

WHEN A GOOD RECOVERY FAILS TO ASSESS A TEST METHOD

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In order to monitor corrosion in the reactor coolant circuit, different metals are measured several times each month. Here the focus is on the determination of sodium (Na). The matrix in the reactor coolant is pure water with lithium (Li) concentration varying from 1 mg L⁻¹ to 5 mg L⁻¹ and boron (B) in the range from 20 mg L⁻¹ to 2500 mg L⁻¹. Sodium can be determined by several methods, flame AAS, flame AES, ICP-AES. In routine use the LOQ for flame AES is around 0.5 µg L⁻¹, for flame AAS around 3 µg L⁻¹ and for ICP-AES around 5 µg L⁻¹. Upper concentration level is 100 µg L⁻¹, but focus is between LOQ and 10 µg L⁻¹. All three methods are used at Ringhals' two process control laboratories.

In the validation study, the focus was on the performance characteristics LOD and recovery using matrices of varying boron concentration. In the validation of each method, the recovery was tested by spiking and a good recovery was obtained. After strict contamination control, the LOQ was mainly limited by the technique used for determining sodium.

When comparing results using the same test method, between the two laboratories on samples from the coolant circuit, one lab typically measured 4 µg L⁻¹ and the other lab measured 15 µg L⁻¹. Both laboratories in this case used flame AES. This discrepancy was investigated and found to be background emission from the boron matrix. The solution, to this interference on the background, was either using a cooler flame, or flame AAS. Also an emission technique with background correction, ICP-AES was found possible to use.