



Retsch GmbH
Retsch-Allee 1-5
42781 Haan
Germany

Phone 02104/2333-100
E-Mail info@retschi.com

www.retschi.com

METHANE IN ROCKS

HOW ANALYTICS HELP UNRAVEL THE SECRETS OF OUR PLANET



ALMA MATER STUDIORUM
UNIVERSITÀ DI BOLOGNA

"Why are we interested in methane deep within rocks? The analysis of methane and its isotopic ratio from tiny fluid inclusions trapped in minerals provides valuable insights into the formation and evolution of our planet. For example, it enables us to distinguish whether methane originated from biological processes, geochemical reactions, or thermal decomposition. This knowledge is crucial for a better understanding of global carbon cycles, energy resources, and environmental developments."

The research group led by Prof. Vitale Brovarone at the University of Bologna, Italy, is dedicated to investigating these questions. The methodological details and validation of the analytics described here have been thoroughly documented in a recent study by Olivieri et al. (2024) [1].

Methodological Approach

To release methane from fluid inclusions, rock samples were ground using Planetary Ball Mill PM 100 and 250 ml jars made of zirconium oxide. The jars were hermetically sealed with a safety closure device in combination with special RETSCH aeration lids (fig. 1), evacuated, and flushed with zero air to avoid contamination. Thanks to the aeration lids, the released gases could be extracted directly after milling without opening the system, see fig. 2.



Fig. 1: Aeration lids offered in different materials and Planetary Ball Mill PM 100

Fig. 2 illustrates the test setup and the most important components of the system. The analysis of methane concentrations was performed using Cavity Ring-Down Spectroscopy (CRDS), a highly precise spectroscopic method. The extraction protocol, artifact minimization strategies, and interlaboratory comparison of similar setups are critically discussed in Olivieri et al. (2024) [1].

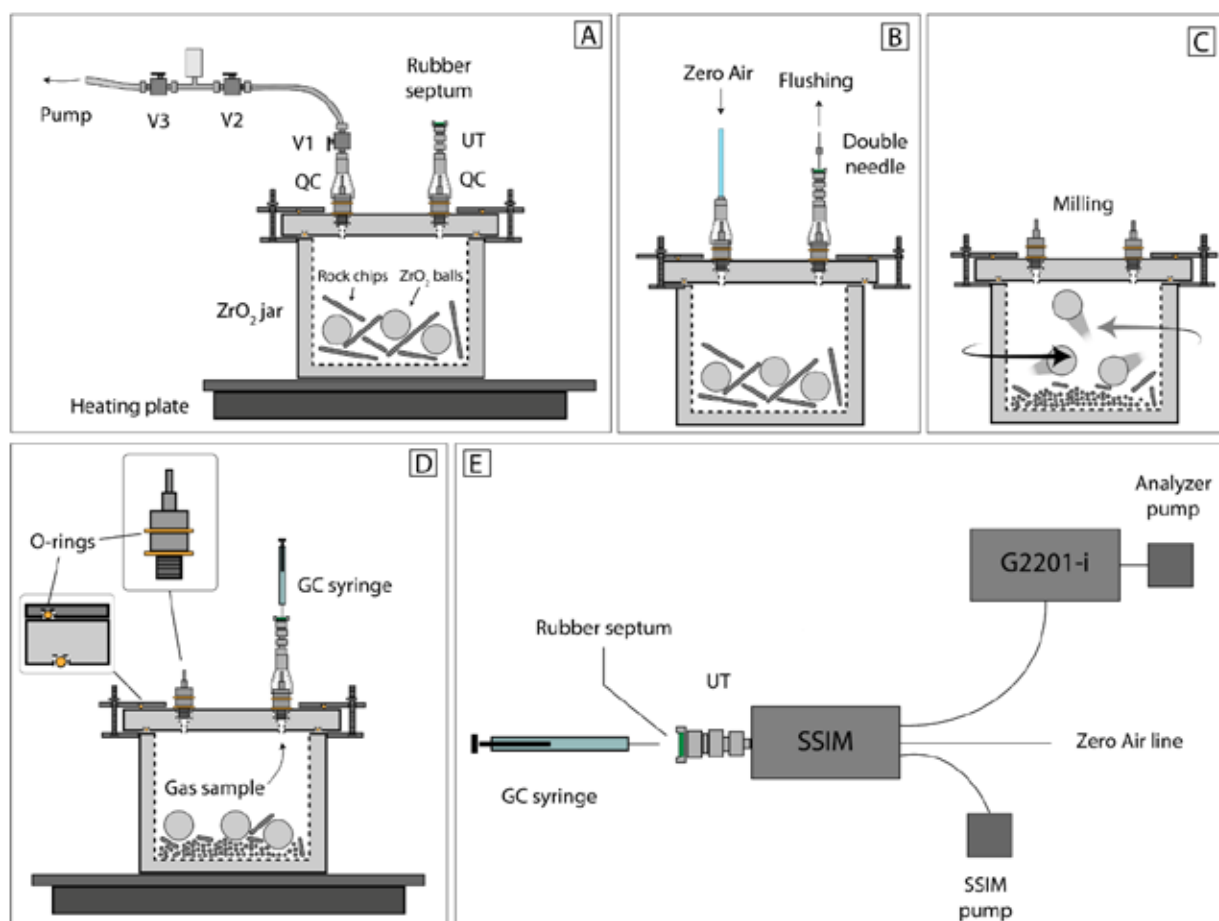


Fig. 2: Overview of the ball milling extraction setup and protocol steps: (A) jar evacuation, (B) flushing with Zero Air, (C) milling, (D) gas extraction, and (E) sample injection for analysis. Key components and connections are indicated. Picture taken from Olivieri et al. (2024) [1].

Results and Conclusion

The methane trapped in rocks can be extracted and analyzed using this setup. By comparing two jar materials (steel and zirconium oxide) under identical conditions, it is also possible to assess whether the vessel material influences the measurement results and how it affects the reproducibility of the analyses. RETSCH Planetary Ball Mills, such as the PM 100, are known for high performance and precision. They accommodate jars and adapters from 1.5 ml vials up to 500 ml jars, with up to four grinding stations. User-friendly settings allow easy control of time, speed, and cycle programs. Equipment options include safety closures, a pressure and temperature control unit, and an aeration lid with interchangeable inlays for use with steel, zirconium oxide, tungsten carbide, or agate jars (see Fig. 1). RETSCH mills and the appropriate accessories provide a reliable basis for safe and reproducible analysis. As shown, this enables precise geochemical investigations and offers an important contribution to better understanding the geological secrets of our planet.



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[1] Olivieri, Orlando Sébastien, et al., *Frontiers in Earth Science* 12 (2024): 1438382.