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Report:

Background

Physically estimated triplet phases (PETP) from 3-beam diffraction experiments appear uniquely suited to identify the correct or "best" phase model among several possible and closely related ones. The experimental work involves measurement of a selected set of triplets of which the model sensitive ones are used for model discrimination. A model sensitive triplet contains at least one structure-factor phase that is significantly influenced by the change in structure.

This novel application of PETP's is being explored in a study of the complex a-D-glucose NaCl . H_2O (2: 1: 1) in space group $P3_1$ with $V = 4.180$ Å^3. A structure model of this complex has been published (Model CA) [1]. However, there exists at least one alternate solution (Model GE) [2]. We have refined both models and identified the differences in structure. Introducing in Model CA the primary difference, which involves an interchange of the Cl and water 0 positions, this model can be refined into Model GE in about 100 refinement cycles. Conversely, interchanging the Cl and water 0 positions in model GE, it can be refined into coincidence with the CA model. There is a peculiar distribution of the phase differences between the two models which makes model sensitive triplets with a triplet phase difference $\Delta\Phi_3 > 45^o$ and with 'amplitudes suitable for phase measurements extremely scarce. These triplets contain only one structure-factor phase that is sensitive to the model, the two others are not. In the great majority of cases where model-sensitive single phases are involved they occur in pairs, each with an intermodel $|\Delta\phi| \sim 180^o$, thus rendering $\Delta\Phi_3 \sim 0$.

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Three-beam intensity profiles were collected for three different crystals, #9, #l0 and #0. Exposure to X-radiation initiates a chemical reaction that eventually induces a rapidly increasing mosaicity, making the crystals unsuited for measurement. Because of this, crystals have to be tested and oriented immediately before the actual measurements. A crystal was replaced when its mosaicity was in a state of rapid increase and there were signs of changes in cell parameters. This usually takes place in about 1.5 days. For any given triplet -H, K, H-K measured, a measurement was also made of the Friedel-related triplet, H, -K, -H+K. From 5 to 30 scans were accumulated for each profile to obtain satisfactory counting statistics. The number of triplet pairs measured for each of the three crystals were: 17 (#9), 15 (#10), and 11 pairs (#0), respectively. The data have been analysed, and triplet phases have been assigned and compared with the values from previous measurements and with those calculated for each structure model. Phases were assigned with a precision of $\pi/8\,(22{,}5^{o})$. For a quantitative comparison with the two models, CA and GE, we have calculated:

$\langle \Delta\Phi_3^2 \rangle^{1/2}_{MM} = [1/n \sum^{n} (\Phi_{3,exp} - \Phi_{3,MM})^2]^{1/2}$, the rms difference between the set of assigned triplet phases in a series of measurements and the corresponding values calculated for model MM

$(\Delta\Phi_3)_{MM,95\%}$ = 95% confidence interval for the difference $(\Phi_{3,exp} - \Phi_{3,MM})$

Results:

Crystal		$\langle \Delta\Phi_3^2 \rangle^{1/2}_{MM}$(deg.)	$(\Delta\Phi_3)_{MM,95\%}$(deg.)
	Model CA	109.4	(82.1 , 164.0)
9	Model GE	20.7	(15.5 , 31.0)
	Model CA	25.7	(19.0 , 39.8)
10	Model GE	72.9	(53.8 , 112.8)
	Model CA	97.6	(69.1 , 165.6)
0	Model GE	23.7	(16.8 , 40.2)

The results show that crystals #0 and #9 are in agreement with Model GE, crystal #10 confirms Model CA. Similar results have been obtained in all previous experiments. At the present stage, one concludes that the set of crystals defines two distinct groups, indicating that both structure models are present, but do not coexist in the same crystal specimen. Further work is required to resolve this interesting case of structural ambiguity,

On the 08.06. at 06 hrs. we had computer problems that were not solved until 1530 hrs. At about 18 hrs. a vacuum failure caused by a thunderstorm and lightning in the area, eventually led to arcing and fatal damage of a high-voltage piezoelectric controller unit in the monochromator vessel. This situation could have been avoided with a safety-circuit in function. This has been installed later. As it were, no more measurements could be made for the rest of our experiment period. In total we lost about 9 shifts of beamtime due to computer failure and the breakdown of the controller unit.

[1] Ferguson, G., Kaitner, B., Connett, B.E. & Rendle, D.F. (1991) *Acta Cryst.* **B47**, 479-484.
[2] Fröhlich, R. Personal communication.