Keywords: principles of instrumental analysis, heat, and kinetic methods.

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They may have other applications as precursors in superconducting ceramic and magnetic field production. The gaseous products of the thermal decomposition in nitrogen are also determined and their magnetic susceptibilities were measured over the temperature range of 76-303K and the magnetic moments were calculated. The results show that 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) are high-spin complexes with weak ligand fields. The solubility value in water at 293K for analysed 4-chlorophenoxyacetates is given in the range of 76-303K and the magnetic moments were calculated. The results show that 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) are high-spin complexes with weak ligand fields. The solubility value in water at 293K for analysed 4-chlorophenoxyacetates is in the order of 10^{-3} mol/dm^3.
Ni(II) ions were synthesized and their properties were studied [10,16,17]. As a continuation of our studies on chlorophenoxyacetates and also on carboxylates [10,18-30] we decided to synthesized 4-chlorophenoxyacetates with Nd(III), Gd(III) and Ho(III) and to study some of those of their properties not to have been investigated so far, such as magnetic properties in the range of 76-303K, thermal stability in air at 293-1173K, solubility in water at 293K and to record their FTIR spectra. Thermal stability investigations give informations about the process of dehydrations about the ways of decompositions, and the magnetic susceptibility measurements let study the kinds of the manner of central ion coordination. The determination of the solubility is valuable because it informs about the practical use of acid for separation of transition metal ions by extraction or ion-exchange chromatographic methods.

Table 1. Elemental analysis data of Nd(III), Gd(III) and Ho(III) 4-chlorophenoxyacetates and their solubility in water at 293K.

| Complex L− = C8H6ClO3 | H% | C% | Cl% | M% | Solubility |
|------------------------|-----|-----|-----|-----|------------|
| NdL−·2H2O             | 3.00 | 7.77 | 39.10 | 38.96 | 14.50 | 14.48 | 19.60 | 22.70 | 5.3·10^-4 |
| GdL−·2H2O             | 2.90 | 7.79 | 38.40 | 38.94 | 14.20 | 14.18 | 20.90 | 19.50 | 7.6·10^-4 |
| HoL−·3H2O             | 3.10 | 8.88 | 37.10 | 37.16 | 14.10 | 14.08 | 21.80 | 21.20 | 8.5·10^-4 |

The contents of M+ were established gravimetrically and by XRF method using spectrometer of X-ray fluorescence with energy dispersion EDXRF-1510 (CANBERRA firm). The IR and FIR spectra of complexes were recorded over the ranges 4000-400cm⁻¹ and 600-100cm⁻¹, respectively, using M-80 and Perkin-Elmer 180 spectrometers. Samples for IR spectra measurements were prepared as KBr discs. FIR spectra were obtained in Nujol mulls sandwiched between polyethylene plates (Table 2). The FTIR spectra of the intermediate and final products obtained from the complex thermal decompositions were also registered.

Experimental details

The 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) were prepared by the addition of the equivalent quantities of 0.1mol·dm⁻³ ammonium 4-chlorophenoxyacetate (pH ≈ 5) to a hot solutions containing the 0.1 mol·dm⁻³ Nd(III), Gd(III) and Ho(III) nitrates (V) and crystallizing at 293K (1h). The solids formed were filtered off, washed with hot water and methanol to remove ammonium ions and dried at 303K to a constant mass.

The contents of carbon and hydrogen in the complexes and in the intermediate and final products obtained from their thermal decompositions were determined by elemental analysis using a CHN 2500 Perkin-Elmer analyzer and the content of chlorine by the Schöniger method (Table 1).

The X-ray diffraction patterns of hydrated and dehydrated complexes, the intermediate and final products of complex thermal decompositions were taken on a HZG-4 (Carl Zeiss Jena) diffractometer using Ni filtered Cu Kα radiation. The measurements were made within the range 2Θ = 4-80° by means of the Debye-Scherrer-Hull method. The relationships between I/I₀ and 2Θ for these complexes are presented in Fig.1.

Table 2. Wavenumbers (cm⁻¹) of COO⁻ bands in the analysed complexes of Nd(III), Gd(III), Ho(III), and Na(I), and that of the COOH band in 4-chlorophenoxyacetic acid.

| Complex L− = C8H6ClO3 | ° C=O | ° as OCO | ° sym OCO | ° as OCO | ° Cl= | ° M=O |
|------------------------|------|----------|-----------|----------|------|-------|
| NdL−·2H2O              | -    | 1571     | 1391      | 2010     | 57   | 449   |
| GdL−·2H2O              | 1572 | 1337     | 235       | 507      | 452  |
| HoL−·3H2O              | 1572 | 1346,1328| 226,244   | 528,563  | 449  | 449   |

The thermal stability and decomposition of the prepared complexes were determined by Pau-lik-Paulik-Erday Q-1500D derivatograph with Derill converter, recording TG, DTG, and DTA curves (Fig.2). The measurements were made at a heating rate of 10K min⁻¹ with a full scale. The samples (100mg) were heated in platinum crucibles in static air to 1173K with a sensitivity of TG-100mg. DTG and DTA sensitivities were regulated by a Derill computer program. The products of decomposition...
were calculated from TG curves and verified by the diffraction pattern registration and IR spectra. The

termogravimetric analysis of DSC/TG was performed at temperature 323-723K using a differential ther-
mos analyzer Netzsch STA 409C 3F. The measurements were carried out under nitrogen flow (99.995%
purity) and temperature increase rates of 1-12K min⁻¹.

Table 3. Temperature ranges of thermal stability of Nd(III), Gd(III) and Ho(III) 4-chlorophenoxyacetates in air.

| Complex   | L⁻ClO⁻ | ΔT/K | Mass loss/% | ΔH/kJ mol⁻¹ | ΔH(ΔH) | T/K  | Mass of residue/% | Final product of decomposition |
|-----------|---------|------|-------------|--------------|---------|------|-------------------|--------------------------------|
| NdL₆·3H₂O | 390-415 | 22.8 | 70.2 412 | 10.39        | 74.8    | 57.6 | 10.39 | NdOCl            |
| GdL₆·3H₂O | 316-412 | 24.3 | 71.8 412 | 10.39        | 74.8    | 57.6 | 10.39 | GdOCl            |
| HoL₆·3H₂O | 312-425 | 24.2 | 70.2 412 | 10.39        | 74.8    | 57.6 | 10.39 | HoOCl            |

ΔT₁ – temperature range of dehydration process, ΔT₂ – temperature range of anhydrous complex decom-
position, n – number of water molecules lost in the one step of dehydration process, ΔH – enthalpy value of
dehydration process, Tₐ – final temperature of decomposition process, ΔH(ΔH) – enthalpy value for
one molecule of water, Tₐ – final temperature of decomposition process

Magnetic susceptibilities of polycrystalline samples of 4-chlorophenoxyacetates of Nd(III),
Gd(III) and Ho(III) were measured by the Gouy method using a sensitive Cahn RM-2 balance. The
samples were placed in a long cylindrical tube which was suspended from an analytical balance.

The sample tube was positioned between the poles of the magnet such that one its end was in the re-
gion of homogenous field and the other end was in the region of zero field. The force exerted on the sample was a function of the volume occupied by the sample in the region of the field gradient. This
force may be written in scalar form as a function of the isotropic volume susceptibility. Measurements
were carried out at a magnetic field strength of 9.9 kOe. The calibrate employed was Hg[Co(SCN)]₂ for
which the magnetic susceptibility was assu-
moved to be 1.644·10⁻⁵ cm³g⁻¹. Correction for dia-
magnetism of the calibrant atoms was calculated
by the use of Pascal’s constants [31,32]. Magnetic
moments were calculated from Eq (1):

\[
\mu = 2B_0 \left( \frac{3}{\mu_B} \right) \theta \cdot T^{-1}.
\]

Table 4. Magnetic data for the studied complexes of Nd(III), Gd(III) and Ho(III), L⁻ = C₆H₅ClO₃

| Complex | L⁻ClO⁻ | T/K | μ /MₖOe | μ /μ₅B | T/K | Z /10⁻⁶ | μ /μ₅B |
|---------|---------|-----|---------|---------|-----|----------|---------|
| NdL₆·2H₂O | 0 = 25 | 76  | 14292  | 2.95  | 76  | 97416  | 7.70  |
| GdL₆·2H₂O | 0 = 8.2 | 123 | 9964  | 3.13  | 123 | 66753  | 7.74  | 123 | 169355 |
| HoL₆·3H₂O | 0 = 35 | 133 | 9335  | 3.15  | 133 | 55665  | 7.71  | 133 | 98520  |
|          | 143   | 8763 | 3.17  | 143  | 8763 | 51821  | 7.70  | 143  | 91856  |
|          | 153   | 8343 | 3.20  | 153  | 8343 | 48289  | 7.69  | 153  | 87225  |
|          | 163   | 7924 | 3.22  | 163  | 7924 | 45099  | 7.67  | 163  | 81917  |
|          | 173   | 7543 | 3.23  | 173  | 7543 | 42622  | 7.68  | 173  | 77229  |
|          | 183   | 7237 | 3.26  | 183  | 7237 | 40172  | 7.67  | 183  | 73389  |
|          | 193   | 7069 | 3.29  | 193  | 7069 | 36455  | 7.71  | 193  | 69831  |
|          | 203   | 6742 | 3.31  | 203  | 6742 | 36669  | 7.72  | 203  | 66386  |
|          | 213   | 6418 | 3.31  | 213  | 6418 | 34733  | 7.70  | 213  | 63506  |
|          | 223   | 6189 | 3.32  | 223  | 6189 | 33252  | 7.71  | 223  | 60683  |
|          | 233   | 5960 | 3.33  | 233  | 5960 | 31771  | 7.70  | 233  | 57972  |
|          | 243   | 5769 | 3.35  | 243  | 5769 | 30518  | 7.71  | 243  | 55713  |
|          | 253   | 5636 | 3.38  | 253  | 5636 | 29293  | 7.70  | 253  | 53680  |
|          | 263   | 5445 | 3.39  | 263  | 5445 | 28467  | 7.76  | 263  | 51985  |
|          | 273   | 5274 | 3.40  | 273  | 5274 | 27470  | 7.75  | 273  | 50182  |
|          | 283   | 5178 | 3.43  | 283  | 5178 | 26701  | 7.78  | 283  | 49049  |
|          | 293   | 5083 | 3.45  | 293  | 5083 | 25933  | 7.80  | 293  | 47693  |
|          | 303   | 4949 | 3.47  | 303  | 4949 | 25192  | 7.82  | 303  | 45943  |

The solubilities of 4-chlorophenoxyacetate-
tes of Nd(III), Gd(III) and Ho(III) in water were measured at 293K. Saturated solutions of the ob-
tained compounds were prepared under isother-
mal conditions. The contents of NdL₆, GdL₆ and
HoL₆ were determined using ASA 880 spec-
tro photometer (Varian). The values of solubilities are presented in Table 1.

Results and Discussion

The complexes of 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) were obtained as polycrystalline products with a metal ion to ligand ratio of 1:3 and the general formula M(C₆H₅ClO₃)₃-
H₂O, where M(II) = Nd, Gd, Ho and n = 2 for Nd
and Gd and n = 3 for Ho. Their colours are fol-
lowing: violet for Nd(III), cream for Ho(III), and

Fig. 2. TG,DTG and DTA curves for Ho(III) 4-chlorophenoxyacetate in air.
white for Gd(III) complexes. In these compounds the f–f electron transitions of the central ions are those of the lowest energy and absorption occurs at relatively high wave lengths that depends on the nature of the metal ion [33,34].

The compounds were characterized by elemental analysis (Table 1), FTIR and FIR spectra (Table 2).

The 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) exhibit similar solid state IR spectra. The band at 1708 cm⁻¹ originating from the HCOO⁻ group, presented in the acid spectrum, is replaced in the spectra of complexes by two bands at 1572-1567 cm⁻¹ and 1346-1328 cm⁻¹, which can be ascribed to the asymmetric and symmetric vibrations of COO⁻ group, respectively [35-38].

The bands with the maxima at 3429-3410 cm⁻¹ characteristic for ν(OH) vibrations [35-40] confirm the presence of crystallization water molecules in the complexes.

The bands of C-H vibrations are observed at 2929-2918 cm⁻¹. The bands of ν(C=C) ring vibrations appear at 1624-1622 cm⁻¹, 1494 cm⁻¹, 1177-1174 cm⁻¹, 1111-1104 cm⁻¹ and 1068-1066 cm⁻¹ [1].

The valency ν(C=C) vibrations band occurs at 734-730 cm⁻¹ and those of the asymmetric and symmetric ν(C=C) at 1082-1086 cm⁻¹ and 1060-1058 cm⁻¹, respectively. The bands at 451-449 cm⁻¹ confirm the ionic metal-oxygen bond [41-50].

These bands change insignificantly their positions which may suggest the different stability of these complexes.

The bands in the range of 118-106 cm⁻¹ are connected with the O–H...O streching vibrations and they change their shapes according to the rise of atomic number of elements and the degree of hydration in 4-chlorophenoxyacetates. The bands at 138-122 cm⁻¹ confirm the internal C-C torsion vibrations and the bands at 283-267 cm⁻¹ arising from the aromatic ring vibrations also change their shapes.

Table 2 presents the values of the two band frequencies of asymmetrical and symmetrical vibrations for carboxylate group of analysed complexes.

The separations of the ν(COO⁻), and ν(COO) modes in the compounds, Δν(COO) and Δν(COO) are smaller than that of the sodium salt (Δν(COO) = 271 cm⁻¹) indicating a smaller degree of M-O ionic bonds in 4-chlorophenoxyacetates compared to that of the sodium salt. For the complexes the shifts of the frequencies of bands of ν(COO⁻), and ν(COO) are lower, and lower or the same, respectively, than those for sodium 4-chloro- phenoxyacetate. Accordingly, taking into account the spectroscopic criteria [36-38,49,51] the carboxylate ions appear to be bidentate groups.

In order to estimate the crystalline forms of the 4-chlorophenoxyacetates the X-ray powder diffraction measurements for the dihydrates were carried out. The diffractogram values suggest them to be polycrystalline compounds with various degree of crystallinity [52] (Fig.1).

The thermal stability of Nd(III), Gd(III) and Ho(III) 4-chlorophenoxyacetates was studied in air at 293-1273 K (Table 3, Fig. 2).

When heated to 1273 K the Nd(III) complex is dehydrated in one step. In the temperature range of 338 – 416 K it loses two water molecules and forms anhydrous salt. The loss of mass calculated from TG curve is equal to 4,88% (theoretical value is equal to 6,68%) which corresponds to the loss of three molecules of water (theoretical value is equal to 6,96%). The anhydrou 4-chlorophenoxyacetate of Ho(III) at 573 – 1223 K is decomposed to Ho₂O₃, which is the final product of thermal decomposition. The mass loss calculated from TG curve is equal to 74,29% which corresponds to the Ho₂O₃ formation (calculated value is 75,73%).

The intermediate product of complex decompositon at 573-912K is HoOCl. The dehydra- tion process is connected with the endothermic effect seen in DTA curve, while the combustion of the organic ligand is accompanied by exothermic one.

Considering the temperature at which the dehydration process of the complexes takes place, and the way by which it proceeds it is possible to assume that the water molecules may be in the outer sphere of complex coordination [41, 53-55].

The IR and FIR spectra recorded for analysed compounds may also suggest that the mole- cules of water are probably bonded by hydrogen bond in outer-sphere of complex. The detailed data obtained from the determination of the complete structures of these complexes can give fair answer concerning above assumption. However, the monocrystals have not been obtained so far, but attempts to obtain them have been made. The results indicate that the thermal decomposition of 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) compounds changes from 2,95 µμ (at 76K) to 3,47 µμ (at 303K) for Nd(III) complex, from 7,70 µμ (at 76K) to 7,82 µμ (at 303K) for Gd(III), and from 10,07 µμ (at 76K) to 10,56 µμ (at 303K) for Ho(III) 4-chlorophenoxyacetates.

In 4-chlorophenoxyacetates of Nd(III), Gd(III) and Ho(III) the paramagnetic central ions remain practically unafected by diamagnetic ligands coordinated around them. The f-electrons cause their paramagnetism which is very weak, and it results from outside influences and they do not partici- pate in the formation of M-O bond. They are in an inner shell characterized by radius 0,35Å [62] to be very small in comparison with the radius of the 5f-shell (~1 Å). Their energy levels are the same as in the free ions. For most of the lanthanide ions the ground state is separated by several hundred of cm⁻¹ from the next higher lying state. Therefore the magnetic properties can be taken as those of the ground state alone. Taking into account this fact lanthanide ions in the comp- ound act in the same way as the free ions. The values of µμ determined for 4-chlorophenoxyace- tates are close to those calculated for Ln³⁺ ions by Hund and Van Vleck (Table 5). Their values at room temperature for analysed complexes are
following: 3.74 μC for Nd; 7.23 μC for Gd and 10.60μC for Ho [63].

From the values of magnetic moments determined for the complexes it appears that energies of 4f electrons in the central ions are not changed compared to those in the free lanthanide ions. Thus, the colours of the complexes stay close to those obtained for Ln 

$^1_{173}$K decompose in three steps. In the first step the colours of the complexes are the same as comparison with the free lanthanide ions, there ions. Their energies are not radically changed in energy of f-f electronic transitions of the central ions. Therefore the metal ligand bonding in lanthanide complexes is mainly electrostatic in nature 

$^2_{64,65}$.

Conclusions

On the basis of the results obtained it appears that 4-chlorophenoxycacetates of Nd (III), Gd(III), and Ho(III) were synthesized as hydrated octet. Therefore the metal ligand bond in lanthanide complexes is mainly electrostatic in nature 

$^4_{64,65}$.

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