

Pavelić K., Karminski-Zamola G., *J. Med. Chem.*, 2005, *in press*.

Keywords: intercalators, heterocyclic compounds, DNA binding

P.06.04.6

Acta Cryst. (2005). A61, C282

Photooxygenation of Rubrene (5,6,11,12-tetraphenylnaphthalene)

Kiyoaki Shinashi^a, Isao Oonishi^b, ^a*Department of Law, Faculty of Law, Chuogakuin University, Kujike 451, Abiko, Chiba, 270-1196, Japan.* ^b*Department of Biomolecular Science, Faculty of Science, Toho University, Miyama 2-2-1, Funabashi, Chiba, 274-8510, Japan.* E-mail: kshinajp@yahoo.co.jp

It is well-known that when polycyclic aromatic hydrocarbons (PAHs) in solution are irradiated by light with their absorption wavelength, they react with molecular oxygen dissolved in solvent to form *endo*-peroxides. This photooxygenation occurs due to 1,4-cycloaddition of singlet oxygen to a PAH molecule in the ground state. When rubrene in benzene solution was irradiated by light, the color of solution gradually changed from reddish orange to colorless. The rubrene peroxide was crystallized from 1,4-dioxane solution and X-ray structure analysis was carried out. A molecular oxygen bonds to 5 and 12 carbon pair of the naphthalene skeleton. Although the naphthalene moiety of rubrene has completely planar structure due to the crystallographic symmetry, that of the peroxide is largely bent at the positions where molecular oxygen binds.

The structure optimization of PAHs including rubrene and their peroxide was performed by applying semi-empirical molecular orbital calculation methods. The optimized structure was coincident with the experimentally determined structure. The photooxygenation reactivity of PAHs including rubrene was well explained by the differences in heats of formation between PAHs and their peroxide.

Keywords: rubrene, photooxygenation, PAH

P.06.04.7

Acta Cryst. (2005). A61, C282

Deuterium Isotope Effect on the Crystal Structures of Pyrene: A Lab XRPD Study

Alastair J. Florence^a, Norman Shankland^a, Kevin S. Knight^b, Xuelian Xu^a, ^a*Department of Pharmaceutical Sciences, University of Strathclyde, Glasgow G4 0NR, UK.* ^b*ISIS Facility, Rutherford Appleton Laboratory, Oxon OX11 0QX, UK.* E-mail: alastair.florence@strath.ac.uk

Laboratory XRPD data were collected from capillary samples of perdeuterated pyrene (C₁₆D₁₀) and protonated pyrene (C₁₆H₁₀) in the range $T = 80\text{--}140\text{K}$ [1]. Unit cell volumes were determined by Pawley refinement (*ca.* 120 reflections to 2 Å resolution), yielding:

(i) $V(T) = (0.1511 \pm 0.008672)T + (1001.9 \pm 1.0)$, $r^2 = 0.9743$, for C₁₆D₁₀ (form I);

(ii) $V(T) = (0.1449 \pm 0.006194)T + (1004.8 \pm 0.7)$, $r^2 = 0.9803$, for C₁₆H₁₀ (form I).

A comparison of slopes ($P = 0.553$) and elevations ($P < 0.0001$) within *Prism v4.02* confirmed that whilst the difference between the slopes is not significant, the perdeuterated crystal structure is significantly more compact than its hydrogenous counterpart at any given T . A likewise comparison of form II C₁₆D₁₀ with form II C₁₆H₁₀ produced the same conclusion.

The quality of the XRPD analysis was assessed against a neutron powder diffraction analysis of C₁₆D₁₀ [2]. The principal differences of note are the superior precision of the best-fit parameters, plus the superior goodness of fit, obtained with the neutron analysis, e.g.:

(i) $V(T) = (0.1496 \pm 0.001047)T + (1002.0 \pm 0.2)$, $r^2 = 0.9993$, for C₁₆D₁₀ (form I; HRPD, ISIS Facility, RAL).

[1] Xu X., *Ph.D. Thesis*, University of Strathclyde, Glasgow, UK, 2005. [2] Love S.W., *Ph.D. Thesis*, University of Strathclyde, Glasgow, UK, 1997.

Keywords: pyrene, deuterium effect, powder X-ray diffraction

P.06.05.1

Acta Cryst. (2005). A61, C282

Single Crystal and the Third Order non Linear of the Adeninium Dinitrate

Karim Bouchouit^a, Noureddine Benali-cherif^b, Bouchta Sahraoui^c,

^a*Département de Chimie, Faculté des Sciences, Université de Jijel.b-Institut des Sciences Exacte.* ^b*Centre universitaire de Khenchela.* ^c*(POMA) Université d'Angers, France.* E-mail: karim.bouchouit@laposte.net

We report the measurement of the degenerate fourth-wave mixing (DFWM) of adeninium dinitrate in aqueous solutions at $\lambda = 532\text{ nm}$ in ps regime with different numbers of π -conjugated bonds. From these measurements, we evaluated the values of the second order hyperpolarizabilities γ , which are about 10^3 larger than the γ value of CS₂. The influence of π -conjugated bonds on the third-order susceptibilities and appropriate figures of merits is discussed. The more important seems to be the possibility of a simultaneous increase of the third-order susceptibilities, together with the decrease of the absorption coefficients that open a possibility of their use as promising materials for laser wavelengths mixing.

In the asymmetric unit of the title compound, C₅H₇N₅²⁺, 2NO₃, the adenine base is diprotonated and cocrystallizes with two nitrate anions. The structure is a layered one, and in each layer all H atoms bonded to O and N atoms are involved in a twodimensional hydrogen-bonding network. Short contacts are observed between parallel layers and ensure the cohesion of the crystals.

Keywords: hydrogen bonding, degenerate four wave mixing, third-order susceptibilities

P.06.05.2

Acta Cryst. (2005). A61, C282

In-situ Diffraction Study of C₆₀ Polymerization under High-pressure High-temperature

Leonel Marques^a, M Mezouar^b, J-L. Hodeau^c, M. Núñez-Regueiro^d, ^a*Departamento de Física and CICECO., Universidade de Aveiro, 3810 Aveiro, Portugal.* ^b*E.S.R.F. 38041 Grenoble, France.* ^c*Laboratoire de Crystallographie, C.N.R.S. 38041 Grenoble, France.* ^d*C.R.T.B.T., C.N.R.S. 38041 Grenoble, France.* E-mail: marques@fis.ua.pt

Polymerization in C₆₀ has been achieved using photochemical and high-pressure high-temperature (HP-HT) methods [1]. Both polymers have the same 2+2 cycloaddition polymeric bond but their crystal structures are very different. HP-HT polymers are ordered while photopolymers have a disordered fcc structure reflecting a random polymerization [2]. In order to understand the origin of bond order in HP-HT polymers and its absence in photopolymers we have performed in situ diffraction studies at the E.S.R.F. [3]. We have found that the applied anisotropic stress selects the bonding direction, which is crucial to obtain ordered polymeric structures since it avoids geometrical frustration inherent to 2+2 cycloaddition bonds in the fcc monomer lattice. The photochemical method having no symmetry-breaking field leads to a frustrated disordered fcc polymer. The highly anisotropic distribution of polymeric bonds in HP-HT polymers should be at the origin of the elastic anisotropy in the C₆₀-derived amorphous samples and the magnetic anisotropy in 2D-ordered polymerized C₆₀.

[1] Núñez-Regueiro M., Marques L., Hodeau J.L., Béthoux O., Perroux M., *Phys.Rev.Lett.*, 1995, **74**, 278. [2] Putzai T., Ozlányi G., Kamarás K., Gránásy L., Pekker S., *Sol. St. Comm.*, 1999, **111**, 595. [3] Marques L., Mezouar M., Hodeau J.-L., M.Núñez-Regueiro, *Phys.Rev.B*, 2003, **68**, 193408.

Keywords: fullerenes, high-pressure X-ray diffraction, polymerization

P.06.06.1

Acta Cryst. (2005). A61, C282-C283

Ortho-halogeno Piedforts

Nikoletta Báthori^{a,b}, László Bihátsi^b, Petra Bombicz^a, Mátyás Czugler^a, ^a*Institute of Structural Chemistry, Chemical Research Center, Hungarian Academy of Sciences, P.O.Box 17. H-1525, Budapest, Hungary.* ^b*Department of Inorganic Chemistry, Budapest University of Technology and Economics, H-1521, Budapest, Hungary.* E-mail: nikol@chemres.hu

In an effort to explore Piedfort pairing in a systematic way four 2,4,6-tris(ortho-halogeno-phenoxy)-[1,3,5]-triazine host molecules