Stepped combustion technique for isotope analysis of SW N in DLC on Si.

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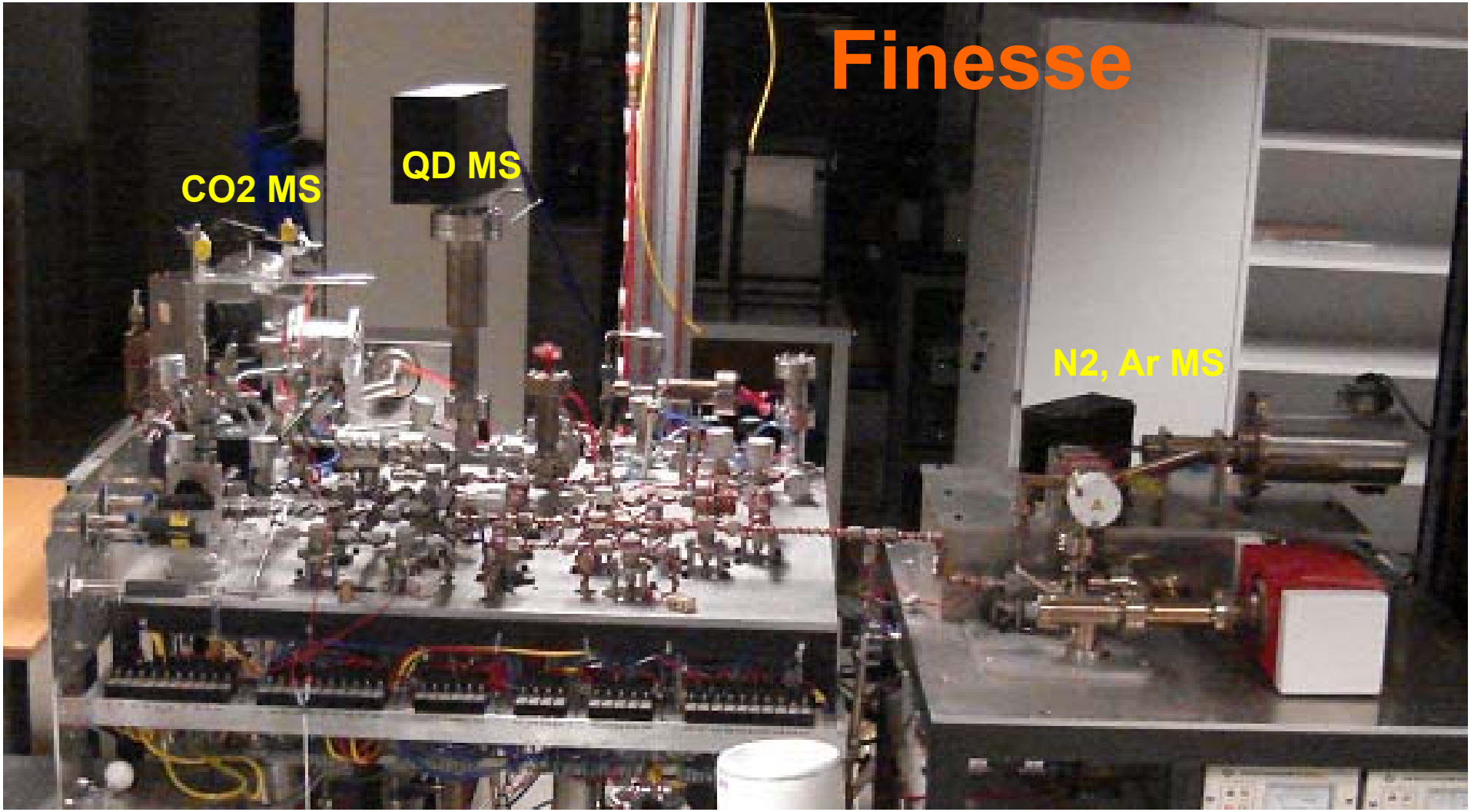
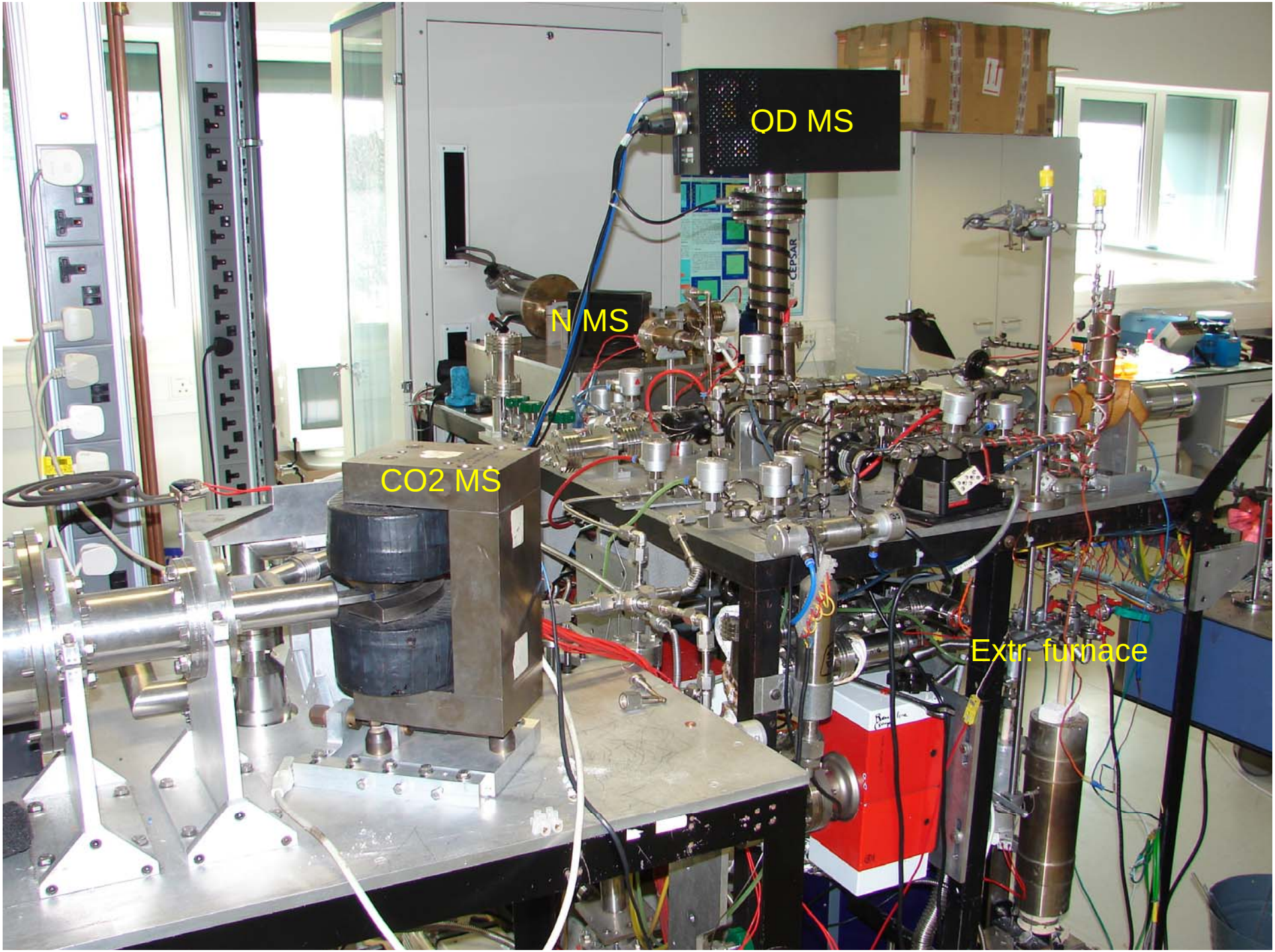
D. S. Burnett

Concentrator targets

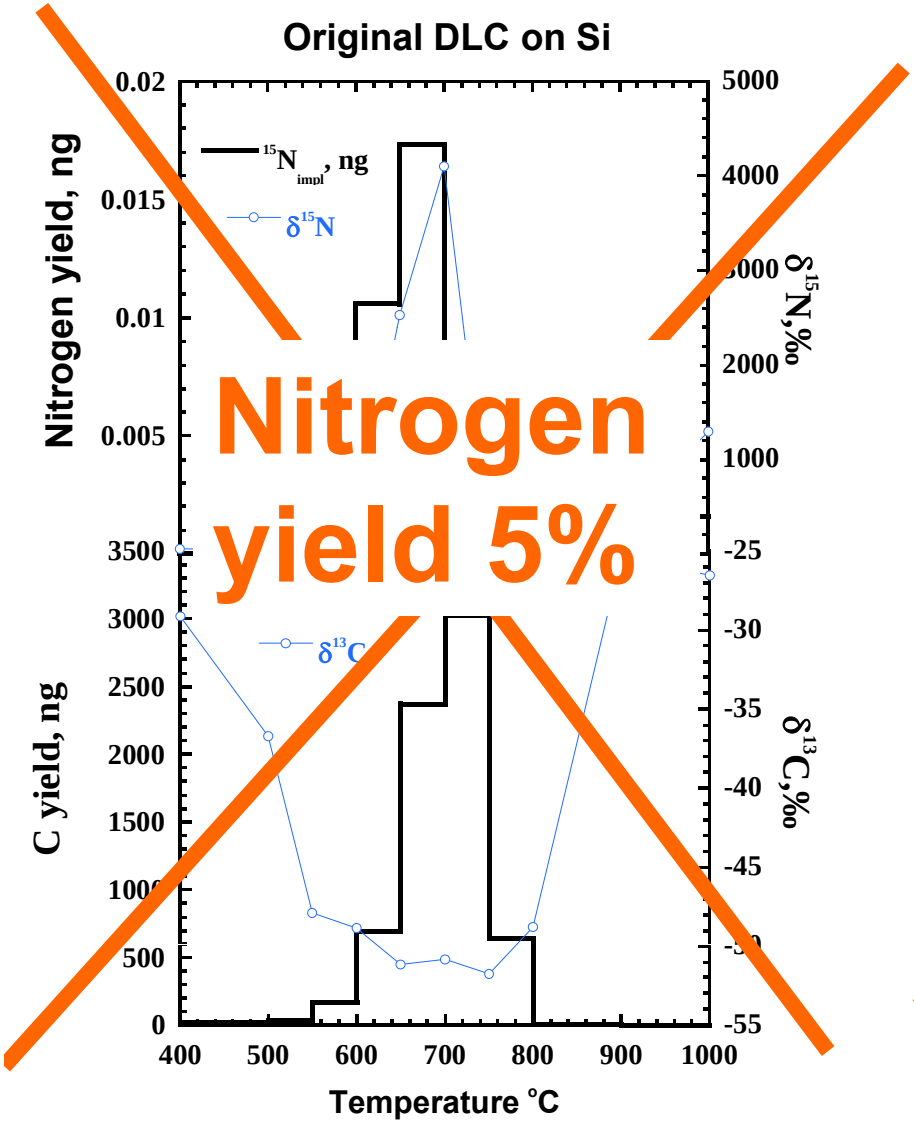
Experimental technique

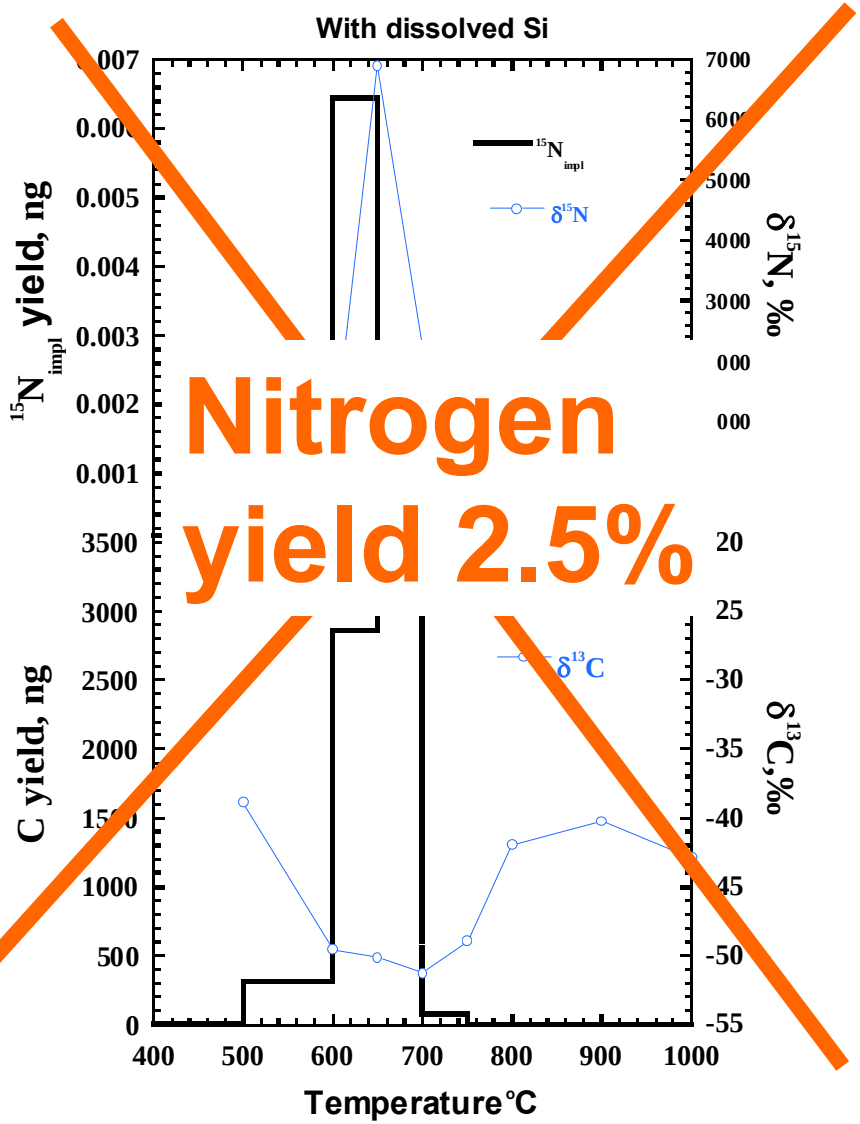



QD MS
N MS
CO2 MS
Extr. furnace

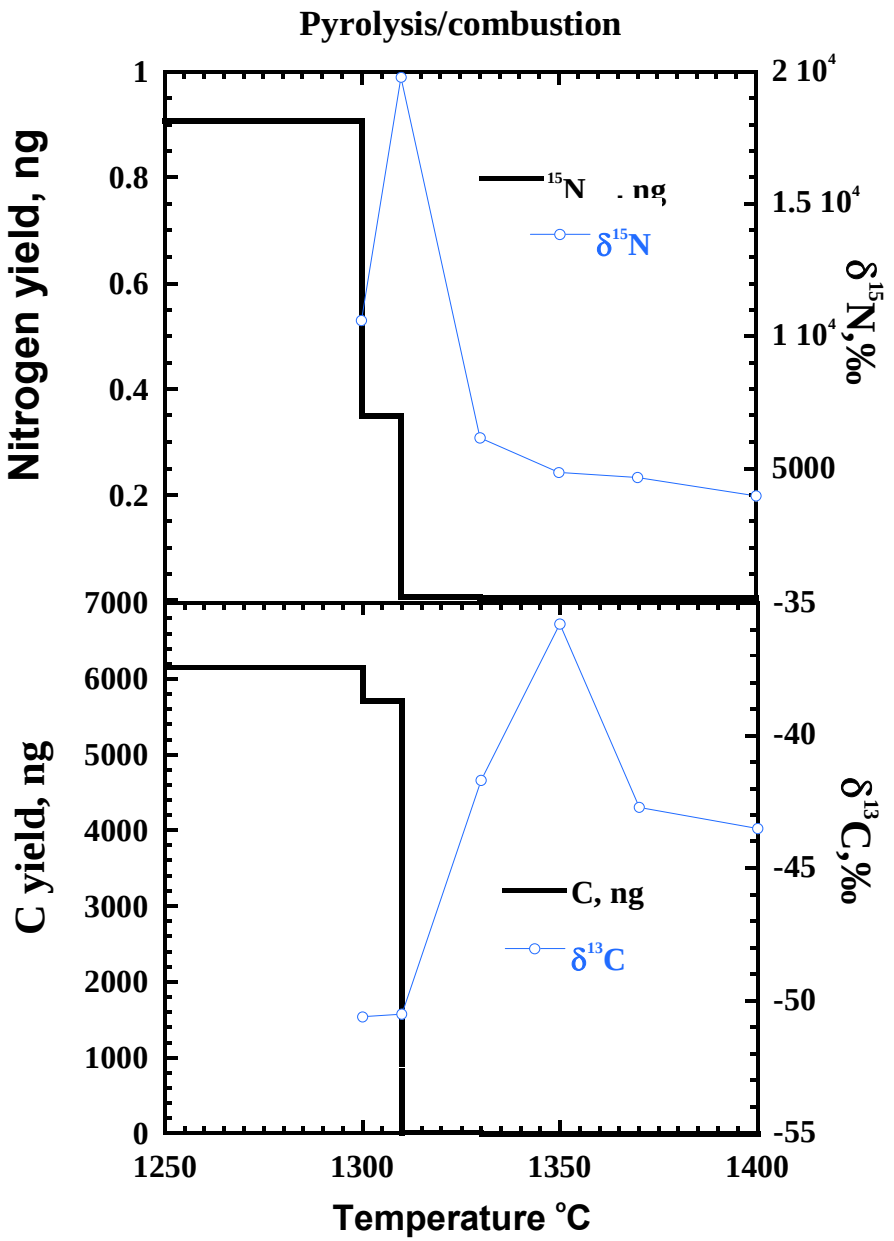
Nitrogen yield problem

Samples with implanted ^{15}N.





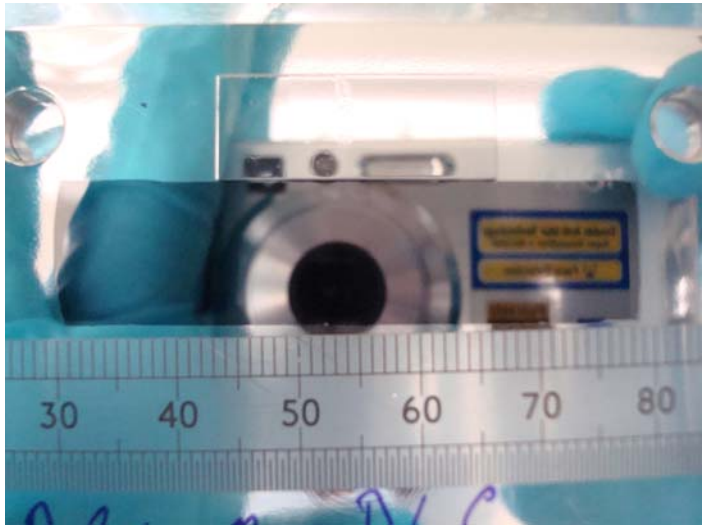
Nitrogen yield solution

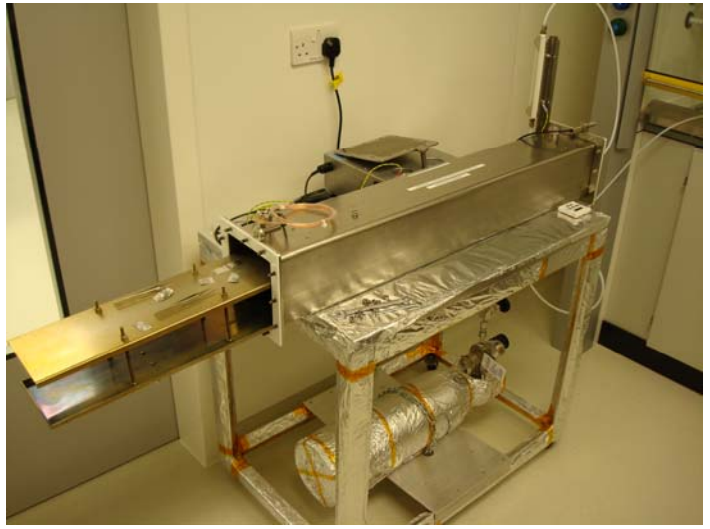


Nitrogen yield ≈100%

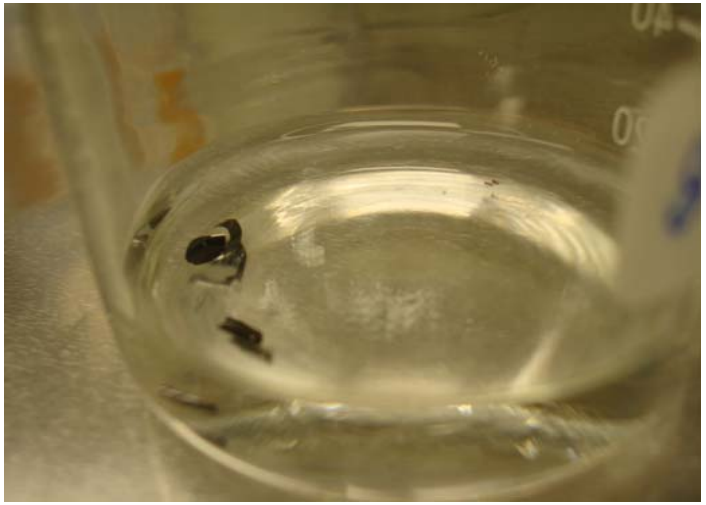
Chemical and cleaning procedures

1. Cleaning of the original DOS in UV/O_3 chamber





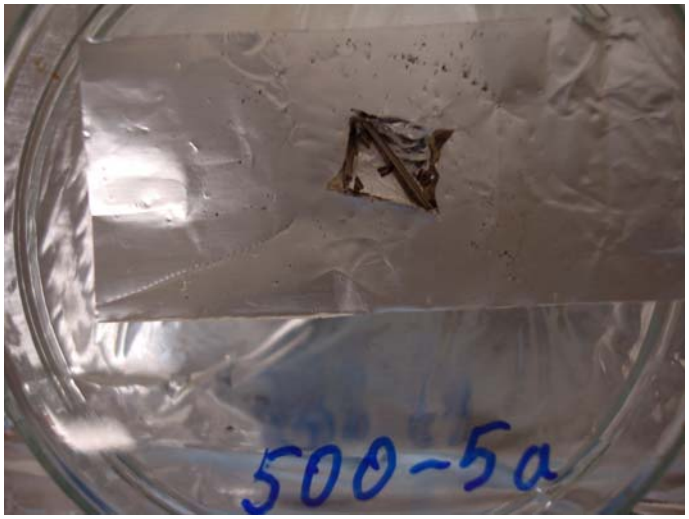
2. Cutting the original plate into smaller pieces and dissolving Si in KOH



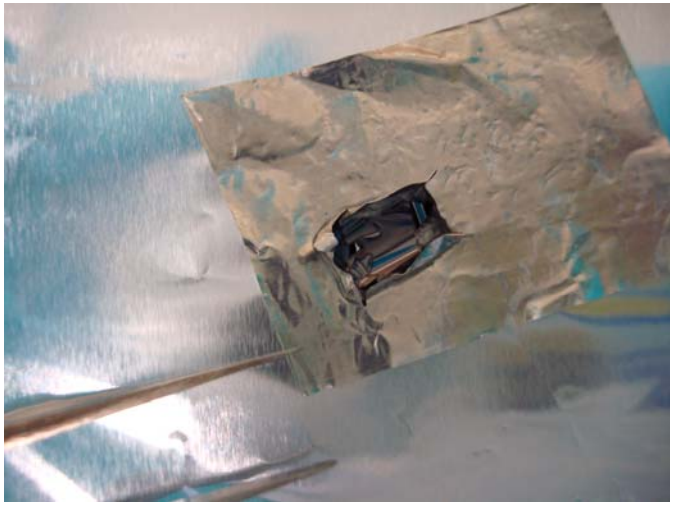
Chemical and cleaning procedures

3. Cleaning the residual DLC film in H_2O_2 and then in isopropanol and acetone

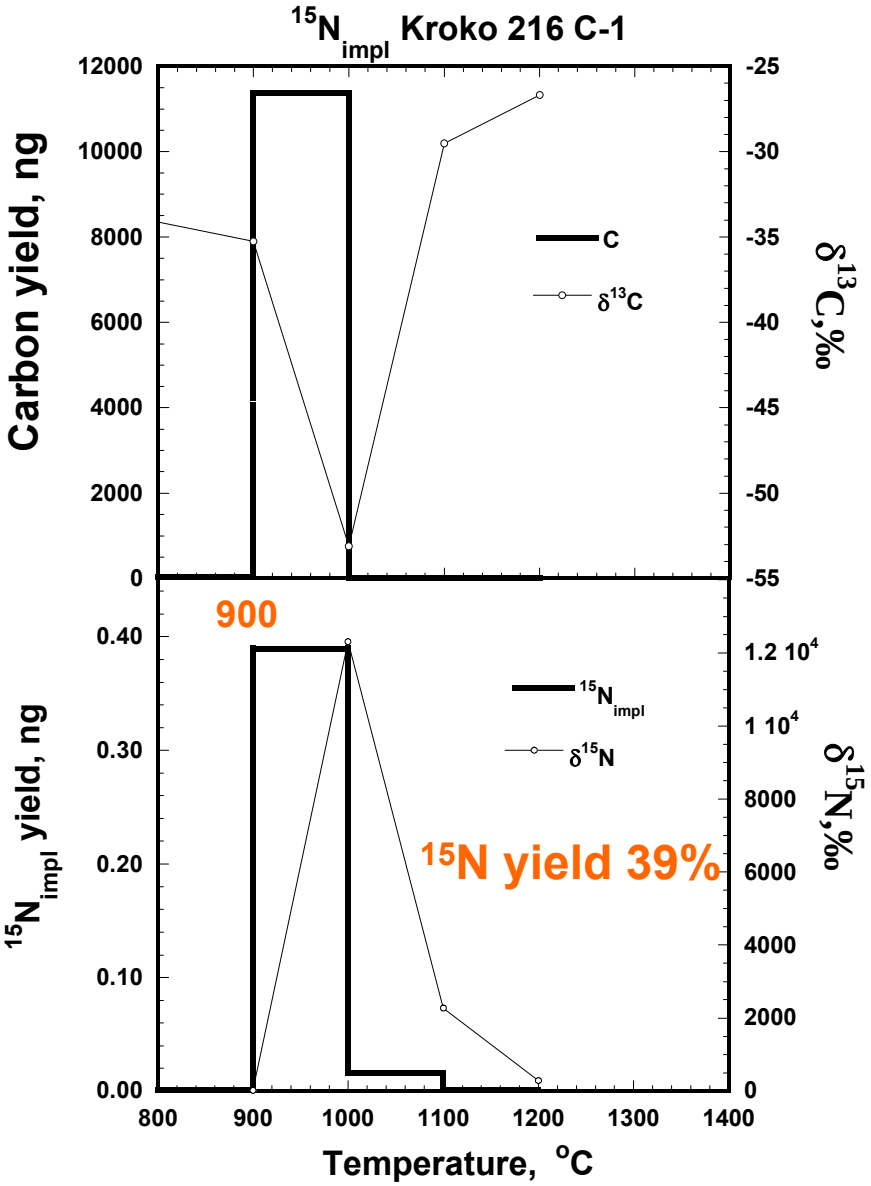
4. Putting the film into cleaned Pt foil and cleaning in UV/O_3 chamber

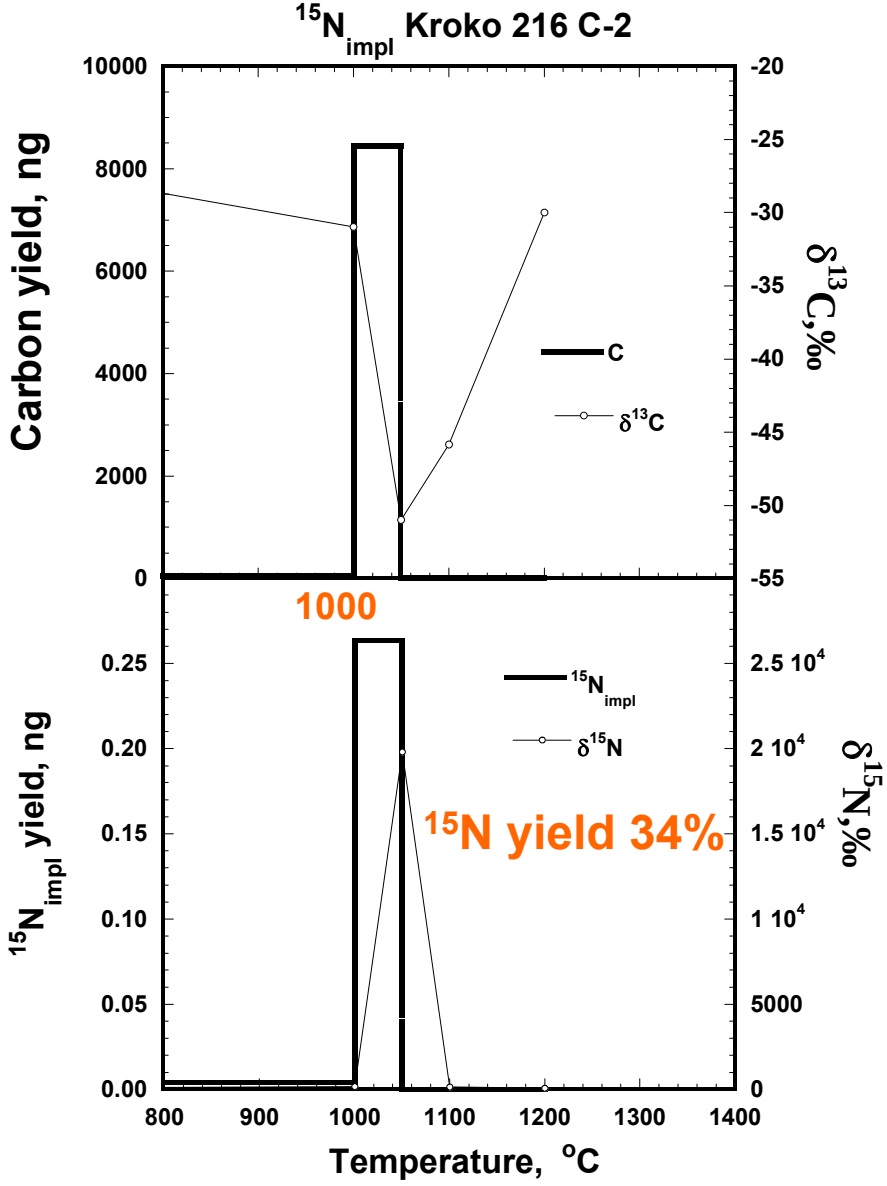


5. Compressing the foil with the DLC film into small container and placing it into vacuum system

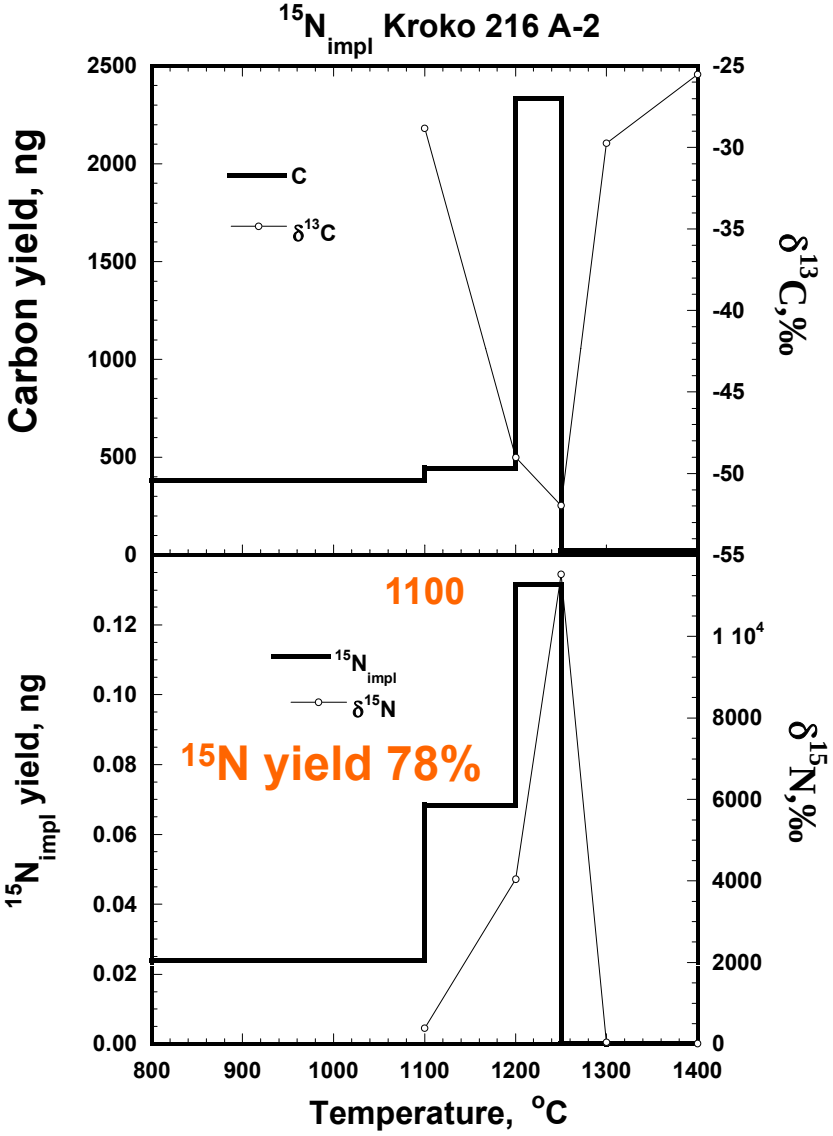


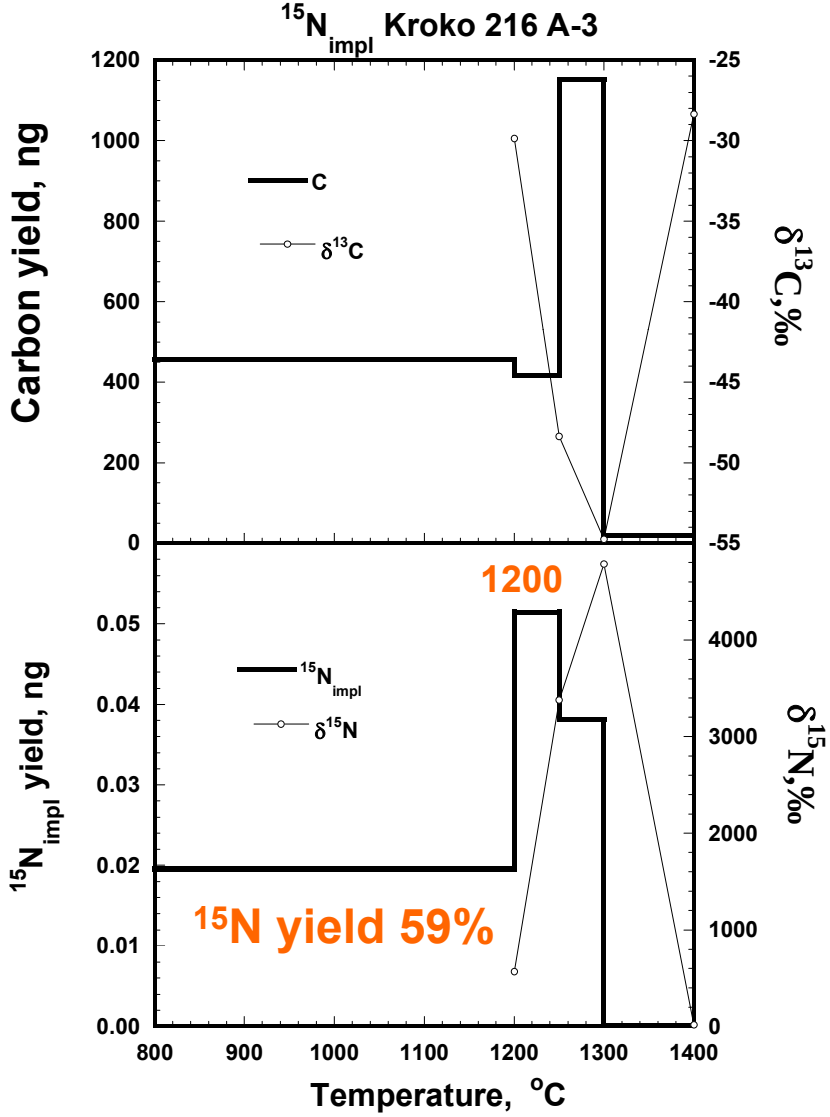
N yield and release temperature



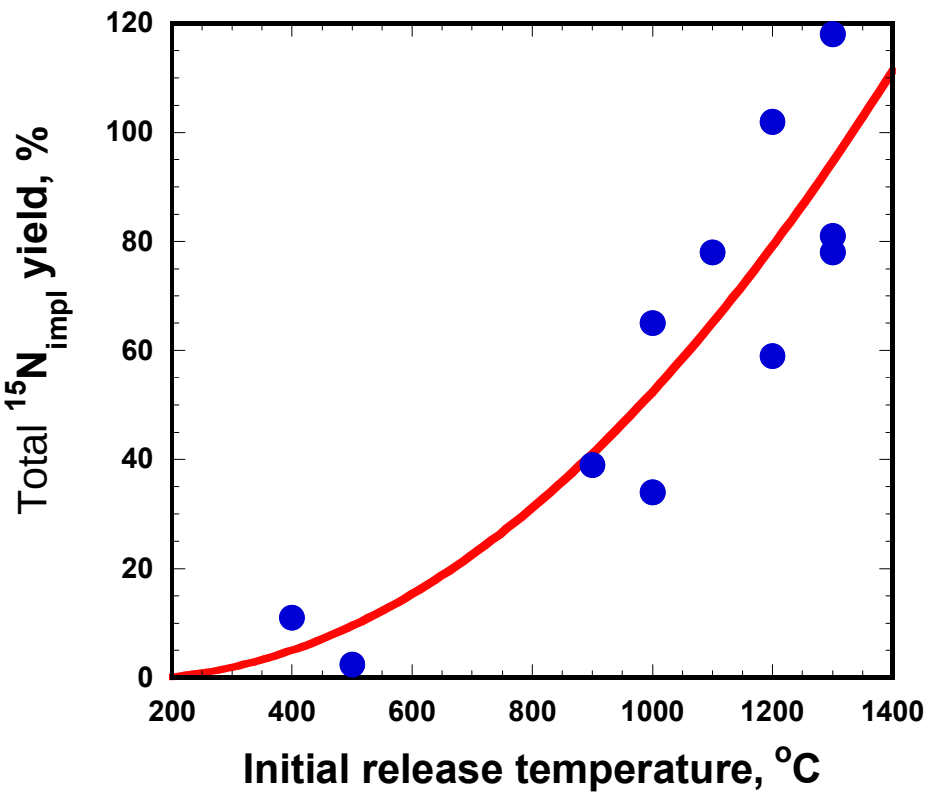


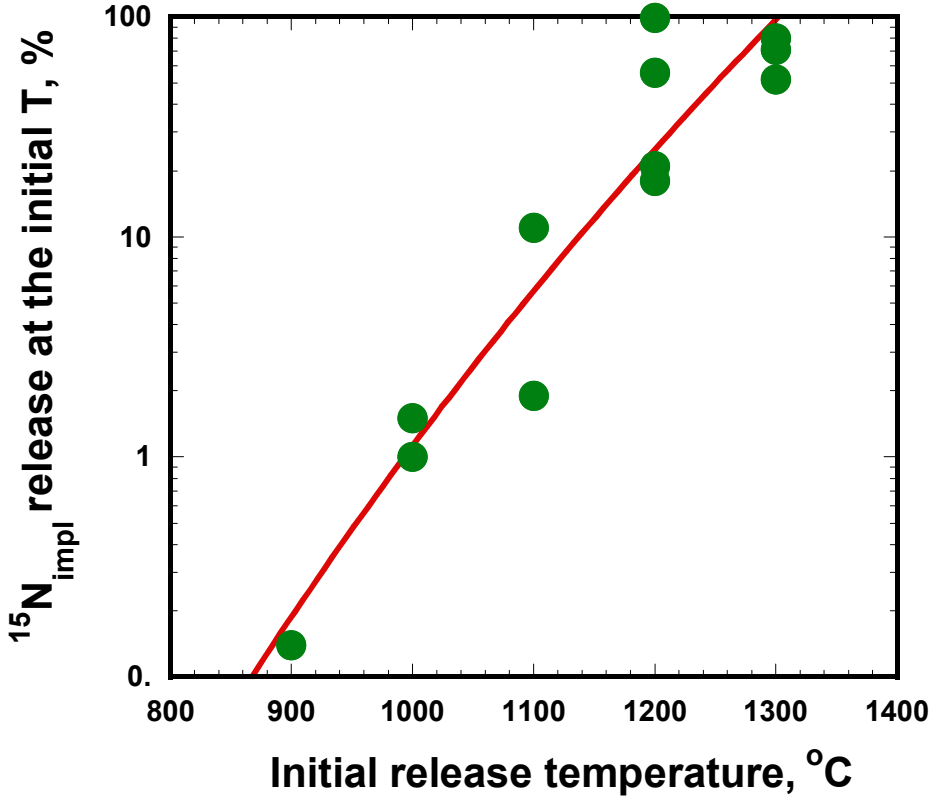
N yield and release temperature



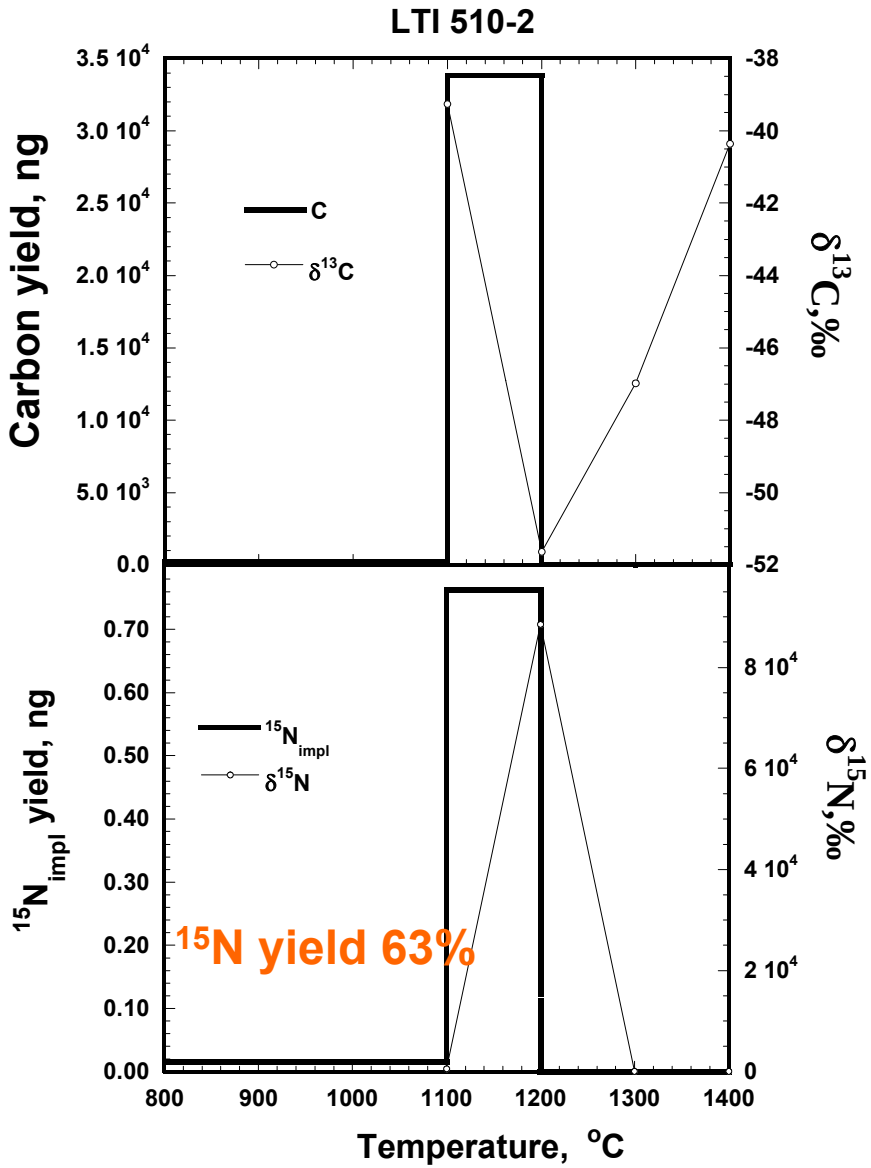


Adjusting release temperature

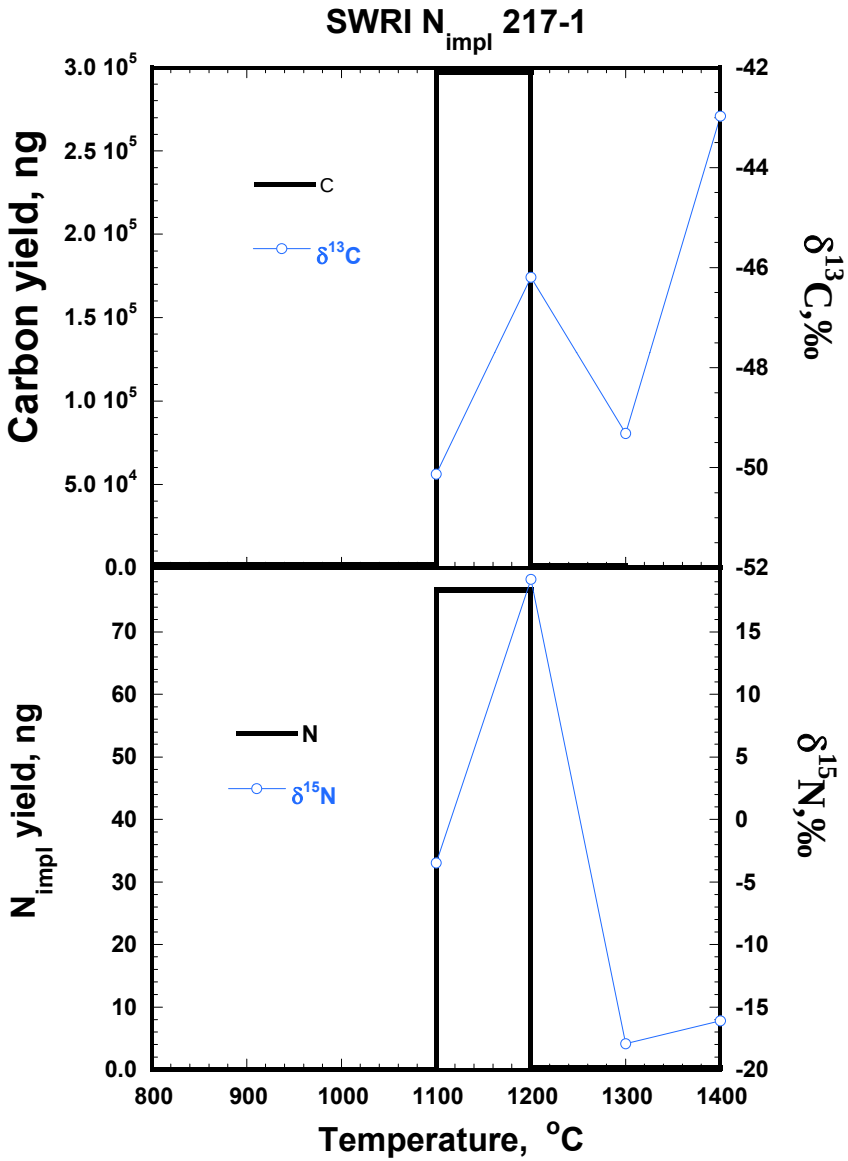


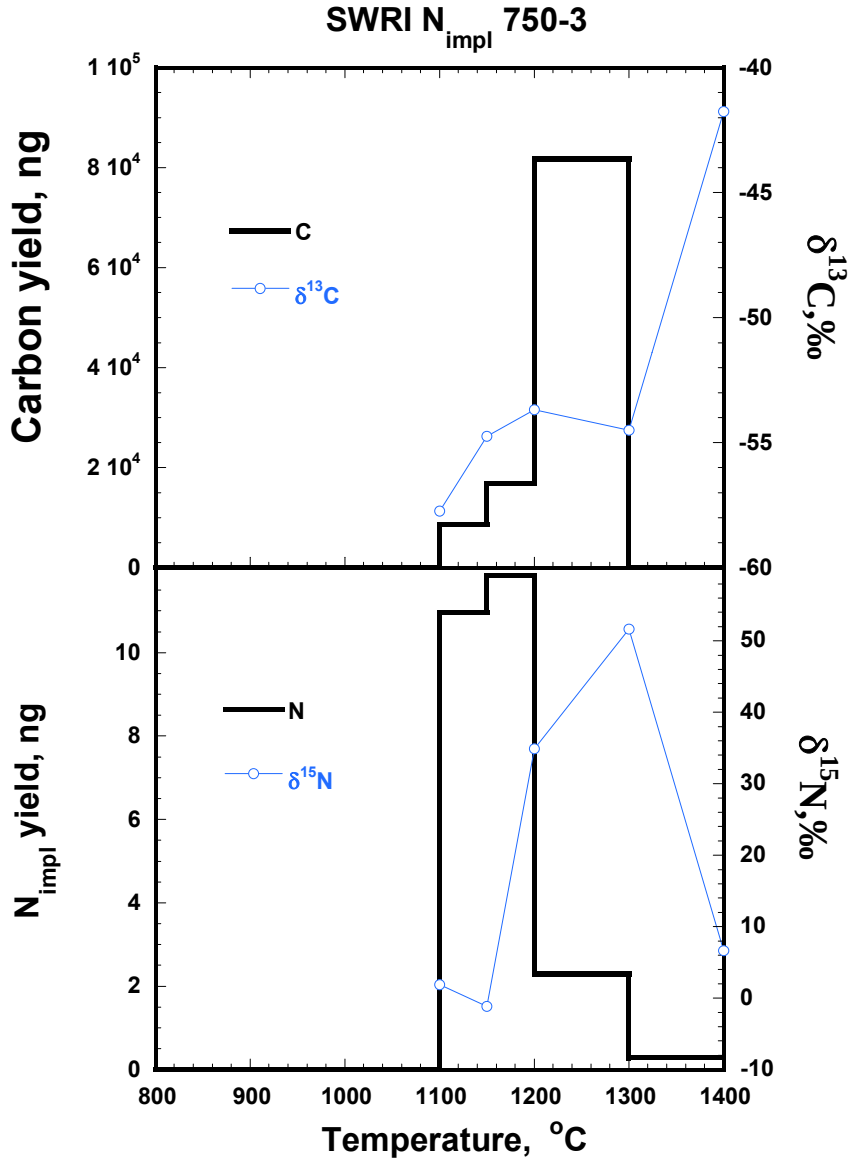


Long-term implant

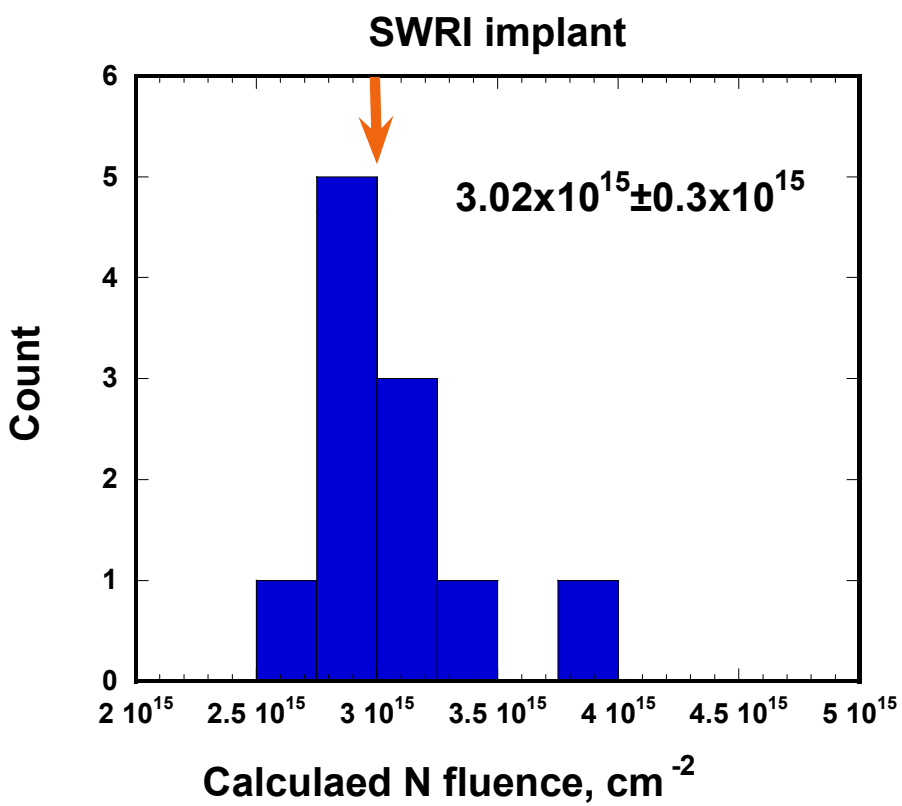


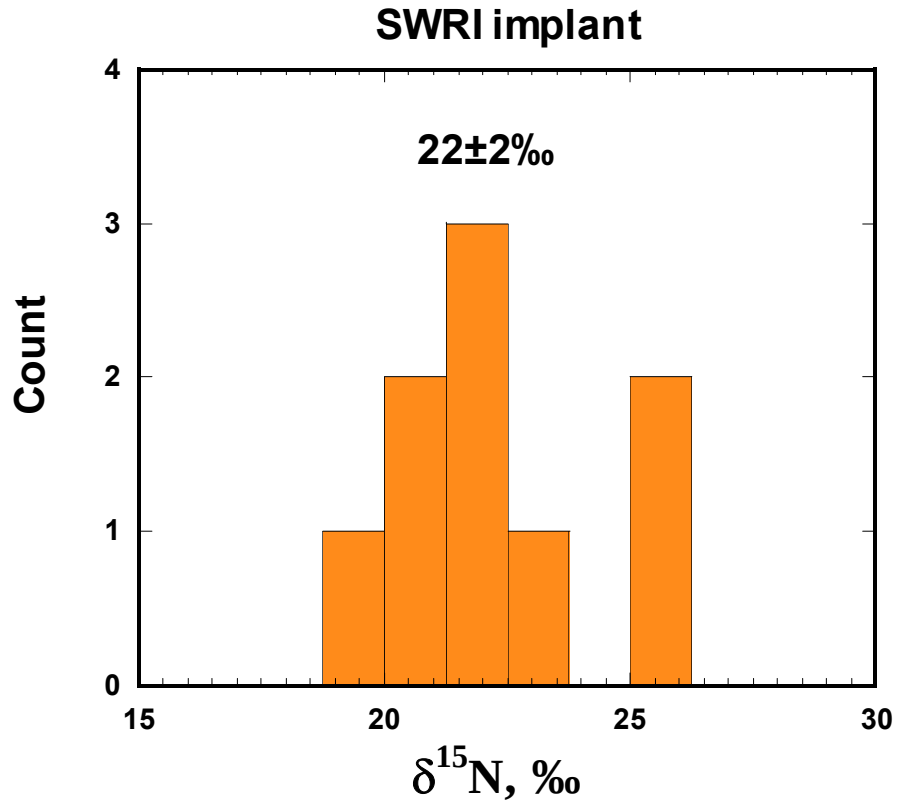
SWRI samples with implanted isotopically normal N





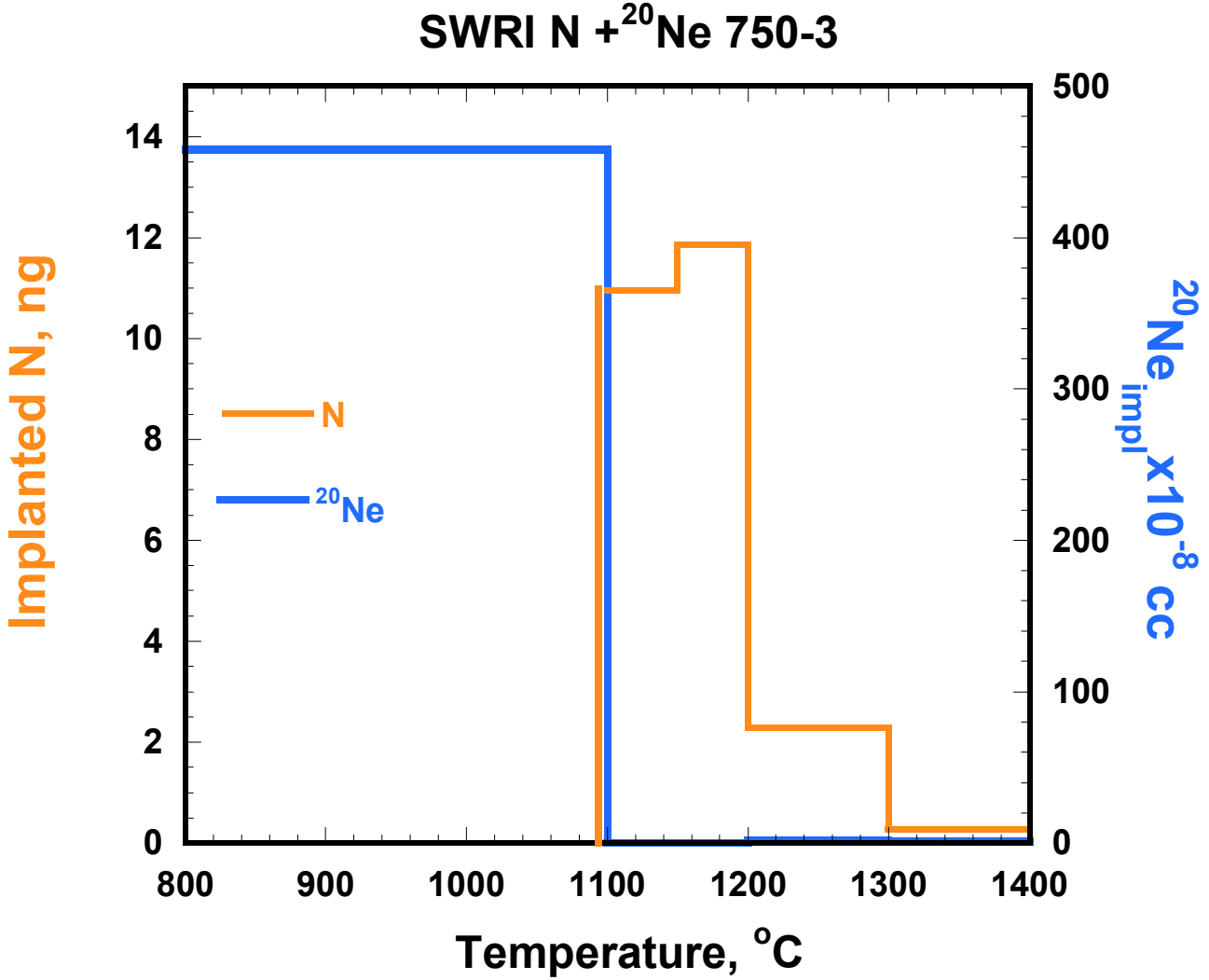
SWRI samples with implanted isotopically normal N





Surface area of the samples is in the range 0.1-1 cm^2

SWRI samples with implanted N and ^{20}Ne



Blanks and contamination

Nitrogen which is released during Genesis sample analyses but is not SW

It comes from:

1. Analytical system blank
2. Surface laboratory contamination
3. Trapped during formation of the DLC layer

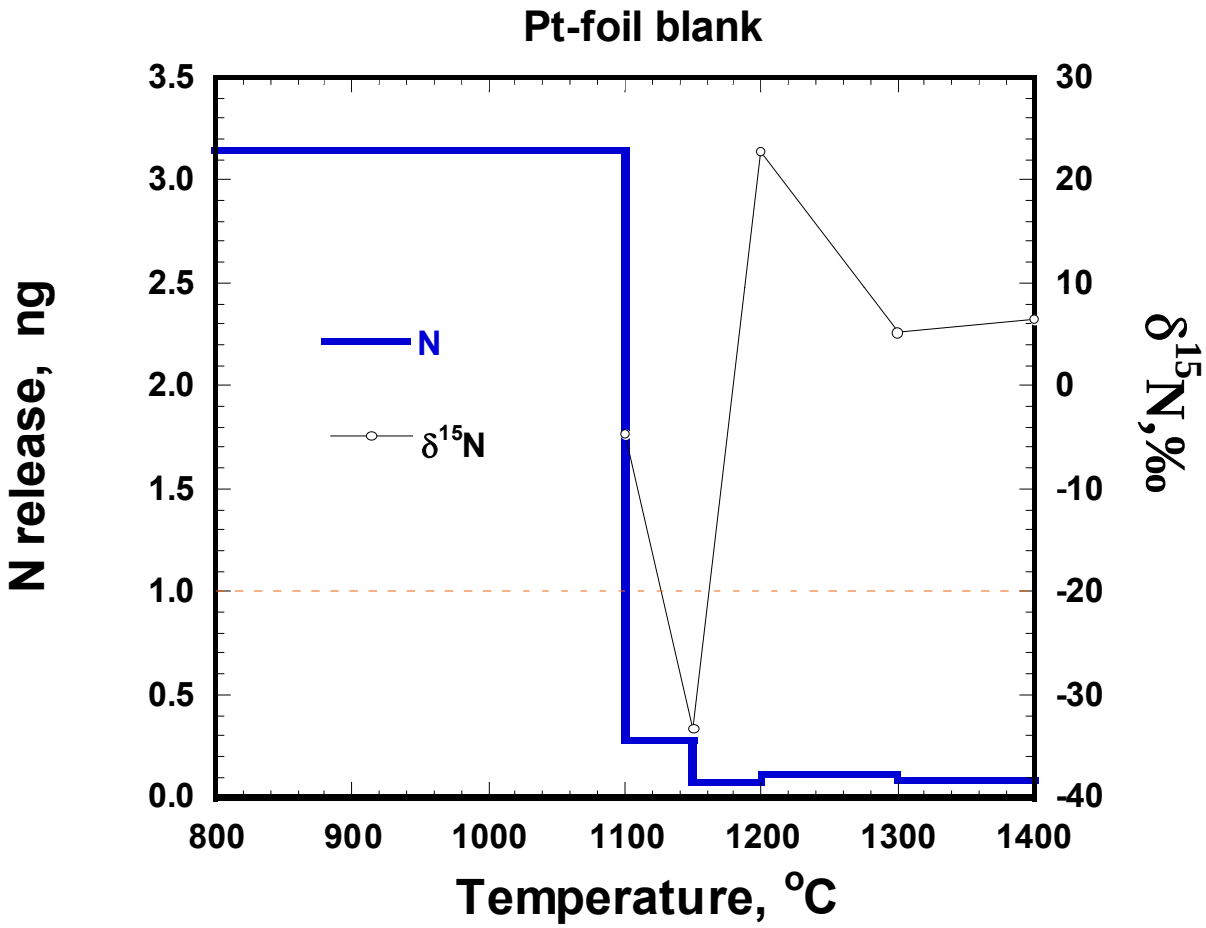
Blanks and contamination

System blank

- Compared to the amount of SW N expected to be released from 1 cm^2 of the concentrator target, ~1 ng, the system blank is low ~ 0.1 ng even at high T.

Blanks and contamination

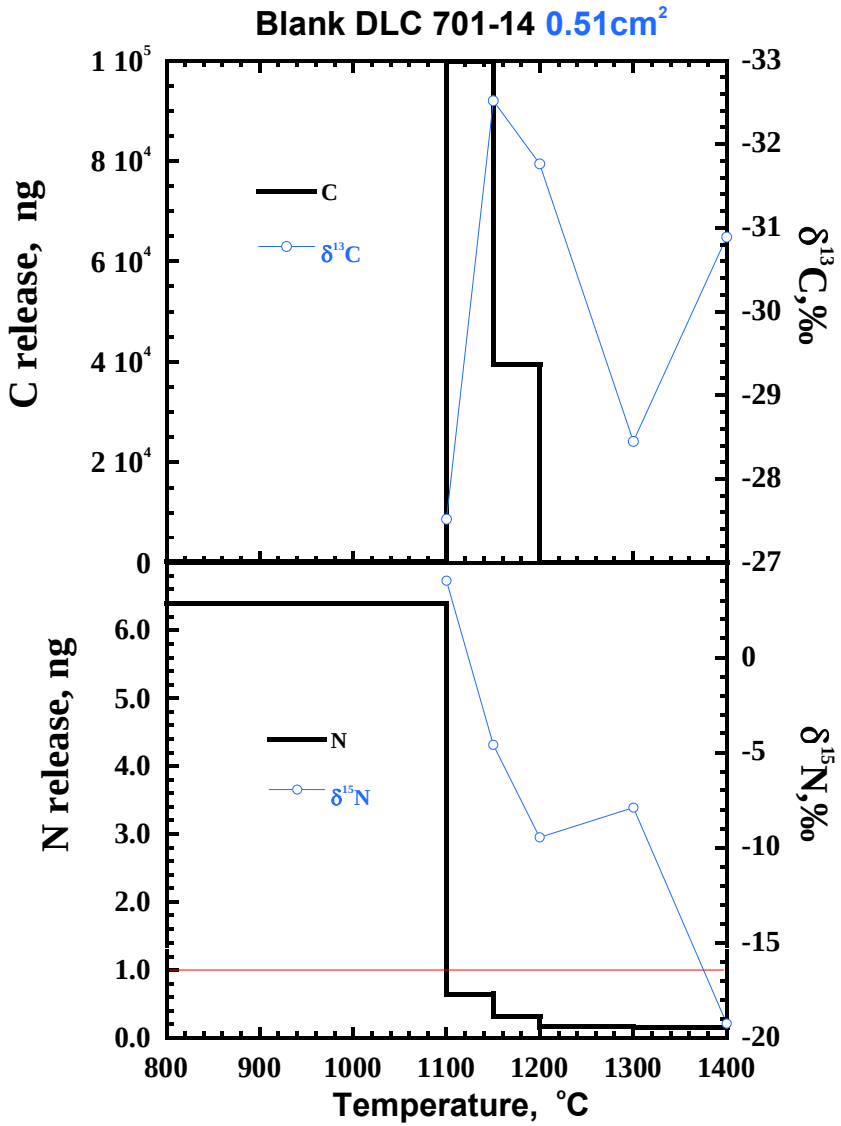
Pt foil blank

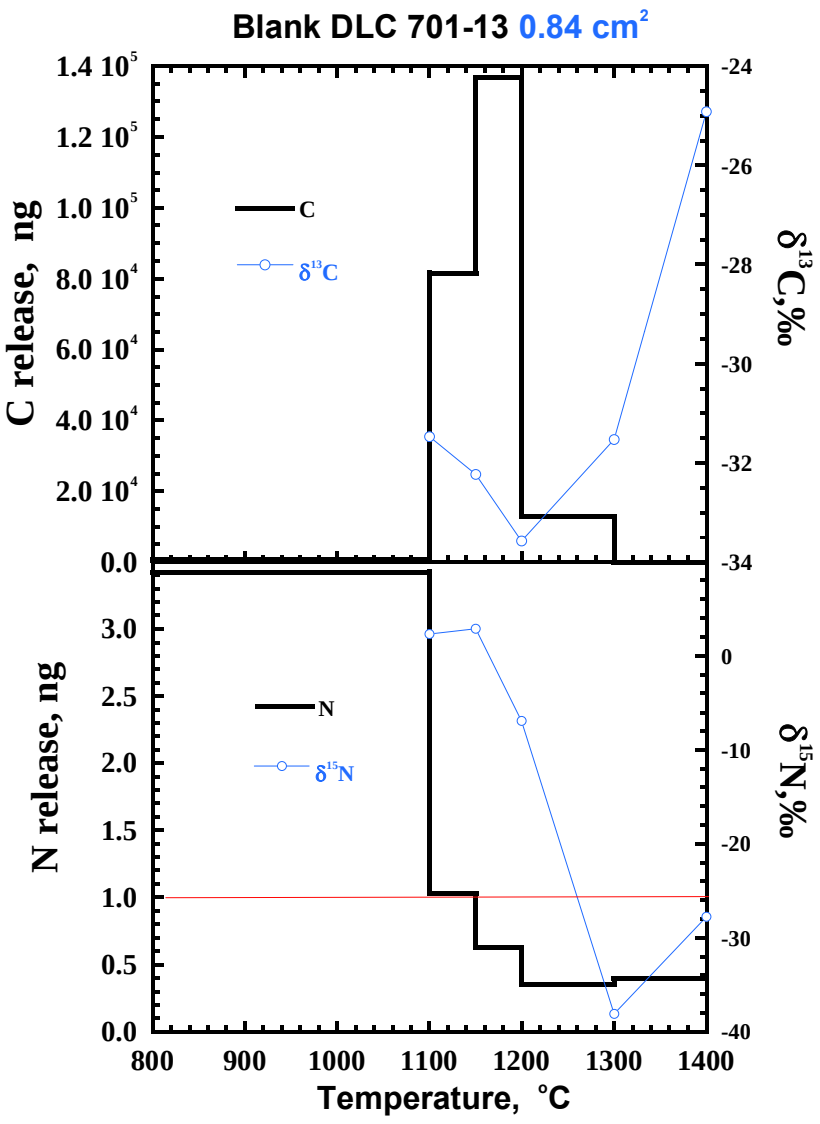


Most of N is released at 1100°C pyrolysis step and seems to come from surface contamination of the Pt foil

Blanks and contamination

Blank DLC

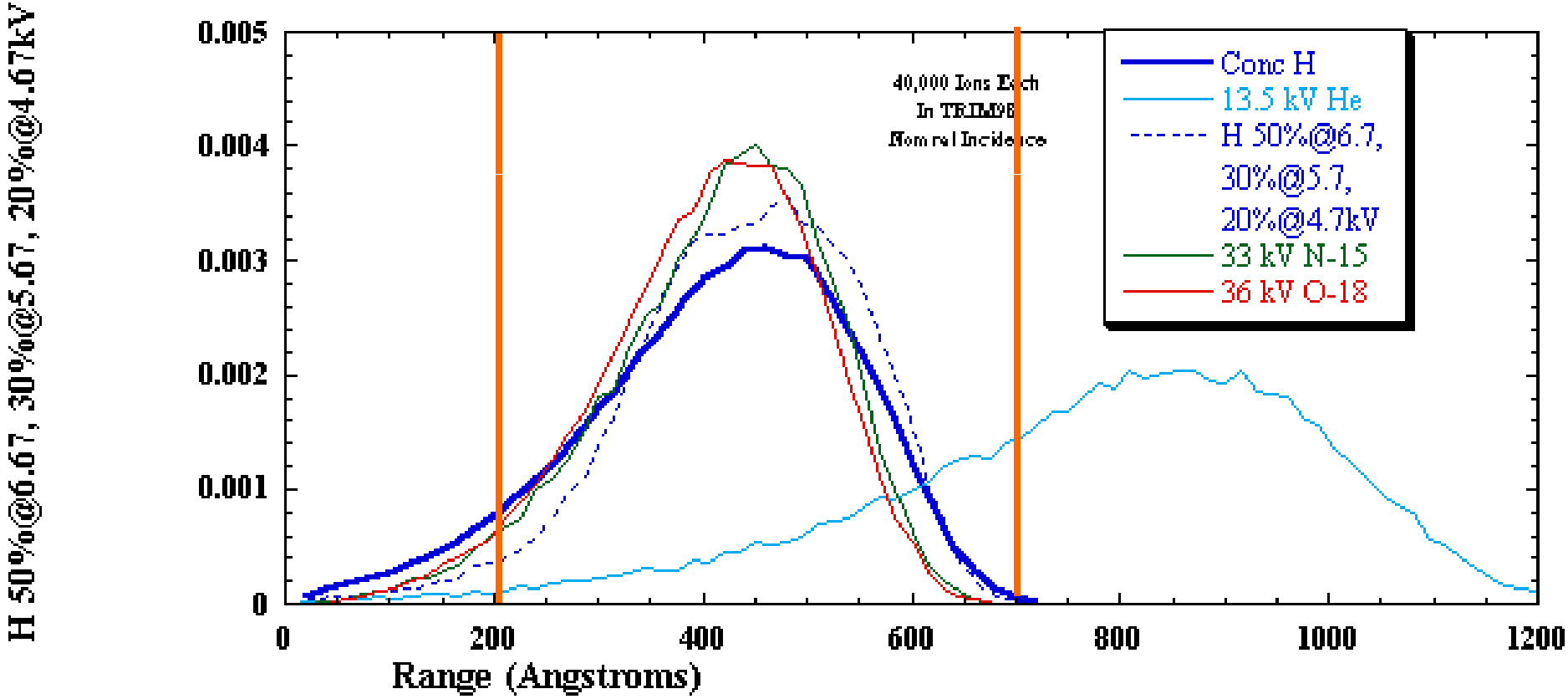




Blanks and contamination

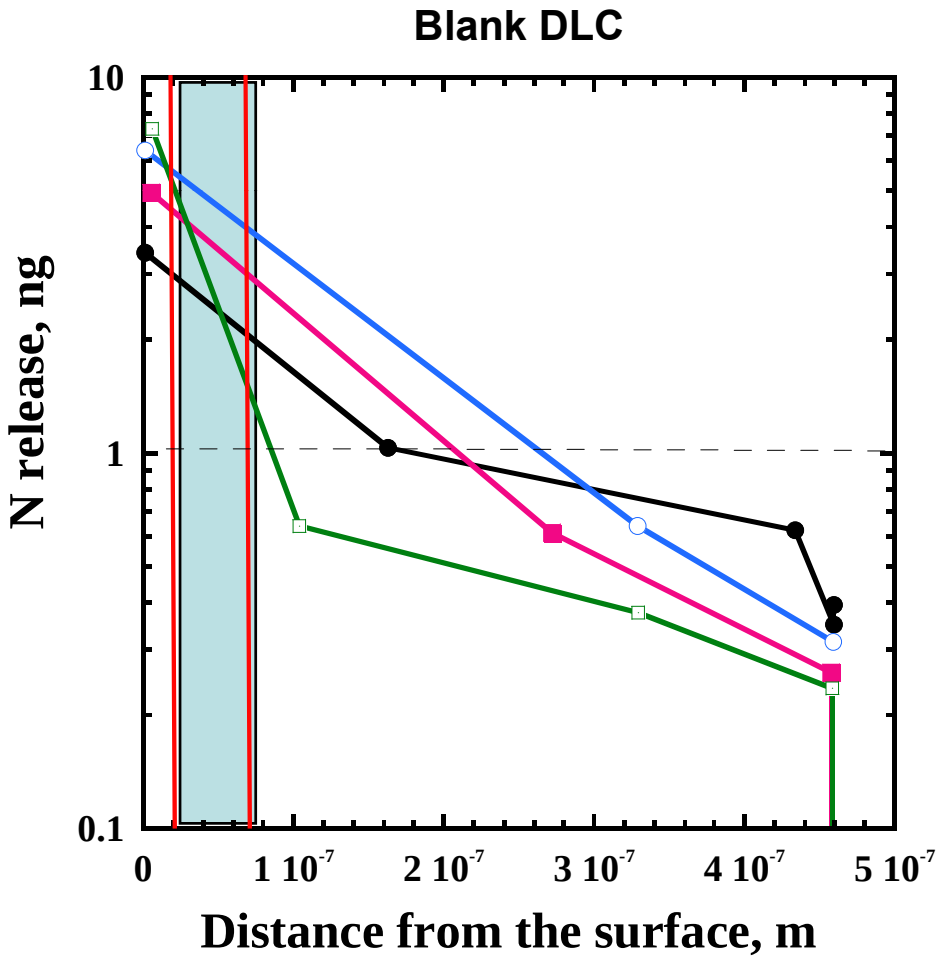
Implantation profile

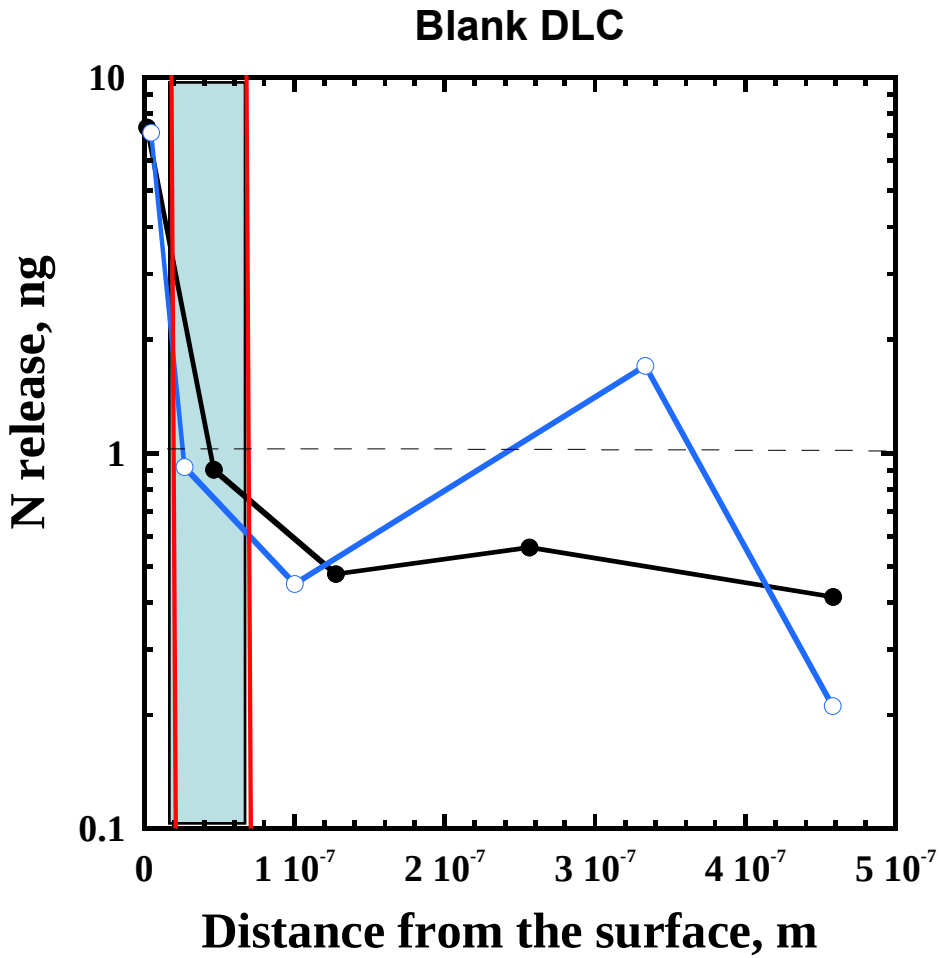
(Provided by Roger Wiens)



Blanks and contamination

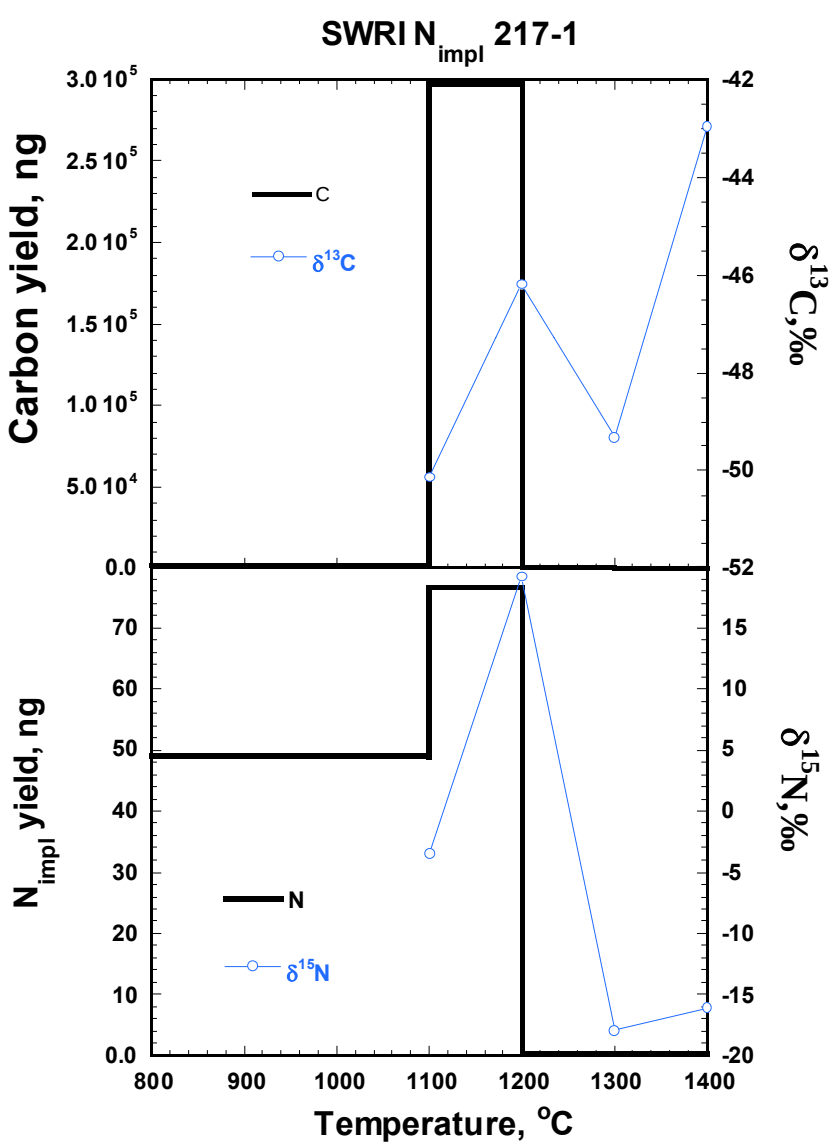
Selective N release

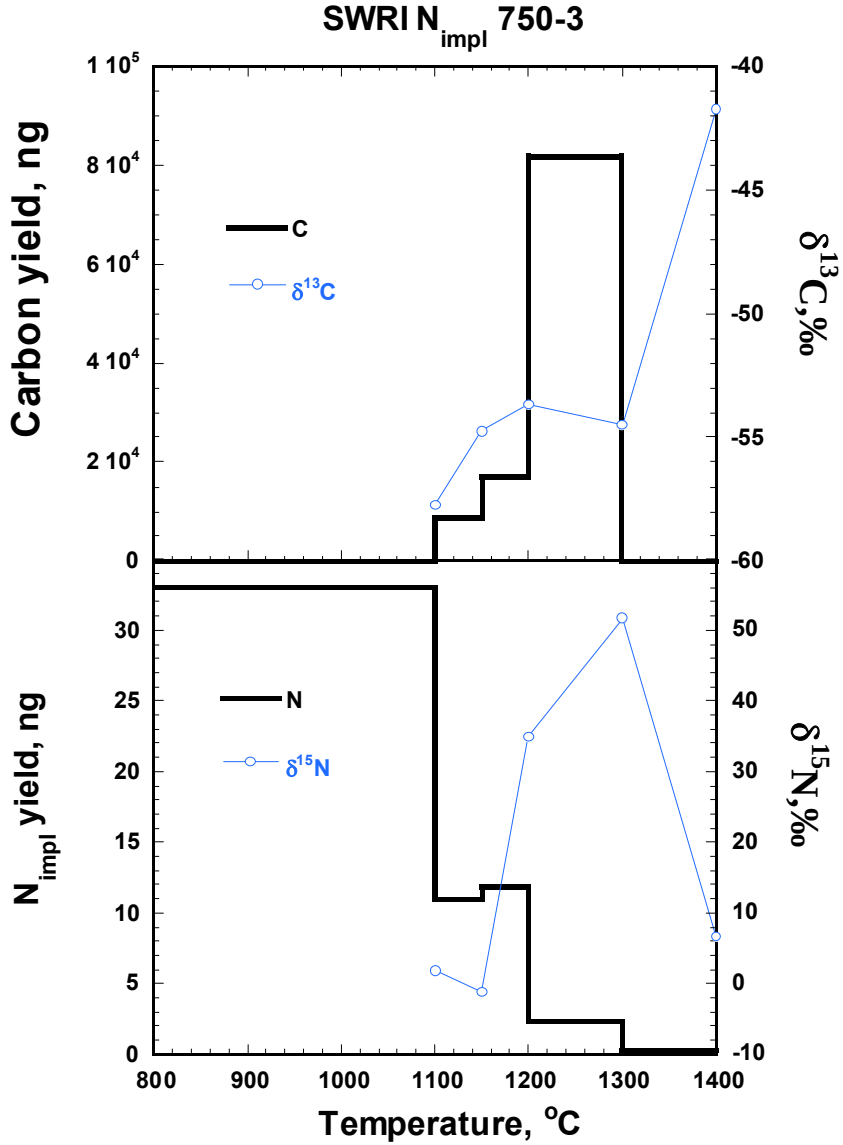




Conclusions

- Stepped combustion technique is suitable for measurements of SW N in DLC on Si concentrator target.
- It provides not more than 50% level of contamination of SW N if 1 cm^2 of the target material is analysed.
- We are almost ready for analyses of real samples


SWRI N impl 217-1
Carbon yield, ng
3.0 10^5
2.5 10^5
2.0 10^5
1.5 10^5
1.0 10^5
5.0 10^4
0.0
C
δ13C
δ13C,‰
-42
-44
-46
-48
-50
-52
N impl yield, ng
70
60
50
40
30
20
10
0.0
N
δ15N
δ15N,‰
15
10
5
0
-5
-10
-15
-20
800
900
1000
1100
1200
1300
1400
Temperature, °C


SWRI N impl 750-3
Carbon yield, ng
1 10^5
8 10^4
6 10^4
4 10^4
2 10^4
0
C
δ13C
δ13C,‰
-40
-45
-50
-55
-60
N impl yield, ng
30
25
20
15
10
5
0
N
δ15N
δ15N,‰
50
40
30
20
10
0
-10
800
900
1000
1100
1200
1300
1400
Temperature, °C