In situ ambient-pressure synthesis of non-stoichiometric Ag_3O: Phase abundance, unit-cell parameters, and *c*/*a* as a function of temperature

Paul J. Schields[1],

SSCI, a Division of Albany Molecular Research Inc., West Lafayette, Indiana 47906

Nicholas Dunwoody,

Tetraphase Pharmaceuticals, Inc., Watertown, Massachusetts 02472

and David Field

324 13 Ave NW, Calgary, AB, Canada T2M 0E8

The crystal structure of Ag_3O is comprised of a two-layer hexagonal close packing of Ag with oxygen occupying two thirds of the octahedral interstices of every other adjacent closest-packed layer (Figure 1.)

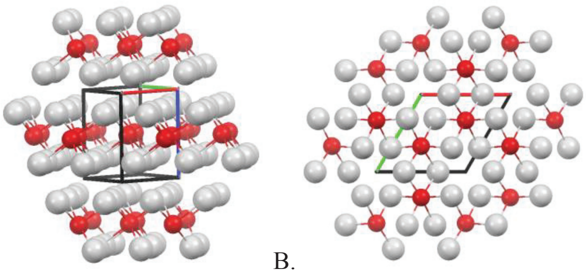

Figure 1. The structure of Ag_3O determined by Beesk et al. (1981); grey balls are Ag and red balls are oxygen; *a* axis is red, *b* axis is green, and vertical *c* axis is blue. A.) perspective view. B.) *c* axis projection.

Ag_3O was produced by heating jet-milled films composed of a mixture of Ag_2O and Ag at ambient pressure. The thermal reaction was analyzed *in situ* with XRPD from -140 to 150°C. Ag_3O was also produced by ball milling and sonication of the jet-milled film at ambient temperature.

Ag_3O was stable up to about 130 °C and, by 300 °C, decomposed to Ag and Ag_2O in air. The *c*/*a* ratio previously reported by Beesk et al. (1981) of Ag_3O is in line with the linear relation of *c*/*a* and unit-cell volumes at room temperature. The oxygen content is hypothesized to be variable and to decrease with increasing temperature. Nonstoichiometric Ag_3O is similar to the suboxides of Ti, Zr, and Hf (Hirabayashi et al.).

Beesk,W, Jones P.G., Rumpel, H., Schwarzmann, and Sheldrick, G.M. (**1981**). "X-ray crystal structure of Ag_6O_2," J. Chem. Soc., Chem. Commun., 664-665.

Hirabayashi, M, Yamaguchi, S., and Arai, T. (**1973**). "Superstructure and Order-Disorder Transformation of Interstitial Oxygen in Hafnium," **35**(2), 473-481.

[1] Corresponding author: paul.schields@amriglobal.com.