PALLADIUM Reference Samples

46 Pd

Sample Specifications

These calibration samples have been made from the purest commercially available palladium ("MARZ" grade). Ferromagnetic impurities have been confirmed to be less than 30 ppm by independent analysis.

In addition to chemical analysis, the magnetic moment of the palladium was measured in a Quantum Design MPMS susceptometer. Direct comparison was made with measurements performed on calibrated palladium samples originally purchased from the National Bureau of Standards (NBS Standard Reference Material 765).

The magnetic gram susceptibility of palladium as a function of temperature is given below.

Temperature (*K*)	Susceptibility ($\chi_g \times 10^{-6} cm^3/g$)
295	5.28
296	5.27
298	5.25
299	5.24

These values were taken from the specification sheet provided with the original SRM 765 palladium samples. They are uncertain to less than ±0.5% and have been corrected for demagnetization.

To convert from χ_g to the magnetic moment (in emu) measured by the MPMS, multiply by the magnetic field (in gauss) and the sample mass (in grams).

Measurement of Samples

In order to get the highest measurement accuracy and reproducibility it is necessary to have the sample rigidly mounted in a magnetically clean holder. To verify the susceptibility of the commercial palladium, a fused quartz tube (4mm OD, 3mm ID) was used with the sample held between two 3mm quartz rods inserted from either end. The holder was 200mm long to avoid the effects of the ends. Two phenolic guides of 7.6mm diameter were made for each end to ensure that the sample was centrally mounted within the sample chamber. One must also measure the magnetic moment of the sample holder with an air gap corresponding to the sample length.

The moment of the air gap is equal to the inverse quartz susceptibility of a piece of quartz equal to the length of the palladium sample and of the diameter of the quartz rods. The volume susceptibility of the air gap is expected to be 1.08 emu/cm^3, which is about 1.7% of the palladium volume susceptibility. One must also consider that both the palladium sample and the sample holder will never be completely free of ferromagnetic impurities. Typically our palladium samples will show a ferromagnetic saturation moment of about 3×10^{-5} emu for a 200 mg sample. If the susceptibility of the 200 mg palladium sample were to be determined from a single point measurement at 1000 Oe for instance, its paramagnetic moment would be 1.05×10^{-3} emu. The presence of its ferromagnetic impurities will add 2.9% to the expected moment. Accordingly, at 10000 Oe the error would be 0.29 %. In order to get the best estimate of the paramagnetic susceptibility of the sample one would determine the slope of the magnetization between 10000 and 20000 Oe.

Handling and Cleaning Samples

Note: The chemicals used in these procedures are very dangerous if not handled properly. Please refer to the chemical manufacturer for relevant safety information and handling instructions prior to use.

Always use plastic tweezers when handling samples. Providing that the samples are not ex-posed to ferromagnetic materials they should not require re-etching. To clean a sample use undiluted reagent grade hydrochloric acid (HCl), nitric acid (HNO_3) and if possible, do the cleaning in a chemical hood to avoid acid fumes. Mix the acids in the ratio 3 nitric to 1 hydrochloric and etch the sample for five minutes at room temperature. To wash, rinse the sample two or three times with distilled water.

The MPMS measures magnetic moment that is proportional to the sample volume and is calibrated for palladium samples of mass between 0.225g and 0.275g. As the sample loses mass due to etching its volume will decrease and there will be a slight inaccuracy in the moment returned. For a sample of mass 0.210g the error is ±0.25%. As the sample loses approximately 20mg per 5 minute etch, it is unlikely that mass loss will be a problem.

Statement of Policy

Quantum Design provides these palladium samples to permit owners of the QD MPMS system to verify the overall calibration of the instrument whenever they so wish. It is not proposed that these samples be considered replacements for the now discontinued palladium reference materials previously obtained from the NBS. For up-to-date information on the availability of alternate susceptibility references, please contact the National Institute of Standards and Technology (formerly NBS) at www.nist.gov.

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