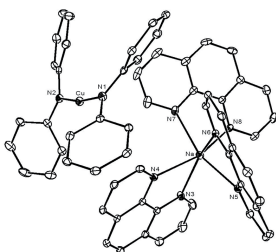


can work as an effective catalyst to carry out the C-N coupling under the same reaction conditions indicating that **2** is the intermediate of the catalytic system. Both  $[\text{Cu}(\text{NPh}_2)_2]^-$  and  $[\text{Cu}(\text{NPh}_2)\text{I}]^-$  were observed by *in situ* electrospray ionization mass spectrometry (ESI-MS) analysis under the catalytic reaction condition indicating that they are intermediates in the reaction. A catalytic cycle was proposed based on these observations. Molecular structure of **2** was determined by single-crystal X-ray diffraction analysis.



**Keywords:** C-N coupling, copper, ESI-MS

## MS52.P01

*Acta Cryst.* (2011) **A67**, C557

### Effect of thermal treatment on characteristics nanodiamonds and diamond blend

Svetlana N. Ivashevskaya,<sup>a</sup> Vasily A. Lebedev,<sup>b</sup> Gennady P. Kopitsa,<sup>c</sup> <sup>a</sup>*Institute of Geology Karelian Research Centre RAS, Petrozavodsk (Russia)*, <sup>b</sup>*Department of Materials Science, Moscow State University, Moscow (Russia)*, <sup>c</sup>*Petersburg Nuclear Physics Institute RAS, 188300 Gatchina (Russia)*. E-mail: ivashevskaja@yahoo.com

Diamond nanoparticles are now the most commonly synthesized nanomaterials. As they have some distinctive properties, such as high adsorptive capacity, high thermal conductivity, high surface energy, hydrophobicity and a large specific surface, they are of great interest as materials with a wide application range. Nanodiamonds (NDs) can be used as adsorbents and catalysts for the production of nanocomposite materials, for growing diamond films, in medicine and biotechnology [1-3]. They can not now be widely used mainly because there are no well-defined standards and the quality of the NDs, offered by different producers, is unstable [4]. Therefore, the study of NDs is important and promising.

The main goal of NDs purification is removal of non-diamond forms of carbon. Practically all known methods are based on the use of the different resistances of diamond and non-diamond forms of carbon to oxidants. To apply them, many technical problems, arising in exothermal reactions at high temperatures and intensive gassing in a limited volume, should be solved [5]. A decisive factor in selecting a purification variant is safety.

The goal of the project was to assess the effect of thermal treatment in vacuum on the structural characteristics and chemical composition of NDs and a diamond blend produced by detonation synthesis. NDs in the form of powders were obtained from two sources: Gansu Liru Lingyun NanoMaterial Co Ltd. (China) and NanoCarbon Research Institute Ltd. (Japan). A diamond blend (brand AIII-A) was offered by Scientific and Technical Enterprise "Sinta" (Belarus).

It is commonly accepted that only a complex of diagnostic techniques gives the possibility to get a true knowledge about the carbon nanostructures properties. Chemical composition was determined by mass-spectrometry (an inductively-coupled plasma mass-spectrometer ICP-MS Xseries2 (ThermoFisher Scientific)) and X-ray spectral analysis (a VEGA II LSH electron microscope (TESCAN OJSC) with an attachment for microprobe analysis); different levels of structural elements aggregation were analysed by SEM (VEGA II LSH), AFM (N'Tegra Aura NT-MDT) and DLS (Malvern Instruments Ltd.) methods; structural parameters of crystallites were analysed by X-ray structural analysis (Thermo Scientific ARL X'TRA Powder X-ray Diffraction System); in order to determine the pair density-density

correlation functions and to estimate parameters of the nanostructure (correlation length, specific surface and fractal dimension) was used SANS.

Our study has shown that thermal treatment under mild conditions is a promising approach to purification of NDs and a diamond blend from the point of view of economy, safety and environmental protection.

*The project was supported by ESD RAS-5.*

[1] O. Shenderova, V.V. Zhirnov, D.W. Brenner, *Crit. Rev. Solid State Mater.*, **2002**, *27*, 227 [2] A.M. Schrand, H. Huang, C. Carlson, J.J.Schlager, *Phys. Chem. B.* **2007**, *111*, 2. [3] V.J. Dolmatov, L.N. Kostrova, *Superhard materials* **2000**, *3*, 82. [4] V. Teslenko <http://rough-polished.com/ru/analytics/31816.html> **2009**. [5] V.G. Sushchev, V.J. Dolmatov, V.A. Marchukov, M.V.Veretennikova [www.rql.kiev.ua/almaz\\_j](http://www.rql.kiev.ua/almaz_j), **2008**.

**Keywords:** carbon, nanotechnology, diamond

## MS52.P02

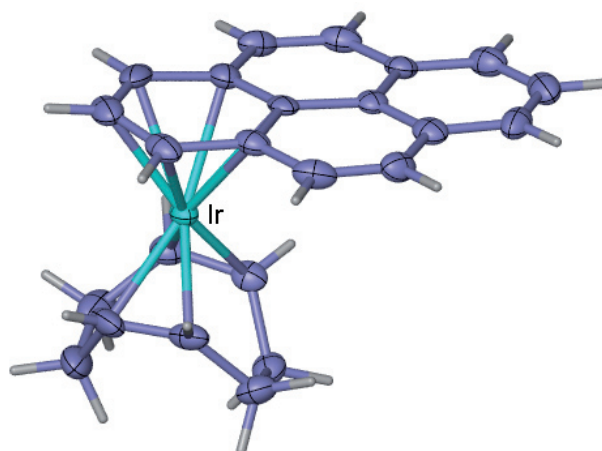
*Acta Cryst.* (2011) **A67**, C557-C558

### Synthesis of nanographene organometallics for deposition on a graphene surface

Andrew Parlett,<sup>a</sup> Paul Raithby,<sup>a</sup> Adelina Ilie,<sup>b</sup> Gavin Jones,<sup>b</sup> Sara Fuentes,<sup>a</sup> Simon Bending,<sup>b</sup> <sup>a</sup>*Department of Chemistry*, <sup>b</sup>*Department of Physics, University of Bath (UK)*. Email: ap292@bath.ac.uk

Graphene's unique electrical and mechanical properties give it a wide variety of possible applications in the future of electronic devices [1]. Being essentially an extended network of interconnected arene rings, it is also of much interest in chemistry. It can be modified chemically via oxidation, (partial) hydrogenation etc to alter the band gap, or else used as a surface on which to adsorb small molecules which can be visualised using AFM and STM microscopy. Such small molecule adsorption can be used for reactions directed by the graphene surface or for functionalising the graphene [2].

We have successfully synthesised a range of organometallic compounds with coordinated polycyclic aromatic hydrocarbons (or nanographenes) such as pyrene which are either novel or found as a new polymorph. These compounds have been characterised using a variety of spectroscopic methods, as well as X-ray crystallography. These compounds will be deposited on to graphite or graphene and imaged using AFM and STM microscopy. This will allow us to optimise the surface coverage of such molecules and analyse their interactions with the surface and each other. By 'sticking' molecules such as these to graphene we hope to be able to alter the electrical, magnetic and optical properties of graphene.



## Poster Sessions

[1] A.K. Geim, **2009**. *Science* 324(5934), 1530-1534. [2] H.L. Lee, *et al.* **2011**. *J. Am. Chem. Soc.* 133, 4447-4454.

**Keywords:** graphite, grganometallic

### MS52.P03

*Acta Cryst.* (2011) **A67**, C558

#### XRD characterization of bulk graphene-based material

Gianfranco Carotenuto,<sup>a</sup> Davide Altamura,<sup>b</sup> Cinzia Giannini,<sup>b</sup> Dritan Siliqi,<sup>b</sup> <sup>a</sup>*Institute of Composite and Biomedical Materials- CNR, Piazzale Tecchio, 80125 Napoli (Italy)*. <sup>b</sup>*Institute of Crystallography - CNR, Via G. Amendola 122/0, 70126 Bari (Italy)*. E-mail: giancaro@unina.it

Graphene is a nanostructure with unique physical properties that can be used to prepare a number of novel functional materials [1]. Bulk-quantities of graphene can be produced by exfoliation of expanded graphite with ultrasounds [2]. The expanded graphite is a commercial material which is obtained by thermal reduction of graphite oxide at temperatures close to 1000°C (graphite oxide is usually prepared by chemical oxidation of natural graphite using a mixture of strong chemical oxidants like: nitric acid, sulfuric acid and potassium permanganate). The degree of exfoliation strictly depends on the graphite oxidation level and the thermal shock treatment undergone by the graphite oxide; such a parameter is of a fundamental importance for the resulting physical properties of the nanostructured material that are prepared by intercalation of graphene with polymers and/or other types of nanostructures (e.g., CNTs, fullerenes, ceramic or metal nanoparticles, etc.).

The graphite oxidation/reduction process can be accurately investigated by wide-angle X-ray powder diffraction (XRD) [3] looking at the shift of the (002) peak in the diffraction pattern [4]. The presence of defects in the graphite structure like oxygen-groups (-OH, -COOH, etc.) and/or intercalated molecules (e.g., H<sub>2</sub>SO<sub>4</sub>) has the effect to modify the interlayer spacing, thus shifting the position of the peaks.

[1] A.K. Geim, K.S. Novoselov, *Nature Materials* **2007**, 6, 183-191. [2] A.P. Yaroshenko, M.V. Savos'kin, A.N. Magazinskii, V.I. Shologon, R.D. Mysyk, *Russ. J. Appl. Chem.* **2002**, 75(6), 861-865. [3] *Quantum Dots: Synthesis and Characterization*, D. Dorfs, R. Krahne, C. Giannini, A. Falqui, D. Zanchet & L. Manna, *Book Title: Comprehensive Nanoscience and Technology (vol. 2)*, Editors: Guozhong Cao, Duncan Gregory, Thomas Nann, Edited by: Elsevier, **2011**, 219-260. [4] Y. Wu, B. Wang, Y. Ma, Y. Huang, N. Li, F. Zhang, Y. Chen, *Nano Res.* **2010**, 3(9), 661-669.

**Keywords:** graphene, graphite oxide, XRD

### MS53.P01

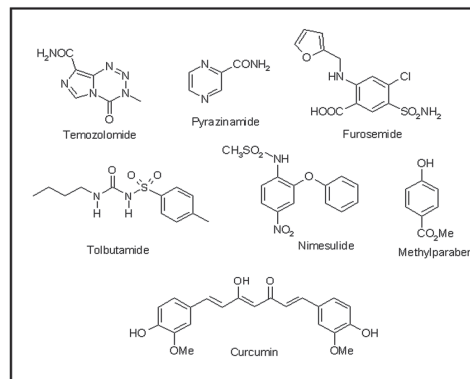
*Acta Cryst.* (2011) **A67**, C558

#### Polymorphs of some common drugs and bioactive agents

Ashwini Nangia, *School of Chemistry, University of Hyderabad, Hyderabad 500 046 (India)*. E-mail: ashwini.nangia@gmail.com

Novel polymorphs of important drugs such as Temozolomide, Pyrazinamide, Furosemide, Tolbutamide and Nimesulide, bioactive agents Methylparaben and Curcumin, and some model sulfonamides and hydroxybenzoic acids will be presented. We have found that screening against a large number of crystallization methods such as solvent-antisolvent, temperature variation and ramping, cofomers

and additives, solventless melt and sublimation techniques, and ionic liquids afforded novel crystalline forms of materials. Success seems to be more a factor of McCrone's famous dictum and the approach is still quite heuristic in terms of which methods work best for what kind of molecule. The success of our methodology will be presented through case studies involving different types of molecules taken up for polymorph search in our group.



[1] N. J. Babu, S. Cherukuvada, R. Thakuria, A. Nangia, *Cryst. Growth Des.* **2010**, 10 1979-1989. [2] N.K. Nath, A. Nangia, *CrystEngComm, CrystEngComm*, **2011**, 13, 47-51. [3] P. Sanphui, B. Sarma, A. Nangia, *J. Pharm. Sci.*, **2011**, DOI: 10.1002/jps.22464. [4] P. Sanphui, N.R. Goud, U.B.R. Khandavilli, S. Bhanoth, A. Nangia, *Chem. Commun.*, **2011**, DOI:10.1039/C1CC10204D.

**Keywords:** crystallization, pharmaceutical, polymorphism

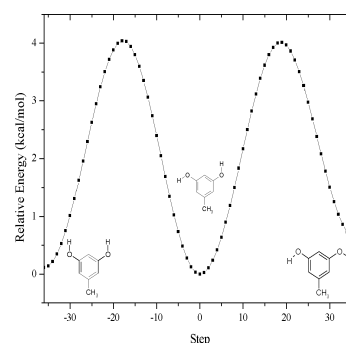
### MS53.P02

*Acta Cryst.* (2011) **A67**, C558-C559

#### High throughput crystallization of orcinol with various N-acceptor cofomers: An alternative approach for exploring the structural landscape

Tejender S. Thakur, Pawel Grobelny, Arijit Mukherjee, Gautam R. Desiraju, *Solid State & Structural Chemistry Unit, Indian Institute of Science, Bangalore 560 012, (India)*. E-mail: tejender@sscu.iisc.ernet.in

An exhaustive search of the structural landscape of orcinol, 5-methyl-1,3-dihydroxybenzene, was carried out with high throughput crystallography. Polymorphs, pseudopolymorphs (solvates) and co-crystals are described. Several packing modes were identified for the orcinol co-crystals with various N-bases. In these several structural variations, the OH group conformations in the orcinol molecule (Figure 1) were found to depend on the choice of co-formers and the crystallization conditions employed. The study provides an alternative and more efficient approach to look into the various possibilities available for co-crystal formation.



**Figure 1** Relaxed potential energy surface scan performed for the OH group rotations in the orcinol molecule.