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The molecular and crystal structure of the title compound, C₂₁H₁₂ClN₃O, has been determined by X-ray single crystal diffraction technique. The compound crystallizes in the triclinic, space group *P-1* with unit cell dimensions $a=8.8342(9)$, $b=10.2301(8)$, $c=11.2401(9)$, $\alpha=76.473(6)^\circ$, $\beta=84.912(7)^\circ$, $\gamma=64.419(6)^\circ$, $V=890.74(13)\text{Å}^3$, $Z=2$, $R_f=0.045$ and $wR_2=0.130$. The crystal structure of the title compound phthalonitrile derivatives, C₂₁H₁₂ClN₃O, is stabilized by π - π stacking interactions.

Calculational studies were performed by using AM1, PM3 semi-empirical and DFT methods. Geometry optimizations of compound have been carried out by using three semi-empirical methods and DFT method and bond lengths, bond and torsion angles of title compound have been determined. Atomic charge distribution have been obtained from AM1, PM3 and DFT. In order to determine conformational flexibility on the molecule, molecular energy profile of the title compound was obtained with respect to the selected torsion angle T(C21-C16-N3-C15), which is varied from -180° to $+180^\circ$ in every 10 via PM3 semi-empirical method.

Keywords: single crystals; crystallographic analysis; DFT

FA4-MS05-P22

Crystal Structures of Ferrocenyl- Phosphazene Derivatives. Asli Ozturk^a, Nuran Asmafiliz^b, Tuncer Hokelek^a, Zeynel Kilic^b. ^a*Hacettepe University, Department of Physics Engineering, 06800 Ankara, Turkey.* ^b*Ankara University, Department of Chemistry, 06100 Ankara, Turkey.*

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In this study, crystal structures of two ferrocenyl-phosphazene derivatives, C₁₅H₂₀Cl₄FeN₅P₃, (I), and C₄₂H₆₂Fe₂N₉P₃, (II), are investigated. The data have been collected with Mo K _{α} radiation on an Enraf-Nonius CAD-4 diffractometer.

spiro(Butane-1,4-diamino)-[N-(1-ferrocenyl-methyl)]-4,4,6,6-tetrachloro-cyclotriphosphazatriene (I) is a mono-ferrocenyl *spiro*-cyclic phosphazene derivative and it belongs to the space group *P bca* with cell parameters $a=16.027(3)$, $b=11.834(8)$, $c=24.111(4)$ Å. It has π - π contact between cyclopentadiene rings [centroid-centroid distance = 3.278(4) Å]. The N-H...N intermolecular hydrogen bonds^[1] link the molecules, forming infinite one dimensional chains running approximately parallel to *c* axis and the intramolecular C-H...N H bonds form a dimerization.

spiro-(Butane-1,4-diamino)-[N,N'-bis(1-ferrocenyl-methyl)]-4,4,6,6-tetrakispyrrolidino-cyclotrip

hosphazatriene (II) is a bis-ferrocenyl *spiro*-cyclic phosphazene derivative including two ferrocenes and it belongs to the space group *P-1* with cell parameters $a=11.494(4)$, $b=15.259(3)$, $c=15.293(3)$ Å and $\alpha=60.98(1)^\circ$, $\beta=75.49(2)^\circ$, $\gamma=71.33(2)^\circ$. It also has π - π contact between cyclopentadiene rings and C-H... π contacts between H atoms of C13, C19, C34 and cyclopentadiene rings (C6/C7/C8/C9/C10) and (C1/C2/C3/C4/C5) and phosphazene ring (P1/N1/P2/N2/P3/N3).

[1] İltter, E. E., Asmafiliz, N., Kılıç, Z., Işıklan, M., Hökelek, T., Çaylak, N. and Şahin, E., *Inorg. Chem.*, 46, 9931-9944, 2007.

Keywords: phosphazenes; ferrocenyl-phosphazenes; hydrogen bonds

FA4-MS05-P23

Solid State Chemistry of Tetrabromo-and Tetrachlorosemiquinone Radical Anions. Krešimir Molčanov^a, Biserka Kojić-Prodić^a. ^a*Rudjer Bošković Institute, Zagreb, Croatia.*

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p-Tetrabromosemiquinone (bromanil) and *p*-tetrachlorosemiquinone (chloranil) radical anions are well-known for their stability. They were extensively studied by EPR, IR and Raman spectroscopies and theoretical methods; an X-ray crystallographic study was also attempted. The crystal structures, however, remained elusive.

Freshly prepared semiquinone crystals are red, but turn green in a few minutes at RT. The colour change is due to disproportionation of the radicals into a quinhydrone-like substance [1]. All previous studies of tetrachlorosemiquinone radical anion salts were done using a green compound.

For the first time, high-resolution data on geometry of semiquinone radicals not participating in charge-transfer interactions (such as coordination bonds or low-barrier hydrogen bonds) has been experimentally obtained. Geometry of the quinoid rings and C–O bond lengths (1.25 – 1.26 Å) differs slightly from the hydrogen bonded semiquinone (C–O bond lengths of 1.29–1.30 Å [1]), and these results agree well with quantum chemical models.

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Keywords: radical salts; accurate geometry; low-temperature data collection

FA4-MS05-P24

Crystal Structure of 2,4,4-Tris(benzylsulfanyl)-1,1-dichloro-3-nitrobuta-1,3-diene. Cigdem Sayil^a, Goksin Aydinli^a, Cemil Ibis^a. ^a*Istanbul University, Faculty of Engineering, Department of Chemistry, 34320 Avcilar-Istanbul, Turkey.*

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The molecule of the title compound, C₂₅H₂₁Cl₂NO₂S₃, is not planar. The three phenyl rings and the butadiene group are inclined at angles of 85.9(1), 61.9(1), 81.4(1)

°. There are numerous publications devoted to the synthesis of halogenobutadienes containing atom of chlorine. They possess a broad spectrum of useful properties: they are employed as monomers for the preparation of valuable polymers and copolymers resistant to heat, light, chemical corrosion, etc. and they show algicidal, bactericidal fungicidal activities[1]. A number of halogenobutadienes manifest high antitumour activity. However, there are a few reports on the crystal structures of nitrobutadiene compounds[2,3]. It is the first publication about single crystal structure of 1,1,3-tris(arylthio)nitrobutadiene derivative.

The title compound, (I), was synthesized from 2-nitropentachlorobutadiene and benzyl mercaptan[4]. It is noted that, our spectroscopic data are in accordance with reported by this article but apparently, title compound is not a 1,1,4-substituted, but a 1,1,3-substituted regioisomer instead. This indication was proven by X-Ray analysis newly. Crystallographic analysis was carried out and the results are presented in this paper.

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Keyword: single-crystal structure analysis; sulfur compounds; synthetic organic chemistry

FA4-MS05-P25

NBO Analysis – A Useful Tool on Interpretation of Results of Crystal Structure Determination.

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General picture of crystal structure of compounds, especially of molecular crystals, can be appropriately complemented by means of Natural Bond Orbital (NBO) analysis, which provides a detailed view on the electronic structure of compounds. NBO analysis is based on a method for optimal transformation of a given wavefunction into a localized form, corresponding to the one-center (“lone pair”) and two-center (“bond”) elements of the Lewis structure picture [1]. NBO analysis provides information on localization of the individual Natural Atomic Orbitals (NAOs), degree of delocalization of the electronic structure, resonance structures, charge balance, bond orders (Wiberg indices) and other useful information on electronic structure of a compound. This method has been successfully applied in our works on several crystal structures of push-pull olefins [2-4], where the charge balance, possible and preferred resonance structures and Wiberg indices have been analysed. The contribution of lone pairs of respective atoms to electronic density of a bifunctional monophenol

[5], pyrazoline chromophore [6], fluoroquinolone [7] and imidazol[2, 1/b]thiazoles [8] (Fig. 1) and deviations from the expected bond orders in those compounds have been analyzed. NBO analysis revealed the impact of electronic redistribution to geometrical parameters in these molecules. It was found that the electronic delocalization caused shortening of the respective single bonds and/or elongating of some double bonds, when compared to typical bond lengths in the crystal structures of similar compounds.

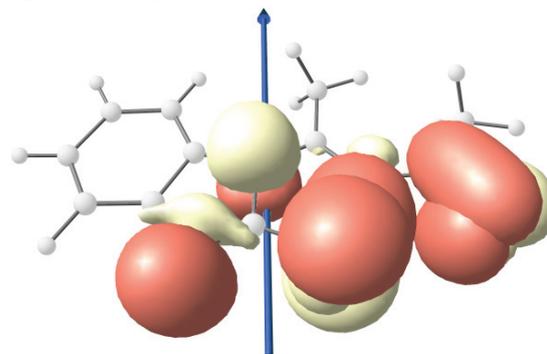


Fig. 1 The contributions of lone pairs of respective atoms to the electronic structure of 2-acetyl-3-methylbenzo[d]imidazol[2, 1/b] thiazole. The arrow represents the direction of total dipole moment.

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Keywords: DFT; electron density distribution in bonds; orbital calculations

FA4-MS05-P26

Hindered Rotation in N-(Acyloxy)-4-methylthiazole-2(3H)-thiones.

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Dynamic processes associated with a topological exchange of substituents in mixed anhydrides composed of carboxylic and cyclic thiohydroxamic acids were unexpectedly slow at ~298 K [1]. At ~320 K signal coalescence occurred for anhydrides derived from cyclic thiohydroxamic acid N-hydroxy-4-methylthiazole-2(3H)-thione and β-disubstituted carboxylic acids. At temperatures significantly above this point, fast exchange of diastereotopic substituents was evident from averaged