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EUROfusion

Methodology to evaluate hydrogen isotopes permeabilities and diffusivities from experiments with a purged permeator setup

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The Q-PETE (Hydrogen Permeation and Transport Experiment) at KIT is set up to investigate hydrogen isotopes permeation through structural materials with specific relevance to the HCPB (helium cooled pebble bed) DEMO blanket breeder zone. The experiment therefore consists of two purged chambers separated by a permeation membrane (made of Eurofer or other steels of interest). The permeated and purged hydrogen is detected (time resolved) with a mass spectrometer. Central objective is the direct determination of material data. The determination of permeation parameters like diffusivity and Sieverts' constants is usually performed by modelling experimental results with a solution of a differential equation, 2nd Fick´s law applied to a flat membrane. Mostly permeation disk or gas release experiments (without purge, measuring the pressure rise) were performed in the past, and according analysis methods were applied.

This contribution will introduce necessary methods for purged permeation experiment analysis: A self-developed branch and bound (B&B) algorithm – least square fit algorithm- consisting of super cycles each with four steps which will calculate the desired diffusion and Sieverts' constant. Signal distortion due to the residence time distribution of the measured gas between the permeation chamber and the analysis station, by re-transformation with a so-called Time Spread Function (TSF), is presented.

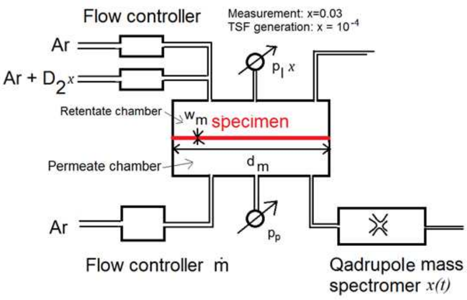

Fig 1: Simplified sketch of Q-PETE experiment. The experimental result for Q-flux is given with eq. (1) $j_{meas,i} = \frac{\dot{m}}{M}\ x(t)$.

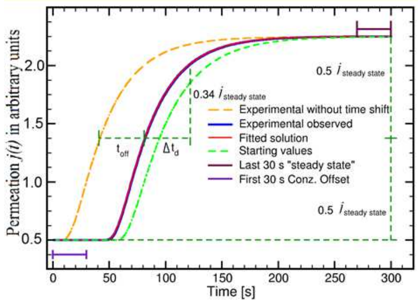

Fig 2: It gives an overview for initial values estimation for B&B algorithm for D_{eff}, c(0) and $_{toff}$. Eq.(2) : $c(0) = K_S\sqrt{x\,p_l}$

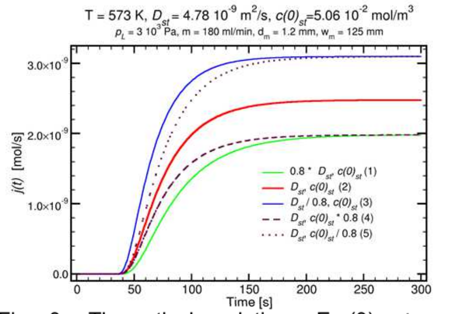

Fig 3: Theoretical solution: Eq.(3): $j_{theo,i} = \frac{D_{eff}\,c(0)d_m{}^2}{w_m 4\,\pi}\left(1+\sum_{k=1}^{\infty}(-1)^k e^{-k^2\pi^2\ D_{eff}(t-t_{off})/w_m{}^2}\right)$

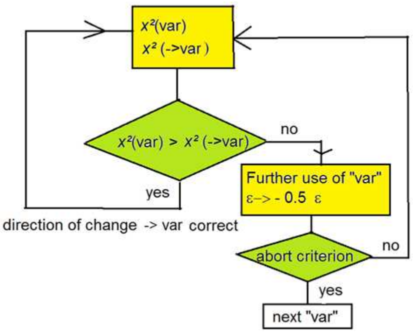

Fig 4: Eq.(3) is a nonlinear transcendent equation with three variables. Is is solved by a B&B algorithm which compares two possible solutions, choosing the better one lower χ^2 .

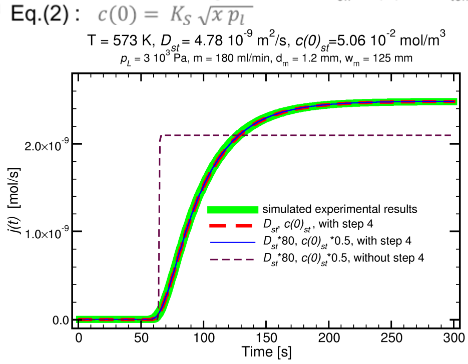

Fig 5: B&B could use only three steps. But bad initial values and numerical problems (shape of χ^2 is parameter space) are forcing a fourth step holding constant the steady state.

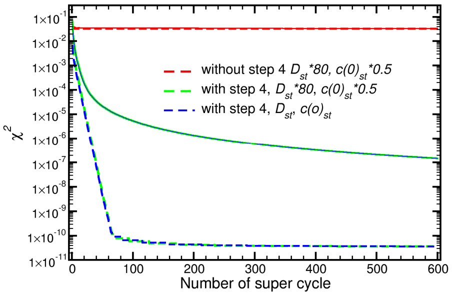

Fig 6: It displays the to Fig 5 coresponding eq.(4) $\chi^2 = \frac{1}{n_m - 1}\sum_{i=1}^{n_m}\frac{(j_{theo,i}-j_{meas,i})^2}{(j_{theo,i}+j_{meas,i})^2}$ values. Note in red a non proper working B&B algorithm.

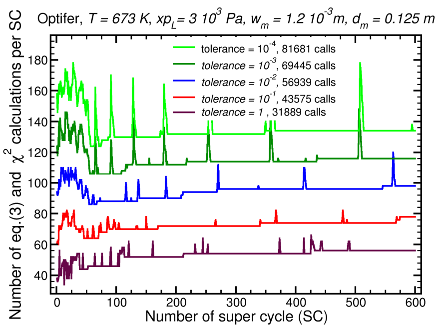

Fig 7:´Result of B&B algorithm optimization procedure. All here displayed solutions own an error less than 10^{-4}. Tolerance is a parameter how fine the variables are treated.

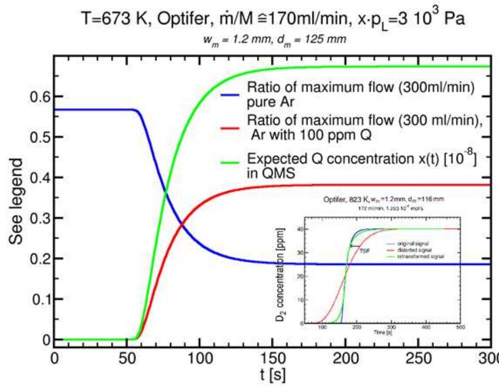

Fig 8: Countermeasure against inter diffusion during transport. The small graphics show original signal by blue line, distorted signal by red. Large graphics shows by blue signal of pure Ar controler and by red that of the calbration gas controler . Green the desired QMS signal.

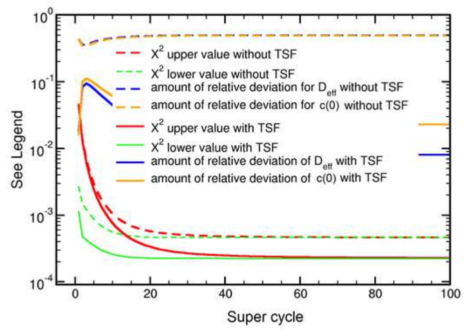

Fig 9: Result of the Q-PETE analysis.. Results with continues lines are using the TSF, dashed lines are results without TSF.

Conclusion: The here presented analysis of a Q-PETE experiment reaches an anlysis error for synthetic data of less than 0.01 %. There are countermeasures against inter diffusion (TSF) and non vanishing Q-concenration in the permeate chamber by e.g. extrapolation to $\dot{m}^{-1} = 0$.

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