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Keywords: molecular recognition, selectivity, weak interactions

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Inclusion of Molecular Iodine into Channels of the Organic Zeolite Gossypol

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Gossypol, a phenolic pigment extracted from cotton seeds, demonstrates unique ability as a host compound and its polymorphism[1] makes gossypol an interesting object of solid supramolecular chemistry.

Channel-type zeolite has been obtained by desolvation of the unstable clathrate of gossypol with dichloromethane. Removal of the solvent molecules does not affect the crystal structure of the host: channels are retained, although they show a slight contraction (4%). Sorption experiments performed in the gas phase or in water showed that the zeolitic form of gossypol can take up molecular iodine. The crystal structure of the gained complex is isomorphic with the structure of the dichloromethane clathrate where hydrogen-bonded columns of gossypol molecules are packed by means of Van der Waals forces into a porous architecture. In the complex, iodine molecules enter the extended cavities of the channels within the zeolitic structure, occupying 1/8 of the possible sites. The host-guest ratio of the obtained product depends on the size of crystallites of the initial zeolite and the absorption temperature. The higher sorption temperature the higher amount of absorbed molecules but the lesser crystallinity of the product. However, the iodine molecules can be removed with the help of vacuum giving back to the initial zeolite.

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Keywords: gossypol, inclusion compound, organic zeolite

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Crystal Structure and Properties of a Tetrakis(tert-butylthio) Butatriene Compound

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1,2,3-butatrienes have long attracted organometallic chemists because of their highly unsaturated structure [1]. Since the butatrienes obtained here keep some energy in their skeletons, they must be reactive molecules, and thus their applications to further organic transformations will be interesting subject [2].

The aim of this work is to synthesis [3] and to determine the crystal structure of Tetrakis(tert-butylthio)butatriene compound. Crystal of Tetrakis(tert-butylthio)butatriene was mounted on a Rigaku R-AXIS Rapid-S Diffractometer with a graphite monochromatized MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). The structure was solved by direct method with SIR92 [4] and refined with Crystals.

Crystal data: C₂₀H₃₆S₄, the compound is monoclinic, space group P2₁/n, $a = 11.061(6)$, $b = 10.850(4)$, $c = 11.271(6) \text{ \AA}$, $\beta = 116.427(12)^\circ$, $V = 1211.51(10) \text{ \AA}^3$, $Z = 4$, $D_{\text{calc}} = 2.219 \text{ g/cm}^3$, $F(000) = 880.00$, $\mu(\text{MoK}\alpha) = 7.86$.

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Keywords: small organic molecules, sulfur compounds, structures of organic compounds

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Crystal Structure of a Tetrakis(4-chlorophenylthio) Butatriene Compound

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Compounds with cumulated double bonds, such as allene or ketene, are fairly reactive because of their strained structures, thus, effective synthesis of these compounds is still challenging in organic chemistry. Construction of three cumulated carbon-carbon double bonds is more difficult problem, and the effective synthetic methods of butatrienes are still very few [1].

The aim of this work is to synthesis [2] and to determine the crystal structure of Tetrakis(4-chlorophenylthio)butatriene compound. Crystal of Tetrakis(4-chlorophenylthio)butatriene was mounted on a Rigaku R-AXIS Rapid-S Diffractometer with a graphite monochromatized MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). The structure was solved by direct method with SIR92 [3] and refined with Crystals.

Crystal data: C₂₈H₁₆Cl₄S₄. The compound is monoclinic, space group P2₁/c, $a = 6.9785(8)$, $b = 8.6803(9)$, $c = 22.884(2) \text{ \AA}$, $\beta = 93.887(6)^\circ$, $V = 1383.0(3) \text{ \AA}^3$, $Z = 2$, $D_{\text{calc}} = 1.495 \text{ g/cm}^3$, $F(000) = 632.00$, $\mu(\text{MoK}\alpha) = 7.47$.

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Keywords: sulfur compounds, small organic molecules, structures of organic compounds

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Structural Insights of a Potential Inhibition Against Leishmania Major

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Leishmaniasis is a tropical disease caused by a protozoal parasite of the order Kinetoplastid. According to the World Health Organization reports, 88 countries are affected, with 12 million infected people and approximately 350 million people at risk. The need for new drugs for the treatment of leishmaniasis infections comes from a lack of safe drugs and the serious secondary effects observed in available chemotherapy. Looking for new bioactive substances [1], potentially useful against leishmaniasis, we used both PRTase adenine phosphoribosyltransferase from *L. tarentolae* and parasite *L. major* as a model system to screen the inhibitory capacity of one small molecule compound from Brazilian plant. The data collection was performed using Enraf Nonius KappaCCD at room temperature. The structure was analyzed from 1425 reflections with $I > 2\sigma(I)$ and refined to R1-values of 0.033 [1]. The molecules are joined in crystal structure through five twice classical O-H...O hydrogen bonds linking the atoms O4-H4...O5ⁱ [$i = -x+3, 0.5+y, -z+1$], O5-H5...O2ⁱⁱ [$ii = x, y, 1+z$], O2-H2...O4ⁱⁱⁱ [$iii = x+2, y-0.5, -z$], O1-H1...O3^{iv} [$iv = x-1, y, z$] and O3-H3...O1^v [$v = -x+2, 0.5+y, -z$] with distances between donor and acceptor of 2.707(1), 2.808(1), 2.780(1), 2.736(1) and 2.843(2) Å, respectively. The theoretical analysis of occupancy factor for the hydrogen atoms of two last ones is consistent to crystallographic model.

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Keywords: leishmaniasis, inhibition, phosphoribosyltransferase

P.06.10.4

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Crystal Structures of trans- and cis-octenes

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n-Alkanes and most of their α - and α,ω -substituted derivatives show a remarkable alternation in their melting points with increasing chain length [1,2]. The same phenomenon also occurs within series of substances with a constant number of C-atoms. One of these isomeric series are the octenes where the melting points of the 2- and 4-octenes are relatively higher than those of 1- and 3-octenes. This holds for the series of *cis*- as well as for the series of *trans*-isomers.

Single crystals of all *trans*-octenes have been grown *in situ* using a miniature zone melting procedure [3], and their X-ray analyses have been carried out. Crystal structures of *cis*-octenes have been determined by X-ray powder diffraction using lattice energies minimisations [4]. The structural similarities and differences between the *trans*- and *cis*-isomers of each serie could be analyzed based on the packing arrangements of hydrocarbon chains and the end groups. The melting point alternation in both isomeric series can be explained based on the calculations of lattice energies.

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Keywords: molecular packing, lattice energy calculations, melting point alternation

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Influence of the p-substituent for the Diastereomeric Resolution of Carboxylic Acids

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The salts of (S)- and (R)-1,4-benzodioxane-2-carboxylic acid with three (S)-1-arylethylamines have been investigated [1]. Their melting points and their solubilities in alcoholic solvents revealed large differences between the benzodioxanecarboxylates of (S)-1-(p-nitrophenyl)ethylamine and (S)-1-(p-methylphenyl)ethylamine. Therefore this latter amines were selected to resolve (\pm)-1,4-benzodioxane-2-carboxylic acid by diastereoselective crystallisation finding that both of them display a high resolution ability for such substrate, which contrasts with the null efficiency of unsubstituted 1-phenylethylamine. The crystal structures of the salts showed that there is correlation between the efficiencies of the optical resolutions of the amines with the resolving reagents and the crystal structures of the salts. A hydrogen bond layer was found to be common to the less soluble salt crystals, consisting of stable columnar structures with planar boundary surface [2]. In contrast, in the corresponding more soluble salts no particularly stabilized crystal structure is formed, only columnar structures are present. These results strongly suggest, that for successful resolution it is necessary realize hydrogen bond layers, consisting of stable columns with planar boundary surfaces, in the crystal of one of the pairs of diastereomeric salts

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Keywords: diastereomeric method, chiral discrimination, crystal engineering

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Structure by 2D NMR and X-ray Crystallography of a Triterpene from *M. imbricata*

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Many specimens of *Maytenus* (Celastraceae) are used in medicine folk in different Brazilian regions and shown a diversity of secondary metabolites, like as flavonoids, glycosides, maitansinoids, alkaloidic and non alkaloidic sesquiterpenes, friedelanes, oleananes, lupanes, quinonoid triterpenes and pentacyclic triterpenes (PCTTs) of the other series. To PCTTs are attributed pharmacological properties like as antiseptic, ant-asthmatic and antimicrobial action, antispermatogenic, antispasmodic, analgesic and ant-ulcer effect, insecticide, antitumoral, moluscicide, allelopathic and anti-inflammatory effect.

The compound was isolated from the powder extract of *Maytenus imbricata*. From the Mass Spectrometry (MS), ¹H and ¹³C NMR and X-ray data it was possible to determine the molecular formula C₃₀H₅₀O₂. The structure of the compound were established by two-dimensional NMR spectroscopic techniques and later confirmed by single crystal X-ray diffraction as 3 β ,30-dihydroxy-lup-20(29)-ene.

The crystal structure shows one molecule in the asymmetric unit. The symmetry was examined carefully and it was concluded that *P4*₁ is the correct space group. The crystal packing is stabilized by two intermolecular hydrogen bonds, which give rise to the formation of five infinite helical chain along *c* per unit cell. Analysis with Mogul program showed all bond length and bond angle between corresponding atom in the molecule are in good agreement with expected.

Keywords: triterpenes, NMR, X-ray crystallography

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The Structure Characterization of Molecules with ESR Spin Labels of Pyroline and Piperidine Type

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A series of molecular structures bearing ESR spin labels of pyroline and piperidine types has been determined. The review of all structures shows common features of hydrogen bonds formed by nitroxyl oxygen atoms and neighboring hydrogen donors. In addition to molecules bearing a single spin label (SSL) [1], the list contains a number of double spin labels (DSL) and three spin labels (TSL). Intentionally rigid spacers bearing the spin labels fix the inter-radical distance in interval 6-30 Å in case of DSLs. The spin label moiety itself remains virtually untouched by any external influences as the chemical composition of spacer, crystal packing, hydrogen bonds, etc.

The double spin label [3,3'-oxybis(ethyleneoxycarbonyl)bis(2,5-dihydro-2,2,5,5-tetramethyl pyrrol-1-yloxy)], i.e. DSL with a poly(ethyleneoxide) spacer was prepared as a paramagnetic tracer for ESRI studies of diffusion processes in polymer gels and concentrated polymer solutions [2]. It is of special interest also for its phase transition at 248 K where the molecules lose their two fold symmetry and the space group transfers with lowering the temperature from Iba2 to Pbc2₁ keeping the molecular stacking virtually unchanged.

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Keywords: spin label, organics, X-ray structure

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Crystallization of the Azithromycin 11,12-hydrogenborate. Can We Have the Control?

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