from Kurucz et al., 1995 [4] was used to recover a correctly refolded protein from inclusion bodies.

A purification procedure comprising three chromatography steps yielded scFv 1696 in the purity necessary for crystallization trials.

The complex was prepared by mixing scFv1696 with an excess of the epitope peptide corresponding to the N-terminus of HIV-2 PR (PQFSLWKR). Monomeric and dimeric forms of the complex were separated by FPLC on a Mono-Q column. In contrast to free scFv 1696 these forms are stable and do not interchange.

In a series of crystallization trials crystals of the complex have been obtained.

For crystallization trials by vapour diffusion method (hanging drop) only monomeric complex of the scFv1696 with epitope peptide was used. Crystals grew spontaneously at 25°C in ammonium sulfate (concentrations: 1.8 - 2.0 M) at pH 4.6. These crystals were very sensitive and flaws on their surface appeared in few days. However, this spontaneous crystallization could not be reproduced with a new batch of the complex, therefore, a streak seeding technique was applied and single crystals of size up to 0.3 x 0.3 x 0.2 mm were obtained.

Solving of 3D structure of the scFv 1696 - epitope peptide complex is expected to lead to antibody-structure-based design of a new class of HIV protease non-active site inhibitors, possibly of different HIV resistance characteristics.


POSTERY

CRYSTALLIZATION AND PRELIMINARY DIFFRACTION STUDY OF HIV-1 PROTEASE COMPLEXED WITH HYDROXYETHYLAMINE INHIBITOR SI

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The HIV-1 protease is essential for replication of infective virus HIV, and therefore is an attractive target for the design of specific inhibitors. In search for new inhibitors substantial effort is placed on understanding the nature of the inhibitor binding modes in the active site, using X-ray diffraction on crystals as the primary source of structural information. Here we report crystallization of HIV-1 protease complexed with a four amino acid pseudopeptidic inhibitor, where the scissile peptide bond is replaced by hydroxyethylamine isostere. This inhibitor, Boc-Phe-[S(CH(OH)CH₂NH)]Phe-Ile-Phe-NH₂, is assigned here SI.

First crystallization trials were based on crystallization studies performed with complex of HIV-1 PR with inhibitors SE, RE, RQ. These inhibitors differ from SI only in the amino acid at P2’ position, carrying Glu or Gln instead of Ile, and in configuration at C4, an hydroxyl bearing carbon of the pseudopeptidic bond. Hanging drop vapor diffusion technique has been used in all experiments. In the case of Glu/Gln containing inhibitors protein - inhibitor mixture of 3 mg/ml HIV PR, 0.544 mM inhibitor (four-fold molar excess over protease) in 50 mM sodium acetate pH 5.6, 1 mM EDTA, 0.05% -mercaptoethanol, 5% DMSO was used. The optimal crystallization conditions found for these crystals are: 1M NH₄H₂PO₄, 100 mM sodium citrate, pH 4.5, temperature 6 - 8°C [1].

In contrast to Glu/Gln containing inhibitors, SI inhibitor addition caused protein precipitation even without presence of salt precipitant. Probable reason is higher hydrophobicity of SI inhibitor. Addition of ammonium phosphate at conditions described above produced no crystal growth and no additional precipitation. Crystals of max.
X-ray studies on 1,4-dihydropyridines have shown a great
dependence of the geometry of the skeleton on the attached
substituents, especially those in the 4 position. We have
studied the crystal structure of dimethyl isopropyl
1-ethoxymethyl-6-methyl-4-(3-nitrophenyl)-1,4-dihydropyridine-2,3,5-dicarboxylate prepared by a modified
Hantzsch procedure.

Crystal data: monoclinic, P2₁/c, a = 9.291(4), b =
9.598(3), c = 27.175(13) Å, β = 99.53(3)°, γ = 0.103 mm⁻¹,
Z = 4, Dᵣ = 1.32 Mg.m⁻³, R(F) = 0.039, wR(F²) = 0.067 for
1420 unique reflections, S = 0.853.

The crystal structure is formed by single molecules
linked via hydrogen bonds. The substituted 1,4-dihydropyridine ring has a flat boat conformation with the N and C
atoms displaced by 0.126(3) and 0.202(3) Å, respectively,
from the plane through the other four C atoms which de-
finesthe base of the boat. The phenyl ring has an almost
planar conformation, with a dihedral angle between the in-
dividual planes of the 1,4-dihydropyridine and phenyl
rings of 87.1(1)°. The 3-nitro substituent on the phenyl
ring is rotated away from the coplanarity with the phenyl
ring by only 3.1°.

**COMPLEXES OF COPPER AND NICKEL
INVOLVING THIODIGLYCOLIC ACID**

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Thiodiglycolic acid as a ligand can be coordinated to
metal centres in different modes.¹ In our previous study we
have used thiodiglycolate dianion as a bridging ligand be-
tween two iron centres and we have found a week antiferro-
magnetic exchange.² We wanted to find if the exchange
was intramolecular only. Because we were not successful
in preparation of single crystals of iron complexes we have
decided for preparation of copper and nickel mono and
binuclear complexes.

Complexes of compositions

µ-[(pmdien)(H₂O)Cu(µ-ttdga)Cu(pmdien)(H₂O)](ClO₄)₂ (1),

[(pmdien)(H₂O)Cu(µ-ttdga)Cu(pmdien)(H₂O)](NO₃)₂ (2),

[(phen)Cu(µ-ttdga)(phen)](NO₃)₂·5H₂O (3),

[(mdpta)Ni(µ-ttdga)Ni(mdpta)](ClO₄)₂ (4),

[Cu(phen)(tdga)(H₂O)]H₂O (5), [Ni(bpy)(tdga)(H₂O)]H₂O (6),

[Ni(phen)(tdga)(H₂O)] (7),

[Ni(1,8-dan)(tdga)(H₂O)] (8)

and [Ni(phen)(tdga)(H₂O)]·H₂O (9),

where

H₂tdga = thiodiglycolic acid,
pmdien = pentamethyldiethylenetriamine,
phen = 1,10-phenanthroline,
mdpta = 3,3’-diamino-N-methylpropylamine,
bpy = 2,2’-bipyridyl,
1,8-dan = 1,8-diaminonaphthalene, nphen = 5-nitro-1,10-phenanthroline, have been prepared.

Temperature dependence of magnetic susceptibility was studied for copper binuclear complexes but no exchange between paramagnetic centres was found. Cyclic voltammetry of (1) and (2) proved possibility of reversible reduction of the complexes.

X-ray analysis of (1) proved coordination number five around CuII centres bridged by thiodiglycolate through one oxygen atom. Coordination is completed by one O atom from water molecule and three N atoms of pmdien. Structure (3) (see Figure) consists also of two pentacoordinated CuII atoms bridged by thiodiglycolate. One copper atom is coordinated through one O atom of the dianion and four N atoms from two phenanthrolines. The second Cu atom is coordinated by two oxygens of both carboxylate groups, S atom of thiodiglycolate and two N atoms of phenanthroline.

Grant support: IG 31703010 from UP Olomouc.


SEMI-EMPIRICAL ANALYSIS OF NICKEL COMPLEXES WITH TRITHIOCYANURIC ACID

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Recently, we prepared and structurally characterized the following complexes involving dianion of trithiocyanuric acid and nitrogen-donor ligands in the coordination sphere:

[Ni(taa)(ttcH)]¹ (Fig. 1), [Ni(bapen)(ttcH)].2H₂O² (Fig. 2) and [Ni(pmdien)(ttcH)]³ (Fig. 3)

(taa = tris(2-aminoethyl)amine, bapen = N,N'-bis(3-aminopropyl)ethylenediamine, pmdien = pentamethyldiethylenetriamine, ttcH₃ = trithiocyanuric acid). In addition to the X-ray structures of 1, 2 and 3 we have performed geometry optimizations of all the complexes. Geometries were optimized by the semi-empirical ZINDO1 method using the HyperChem 5.0 molecular modelling program package. As can be seen from the Table (see below) calculated bond lengths and angles are in good agreement with experimentally determined values.

Acknowledgement: We thank the Grant Agency of the Czech Republic for support (203/98/P220).

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**CRYSTAL STRUCTURE OF HALOALKANE DEHALOGENASE LINB FROM Sphingomonas paucimobilis UT26 AT 1.6 Å RESOLUTION**

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Haloalkane hydrolitic dehalogenase LinB from *Sphingomonas paucimobilis* UT 26 is the enzyme releasing chloride or bromide anion from *n*-halogenated alkanes. The enzyme was crystallized using the hanging-drop vapor-diffusion method at 278 K. The best crystals were obtained by micro-seeding with a PEG 6000 precipitant. They diffract to at least 1.6 Å using synchrotron X-ray under cryogenic (100 K) conditions. The crystal belongs to the orthorhombic space group P2₁2₁2 with unit-cell parameters a=50.29, b=71.70, c=72.73 Å. The crystal structure of LinB has been solved by molecular replacement with known structure of dehalogenase from *Rhodococcus* species and refined at 1.6 Å resolution to crystallographic R-factor of 16.5%. The monomeric enzyme is a spherical molecule and is composed, alike as the other members of the protein superfamily of the a/b-hydrolases, to two domains. Domain I has a/b type structure with central eight-stranded twisted b-sheet, domain II lies like a cap on top of domain I and consists mainly of α-helices.

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**PREPARATION AND CRYSTAL STRUCTURE OF LEAD TUNGSTEN OXIDE**

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Lead tungsten oxide (Pb₂WO₅) was prepared by solid state reaction of powdered WO₃ and PbO at 890°C [1]. Phase transformation take place during cooling of the product at 330°C [2]. Low temperature form of Pb₂WO₅ was created by this way. A good single crystal of the suitable size for X-ray structure measurement was chosen from the polycrystalline sample.

Intensity data were collected at room temperature on an Enraf-Nonius CAD4 diffractometer (Cu-Kα λ = 1.54056 Å).

The structure was solved by the direct method using the program SIR92 [3]. The lead and the tungsten atoms were refined anisotropically, the oxygen atoms were refined isotropically only. The minimised function was $w(Fo - Fc)^2$, where $w$ is Chebyshev polyonm. [4].

Crystal data for lead tungsten oxide stable below 330°C:

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Formula</td>
<td>Pb₂WO₅</td>
</tr>
<tr>
<td>Mol.wt.</td>
<td>678.245</td>
</tr>
<tr>
<td>Crystal system</td>
<td>monoclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 21/a</td>
</tr>
<tr>
<td>a, Å</td>
<td>12.679(6)</td>
</tr>
<tr>
<td>b, Å</td>
<td>5.856(1)</td>
</tr>
<tr>
<td>c, Å</td>
<td>7.273(6)</td>
</tr>
<tr>
<td>V, Å³</td>
<td>143.55(14)</td>
</tr>
<tr>
<td></td>
<td>146.25</td>
</tr>
</tbody>
</table>

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The tungsten atom is surrounded by four oxygen atoms. The fifth oxygen is connected with two atoms of lead and placed in the tungsten atom coordination sphere. Atoms of oxygen form deformed trigonal bipyramid around the tungsten atom.


**CYCLOSPORINS OF SYMMETRY P2₁ - GROUP OF PSEUDOISOMORPHOUS CLATHRATES**

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Cyclosporins, especially cyclosporin A (Cs A), are fungal metabolites widely used as an immunosuppressive drugs in transplantation surgery. The study of different cyclosporin solvates and phase modifications is very important for formulation of new medicinally applicable immunosuppressive preparates.

Several solvates of cyclosporin A, which crystallize in P2₁, P2₁2₁2₁ and P4₁ space groups are known. The P2₁ symmetry is suitable not only for cyclosporin A but for the other cyclosporins as well. In addition it is possible to incorporate some solvents to cyclosporin backbone and series of pseudoisomorphous structures crystallize in this way. Till now seven cyclosporin clathrates are known (6 of them were solved in our Laboratory):

The common characteristics of these clathrates are: Conformation of the basic cyclosporin backbone does not change. The solvent is fixed in a backbone cavity (volume about 430 - 560 Å³) by van der Waals forces only, no hydrogen bond contacts were found. The solvent thermal-motion parameters are very high, sometimes it is impossible to localize the solvent precisely even from low-temperature data.

The authors wish to thank Prof. Z. Žák and Dr. I. Císařová for low temperature measurement.


**APPLICATION OF MODERN COMPUTER GRAPHIC METHODS FOR ELECTRON DENSITY VISUALIZATION**

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Several methods of modern computer graphics were used for developing a program for fast 3D electron densities visualization. Following tools and methods were applied:

a) OpenGL graphic language [1] - a computer API designed originally by SGI company for communication between software and graphic hardware. Today, very fast graphic cards which accept this API are available not only for expensive SGI machines but for inexpensive PC as well.

b) Marching cube algorithm [2] - a mathematical algorithm for visualization voxelized information like results of X-ray tomography in medicine. Application of this algorithm to ELD map leads to very fast construction of 3D objects which can represent those ELD map.
c) Stereographic visualization [3]. Stereographic visualization is a method for computation of two images for left and right human eye. This two images could then be delivered separately (usually by help of LCD glasses) to correct eye. The brain can see the object like real 3D one.

All these methods were combined in Marching Cube ELD program. This program is able to read output from CRYSTALS [4] crystallographic package. The program is capable to visualize ELD in several ways, manipulate with the map and add new atoms to the map. The solvation of several cyclosporin A crystal forms was determined by this way. New functions of this program, especially input from other systems then CRYSTALS, are under development.


Ag, Au AND Pd COMPLEXES WITH N-BASES: STRUCTURES AND VIBRATIONAL SPECTRA

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Vibrational spectral studies of interaction between adsorbates and surfaces of SERS (Surface-enhanced Raman Scattering) active metals are the subject of interest. To facilitate elucidation of the structure of the surface species on Ag, Au and Pd colloids by comparison of SERS spectra of studied molecules adsorbed on colloids with vibrational (Raman and infrared) spectra of analogous synthetic complexes of molecules with SERS active metals, it is very useful to know the structure of this complexes.

During the work, five new complexes were prepared. The central atoms were SERS active metals: Ag, Au and Pd. Ligands were studied bases, i.e. acridine (acr), bipyridine (bpy) and phthalazine (pht). Charge compensating ions are chlorides or nitrates. Silver coordination sphere is pseudotetrahedric in all cases, gold and palladium coordination spheres are tetragonal. It corresponds to d¹⁰ electron configuration on Ag⁰ and d⁰ configuration on Au³⁺ and Pd²⁺.

The structures of the two complexes prepared recently, [Ag(acr)₂NO₃] and [Au(pht)Cl₃], are given in Fig.1 and 2. Complexes [Ag₂(bpy)₂(NO₃)₂], and polymeric [Ag₂⁺(H₂O)(NO₃)(pht)₂].NO₃.H₂O, are an example how the combination of vibrational and structural information was used for determination of the surface species structure. The structure of the last complex - [Pd(bpy)Cl₂].1/2C₇H₈O₂ - helped to decide between chemisorption and physisorption of bipyridine on the surface of Pd colloid.

All complexes were characterized by X-ray analysis, infrared spectroscopy and Raman scattering. Some of them were characterized by DSC measurements.


Acknowledgement: Financial support of this work by 203/97/0259 awarded by GA of Czech Republic and grant “Creative activity of the students” – G4 FRVS 1713/99 awared by Ministry of Education of Czech Republic.

PROGRAM FOR STRUCTURE DETERMINATION OF 1D CRYSTALS

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The description of a simple demonstration program for structure determination of (hypothetical) one-dimensional crystals will be given. The program, called mc.xls, is a MS Excel 5.0 application, i.e. it uses Excel sheets to provide input and output and Excel modules with Visual Basic code to run.

The program gives the opportunity to try the single crystal structure determination on simple examples. Working within one dimension, the Fourier transforms can be drawn as simple charts that can be understood intu-
In the fourth step you have to solve the phase problem by means of Patterson synthesis. The output has two parts: graphical (see Chart 1) and numerical in the form of peaks table.

In the fifth step you refine the structure in the direct space using repeated Fourier synthesis. You can choose any of five standard types of Fourier synthesis: Patterson synthesis based on $|F_{obs}|^2$, checking Patterson on $|F_{calc}|^2$, Fourier on $F_{obs}$ checking Fourier on $F_{calc}$, and difference Fourier on $(F_{obs}-F_{calc})$. Each type of synthesis gives the graphical output similar to that in Chart 1.

The sixth step consists in refinement in the reciprocal space by means of Least Squares method. The last two steps are usually combined and repeated several times to get the final structure model. At the end, when you think the structure is complete, you can calculate distances and draw a simple picture.

The program provides graphical user interface and contains a help system including small demo. To use it, just a basic knowledge of computers and single crystal X-ray structure determination should be enough. The latest version, which runs under MS Windows 3.1 or higher and MS Excel 5.0 or higher, is freely available at request to mirek@natur.cuni.cz.

QUANTITATIVE XRD PHASE ANALYSIS OF FLY ASH COMPOSITES

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The work summarises the results of the research of fly ashes, their mixtures and composites, aimed at their quantitative phase analysis. The percentages of individual components were determined using several methods of X-ray powder diffraction. A procedure of the preparation and application was developed for each of them.

The RIR values were obtained for each phase by evaluation of diffraction patterns of 1 : 1 analyte - corundum mixtures, of non-binary mixtures, and by modelling diffraction patterns of binary 1 : 1 mixtures; based on known crystal structure of the phases involved.

The application of the internal standard method required preparation of several artificial mixtures for each phase. These were used to measure the diffraction peak intensity and derive the linear regression parameters of the dependence of diffraction intensities on the weight percentage of the analyte.

The external standard method proved to be ineligible for the available diffractometer arrangement.

To apply the XQPA method (Weiss et al., 1983), two databases were composed. The first one contains d-values and corresponding intensities of modelled pure phases, the second one was formed from real values obtained by measurement of reference samples of pure phases.

All methods were applied on the 30 artificial test mixtures, whose composition was close to real fly ashes and materials derived of them. The acquired results indicate that the RIR method is very well applicable both with that the factors derived from reference 1:1 phase - corundum mixtures, and factors derived from modelled mixtures. The XQPA method is also applicable to the quantitative analysis of fly ash mixtures, yielding better results when the database of modelled reference materials is used. Both above methods can determine only the relative proportions of individual crystalline components, as there are always some amorphous components in the real mixtures.

The percentage of the amorphous component in the mixture can be determined using the internal standard method, as proved by the results obtained. A disadvantage of this method is the very time-consuming preparation all calibration mixtures. Therefore, a combination of a direct method (RIR or XQPA) with the internal standard method is recommended. The results from direct methods must be recalculated to the absolute values of phase percentages,
using the determination of the major components by the internal standard method.

All procedures were tested with four real fly ash mixtures. The absolute error values achieved with artificial test mixtures varied in most cases between 5 and 10%.

As a conclusion, the work presents an optimum analytical scheme of XRD quantitative phase analysis of fly ashes, including also the time requirements of each method involved.

### MORFOLOGICKÉ TYPY MONAZITU Z VEPORIKA (ZÁPADNÉ KARPATY)

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Monazit je hlavný nositeľ prvkov ľahkých vzácných zemin (La, Ce, Pr, Nd, Sm, Eu) v kryštaliniku Západných Karpát. Patrí k frekventovaným akcesorickým minerálom v niektorých typoch magmatitov a metamorfitov. V juhozápadnom veporiku (oblasť severne od Lučenca) vytvára kontrastné anomálné koncentrácie vo zvetralinách, v ktorých obsahy niekoľkonásobne prevyšujú bežné obsahy akcesorického monazitu v horninách. Možno opodstatnené predpokladať, že pôvod monazitov je spojený s minerogenetickými (pravdepodobne hydrotermálnymi) procesmi, ktoré umožnili migráciu a koncentrovanie prvkov vzácných zemin.

Ideálizovaný chemický vzorec monazitu je (Ca, Ce, La, Pr, Nd, Sm)PO₄. Minerál monazit je izomorfná zmes, ktorá sa skladá spravidla z troch zložiek: monazitu (CePO₄), huttonitu (ThSiO₄) a brabantitu (CaTh(PO₄)₂). [1]. Jeden jemnoklinický (bodová grupa 2/m), súmernosť kryštálové štruktúry je P₂₁/m. V štruktúre sa nachádzajú PO₄ tetraédre a REEO₉ polyédre ktoré vytvárajú tzv. polyédricko - tetraédrické reťazce. Reťazce sú rovnoznejšie s [001], základná bunka obsahuje 4 vzorcové jednotky [2].

Pre výskum monazitu sme získali koncentráty z kryštalov, ktorých sme vyseparovali monominerálne frakcie monazitu. Morfologické štúdium sme robili na súboroch minimálne 200 monokryštalov resp. zrastov. Priebeh prívech monazitu v koncentrátoch sa pohybuje v rozmedzí 0,1 - 2 mm ale nie sú zriedkavé ani 3-4 mm kryštály, zrasty dvoch jedincov a polykryštálové zrastové agregáty.

Kryštály sme zaradili do šiestich morfologických skupín (izometrické, izometricko-tabuľkovité, tabuľkovité, tabuľkovito-prizmatické, prizmatické, prizmaticko-izometrické; obr. 1). Prevládajúcu morfologický typom sú izometrické a izometricko-tabuľkovité kryštály (60 - 90 %).

Zrasty monazitov sú najčastejšie podľa (100) a (001) a sú pomerné hojné. V študovalých vzorkoch sme našli penetračné zrasty viacerých (obr. 2) rôzne velkých jedúcinnov, ktoré však vždy tvorili rovnako farebné variety.

Monazit na študovanom území vystupuje v pomerné širokej škále farieb, pričom jednotlivé farebné variety sú kvantitatívne rovnako zastúpené. Vo všetkých vzorkoch prevládajú kryštály žltomodrej farby (v priemere od 53 - 73 %). Zostatok sú tmavé, zelenkasté a červenkasté monazity.

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COMBINATION OF MÖSSBAUER SPECTROSCOPY AND X-RAY POWDER DIFFRACTION - A POWERFUL TOOL FOR THE CHARACTERIZATION OF IRON OXIDES QUA PRODUCTS OF THERMAL TRANSFORMATIONS

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FeO, Fe3O4 (magnetite), α-Fe2O3 (hematite), β-Fe2O3, γ-Fe2O3 (maghemite) and ε-Fe2O3 can be formed as products of the thermally induced processes (oxidation, the thermal decomposition of iron salts and minerals or solid-state reactions of iron compounds [1–5]). Majority of these processes is accompanied by the mutual structural transformations of the individual oxide forms. Thus the miscellaneous mixture of iron oxides can be present in the thermally treated samples. The combination of Mössbauer spectroscopy and x-ray powder diffraction is a very effective technique for the identification of the individual phases and determination of the reaction mechanism.

The quantitative results accessible by both methods yield important information for the study of reaction kinetics. The different well crystalline forms as well as the amorphous or superparamagnetic nanoparticles of iron oxides can be isolated by the appropriate choice of the heat treatment conditions and the following chemical separation. Their magnetic characterization, the relation between the structure and magnetic properties, particles size, structure defects, temperatures of magnetic transitions can be successfully investigated using Mössbauer measurements in a wide range of temperatures (4.2–1200 K) and x-ray powder diffraction analysis.

In this work, our previous published and literary data respecting the structural and magnetic properties of iron oxides, their hyperfine Mössbauer parameters, processes of their formation and the mechanism of thermal transformation will be reviewed and summarized. The possibility to discern the individual oxide phases using Mössbauer spectroscopy and x-ray powder diffraction, advantages and disadvantages of both methods will be discussed.