

# Methods

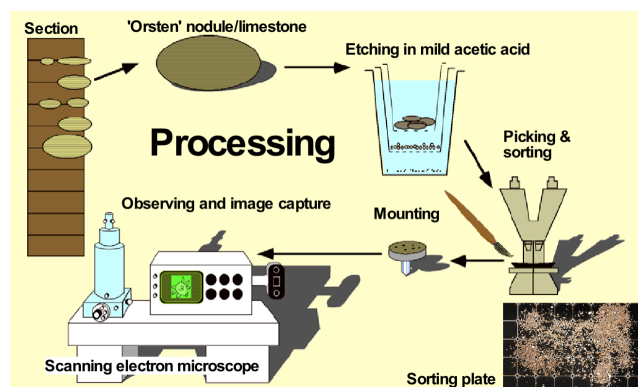
The most important and pioneering work was done by Klaus J. Müller and his field work already in the 1970s. The major amount of nodular limestone material yielding fossils in 'Orsten' preservation came from a quarry near a former manor house Gum at the south western edge of the hill Kinnekulle at the southern margin of Lake Vänern, now a nature reserve and closed to the public. More details will be added in a separate part in due course. Remarkably, no further excursions yielded major amounts of fossils with preserved "soft parts" (exception: some material collected by Japanese colleagues, but mainly unpublished, see Maeda et al. 2011). Again, the principle way of how to recognize productive rock in the field, and further processing the rock was developed by Klaus. Subsequent investigations, however, were progressively advanced and modified.

## 1. Collecting and processing 'Orsten' fossils

'Orsten' fossils are phosphatized – this is a fluoric apatite, similar to our tooth material. In the field, one cannot see anything of the secondarily preserved soft cuticular parts of or entire fossils – impossible due to their size –, but Klaus used the "trick" by discovering the shiny phosphatic shield halves of phosphatocopids. This worked fine in southern Öland, but according to our observations, much less so in Gum, for example. Another way, but Klaus was excellent in this was to inspect the colour, size of the crystallites, and consistency. With this he was able to spot fine differences and could immediately recognize of a nodule or piece of it would be successful. This also worked with the material we received in Moscow, where he selected four small pieces, which yielded four tiny tardigrades, our 100 kg were just empty. The numerous calcified *Agnostus* shields are, however, no indicator. Dieter once collected 'Orsten' nodules from a road cut, but all were just "empty".

The entire process is illustrated on the right side for the method developed for Orsten material from Sweden. Klaus has described general aspects in several papers, which are summarized here. Material from different areas may require adaptation to specific differences.

The first steps were done in the so-called acid laboratory: First the 1-2 kg samples of nodules (or complete small orstenar) were crushed to pieces of walnut size and put onto sieves fitting into 10 l household buckets filled with diluted acetic acid. Klaus always used not more than 10% acetic acid. He also used some buffering to be sure that no fossils are lost. Klaus had tested sieves of different size, but below 100 µm too much dirt (non-animal material) remained on the sieves. Therefore he selected one coarser and a smaller sieve, preferably of a mesh size of 200 µm and 100 or 50 µm and left the rock pieces 10-14 days in the buckets and then washed the residues to get rid of remaining acid. Fossils in 'Orsten' preservation are extremely brittle and experience showed that phosphatic matter became also eroded after a while, so too strong treatment



destroys all the material = success 0%. Thereafter the residues were dried and processed further. Other colleagues, mainly the conodont workers used higher solutions – the primarily phosphatic conodonts seem to be more resistant against the acid. Others also put an entire huge rock sample into a basin filled with strong acid and left it there for half a year. Buffering should happen by the eroding surface, but to our experience exactly this happens and would not allow any fossil to survive.

The further steps are:

- Pre-sorting: this occurred in sorting plates under a sorting microscope (Klaus preferred a special handmade Zeiss model); the fossil fragments are spread into a flat rectangular plate with a grid on the surface and holes in the center of each square and sorted out using a fine brush;
- selection of promising specimens: the selected material was again inspected for specimens of interest, which were pushed through the holes into Franke cells, which is placed underneath the sorting plate in a kind of carrier, which has a hole in the center and from there selected and the best glued onto SEM stubs; the rest was archived in small bottles and in the Franke cells;
- the next step is a detailed check of the Franke cells for 3D fossils rather than small shellies and conodonts (if they haven't remained in the remains already); both these elements are also phosphatized and interesting, but were not the direct scope of our 'Orsten' research; the material may also contain other phosphatized fossils such as brachiopod shells and also sponge spicules (secondarily changed into phosphate or primarily silicified); in order to save money, Klaus decided not to mount each specimen onto a single stub, but to mount several, in general 10 specimens on a stub in some distance from the center, this was to avoid blocking a specimen by another more in the front, when the stub was tilted;
- coating of the stubs: the stubs were initially carbon-coated, later gold-coated, and placed into the SEM for inspection; use of the turning and tilting units were invaluable tools for 'Orsten'-type fossils because due to gluing at least one side is disguised, but one can view the specimens from all sides and directions.

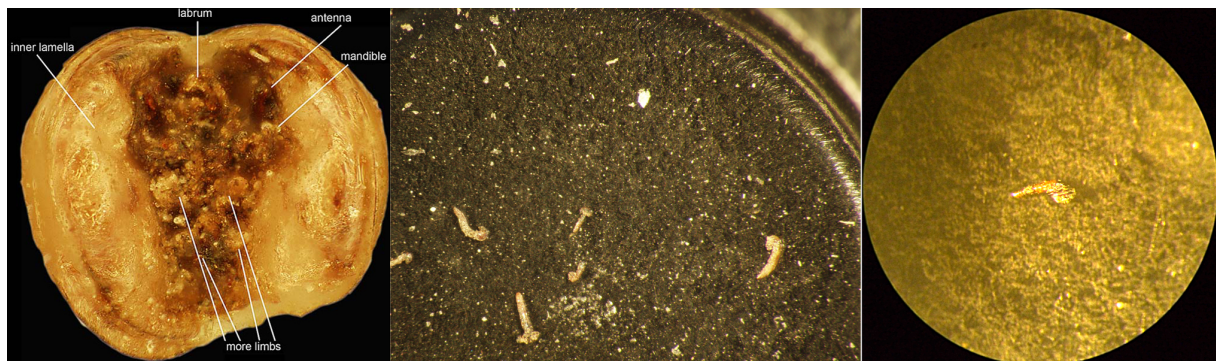
There are two major difficulties with regard to succeeding in attaining Orsten-type fossils:

1. the discovery of suitable rock in the field and
2. the picking and sorting quality; many underestimate particularly the latter aspect, which requires
  - a) a very good microscope and skills in recognizing the specimens – and
  - b) organized working. adequate documentation is required.

Another thing we known know is that limestones having yielded 'Orsten'-type fossils are not necessarily nodular like those from Sweden. The material from Siberia is not nodular but layered and more crust-like. The material we collected in Australia was from nodules, but these were heavily weathered and this may have be the main reason that we failed to find much many fossils in 'Orsten' preservation. May be even that the hard grounds were the only successful rocks. More about the Chinese rocks and their specifities will be added in due course.

Investigation of 'Orsten' fossil may also need some considerations: First of all, by light microscopy stage of processing the material, one cannot even identify if a fossil is well preserved. Such fossils are not only tiny, but shiny and appear to look nice, details cannot be seen very well. Yet, as you may see from the left images below, the ventral details of the body and limbs of this larval phosphatocopine are ill-preserved, only the soft membrane,

inner lamella that spans between out shield rim and body appears nice. We uphold that investigations are largely restricted, so far, to scanning electron microscopy SEM – "our eye" on this minute world.



Lastly a word to accuracy: The sorting process under a microscope is time-consuming and should be done very carefully by skilled persons. People not trained in differentiated viewing will not find much under the microscope. Klaus was extremely focused on and successful in finding persons for this duty. He also let the limestone pieces being cleaned from recent dirt and contamination, yet he could not avoid that his assistants picked pieces of extant plant and animal material, mainly insects and mites, and glued them onto the stubs. Frequently, also a subsequent contamination with plant hairs from the room flowers occurred.