Ref: JCT_2018_153
Title: Thermal-alteration interphase transformations in natural and synthetic arsenic sulfide As4S4 polymorphs
Journal: The Journal of Chemical Thermodynamics
Corresponding Author: Corresponding Author: Oleh Shpotyuk
Co-authors: Co-authors: Andrzej Kozdras, Peter Balaz, Zdenka Bujnakova, Yaroslav Shpotyuk
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Kind regards,
R D Weir
Editor
The Journal of Chemical Thermodynamics

Abstract:
Thermal-alteration interphase transformations in natural (realgar α-As4S4 of two mineral origins) and synthetic (commercial powdered high-temperature β-As4S4 modification synthesized from elemental constituents and subjected to high-energy mechanical ball milling) arsenic monosulfide polymorphs are studied exploring the temperature-modulated DSC TOPEM® method. Specific heat capacity and non-reversing heat flow variation in realgar α-As4S4 demonstrate two endothermic events, these being ascribed to interphase α→β transformation at ~270-275oC, and melting of this newly-formed high-temperature β-As4S4 phase at 308-309oC. This polymorph originated from thermal-alteration of mineral realgar possesses congruent melting in contrast to synthetic β-As4S4 polymorph, which always show non-equilibrium melting due to accompanied generation of compositionally-authentic amorphous phase. Calorimetric studies on synthetic β-As4S4 polymorph in powdered coarse-grained and milled states show complicated non-equilibrium melting in principally different crystalline-amorphous environments along with crystal-to-glass transformations. Structural-chemical heterogeneity at β-As4S4 crystallites results in incongruent double-peak melting through two endothermic events at ~305oC and ~315oC. The amorphous phase formed under high-energy milling of synthetic β-As4S4 possesses a dual nature due to stabilization of As-rich glassy substances with low- and high-temperature glass transition mid-points. This process in the powdered synthetic β-As4S4, identified as re-amorphization of initial amorphous phase and direct vitrification from β-As4S4 crystallites, was parameterized as compared to calorimetric thermal-alteration events in orpiment As2S3 mineral.

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Compose

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Dear Dr. Balitska,
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Kind regards,
R D Weir
Editor
The Journal of Chemical Thermodynamics

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The paper “Thermal-alteration interphase transformations in natural and synthetic arsenic sulfide As_4S_4 polymorphs” by Oleh Shpotyuk et al. presents an important insight on interphase transformations in synthetic β-As_4S_4 as compared with minerals of realgar α-As_4S_4 (provided from two different mines) and orpiment As_2S_3. The most essential findings concern conclusion on incongruent double-peak melting of β-As_4S_4 polymorphs in respect to congruent melting of natural minerals, the effect attributed to crystalline-amorphous heterogeneity in synthetic polymorph. Other important finding is that thermal alterations in β-As_4S_4 affected to high-energy milling include re-amorphization and vitrification, resulting in dual nature of amorphous phase with low and high glass transition mid-point temperatures.

In view of novelty and importance of these findings, this paper is surely worth publication in JCT (Journal of Chemical Thermodynamics).

I only suggest minor revisions before publication this paper to improve its soundness for JCT readers.

- in “Results and Discussion” part, the authors should underline an essential difference in thermodynamics parameters of amorphized arsenic monosulfide As4S4 and arsenic trisulfide As2S3 compounds as follows from their DSC-TOPEM study with the same comparison as given by Brazhkin with co-authors (ref. [20]) extracted from XRD study on pressure-induced amorphization in As4S4.

- improvement of English especially in the Discussion part is recommended.

Conclusion: publication after minor revision

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I hope that you will consider The Journal of Chemical Thermodynamics as a potential journal for your own publications in the future.

Kind regards,

R D Weir
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