

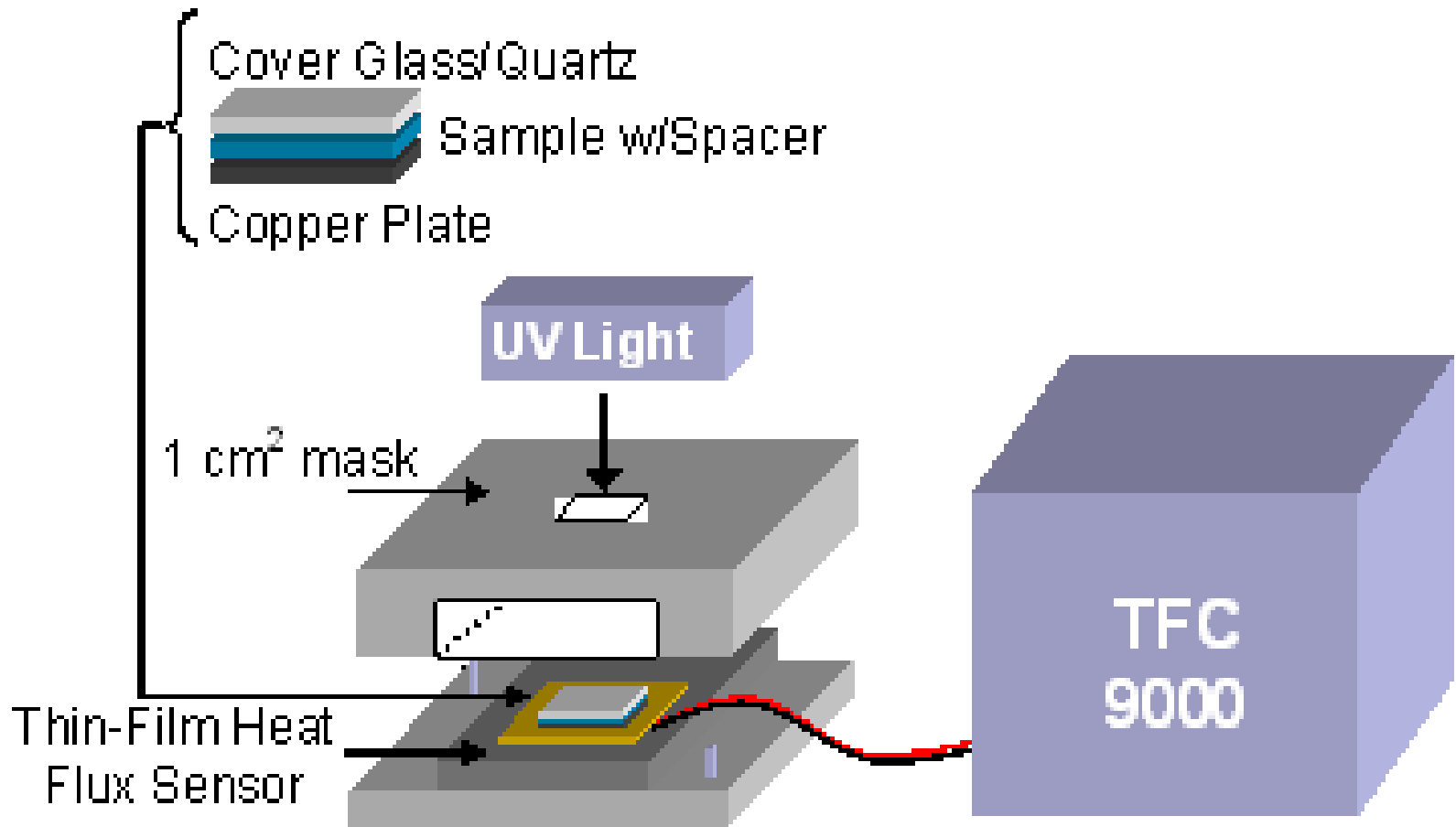
The Use of Thin Film Calorimetry in Profiling Norrish Type I and Norrish Type II Photoinitiators

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Celeste Diener

Open Test Station



TFC-9000



Dual Test Station



Create Temperature set points

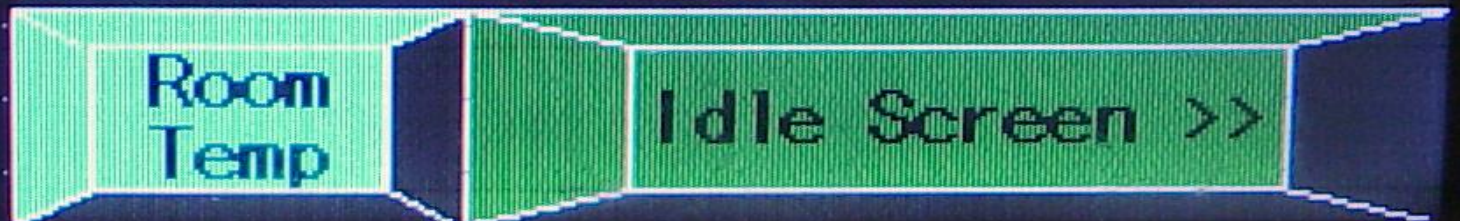
Temperature Display:



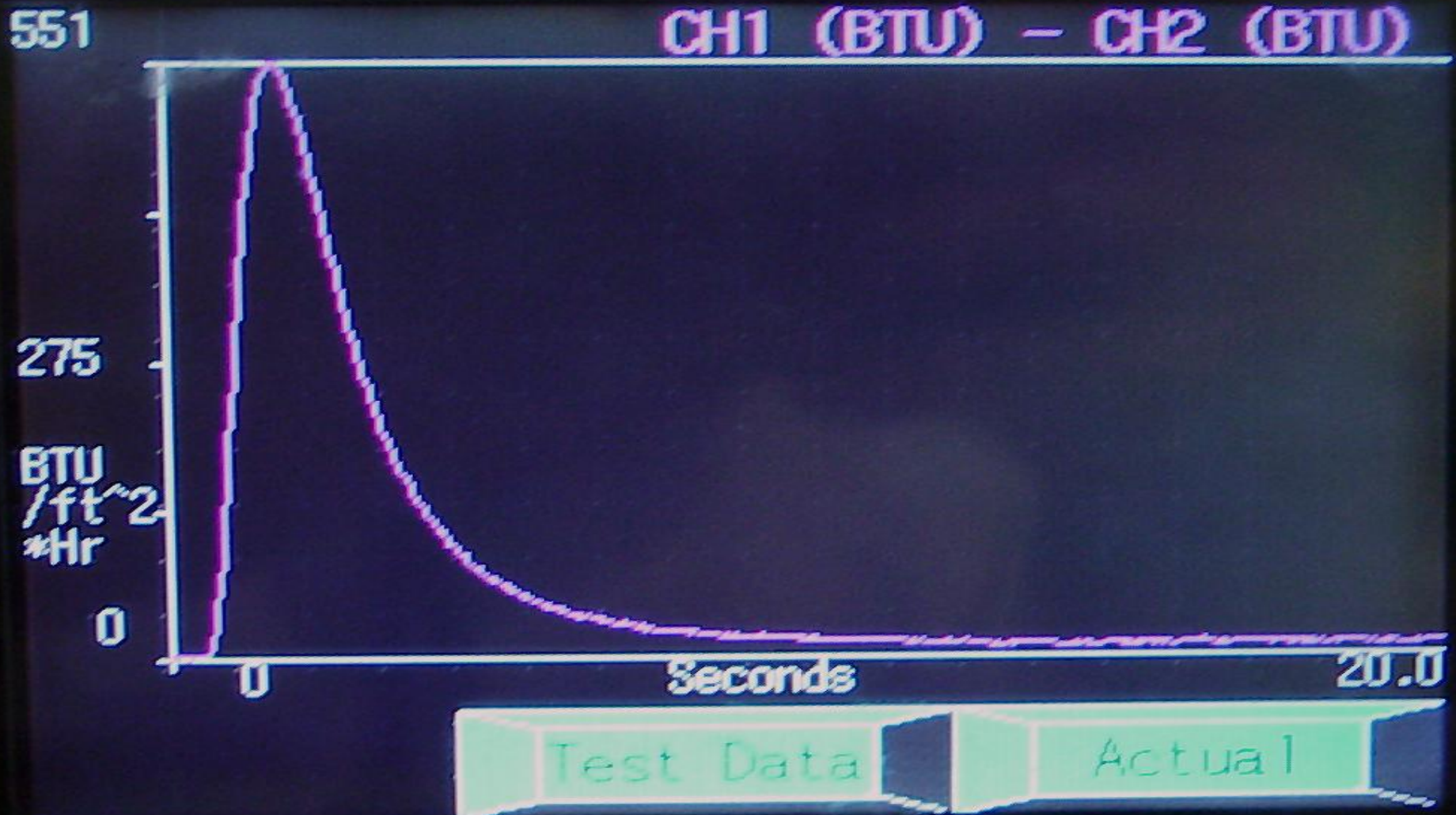
Currently: C

Start Test Temp Setpoint: 28.0 C

Test Length Setpoint: 0 Minutes
20 Seconds



Real Time Graphing



On Line Test Results

TEST RESULT DATA

Testing

Test Length (Seconds): 10.3
Time to Peak (Seconds): 8.39
Peak Heat: 2 BTU 2110 J
Total Heat*: 0.000 BTU* 0 J*
Slope: 0 * ft²/hr
Start Temp1: 20.6 End Temp1: 0.0
Start Temp2: 220.0 End Temp2: 0.0
Heat1: 0 BTU/F²*hr 0 J/gr
Heat2: 0 BTU/F²*hr 0 J/gr
Heat1-2: 0 BTU/F²*hr 0 J/gr
Temp #1: 20.7C Temp #2: 220.0C

Stop

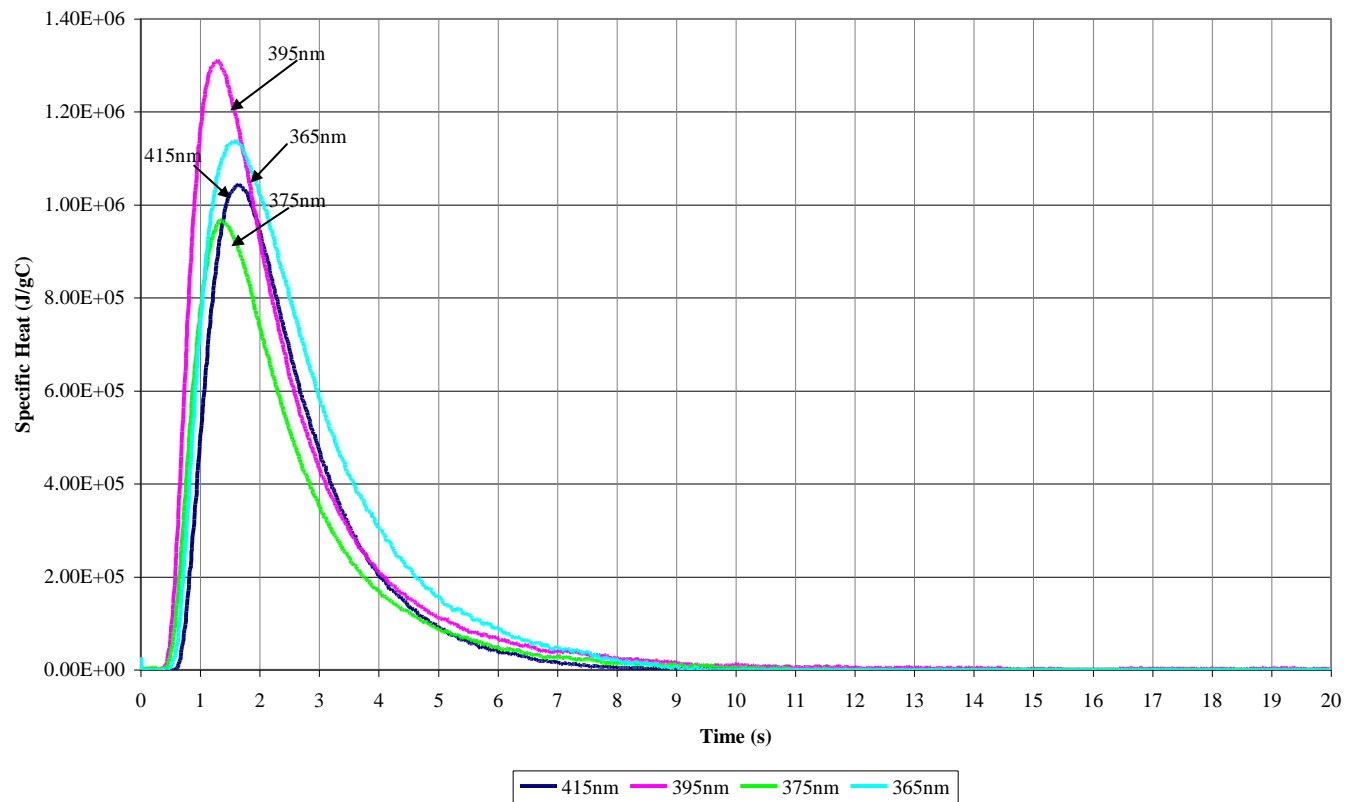
Transfer

New

Graph

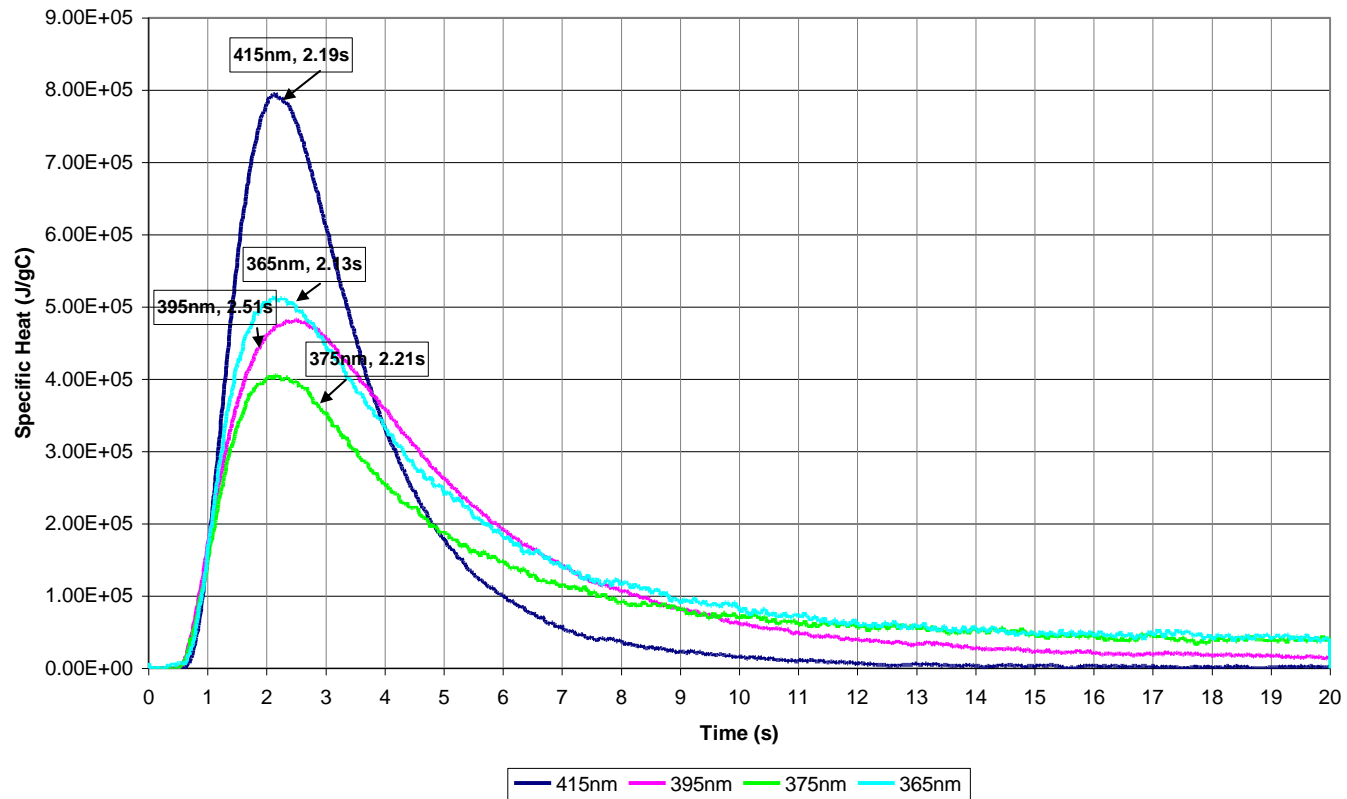
Exported Test Results

CN971A80(Urethane Acrylate)/IRGACURE 369(4%)
Trial 2



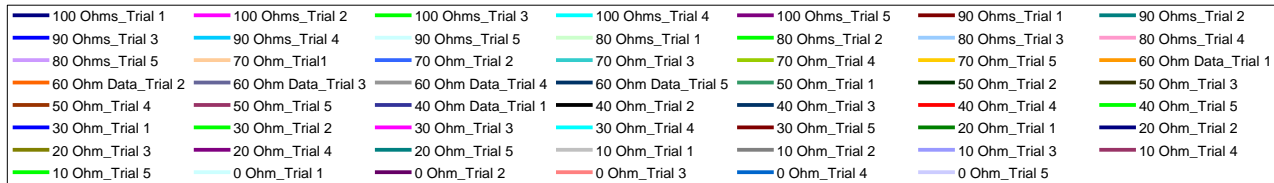
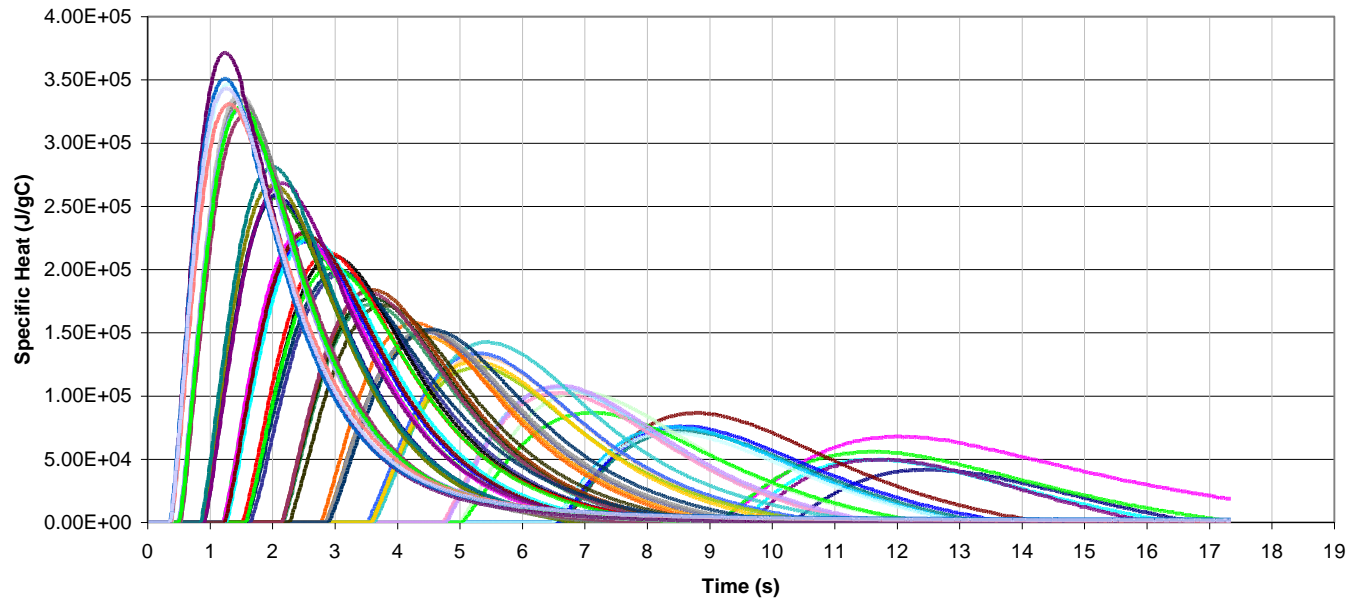
Exported Test Results

EBECRYL 3720(Bisphenol-A epoxy diacrylate)/ITX(1%)/EDAB(5%)
Trial 2



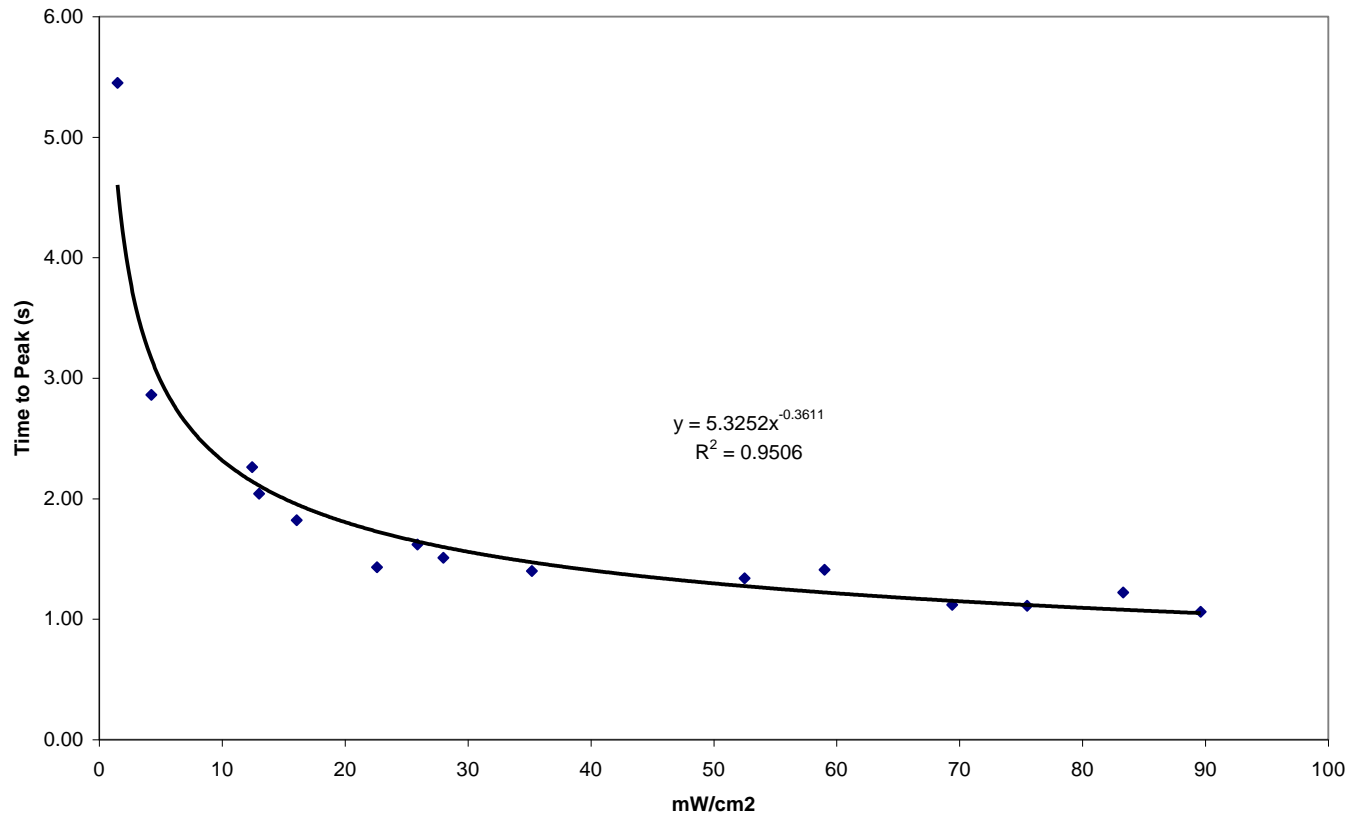
Intensity Study

0-100 Ohm Resistance
Trials 1-5



Power vs. Time to peak

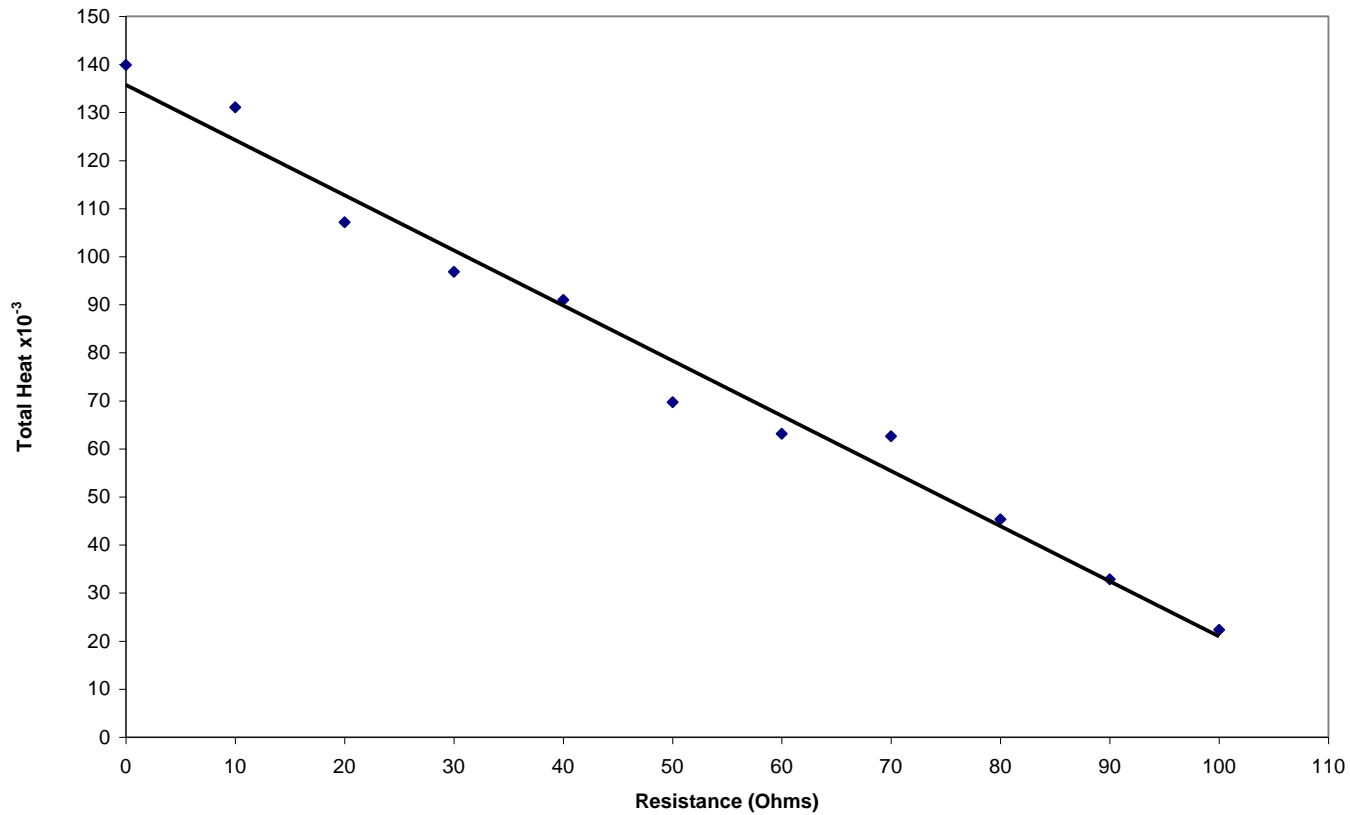
Time to Peak vs. mW/cm2



Intensity vs. Total heat

Intensity Studies_Total Heat Released

$$y = -1.1476x + 135.74$$
$$R^2 = 0.9823$$



Fast Data Collection

22 objects (Disk free space: 236 GB) 9.88 MB Local intranet

| Name | Size | Type | Date Modified |
|-------------------------|----------|------------------------|---------------------|
| 415nm_Trial 1.txt | 33 KB | Text Document | 10/18/2007 8:46 AM |
| 415nm_Trial 2.txt | 33 KB | Text Document | 10/18/2007 8:49 AM |
| 415nm_Trial 3.txt | 34 KB | Text Document | 10/18/2007 8:51 AM |
| 415nm_Trial 4.txt | 34 KB | Text Document | 10/18/2007 8:54 AM |
| 415nm_Trial 5.txt | 33 KB | Text Document | 10/18/2007 8:57 AM |
| 395nm_Trial 1.txt | 33 KB | Text Document | 10/18/2007 9:02 AM |
| 395nm_Trial 2.txt | 33 KB | Text Document | 10/18/2007 9:05 AM |
| 395nm_Trial 3.txt | 33 KB | Text Document | 10/18/2007 9:08 AM |
| 395nm_Trial 4.txt | 33 KB | Text Document | 10/18/2007 9:11 AM |
| 395nm_Trial 5.txt | 33 KB | Text Document | 10/18/2007 9:13 AM |
| 375nm_Trial 1.txt | 33 KB | Text Document | 10/18/2007 9:17 AM |
| 375nm_Trial 2.txt | 33 KB | Text Document | 10/18/2007 9:20 AM |
| 375nm_Trial 3.txt | 33 KB | Text Document | 10/18/2007 9:23 AM |
| 375nm_Trial 4.txt | 33 KB | Text Document | 10/18/2007 9:25 AM |
| 375nm_Trial 5.txt | 33 KB | Text Document | 10/18/2007 9:28 AM |
| 365nm_Trial 1.txt | 33 KB | Text Document | 10/18/2007 9:31 AM |
| 365nm_Trial 2.txt | 33 KB | Text Document | 10/18/2007 9:34 AM |
| 365nm_Trial 3.txt | 32 KB | Text Document | 10/18/2007 9:36 AM |
| 365nm_Trial 4.txt | 33 KB | Text Document | 10/18/2007 9:39 AM |
| 365nm_Trial 5.txt | 33 KB | Text Document | 10/18/2007 9:41 AM |
| Formulation No. 178.xls | 8,418 KB | Microsoft Excel Wor... | 10/18/2007 11:25 AM |
| Formulation No. 178.doc | 1,052 KB | Microsoft Word Doc... | 10/18/2007 11:44 AM |

First test at 10/18/2007 8:46AM -
→ Report at 10/18/2007 11:44AM

Norrish Type I Reaction

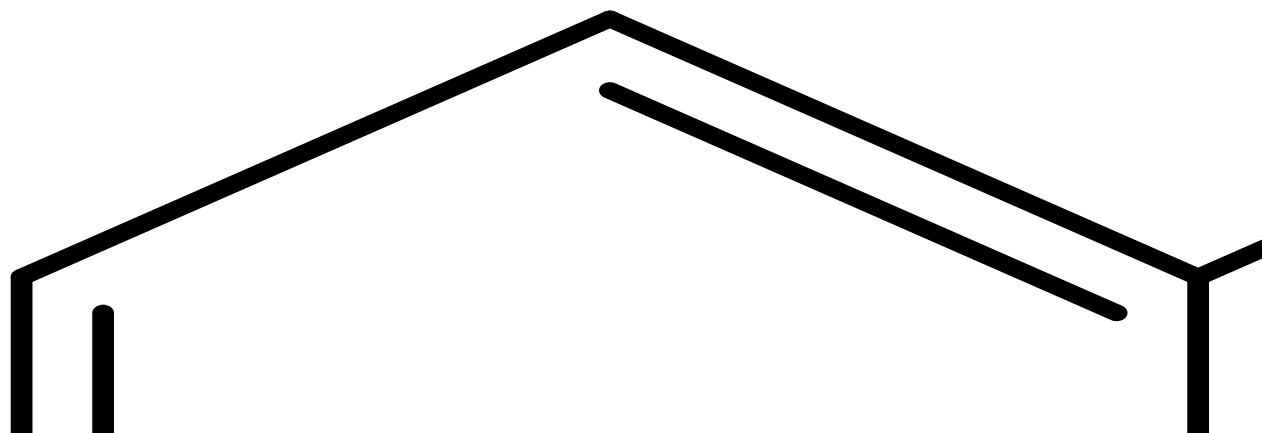


Table 1: TFC data for the 415nm* LED with the polyester acrylates
CN2262

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|------------------------------|--------------|--------------|--------------------------------------|
| ITX | 0.5 | 2.93 | 43 | 5.07 |
| | 1 | 3.05 | 51 | 5.55 |
| | 1.5 | 2.86 | 45 | 5.73 |
| TPO | 0.5 | 2.18 | 196 | 15.5 |
| | 1 | 2.70 | 125 | 14.3 |
| | 2 | 1.54 | 347 | 20.6 |
| | 3 | 1.40 | 407 | 19.2 |
| | 4 | 1.19 | 404 | 23.2 |
| Irgacure 369 | 1 | 3.09 | 101 | 11.0 |
| | 2 | 2.33 | 120 | 14.2 |
| | 3 | 1.80 | 109 | 16.1 |
| | 4 | 1.64 | 143 | 12.0 |

*415nm wavelength did not cure BP/EDAB series

Table 2: TFC data for the 395nm LED with the polyester acrylates
CN2262

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5$ <i>(J/g°C)</i> |
|------------------|------------------------------|--------------|--------------|---|
| BP | 4 | 10.42 | 39 | 2.10 |
| | 5 | 9.09 | 55 | 2.28 |
| | 6 | 9.04 | 50 | 2.08 |
| ITX | 0.5 | 2.37 | 105 | 7.74 |
| | 1 | 2.21 | 108 | 8.14 |
| | 1.5 | 2.04 | 102 | 7.73 |
| TPO | 0.5 | 1.31 | 546 | 22.5 |
| | 1 | 1.45 | 444 | 20.7 |
| | 2 | 1.08 | 695 | 25.2 |
| | 3 | 0.95 | 618 | 29.0 |
| | 4 | 0.99 | 598 | 26.6 |
| Irgacure 369 | 1 | 1.77 | 302 | 17.0 |
| | 2 | 1.46 | 344 | 20.1 |
| | 3 | 1.16 | 209 | 15.1 |
| | 4 | 1.08 | 306 | 18.2 |

Table 3: TFC data for the 375nm LED with the polyester acrylates CN2262

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5$ <i>(J/g°C)</i> |
|------------------|------------------------------|--------------|--------------|---|
| BP | 4 | 4.42 | 149 | 3.28 |
| | 5 | 4.52 | 175 | 3.38 |
| | 6 | 4.31 | 164 | 2.98 |
| ITX | 0.5 | 2.62 | 187 | 6.14 |
| | 1 | 2.33 | 57 | 5.17 |
| | 1.5 | 2.03 | 54 | 5.46 |
| TPO | 0.5 | 1.51 | 434 | 19.6 |
| | 1 | 1.61 | 316 | 19.6 |
| | 2 | 1.32 | 557 | 21.8 |
| | 3 | 1.02 | 379 | 24.0 |
| | 4 | 1.08 | 440 | 24.5 |
| Irgacure 369 | 1 | 1.53 | 279 | 23.6 |
| | 2 | 1.31 | 328 | 24.7 |
| | 3 | 1.13 | 289 | 22.0 |
| | 4 | 1.06 | 334 | 23.8 |

Table 4: TFC data for the 365nm LED with the polyester acrylate CN2262

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5$ <i>(J/g°C)</i> |
|------------------|------------------------------|--------------|--------------|---|
| BP | 4 | 3.70 | 287 | 4.71 |
| | 5 | 3.62 | 310 | 4.97 |
| | 6 | 3.68 | 284 | 4.06 |
| ITX | 0.5 | 2.12 | 115 | 8.95 |
| | 1 | 2.05 | 89 | 6.26 |
| | 1.5 | 2.13 | 71 | 5.45 |
| TPO | 0.5 | 1.41 | 540 | 22.0 |
| | 1 | 1.49 | 399 | 22.0 |
| | 2 | 1.24 | 635 | 25.8 |
| | 3 | 1.15 | 570 | 25.7 |
| | 4 | 1.29 | 508 | 25.2 |
| Irgacure 369 | 1 | 1.32 | 410 | 23.6 |
| | 2 | 1.25 | 439 | 24.7 |
| | 3 | 1.08 | 408 | 22.0 |
| | 4 | 1.11 | 376 | 23.8 |

Table 5: TFC data for the 415nm LED with the Bisphenol-A epoxy diacrylate Ebecryl 3720

| <i>Initiator</i> | <i>Conc. (wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|--------------------|--------------|--------------|--------------------------------------|
| ITX | 0.5 | 2.00 | 254 | 8.54 |
| | 1 | 2.28 | 218 | 7.93 |
| | 1.5 | 2.43 | 179 | 6.72 |
| TPO | 0.5 | 3.10 | 159 | 11.3 |
| | 1 | 2.13 | 321 | 12.7 |
| | 2 | 1.69 | 457 | 14.3 |
| | 3 | 1.53 | 509 | 14.8 |
| | 4 | 1.65 | 480 | 13.7 |
| Irgacure 369 | 1 | 2.03 | 221 | 10.4 |
| | 2 | 1.49 | 389 | 11.3 |
| | 3 | 1.35 | 418 | 12.2 |
| | 4 | 1.33 | 492 | 12.6 |

Table 6: TFC data for the 395nm LED with the Bisphenol-A epoxy diacrylate Ebecryl 3720

| <i>Initiator</i> | <i>Conc. (wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|--------------------|--------------|--------------|--------------------------------------|
| BP | 4 | 7.53 | 101 | 3.23 |
| | 5 | 7.62 | 135 | 3.17 |
| | 6 | 5.76 | 166 | 3.29 |
| ITX | 0.5 | 1.95 | 296 | 8.57 |
| | 1 | 2.52 | 131 | 4.71 |
| | 1.5 | 2.52 | 84 | 3.37 |
| TPO | 0.5 | 1.61 | 557 | 15.2 |
| | 1 | 1.35 | 683 | 15.1 |
| | 2 | 1.35 | 741 | 14.7 |
| | 3 | 1.51 | 582 | 12.8 |
| | 4 | 1.31 | 570 | 14.6 |
| Irgacure 369 | 1 | 1.33 | 484 | 13.3 |
| | 2 | 1.24 | 633 | 12.4 |
| | 3 | 1.10 | 609 | 14.3 |
| | 4 | 1.11 | 673 | 15.9 |

Table 7: TFC data for the 375nm LED with the Bisphenol-A epoxy diacrylate Ebecryl 3720

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | <i>t_{max}</i> (s) | <i>Slope</i> | <i>c_{max}</i> x10 ⁵ <i>(J/g°C)</i> |
|------------------|------------------------------|----------------------------|--------------|---|
| BP | 4 | 3.96 | 424 | 4.32 |
| | 5 | 3.92 | 433 | 4.24 |
| | 6 | 3.78 | 448 | 4.55 |
| ITX | 0.5 | 2.35 | 122 | 4.30 |
| | 1 | 2.20 | 69 | 3.98 |
| | 1.5 | 2.42 | 37 | 3.71 |
| TPO | 0.5 | 1.83 | 459 | 14.2 |
| | 1 | 1.62 | 542 | 13.9 |
| | 2 | 1.51 | 491 | 13.6 |
| | 3 | 1.67 | 348 | 96.6 |
| | 4 | 1.72 | 267 | 76.5 |
| Irgacure 369 | 1 | 1.39 | 462 | 13.1 |
| | 2 | 1.37 | 487 | 12.6 |
| | 3 | 1.34 | 449 | 12.8 |
| | 4 | 1.35 | 391 | 12.9 |

Table 8: TFC data for the 365nm LED with the Bisphenol-A epoxy diacrylate Ebecryl 3720

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5$ <i>(J/g°C)</i> |
|------------------|------------------------------|--------------|--------------|---|
| BP | 4 | 2.97 | 630 | 4.88 |
| | 5 | 3.41 | 538 | 4.33 |
| | 6 | 3.09 | 522 | 4.76 |
| ITX | 0.5 | 2.33 | 181 | 5.67 |
| | 1 | 2.11 | 104 | 4.97 |
| | 1.5 | 2.04 | 64 | 4.94 |
| TPO | 0.5 | 1.66 | 547 | 14.8 |
| | 1 | 1.50 | 668 | 14.8 |
| | 2 | 1.55 | 587 | 14.1 |
| | 3 | 1.57 | 489 | 13.1 |
| | 4 | 1.69 | 332 | 9.34 |
| Irgacure 369 | 1 | 1.38 | 529 | 13.7 |
| | 2 | 1.56 | 358 | 10.7 |
| | 3 | 1.44 | 347 | 11.7 |
| | 4 | 1.63 | 235 | 9.03 |

Table 9: TFC data for the 415nm LED with the urethane acrylates CN971A80

| <i>Initiator</i> | <i>Conc. (wt%)</i> | <i>t_{max}(s)</i> | <i>Slope</i> | <i>c_{max} × 10⁵ (J/g°C)</i> |
|-------------------------|-------------------------------|----------------------------------|---------------------|--|
| ITX | 0.5 | 4.04 | 53 | 3.79 |
| | 1 | 4.07 | 50 | 3.36 |
| | 1.5 | 3.77 | 46 | 3.18 |
| TPO | 0.5 | 2.92 | 164 | 10.3 |
| | 1 | 2.26 | 264 | 12.0 |
| | 2 | 1.84 | 343 | 13.2 |
| | 3 | 1.77 | 392 | 12.8 |
| | 4 | 1.59 | 390 | 14.1 |
| Irgacure 369 | 1 | 2.60 | 170 | 8.36 |
| | 2 | 2.04 | 221 | 9.97 |
| | 3 | 1.84 | 288 | 10.5 |
| | 4 | 1.76 | 304 | 10.4 |

Table 10: TFC data for the 395nm LED with the urethane acrylate CN971A80

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|------------------------------|--------------|--------------|--------------------------------------|
| BP | 4 | 10.02 | 50 | 1.72 |
| | 5 | 8.37 | 57 | 1.92 |
| | 6 | 8.76 | 65 | 1.94 |
| ITX | 0.5 | 2.96 | 97 | 5.08 |
| | 1 | 3.18 | 65 | 3.52 |
| | 1.5 | 3.44 | 50 | 2.79 |
| TPO | 0.5 | 1.74 | 406 | 12.3 |
| | 1 | 1.38 | 533 | 15.0 |
| | 2 | 1.33 | 532 | 15.4 |
| | 3 | 1.36 | 552 | 14.3 |
| | 4 | 1.25 | 525 | 15.9 |
| Irgacure 369 | 1 | 1.66 | 362 | 10.6 |
| | 2 | 1.31 | 428 | 14.2 |
| | 3 | 1.33 | 503 | 13.8 |
| | 4 | 1.99 | 510 | 13.1 |

Table 11: TFC data for the 375nm LED with the urethane acrylate CN971A80

| <i>Initiator</i> | <i>Conc. (wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|------------------------|--------------|--------------|--------------------------------------|
| BP | 4 | 4.30 | 163 | 2.39 |
| | 5 | 4.85 | 173 | 2.69 |
| | 6 | 4.99 | 175 | 2.65 |
| ITX | 0.5 | 2.82 | 60 | 3.49 |
| | 1 | 2.74 | 48 | 2.99 |
| | 1.5 | 2.65 | 29 | 2.96 |
| TPO | 0.5 | 1.95 | 342 | 12.4 |
| | 1 | 1.65 | 423 | 14.6 |
| | 2 | 1.67 | 396 | 13.2 |
| | 3 | 1.78 | 305 | 10.3 |
| | 4 | 1.69 | 271 | 8.59 |
| Irgacure 369 | 1 | 1.65 | 402 | 12.6 |
| | 2 | 1.46 | 417 | 12.8 |
| | 3 | 1.48 | 480 | 6.20 |
| | 4 | 1.53 | 391 | 9.64 |

Table 12: TFC data for the 365nm LED with the urethane acrylate CN971A80

| <i>Initiator</i> | <i>Conc.</i> <i>(wt%)</i> | $t_{max}(s)$ | <i>Slope</i> | $c_{max} \times 10^5 (J/g^{\circ}C)$ |
|------------------|------------------------------|--------------|--------------|--------------------------------------|
| BP | 4 | 3.86 | 272 | 3.09 |
| | 5 | 3.97 | 251 | 3.42 |
| | 6 | 4.58 | 227 | 2.86 |
| ITX | 0.5 | 2.83 | 83 | 4.23 |
| | 1 | 2.77 | 53 | 3.64 |
| | 1.5 | 2.61 | 42 | 3.80 |
| TPO | 0.5 | 1.74 | 380 | 14.0 |
| | 1 | 1.58 | 493 | 14.5 |
| | 2 | 1.38 | 472 | 16.0 |
| | 3 | 1.58 | 426 | 13.9 |
| | 4 | 1.71 | 331 | 11.0 |
| Irgacure 369 | 1 | 1.51 | 493 | 14.5 |
| | 2 | 1.45 | 423 | 14.6 |
| | 3 | 1.55 | 394 | 12.8 |
| | 4 | 1.63 | 310 | 11.5 |

Conclusion

- The TFC-9000 is able to measure curability with ease and efficiency
- LED Curing is viable and achievable with the correct photoinitiators for the wavelength