was cleaved off by treatment with base [11]. Newer developments have involved the introduction of two to four trimethylsilyl moieties, and the crystal structure of 1,1',3-tris-(trimethylsilyl)-2(*N*,*N*-dimethylamino-1-ethyl)ferrocene has been reported [13]. From the point of view of applications as ligands for more than one metal centres, we thought it of interest to prepare chiral homoannular trisubstituted ferrocenes with at least three donor sites, and we now wish to report on the synthesis of this type of compounds, their conformation in solution, and their complexing properties.

Results and discussion

The synthesis of the compounds **3** and **5** is straightforward, and is illustrated in Fig. 1. The lithiation of (*R*)-(*N*,*N*-dimethylamino-1-ethyl)ferrocene (**1**) by butyllithium in ether is highly stereoselective, producing the (*R*,*R*)- and the (*R*,*S*)-2-lithio derivative in 92% diastereomeric excess [14–15]. This selectively is due to a chelating effect of the nitrogen lone pair which stabilizes the lithium on the nucleus in the (*R*,*R*)-diastereoisomer. The same effect was observed previously with other metallocenes such as cymantrene, but must be very subtle; replacement of the α-hydrogen by deuterium completely changes the site selectivity of the lithiation in cymantrene derivatives [16]. The reaction of the lithio compound with chlorodiphenylphosphine produces Kumada's ligand **2**, while sulfides **4** are formed by the reaction with

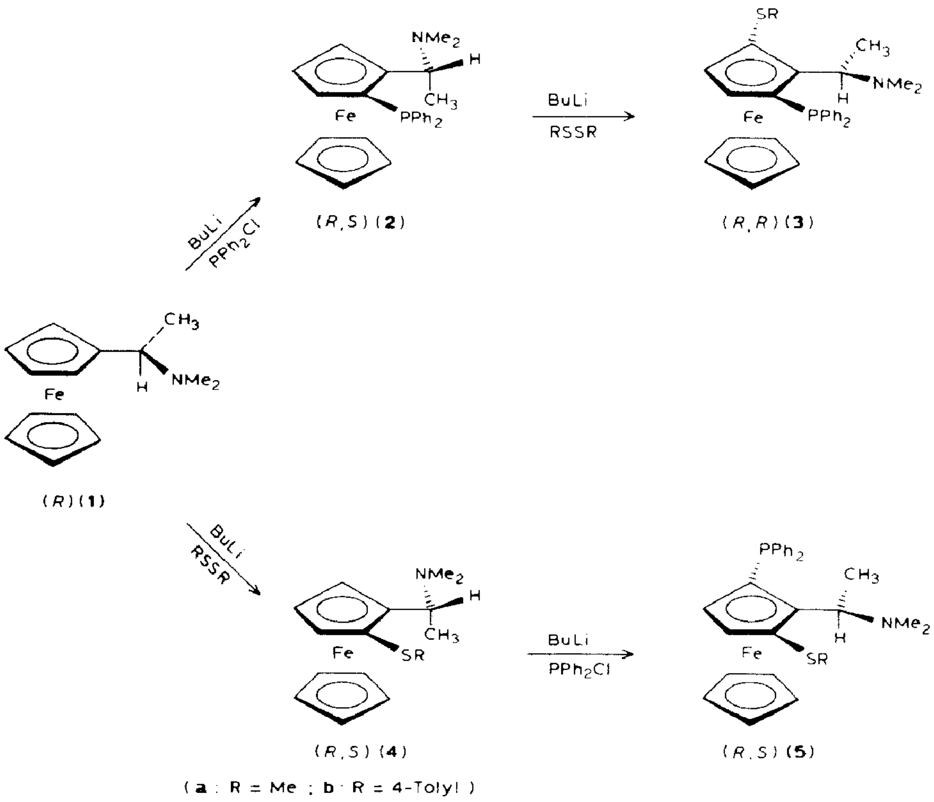


Fig. 1. Synthesis of the trisubstituted ferrocene derivatives 3 and 5.

Table 1

Yields and properties of trisubstituted ferrocenes **3** and **5** and the nickel(II) complex of **5b**

Comp.	Yield (%)	M.p. (°C)	$[\alpha]_D^{20}$ (c 1)	Formula	Analysis (found (calcd.) (%))		
					C	H	N
3a	29	oil	−100.6° (EtOH)	$C_{27}H_{30}FeNPS$	66.3	6.0	3.0
					(66.5)	(6.2)	(2.9)
3b	20	oil	+30.4° (EtOH)	$C_{33}H_{34}FeNPS$	70.0	6.1	2.2
					(70.3)	(6.0)	(2.5)
5a	40	90–93	−182.3° (EtOH)	$C_{27}H_{30}FeNPS$	66.4	6.3	2.9
					(66.5)	(6.2)	(2.9)
5b	62	52–53	+102.2° (EtOH)	$C_{33}H_{34}FeNPS$	70.2	6.0	2.4
					(70.3)	(6.0)	(2.5)
6	78	166–169	+133.8° ($CHCl_3$)	$C_{33}H_{36}Cl_2FeNNiOPS$	55.5	4.9	1.9
					(55.7)	(5.1)	(2.0)

disulfides [17]. The second lithiation is highly site selective as well, as no 3- or heteroannular substituted isomers of the desired compounds could be isolated, and the mixed phosphorus/sulfur ferrocenylethylamines were obtained optically pure. The results of the syntheses and the properties of the products are listed in Table 1.

In order to gain insight into the ability of the trisubstituted ferrocenes to form complexes with metals, we have studied their conformation in solution by NMR methods. The general features of the ^1H and ^{13}C NMR spectra are summarized in Table 2.

The ^1H NMR pattern is as expected for these compounds, but an interesting coupling to phosphorus is observed in the ^{13}C NMR spectra. Thus, in the two (R,R) diastereoisomers **3**, the CH of the ethyl side chain shows a large coupling constant J(C–P) (12.0 and 8.8 Hz, respectively), while the methyl group shows only a small coupling, if any, formally over four bonds (3.0 and < 0.1 Hz, respectively). The situation is reversed in the case of the (R,S)-diastereoisomers **5**: No couplings of phosphorus with the CH groups (formally three bonds) is observed, but there are large couplings to the methyl group (16.8 and 13.2 Hz, respectively), formally over four bonds.

There are some examples in the literature of long range couplings of phosphorus to other nuclei, but there is no generally accepted theory of the coupling mechanism. Interactions through bonds as well as through space have been discussed [18]. Summarizing published data, Quin has suggested that the main mechanism for long range couplings is through space, and that the direction of the phosphorus lone pair determines the magnitude of the coupling [19]. Atoms to which the lone pair is directed, show larger couplings than atoms from which the lone pair is turned away. We therefore conclude that the most stable conformations of the trisubstituted ferrocenes are as depicted in Fig. 2, with the hydrogen (as the least bulky substituent of the ethyl CH) pointing downwards to the ferrocene (*endo*). In these conformations, the phosphorus lone pair is directed clearly towards the methyl group in the (R,S) isomers, whereas in the (R,R) compounds, it points towards the carbon atom of the CH group. Interpretation of the coupling pattern by analogy to the well-known W coupling of protons [20] is not very convincing, as the structure has not much similarity to the W shape. The different substituents at sulfur (methyl vs. 4-methyl-

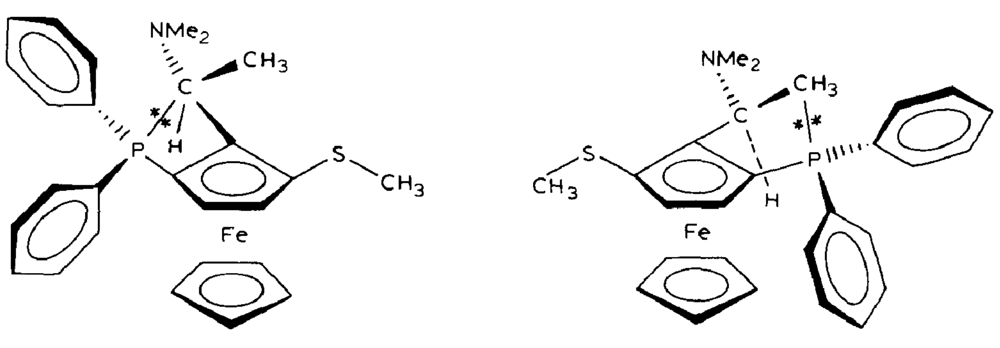
Table 2

NMR data for the compounds **3**, **5**, **6**, and the rhodium(I) complex **7** of ligand **5a**. δ values in $CDCl_3$. Coupling constants H–H in ^1H NMR, C–P in ^{13}C NMR

Comp.		CpH	Cp(4,5)(^1H) Cp(1–5)(^{13}C)	Me	CH	NMe_2	PPh_2	*R*
3a	^1H	3.97	3.87 4.35	1.64 (7.0 Hz)	4.11	1.82	7.05–7.60	2.34
	^{13}C	71.0	70.5 70.8 77.0 86.4 (6.5 Hz) 97.0 (19.0 Hz)	12.8 (3.0 Hz)	59.1 (12.0 Hz)	40.3	132.4 135.2 (both 23 Hz) 126.0–129.0	19.3
	^{31}P						−23.1	
3b	^1H	4.03	3.94 4.43	1.50 (7.2 Hz)	4.33	1.64	7.14–7.58	7.00 7.1 2.24
	^{13}C	71.4	72.3 (3.1 Hz) 76.9 79.9 80.8 (11.2 Hz) 102.4 (19.8 Hz)	10.1 (< 0.1 Hz)	59.3 (8.8 Hz)	39.2	127.1–140.6 (overlap, not resolved)	20.9
	^{31}P						−21.9	
5a	^1H	3.92	3.88 4.46	1.89 (7.0 Hz)	3.68	1.84	7.21 7.30 750	2.27
	^{13}C	78.0	72.0 76.2 (21.5 Hz) 86.6 (5.0 Hz) 99.8 (22.0 Hz)	17.8 (16.8 Hz)	58.3 (< 0.1 Hz)	42.2	127.5 132.7 134.9 138.8 139.5 (both 22 Hz)	19.6
	^{31}P						−22.1	
5b	^1H	4.00	4.04 4.64	1.92 (7.0 Hz)	3.46	1.62	7.20–7.60	7.01 7.11 2.27
	^{13}C	71.3	72.1 (5.3 Hz) 76.1 77.9 79.6 (5.3 Hz) 102.4 (19.8 Hz)	20.0 (13.2 Hz)	57.0 (< 0.1 Hz)	42.6	127.3–139.9 (overlap, not resolved)	20.9
	^{31}P						−22.4	
6	^1H	3.93	4.52 5.07	2.50	5.00	2.07 2.16	7.07–7.56	2.27
	^{13}C	72.5	75.8 78.8 (26.2 Hz) 80.3 81.6 86.7 (25.0 Hz)	19.5 (21.2 Hz)	60.0	36.1 42.4	126.2–138.2 (assignment impossible)	20.8
	^{31}P						−23.8	
7	^1H	4.08	4.00 4.53	1.31	3.78	2.82	7.30–7.60 8.26	2.35
	^{13}C	71.6	69.6 (12.0 Hz) 72.5 85.8 94.4 (15.0 Hz)	18.3	58.6	42.9	127.0–126.3 (overlap, not resolved)	20.7
	^{31}P						+26.2 (*J*(Rh–P) 125 Hz)	

phenyl) are responsible only for a fine tuning of the conformation, but do not change its general features.

To confirm these results, we examined NOE effects by the two dimensional technique in the rotating frame (ROESY [21–24]). In the (*R*,*R*) isomers, the phenyl groups of the diphenylphosphino moiety show a strong NOE effect for the methyl groups of the dimethylamino substituent, whereas in the case of the (*R*,*S*) compounds they show a strong interaction with the methyl group of the $CH-CH_3$ unit. This confirms the conformations shown in Fig. 2. Only weak NOE effects are found between the dimethylamino group and the unsubstituted cyclopentadienyl ring,



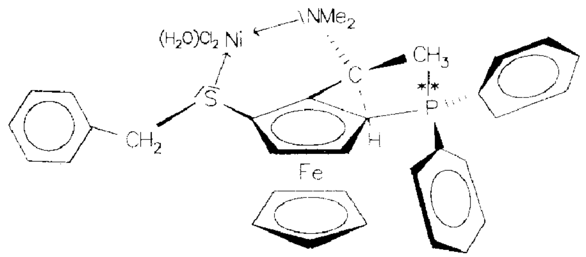
(*R*,*R*) (**3a**)　　(*R*,*S*) (**5a**)

Fig. 2. Conformations of the compounds **3** and **5**, as indicated by NMR data.

suggesting that this substituent does, indeed, avoid the sterically crowded *endo* region of the ferrocene system. The NOE effects between the methyl of the $CH-CH_3$ unit and the cyclopentadienyl are only slightly stronger, and suggest that this group also has an *exo* tendency. This corresponds exactly to the results for the disubstituted compounds **2** and **4**. On the other hand, stronger interactions occur between the methyl groups at sulfur and nitrogen in the (*R*,*S*) diastereoisomers **5**, in exactly the same way as in the disubstituted derivatives **4**. In **3a**, there is a strong NOE between the dimethylamino and the diphenylphosphino group, just as in Kumada's ligand **2**, but none between the methyl groups at sulfur and nitrogen. Thus, the conformation of the trisubstituted ferrocenes closely resembles that of the corresponding disubstituted starting compounds.

The properties of the trisubstituted ferrocenes as ligands for metals must also be influenced by the conformations. It seems evident from Fig. 2 that the (*R*,*R*) isomers should be able to form chelates with phosphorus and nitrogen without great conformational changes, leaving the sulfur free for further coordination. The reverse can be expected for the (*R*,*S*) compounds, which should prefer to form chelates with sulfur and nitrogen. The site selectivity can be expected, however, to depend strongly on the metal chosen. We have prepared complexes of **5a** with rhodium(I) and of **5b** with nickel(II), which seemed of considerable interest in respect of possible catalytic applications of the ligands, to study the selectivity problem.

The nickel(II) complex **6** was prepared by reaction of the ligand **5b** with hydrated $NiCl_2$ in ethanol, and obtained as stable solid. The most interesting features of its NMR spectra are the appearance of the signal from the CH_3 carbon of the ethyl group as a doublet with J(C–P) 21.2 Hz, which clearly shows that the phosphorus lone pair is not involved in bonding but still points towards the methyl carbon, and the strong splitting of the signals from the CH_3 groups of the dimethylamino residue, which appear as very broad signals at δ 36.1 and 42.4 ppm. In the proton spectra the corresponding signals are observed at 2.07 and 2.16 ppm. Similar splitting effects have been found for ferrocene derivatives with sulfur substituents in cases where there is chelation [25], and we thus assume that both sulfur and nitrogen coordinate to nickel, leaving the phosphorus free, just as expected for the most stable conformation of the ligand. The conclusion is supported by the fact that the ^{31}P resonance appears at nearly the same position as that for the free ligand (δ



6

Fig. 3. Probable structure of the nickel complex **6**, as indicated by NMR data.

−23.8 ppm). The assignment of the broad signals in the ^1H NMR is based on a two-dimensional CH correlation [26,27], and listed in Table 2. The probable structure, as derived from the NMR data, is shown in Fig. 3.

Coordination to rhodium(I) was studied under conditions similar to those used for asymmetric hydrogenation. Thus, $[Rh(COD)Cl]_2$ (COD = 1,5-cyclooctadiene) was treated with two equivalents of the ligand **5a** in methanol, and after the evaporation of the solvent, the product was dissolved in $CDCl_3$ for NMR measurements. A broad doublet is observed in the ^{31}P NMR spectrum at δ 25.5 ppm, with $^1J(P-Rh)$ 125 Hz (in addition to a signal from the uncomplexed ligand (δ −22.6 ppm)), a value expected for phosphorus coordinated to rhodium [28–31]. The solution deterioates rapidly; after some hours, three additional doublets can be detected (δ 17.7 and 21.4 ppm). The question of whether chelation has occurred is more difficult to answer. The ^{13}C NMR spectrum confirms the coordination of phosphorus to rhodium, since there is no long range coupling to the CH_3 of the ethyl group. The NMe_2 carbons give rise to a broad signal at 42.9 ppm, but with no significant splitting. This group also gives a singlet in the proton spectrum. We therefore suggest that the nitrogen is not involved in coordination, although the extreme broadening of th NMe_2 carbons and the CH of the ethyl group is difficult to explain. As in the case of the nickel complex, the ^1H NMR spectrum shows broadened signals, which were assigned by a combination of CH correlation and COSY spectra, the data are shown in Table 2, but are not sufficient to allow assignment of a definite structure to the complex; if the ligand is only monodentate, the complex could still be dimeric, as is the starting material $[Rh(COD)Cl]_2$.

Our conclusion is that conformational effects do play an important role in determining the coordination mode of such trisubstituted ferrocene derivatives. Although chelation of phosphorus and nitrogen with rhodium might be expected, it probably does not occur because it would result in the methyl group and not the hydrogen of the CH_3–CH unit pointing downwards (*endo*), resulting in severe crowding in the region of the unsubstituted cyclopentadienyl ring. Tridentate chiral ligands with phosphorus, sulfur, and nitrogen binding sites derived from amino acids have been shown to form P–N-chelates with $PdCl_2$ since the comparatively flexible structure does not introduce steric effects [32]. On the other hand, chelation with sulfur and nitrogen does not require any substantial change in the conformation of the ligand, and occurs readily with nickel.

We have examined the possibility of using the ligands in asymmetric hydrogenations with rhodium. Compared with, e.g., Kumada's ligand, the introduction of the sulfur substituent slows down the reaction rate considerably; several days are required for the completion of the hydrogenation of *N*-acetyl-2-amino-cinnamic acid. The enantiomeric excess does not exceed 70%, and the ligands are therefore of no practical interest in asymmetric hydrogenations. Their behaviour in Grignard cross coupling reactions [32] is currently under investigation, as well as the coordination of a second metal to the binding sites left. Preliminary results show that silver(I) can be introduced into the nickel complex in the case of **5b**, and the resulting compound exhibits highly complex redox properties as indicated by cyclovoltammetry. Performing two subsequent reactions on the same substrate is a conceivable application of such trinuclear compounds.

Experimental

NMR spectra were recorded with Bruker WP 200 and Bruker AM 360 spectrometers. Optical rotations were measured with a Roussel-Jouan Digital 71 polarimeter. Reactions involving lithiated compounds were performed under nitrogen.

The starting compound (*N*,*N*-dimethylamino-1-ethyl)ferrocene was pepared [33] and resolved [34] as previously described, to give enantiomerically pure (*R*)-(*N*,*N*-dimethylamino-1-ethyl)ferrocene **1** ($[\alpha]_D^{22} = +14.8°$ (*c* 1.0, ethanol)). Subsequent metalation and reaction with chlorodiphenylphosphine gave **2** [11], or with dimethyl disulfide **4a** and with di-(4-methylphenyl)disulfide **4b** [17].

(R,R)-1-Methylthio-3-diphenylphosphino-2-(N,N-dimethylamino-1-ethyl)ferrocene 3a and (R,R)-1-(4-methylphenyl)thio-3-diphenylphosphino-2-(N,N-dimethylamino-1-ethyl)-ferrocene 3b

To a solution of 4.41 g (10 mmol) of (*R*,*S*)-1-diphenylphosphino-2-(*N*,*N*-dimethylamino-1-ethyl)ferrocene (**2**) in 50 ml of dry diethyl ether were added 10.0 ml (15 mmol) of a 1.5 *M* solution of butyllithium in hexane. The mixture was heated under reflux for 2 h then a solution of 15 mmol of the disulfide (dimethyl disulfide to form **3a**, di-(4-methylphenyl)disulfide to form **3b**) in 50 ml of dry diethyl ether was added dropwise at room temperature. Stirring was continued for 18 h then 50 ml of water were added, and the organic layer was shaken with 1 *n* NaOH (40 ml) and water (50 ml). The organic layer was separated, dried (Na_2SO_4), and evaporated. The residue was purified by chromatography [silica gel 60 (0.063–0.200 mm) (100 g), hexane/diethyl ether 5/1]. R_f(**3a**): 0.75; R_f(**3b**): 0.70. Yields and properties are listed in Table 1, and NMR spectra in Table 2.

(R,S)-1-Methylthio-3-diphenylphosphino-2-(N,N-dimethylamino-1-ethyl)ferrocene 5a and (R,S)-1-(4-methylphenyl)thio-3-diphenylphosphino-2-(N,N-dimethylamino-1-ethyl)-ferrocene 5b

To a solution of 10 mmol of (*R*,*S*)-1-methylthio-2-(*N*,*N*-dimethylamino-1-ethyl)ferrocene **4a** or (*R*,*S*)-1-(4-methylphenyl)thio-2-(*N*,*N*-dimethylamino-1-ethyl)ferrocene **4b** in 50 ml of dry diethyl ether were added 12 ml (18 mmol) of a 1.5 *M* solution of butyllithium in hexane. The mixture was refluxed for 4 h then a solution of chlorodiphenylphosphine (7.9 g, 18 mmol) in 80 ml of dry diethyl ether was added dropwise at room temperature. Stirring was continued for 16 h, 50 ml of water were added, and the organic layer was shaken twice with water (50 ml) then dried (Na_2SO_4). After evaporation of the solvent, the residue was recrystallized from hexane. Yields and properties see Table 1, NMR spectra Table 2.

*Nickel(II) complex **6** of ligand **5b***

To a solution of 2.87 g (5 mmol) of **5b** in 30 ml of ethanol was added a solution of 1.16 g (4.9 mmol) of $NiCl_2(H_2O)_6$ in 20 ml of ethanol. After 20 min stirring the solution was concentrated to 15 ml and 100 ml of hexane were added. The solid which separated was filtered off and purified by dissolution in ethanol and reprecipitation by addition of cyclohexane. Yield and properties are listed in Table 1, NMR spectra in Table 2.

*Rhodium(I) complex of **5a** and asymmetric hydrogenation*

To a solution of 50.2 mg (0.114 mmol) of $[Rh(COD)Cl]_2$ (COD = 1,5 cyclooctadiene) in 50 ml of ethanol, 111.0 mg (0.228 mmol) of **5a** were added under nitrogen. For the recording of NMR spectra the mixture was stirred for 20 min, volatile compounds then evaporated off under vacuum, and the residue dissolved in $CDCl_3$. The results are shown in Table 2. For asymmetric hydrogenation, 4.10 g (20 mmol) of *N*-acetyl 2-aminocinnamic acid were added and the nitrogen was replaced by hydrogen. The hydrogen uptake was complete after 5 d, and 10 g of strongly acidic ion exchange resin (Dowex 50 W) was added to the mixture and stirring was continued for 1 h. The solids were filtered off and washed carefully with hot ethanol and the combined ethanolic solutions were evaporated. The residue was extracted with hexane, and the residual *N*-acetyl-phenylalanine (3.73 g, 90%) was analyzed by polarimetry (ee 70%).

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