

# A Beryllium Dome Specimen Holder for XRD Analysis of Air Sensitive Materials

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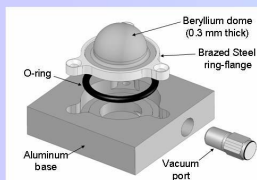
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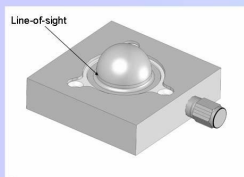
## INTRODUCTION

- We needed a way to analyze materials that were:
  - highly hygroscopic (e.g. rare-earth halides)
  - quick oxidizers (e.g. Er, Li metal)
- Our Wish List
  - easy to assemble inside glove-box
  - maintain inert conditions of glove-box
  - minimum of XRD artifacts from holder
  - excellent seal (below  $10^{-8}$  torr)
  - minimize sample displacement issues

## DESIGN

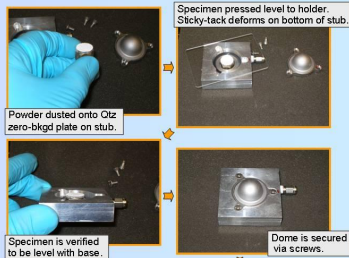


Our design employs a Be-dome brazed to a stainless-steel flange. A compression O-ring seals the system to the Al base via 3 bolts. A vacuum port is available or can be sealed off.

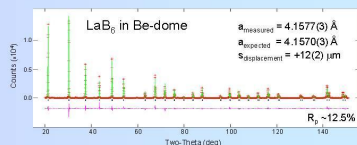
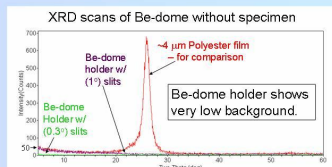


The Be-dome flange sits below the line-of-sight of the Al base. This prevents beam blockage at low angles so the X-Ray beam sees the specimen unhindered. The Al base defines the zero plane.

## SPECIMEN PREP

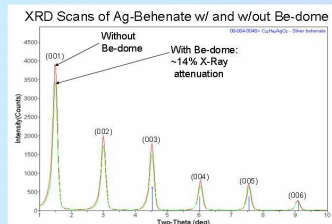


## EVALUATION



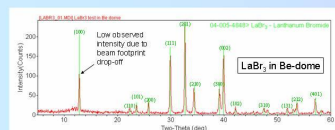
LaBr<sub>3</sub> (SRM 660) powder run in Be-dome shows good fit to predicted structure. There are essentially no observed specimen holder artifacts out to 150° 2θ.

## EVALUATION

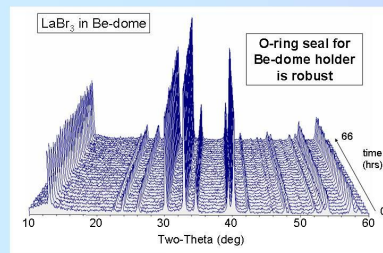


Use of 0.3° slits reduces scatter sufficiently to observe low angle peaks down to 1° 2θ. There is ~14% beam attenuation from the Be-dome.

## RESULTS: LaBr<sub>3</sub>

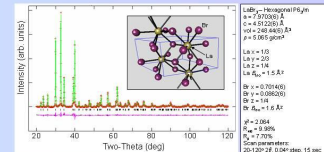


20 minute scan shows good signal and easy phase ID.

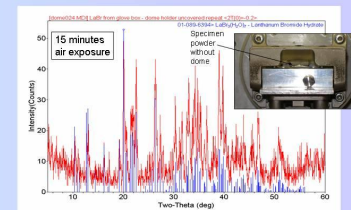


No significant change in the XRD pattern during 66 hour scan series indicates a leak-tight seal, preserving the inert atmosphere within the Be-dome specimen holder.

## RESULTS: refinement



Rietveld refinement via GSAS is straight-forward, yielding precise refined parameters.



After only 15 minutes of air exposure, LaBr<sub>3</sub> powder has completely decomposed to LaBr<sub>3</sub> · 7(H<sub>2</sub>O)

## SUMMARY

- A Be-dome specimen holder has been designed and fabricated for use with highly reactive materials that require assembly in a glove-box.
- The specimen holder has a robust seal and no artifacts in the 1-150° 2θ range. The design also minimizes specimen displacement errors.
- Rietveld structure refinement of XRD scans is straight-forward, yielding quality results.

## Acknowledgments

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