THE COORDINATION COMPOUNDS OF NICKEL WITH GLYCINE AND WITH AN N–N LIGAND

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The compounds $[Ni(Gly)_2 N–N].nH_2O$ ($n = 1, 2$; Gly = glycine anion; N–N — ethylenediamine 1,10-phenanthroline, α,α'-bipyridyl) and $K[Ni(Gly)_3].3 H_2O$ were prepared. On the basis of their magnetochemical and spectral properties, the compounds obtained can be characterized as octahedral nickel(II) complexes. Cyclic voltammetry was employed to study the redox properties of selected compounds and possible electrochemical and chemical oxidation were studied. The results obtained permitted comparison with the properties of nickel(II) complexes with dipeptides or tripeptides as ligands.

The blue octahedral diaqua-bis(glycinato) nickel(II) complex, $[Ni(H_2O)_2(Gly)_2]$, has been known for some time[1-3], and contains water molecules bonded in the *trans* position. It has recently been demonstrated[4,5] that nickel(II) complexes with dipeptides, Gly-Gly (glycyl-glycine) and Gly-Ala (glycyl-α-alanine), and tripeptides, Gly-Gly-Gly (triglycine) and Gly-Gly-Ala (glycyl-glycyl-α-alanine), as ligands can be oxidized to the corresponding nickel(III) compounds. As nickel compounds containing nickel in a higher oxidation state with bidentate ligands of the N–N types are known, where N–N is α,α'-bipyridyl (bpy) — for example[6], $[Ni(bpy)_3]$-$(ClO_4)_3$ or ethylenediamine (en) — for example[7] $[Ni(en)_3]Cl_3$, we were interested to learn whether compounds of the type $[Ni(Gly)_2 N–N]$ and $K[Ni(Gly)_3]$ could be converted to the corresponding compounds of nickel in a higher oxidation state. As the characteristics of the initial nickel(II) complexes were not found in the literature, the method for their preparation and some of the results obtained from physico--chemical study of these compounds will also be given here.

EXPERIMENTAL

Ethylenediamine was the product of the Downs Development Co (U.K.). and the other chemicals all of p.a. purity (Lachema Brno, Czechoslovakia), were used without further modification, The infrared absorption spectra were recorded on a Specord IR 75 instrument using the Nujol technique and the electronic absorption spectra were obtained using a Specord M 40 spectrometer (Carl Zeiss, Jena). The magnetic susceptibility was measured using the Gouy method on a magnetic balance constructed in the laboratory; $CoHg(NCS)_4$ was used for calibration. Thermal decomposition was studied on a Q 1500 D Derivatograph (MOM Budapest) with a sample

weight of 120—140 mg and temperature increase of 2·5°C/min. Cyclic voltammetry was carried out using a PA 3 polarographic analyzer (Laboratorní přístroje, Prague) with polarization from 0 to 1·3 V and a scan rate of 0·2 V s⁻¹. The complex concentration was 5—10 mmol l⁻¹; because of the low concentration of the sample, the ionic strength I was adjusted to a value of only 0·01 mol l⁻¹ using an NaCl solution (it was demonstrated that a higher concentration of inert electrolyte does not affect the cyclic voltammetry). The pH value was adjusted using an NaOH solution to a value of 10·0 and a gC/SCE/Pt electrode system was employed.

X-ray studies were carried out using a TUR M 62 instrument and HZG-4/C diffractometer (Präzisionsmechanik, Freiberg G.D.R.). Single crystals were studied (basic parameters and space group determination) using a PDK-1 precession chamber (Dioptra, Kroměříž Czechoslovakia) and WBG 2 Weissenberg instrument (Präzisionsmechanik, Freiberg). A computer was not used for the computation.

$[Ni(Gly)_2 bpy].2 H_2O$

A solution containing 0·4 g (2·56 mmol) of bpy in 10 ml of ethanol was added to a solution of 0·45 g (11·85 mmol) $[Ni(H_2O)_2(Gly)_2]$ (prepared according to ref.[1]) in 10 ml of warm water. The dark purple solution formed was stirred at laboratory temperature for half an hour and then evaporated on a water bath at 70°C; the precipitated crystals were filtered, thoroughly washed with ethanol and dried in a vacuum dessicator over KOH after washing with ether.

$[Ni(Gly)_2 phen].H_2O$

This substance was obtained by reaction of 0·45 g (1·85 mmol) of $[Ni(H_2O)_2(Gly)_2]$ dissolved in water and 0·4 g (2·02 mmol) 1,10-phenanthroline (phen) dissolved in ethanol, using the procedure described above.

$[Ni(Gly)_2 en].H_2O$

An amount of 0·45 g (1·85 mmol) $[Ni(H_2O)_2(Gly)_2]$ was dissolved in 10 ml of warm water and mixed with a solution of 0·15 ml (2·24 mmol) of ethylenediamine in 10 ml ethanol. The reaction mixture was treated in the same way as for $[Ni(Gly)_2 bpy].2 H_2O$ and the precipitated crystals obtained from the evaporated solution after standing at laboratory temperature for 1 week were filtered and washed with ethanol and ether on the frit.

$K[Ni(Gly)_3].3 H_2O$

A solution of 2·4 g (9·88 mmol) $[Ni(H_2O)_2(Gly)_2]$ in 10 ml of water was mixed with a solution of 1·5 g (20 mmol) glycine and 1·2 g (21 mmol) KOH in water. The reaction mixture was evaporated on a water bath and then left to crystallize. The dark blue precipitated crystals were filtered and washed with ethanol and ether on the frit.

A substance with the composition $[Ni(Gly)_2 bpy].4 H_2O$ was prepared in a similar manner as $[Ni(Gly)_2 bpy].2 H_2O$; the crystals precipitated from the condensated solution were not washed with ethanol, which dehydrates the product. This substance was employed to study the crystal structure as it forms well-developed crystals.

Electrochemical oxidation of $[Ni(Gly)_2 bpy].2 H_2O$. An amount of 1 g of $[Ni(Gly)_2 bpy]$ was dissolved in 14 ml of water and mixed with a solution of 0·6 g of KOH in 1 ml of water. This mixture was electrolyzed with constant stirring at laboratory temperature using a stabilized BS 554 source (Tesla) at platinum electrodes at a voltage of 2·5 V and current density of 0·1 A.

The reaction product, in the form of a black diamagnetic powder, was removed from the electrode at one hour intervals to a beaker containing water and can be stored at a temperature of $0°C$. Drying yields a grey paramagnetic substance with the composition [Ni(Gly)bpy(OH)].

Chemical oxidation. To a solution of 0.5 g (1.25 mmol) [Ni(Gly)$_2$bpy].2 H_2O in 5 ml water was added 0.15 g (2.7 mmol) KOH and 0.3 g $K_2S_2O_8$ (1.11 mmol) in small amounts with stirring. The reaction mixture was stirred at laboratory temperature for 15 min, and the precipitated solid substance was filtered off and thoroughly washed with water on the frit.

The chemical compositions of the substances prepared are given in Table I and the results of magnetochemical measurements are listed in Table II. Table III gives the electronic absorption spectra.

RESULTS AND DISCUSSION

The nickel(II) complexes were prepared from $[Ni(H_2O)_2(Gly)_2]$ through replacement of a water molecules by a bidentate N–N ligand, yielding the compound $[Ni(Gly)_2N-N].nH_2O$ ($n = 1, 2$; N–N = en, bpy, phen). A study of the thermal stability of $[Ni(H_2O)_2(Gly)_2]$ revealed[8] that heating above $120°C$ leads to splitting off of both water molecules. A further increase in the temperature leads to thermal decomposition of the anhydrous salt. As the results of X-ray structural analysis

TABLE I

The chemical compositions of the prepared substances

Compound	Formula M_r	Found/calculated			
		% Ni	% C	% H	% N
[Ni(H$_2$O)$_2$(Gly)$_2$]	C$_4$H$_{12}$NNiO$_6$	23·71	19·70	5·34	11·57
	242·9	24·17	19·78	4·98	11·53
[Ni(Gly)$_2$bpy].2 H$_2$O	C$_{14}$H$_{20}$N$_4$NiO$_6$	13·93	42·24	4·75	14·41
	399·1	14·71	42·13	5·03	14·04
[Ni(Gly)$_2$bpy].4 H$_2$O	C$_{14}$H$_{24}$N$_4$NiO$_8$	13·25	39·00	5·59	13·36
	435·1	13·50	38·64	5·56	12·88
[Ni(Gly)$_2$phen].H$_2$O	C$_{16}$H$_{18}$N$_4$NiO$_5$	14·50	46·80	4·35	13·88
	405·0	14·49	47·44	4·48	13·83
[Ni(Gly)$_2$en].H$_2$O	C$_6$H$_{18}$N$_4$NiO$_5$	20·77	25·23	5·98	19·51
	284·9	20·60	25·29	6·37	19·66
K[Ni(Gly)$_3$].3 H$_2$O	C$_6$H$_{18}$KN$_3$NiO$_9$	15·64	19·28	3·97	10·87
	374·0	15·70	19·27	4·85	11·24
[Ni(Gly)bpy(OH)]	C$_{12}$H$_{13}$N$_3$NiO$_3$	19·87	47·18	3·83	13·95
	305·9	19·19	47·11	4·28	13·73

indicated[3] that the water molecules are bonded in the *trans* position; replacing them by a bidentate N–N ligand can lead to the formation of polymer arrangement *I* or to reorganization of the coordination sphere, *II*. Preparation of substances with

I

II
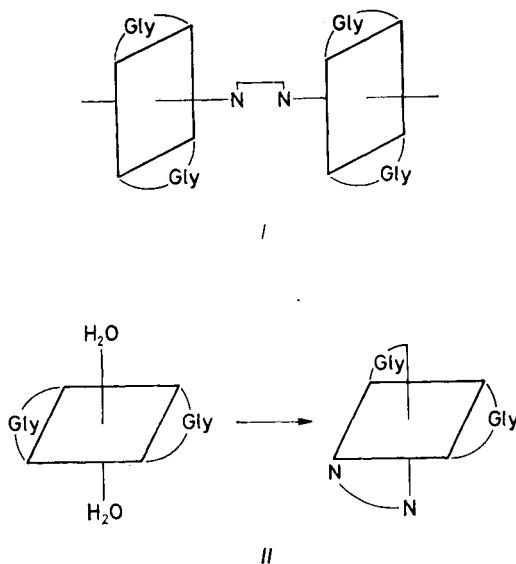
the compositions $[Ni(Gly)_2bpy].2\,H_2O$, $[Ni(Gly)_2phen].H_2O$, and $[Ni(Gly)_2en]$. .H_2O indicated that the molecules of water in the inner sphere of the complex can be replaced by a bidentate N–N ligand. The magnetic moments of these substances (Table II) have values characteristic for octahedral nickel(II) complexes. It follows from the diagram of splitting of the d-orbitals that the central atom should then have two unpaired electrons. Because of the magnitude of the orbital contribution, the μ_{ef} values vary in the interval 2·9 – 3·4 BM. The electronic absorption spectra of the substances prepared are also typical for octahedral nickel(II) complexes. The strong absorption maximum at c. $28\,000\,cm^{-1}$ corresponds to the electron transition $^3A_2 \rightarrow {}^2T_1(P)$ and the other, weaker maxima at c. $17\,000\,cm^{-1}$ and $10\,000\,cm^{-1}$ to the $^2A_2 \rightarrow {}^3T_1(F)$ and $^3A_2 \rightarrow {}^3T_2$ transitions. As the electronic absorption spectra of these compounds are very similar to that of *trans*-$[Ni(H_2O)_2(Gly)_2]$, it would appear that these substances exist as the trans(O) isomers.

It follows from the thermogram of $[Ni(Gly)_2N–N].nH_2O$ (Table II) that water is removed at a lower temperature than for $[Ni(H_2O)_2(Gly)_2]$. Thermal decomposition of these complexes begins with splitting off of a water molecule to form the anhydrous salt. A further increase in the temperature then leads to decomposition, beginning at 255°C for the bipyridyl complex, 250°C for phenanthroline and 205°C for the $[Ni(Gly)_2en].H_2O$ complex.

Study of the infrared absorption spectra yields information on the coordination of the bidentate ligand. The infrared spectrum of $[\text{Nibpy(Gly)}_2].2\,H_2O$ contains both the absorption maxima characteristic for the glycinate anion[9-11], notably $\nu_{as}(NH_2)$

TABLE II

Some of the properties of the prepared compounds

Compound	μ_{ef}, BM	GTA[a]
$[\text{Ni}(H_2O)_2(\text{Gly})_2]$	3·19	120 (180)
$[\text{Ni}(\text{Gly})_2\text{bpy}].2\,H_2O$	3·10	105 (140)
$[\text{Ni}(\text{Gly})_2\text{phen}].H_2O$	3·10	100 (160)
$[\text{Ni}(\text{Gly})_2\text{en}].H_2O$	2·92	60 (125)
$K[\text{Ni}(\text{Gly})_3].3\,H_2O$	2·69	100 (130)
$[\text{Ni}(\text{Gly})\text{bpy}(OH)]$	2·91	—

[a] The values correspond to the initial (final) dehydration temperature (°C).

TABLE III

The electronic absorption spectra of the studied complexes

Compound	$\tilde{\nu}\,cm^{-1}$	Electronic transition	Δ, cm^{-1} B, cm^{-1}
$[\text{Ni}(H_2O)_2(\text{Gly})_2]$	26 960	$^3A_2 \rightarrow {}^3T_1(P)$	13 070
	16 320	$^3A_2 \rightarrow {}^3T_1(F)$	270
	10 500	$^3A_2 \rightarrow {}^3T_2$	
$[\text{Ni}(\text{Gly})_2\text{bpy}].2\,H_2O$	28 500	$^3A_2 \rightarrow {}^3T_1(P)$	13 750
	17 720	$^3A_2 \rightarrow {}^3T_1(F)$	330
	10 000	$^3A_2 \rightarrow {}^3T_2$	
$[\text{Ni}(\text{Gly})_2\text{phen}].H_2O$	30 680	C.T.	
	29 200	$^3A_2 \rightarrow {}^3T_1(P)$	14 180
	17 560	$^3A_2 \rightarrow {}^3T_1(F)$	280
	10 000	$^3A_2 \rightarrow {}^3T_2$	
$[\text{Ni}(\text{Gly})_2\text{en}].H_2O$	27 960	$^3A_2 \rightarrow {}^3T_1(P)$	13 500
	17 360	$^3A_2 \rightarrow {}^3T_1(F)$	320
	10 500	$^3A_2 \rightarrow {}^3T_2$	
$K[\text{Ni}(\text{Gly})_3].3\,H_2O$	28 240	$^3A_2 \rightarrow {}^3T_1(P)$	13 770
	16 580	$^3A_2 \rightarrow {}^3T_1(F)$	235
	10 500	$^3A_2 \rightarrow {}^3T_2$	

$3\ 320\ cm^{-1}(s)$, $\nu_{sym}(NH_2)$ $3\ 250\ cm^{-1}(s)$, $\nu(OH)$ $2.290\ cm^{-1}(s)$, $\nu_{as}(COO^-)$, $1\ 570\ cm^{-1}(s)$, $\nu_{sym}(COO^-)$ $1\ 405\ cm^{-1}(s)$ and vibrations corresponding to the Ni—N bond at $520\ cm^{-1}(m)$, as well as maxima corresponding to the coordination of bipyridyl. It has been pointed out[12] that these are primarily maxima at wavenumbers of $1\ 590\ cm^{-1}(s)$, $1\ 160\ cm^{-1}(m)$, and $1\ 025\ cm^{-1}(m)$. Other absorption maxima characteristic for bipyridyl have wavenumbers of $1\ 225\ cm^{-1}(s)$, $980\ cm^{-1}(m)$, and $770\ cm^{-1}(s)$. The other absorption maxima characteristic for bipyridyl overlap with the absorption maxima of glycinate.

Similarly, the infrared spectrum of $[Ni(Gly)_2phen].H_2O$ contains both the absorption maxima typical for glycinates $(\nu_{as}(NH_2)$ $3\ 380\ cm^{-1}(s)$, $\nu_{sym}(NH_2)$ $3\ 280\ cm^{-1}(s)$, $\nu(OH)$ $2\ 270\ cm^{-1}(s)$, $\nu_{as}(COO^-)$ $1\ 570\ cm^{-1}(s)$, $\nu_{sym}(COO^-)$ $1\ 410\ cm^{-1}(s))$ and for the Ni—N bond with a vibration at $505\ cm^{-1}(w)$ as well as maxima characteristic for 1,10-phenanthroline[12] with wavenumbers of $1\ 325\ cm^{-1}(s)$, $1\ 250\ cm^{-1}(m)$, $1\ 230\ cm^{-1}(m)$, and $850\ cm^{-1}(s)$. The other absorption maxima of phenanthroline overlap with those of glycinate.

In the spectrum of $[Ni(Gly)_2en].H_2O$, the following absorption maxima were assigned to coordinated glycinate: $\nu_{as}(NH_2)$ $3\ 330\ cm^{-1}(s)$, $\nu_{sym}(NH_2)$ $3\ 200\ cm^{-1}(s)$, $\nu(OH)$ $2\ 300\ cm^{-1}(m)$, $\nu_{as}(COO^-)$ $1\ 570\ cm^{-1}(s)$, $\nu_{sym}(COO^-)$ $1\ 400\ cm^{-1}(s)$ and to the valence vibration of Ni—N corresponding to a maximum at $510\ cm^{-1}(w)$. Ethylenediamine has absorption maxima at $\nu(NH_2)$ $3\ 290\ cm^{-1}(s)$ and $\delta(NH_2)$ $1\ 030\ cm^{-1}(s)$.

The temperature dependence of the magnetic susceptibility of the complexes prepared obeys the Curie–Weiss law with a very small ϑ value (for $[Ni(Gly)_2en].H_2O$, $\vartheta = 3\cdot6$ K and $g = 3\cdot29$, for $[Ni(Gly)_2bpy].2\ H_2O$, $\vartheta = 1\cdot48$ K and $g = 3\cdot06$). Fig. 1 depicts the dependence of the magnetic suceptibility and μ_{ef} on the temperature in the interval 95 K to 293 K. It can be seen that the magnetic moment is constant over the whole measured interval of temperatures and has a value corresponding
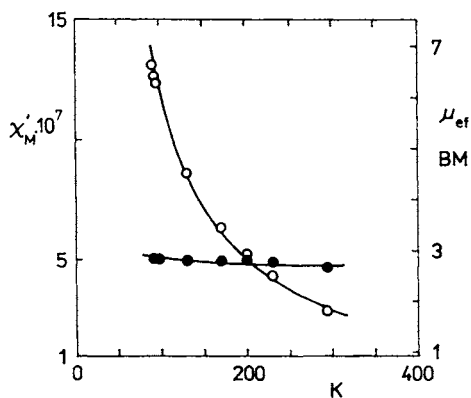

FIG. 1

The temperature dependence of the molar magnetic susceptibility X'_M ○ and magnetic moment μ_{ef} ● of the $[Ni(Gly)_2en].H_2O$ complex

to octahedral coordination. As a polymeric complex would apparently involve metal–metal interactions that would affect the temperature dependence of μ_{ef}, it can be assumed that the studied compounds are mononuclear complexes (monomers). Their relatively high solubility is also in agreement with this conclusion.

The crystal structure was studied using $[\text{Ni(Gly)}_2\text{bpy}].4\,\text{H}_2\text{O}$ which forms well--developed crystals. This substance crystallizes in the orthorhombic system, with a space group of *Fmmm* or *F222* or *Fmm2*, with parameters $a = 2\cdot825(5)$, $b = 4\cdot039(5)$, and $c = 0\cdot746(5)$ nm (parameters a, b, and c were refined on the basis of 53 measured and indexed lines), with an elementary cell volume of $V = 8\cdot51$ nm^3. For $Z = 16$, $\varrho_{\text{theor}} = 1\cdot36\,.\,10^3$ kg m^{-3}, $\varrho_{\text{exp}} = 1\cdot42\,.\,10^3$ kg m^{-3}.

Similarly, magnetochemical studies and the character of the electronic spectra of $\text{K}[\text{Ni(Gly)}_3].3\,\text{H}_2\text{O}$ indicate that this compound is an octahedral nickel(II) complex with the following wavenumbers of characteristic maxima in the infrared absorption spectra: $\nu_{\text{as}}(\text{NH}_2)$ $3\,200 - 3\,500$ cm^{-1}(s, sh), $\nu_{\text{sym}}(\text{NH}_2)$ $3\,020$ cm^{-1}(s), $\nu(\text{OH})$ $2\,300$ cm^{-1}(m), $\nu_{\text{as}}(\text{COO}^-)$ $1\,585$ cm^{-1}(s), $\nu_{\text{sym}}(\text{COO}^-)$ $1\,420$ cm^{-1}(m), $\nu(\text{Ni}-\text{N})$ 520 cm^{-1}(m). This compound is dehydrated in the temperature interval from 100 to 130°C (for 3 H$_2$O, Δm calculated 14·5%, found 13·5%).

The redox characteristics of these substances were first studied using cyclic voltammetry. The curve for the oxidative decomposition of the base electrolyte containing $[\text{Ni(Gly)}_2\text{phen}].\text{H}_2\text{O}$ has an anodic shoulder at $I_P^A = 9\cdot5$ μA corresponding to 118 μA cm^{-2} ($0\cdot02$ V s^{-1}). The cathodic peak is smaller and visible only for higher depolarizer concentrations. The potential difference between the anodic and cathodic peaks ($E_P^A - E_P^C$) is greater than would correspond to exchange of a single electron and increases with increasing polarization rate: ΔE_P ($0\cdot1$ V s^{-1}) $= 0\cdot14$ V ($E_P^C = 0\cdot96$ V, $E_P^A = 1\cdot1$ V); ΔE_P ($0\cdot5$ V s^{-1}) $= 0\cdot325$ V ($E_P^C = 0\cdot874$ V, $E_P^A = 1\cdot2$ V). Quotient $\log I_P/\log V = 0\cdot5$. The Faradaic current is very slightly smaller than would correspond to exchange of a single electron. This is thus an irreversible diffusion--controlled oxidation. Evaluation of the results is complicated by deposition of the solid product on the electrode. For $[\text{Ni(H}_2\text{O)}_2(\text{Gly})_2]$ only a very slight increase in the anodic current was found at potentials corresponding to decomposition of the base electrolyte.

Preparative electrochemical oxidation of $[\text{Ni(Gly)}_2\text{bpy}].2\,\text{H}_2\text{O}$ yielded a black, unstable substance that is diamagnetic when damp. Filtration under vacuum and washing on the frit with ethanol or drying under vacuum in a dessicator leads to a colour change from black to light and the substance becomes paramagnetic. Thus, the unstable, black, diamagnetic complex decomposes to yield a light grey paramagnetic substance with the composition $[\text{Ni(Gly)bpy(OH)}]$. The same final product was obtained from chemical oxidation of $[\text{Ni(Gly)}_2\text{bpy}].2\,\text{H}_2\text{O}$ using $\text{K}_2\text{S}_2\text{O}_8$. Attempts to carry out anodic oxidation of a Ni(Gly)$_2$–K(Gly) mixture were not successful; no solid substance was precipitated and no changes in the solution were observed. Electrochemical or chemical ($\text{K}_2\text{S}_2\text{O}_8$) oxidation of a solu-

tion of $[Ni(Gly)_2en].H_2O$ results in a change in the solution colour from the original blue to black. However, the dissolved substance could not be isolated from solution without a change in composition.

It thus follows that the prepared compounds of the $[Ni(Gly)_2N-N]$ type undergo redox reactions but the products formed are unstable and readily decompose to form the nickel(II) compounds. On the other hand, Margerum et al.[4,5] employed electrochemical oxidation of nickel(II) complexes with Gly-Gly dipeptides to prepare relatively stable nickel(III) coordination compounds. Comparison of the two types of complex indicated that, while the ligand in the group of compounds prepared here was the $CH_2(NH)_2COO^-$ anion, sometimes in association with the molecular N-N ligand (e.g. bpy), dipeptides are bonded as tridentate ligands[4,5] to the central atom through the amino group, oxygen atom of the COO^- group and nitrogen atom of the deprotonated peptide bond, in which the electron density is greatly increased on the nitrogen atom, increasing its ability to form dative σ-bonds. The arrangement here is similar to that for nickel(III) complexes with dioximes[13,14] —
i.e. contains the $-C=N-\overset{(-)}{O}-N=$ group. Ligands that have marked σ-donor properties increase the electron density in the vicinity of the central atom and thus contribute to an increase in its stability in the higher oxidation state. It should be realized that nickel prefers ligands containing nitrogen atoms so that the suitability of peptides or oximes as ligands stabilizing nickel in the higher oxidation state becomes obvious.

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Translated by M. Štulíková.