TCD Calibration of the AutoChem Using High-Purity Metal Oxides

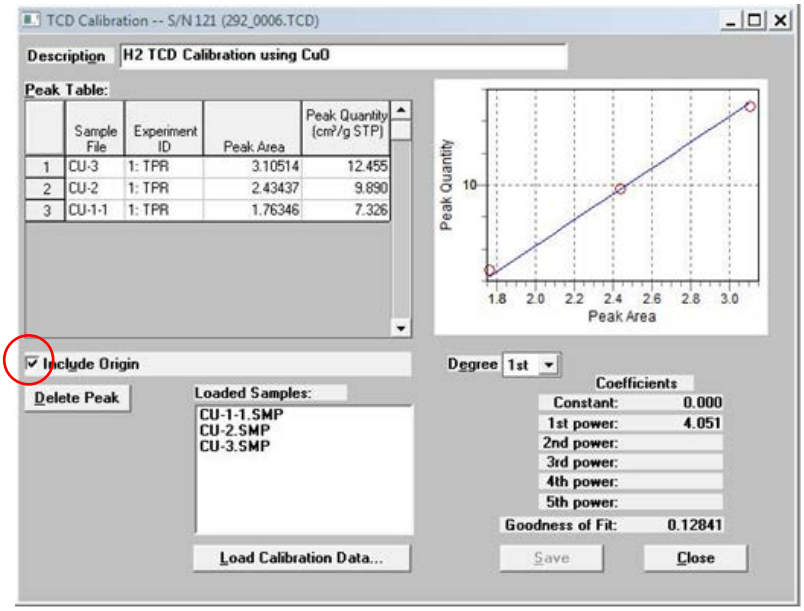
A high-purity metal oxide can be used to calibrate for hydrogen gas using Temperature Programmed Reduction (TPR). High-purity copper oxide (cupric oxide) is recommended due to the large amount of hydrogen gas consumed during its reduction. At least three separate experiments should be performed using three different sample weights and a highly efficient cold trap. If the theoretical amount of hydrogen gas consumed for each sample weight* is used to create a TCD Calibration, the calibration can then be used to determine hydrogen gas volume.

Quantities of 0.0260, 0.0351, and 0.0442 grams of 99.995% copper oxide were used to generate three TPR experiments. The specific reaction is $CuO + H_2 \rightarrow Cu + H_2O$.

The theoretical amount of hydrogen gas consumed for this experiment is 281.78 cm^3 per gram of sample. The adjusted volumes for each experiment were calculated* and the resulting peak areas were integrated.

Sample File Name	CuO Weight (g)	Volume of H_2 Gas (cm^3)
CU-1.SMP	0.0260	7.326218
CU-2.SMP	0.0351	9.890394
CU-3.SMP	0.0442	12.45457

A new TCD Calibration was created using the theoretical hydrogen gas volumes.



*Click here to access a worksheet designed for calculating the theoretical hydrogen volume for different metal oxide weights.

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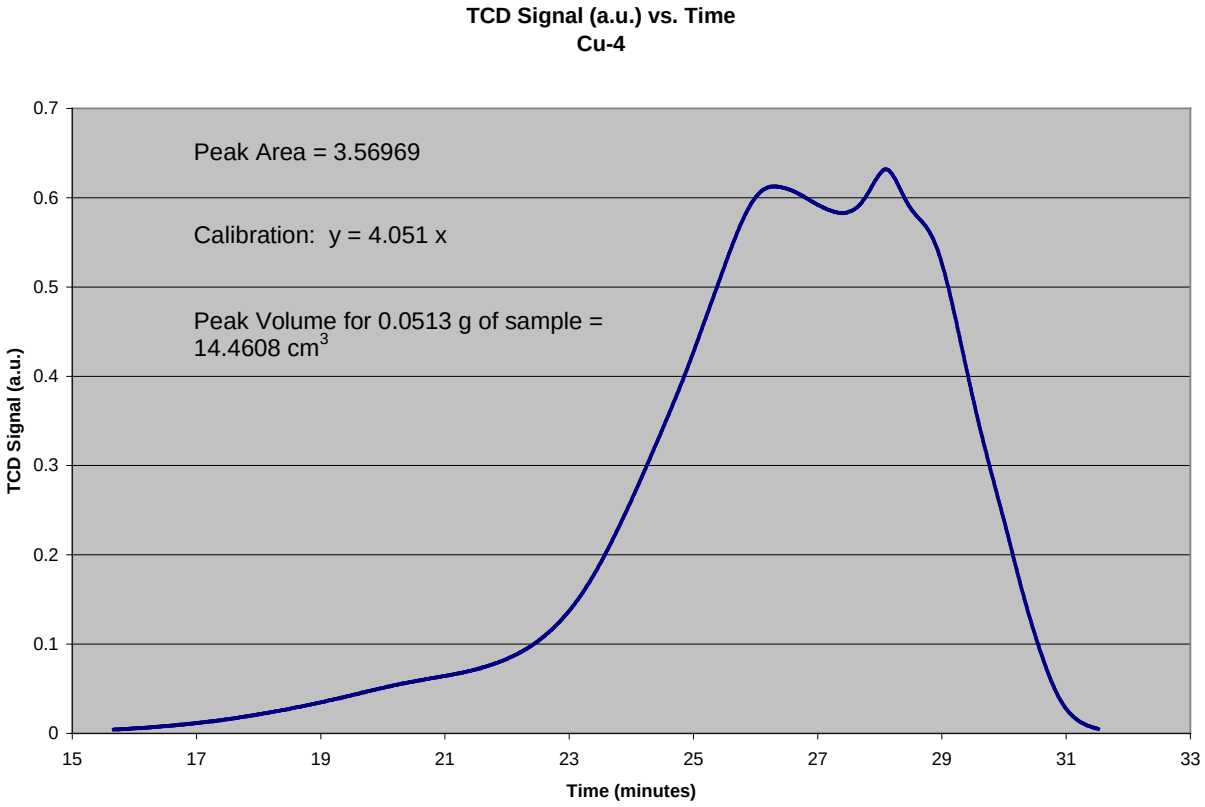
This calibration was then used to test a copper oxide TPR.



The sample file used a CuO weight of 0.0513 grams and consumed 14.46 cm^3 of hydrogen gas or 281.89 cm^3 hydrogen gas per gram of copper oxide. Again, the theoretical amount of hydrogen gas for this reduction is 281.78 cm^3 so the experimental hydrogen consumption was 100.04% of theoretical.

Recommendations:

- ✓ Choose a metal oxide with a reduction temperature similar to the experiments you plan to run.
- ✓ When performing experiments at varying pressures, create a different calibration for each pressure.
- ✓ Select the **Include Origin** option when creating the TCD calibration. This ensures that an area of zero equals a volume of zero.