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**SULPHONATES AND CARBOXYLATES OF (*E*)- AND (*Z*)-3-(HYDROXY-METHYLENE)-*trans*-HEXAHYDRO-2(3*H*)-BENZOFURANONE**

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Received April 29th, 1986

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Sodium salt of 3-(hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone has been found to be formed as the (*Z*)-isomer in solid state and to exist as the (*E*)-isomer or a mixture of the both isomers in polar solvents. Some sulphonates and carboxylates of (*E*)-3-(hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone and (*Z*)-3-(hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone have been prepared by reactions of the corresponding isomers of this sodium salt with sulphonyl and acyl chlorides, respectively. The configuration of the isomeric derivatives was confirmed by  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra.

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In the previous papers<sup>1,2</sup> we dealt with behaviour and configuration of sodium salts of 3-(hydroxymethylene)dihydro-2(3*H*)-furanone (*I*) and 3-(hydroxymethylene)-5-methyldihydro-2(3*H*)-furanone (*II*). Now we have tried to apply our experience to the study of configuration and to preparation of derivatives of sodium salt of 3-(hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone (*III*), which was prepared for the first time by Minato and Horibe<sup>3</sup> during a synthesis of 3-methylene-*trans*-hexahydro-2(3*H*)-benzofuranone. Later sulphonylations<sup>4,5</sup> of this salt gave sulphonates with various ratios of the (*E*)- and (*Z*)-isomers which were separated by means of chromatography or crystallization. The previous papers dealing with the sodium salt *III* did not contain any discussion of its configuration.

Our results showed that the sodium salt *III* prepared in principle according to ref.<sup>6</sup> is formed in only one modification, *viz.* as the (*Z*)-isomer. Its configuration, in solid state in the absence of air humidity, is stable in contrast to that of the salts *I* and *II*, and it is not changed into the (*E*)-isomer even after several hours heating at 110°C or after suspending or dissolving in polar solvent and recuperation of the solid by evaporation of the solvent. But in polar solvents (aqueous acetone, aqueous tetrahydrofuran, methanol) an equilibrium is established which, depending on the solvent type, is markedly shifted in favour of the (*E*)-isomer. In aqueous solution the sodium salt *III* exists exclusively as the (*E*)-isomer. An addition of lithium salt (*e.g.*, LiCl) to methanolic solution of the sodium salt *III* causes an increase in the amount of the (*Z*)-isomer, which agrees with previous papers<sup>7,8</sup> describing the behaviour of alkali  $\beta$ -oxoenolates. The retention of configuration in solid state was

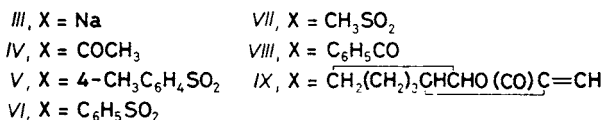
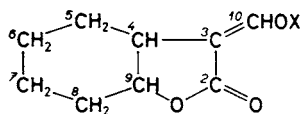
followed by means of IR spectra (mainly in the region of  $700-800\text{ cm}^{-1}$ ), which contain the out-of-plane deformation vibrations of the  $=\text{C}-\text{H}$  bond, and by comparison of the Debye-Scherrer diagrams which were identical for all the sodium salts *III* of different origin. The proportion of the individual isomers in aqueous and methanolic solutions was followed by means of  $^1\text{H}$  NMR spectra, the vinylic proton signal being used as decisive; owing to the anisotropy the (*E*)-isomer is shifted by 0.6 ppm downfield as compared with the corresponding signal of the (*Z*)-isomer. The results of preparations also agree with the proportions observed with the isomers of the sodium salt *III*. If the sodium salt *III* is suspended in anhydrous diethyl ether and treated with acetyl chloride at  $-15^\circ\text{C}$ , the only product is (*Z*)-3-(acetoxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*Z*)-*IV*). If, however, the sodium salt *III* is dissolved in a 2 : 1 mixture of tetrahydrofuran and water and the reaction is carried out in the presence of equimolar amount of triethylamine at room temperature, then the only product is (*E*)-3-(acetoxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*IV*) (*cf.* ref.<sup>9</sup>).

The results obtained were applied to elaboration of preparation methods of the pure (*E*)- and (*Z*)-carboxylates and -sulphonates of *III* (refs<sup>10,11</sup>). The reactions of solutions of the sodium salt *III* in aqueous tetrahydrofuran (2 : 1) with solutions of 4-toluenesulphonyl chloride, benzenesulphonyl chloride, methanesulphonyl chloride, acetyl chloride, and benzoyl chloride in the same solvent in the presence of equimolar amount of triethylamine gave pure (*E*)-3-(4-toluenesulphonyloxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*V*), (*E*)-3-(benzenesulphonyloxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*VI*), (*E*)-3-(methanesulphonyloxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*VII*), (*E*)-*IV*, and (*E*)-3-(benzoyloxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*VIII*), respectively. Besides the derivatives mentioned a certain amount of (*E, E*)-3,3'-(oxydimethylidene)bis(*trans*-hexahydro-2(3*H*)-benzofuranone) ((*E, E*)-*IX*) was also formed in the reactions, reaching up to 12% yield in the case of the methanesulphonate (*E*)-*VII*. The ether (*E, E*)-*IX* can be prepared in high yield by the reaction of the sodium salt *III*, *e.g.*, with (*E*)-*V* in aqueous acetone.

If the suspension of sodium salt *III* in anhydrous diethyl ether is treated with the corresponding sulphonyl or carbonyl chlorides at  $-10$  to  $-15^\circ\text{C}$ , the reaction gives the respective pure isomers (*Z*)-*V*, (*Z*)-*VI*, (*Z*)-*VII*, (*Z*)-*IV*, and (*Z*)-*VIII*. No formation of the ether (*E, E*)-*IX* or of its isomers (*E, Z*)-*IX* and (*Z, Z*)-*IX* was observed in this reaction. The ether (*E, Z*)-*IX* can be prepared (in a mixture with (*E, E*)-*IX*) by the reaction of (*Z*)-*V* with the sodium salt *III* in aqueous acetone.

The substances prepared were identified by means of  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra whose characteristic signals indicated (as in the previous papers<sup>1,2</sup>) the configuration at the double bond. In the  $^1\text{H}$  NMR spectra the decisive signals for determination of the configuration are those of the vinylic protons which are shifted downfield by

about 0.70 to 0.90 ppm in the case of the (*E*)-isomers. Some of the derivatives also exhibit a marked difference in the magnitude of the  $^4J_{\text{H,H}}$  allyl spin-spin coupling constant<sup>12</sup>. The values of chemical shifts of the characteristic signals are given in Table I.



The <sup>13</sup>C NMR spectra show indicative differences in chemical shifts of C(2) and C(10) carbon atoms: with the (*E*)-isomer the C(2) carbon atom signal is shifted downfield by 3.0 to 4.3 ppm and that of C(10) by 4.1 to 5.0 ppm. The spectra of the

TABLE I  
Characteristic signals in the <sup>1</sup>H NMR spectra of the compounds prepared<sup>a</sup>

Compound	$\delta(\text{C}=\text{C}-\text{H})$	$^4J_{\text{H,H}}$	$\delta(\text{CH}_3)$
( <i>E</i> )-III <sup>b</sup>	8.54	2.5	—
( <i>Z</i> )-III <sup>b</sup>	7.93	1.0	—
( <i>E</i> )-IV	8.11	3.0	2.18
( <i>Z</i> )-IV	7.36	2.8	2.28
( <i>E</i> )-V	7.53	3.0	—
( <i>Z</i> )-V	6.72	2.8	—
( <i>E</i> )-VI	7.57	3.0	—
( <i>Z</i> )-VI	6.66	3.0	—
( <i>E</i> )-VII	7.51	3.0	3.12
( <i>Z</i> )-VII	6.74	3.0	3.17
( <i>E</i> )-VIII	8.41	3.0	—
( <i>Z</i> )-VIII	7.60	3.0	—
( <i>E, E</i> )-IX	7.37	3.0	—
( <i>E, Z</i> )-IX <sup>c</sup>	7.39	3.0	—
	6.48	2.6	—

<sup>a</sup> The spectra were measured in C<sup>2</sup>HCl<sub>3</sub>, if not otherwise stated; <sup>b</sup> measured was the (*E*) and (*Z*) isomers mixture in a mixture of <sup>2</sup>H<sub>2</sub>O and hexadeuterioacetone (1 : 2); <sup>c</sup> the upper and the lower values correspond to the (*E*) and (*Z*) parts of ether IX, resp.

substances prepared are given in Table II. The C(4) through C(9) carbons signals of the cyclohexane ring were assigned with the help of comparison of the values measured for the 3-methylenesubstituted lactones *IV–VIII* with the chemical shift values of the corresponding atoms in the non-substituted *trans*-hexahydro-2(3*H*)-benzofuranone (*X*) which were assigned with the use of the shift increments for the conformation-stabilized cyclohexanes according to Schneider and Hoppen<sup>13</sup>. Owing to scarcity of the shift increment values, the molecule of *e,e*-*trans*-2-methylcyclohexyl acetate was used as a model in the calculation. The calculated and the found values are compared in Table III. An insufficiency of the model made itself felt in the assignment of the signals of the C(5) and C(8) atoms, which was caused by operation of some other effects different from the electronic ones which are simulated relatively well by the model used. However, we presume that introduction of a substituent into the 3-position of lactone *X* will affect the chemical shift of the C(5) carbon atom more strongly: the signal of C(5) will show greater changes with changing configuration of the substituent than that of C(8). The signal assigned to C(5) exhibits a distinct downfield shift in the (*E*)-isomers, *viz.* by 0.7 to 1.2 ppm as compared with the (*Z*)-isomers. The differences between C(5) and C(8) in the coupling constants  $^1J_{C,H}$  and  $^1J_{C,C}$  are not convincing, but the lanthanoid-induced shift of the  $^{13}\text{C}$  NMR

TABLE II  
 $^{13}\text{C}$  NMR spectra<sup>a</sup> of the compounds prepared (the atoms of the methylenelactone skeleton)

Compound	C(2)	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)	C(10)
( <i>E</i> )- <i>IV</i>	171.00	115.35	47.64	28.81	25.35	23.93	30.32	82.40	140.69
( <i>Z</i> )- <i>IV</i>	166.94	114.86	46.89	25.95	24.64	23.78	30.20	83.25	136.59
( <i>E</i> )- <i>V</i>	169.81	118.74	47.52	26.43	25.13	23.75	30.17	83.40	141.33
( <i>Z</i> )- <i>V</i>	165.66	118.71	47.08	25.76	24.57	23.67	30.17	82.91	136.51
( <i>E</i> )- <i>VI</i>	169.81	119.04	47.52	26.40	25.13	23.71	30.13	83.43	141.25
( <i>Z</i> )- <i>VI</i>	165.48	118.92	46.85	25.50	24.31	23.45	29.94	82.76	136.25
( <i>E</i> )- <i>VII</i>	169.77	118.56	47.56	26.51	25.17	23.78	30.17	84.47	140.99
( <i>Z</i> )- <i>VII</i>	166.72	119.72	47.23	25.81	24.64	23.75	30.32	83.17	136.18
( <i>E</i> )- <i>VIII</i>	170.85	115.87	47.71	27.07	25.46	23.90	30.28	83.32	140.95
( <i>Z</i> )- <i>VIII</i>	167.23	115.31	46.82	25.91	24.64	23.78	30.24	83.17	136.55
( <i>E, E</i> )- <i>IX</i>	171.04	113.71 <sup>b</sup>	47.52	26.99	25.50	23.97	30.28	83.55	149.95 <sup>b</sup>
		113.59							149.88
( <i>E, Z</i> )- <i>IX</i> <sup>c</sup>	171.53	113.59	47.71	26.77	26.02	24.64	30.31	84.03	149.50
	167.08	112.88	46.66	26.43	25.50	23.82	30.20	83.39	145.77

<sup>a</sup> Measured were 0.5M solutions in  $\text{C}^2\text{HCl}_3$ ; experimental error  $\pm 0.03$  ppm; <sup>b</sup> the presence of both the possible diastereomers makes itself felt; <sup>c</sup> the upper and the lower values correspond to the (*E*) and (*Z*) parts of the ether, resp.

spectra of lactone *X* measured with  $\text{Eu}(\text{FOD})_3$  supports our assignment by a greater signal shift for C(8) than for C(5). The values obtained by measuring the NMR spectra with the shift reagent are presented in Table III.

### EXPERIMENTAL

The temperature data are not corrected. The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured with a Tesla BS 467 (60 MHz) and a Tesla BS 567 (100.034 and 25.156 MHz) apparatus, respectively, hexamethyldisiloxane or sodium 4,4-dimethyl-4-silapentanesulphonate being used as the internal standards. The chemical shifts are related to tetramethylsilane. The lanthanoid-induced shifted NMR spectra were measured in  $\text{C}^2\text{HCl}_3$  with tris(1,1,1,2,2,3,3-heptafluoro-7,7-dimethyl-4,6-octadionato)europium ( $\text{Eu}(\text{FOD})_3$ ). The slope of the dependence on the concentration ratio of the shift reagent and lactone *X* was calculated from three measurements with  $[\text{Eu}(\text{FOD})_3]/[\text{lactone}] = 0.13, 0.22, 0.33$ . The IR spectra were measured with a PE-783 apparatus in Nujol suspensions. The powder diagrams were obtained in a chamber of 57.3 mm diameter with  $\text{Cu K}_\alpha$  radiation, the tube voltage of 40 kV, 30 mA current, 10 h exposition. The chemical shifts

TABLE III

Comparison of the chemical shifts ( $^{13}\text{C}$  NMR) calculated for *e,e*-*trans*-2-methylcyclohexyl acetate with the measured spectrum of lactone *X*, and the slope *k* of the dependence of induced shift of individual carbon atoms on the concentration ratio of the shift reagent and lactone *X*

Atom	C(3)	C(4)	C(5)	C(6)	C(7)	C(8)	C(9)
$\delta_{\text{calc.}}$	34.6 <sup>a</sup>	38.6 <sup>b</sup>	33.5	25.3	24.6	32.0	81.7
$\delta_{\text{exp.}}^c$	35.7	44.8	28.3	25.3	24.0	30.2	85.1
$k^d$	11.87	6.60	2.64	1.70	1.70	3.21	8.86

<sup>a</sup>  $\delta(\text{CH}_2)$  of *trans*-decaline was used; <sup>b</sup>  $\delta(\text{CH})$  of *trans*-decaline is 44.0; <sup>c</sup> measurements in  $\text{C}^2\text{HCl}_3$ ; <sup>d</sup> ppm.

TABLE IV

The additive increments for calculation of chemical shifts of the signals in the  $^{13}\text{C}$  NMR spectra of substituted cyclohexanes

Substituent	Position <sup>a</sup>			
	1	2	3	4
$\text{CH}_3$	5.9	8.2	-0.7	-0.7
$\text{OCOCH}_3$	45.8	5.0	-2.4	-1.7

<sup>a</sup> The substituent always is in the equatorial position.

of the  $^{13}\text{C}$  NMR spectra of *e,e*-*trans*-2-methylcyclohexyl acetate were calculated from the relation

$$\Delta\delta_i = B + \sum_j A_{i,j},$$

where  $B$  means the chemical shift of cyclohexane (27.7 ppm), and  $A_{i,j}$  are additive increments of substituents depending on their position. The  $A_{i,j}$  values used are given in Table IV.

TABLE V  
The derivatives of (*E*)- and (*Z*)-3-(hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone prepared

Compound	M.p., °C (solvent)	Yield %	Formula (m. mass)	Calculated/found	
				% C	% H
( <i>E</i> )- <i>IV</i>	oil	70	$\text{C}_{11}\text{H}_{14}\text{O}_4$ (210.2)	62.85 62.79	6.71 6.68
( <i>Z</i> )- <i>IV</i>	65–68.5 ( $\text{C}_6\text{H}_{14}$ )	62	$\text{C}_{11}\text{H}_{14}\text{O}_4$ (210.2)	62.85 62.81	6.71 6.62
( <i>E</i> )- <i>V</i>	87–89 ( $\text{C}_6\text{H}_6$ )	65	$\text{C}_{16}\text{H}_{18}\text{O}_5\text{S}$ (322.4)	59.61 59.55	5.63 5.53
( <i>Z</i> )- <i>V</i>	157–158.5 ( $\text{C}_6\text{H}_{14} + \text{CHCl}_3$ )	80	$\text{C}_{16}\text{H}_{18}\text{O}_5\text{S}$ (322.4)	59.61 59.48	5.63 5.50
( <i>E</i> )- <i>VI</i>	44–52	67 <sup>a</sup>	$\text{C}_{15}\text{H}_{16}\text{O}_5\text{S}$ (308.3)	58.43 58.46	5.23 5.11
( <i>Z</i> )- <i>VI</i>	115–117.5 ( $\text{C}_6\text{H}_{14} + \text{CHCl}_3$ )	62	$\text{C}_{15}\text{H}_{16}\text{O}_5\text{S}$ (308.3)	58.43 58.32	5.23 5.08
( <i>E</i> )- <i>VII</i>	103–106 ( $\text{C}_4\text{H}_{10}\text{O} + \text{C}_2\text{H}_5\text{OH}$ )	60	$\text{C}_{10}\text{H}_{14}\text{O}_5\text{S}$ (246.3)	48.77 48.61	5.73 5.69
( <i>Z</i> )- <i>VII</i>	108–109 ( $\text{C}_4\text{H}_{10}\text{O}$ )	63	$\text{C}_{10}\text{H}_{14}\text{O}_5\text{S}$ (246.3)	48.77 48.80	5.73 5.58
( <i>E</i> )- <i>VIII</i>	163–165 ( $\text{C}_4\text{H}_{10}\text{O} + \text{C}_2\text{H}_5\text{OH}$ )	88	$\text{C}_{16}\text{H}_{16}\text{O}_4$ (272.3)	70.58 70.42	5.92 5.83
( <i>Z</i> )- <i>VIII</i>	136–139 ( $\text{C}_4\text{H}_{10}\text{O} + \text{C}_2\text{H}_5\text{OH}$ )	72	$\text{C}_{16}\text{H}_{16}\text{O}_4$ (272.3)	70.58 70.51	5.92 5.76
( <i>E, E</i> )- <i>IX</i>	250–252 ( $\text{C}_2\text{H}_5\text{OH}$ )	90	$\text{C}_{18}\text{H}_{22}\text{O}_5$ (318.4)	67.91 68.05	6.97 6.96
( <i>E, Z</i> )- <i>IX</i>	256.5–257 ( $\text{C}_2\text{H}_5\text{OH}$ )	44	$\text{C}_{18}\text{H}_{22}\text{O}_5$ (318.4)	67.91 68.07	6.97 6.94

<sup>a</sup> Purified by column chromatography.

The starting *trans*-hexahydro-2(3*H*)-benzofuranone (*X*) was prepared according to ref.<sup>14</sup> by reaction of cyclohexene oxide with diethyl malonate. B.p. 133°C at 1.6 kPa.  $n_D^{20} = 1.4772$ .

Sodium Salt of 3-(Hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone (*III*)

A solution of 7.0 g (0.05 mol) lactone *X* and 5 ml (0.06 mol) ethyl formiate in 150 ml diethyl ether was added dropwise to a suspension of 1.2 g (0.05 mol) sodium in 150 ml anhydrous diethyl ether with stirring. After 20 h stirring, the cream-coloured precipitate was collected by suction, washed with 3 × 50 ml anhydrous diethyl ether, and dried in a dessiccator. Yield 9.45 g (99%) sodium salt (*Z*)-*III*. IR spectrum (Nujol): 720, 740, 783  $\text{cm}^{-1}$ .

Sulphonates and Carboxylates of (*E*)-3-(Hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*IV*, (*E*)-*V*, (*E*)-*VI*, (*E*)-*VII*, (*E*)-*VIII*)

A solution of 0.02 mol sodium salt *III* and 3 ml triethylamine in 90 ml tetrahydrofuran-water mixture (2 : 1) was added dropwise to a solution of 0.02 mol corresponding sulphonyl or carbonyl chloride in 60 ml of the same solvent with stirring at room temperature. The reaction mixture was stirred at room temperature for 6–12 h, then it was concentrated in vacuum, and the aqueous residue was extracted with 5 × 20 ml chloroform. The combined extracts were dried with  $\text{Na}_2\text{SO}_4$  and chloroform was removed to give 50–80% yields of the raw products which were purified by crystallization or distillation. The physico-chemical characteristics are presented in Table V.

Sulphonates and Carboxylates of (*Z*)-3-(Hydroxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*Z*)-*IV*, (*Z*)-*V*, (*Z*)-*VI*, (*Z*)-*VII*, (*Z*)-*VIII*)

A suspension of 0.02 mol sodium salt *III* in 150 ml dry diethyl ether was cooled to –10 to –15°C and then treated with 0.02 mol of the respective sulphonyl or carbonyl chloride (4-toluenesulphonyl chloride was dissolved in 30 ml ether). The reaction mixture was stirred at this temperature for 10–20 h, whereupon it was concentrated in vacuum, mixed with 50 ml water, and extracted with 5 × 20 ml chloroform. The combined extracts were dried with  $\text{Na}_2\text{SO}_4$ , and chloroform was removed to give 60–85% yields of the raw products which were purified by crystallization. The physico-chemical characteristics are given in Table V.

(*E,E*)-3,3'-(Oxydimethyldiylidene)bis(*trans*-hexahydro-2(3*H*)-benzofuranone) ((*E,E*)-*IX*)

A solution of 1.6 g (5 mmol) (*E*)-3-(4-toluenesulphonyloxymethylene)-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E*)-*V*) and 1.2 g (6 mmol) sodium salt *III* in a mixture of acetone and water (2 : 1) was stirred at room temperature 48 h. Then it was concentrated in vacuum, the precipitate was collected by suction, washed with water, and dried. Yield 1.72 g (90%) raw (*E,E*)-*IX* which was purified by recrystallization from ethanol.

Mixture of (*E,E*)- and (*E,Z*)-3,3'-(Oxydimethyldiylidene)bis-*trans*-hexahydro-2(3*H*)-benzofuranone ((*E,E*)-*IX* and (*E,Z*)-*IX*)

A solution of 1.5 g (8 mmol) sodium salt *III* in 50 ml water was added dropwise to a solution of 2.3 g (7 mmol) 4-toluenesulphonate (*Z*)-*V* in 150 ml acetone, and the mixture was stirred at room temperature 24 h. Then it was concentrated in vacuum, the precipitate formed was collected by suction, washed with water, and dried. Yield 1.43 g raw mixture of (*E,E*)-*IX* and (*E,Z*)-*IX* in the ratio 2 : 3. The mixture was separated on a 30 cm silica gel column (40–100  $\mu\text{m}$ ) with dichloromethane containing 6% ethyl acetate: the (*E,Z*)-*IX* ether is eluted from the column as the first product.

*The authors are indebted to Dr M. Holík, Research Institute of Pure Chemicals, o.p. Lachema Brno, for the measurements of 2 D and INADEQUATE NMR spectra, to Dr M. Černík and Dr Z. Žák, Department of Inorganic Chemistry, University J. E. Purkyně, Brno, for the measurements of the IR spectra and powder diagrams, and to Mrs J. Ondráková for her skilful and self-denying technical assistance.*

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Translated by J. Panchartek.