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Development of a new type of crucible for studying thermal properties of pressurized samples involving gases by calorimetry, application to planetary science and energy transport and storage

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Commercial crucibles used in common Differential Scanning Calorimeters (DSC) are loaded with solid and/or liquid samples at atmospheric pressure and ambient temperature. Even if some crucibles can support pressure increases (for instance up to 150 bars for disk-type DSC) due to temperature variations, the sample is introduced in the crucible always at atmospheric pressure.
Is it possible to extend the application of DSC to measure the thermal properties of pressurized samples including gases by developing a new type of crucible?

In this work, we present the approach currently applied at CTP for the design, realization, loading, and testing of such a crucible, with emphasis on perspectives of further improvements. About 10 prototypes have been realized in our workshop by now; their reliability has been tested by measuring the solid-solid and solid-liquid transitions of acetonitrile and the sublimation of carbon dioxide. Each prototype has been conceived for overcoming the drawbacks of the previous one following a trial-and-error approach.

The DSC we are using for comparing thermal properties of known chemicals obtained with the new crucibles is a NETZSCH 3500 Sirius DSC capable of working down to liquid nitrogen temperature. This apparatus will be used for evaluating the thermodynamic behavior of mixtures of interest for energy transition and planetary science down to cryogenic temperatures. The main goal of this work is to determine if co-crystals (naturally occurring in celestial bodies [1]) can be seen as a valid alternative for the transport and storage of hydrogen and methane on Earth, chemicals that are in the gaseous state at ambient temperature.

The experimental protocol in place for testing the new crucibles has been previously defined by comparing experimental thermal properties (melting temperature, heat of solid-solid and solid-fluid transitions) measured with the NETZSCH DSC to literature values for known systems like water, benzene, acetonitrile, and benzene + acetonitrile mixture, this latter presenting co-crystals [2].

Results obtained with the last versions of the new crucibles on sublimation of carbon dioxide are promising. The trial-and-error approach has revealed the most critical factors that have to be considered in the design and the realization of the crucibles, and in the loading of these with gaseous components in order to obtain physically sounded and reliable results. Nevertheless, further improvements have been identified and will be implemented in the next future.

[1] M.L.CABLE; T.RUNCEVSKI; H.E.MAYNARD-CASELY; T.H.VU and R.HODYSS **"Titan in a test-tube: Organic co-crystals and implications for Titan mineralogy"** *Acc.Chem.Res.2021, 54, 3050-3059*

[2] C.A.MCCONVILLE, Y.TAO, H.A.EVANS, B.A.TRUMP, J.B.LEFTON, W.XU, A.A.YAKOVENKO, E.KRAKA, C.M.BROWN and T.RUNCEVSKI **« Peritectic phase transition of benzene and acetonitrile and formation of a co-crystal relevant to Titan, Saturn's icy moon »** *Chem.Commun.2020, 56, 13520-13523*

Preferred type of contribution:

☐ **Poster**

☒ **Oral**

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