What contribution does neutron single-crystal diffraction make to chemical crystallography? KOALA, COBRA and LAUEG yielded structures published for maximum impact for chemistry and crystallography

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From inception, KOALA was intended to be used for chemical crystallography in addition to the well established range of physics-based applications then typical at neutron facilities. An open consultation across the potential user community had revealed significant demand for a single-crystal instrument suitable for chemical crystallography applications and the advantages of a Laue image plate system were apparent with an instrument based on VIVALDI at the ILLwas commissioned. The chemistry community has provided significant demand for instrument time across a broad range of studies from the occasional (once every 3-5 year) “unprecedented” one-off structure for which neutrons are the key through to systematic studies with variations of counter-ion, temperature or pressure or indeed combinations of these parameters. Deep insights into chemistry have been obtained across the full range of studies undertaken.

Critical to the success of KOALA in the chemical crystallography realm have been the implementation of a COBRA™ cryostream to facilitate data accumulation from air, moisture and temperature sensitive compounds, and the development of the data reduction interface LAUEG [1-2]. These improvements have made feasible the completion of optimised neutron single-crystal diffraction studies in a timeframe which sees them available for publication when the chemistry which has prompted the structure determination is published. It is notable that KOALA has produced a high proportion of the neutron single-crystal studies reported in the Cambridge data base across its years of operation (with many more to come). The impact factor of the journals in which the chemical crystallography papers from KOALA have appeared has significantly exceeded the initial ambition for the instrument and today we regularly submit manuscripts reporting structures from KOALA to Tier One general science journals such as Nature and Science where they are well received with papers appearing in Nature, Nature Chemistry and Nature Communications with many of the remaining papers appearing in premiere journals, notably J.A.C.S. and Angewandte Chemie.

It is important to understand that there is a significant range in the crystallographic “quality” of the structures which can be derived with the optimal exploitation of the data available for any given crystal being critical to the contribution which the neutron structure determination can make to the overall scientific findings. The “routine” approach to structure determination is often inadequate to the scientific task and a more nuanced approach to extracting valid scientific content from the hard won data will yield important chemical insights. On occasion, low resolution data only are obtainable but in many instances these data are precisely those that speak to the question at issue.

At the time this abstract is being written, installation of the replacement instrument for KOALA is in progress. We have optimised aspects of the instrument which could be improved and importantly, the successful approach of standardization of components applied to all of the other instruments at ACNS is being implemented for the replacement.

This talk will survey published results from KOALA (highlighting in particular the non-standard methodologies which have yielded our greatest successes) and draw comparisons with other instruments, setting out the successes, opportunities and challenges we have in exploiting the facilities available to us at the major central science facilities. The successes in chemical crystallography at this neutron facility have been underpinned by the fundamental crystallographic education of the instrument scientists. How to transmit this knowledge to potential successors and capitalise on the existing learning is an important aspect of the manner of operation of our instrumentation.