

# Single-Crystal X-ray Crystallography Measurements of Pyrophoric Materials

## Motivation

Air-sensitive and otherwise unstable or short-lived molecular entities are a day-to-day reality in many fields of synthetic chemistry.

Pyrophoric materials can combine this inherent instability with an element of risk. The challenges that must be overcome to analyse these materials ensure that any result is remarkable and may provide insights into the unstable nature of the material.

## Analysis of Pyrophoric Crystals

To prevent the spontaneous ignition of a pyrophoric material the crystals themselves must be handled with care and in a safe environment. Minimising contact with air can be achieved by extracting the crystals in an inert atmosphere using standard Schlenk-line techniques.

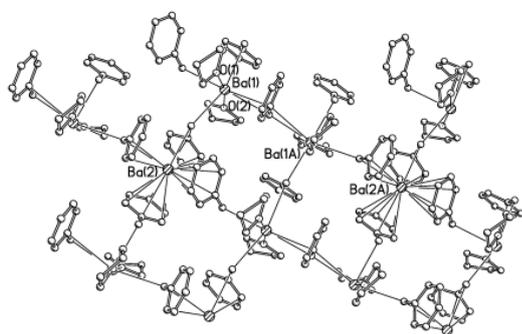


Figure 1: The crystal structure of benzyl barium, an extremely pyrophoric organometallic salt

The use of an appropriate perfluorinated oil to coat the sample provides a barrier to the air while the crystals are examined and mounted on a loop under an optical microscope. Once mounted on the diffractometer a cooling device ensures that the sample is in an inert atmosphere throughout the data collection and the low collection temperature (typically 150 K) further reduces the risk of the sample igniting.

## Benefits of Single-Crystal X-ray Crystallography

As the sample submitted will ideally comprise large single crystals with a relatively small surface area, single-crystal X-ray crystallography is one of the safest ways to analyse the solid state properties of materials of this kind. The added protection afforded by the use of Schlenk-line techniques, perfluorinated oils and the diffractometer-mounted cooling device ensure that the sample is unlikely to degrade during handling or data collection.

## References

K. Izod, P. G. Waddell, *Organometallics* (2015), **34(12)**, 2726