Supplementary material

In the supplementary Fig. 1 a thin section from the stalagmite BU-U is shown. It refers to the same growth interval as the investigated samples. We show a typical part of the 2 cm x 4 cm large thin section with a thickness of about 30 µm. In general, only few inclusions with a diameter above 5 µm are visible. However, a significantly larger number of small inclusions below 1 µm in diameter are obvious. They show an elongated or spherical shape, which is often found in the case of water-filled inclusions (Schwarcz et al., 1976).

The grain size distribution is of special interest with regard to the type of opened inclusions. We show representative distributions for the two discussed extraction methods. Fig. 2 displays the results for the squeezing in the copper tube and Fig. 3 for the crushing in the steel cylinder. The grain size distribution is determined after extraction of water and noble gases using 4 different sieves with mesh sizes ranging from 2 mm down to 63 µm. The crushed speleothems are poured on the sieves, which are shaken by hand. The remaining coarse fraction is weighed by a high precision scale (Precisa 610 MC-FR).

More than 50 % of the total sample yields grains larger than 630 µm and only 14 % smaller than 200 µm in the case of the copper tube extraction. In contrast, more than 40 % is smaller than 200 µm and even 13 % is milled to particles smaller than 63 µm in the case of the crushing cylinder. A further evidence for the more efficient extraction is the absence of grains larger than 2 mm.
Figure captions

Fig. 1: Thin section of stalagmite BU-U from the same growth period as the
investigated samples for NGT determination.

Fig. 2: Grain size distribution of a sample from BU-U squeezed inside the copper tube
by a vice. The given distribution is also typical for other stalagmite samples treated in the
same way. The uncertainties refer to the measurement.

Fig. 3: Grain size distribution of BU-U samples crushed in the steel cylinder by hitting
60 times with the steel ball and two additional samples treated similarly. The uncertainties
reflect the standard deviation of these samples.

Fig. 4: Structure of the stalagmite BU-U from Bunker Cave. Many parts with a milky
white appearance can be seen. They indicate a high fraction of water filled inclusions. On the
upper left side a photo with higher magnification shows crystalline features. The sample for
noble gas analysis was taken from the right stalagmite side. The growth axis inclines towards
the upper right edge.

References

Acta 40, 657 - 665.
Fig. 2

- <63 µm
- 63 µm-200 µm
- 200 µm-630 µm
- 630 µm-2.0 mm
- > 2.0 mm

Bar chart showing weight percentage for different size categories.
Fig. 3

1. <63 μm
2. 63 μm-200 μm
3. 200 μm-630 μm
4. 630 μm-2.0 mm
5. > 2.0 mm

weight-%
Fig. 4