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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, friedelanones, 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 Pyrroline and Piperidine Type

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A series of molecular structures bearing ESR spin labels of pyrroline 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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Crystallization of the Azithromycin 11,12-hydrogenborate. Can We Have the Control?

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