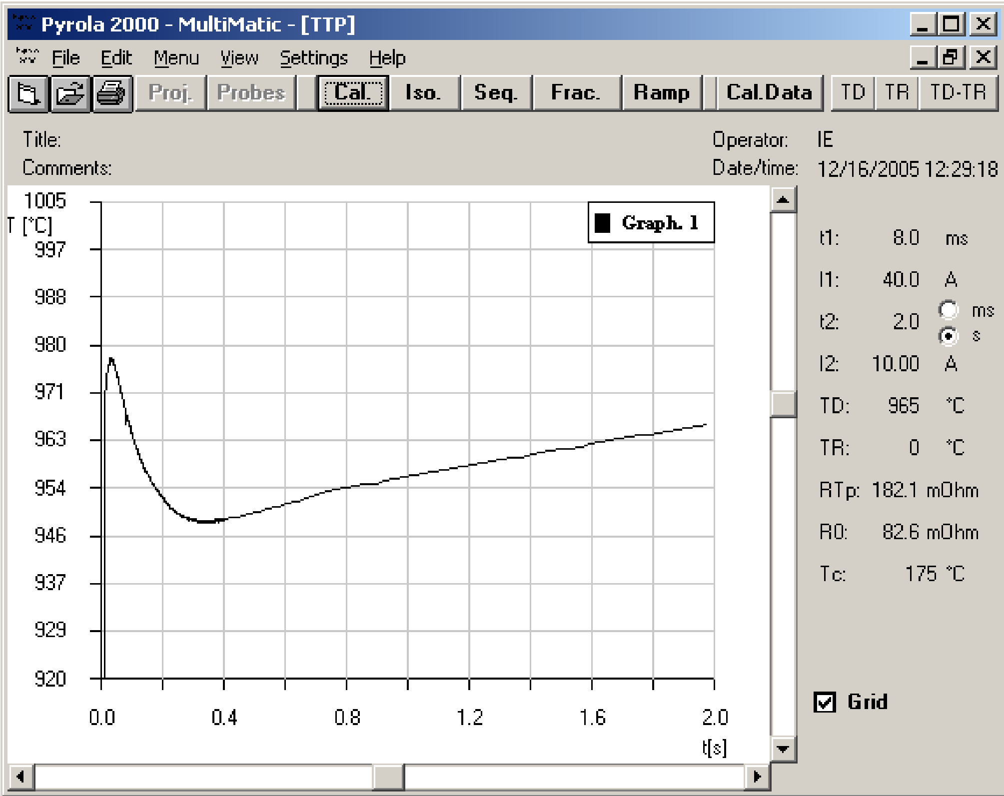
Pyrola Calibration Sequence.

The manual gives basic guidance on how recalibrations are to be performed and this document illustrates a typical calibration of a new filament. When recalibrating or calibrating a new filament, either remove the TTPs to be recalibrated or all stored TTPs if a new filament has been fitted. One calibration must be performed at a temperature between 975℃ and 1000℃ to allow the controller to calculate some basic parameters. Apart from this constraint, the temperatures chosen for other calibrations appear to be at the discretion of the operator. It is thought that ensuring that the calibration temperatures bracket the intended operating range is a sensible policy since early work showed that performance at 300℃ was rather poor when the lowest calibration temperature was 500℃. Whether this was a real effect or a manifestation of operator incompetence is not known. For this reason, this example has target calibration temperature of 1000°, 900°, 750°, 600°, 450° and 300℃ since it was intended to perform fractional pyrolysis at 100° intervals between 300° and 700℃.

1. Cal at 1000℃.

1.1 Initial Results.

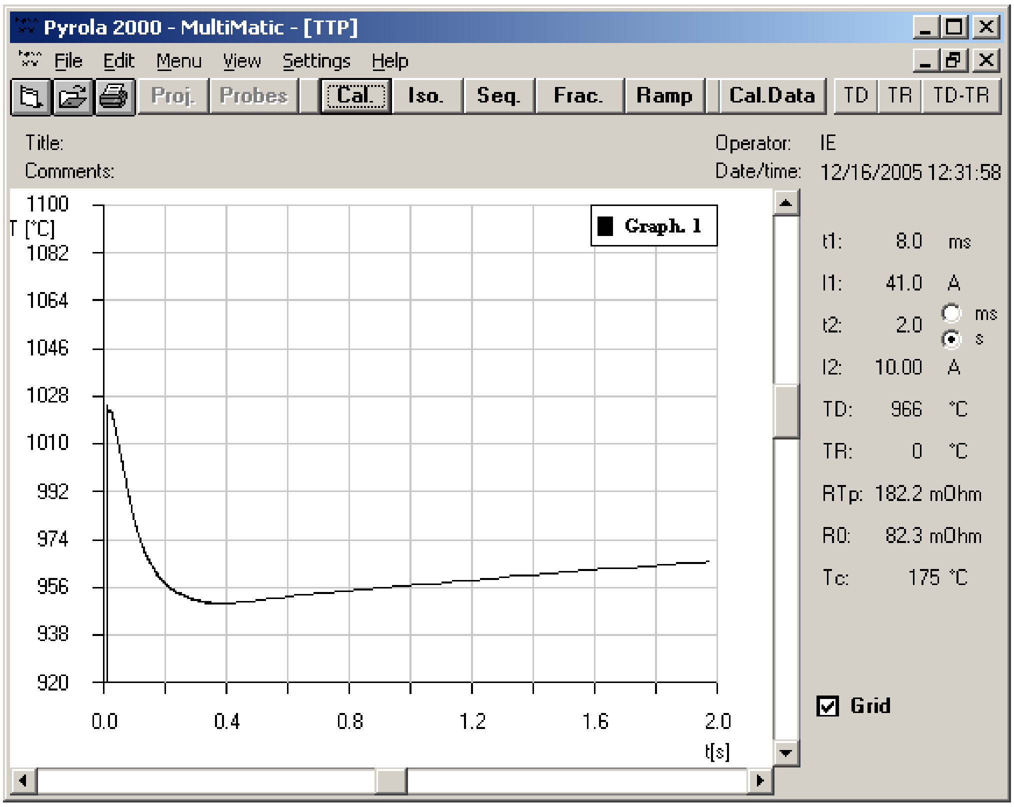
Setting the temperatures and times according to the manual gives the following typical result:



Here, neither I1 nor I2 are sufficiently high since the initial temperature pulse does is greater than 20℃ below the target temperature and the temperature at the end of T2 is 35℃ below the target temperature. Achieving the correct values for I1 and I2 are a matter of experience and educated guesswork. Ultimately an iterative approach is required with progressively smaller adjustments of I1, I2 or both I1 and I2 as the desired calibration temperature is approached.

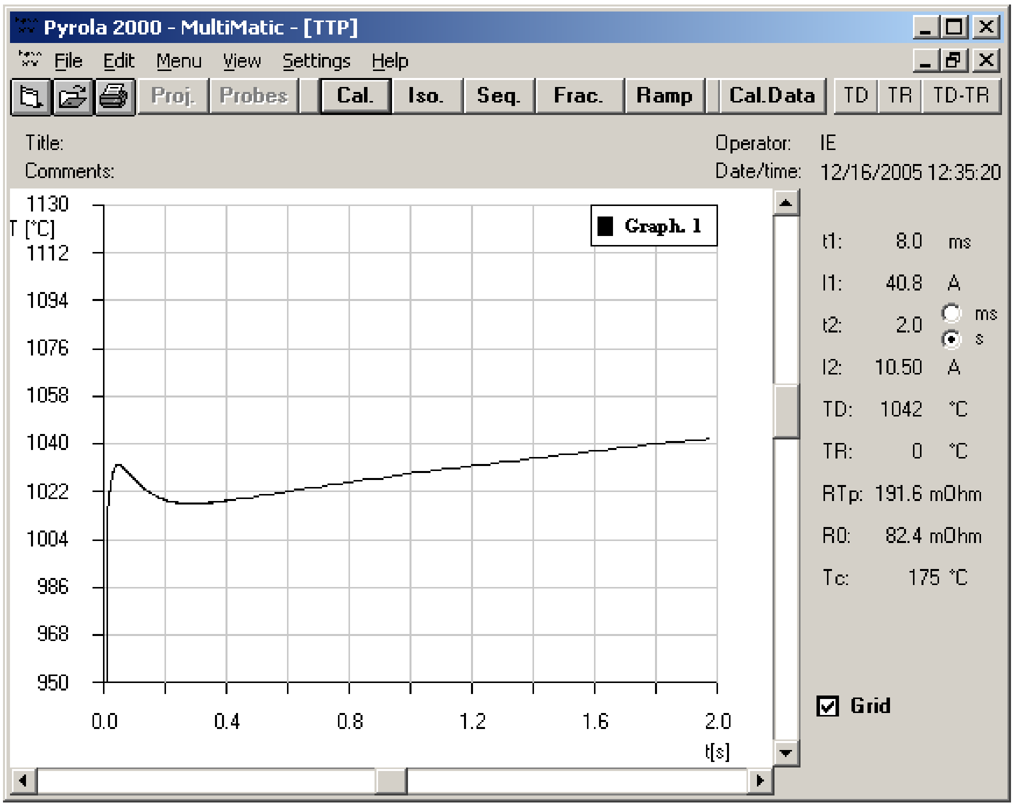
1.2 Iterative Approach to 1000℃.

Increasing I1 to 41A yields the following result:



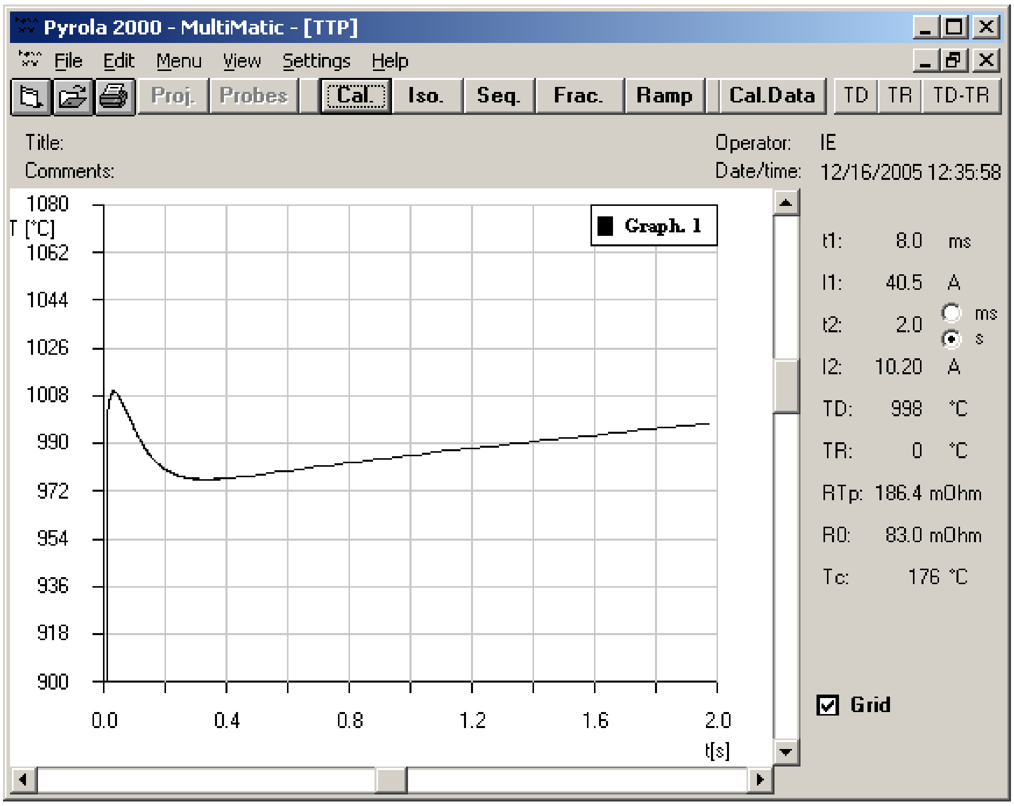
Now I1 is a little high as the initial pulse exceeds 1000℃. I2 was not altered and remains too low.

Decreasing I1 to 40.8A and increasing I2 to 10.50A yields the following result:



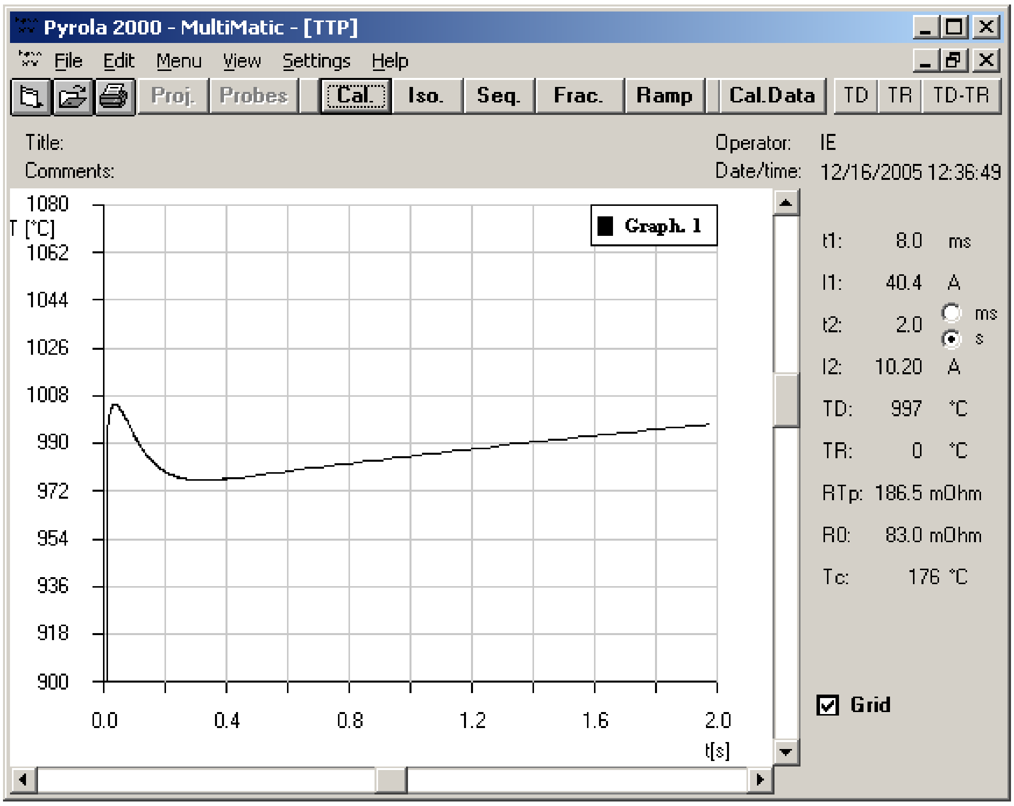
It is seen that I1 is still too high, the initial pulse comfortably exceeding the target temperature, as is I2 since the end temperature now exceeds the required calibration temperature by greater than 40°. The required value for I2 can be assumed to be somewhere between 10.0 and 10.5A.

Reducing I1 to 40.5A and reducing I2 to 10.20A yields the following result:



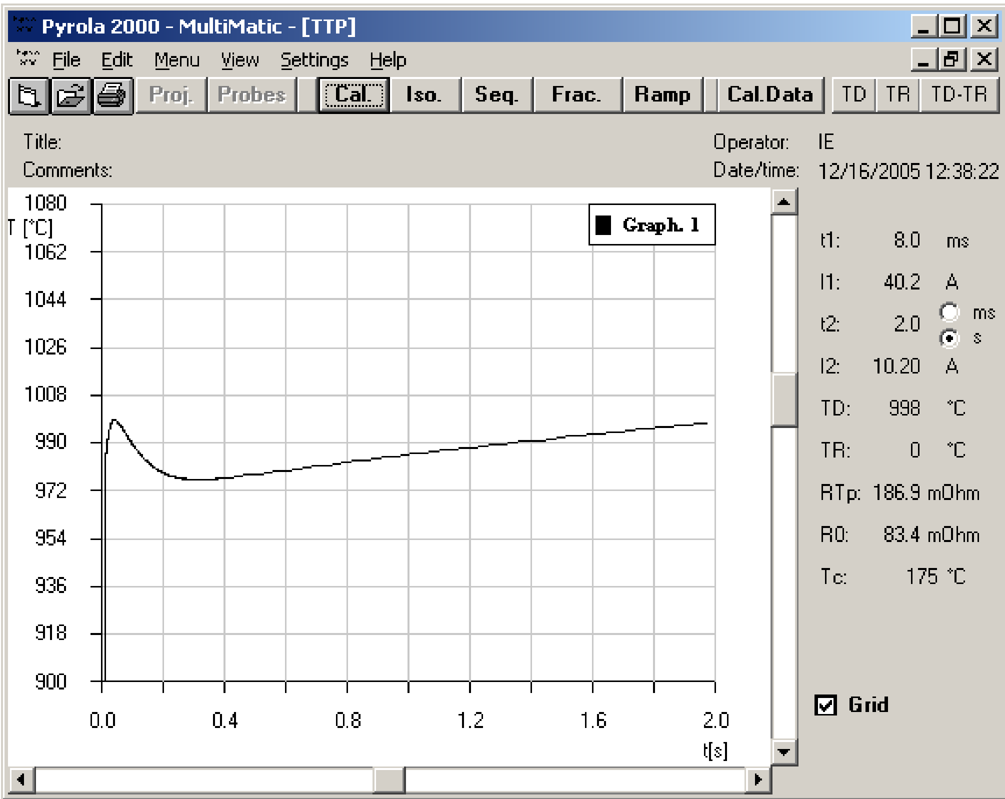
Now the initial pulse overshoot is reduced to approximately 10° and the final temperature is acceptable, indicating that the value used for I2 in this test is possibly correct. Note that, the value of I2 may need adjustment once I1 is correctly set as there is some interaction between the two current settings.

Reducing I1 by 0.1A and leaving I2 unchanged yields the following result:

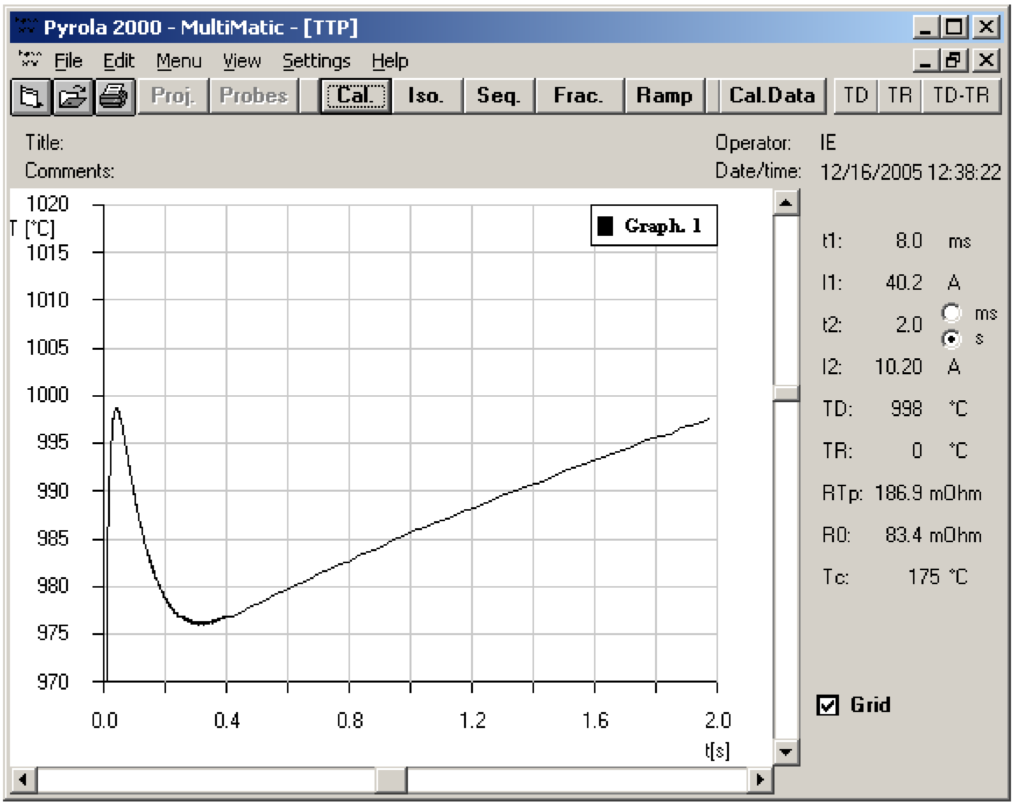


The initial pulse is still a little high and the final temperature is still acceptable.

Further reduction of I1 to 40.2A yields the following result:

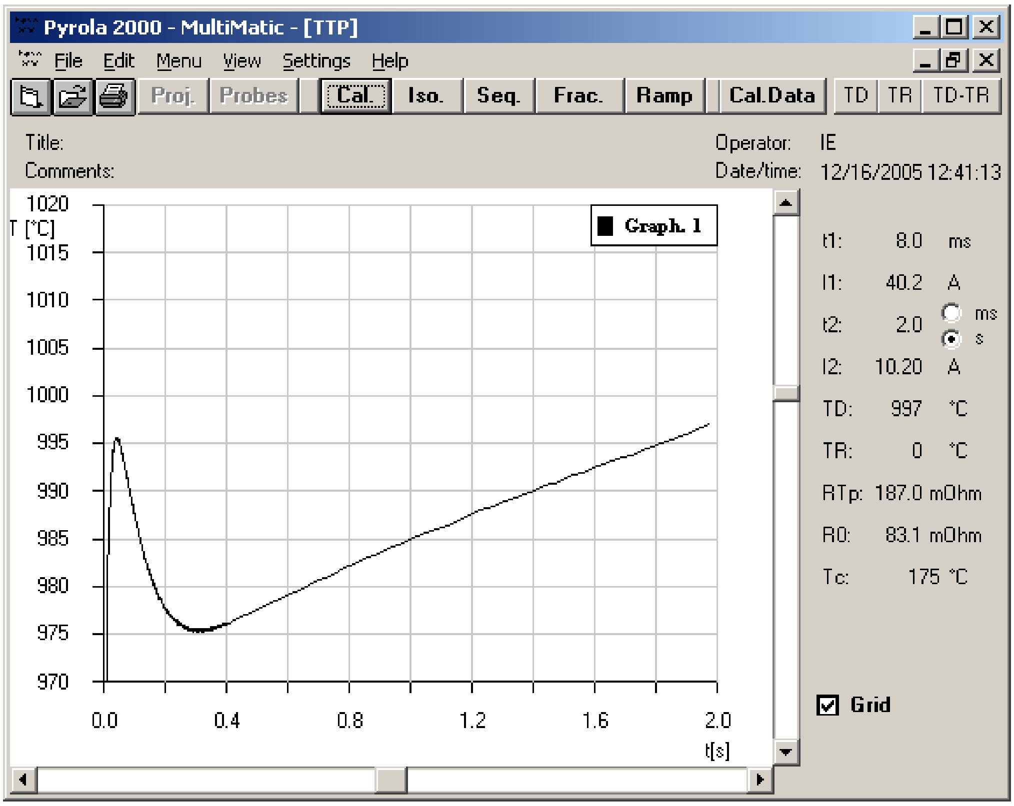


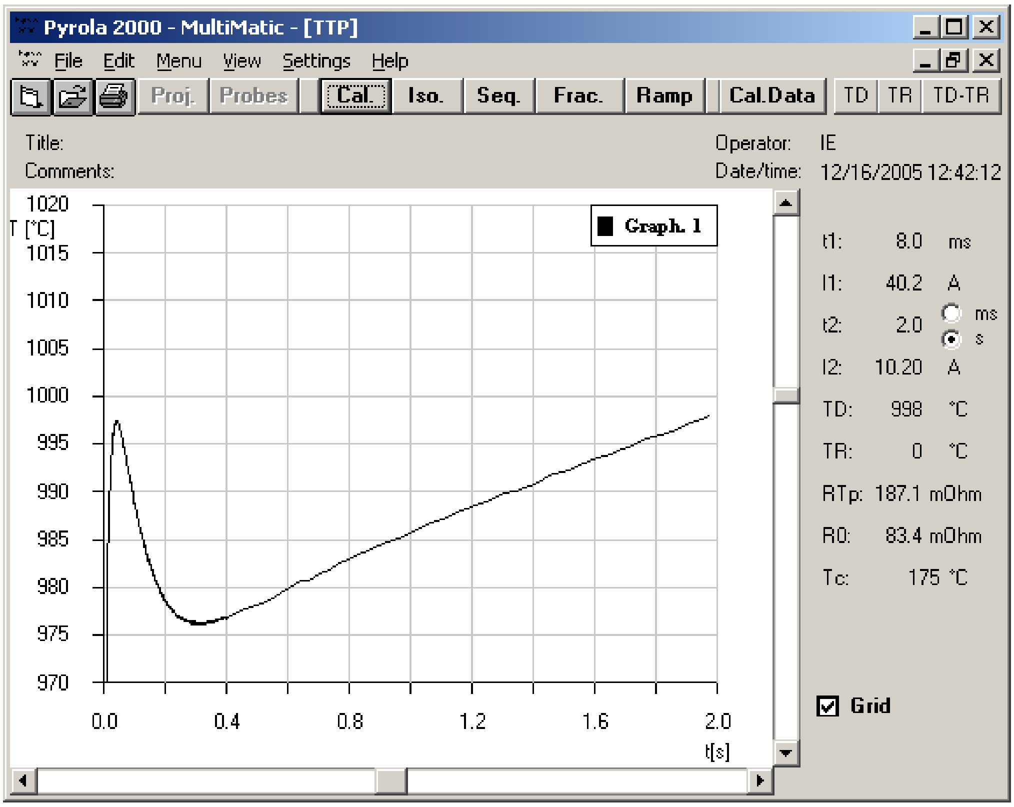
The results obtained now appear acceptable with the initial pulse not exceeding the desired calibration temperature and the final temperature still being close to the desired calibration temperature. Note that, this plot can be expanded in settings, preferences to give any desired combination of x- and y-scales. Adjusting the y-scale to give 50℃ span give the plot shown below:

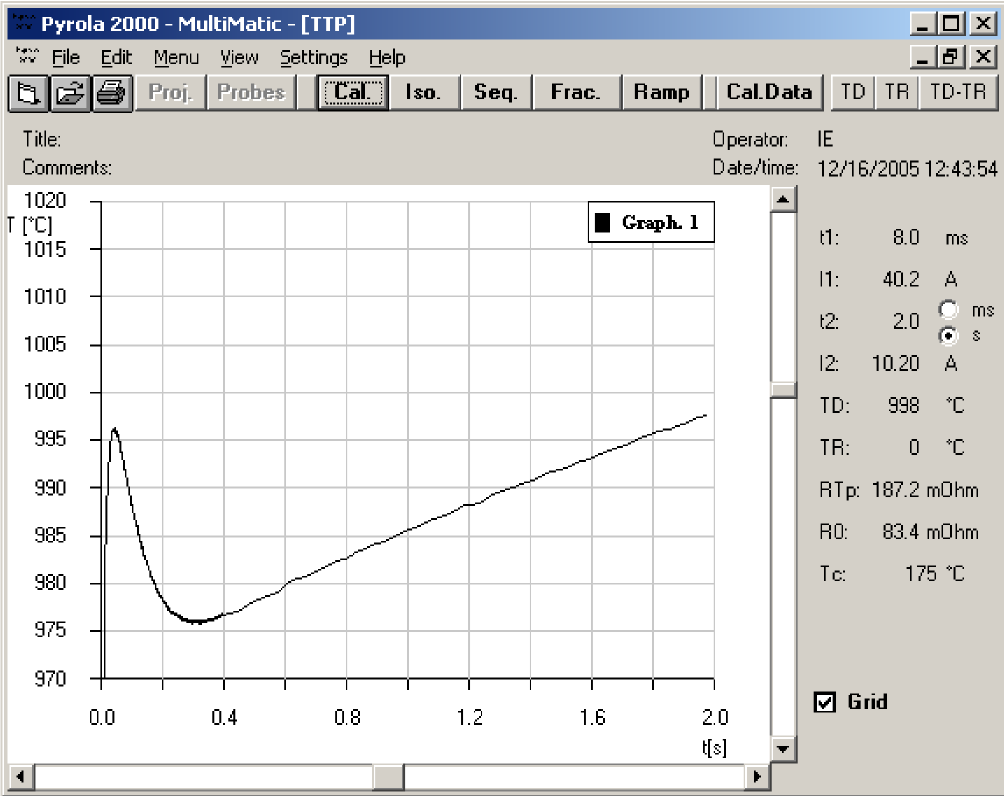


With an expanded y-scale, unwanted temperature excursions above the target temperature are seen easily. The advice given in the manual can be summarised as the temperature after the first pulse and the temperature at the end of the pyrolysis should be about equal. The final piece of advice is to assess the reproducibility of the settings by running repeat runs. It should be noted that a sufficient amount of time should be allowed between runs to allow the filament to cool to the equilibrium starting temperature. This temperature cannot be measured and will depend upon the pyrolysis temperature used, the chamber temperature, the carrier gas and the carrier gas flow. About 60 seconds was typically used. It is possible to monitor the progress of the equilibration when at least two TTps have been obtained by studying the R0(act) and the d value in the Calibration of filament screen. However at this early stage in the calibration, a certain amount of common sense must be used. The results of several repeats is shown below:

1.3 Check Reproducibility.







These three plots, all at similar y-scale expansions, show that the unit is behaving reproducibly under this set of conditions.

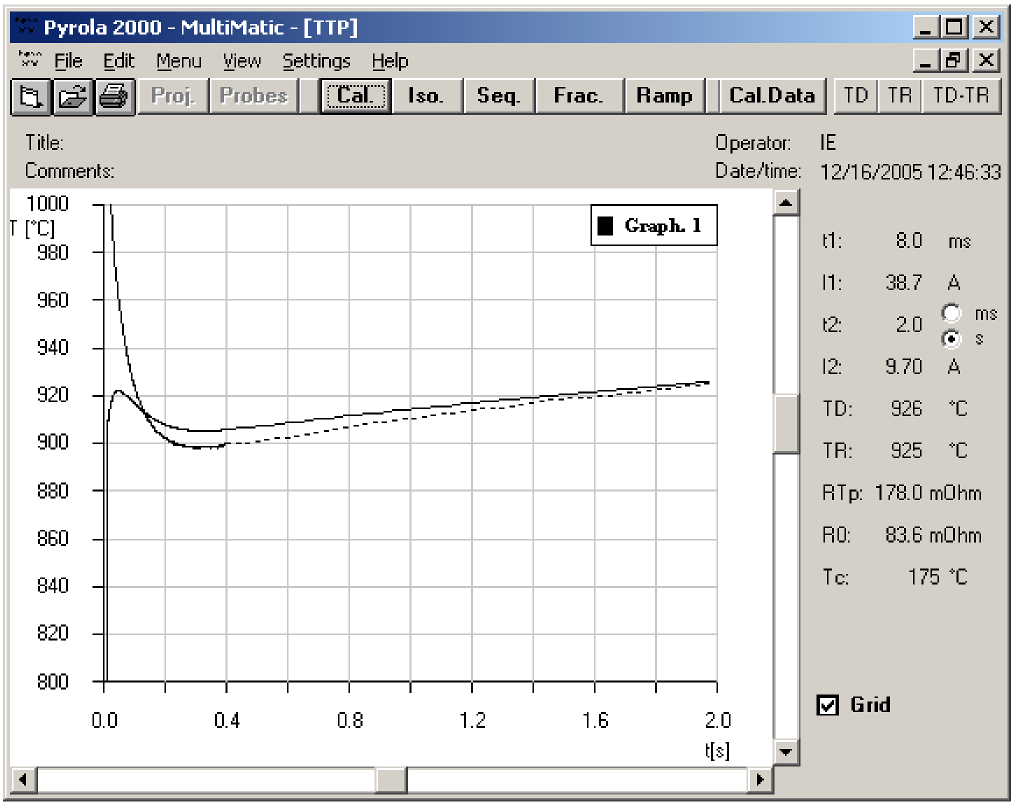
The current parameters must be stored by clicking the Save TTP button in the Calibration of Filament screen before progressing. Note that, the actual temperature obtained as measured by TD will be the calibrated temperature value. Also note that, at this early stage in the calibration, no TR value is presented. This value only appears once a calibration at a second temperature has been performed.

2.0 Calibration at 900C.

It is suggested in the manual that reducing I1 by 1.5A and I2 by 0.5A will reduce the filament temperature by 100℃. use these values as a rule of thumb only.

2.1 Initial Results.

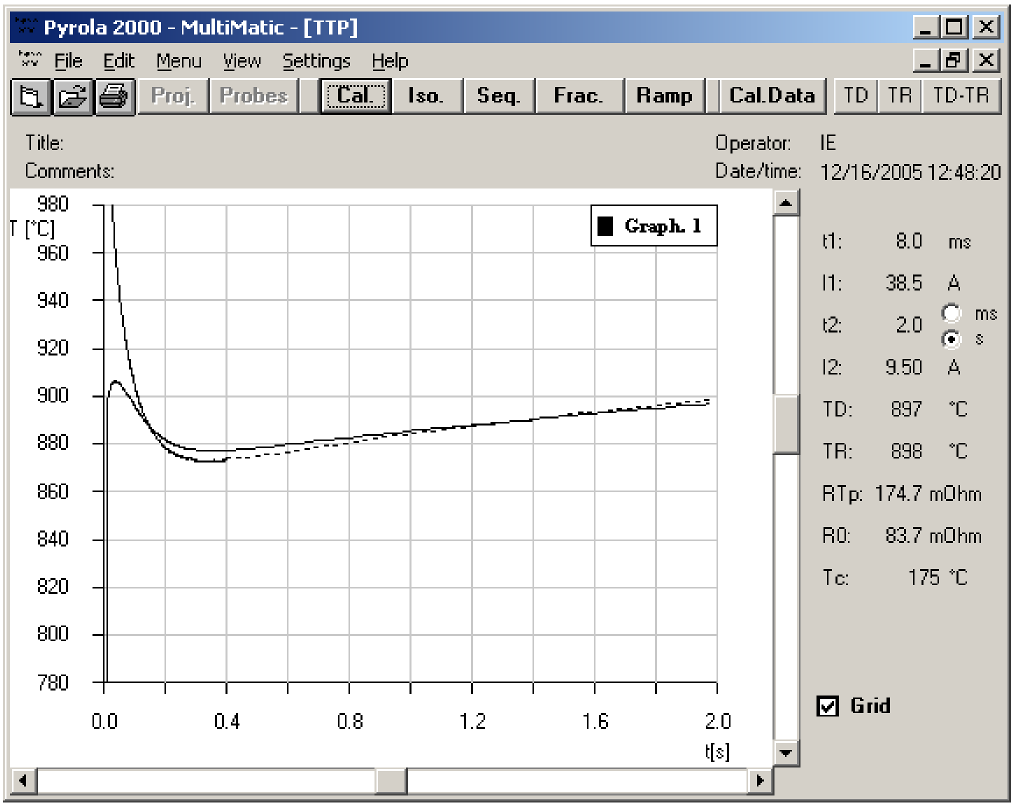
The result below shows the plot obtained by reducing the I1 and I2 values appropriate for calibration at 1000℃ by the values specified in the manual:



This result shows that both I1 and I2 are too high as seen by both the initial pulse and final temperature comfortably exceeding the desired value of 900℃. Note that, having started to calibrate at a second temperature, the TR value now indicates a sensible temperature. In addition, note that, at temperature in excess of 550℃, the temperature performance during calibration is assessed using the solid line TD plot and the dashed line TR plot is ignored.

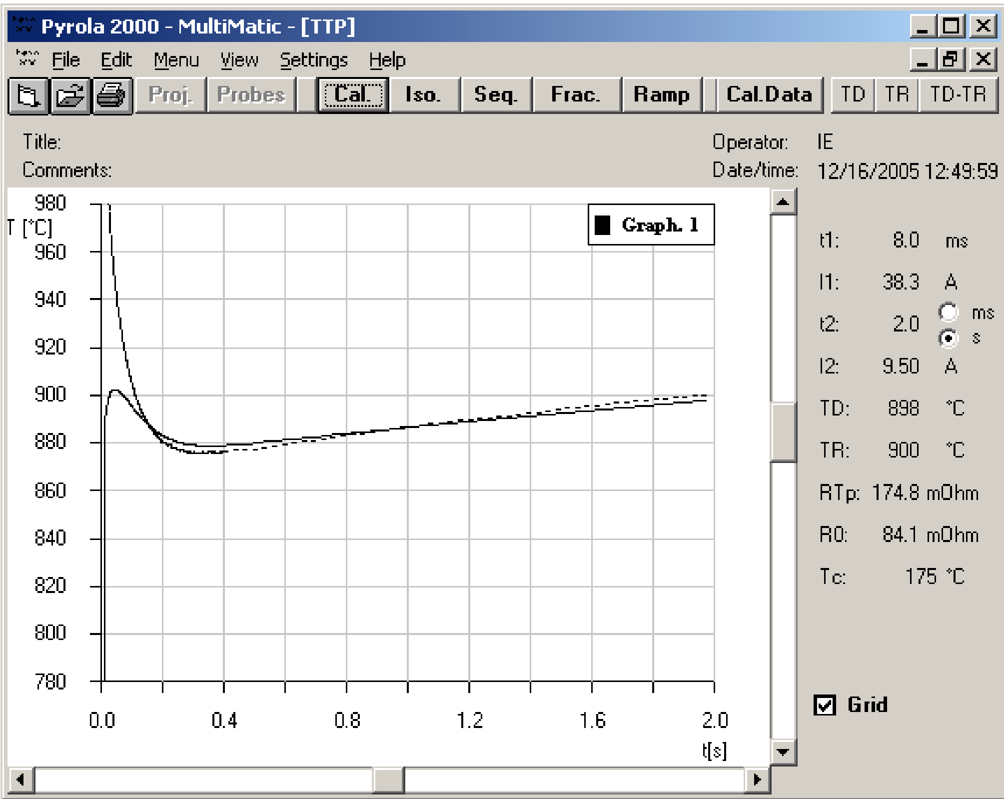
2.2 Iterative Approach to 900°C.

Both I1 and I2 were reduced by 0.2A. The result is shown below:



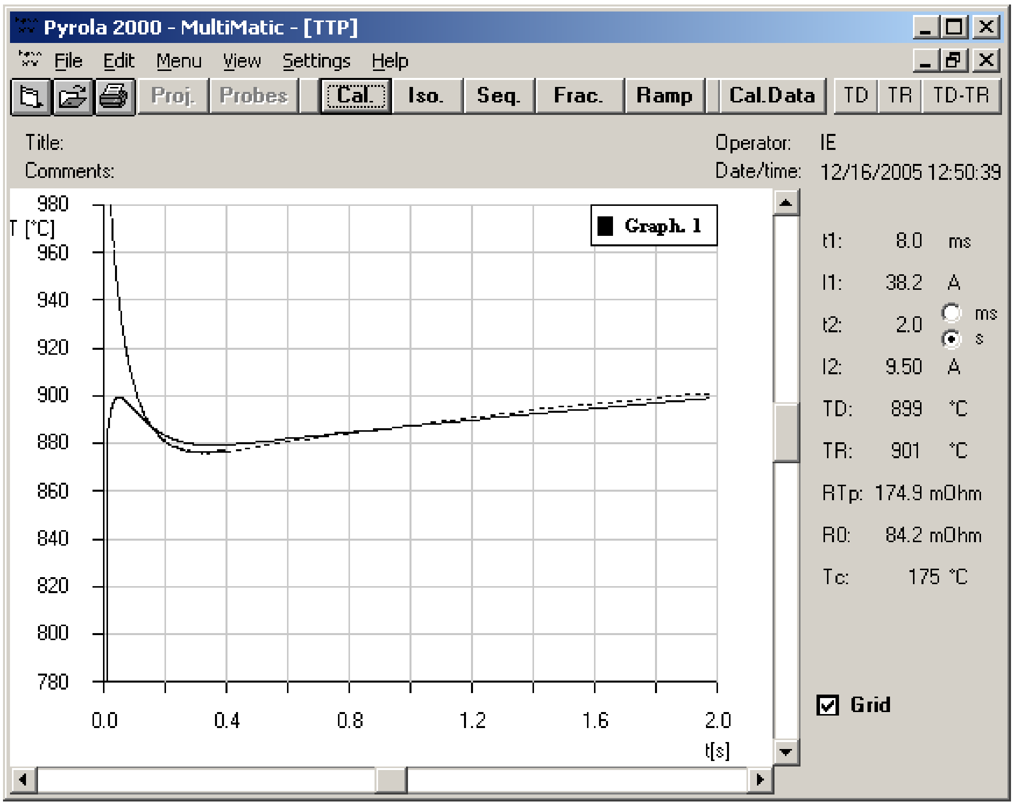
I1 is still too high but I2 appears satisfactory.

A further reduction of I1 to 38.3A yields the following result:



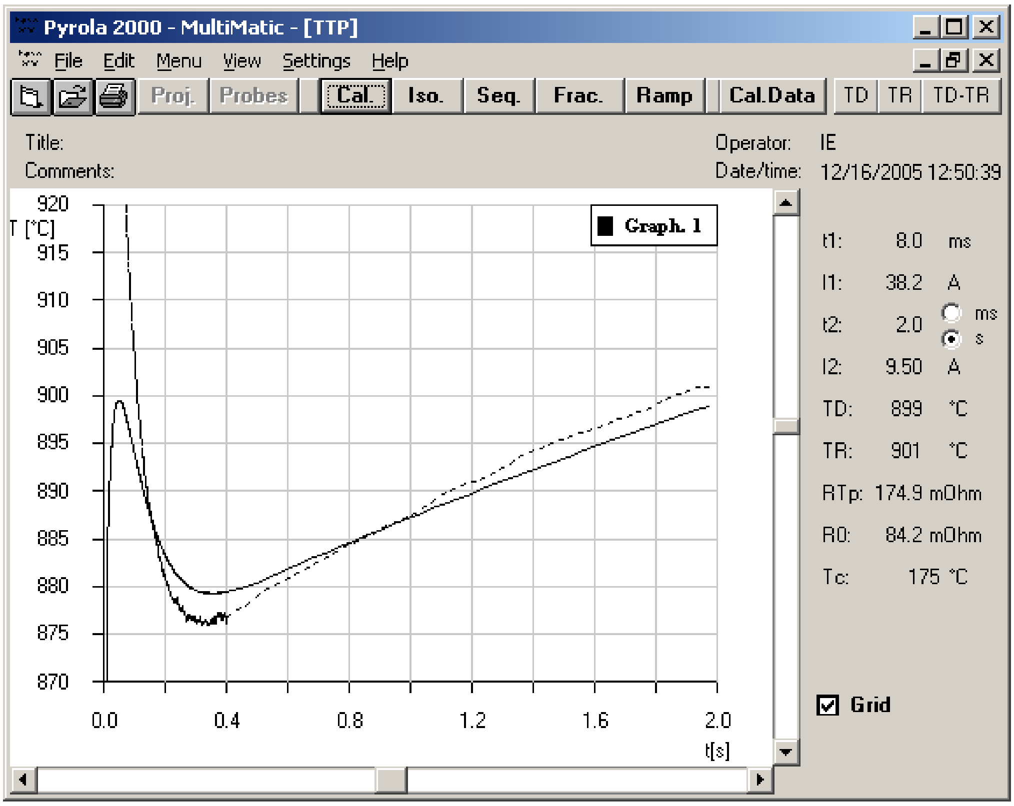
Once again, I1 is still too high and I2 is satisfactory.

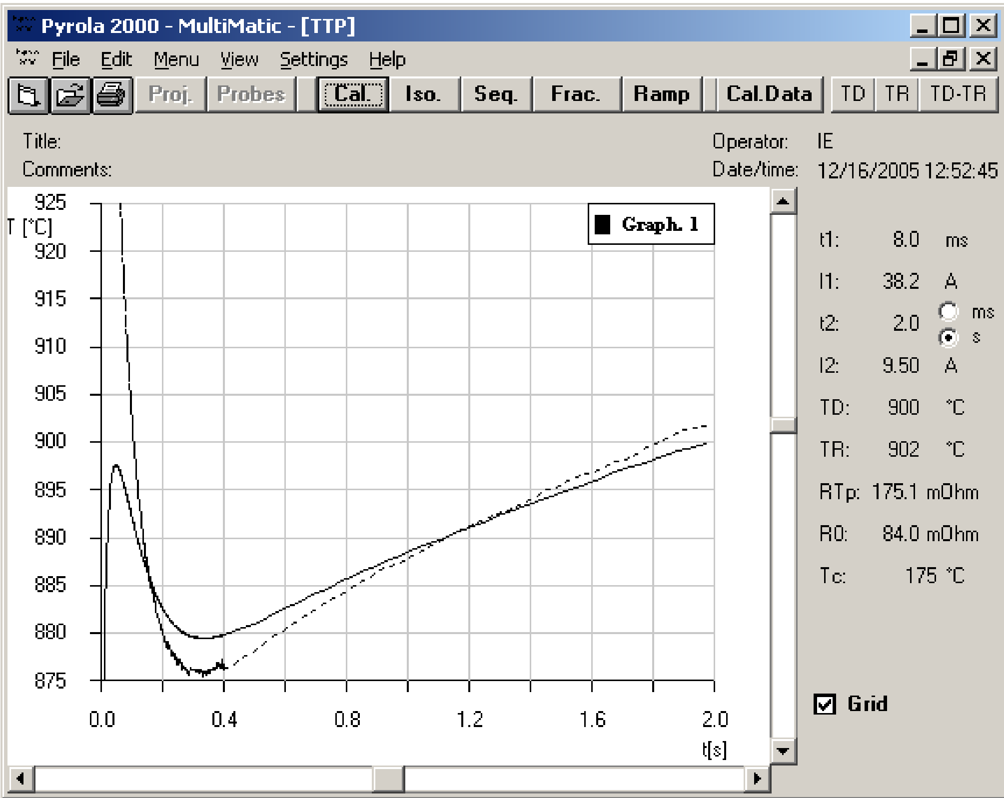
A further reduction of I1 to 38.2A yields the following result:

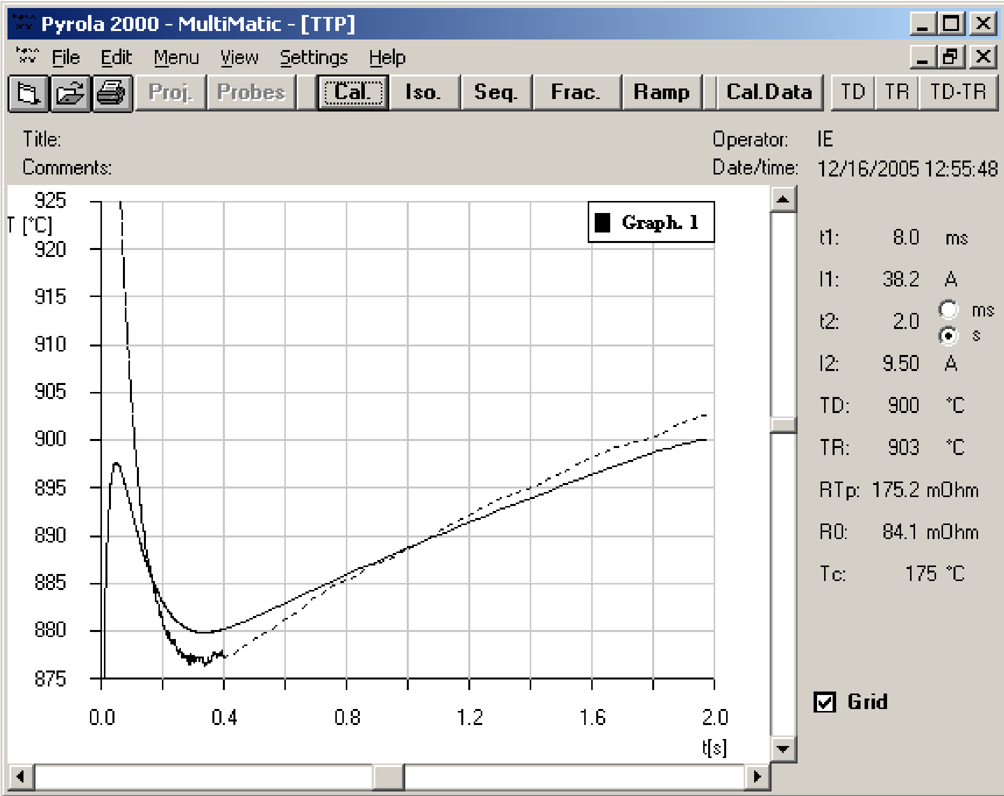


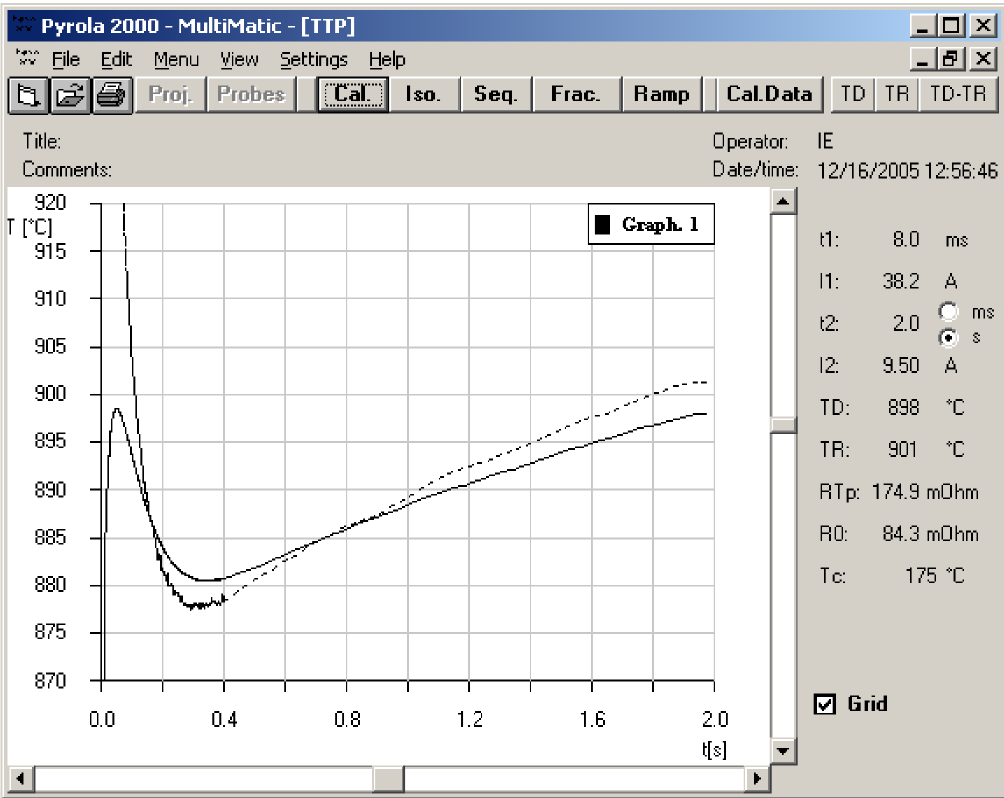
The temperature achieved during the initial pulse and the final temperature is almost equal and the absolute values obtained are very close to the desired temperature. These I1 and I2 values can be considered satisfactory for calibration at 900°C. The TTP for this temperature is saved and the reproducibility examined by repeat runs at increased y-scale as described previously.

2.3 Check Reproducibility.






Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 12:55:48
Graph. 1
T [°C]
925
920
915
910
905
900
895
890
885
880
875
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
t1: 8.0 ms
I1: 38.2 A
t2: 2.0
ms
s
I2: 9.50 A
TD: 900 °C
TR: 903 °C
RTp: 175.2 mOhm
R0: 84.1 mOhm
Tc: 175 °C
Grid


Pyrola 2000 - MultiMatic - [TTP]
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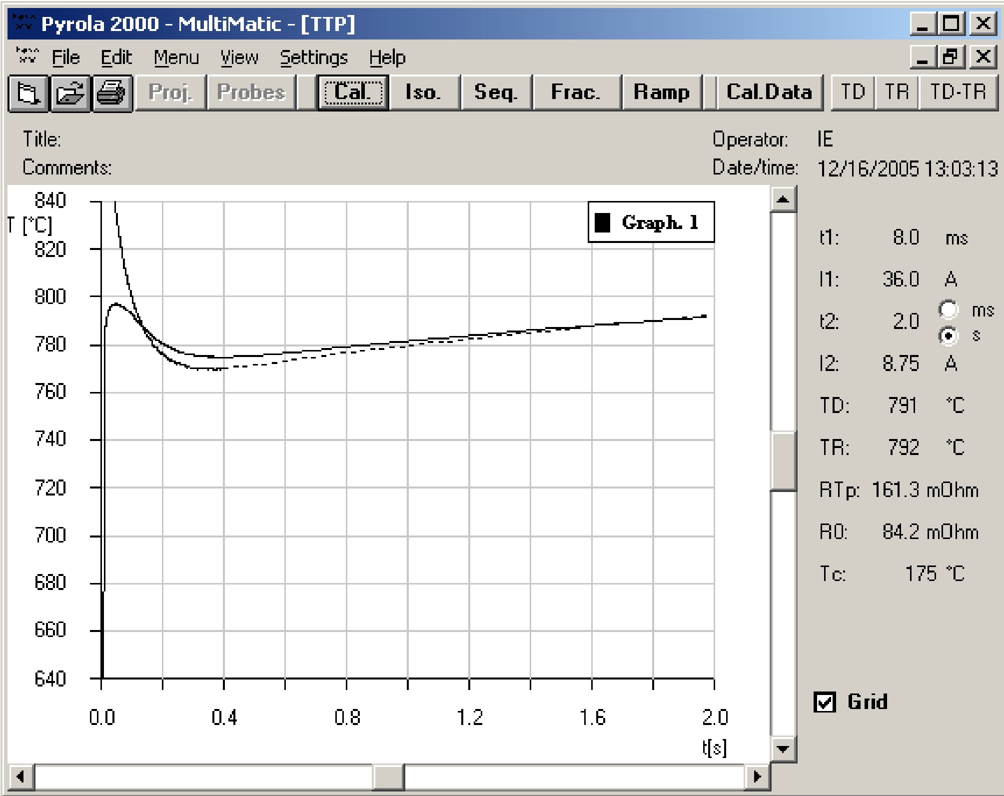
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 12:56:46
Graph. 1
T [°C]
920
915
910
905
900
895
890
885
880
875
870
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
t1: 8.0 ms
I1: 38.2 A
t2: 2.0
ms
s
I2: 9.50 A
TD: 898 °C
TR: 901 °C
RTp: 174.9 mOhm
R0: 84.3 mOhm
Tc: 175 °C
Grid

3.0 Calibration at 750.

3.1 Initial Results.

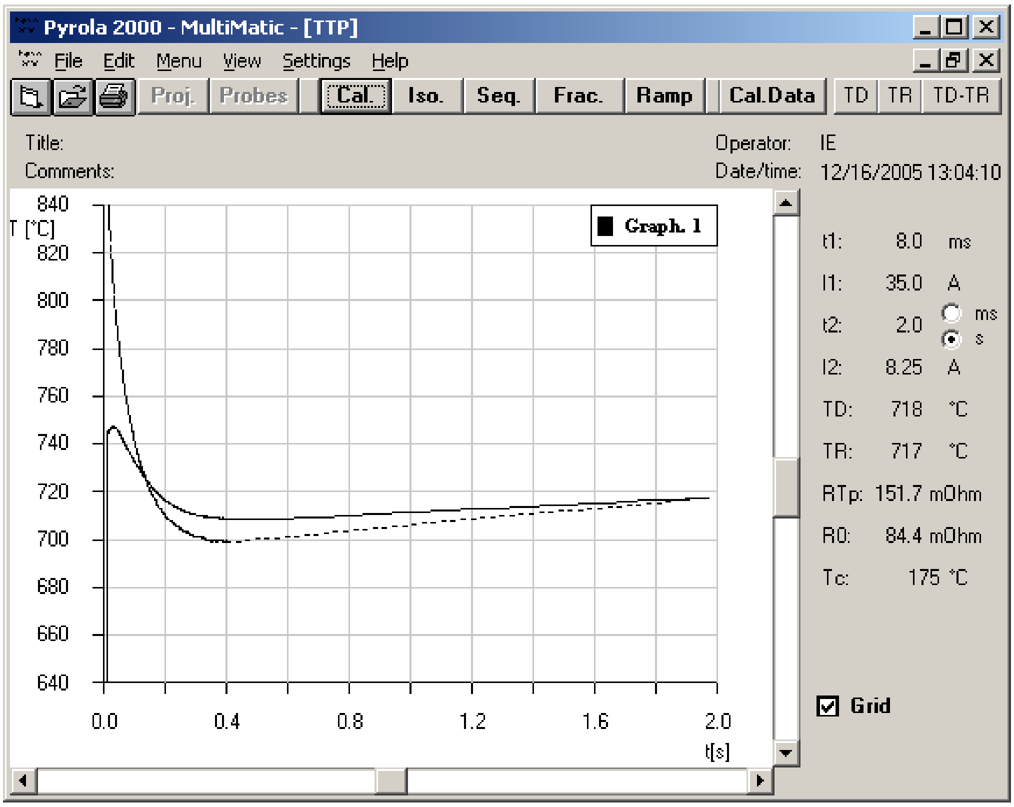
In order to obtain the result, it was assumed that, if reducing I1 and I2 by 1.5A and 0.5A respectively produced a theoretical reduction in calibrated temperature of 100℃, reducing I1 by 2.25A and I2 by 0.75A might produce a reduction of 150℃ in calibrated temperature. The result obtained is shown below:



The magnitude of the reduction in temperature is not as large as was expected. Therefore further reduction in I1 and I2 are required since both the first pulse and the final temperatures are too high.

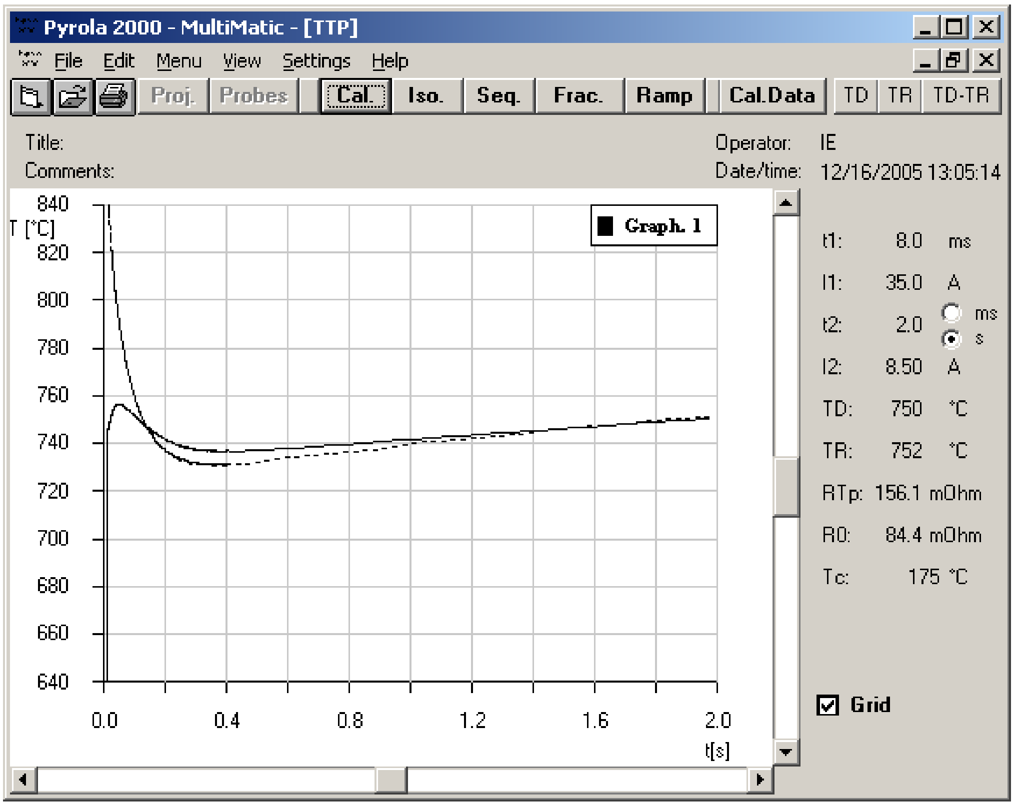
3.2 Iterative Approach to 750℃.

Reducing I1 by 1A and I2 by 0.5A yielded the following result:



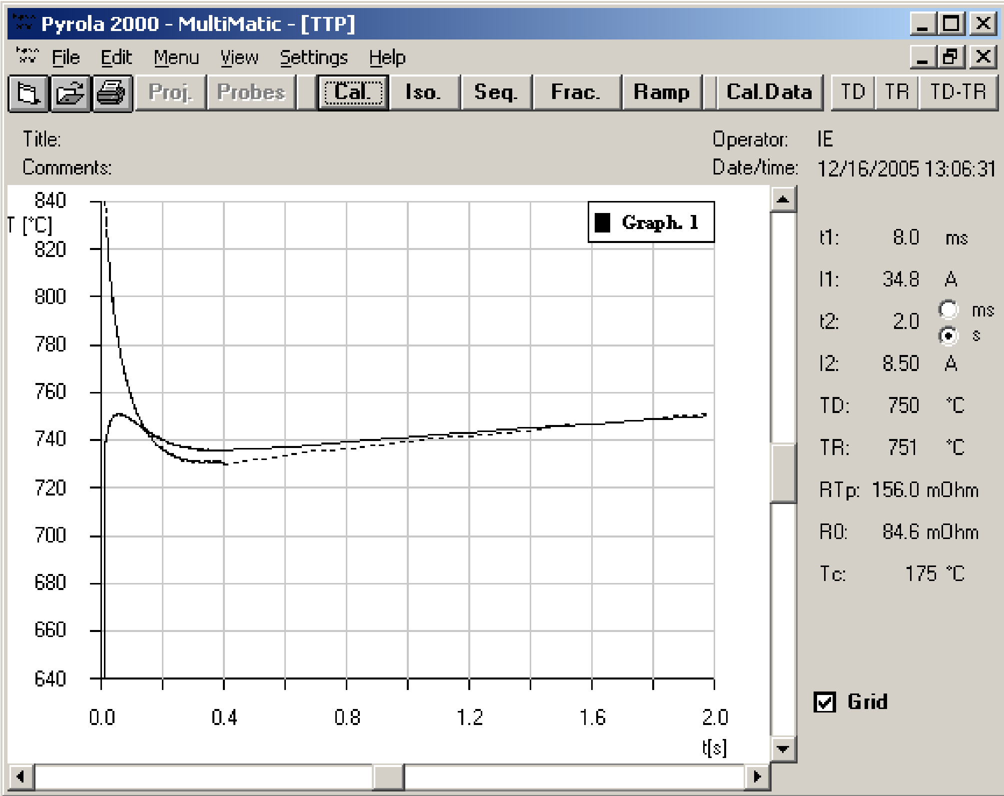
In this instance, an I1 value of 35.0A produces an initial pulse temperature just below 750°; however, an I2 value of 8.25A is too low as the final temperature is significantly below 750℃.

Leaving I1 at 35.0A and increasing I2 to 8.50A yielded the following result:



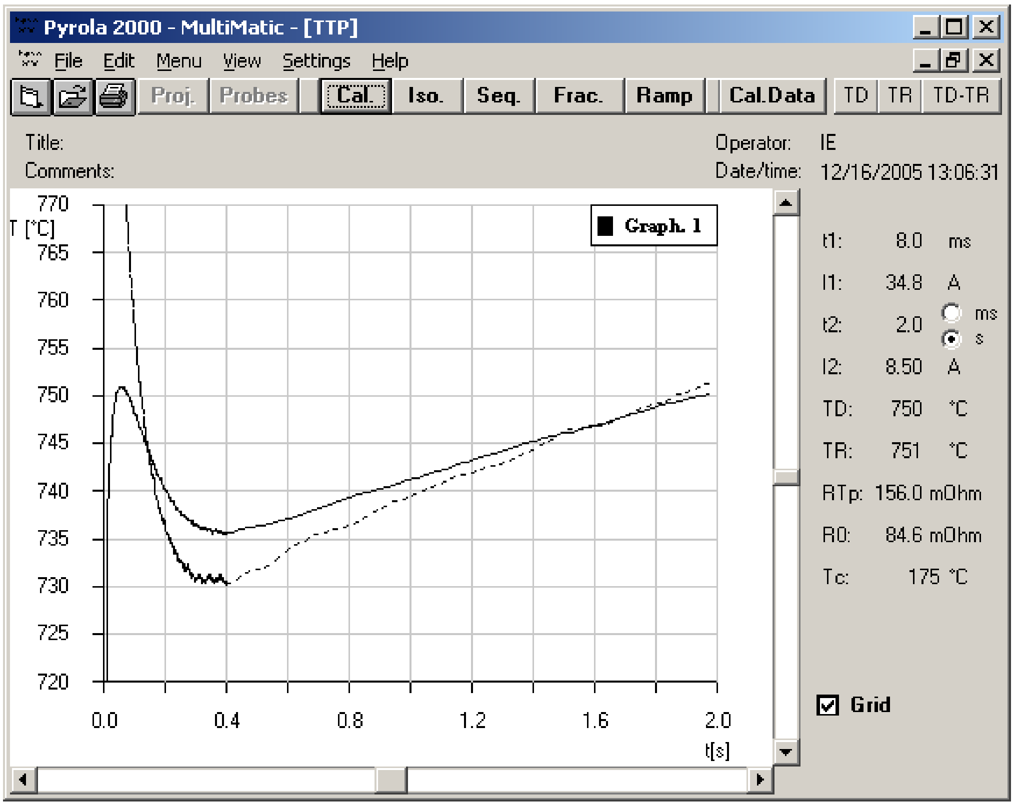
Here, the effect of increasing I2 has brought the final temperature to a satisfactory value but has also increased the temperature obtained during the overshoot from the first pulse such that this temperature almost reaches 760℃.

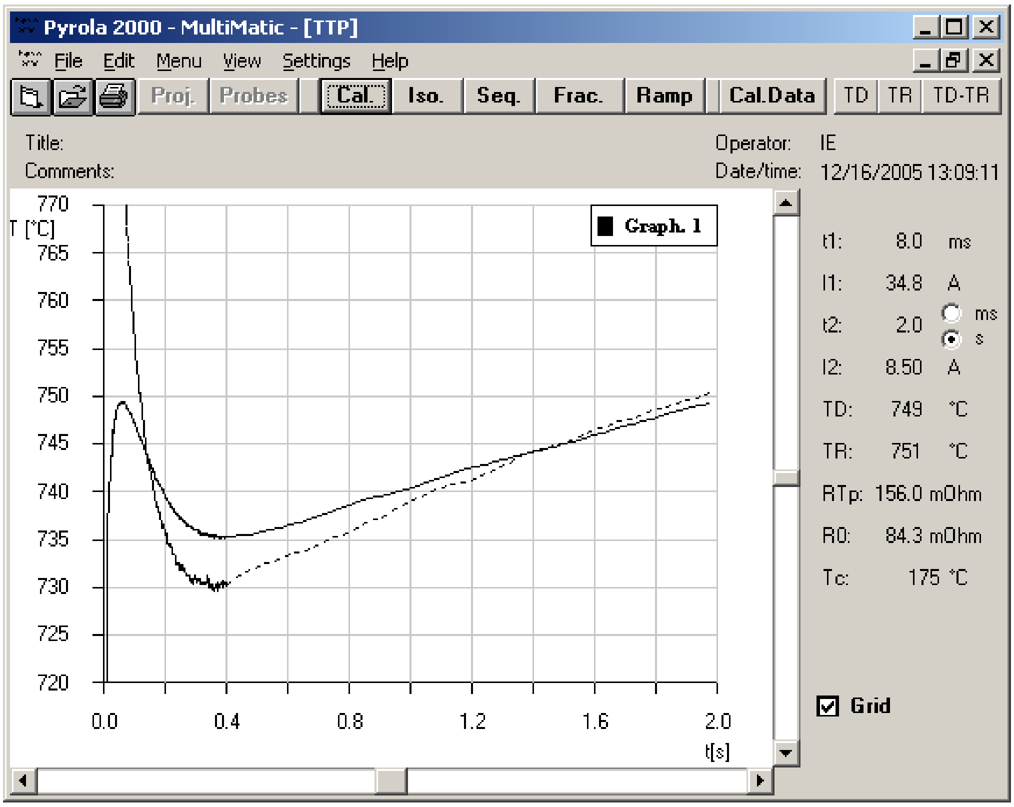
To reduce the initial overshoot, I1 is reduced while maintaining I2 at the previous value. The following result was obtained:

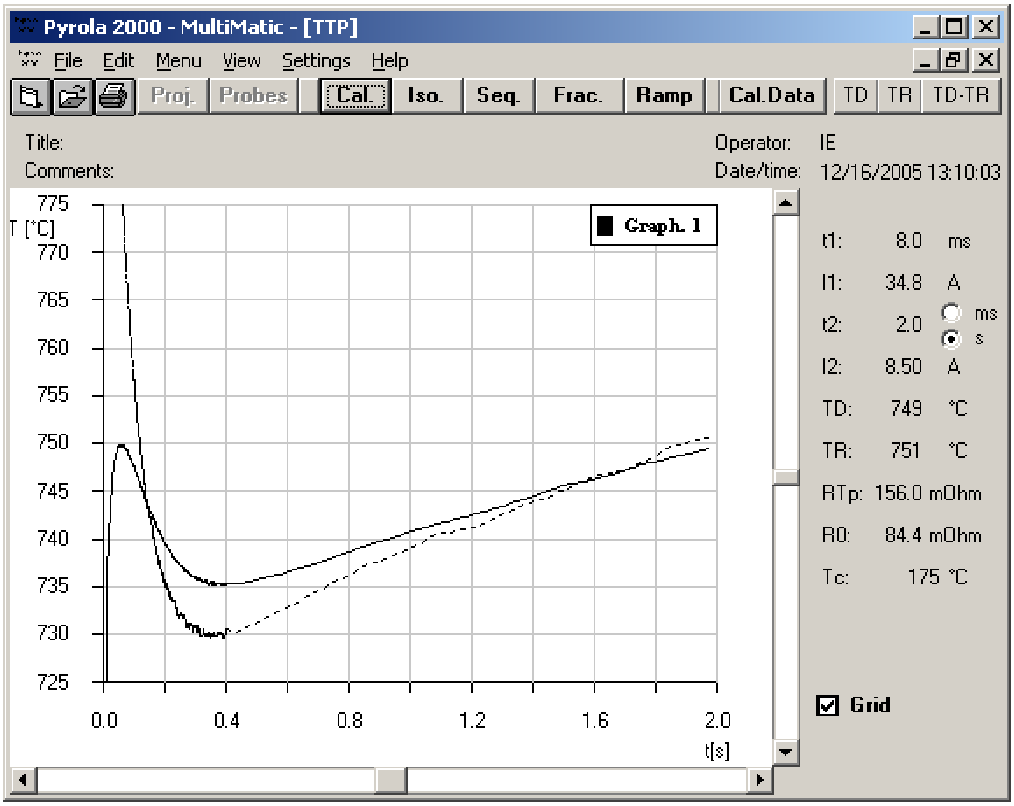


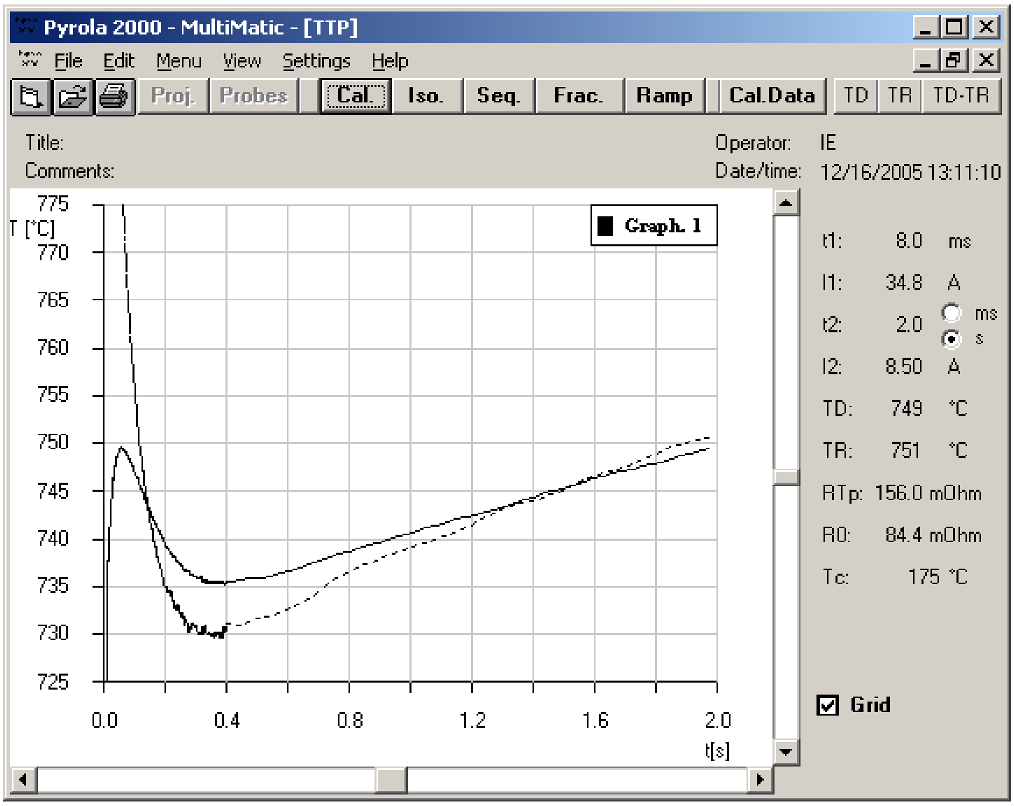
With these settings of I1 and I2, satisfactory performance is obtained at a calibration temperature of 750°C. As before, the TTP is saved and the reproducibility examined at increased y-scale expansion.

3.3 Check Reproducibility.





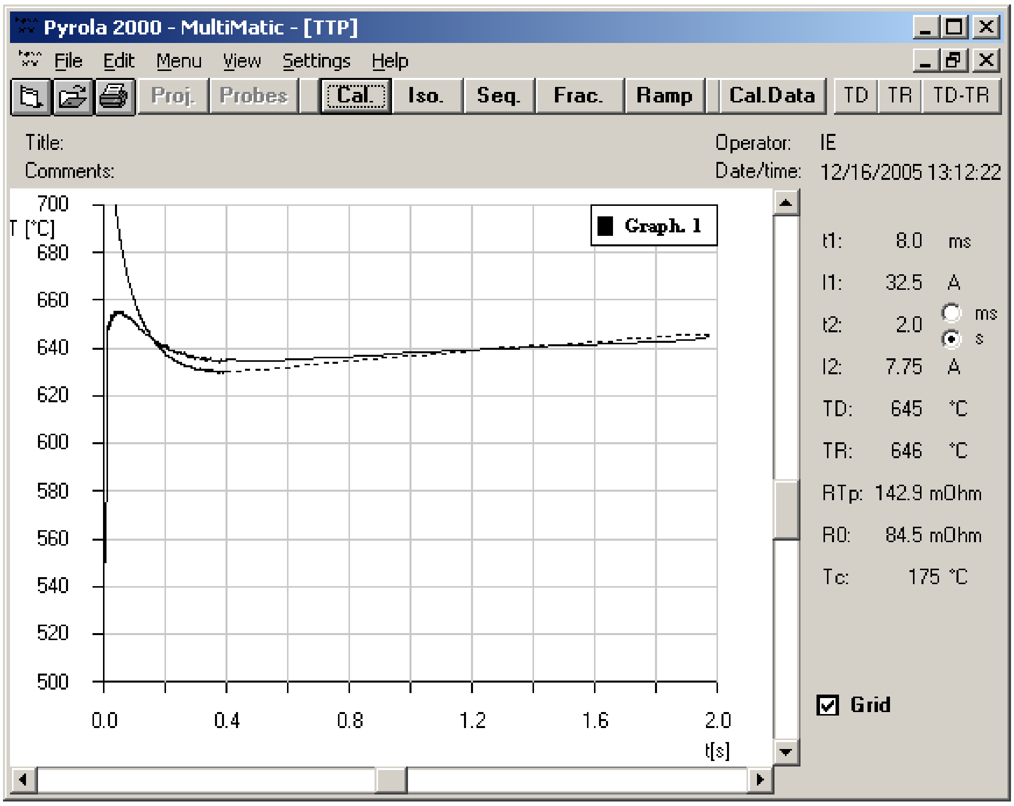

Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 13:10:03
T [°C]
775
770
765
760
755
750
745
740
735
730
725
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
Graph. 1
t1: 8.0 ms
I1: 34.8 A
t2: 2.0 ms s
I2: 8.50 A
TD: 749 °C
TR: 751 °C
RTp: 156.0 mOhm
R0: 84.4 mOhm
Tc: 175 °C
Grid


Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 13:11:10
T [°C]
775
770
765
760
755
750
745
740
735
730
725
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
Graph. 1
t1: 8.0 ms
I1: 34.8 A
t2: 2.0 ms s
I2: 8.50 A
TD: 749 °C
TR: 751 °C
RTp: 156.0 mOhm
R0: 84.4 mOhm
Tc: 175 °C
Grid

4.0 Calibration at 600°C.

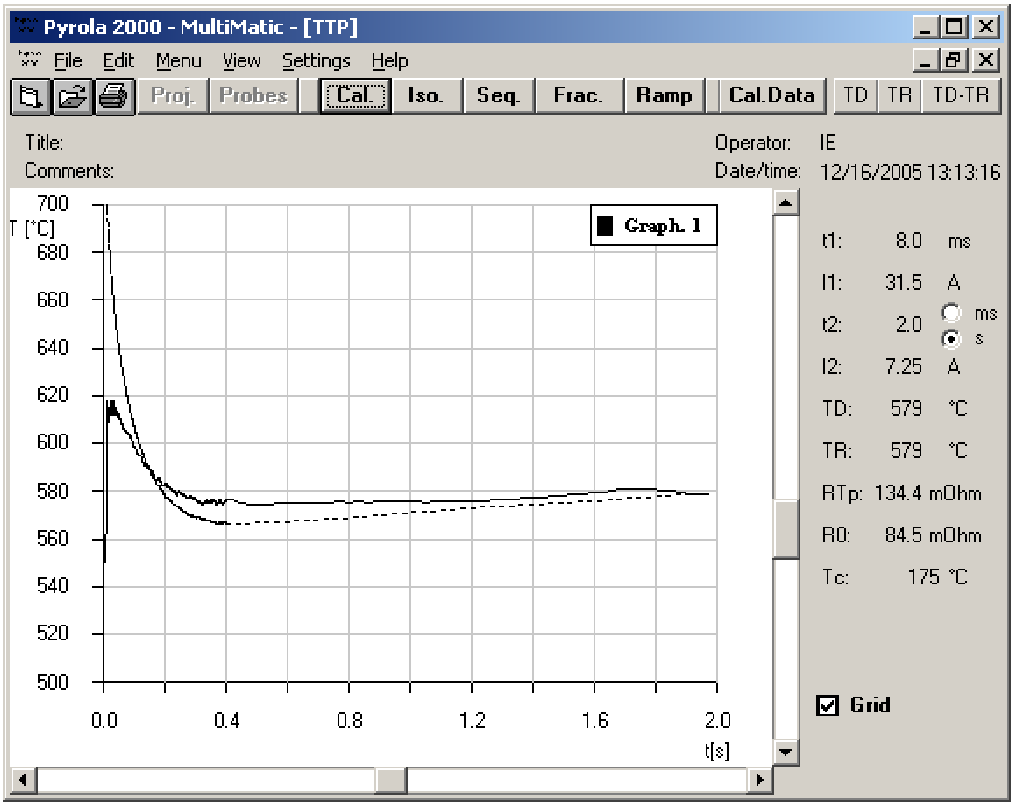
4.1 Initial Results.

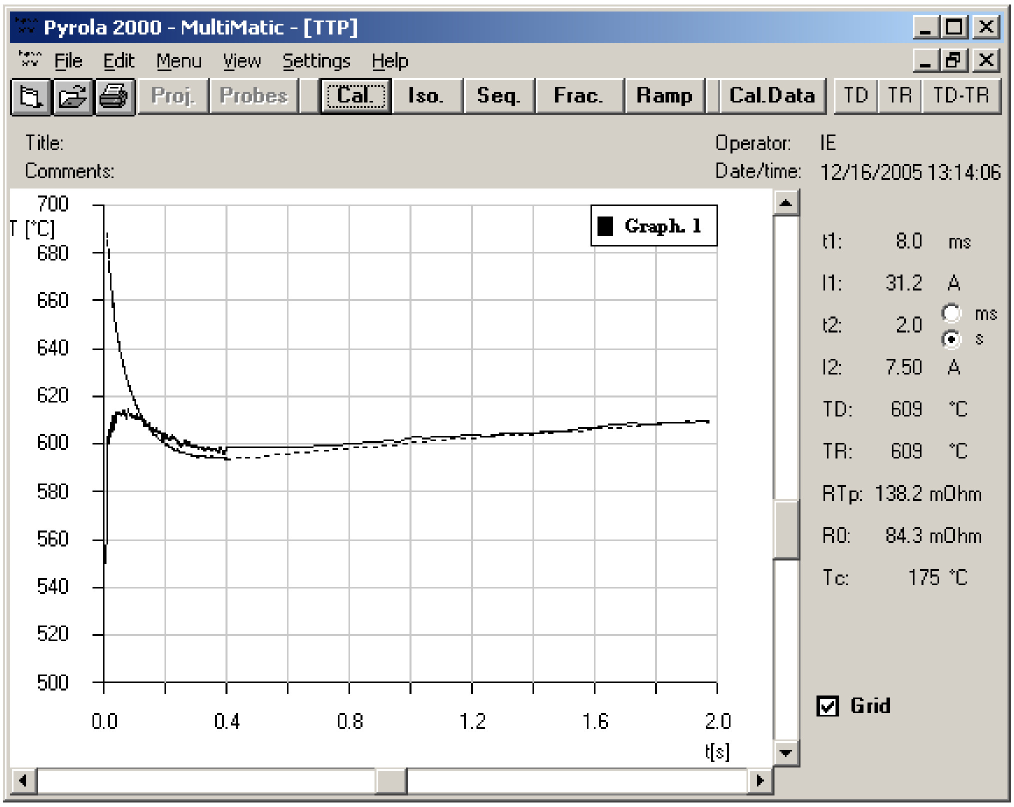
At a calibration temperature of 600°C, the TD plot can still be used. After appropriate reductions in I1 and I2, the following result was obtained:

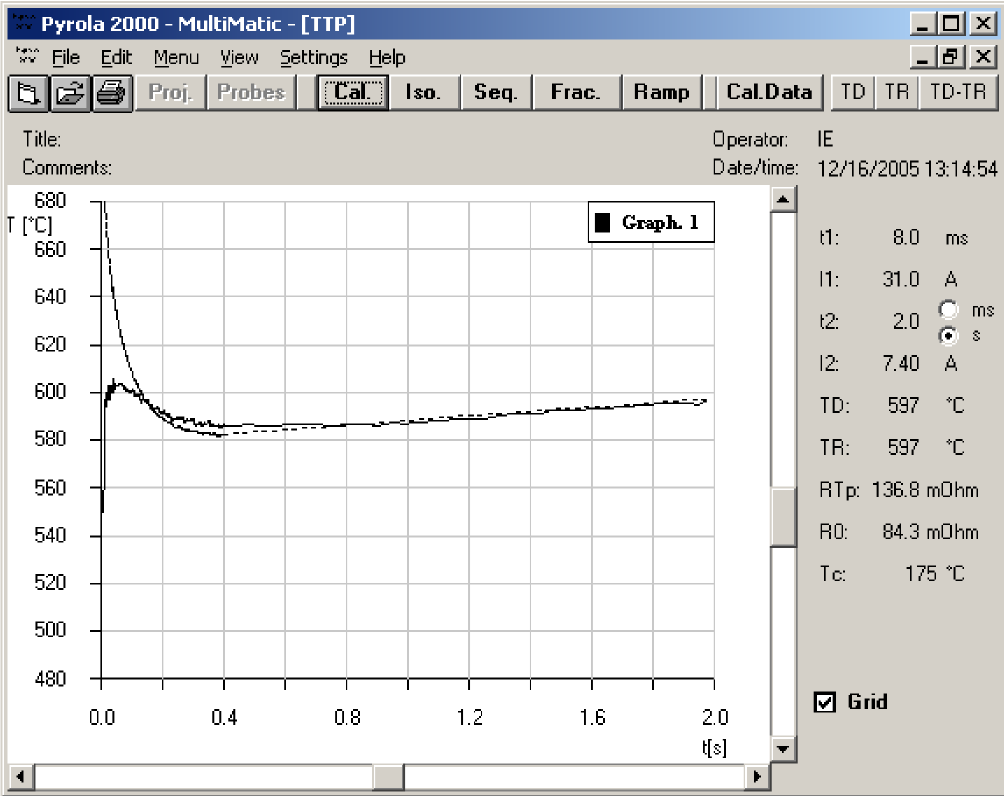


4.2 Iterative Approach to 600℃.

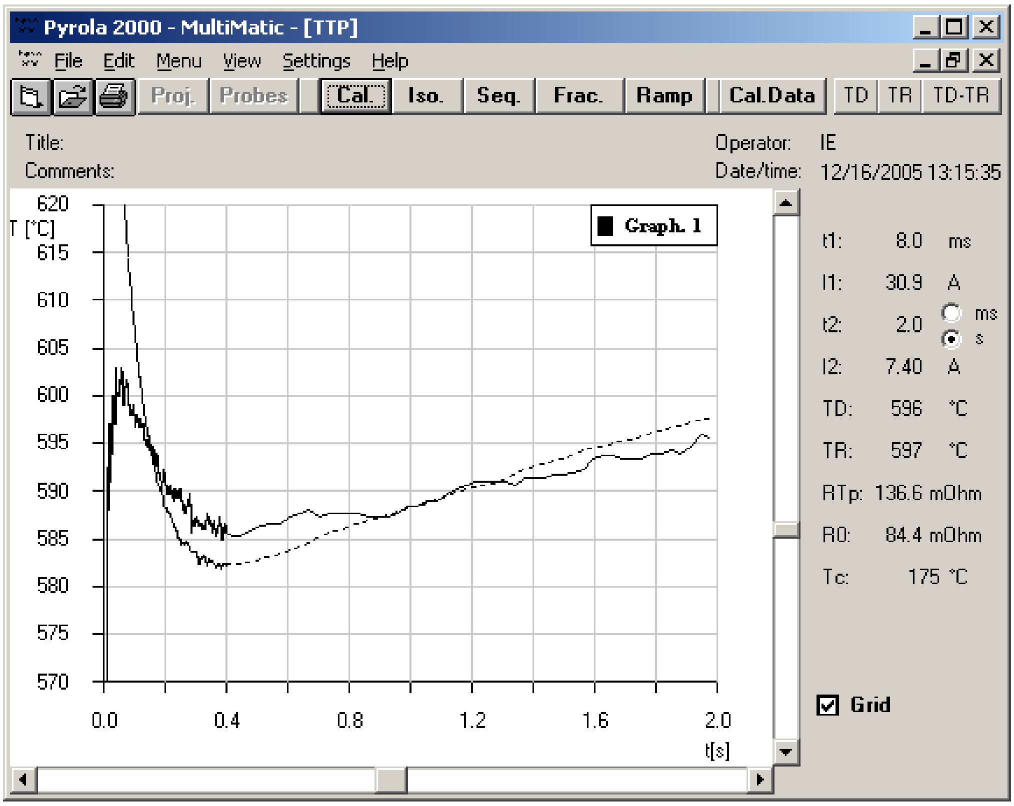
In the manner previously described, the values of I1 and I2 were adjusted until satisfactory performance was obtained at the calibration temperature.

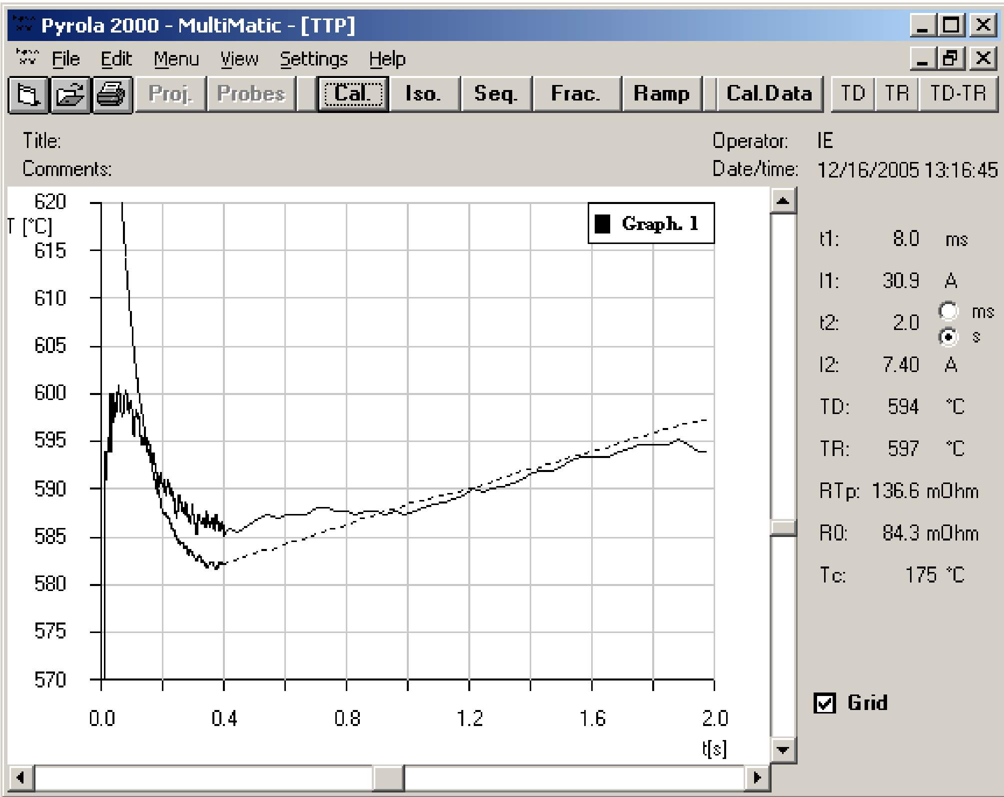


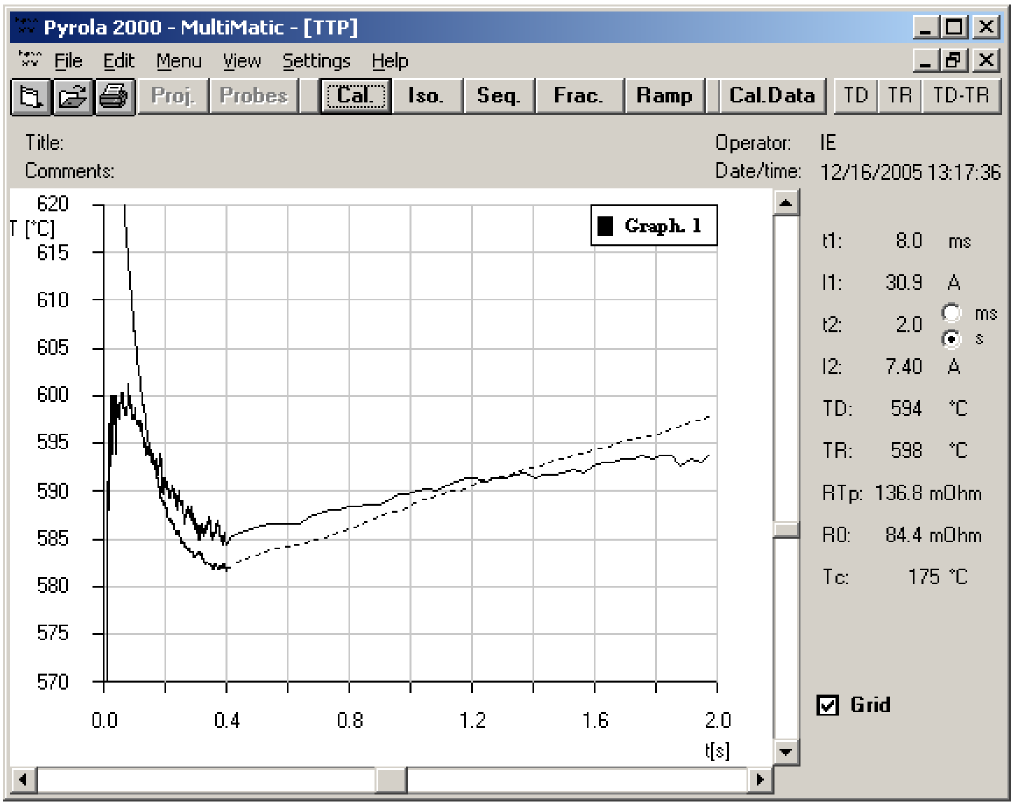


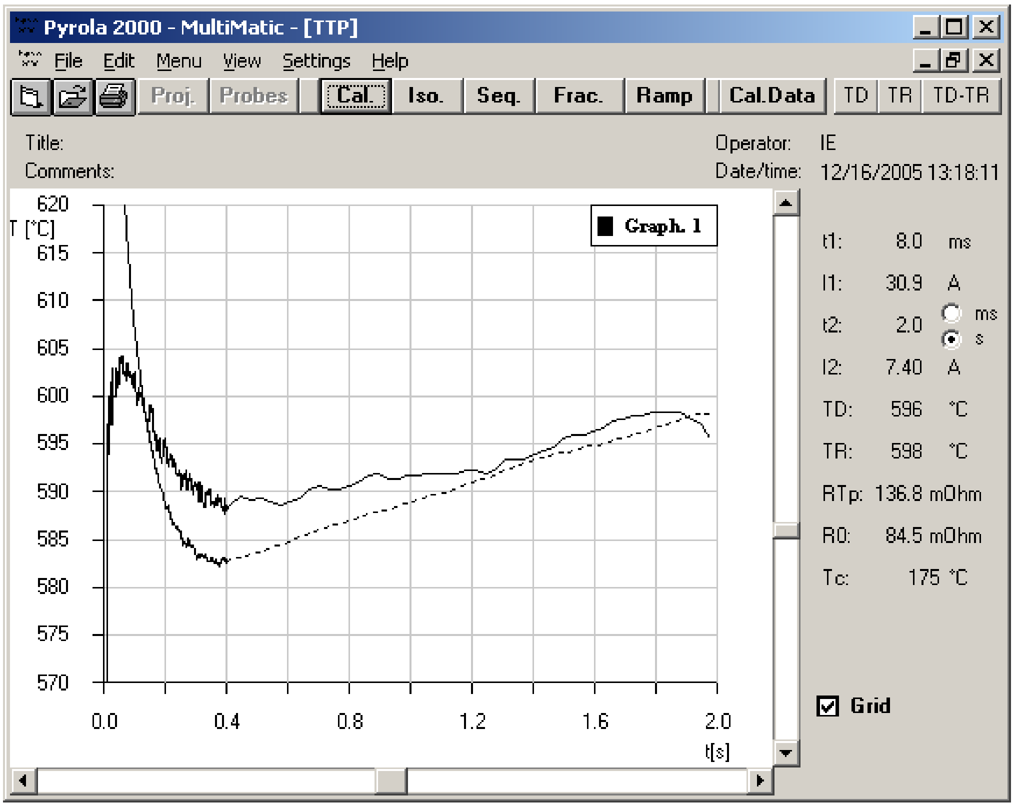

Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 13:14:54
T [°C]
680
660
640
620
600
580
560
540
520
500
480
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
Graph. 1
t1: 8.0 ms
I1: 31.0 A
t2: 2.0 ms s
I2: 7.40 A
TD: 597 °C
TR: 597 °C
RTp: 136.8 mOhm
R0: 84.3 mOhm
Tc: 175 °C
Grid

4.3 Check Reproducibility.





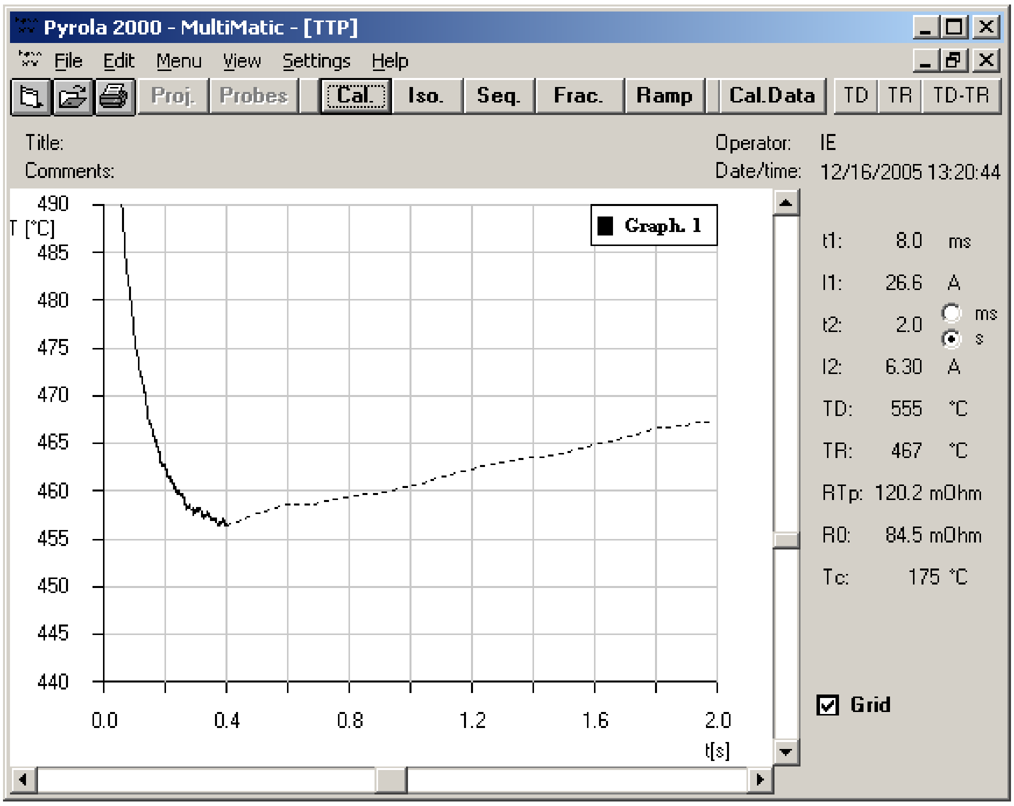

Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 13:17:36
Graph. 1
T [°C]
620
615
610
605
600
595
590
585
580
575
570
0.0 0.4 0.8 1.2 1.6 2.0
t[s]
t1: 8.0 ms
I1: 30.9 A
t2: 2.0 ms s
I2: 7.40 A
TD: 594 °C
TR: 598 °C
RTp: 136.8 mOhm
R0: 84.4 mOhm
Tc: 175 °C
Grid


Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 13:18:11
Graph. 1
T [°C]
620
615
610
605
600
595
590
585
580
575
570
0.0 0.4 0.8 1.2 1.6 2.0
t[s]
t1: 8.0 ms
I1: 30.9 A
t2: 2.0 ms s
I2: 7.40 A
TD: 596 °C
TR: 598 °C
RTp: 136.8 mOhm
R0: 84.5 mOhm
Tc: 175 °C
Grid

5.0 Calibration at 450C.

5.1 Initial Result.

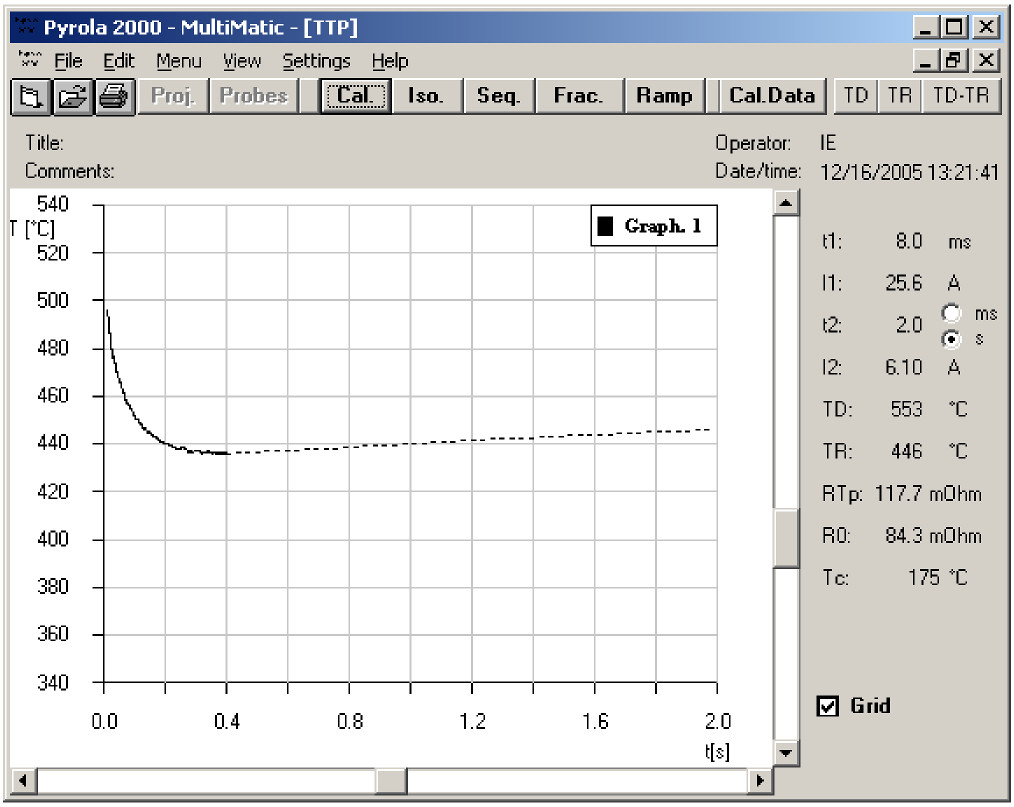
At target temperatures below 550°C, the results should be interpreted on the basis of the trace obtained from TR since the optical temperature sensor is inoperative at temperatures below this value. Note that the trace does not display the TD plot since it is essentially a flat line at about 550°. If the y-axis encompassed this temperature, this plot would be seen but it contains no meaningful data. Setting I1 to 26.6A and I2 to 6.3A yielded the following result:



Both I1 and I2 are too high since the plot is, at all times, above the target temperature of 450°C.

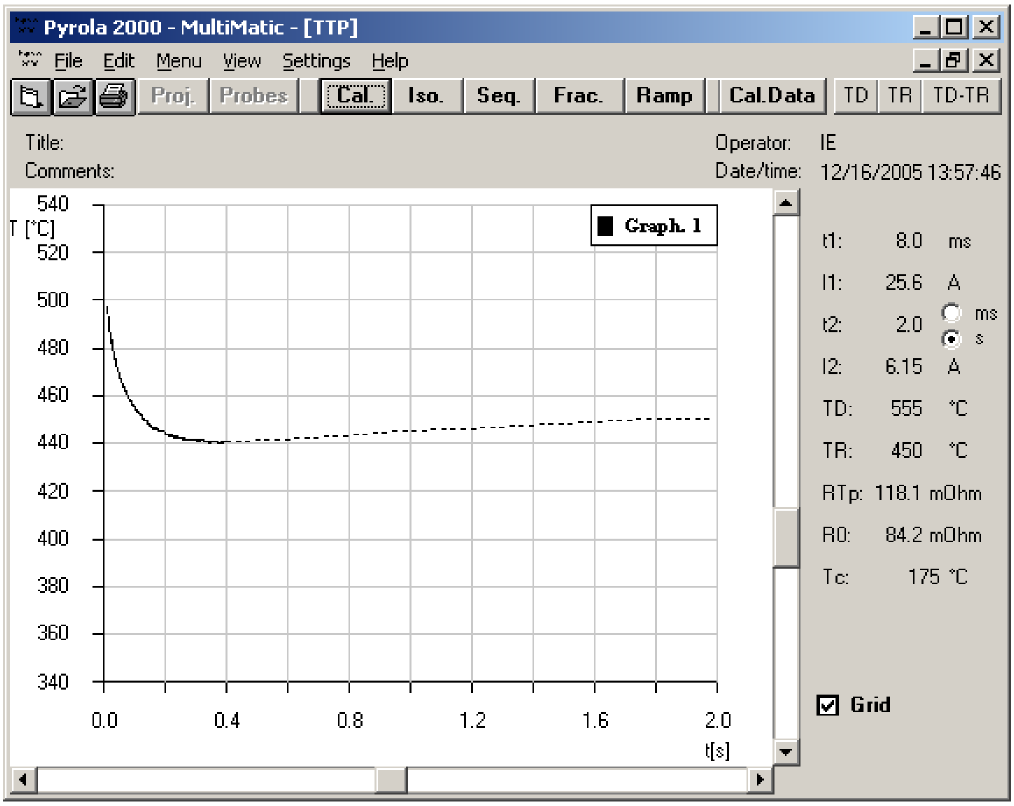
5.2 Iterative Approach to 450℃.

Reducing both I1 and I2 gave the following result:



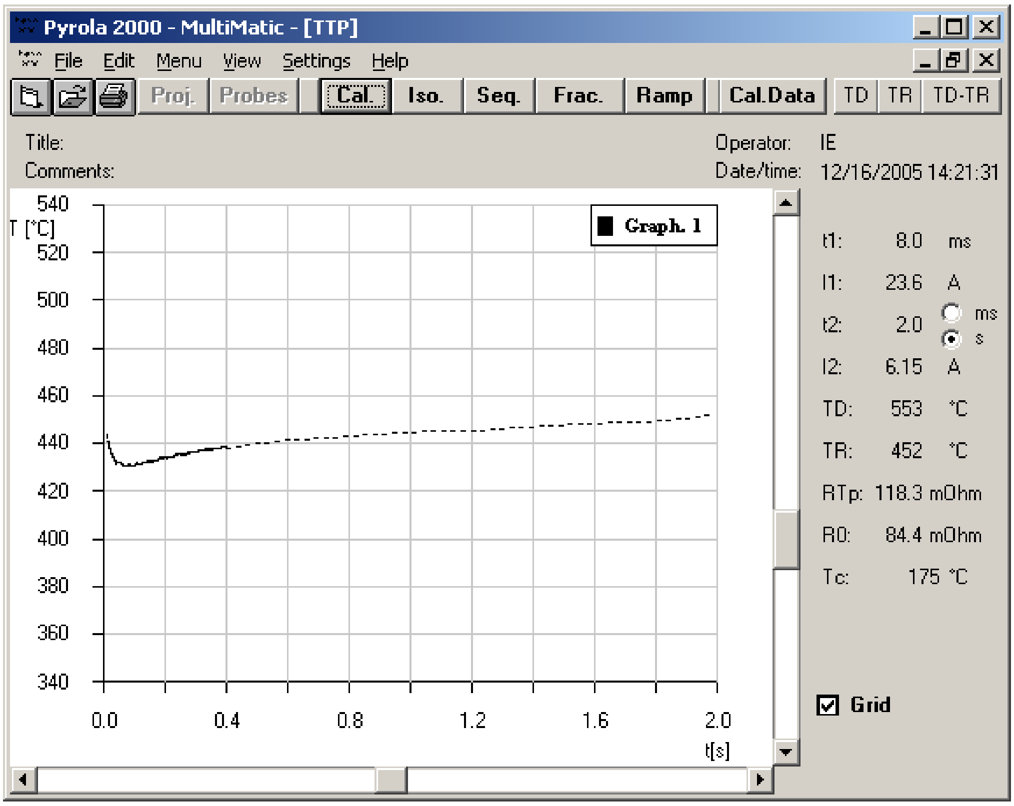
Note the difference in style when studying the TR plot. The temperature appears to drop from an initially high value. Why this should occur is not understood. However, it is safe to say that I2 is a little low.

Increasing I2 by 0.05A gave the following result:



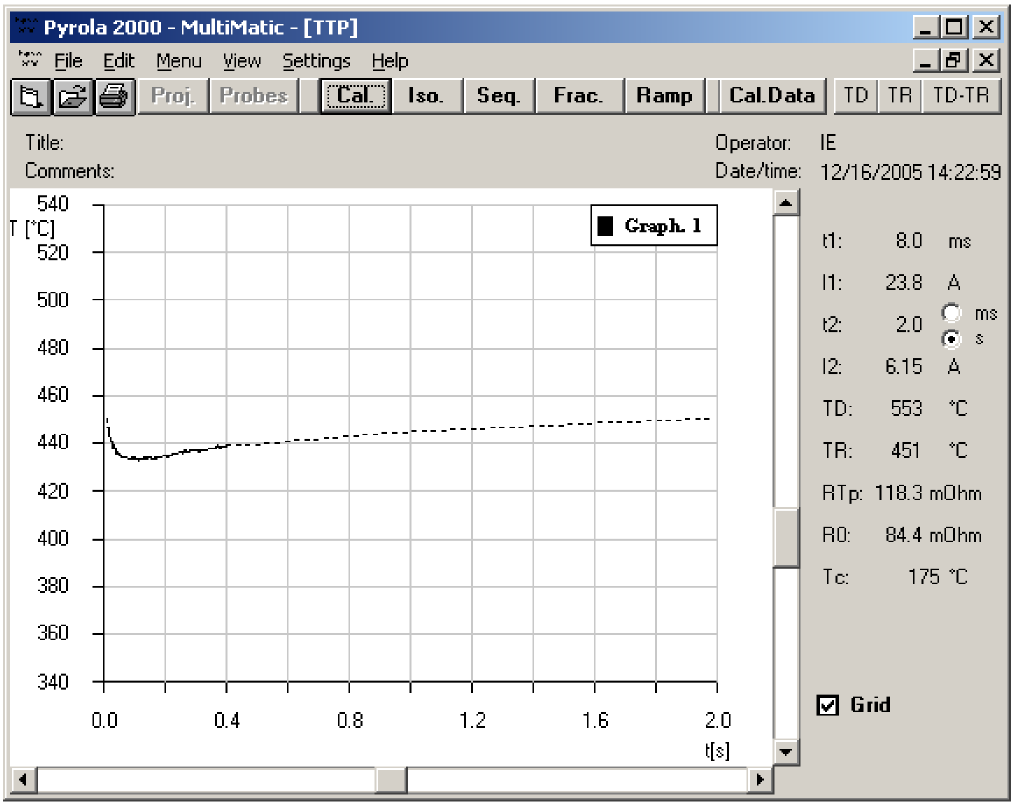
Note that, as the pyrolysis temperature falls, smaller changes in both I1 and I2 are needed to effect the desired change. This should not be too surprising if the dynamics of the system in terms of heat losses are considered. Now the I2 value is judged to be satisfactory. In the past, this type of result at temperatures below 550℃ had been deemed satisfactory. However, this may not be the case as the following plots illustrate. The effect of varying I1 was explored in an attempt to improve the general appearance of the plot.

I1 was decreased significantly in an attempt to reduce the apparent overshoot on the initial pulse with the following result:



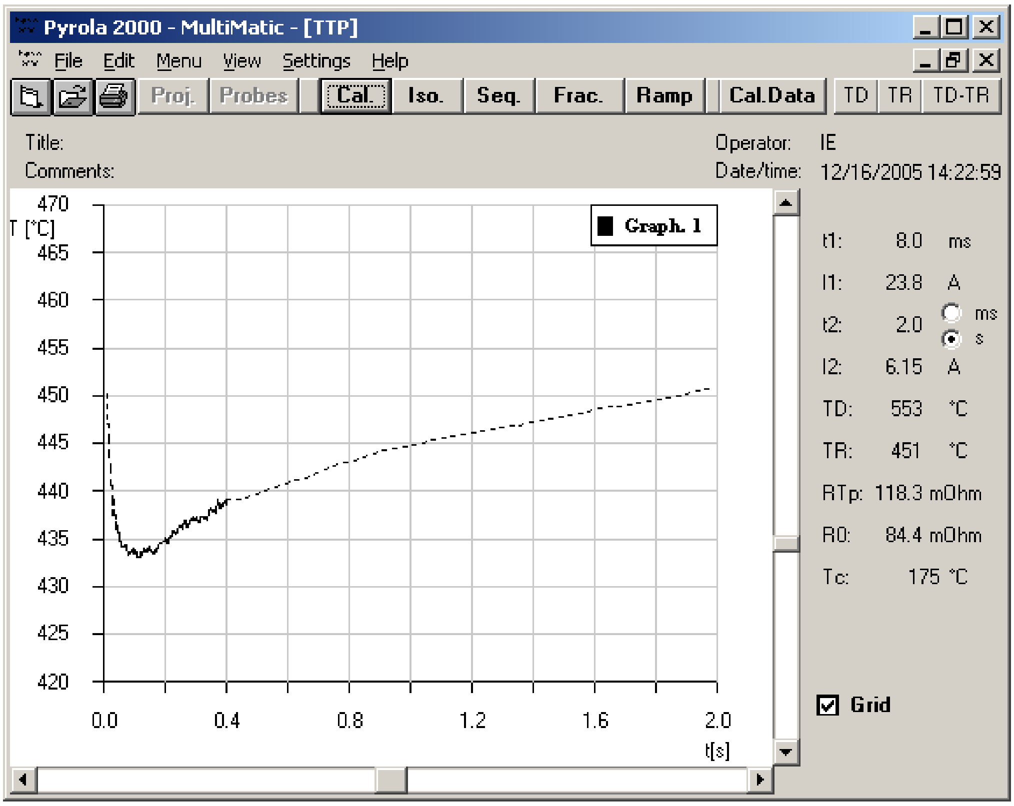
This appears much more satisfactory in terms of potential analytical performance in that the apparent overshoot is much reduced. It was decided to adjust the I1 value until the criteria that are used on TD plots were met, namely to ensure that the temperature achieved during the initial pulse did not exceed the desired calibration temperature. However, in this instance, it was judged that I1 was too low since the initial pulse only just exceeds 440°C.

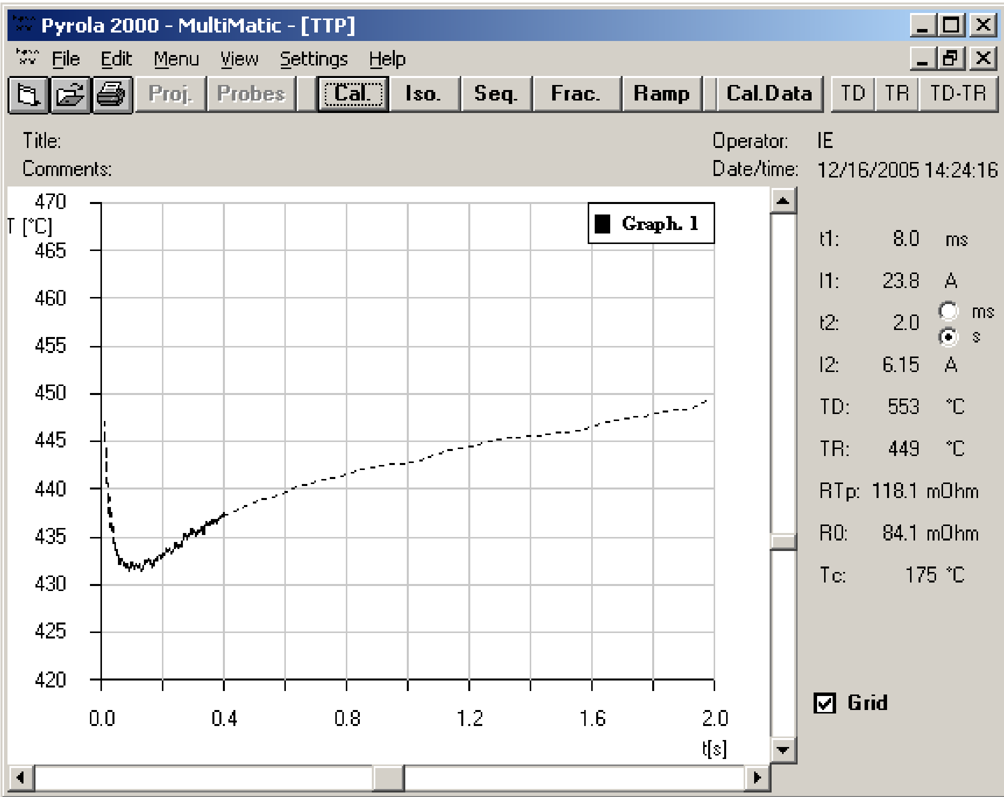
Increasing I1 by 0.2A and leaving I2 unchanged gave the following result:

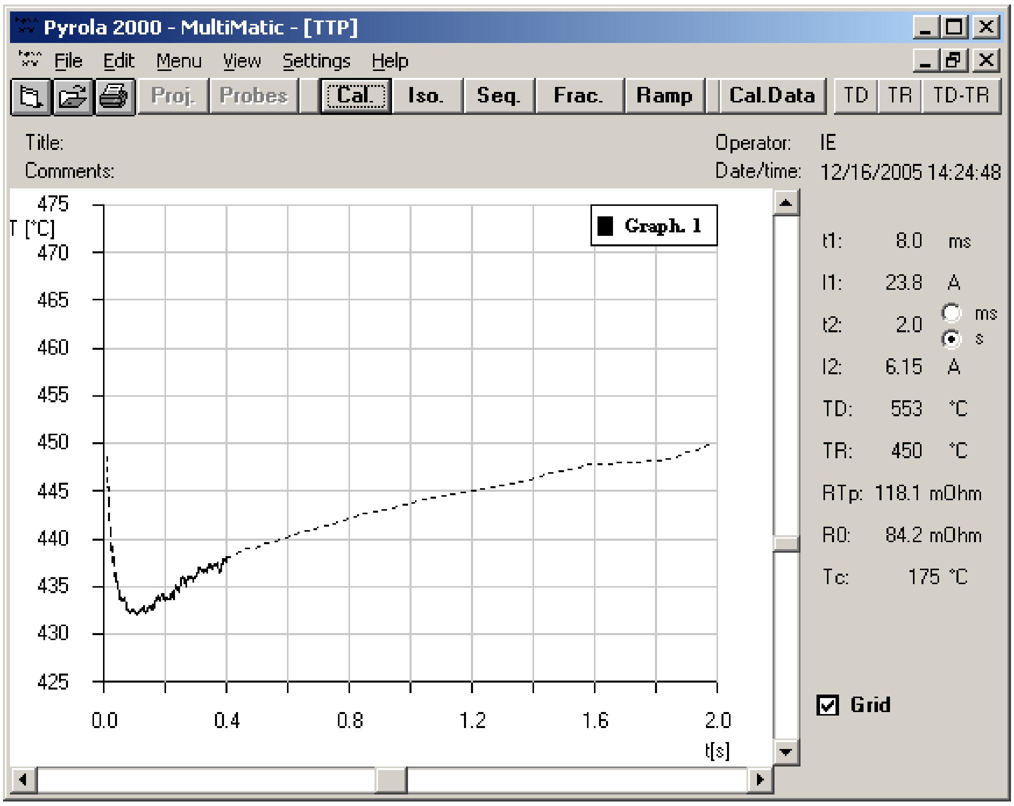


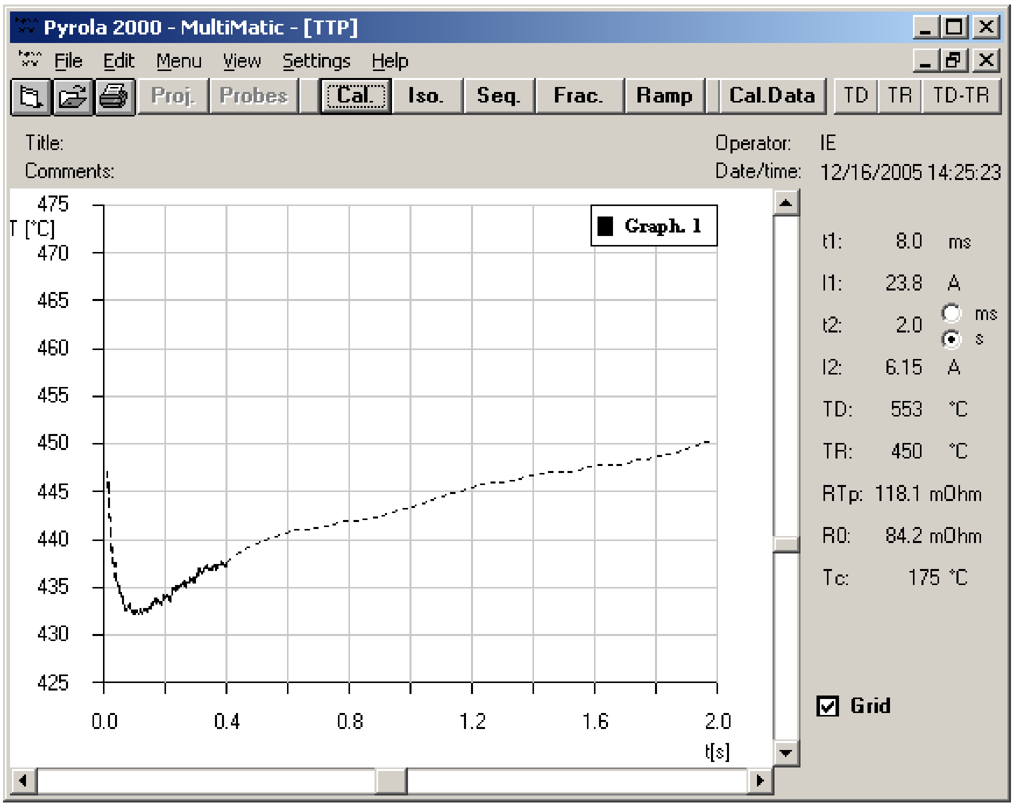
Here the initial pulse just reaches 450℃ and the final temperature, as set by I2 is satisfactory. It was decided to use this approach during calibration at 300℃.

5.3 Reproducibility.





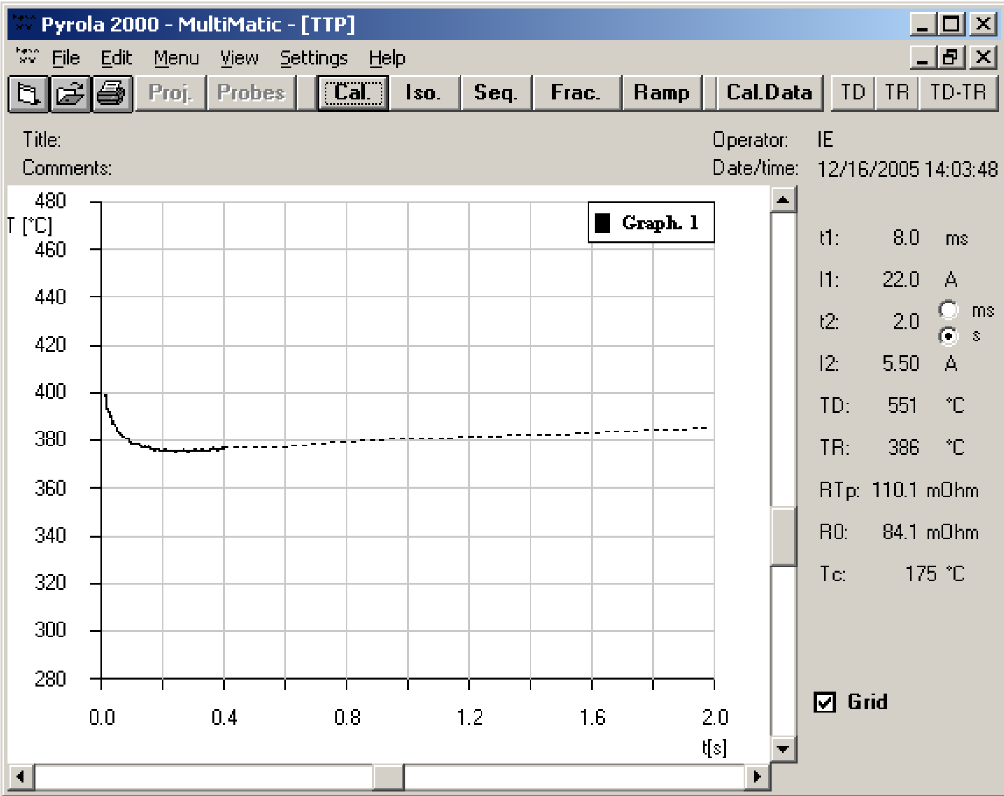

Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 14:24:48
Graph. 1
T [°C]
475 470 465 460 455 450 445 440 435 430 425
0.0 0.4 0.8 1.2 1.6 2.0
t[s]
t1: 8.0 ms
I1: 23.8 A
t2: 2.0 ms s
I2: 6.15 A
TD: 553 °C
TR: 450 °C
RTp: 118.1 mOhm
R0: 84.2 mOhm
Tc: 175 °C
Grid


Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 14:25:23
Graph. 1
T [°C]
475 470 465 460 455 450 445 440 435 430 425
0.0 0.4 0.8 1.2 1.6 2.0
t[s]
t1: 8.0 ms
I1: 23.8 A
t2: 2.0 ms s
I2: 6.15 A
TD: 553 °C
TR: 450 °C
RTp: 118.1 mOhm
R0: 84.2 mOhm
Tc: 175 °C
Grid

6.0 Calibration at 300°C.

6.1 Initial Result.

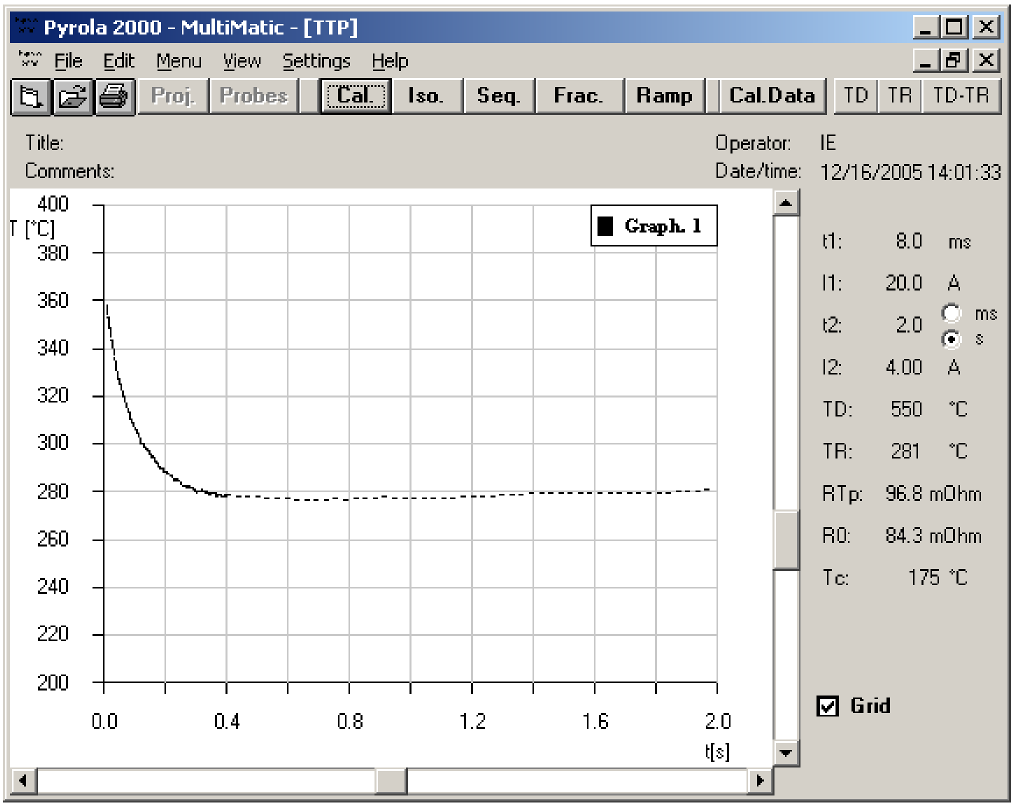
Once again, the TD trace is absent from the plot as it has no meaning at temperatures below 550°C. Setting I1 to 22.0A and I2 to 5.50A yielded the following result:



Both I1 and I2 are too high and the adjustment of I1 should be done to ensure that the initial pulse does not exceed the desired calibration temperature.

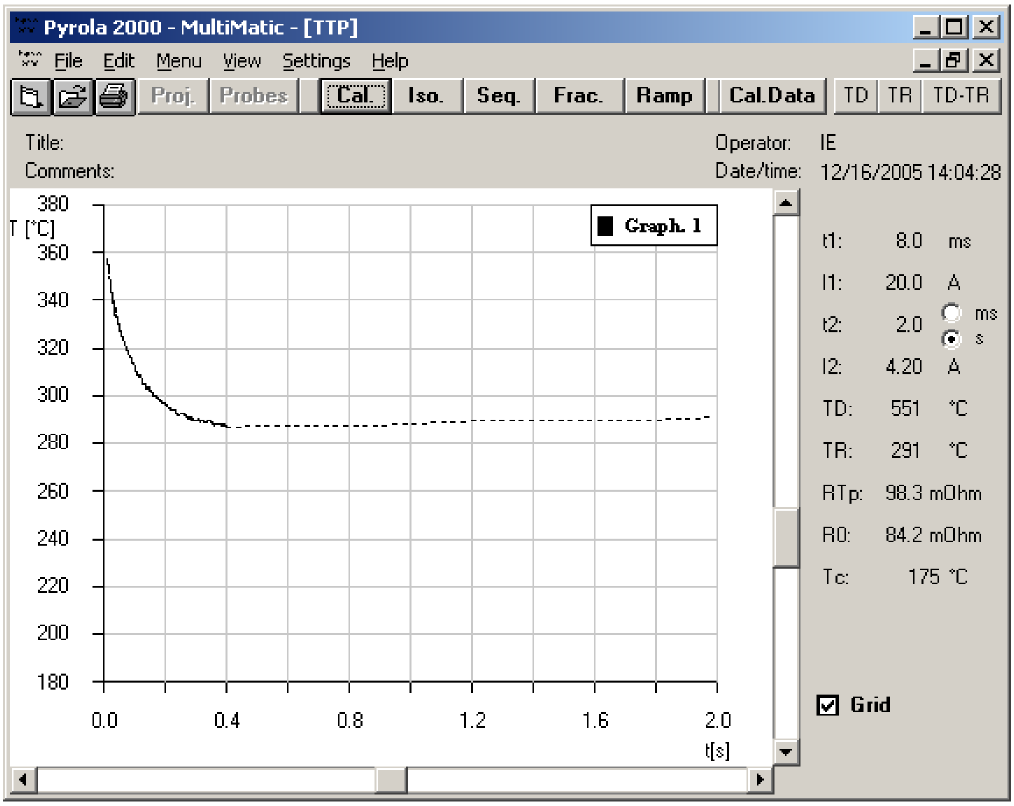
6.2 Iterative Approach to 300℃.

Reducing I1 to 20.0A and I2 to 4.00A yielded the following result:



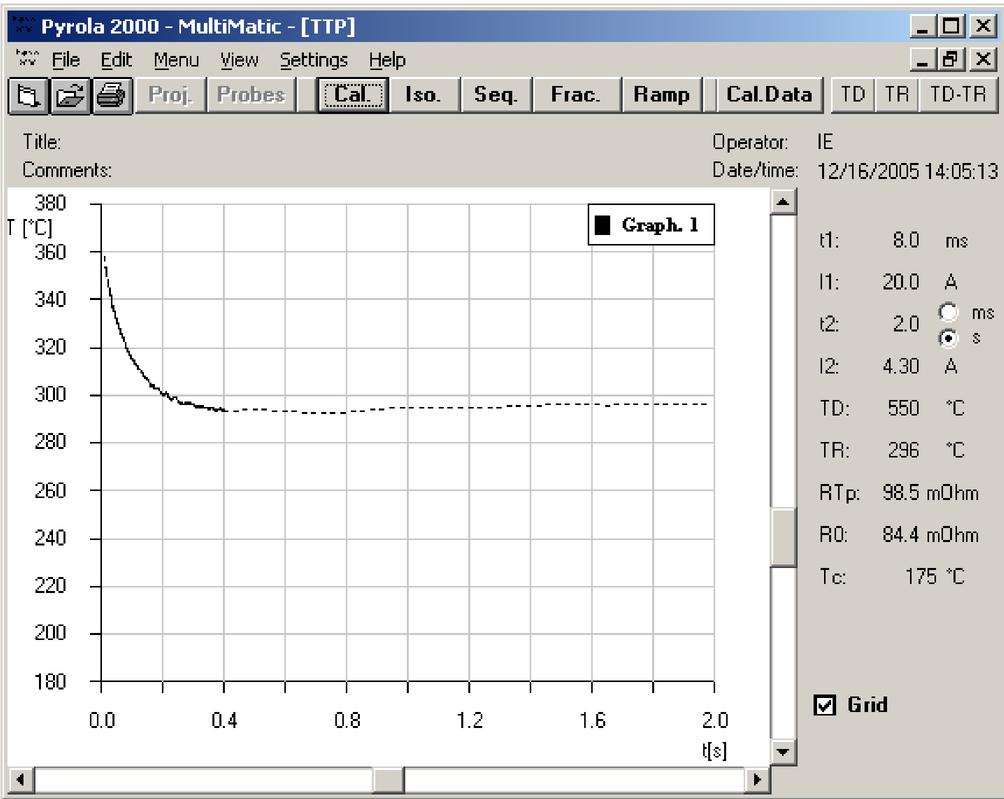
Here, I1 is still too high as seen by the initial pulse temperature achieving almost 360℃ and I2 is now a little too low for the desired calibration temperature of 300℃.

Increasing I2 to 4.20A yielded the following result:



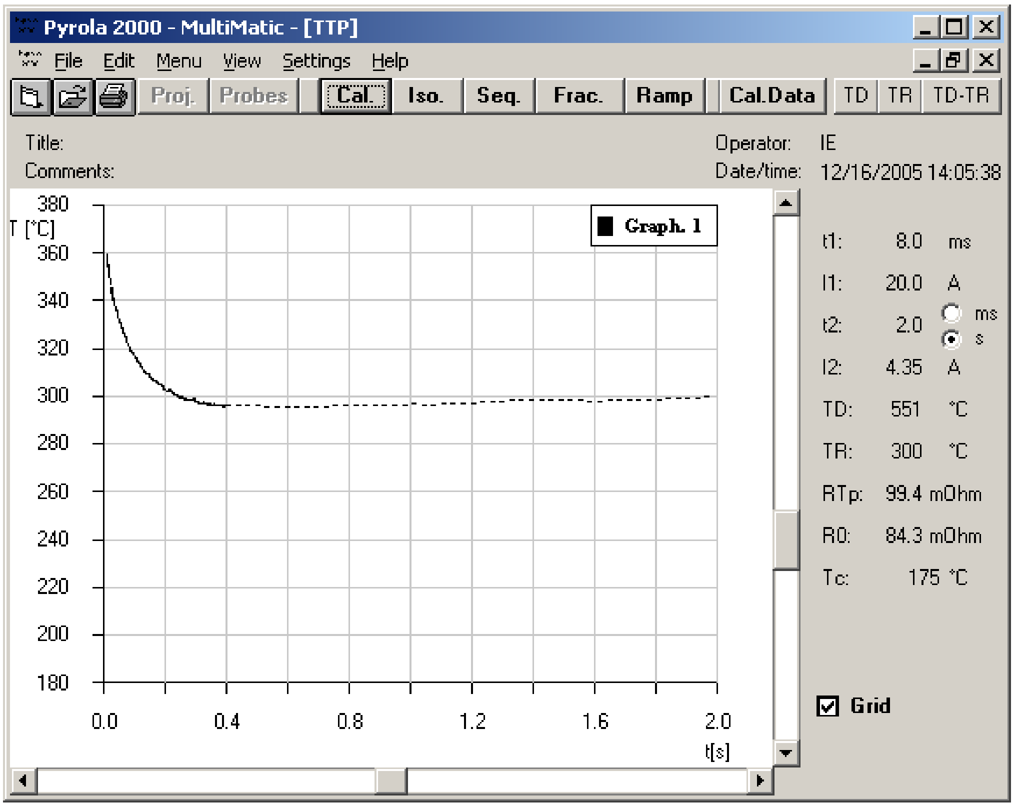
The final temperature has increased by 10° so I2 is still too low.

Increasing I2 by 0.10A yielded the following result:



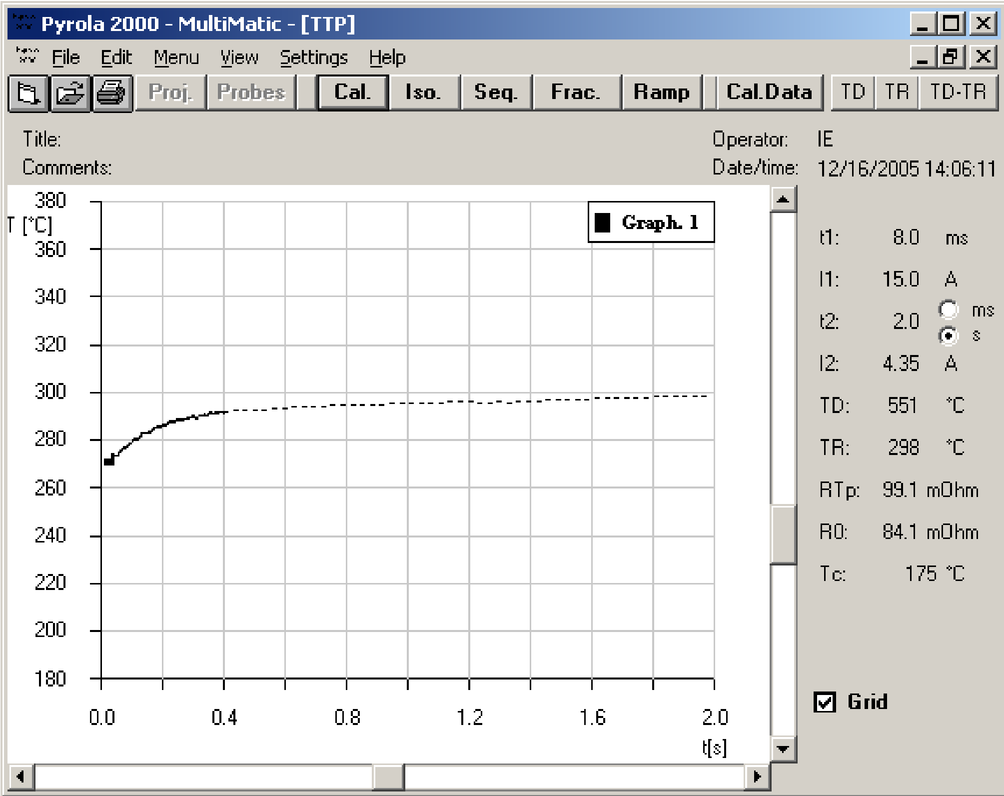
Now the final temperature is acceptable, achieving a temperature only 4° below the desired calibration temperature. However, another small increase in I2 may be beneficial.

Increasing I2 by 0.05A yielded the following result:



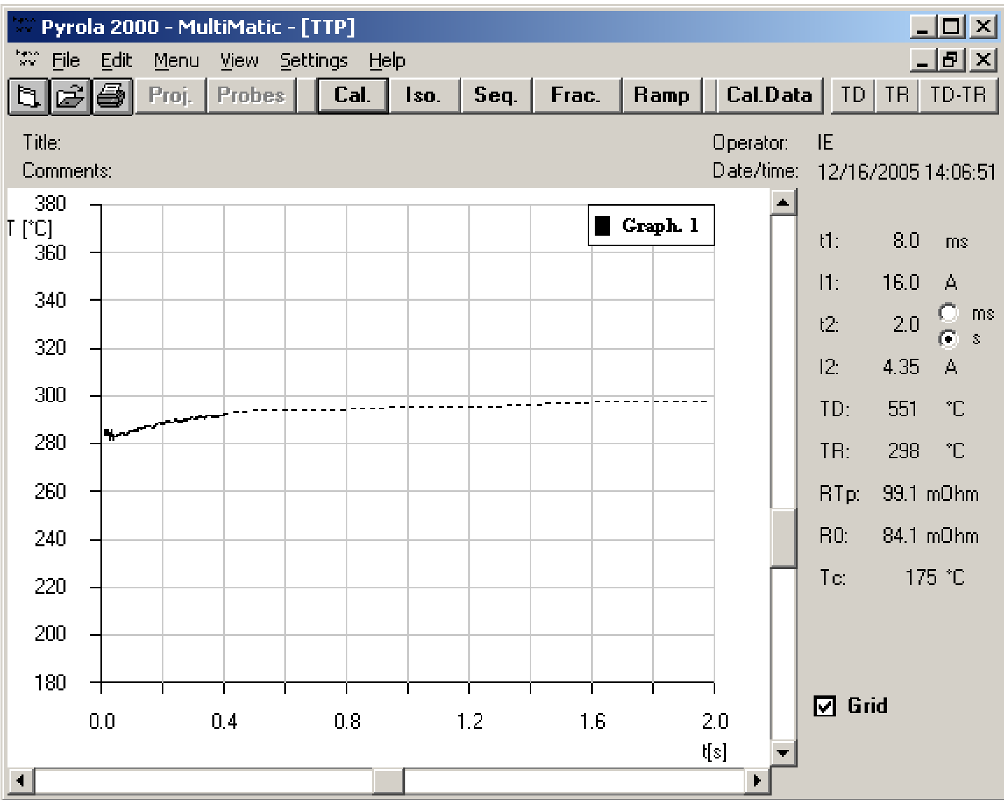
The final temperature achieved with this setting of I2 coincides with the desired calibration temperature.

I1 is now reduced to ensure that the temperature achieved during the initial pulse does not exceed the calibration temperature. The plot obtained is shown below:



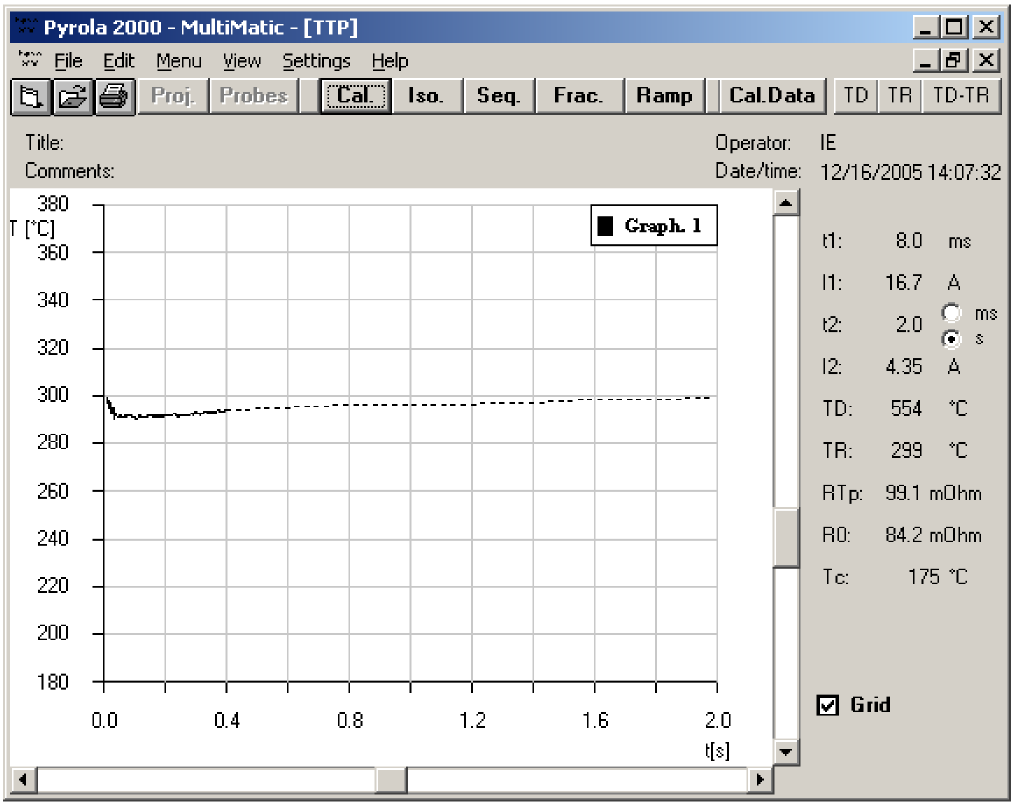
This plot shows that a gross reduction in I1 gives an initial pulse temperature that is too low for the desired calibration.

Increasing I1 produces the plot below:



Although the initial pulse temperature has increased, it was still judged to be lower than would be ideal.

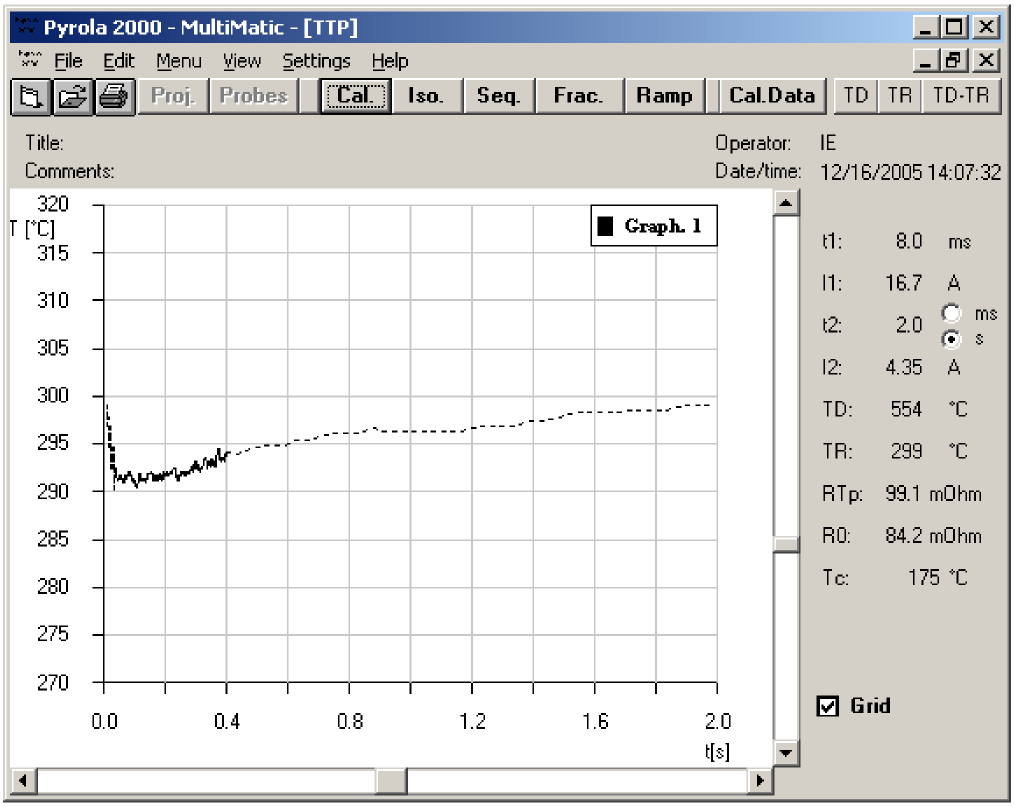
A further increase in I1 produced the following result:

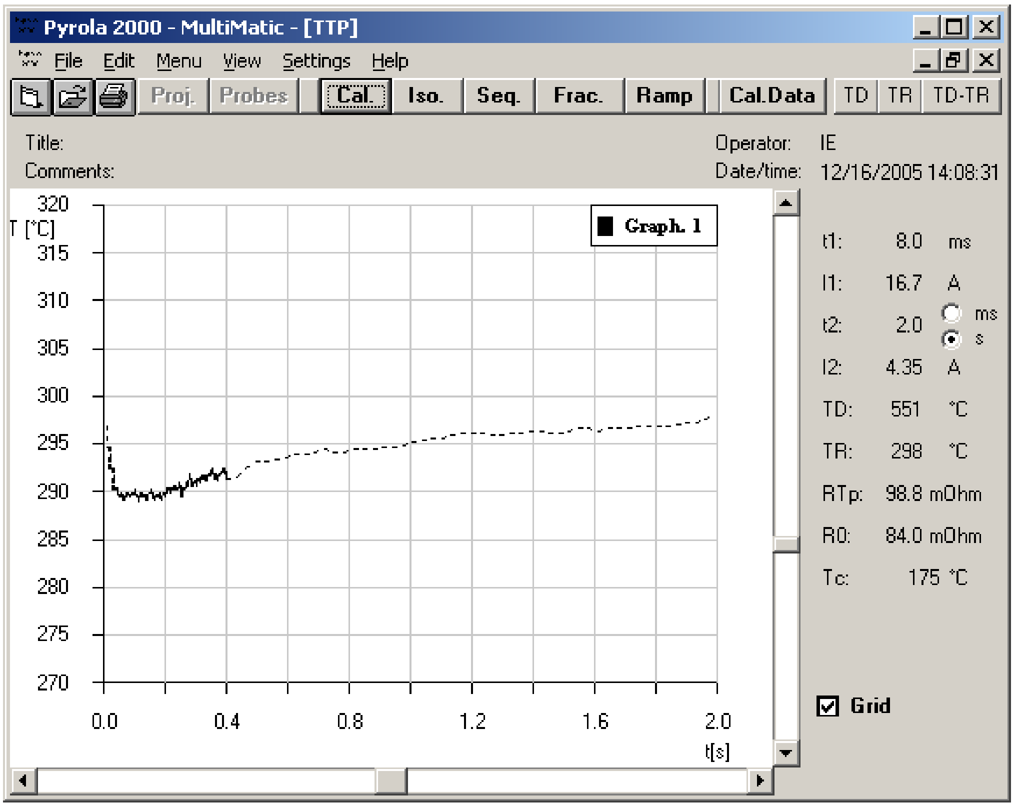


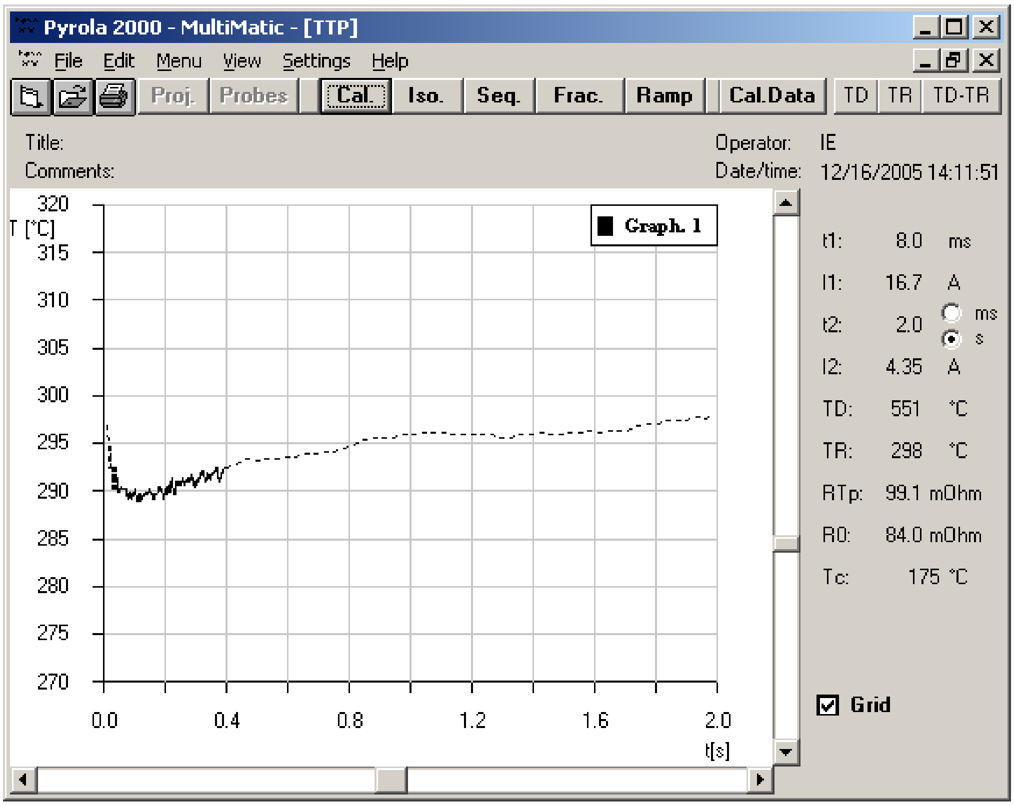
Here, the initial pulse temperature coincides with the desired calibration temperature and the final temperature obtained is still satisfactory and the TTP was saved. These settings were used to investigate the reproducibility of the temperature plot at a calibration temperature of 300℃.

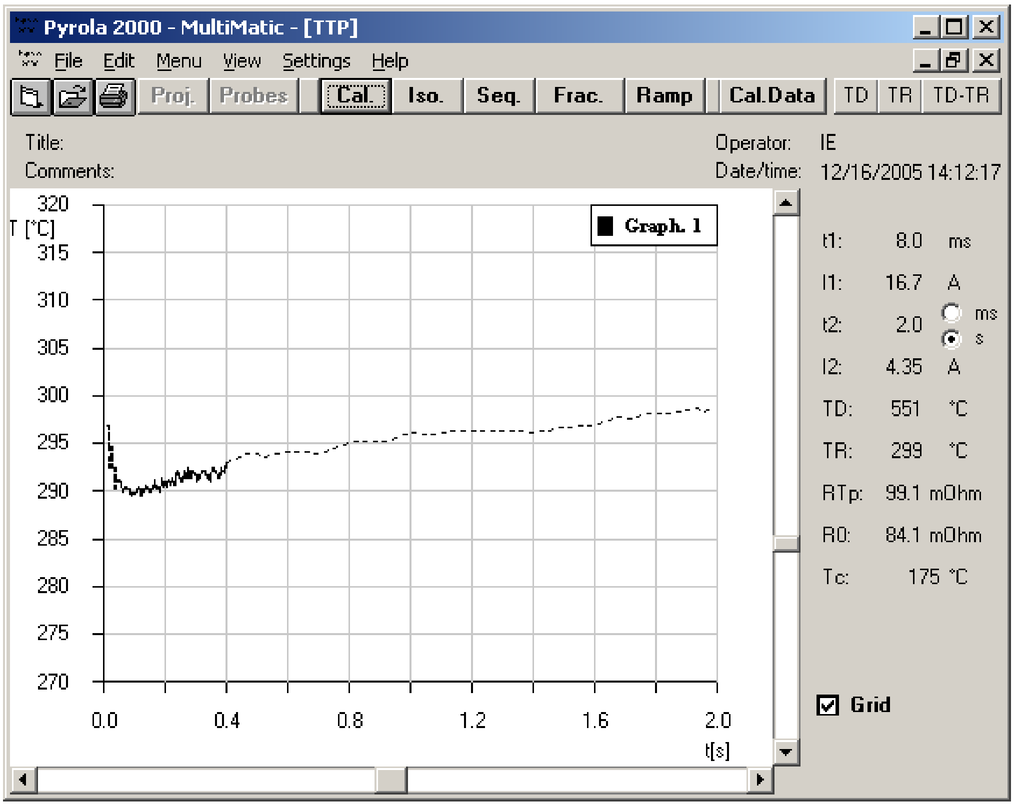
6.3 Reproducibility.

As previously, increasing the y-scale and performing repeat pyrolyses yielded the following set of plots:






Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 14:11:51
Graph. 1
T [°C]
320
315
310
305
300
295
290
285
280
275
270
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
t1: 8.0 ms
I1: 16.7 A
t2: 2.0 ms s
I2: 4.35 A
TD: 551 °C
TR: 298 °C
RTp: 99.1 mOhm
R0: 84.0 mOhm
Tc: 175 °C
Grid


Pyrola 2000 - MultiMatic - [TTP]
File Edit Menu View Settings Help
Proj. Probes Cal. Iso. Seq. Frac. Ramp Cal.Data TD TR TD-TR
Title:
Comments:
Operator: IE
Date/time: 12/16/2005 14:12:17
Graph. 1
T [°C]
320
315
310
305
300
295
290
285
280
275
270
0.0
0.4
0.8
1.2
1.6
2.0
t[s]
t1: 8.0 ms
I1: 16.7 A
t2: 2.0 ms s
I2: 4.35 A
TD: 551 °C
TR: 299 °C
RTp: 99.1 mOhm
R0: 84.1 mOhm
Tc: 175 °C
Grid

7.0 Conclusion.

Although this procedure may seem long-winded, the steps described here are no more numerous than those described in the manual. For reproducible pyrolysis results, it is important that the calibration be performed with some care.

The guidance in the manual that indicates that the system be recalibrated if either the chamber temperature or gas flow is changed is probably sound advice. Obviously a recalibration is necessary if the filament is changed since, although the filaments are designed to be as close to one another as possible in terms of performance, there will be detail differences in either filament resistance or the contact resistance between the limbs of the filament holder and the filament when the filament is replaced. Remember that resistance changes of a few milli-ohms will be significant in this device.