

physico-chemical properties.

NN414 is a very weak acid with $pK_a = 8.5$, $\log P = 1.6$, and molar weight $M_w = 291.8$ g/mol. Two true polymorphs, A and B, of this compound have been identified. Polymorph A crystallizes in needleshaped crystals with a triclinic unit cell by precipitation from a variety of solvents such as acetic acid, acetonitrile, diluted ammonia, methanol, N-methyl-pyrrolidone, 1-propanol, or 2-propanol [1]. Polymorph B forms prismatic crystals by precipitation from methanol or ethanol, and this unit cell is rhombohedral [1]. Mixtures of A and B can also be obtained. Both polymorphs are highly crystalline. Polymorph A melts at approximately 257°C whereas polymorph B melts at approximately 269°C [1].

To establish the thermodynamic relationship between A and B, different experiments concerning crystallization, density of mass, solubility and melting behaviour were carried out using hot stage microscopy, He-pycnometry, intrinsic solubility, and differential scanning calorimetry [2]. The results of these experiments unanimously point to an enantiotropic relationship between A and B, with A being thermodynamically stable at ambient conditions, and B being the stable polymorph at elevated temperatures. A transition point temperature between A and B has been estimated to $T_{trans} = 215^\circ\text{C} \pm 15^\circ\text{C}$ from the differences in melting enthalpies.

[1] Jensen A.F., Junager F., Jessen C. U., Kornø H. T., *International Patent Application*, 2004, WO2004005299. [2] Bernstein J., Davey R. J., Henck J.-O., *Ang. Chem. Intl. Ed.*, 1999, **38**, 3440-3461.

Keywords: polymorphism, phase transition, drug molecule

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Epimerization of α -amino Nitriles to Single Stereoisomers in the Solid State

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Enantiomeric or diastereomeric enrichment to a single isomer has had only limited success to date. We have found that a diastereomeric mixture of α -amino nitriles, which was prepared by the diastereoselective Strecker reaction using the amino alcohol as a chiral auxiliary, thermally epimerizes to a single stereoisomer in the solid state. X-ray structure analyses have shown that the α -amino nitrile, [1*S*,2*R*,(*SR*)]-N-cyano(phenyl)methyl-1-aminoindan-2-ol, epimerizes at 65 °C to give a single diastereomer with an (*S*)-configuration ((*S*)-isomer) at the α position to the nitrile moiety. Namely the (*R*)-isomer is thermally unstable and the (*S*)-isomer is stable in the solid state. In DMSO solution, the diastereomerically pure (*S*)-isomer epimerizes at room temperature to give a 1 : 1 mixture of the (*S*)- and (*R*)-isomers. Therefore the cause of thermal instability of (*R*)-isomer in the solid state should be ascribed to the crystal structure. In the (*R*)-isomer crystal there are two hydrogen bonds, an intramolecular N-H...O and an intermolecular CN...HO bonds which promote dissociation of the cyanide anion. On the other hand, the intramolecular O-H...N bond in the (*S*)-isomer crystal retards the dissociation of the cyanide anion. As a result, the (*R*)-isomer selectively epimerizes to the (*S*)-counterpart via an iminium or imine intermediate.

Keywords: epimerization, solid state isomerization, diastereomeric enrichment

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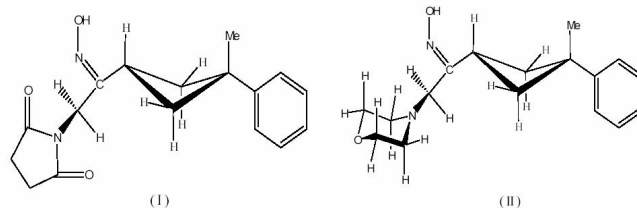
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Two Oxime Derivatives Including Succinimid and Morpholin Groups

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The title compounds, 1-methyl-1-phenyl-3-[1-hydroxyimino-2-succinimido] ethyl] cyclobutane, $C_{17}H_{20}N_2O_3$, (I), and 1-(3-methyl-3-phenylcyclobutyl)-2-morpholin-4-yl-ethanone oxime, $C_{17}H_{24}N_2O_2$, (II), crystallize in space group P2₁/c, [1]. Each compound contains a cyclobutane ring, an oxime group and a benzene ring [2]. The cyclobutane ring in (II) is more puckered than in (I). In (II), morpholin

ring adopts a chair conformation. Although the oxime moiety in (I) has an E configuration, the oxime moiety in (II) has a Z configuration. The molecules in (I) are linked by O-H...O and C-H... π (benzene) interactions, forming a two-dimensional network, while the molecules in (II) are connected by O-H...N interaction.



[1] Özdemir N., Dinçer M., Yılmaz İ., Çukurovalı A., *Acta Cryst.*, 2004, **E60**, o145-o147. [2] Ahmedzade M., Çukurovalı A., Koparır M., *J. Chem. Soc. Pak.*, 2003, **25**, 51-55.

Keywords: crystal structures, organic molecule, drug action

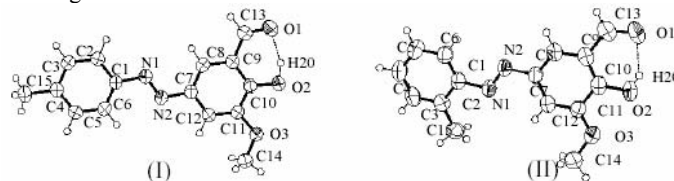
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3-Methoxy-5-(4-methylphenyldiazenyl)salicylaldehyde and 3-methoxy-5-(2-methylphenyldiazenyl)salicylaldehyde

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The two title molecules, both $C_{15}H_{14}N_2O_3$, are roughly planar and display a *trans* conformation with respect to the -N=N- double bond, as found for other diazene derivatives. In both compounds, there are intramolecular O-H...O hydrogen bonds and the crystal packing is governed by weak intermolecular C-H...O hydrogen bonds and π - π stacking.



The structures of both (I) and (II) (Figs. 1 and 2) contain two essentially planar fragments, *viz.* one monosubstituted (C1-C6) and one trisubstituted phenyl ring (C7-C12). The aromatic rings are in a *trans* conformation with respect to the azo double bond. The C14-O3 bond length [1.413(2)Å in (I) and 1.429(4)Å in (II)] is approximately equal to that usually associated with a methyl C-O bond in a methoxy group attached to an aromatic ring (1.424Å; Allen *et al.*, 1987).

[1] Allen F. H., Kennard O., Watson D. G., Brammer L., Orpen A. G., *J. Chem. Soc. Perkin Trans.*, 1987, **2**, S1-19.

Keywords: azo groups, π - π stacking, aromatic ring

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Crystal Structure of 2-cyclohexyl-5-formyl-6-(4-bromophenyl) Imidazo[2,1-b] [1,3,4] Thiadiazole

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1,3,4-thiadiazole nucleus is associated with a broad spectrum of biological activities, possibly due to built in toxophoric thioamide (S-C=N-) unit. Biosteric nature with biologically significant thiazole moiety and its non-carcinogenic nature. A lot of work on the synthesis and biological activities of condensed imidazo(b) thiazoles has been reported since the discovery of novel broad spectrum anthelmintic, Tetramisole. The trend has been shifted to explore the drugs containing biosteric thiadiazole ring in place of thiazole ring of tetramisole *viz.*, imidazo (2,1-b) -1,3,4-thiadiazoles and their derivatives. The title compound screens them for their pharmacological activities.

Title compound will be good intermediates to synthesise various pharmacologically active compounds.

The compound crystallizes as colourless plates in monoclinic with space group $P2_1/n$ and cell dimensions $a=8.493(7)\text{\AA}$, $b=17.071(1)\text{\AA}$ and $c=11.875(1)\text{\AA}$. The structure was solved by SHELX97 program (Sheldrick 1997) and refined to a R factor of 0.0597. The details of the weak interactions are discussed in this paper.

Keywords: X-ray crystallography, small molecules, organic compounds

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Structure of 2-[4-(Dimethylamino)phenyl]-4,5-diphenyl-1H-imidazole Isopropanol Solvate

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Different types of organic compounds have been synthesized using microwave-assisted organic synthesis [1] and Usyatinsky & Khmel'nitsky [2] have reported the use of this technique in the preparation of 2,4,5-substituted imidazoles. Their synthetic procedure involved the condensation of 1,2-diaryethandienones with aldehydes and ammonium acetate as the source of ammonia with an acidic support (acidic silica) in a microwave oven. We synthesized 2-[4-(dimethylamino)phenyl]-4,5-diphenyl-1H-imidazole using a similar technique in the absence of the acidic support media. Recrystallization of the reaction product from isopropanol afforded the title compound, as shown by single-crystal X-ray structure determination. The title compound, $C_{23}H_{21}N_3 \cdot C_3H_8O$, crystallizes with two independent molecules and two solvent molecules in the asymmetric unit. These are connected through hydrogen bonds between the NH group of the imidazole ring and the O atom of the isopropanol solvent molecule, as well as between the N atom of the imidazole ring and the OH group of the isopropanol solvent molecule.

[1] Lidström P., Tierney J., Wathey B., Westman J., *Tetrahedron*, 2001, 57, 9225-9283. [2] Usyatinsky A. Ya., Khmel'nitsky Y., *Tetrahedron Lett.*, 2000, 41, 5031-5034.

Keywords: microwave materials, organic structures, hydrogen bonds

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Substituents at Oxygen Influence N,O-Distances in Thiohydroxamates

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The N,O bond in O-derivatives of N-(hydroxy)thiazole-2(3H)-thiones may selectively be cleaved upon thermal (60–80 °C) or photochemical (300–350 nm) excitation. This circumstance along with additional useful chemical properties causes N-oxy-substituted thiazolethiones to be unprecedentedly versatile and efficient precursors for the generation of reactive intermediates such as carbon or oxygen centered radicals [1].

In view of the fact that N-alkoxycarbonyloxy-, N-acyloxy-, and N-alkoxy-derivatives of thiazole-2(3H)-thiones differ significantly in their stability, selected candidates of each set of thiones (a total of 14 compounds) were investigated by single-crystal X-ray diffraction at temperatures ranging between 298 K and 100 K.

The results of the study indicate that the N,O bond length in N-oxy-substituted thiazole-2(3H)-thiones increases along the series of substituents at oxygen H ~ alkyl < acyl < alkoxy-carbonyl [2,3].

[1] Hartung J., Schwarz M., Svoboda I., Fuess H., Duarte M.T., *Eur. J. Org. Chem.*, 1999, 1275. [2] Hartung J., Schneiders N., Bergsträsser U., *Acta Cryst.*,

2005, E61, o421. [3] Hartung J., Bergsträsser U., Schneiders N., Altermann S., Svoboda I., Fuess H., *in preparation*.

Keywords: heterocyclic compounds, single-crystal X-ray diffraction, nitrogen oxygen bond

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X-ray Induced Radiation Damage in Taurine – a Combined X-ray and Raman Study

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Taurine is one of the lesser-known amino acids, nevertheless, it plays a role in the body and is essential to newborns of most mammal species, and it also protects our bodies against damage from radiation. Along with methionine, cystine and cysteine, it is a sulphur amino acid. The taurine molecule ($\text{NH}_3^+-\text{CH}_2-\text{CH}_2-\text{SO}_3^-$) is small and therefore a well suited model compound for this family of amino acids, as well as for describing the radiation damage in more complex sulphur containing amino acid systems.

Taurine exposed to ^{60}Co gamma rays have been shown to produce traces of different radicals [1], which are detectable with EPR. However, when taurine is irradiated with x-rays, an anisotropic change in the lattice constants is observed, where the crystal expands mainly along the c-axis of the unit cell. The system has been investigated with single crystal diffraction data at 120K and at room temperature, and separately with time resolved high resolution powder diffraction in combination with Raman spectroscopy. The Raman spectra reveal a red shift of the S-O stretching frequency as a function of exposure time. The single crystal data show a continuous rise of charge located approximately 1Å away from one of the oxygen atoms. This finding along with the Raman observation suggest the existence of an x-ray induced proton transfer within the taurine crystals rather than pure radical formation.

[1] Bulut A., Karabulut B., Tapramaz R., Koksall F., *Radiat. Phys. Chem.*, 2000, 58(2), 149-52.

Keywords: diffraction, radiation damage, Raman spectroscopy

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Crystal Structure of 2-Nitro-3,4,4-trichloro-1-mono(dodecyl thio)-1-mono[(4-fluorophenyl)piperazine]-1,3-butadiene Compound

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Some thio substituted halodiene derivatives show an excellent biological activities such as herbicides, insecticides, fungicides etc. from US-Patent [1]. It is known that piperazine compounds are important chemicals of clinical chemistry. Also, piperazine compounds were used in gen-transfer [2].

The aim of this work is to synthesis and to determine the crystal structure of 2-Nitro-3,4,4-trichloro-1-mono(dodecylthio)-1-mono[(4-fluorophenyl) piperazine]-1,3-butadiene compound.

Crystal structure of 2-Nitro-3,4,4-trichloro-1-mono(dodecyl thio)-1-mono[(4-fluorophenyl)piperazine]-1,3-butadiene compound was mounted on an Rigaku R-AXIS Rapid-S Diffractometer with a graphite monochromatized $\text{MoK}\alpha$ radiation ($\lambda=0.71073\text{\AA}$). The structure was solved by direct method with SIR92 and refined with Crystals [3].

Crystal data : $C_{26}H_{37}Cl_3FN_3O_2S$, triclinic, $P\bar{1}$, $a=7.0933(8)$, $b=8.3802(6)$, $c=27.40930(10)\text{\AA}$, $\alpha=76.418(4)$, $\beta=77.526(4)$, $\gamma=71.231(4)^\circ$ $V=1481.955(5)$, $Z=2$, $D_x=1.302\text{ g/cm}^3$, $F(000)=612$, $\mu(\text{MoK}\alpha)=4.13\text{ cm}^{-1}$.

[1] Diamond Alkali Company (Ert.H.Bluestone), U.S. Pat. 3021370, *Chem. Abst.*, 1962, 57, 3293c. [2] Zhao S., Miller A.K., *Tetrahedron Lett.*, 1996, 37, 4463. [3] Altomare A., Cascarano G., Giacovazzo C., Guagliardi A., Burla M., Polidori G., Camalli M., SIR92, *J. Appl. Cryst.*, 1994, 27, 435.

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