

REPRODUCIBILITY OF THE SHIMADZU DTG-60

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Introduction

The Shimadzu DTG-60 is a Simultaneous Differential Thermogravimetric analyzer which combines a heat -flux type DTA with a *Roberval* type TGA which measure both changes in physical state of a sample, as well as weight change, as a function of temperature, over time. This is accomplished by heating both the reference and sample at the same rate, in a single furnace, with a controlled atmosphere. The temperature of each side is compared and the difference output and recorded ($T_s - T_r = DT$). As the sample goes through a phase transition (T_g , T_m , T_c , cure, etc.), the sample will either absorb or radiate heat. Since the DT is proportional to changes in heat, the signal is output in μV . As these changes occur, the sample weight is being read by the *Roberval* balance, and changes in weight are recorded in mg.

Hypothetical

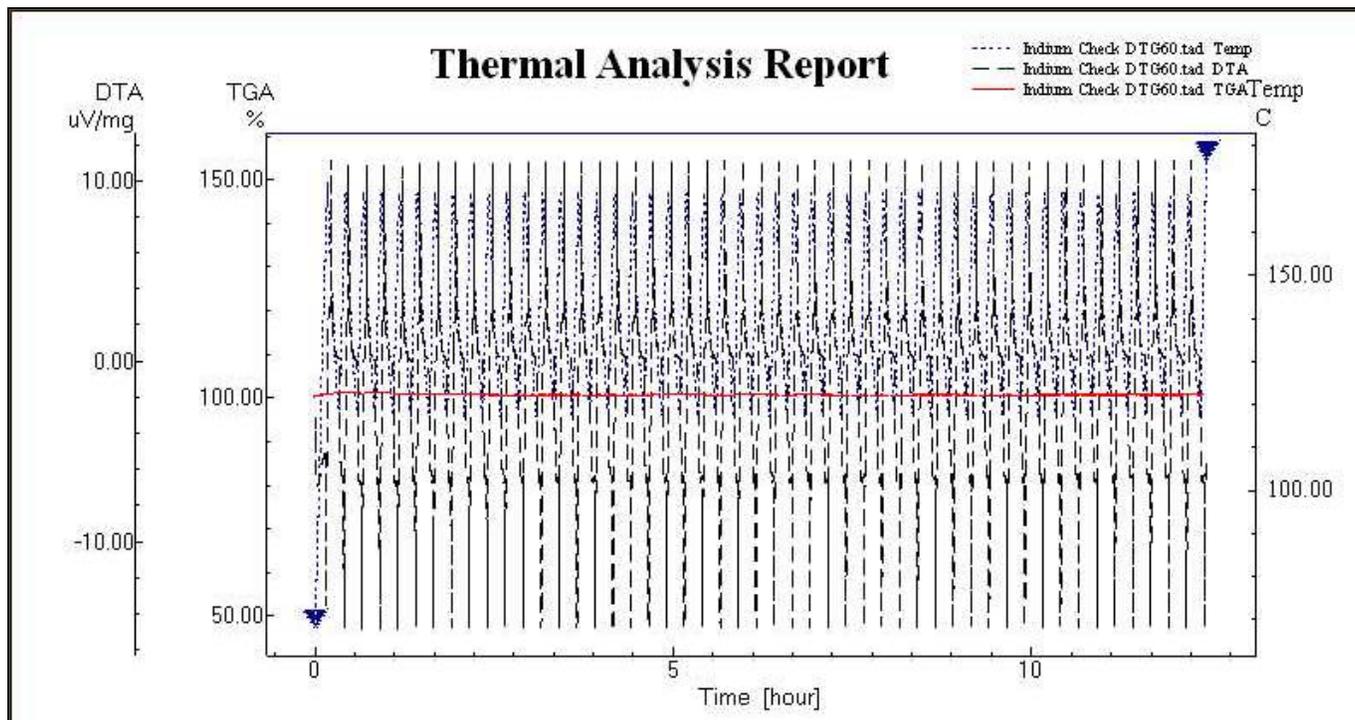
To ensure that the results being reported by the DTG are reliable, an instrument must meet certain criteria. Accuracy, precision, and the ability to repeat analyses are important to any user doing measurements using thermal instrumentation. The DTG-60, when properly calibrated, brings to the user precision (1%), accuracy ($\pm 1.0^\circ C$) and repeatability that can be relied on to produce results of the highest caliber.

Experimental

A typical DTG-60 was used in this experiment to check the accuracy and repeatability of the Shimadzu DTG. The DTG-60 furnace was cleaned by using an oxidative atmosphere flowing at 100 ml/min at 800°C for one (1) hour. The DTG-60 was then calibrated according to published Shimadzu criteria. Upon calibration, an Indium (In) wire standard (99.999% pure) was run 50 times in the DTG. The analysis program employed was a 2 step program. The unit was heated from ambient to 175°C at a gradient heating rate of 10°C/min. The unit was then cooled from 175°C to 100°C at a gradient cooling rate of 10°C/min. This program was repeated 50 times. A Nitrogen (N₂) atmosphere flowing at 50 ml/min was maintained during all 50 cycles. Indium has a recorded melt peak temperature of 156.6°C. The chart below details our findings:

Peak Number	Peak Temp (oC)	Peak Number	Peak Temp (oC)	Peak Number	Peak Temp (oC)
1	156.11	19	156.34	37	156.42
2	156.32	20	156.27	38	156.37
3	156.39	21	156.21	39	156.32
4	156.27	22	156.41	40	156.29
5	156.25	23	156.40	41	156.24
6	156.23	24	156.31	42	156.19
7	156.40	25	156.26	43	156.36
8	156.34	26	156.20	44	156.36
9	156.33	27	156.38	45	156.28
10	156.25	28	156.37	46	156.21
11	156.19	29	156.29	47	156.41
12	156.41	30	156.25	48	156.19
13	156.34	31	156.22	49	156.29
14	156.36	32	156.19	50	156.26
15	156.30	33	156.37	Avg	156.30
16	156.18	34	156.33	SD	0.09
17	156.41	35	156.26	CV	0.06%
18	156.37	36	156.21		

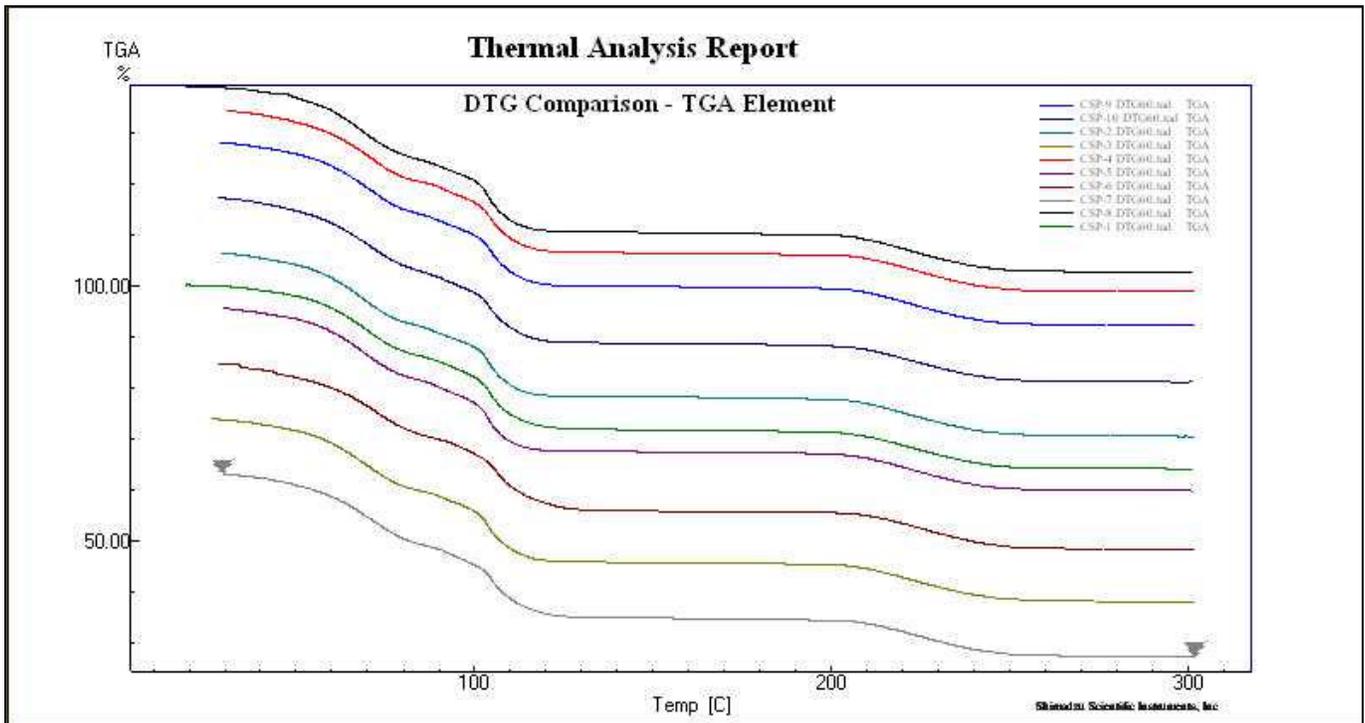
Seen on the next page is the plot of the fifty (50) DTG analysis runs from which the above chart was taken.



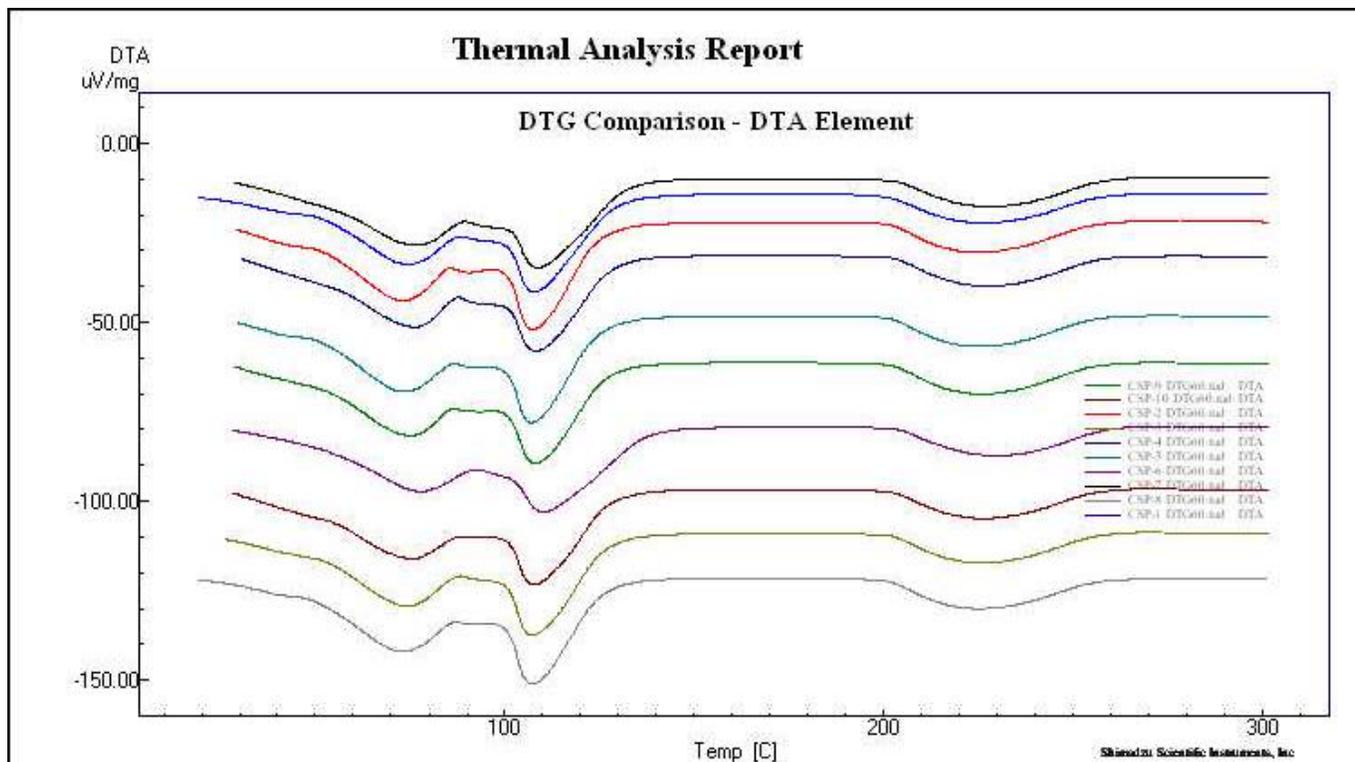
In a second experiment ten (10) separate Cupric Sulfate Pentahydrate ($\text{Cu}_2\text{SO}_4 \cdot 5\text{H}_2\text{O}$) samples were run. The analysis program employed was a single step program. The unit was heated from ambient to 300°C at a gradient heating rate of $10^\circ\text{C}/\text{min}$. A Nitrogen (N_2) atmosphere flowing at 50 ml/min was maintained during all 10 analysis runs. Cupric Sulfate Pentahydrate has three (3) recorded weight loss regions which show the evolution of the water molecules in a ratio sequence of 2:2:1. We also analyzed the endothermic peaks associated with the evolution of the water molecules. The chart on the next page details our findings:

Run	WL-1 %	WL-2 %	WL-3 %	WL-T %	Peak-1 °C	Peak-2 °C	Peak-3 °C
1	14.637	13.758	7.627	36.022	74.89	108.88	226.55
2	14.435	13.934	7.405	35.774	74.33	107.74	224.98
3	14.802	13.428	7.437	35.704	74.61	107.55	226.17
4	14.464	13.555	7.387	35.407	75.69	108.66	227.89
5	14.551	13.728	7.458	35.737	73.75	107.38	225.60
6	14.286	13.716	7.369	35.371	73.26	108.61	226.30
7	14.733	13.674	7.294	35.701	75.82	109.02	226.43
8	14.732	13.954	7.498	36.184	73.33	107.74	225.45
9	14.324	14.056	7.370	35.750	75.52	108.53	226.24
10	15.127	13.514	7.408	36.050	74.95	108.16	226.51
Avg	14.609	13.732	7.425	35.770	74.62	108.23	226.21
SD	0.25	0.20	0.09	0.26	0.94	0.59	0.79
CV	1.73%	1.48%	1.21%	0.73%	1.26%	0.55%	0.35%

Seen below is the plots of the ten (10) DTG analysis runs from which the above chart was taken. First is the TGA curves:



Next is the DTA curves:



Conclusion

As can be easily determined from the data, the DTG-60 provides excellent precision and detailed repeatability. The statistics presented are well within Shimadzu published specifications detailed above. It would be difficult to find similar instrumentation on the market today which can compare itself to these results. In conclusion, if the DTG-60 is properly calibrated, then the sample analysis data can be relied upon to be correct.