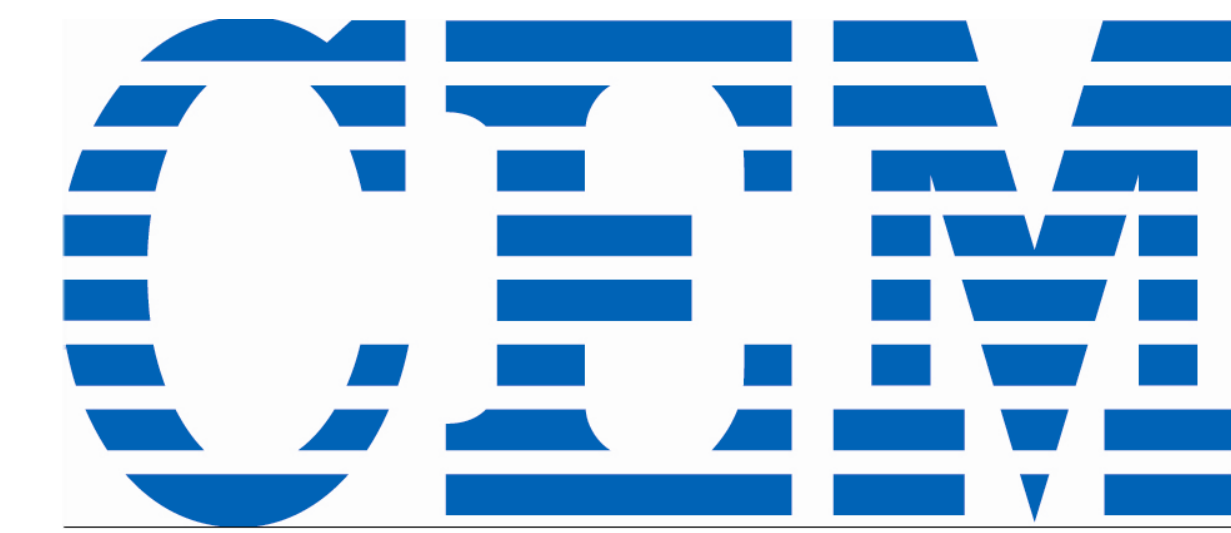


# Quantitative Trace Metals Analysis using Hydrofluoric Acid Alternatives

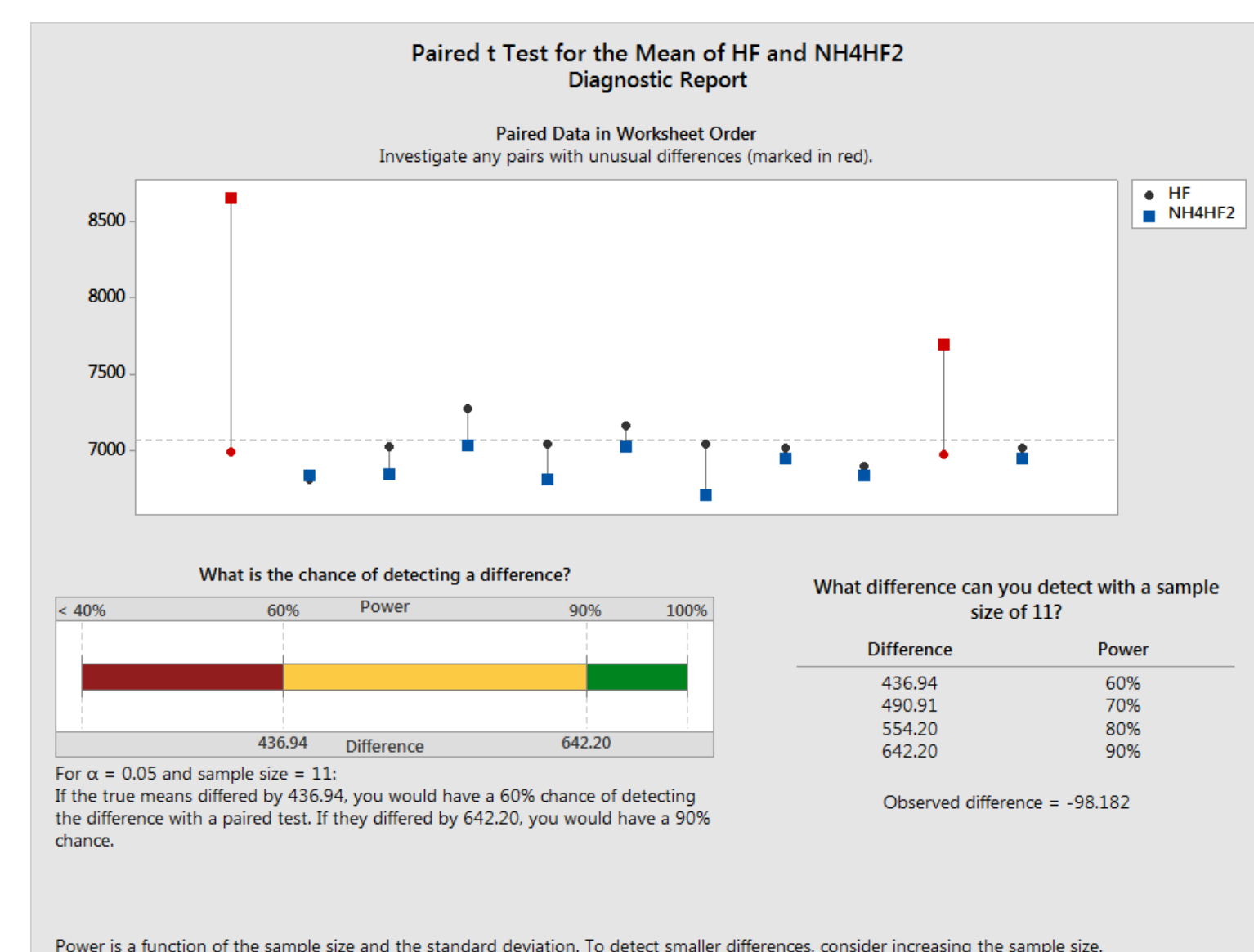
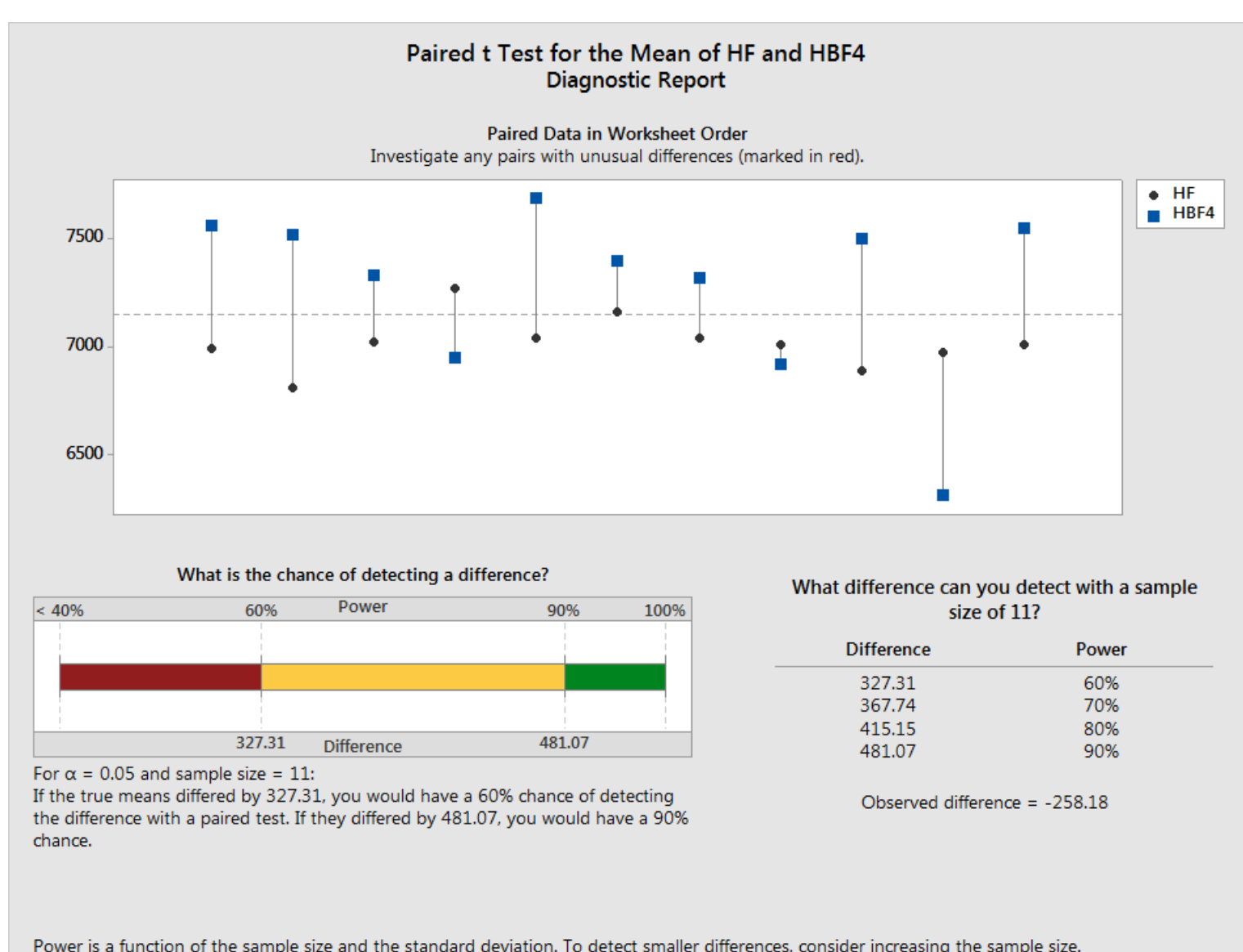


Jane Ramsey and Tom Connell - E. I. du Pont de Nemours and Company  
Daniel Iversen, and Michael Karney - CEM Corporation

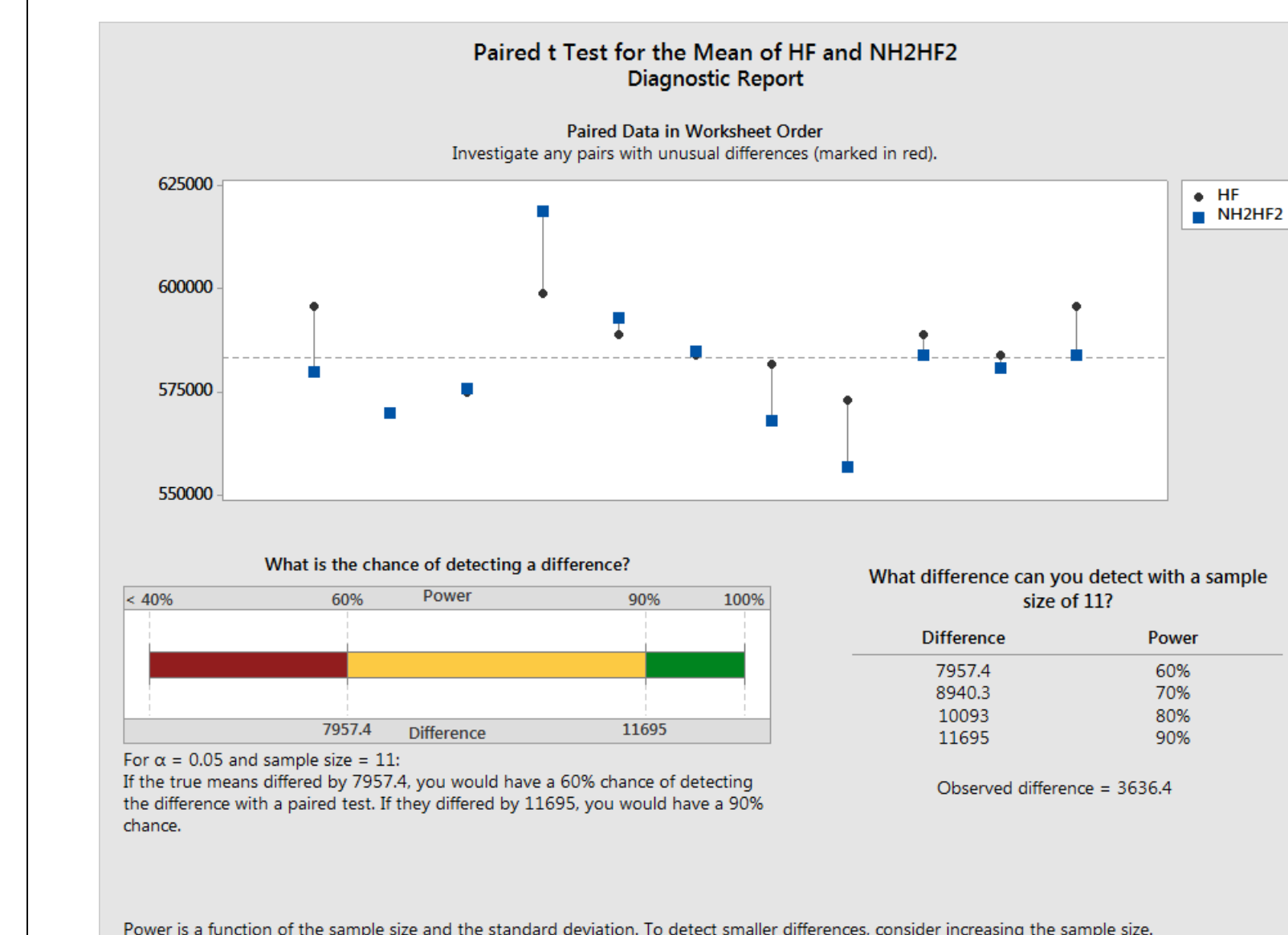


Researchers from CEM and DuPont have worked together to determine if it is possible to use a fluoride surrogate other than hydrofluoric acid (HF) in sample preparation. Total dissolution of a sample matrix is critical to ensure accurate data, and this study investigated two promising, commercially available HF alternatives. Our team used titanium dioxide with trace levels of aluminum and silicon as target analytes for this experiment. Ammonium bifluoride ( $\text{NH}_4\text{HF}_2$ ) and fluoroboric acid ( $\text{HBF}_4$ ) were chosen as fluoride sources and validated against digestions with HF. All samples were prepared with a MARS 6 microwave digestion unit and analyzed with a Perkin Elmer Optima 5300 ICP-OES.

## Trace Al in $\text{TiO}_2$



## Bulk analysis of Ti



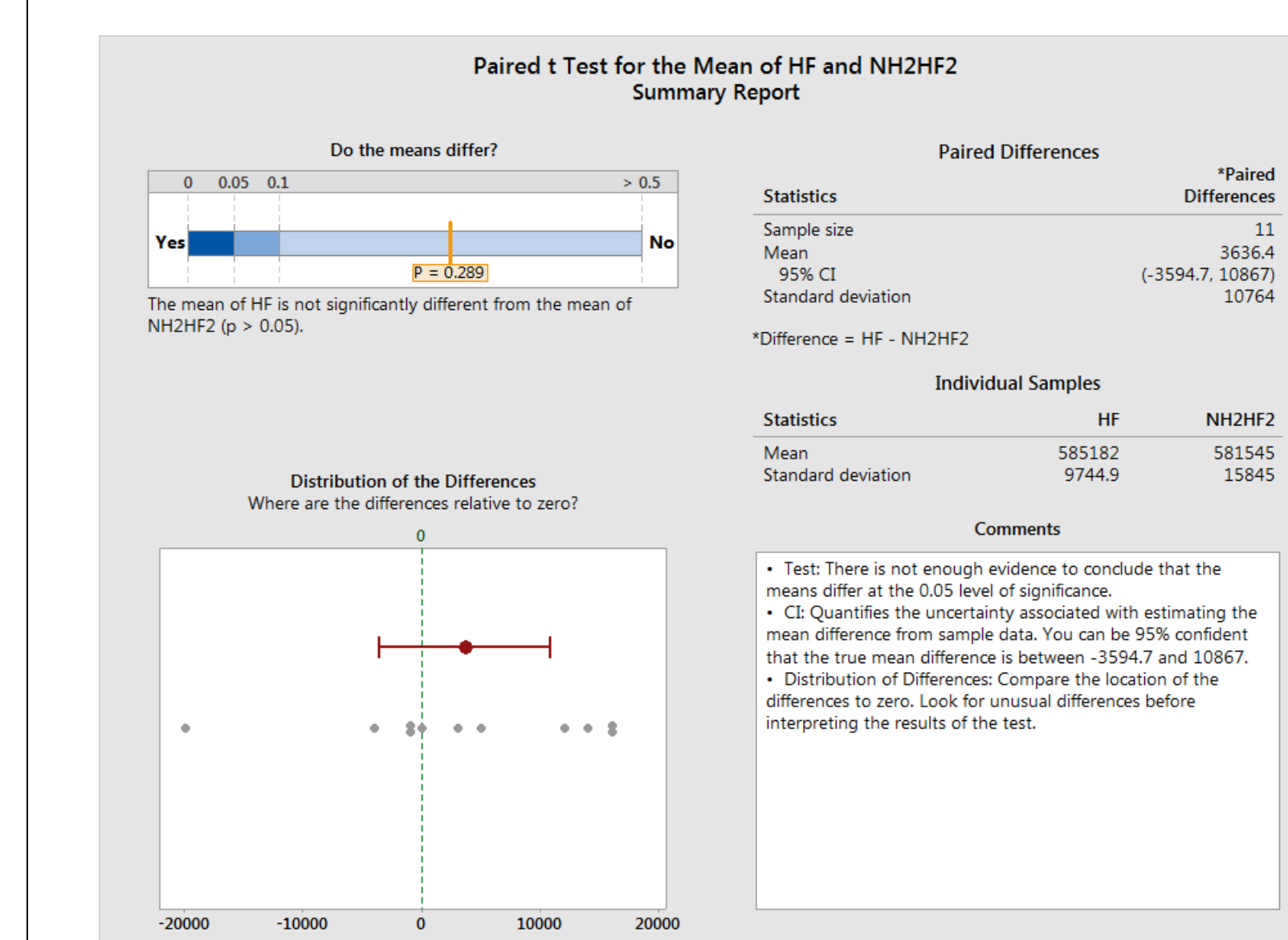
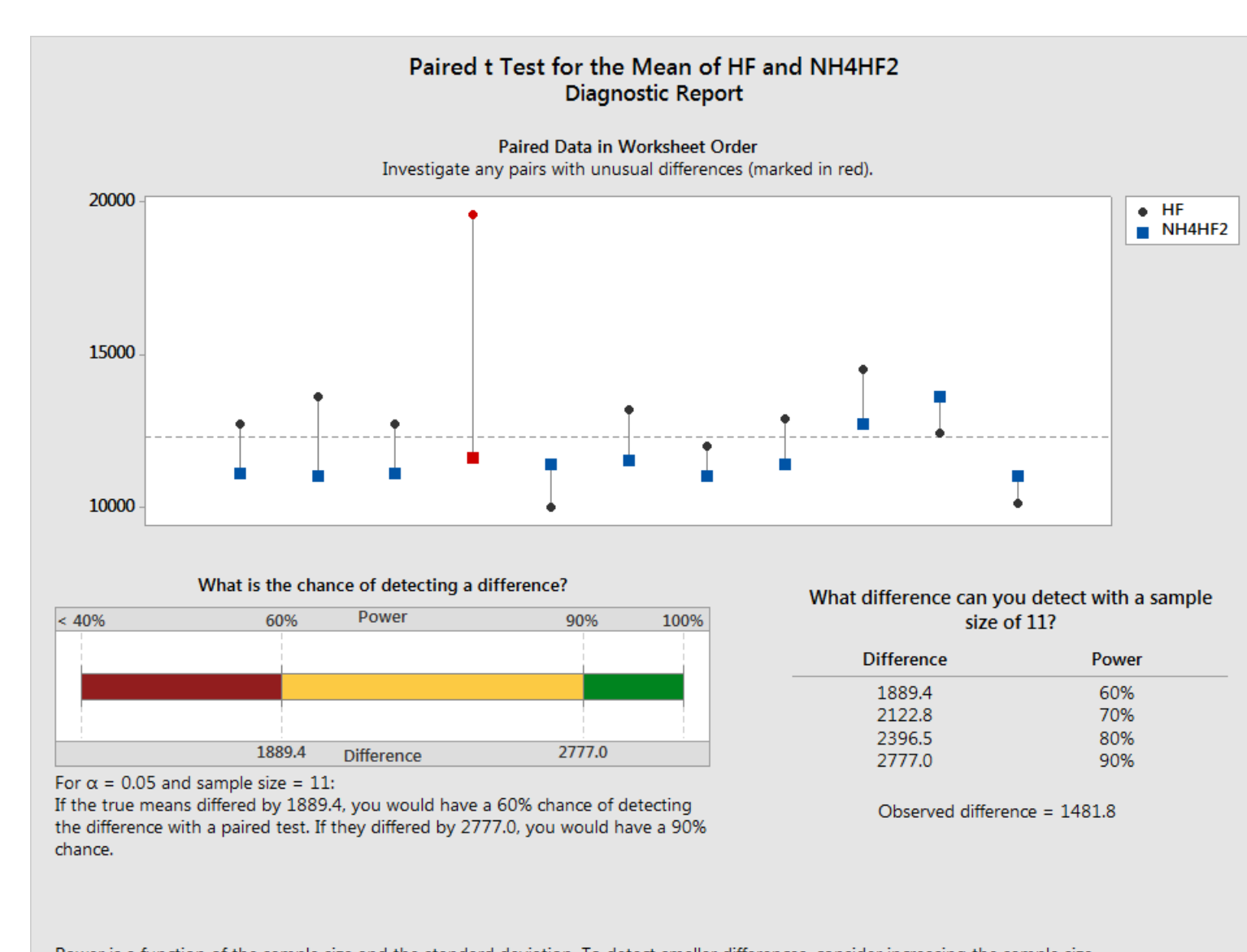
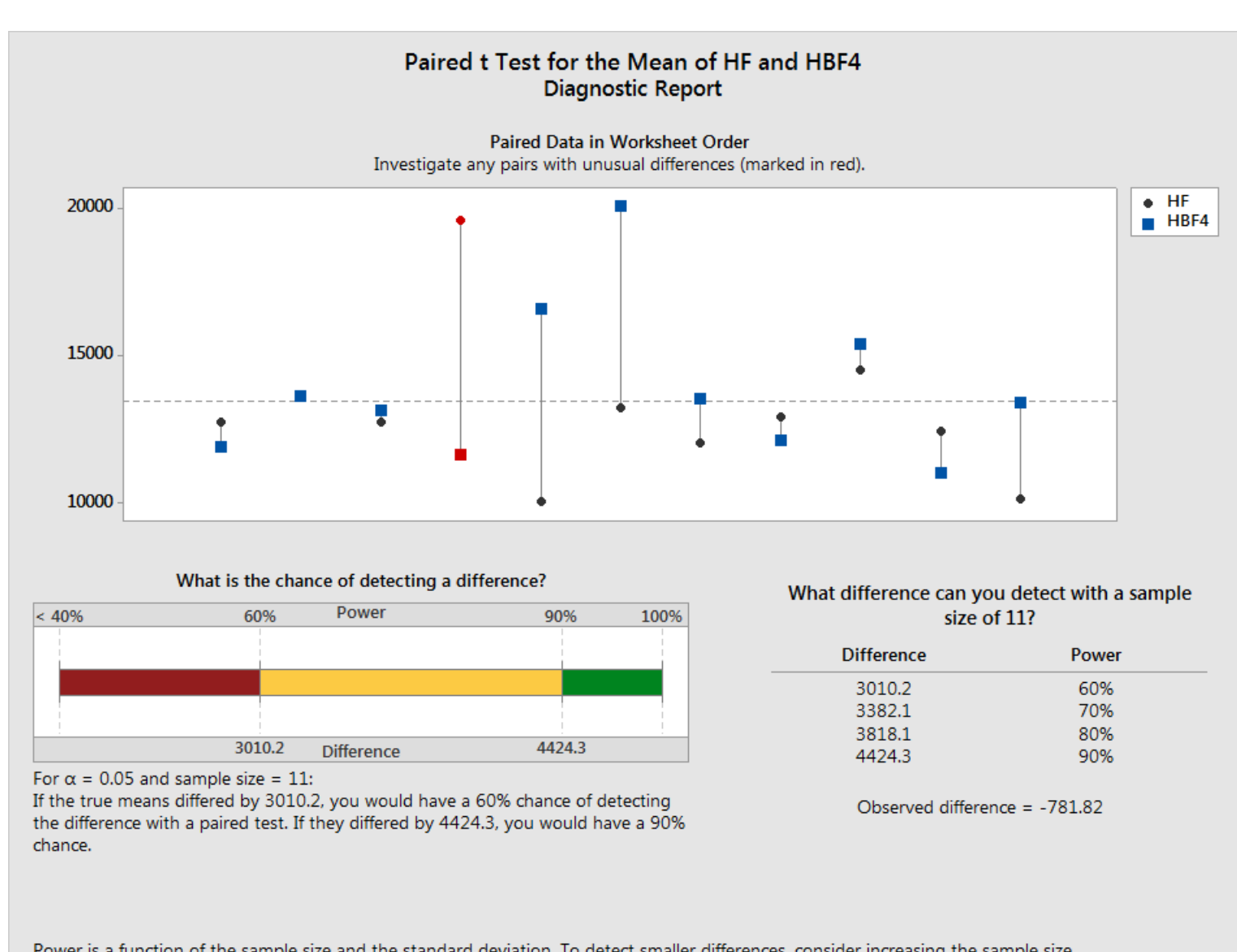
## Experimental Design

Each sample was prepared and analyzed 12 times using HF,  $\text{NH}_4\text{HF}_2$  and  $\text{HBF}_4$  as the principal solvent. A MARS 6 Microwave Digestion System was used ramping to a temperature of 230°C and holding for 30 minutes.

## Observations:

- A Paired t Test was used to determine if the data was the same or different from the HF results.
- The experiment worked with the  $\text{NH}_4\text{HF}_2$  and  $\text{HBF}_4$ . The data suggest that the all digested samples are statistically the same.
- HF out performed the other solvents in some cases.

## Trace Si in $\text{TiO}_2$



## Conclusions:

- HF is still the best solvent for total dissolution of titanium dioxide.
- $\text{HBF}_4$  works well for trace metals recoveries for Al and Si.
- $\text{NH}_4\text{HF}_2$  works as a suitable replacement for the total dissolution of titanium dioxide for trace metals analysis.