

P16.04.16*Acta Cryst.* (2008). A64, C585**A novel approach to specifically crystallize anhydrous compounds: Crystal structure of adenine**

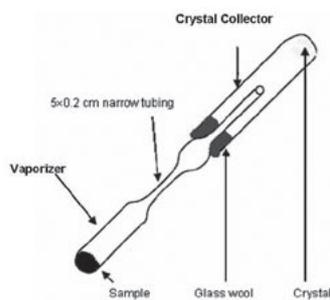
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Crystallization without solvent incorporation is a key requirement to study anhydrous forms of compounds. The structures of all the anhydrous nucleobases were not available as adenine was very hard to crystallize without solvent incorporation. X-ray powder diffraction data both from conventional source as well as synchrotron radiation was found inadequate to solve the structure in the anhydrous form. Crystals of anhydrous adenine were grown by a new approach using the principle of sublimation and gradient cooling. Anhydrous adenine crystallizes in a monoclinic space group $P2_1/c$ with $a=7.891(3)$, $b=22.242(8)$, $c=7.448(3)\text{\AA}$, $\beta=113.193(5)^\circ$, $V=1201.6(3)\text{\AA}^3$ and $Z=8$.

There are two molecules in the asymmetric unit linked via N-H \cdots N hydrogen bonds. The crystal structure is stabilized by two additional sets of N-H \cdots N hydrogen bonds, one across the center of symmetry connecting the imidazole moiety to the pyridine moiety and the other linking the next asymmetric unit resulting in a sheet motif.

Keywords: crystallization, X-ray powder diffraction, synchrotron radiation

**P16.13.17***Acta Cryst.* (2008). A64, C585**Polymorphism and crystal structure of BPT propyl ester in various solvents**Mitsutaka Kitamura¹, Takayuki Hara^{1,2}, Kousuke Adachi¹, Midori Takimoto-Kamimura²

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BPT propyl ester (Propyl 2-(3-Cyano-4-(2-methylpropoxy)-phenyl)-4-methyl-thiazole-5-carboxylate) (Pr-est(CN) and propyl 2-(4-(2-methylpropoxy)-phenyl)-4-methyl-thiazole-5-carboxylate) (Pr-est(H)) were synthesized, and solvent effect in the polymorphic crystallization was investigated by rapid cooling crystallization method. The crystallization for Pr-est(CN) was carried out from ethanol (EtOH), acetonitrile (MeCN), and cyclohexane (c-Hxn) solutions. From EtOH and c-Hxn solutions at high initial concentrations, meta-stable form first appeared, and after then, the transformation to a stable form occurred. On the other hand, at low concentrations the stable form crystallized directly. The crystallization from MeCN solutions resulted in only a stable form, indicating the solvent effect on the polymorphism. X-ray analysis indicated that both of the stable and meta-stable forms are constructed by stacking the sheet structures of the molecules. In the meta-stable form, no hydrogen bonding is observed, whereas the stable form

is stabilized by the hydrogen bonding through nitrile and carbonyl group. Polymorphic crystallization behavior was also examined for Pr-est(H) in MeOH, EtOH and MeCN solutions. In this system only one form crystallized in each solvents, i.e. no solvent effect appeared. The crystal is constructed by stacking the zigzag sheets of the molecules, and the hydrogen bonding between carbonyl group and hydrogen atom in phenyl ring is observed. These results indicate that for the formation of polymorphs the nitrile group in Pr-est(CN) is very important, because it contributes to the formation of distinctive hydrogen bonding and the thermodynamic stability of the solid phase.

Keywords: crystallization, polymorphic structure, pharmaceuticals

P16.13.18*Acta Cryst.* (2008). A64, C585**Photoinduced phase transition between polymorphic crystals of a photochromic diarylethene**Seiya Kobatake^{1,2}, Itsuka Yamashita¹

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Diarylethenes undergo thermally stable photochromic reactions even in the crystalline phases. 1,2-Bis(2-methyl-6-nitro-1-benzothiophen-3-yl)perfluorocyclopentene (1a) has two kinds of polymorphic crystals, which are named as 1a-alpha and 1a-beta. Both of crystals change color upon irradiation with ultraviolet light. Single crystals of 1a-alpha are obtained by recrystallization from hexane. When the alpha-crystals were heated over 180 degree C, the crystals changed to beta-crystals by a phase transition. The difference of the crystal structures of 1a-alpha and beta was confirmed by X-ray crystallographic analysis. At 170 degree C, alpha-crystals do not change to the crystal structures of 1a-beta. However, upon irradiation with ultraviolet light, the alpha-crystals took place the phase transition to form beta-crystals even at 170 degree C. It is due to the decrease in the phase transition temperature because of the presence of a few ten mol% of the closed-ring isomers. The beta-crystal has a needle-like crystal form. The contact angle to water in the beta-crystals was larger than that of alpha-crystals. The thin film of polystyrenes doped 1a was prepared. The contact angle changed from 134 degree to 154 degree by the phase transition to form the beta-crystals even in the film.

Keywords: photochromism, polymorphism, phase transition

P16.14.19*Acta Cryst.* (2008). A64, C585-586**In situ observation of CH₄ hydrate growth by neutron powder diffraction**Akinori Hoshikawa¹, Hiroshi Fukazawa², Naoki Igawa², Hiroki Yamauchi², Yoshinobu Ishi³, Toru Ishigaki¹

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