Exploring new ways to solve old problems; the hunt for a replacement to the 80°C self-heat test

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80°C self-heat test

Extreme Heat Flow Calorimetry

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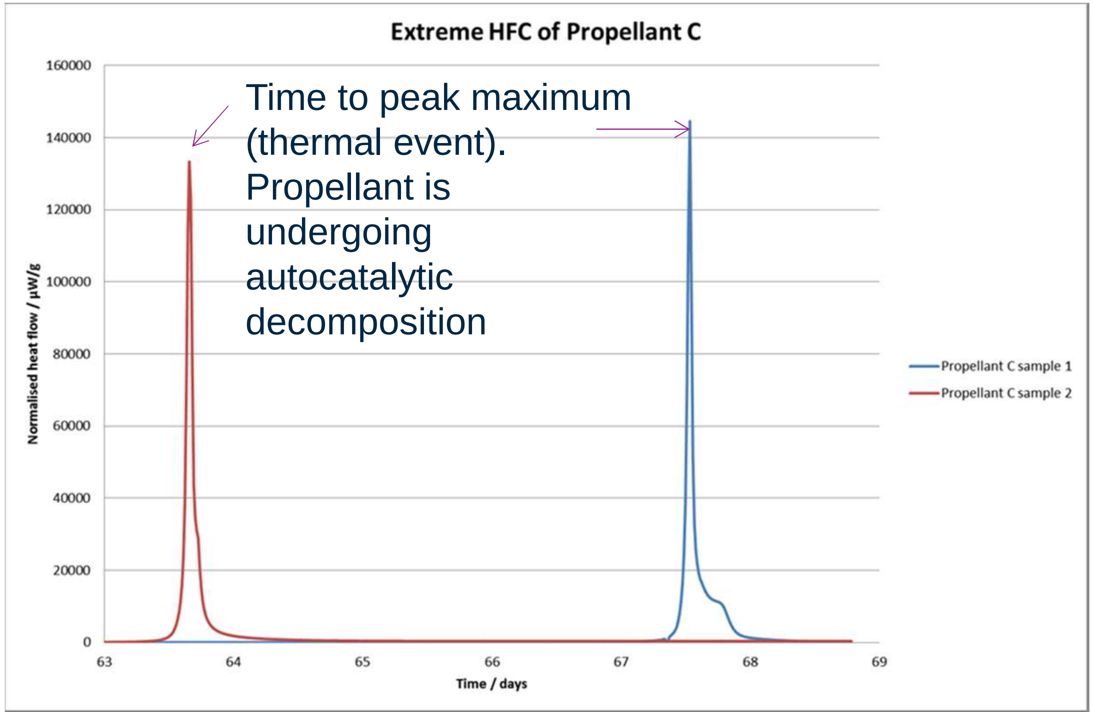
Typical extreme HFC results



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Aims

- Could “extreme HFC” replace the 80°C self-heat test?
- Can extreme HFC detect changes upon ageing for a particular nitrate ester based propellant?

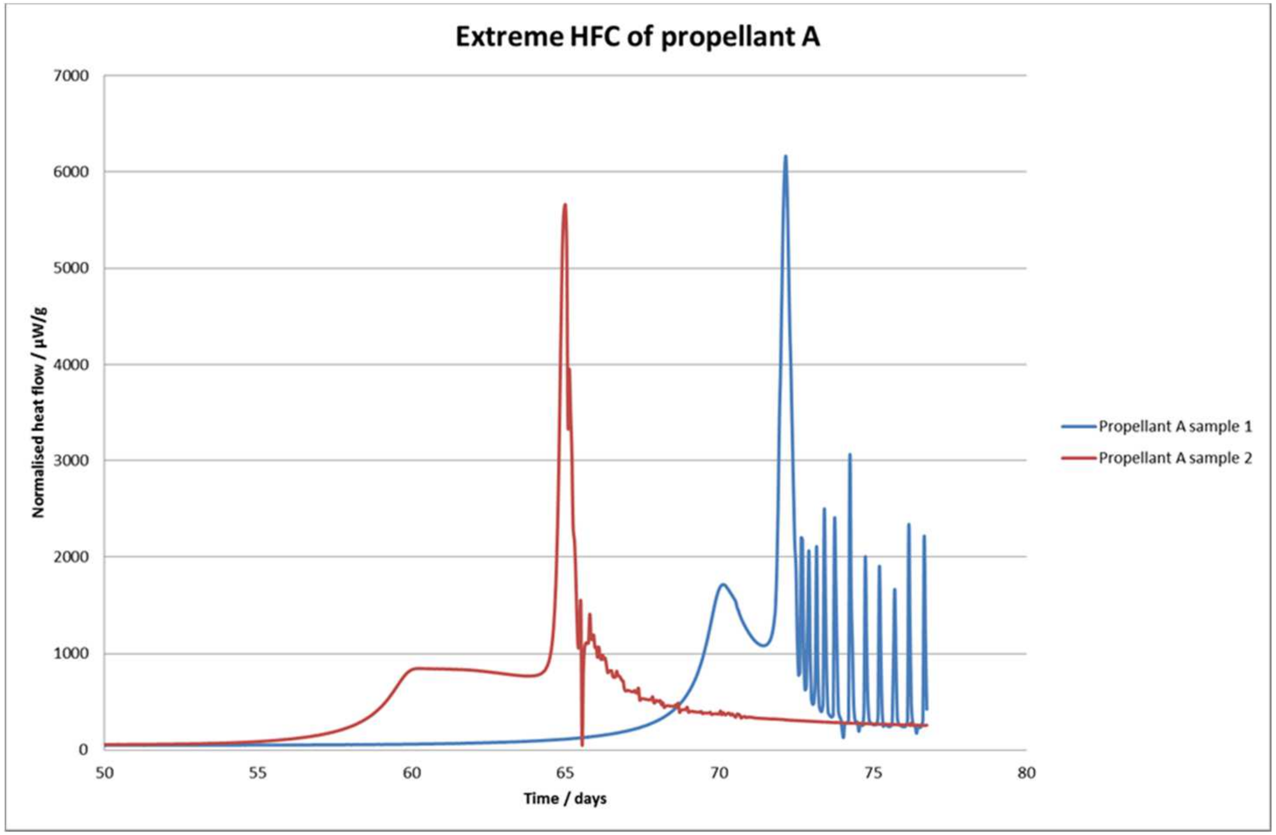
Samples

Sample	Details	Form of sample
Propellant A	Simple nitrate ester containing propellant	Propellant swarfed into small needle like flakes
Propellant B	Simple nitrate ester containing propellant which has been deliberately designed to be unstable for research purposes	Small grains
Propellant C	Complex hybrid nitrate ester containing propellant	Chopped into small cubes
Tetryl	NATO grade tetryl	Coarse powder

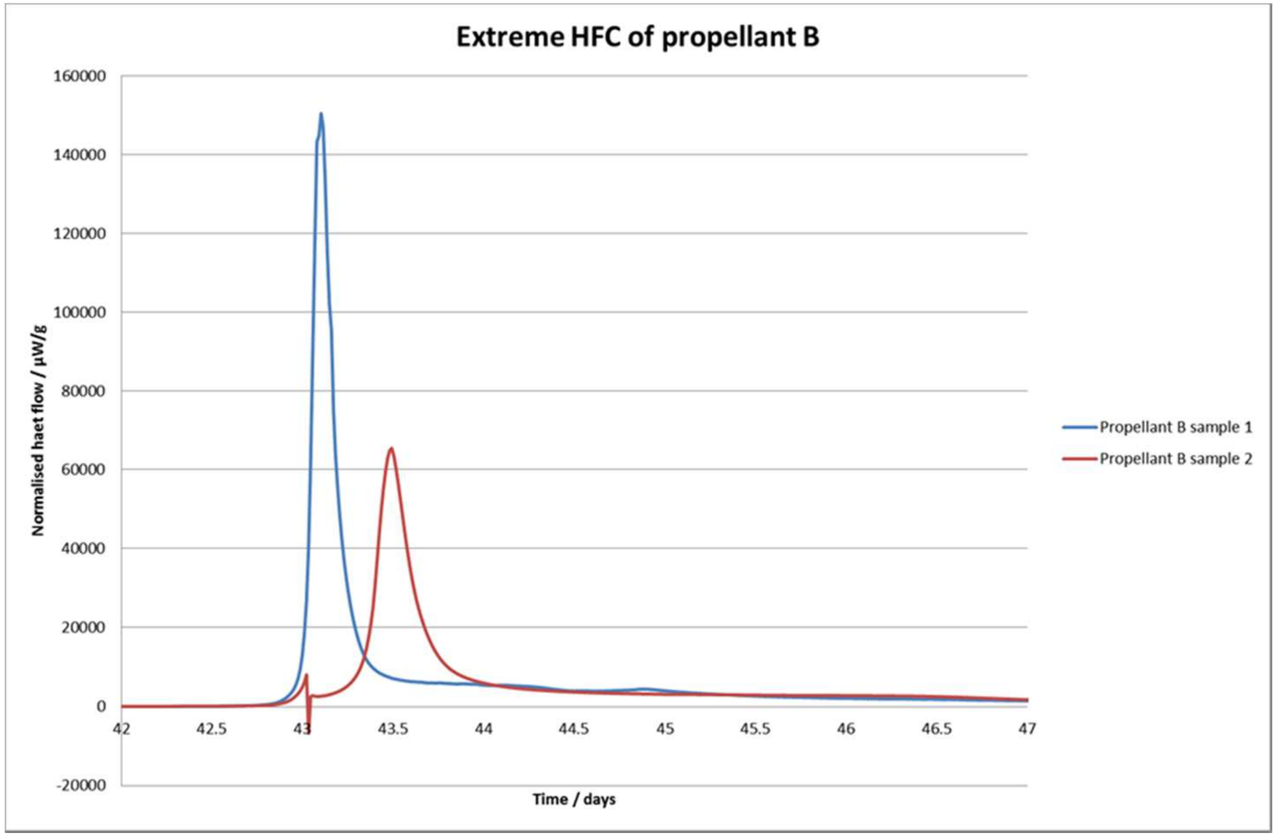
Ageing of propellant C

Ageing time at 80°C	Notes
7 days	Propellant aged in insolation. No extreme HFC
7 days	Propellant aged with boron potassium nitrate (BPN) but the two materials were kept separate from each other as they were in individual glass vials and separated by a distance of about 2cm. No extreme HFC
14, 21 and 28 days	Propellant aged in insolation
14, 21 and 28 days	Propellant aged with BPN but the two materials were kept separate from each other as they were in individual glass vials and separated by a distance of about 2cm

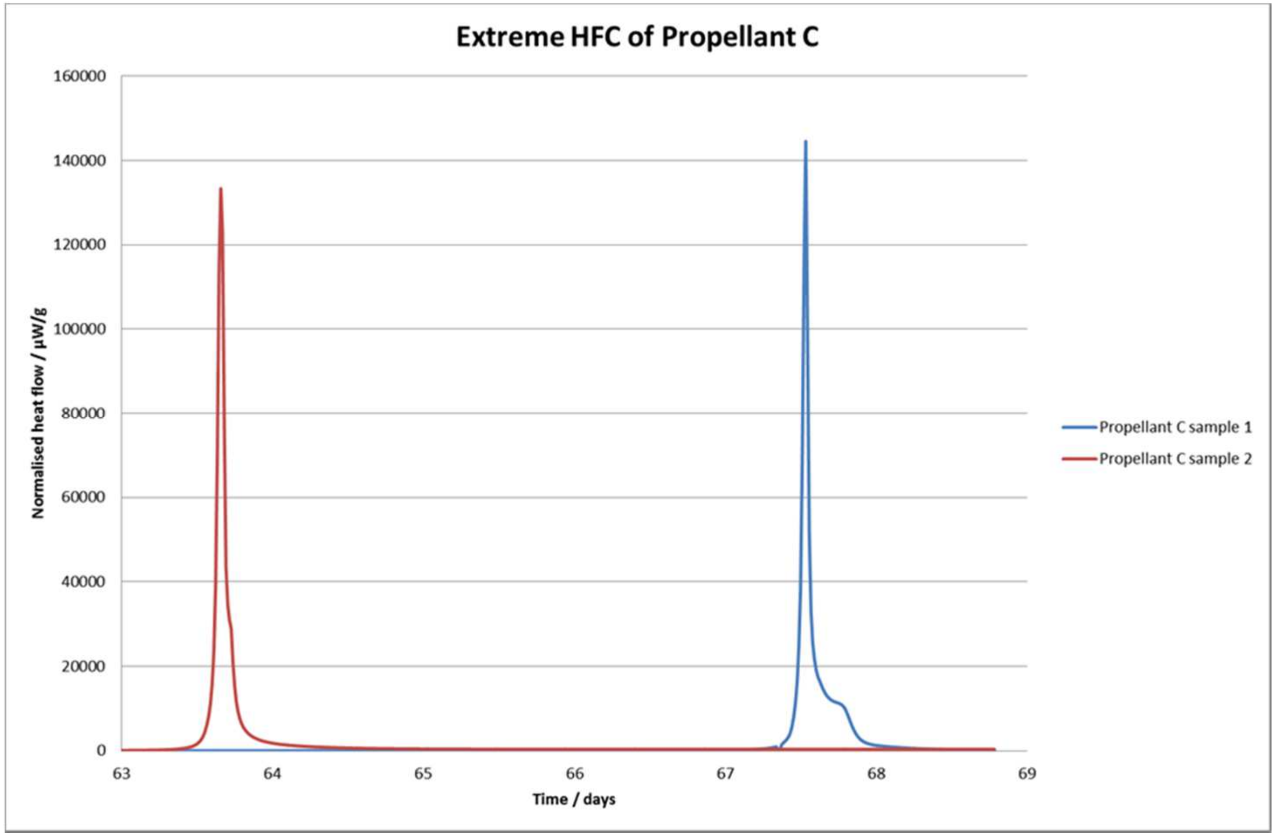
Extreme HFC at 80°C results – propellant A



Extreme HFC at 80°C results – propellant B



Extreme HFC at 80°C results – propellant C



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Summary of 80°C data

Sample	Time to maxima of first thermal event (days)	80°C self-heat test result (days)
Propellant A	70.0, 65.0	63.8, 63.8
Propellant B	43.1, 43.5	40 ± 2
Propellant C	63.7, 67.5	61.0 – 64.2

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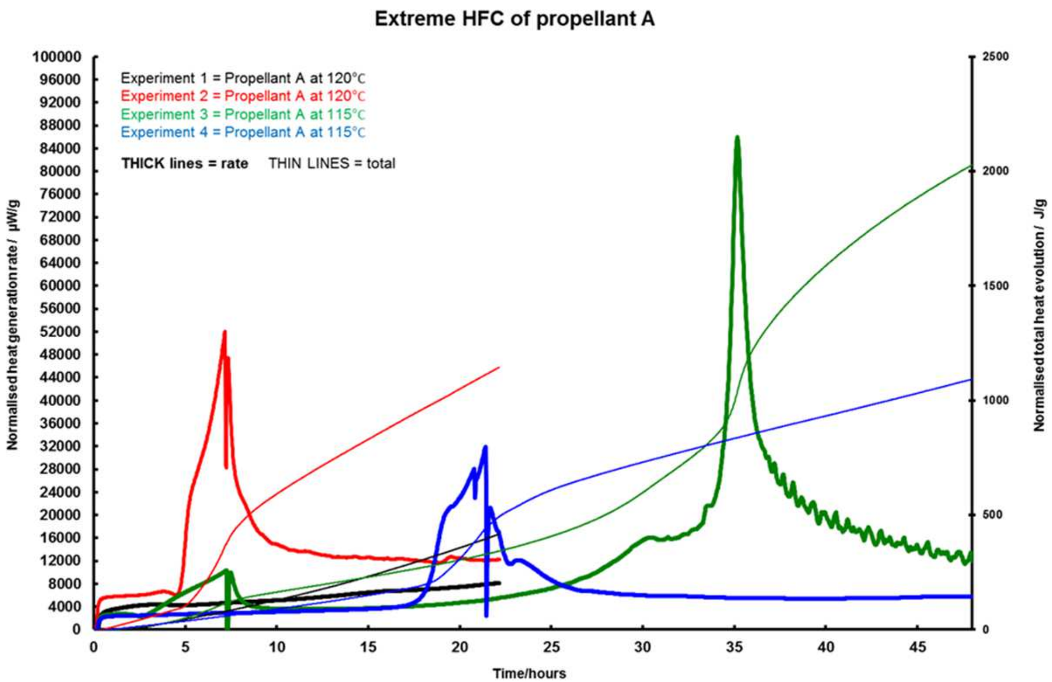
Extreme HFC at 120°C and 115°C results – propellant A

Time to maxima of first thermal event at 120°C (hours)	Time to maxima of first thermal event at 115°C (hours
5.5, no result	19.6, 30.3



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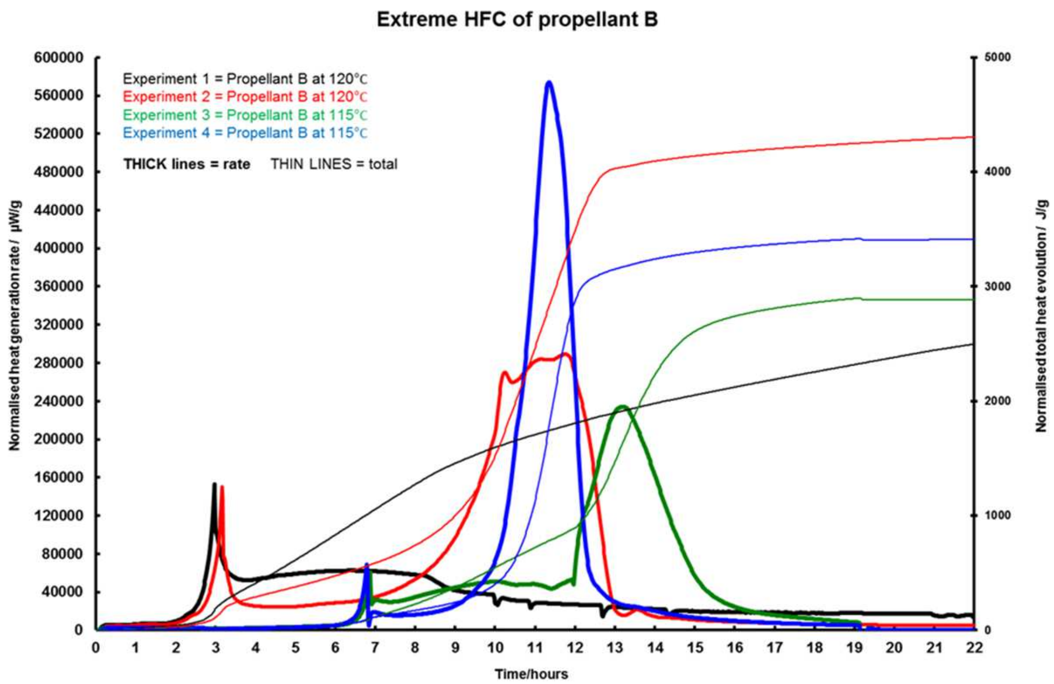
Extreme HFC at 120°C and 115°C results – propellant B

Time to maxima of first thermal event at 120°C (hours)	Time to maxima of first thermal event at 115°C (hours
3.0, 3.2	6.9, 6.8



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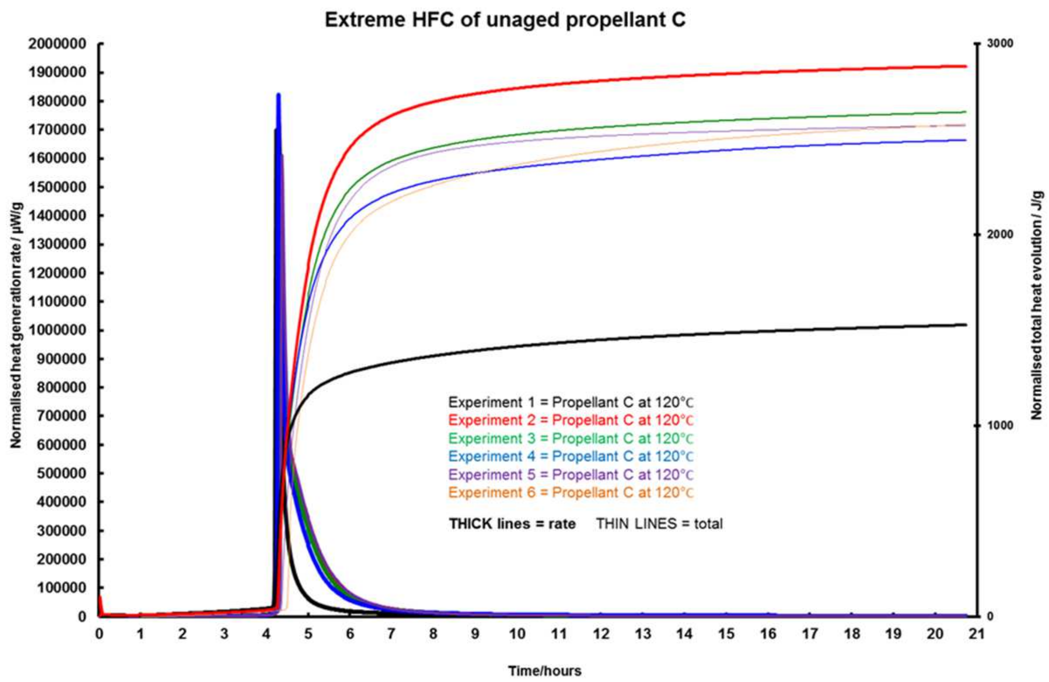
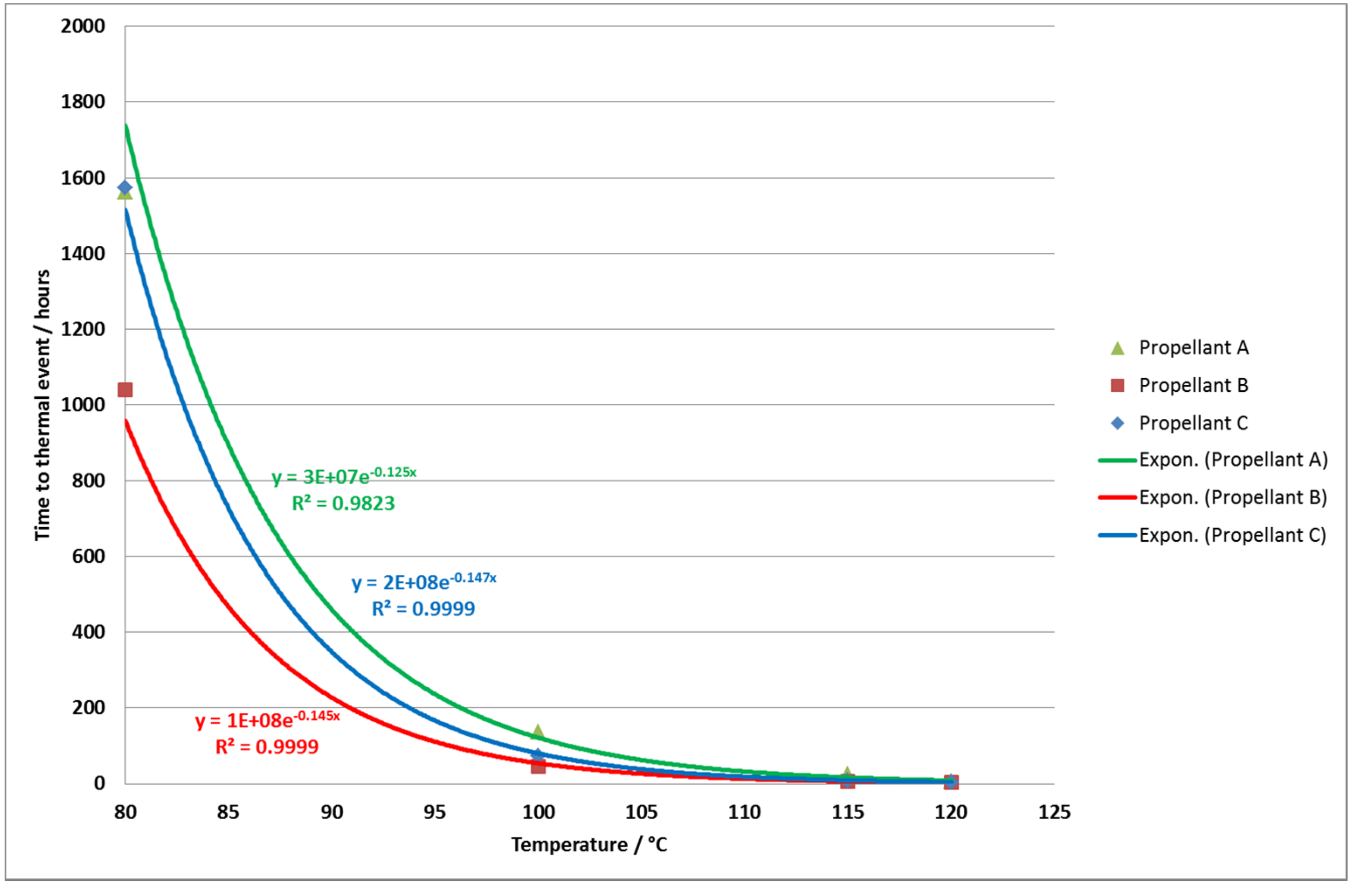
Extreme HFC at 120°C and 115°C results – propellant C

Time to maxima of first thermal event at 120°C (hours)	Time to maxima of first thermal event at 115°C (hours
4.4 ± 0.10	8.10 ± 1.34



Comparison of all results



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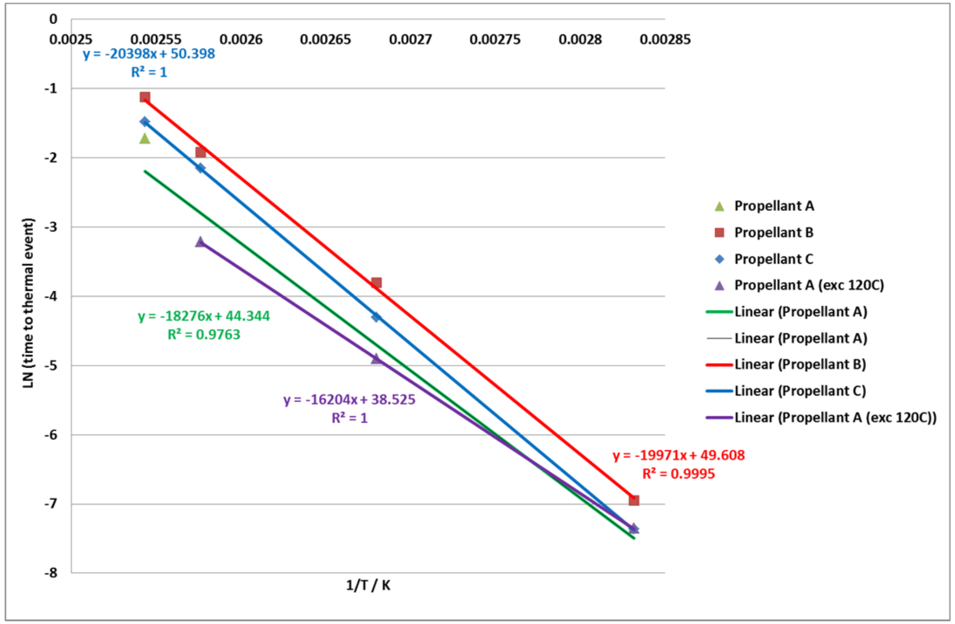
Comparison of all results

Propellant	Apparent activation energy ($kJMol^{-1}$)
A	153 (all data) 135 (excluding 120°C data)
B	166
C	170

$$K = Ae^{\frac{-EA}{RT}}$$



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Predicted time vs measured time to decomposition at 80°C

Sample	Predicted time to decomposition at 80° C (hours)	Measured time to decomposition at 80° C (hours)
Propellant A	4040	1562
Propellant B	857	1039
Propellant C	1723	1574

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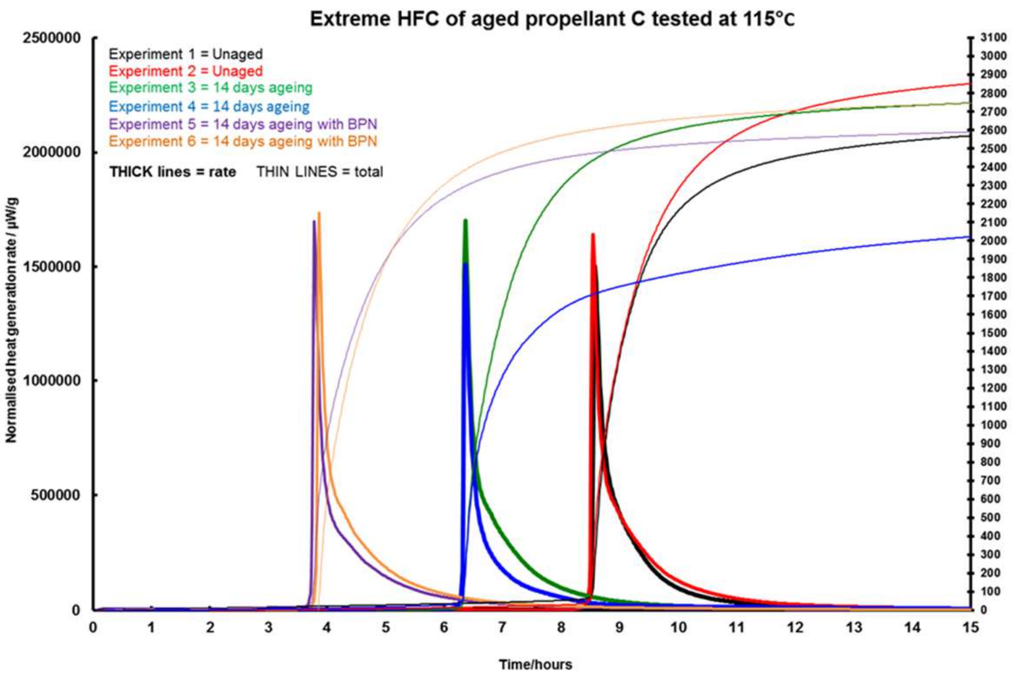
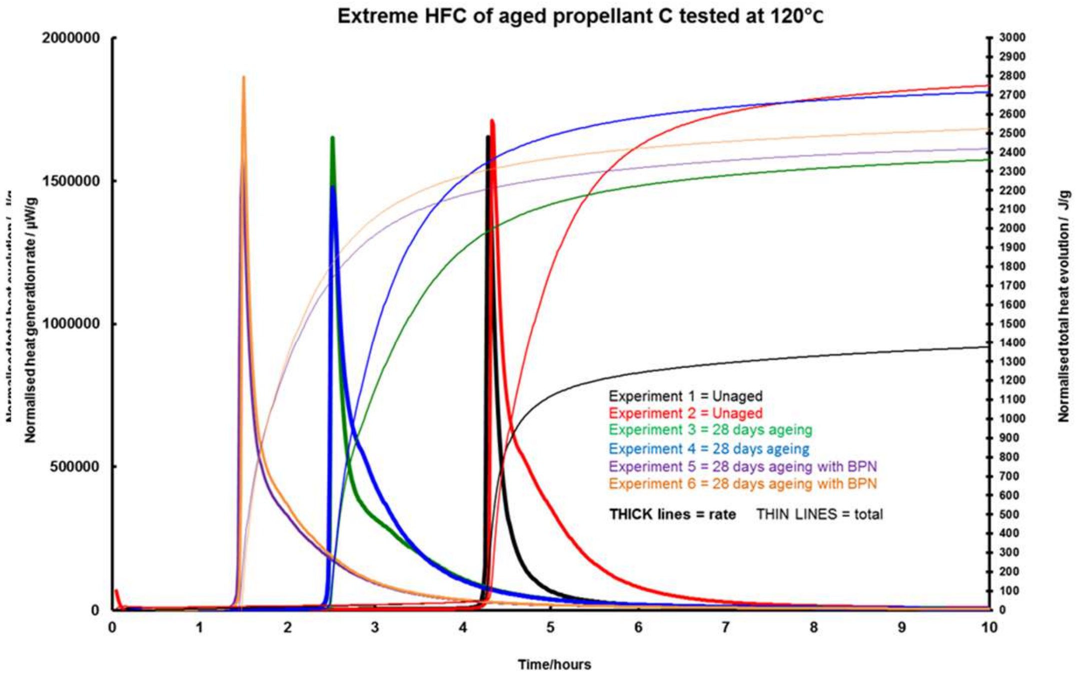
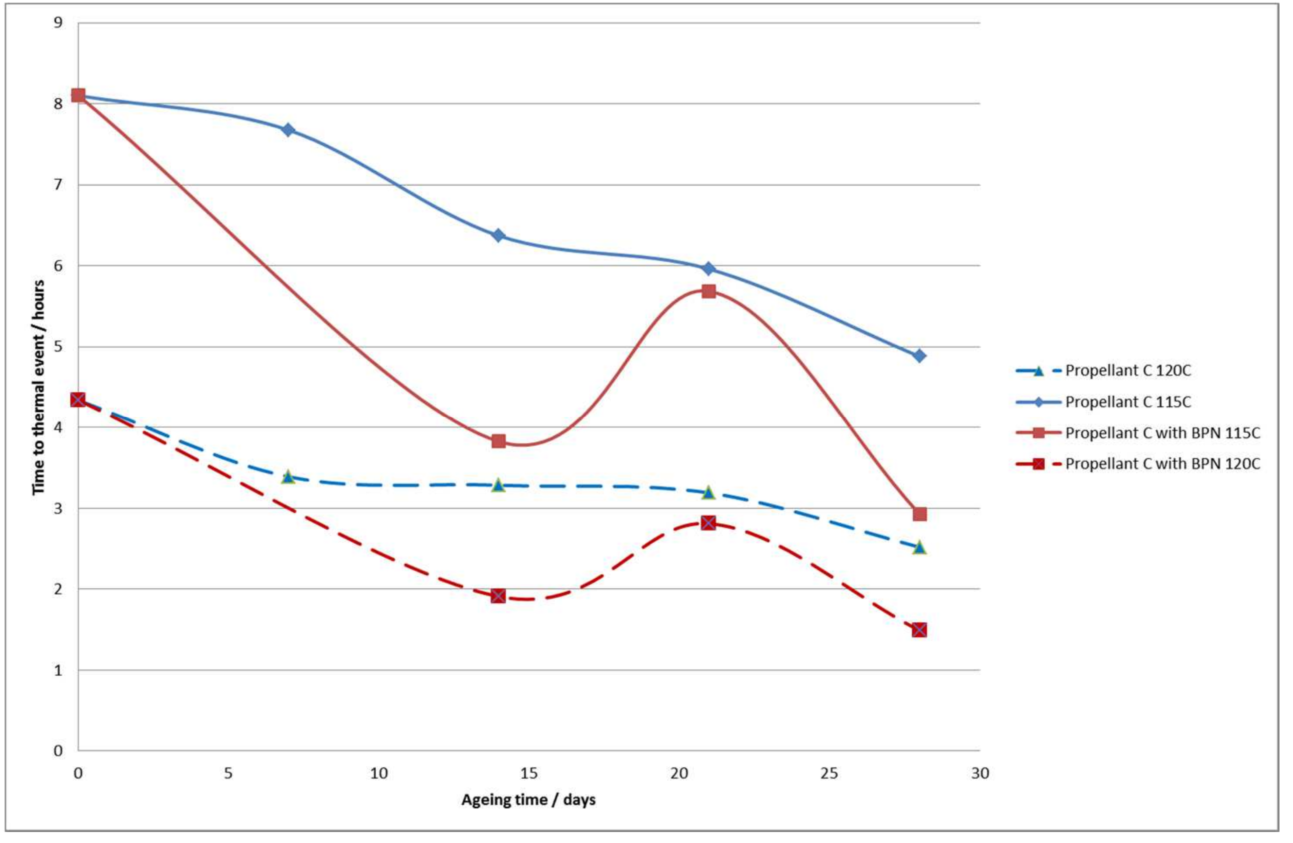
Extreme HFC – aged Propellant C samples





Extreme HFC – summary of aged Propellant C samples data



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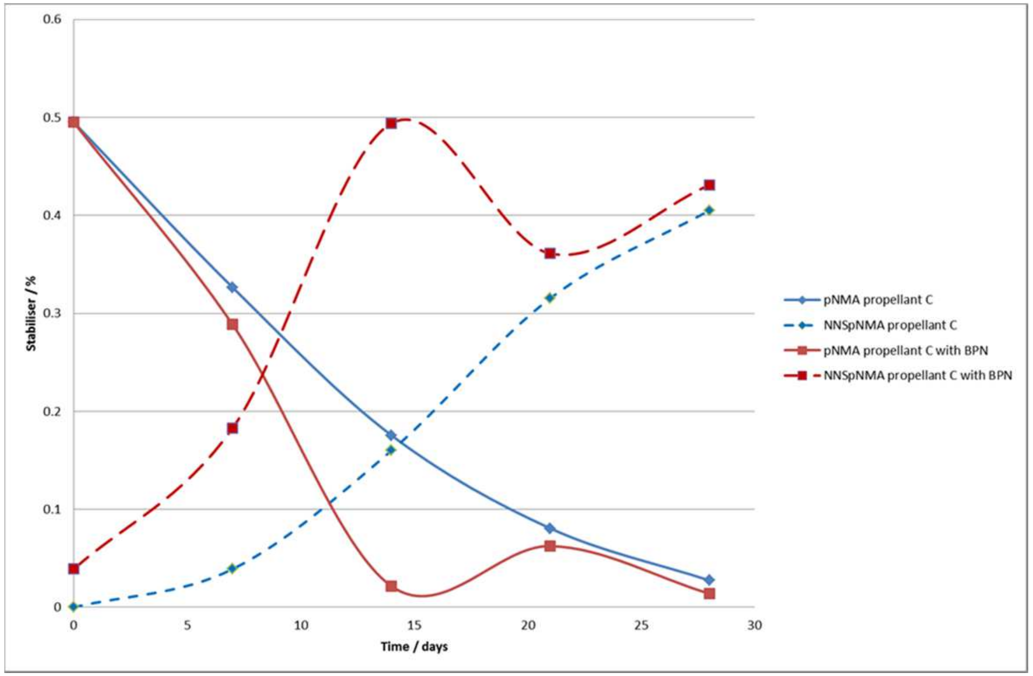
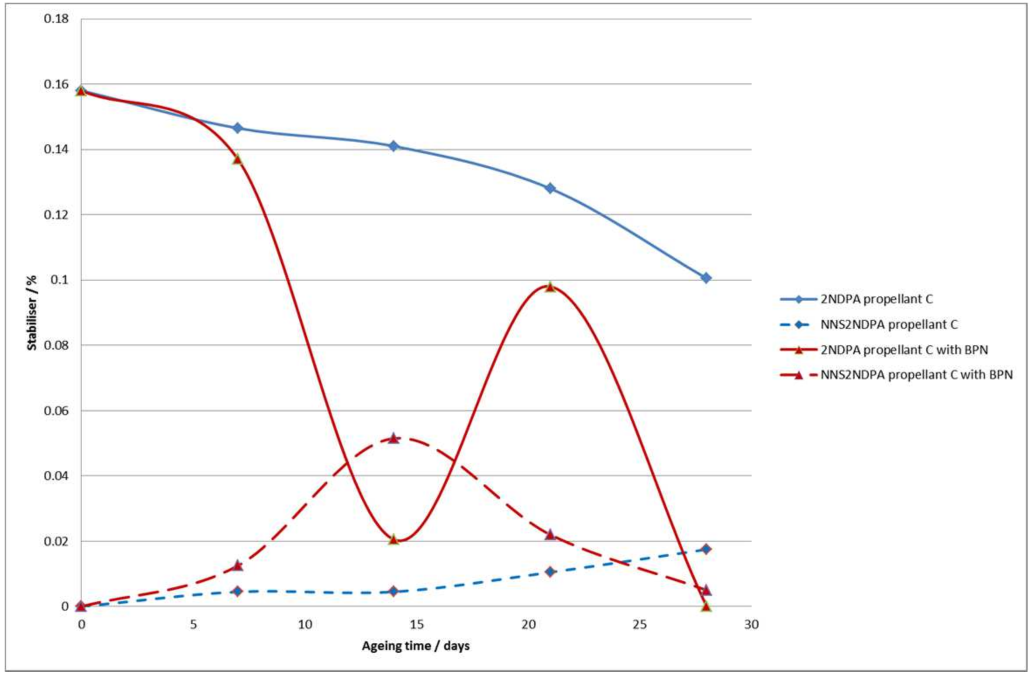
Aged propellant C samples – stabiliser content

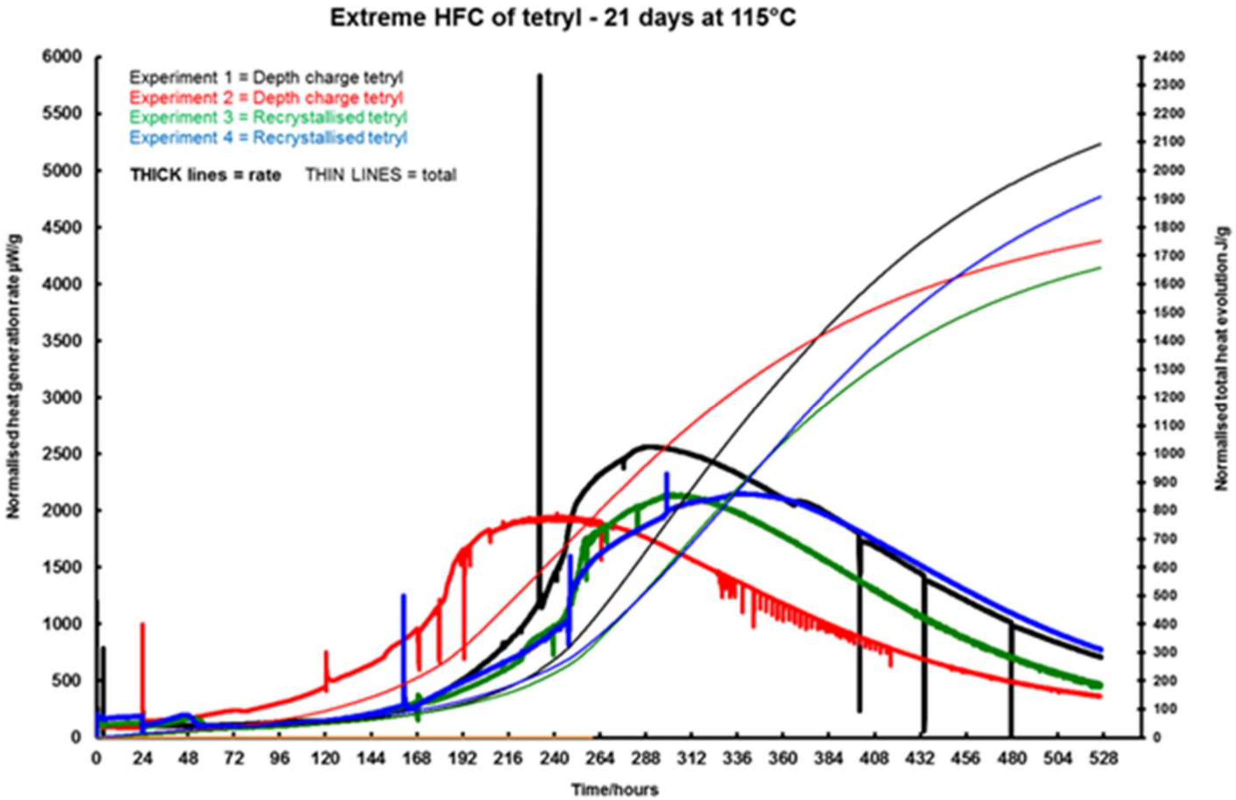


pNMA and NNSpNMA



2-NDPA and NNS-2-NDPA

Extreme HFC results - tetryl



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Conclusions

- Extreme HFC can determine the thermal stability of nitrate ester based propellants
  - The results from the extreme HFC tests compare well with the data from the 80°C self-heat test
- Full decomposition of materials such as tetryl can be followed but technique did not identify any differences between samples and gas generation was an issue
- Extreme HFC can detect changes upon ageing in a particular propellant
  - Additional research is required on a greater range of propellants to confirm this.
  - Trials should be conducted to see if extreme HFC can be conducted at 115°C rather than 80°C in order to increase the throughput of samples using this technique
  - The influence on the form of a sample on the results from extreme HFC should also be investigated
- Extreme HFC shows great promise as potential replacement techniques for the 80°C self-heat test.
  - Future work should concentrate on determining if this is the case for a range of nitrate ester based compositions in the UK inventory

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