ESRF	**Experiment title:** Crystal Structure of Sodium ADP from Powder XRD Data	**Experiment number:** CH1205
Beamline: BM16	**Date of experiment:** from: 02-09-01 to: 04-09-01	**Date of report:** 27-02-02
Shifts: 6	**Local contact(s):** Dr. M. Brunelli	*Received at ESRF:*

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Report:

The title compound did not yield a powder diffraction pattern suitable for crystal structure solution. However, powder diffraction patterns suitable for crystal structure solution, for three biologically important compounds, with previously unknown crystal structures, were collected. The crystal structures of all three have been successfully determined using the following steps:

1. Indexing using the Crysfire suite.[1]
2. Crystal structure global optimisation using a simulated annealing technique,[2] implemented in the program DASH.[3]
3. Restrained Rietveld refinement using GSAS.[4]

Tryptamine Free Base

The previously unknown crystal structure of the biogenic compound tryptamine, in the form of a free base ($C_{10}H_{12}N_2$), Fig. 1, has been solved using simulated annealing followed by restrained Rietveld refinement (space group $P2_12_12_1$, a = 12.28594(6)Å, b = 8.53351(4)Å, c = 8.49385(4)Å, Z = 4, final reduced-χ^2 = 5.236).[5] The crystal structure reported here shows efficient packing involving weak intermolecular hydrogen bonding and distinctive herring bone type packing patterns.

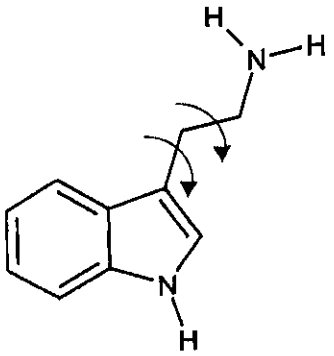

Fig. 1 Atomic connectivity in the tryptamine molecule. The arrows indicate torsion angles that were allowed to vary during simulated annealing. Hydrogen atoms connected to carbon atoms are omitted for clarity.

Vitamin K_3

Vitamin K_3, menadione, $C_{11}H_8O_2$, Fig. 2, has been solved using simulated annealing and restrained Rietveld refinement. The crystal structure was solved in a monoclinic cell, a = 11.128(1), b = 20.663(2), c = 7.4476(5)Å and $\beta = 97.983(9)°$, space group $P2_1/a$, Z = 4, $\chi^2 = 11$. The structure consists of stacked chains of molecules lying head-to-tail shown in Fig. 3.

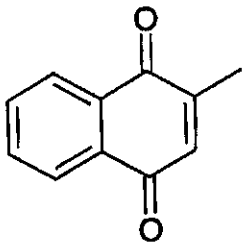

Fig. 2 Atomic connectivity in the vitamin K_3 molecule.

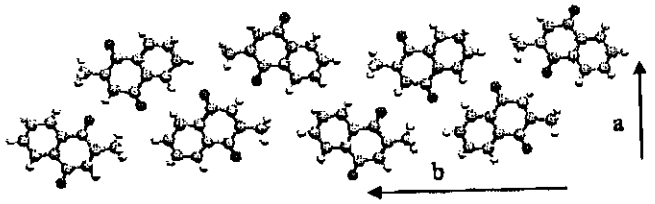

Fig. 3 Chains of vitamin K_3 molecules.

Bispyralozone

Bispyralozone, $C_{20}H_{18}N_4O_2$, Fig. 4, is a reagent used for the determination of cyanide.[6] The crystal structure was solved using an orthorhombic cell, a = 21.145(6), b = 18.839(3) and c = 8.742(1)Å, space group Pbca, Z = 4. The molecule has two chiral centres and simulated annealing runs in which the molecules were assigned one S configuration and one R configuration consistently ended with lower profile χ^2 values. Initial refinements indicate efficient packing and a good profile fit with $\chi^2 \sim 3.5$.

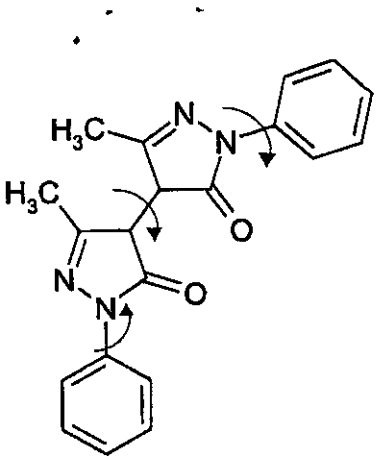

Fig. 4 Atomic connectivity in the bispyralozone molecule, the arrows represent the torsion angles that were allowed to vary during simulated annealing.

References

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2. David, W. I. F., Shankland, K. & Shankland, N. (1998). *Chem. Commun.*, **8**, 931-932.
3. Cambridge Crystallographic Data Centre (2001). DASH – a program for structure solution from powder diffraction data, CCDC, 12 Union Road, Cambridge, UK.
4. Larson, A. C. & Von Dreele, R. B. (1994). "General Structure Analysis System (GSAS)", Los Alamos National Laboratory Report LAUR 86-748.
5. Nowell, H., Attfield, J. P. & Cole, J. C., in preparation.
6. Epstein, J. (1947) *Analytical Chemistry*, **19**, 272-274.