CN analysis in very small samples using the Integra2

The Inetgra2 has expanded capability to measure C and N by elemental analysis – isotope ratio mass spectrometry (EA-IRMS). The extended range modifications now enable researchers to get down to sample sizes as low as 2.5 µg C and 1.5 µg N whilst maintaining excellent precision. These modifications are invaluable for researchers wishing to measure small samples, samples which contain limited organic content, or samples with large C:N ratios.

The extended range set up reduces the amount of helium used to carry the sample through the system, thus reducing the dilution of the gas species of interest, whilst simultaneously offering researchers a reduction in operating costs due to the low consumption of helium. Extensive tests have been carried to ensure the background concentrations remain low. This application note details the hardware configuration alongside the operational conditions and timings necessary for the extended range set up.
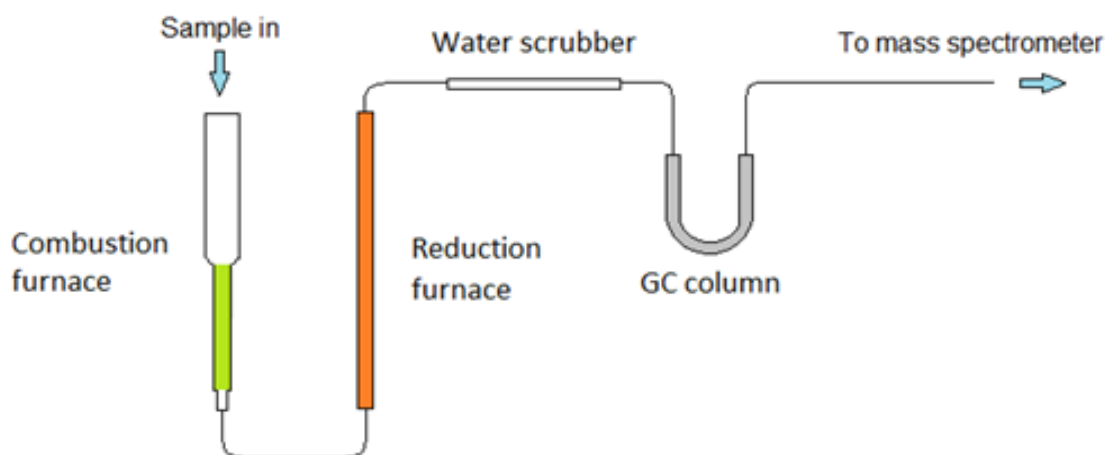
Figure 1: The Integra2

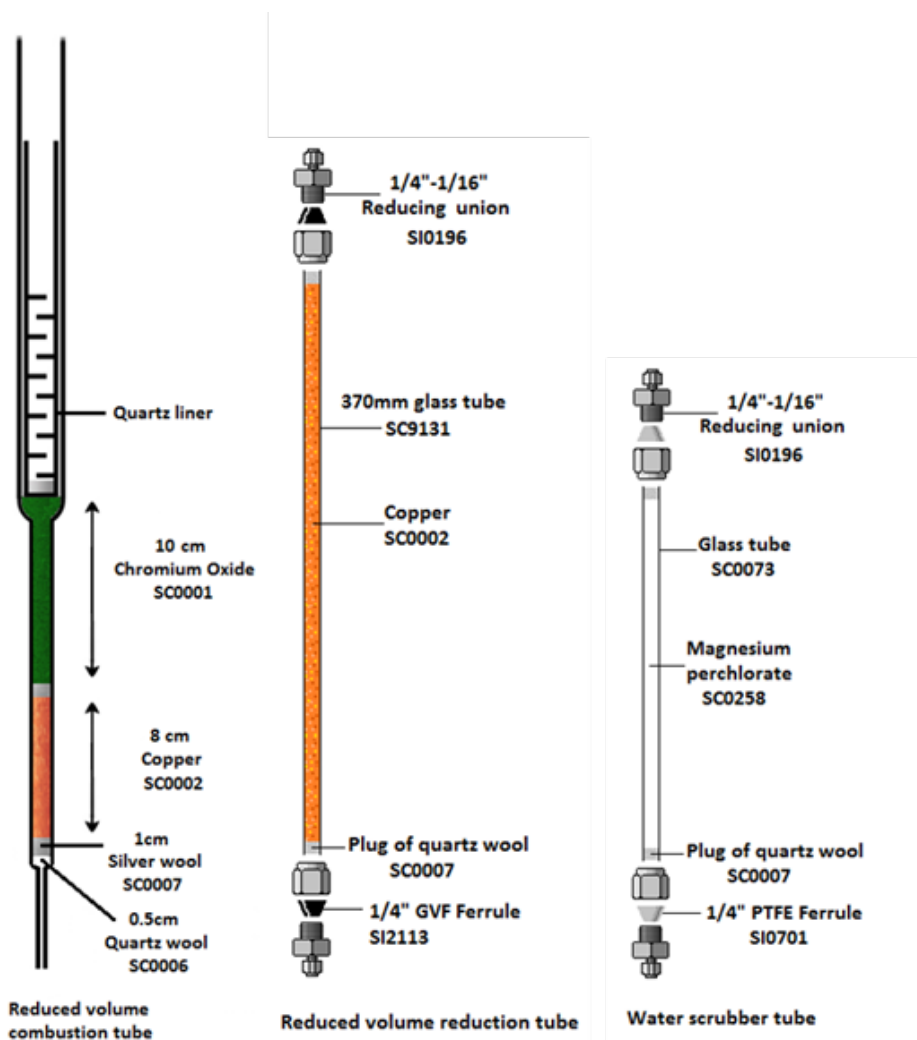Figure 2: Schematic overview of the extended range configuration

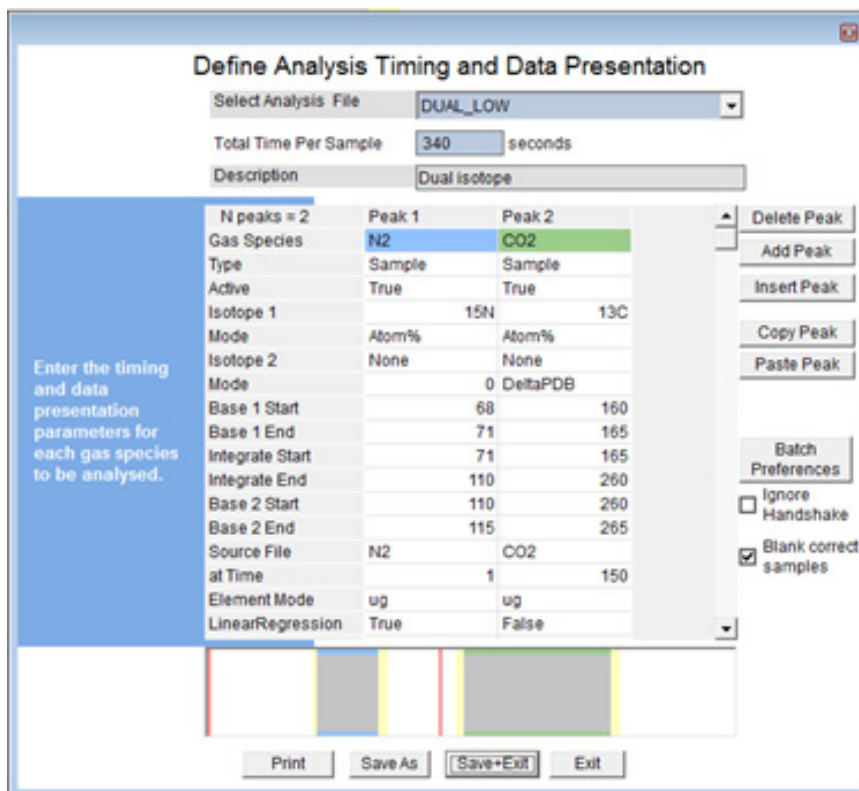Figure 3: The columns used in the extended range configuration

Figure 4: Analysis timings

Operating conditions - Temperatures, Pressures and Flows

Combustion Tube	1050°C
Reduction Tube	600°C
GC Oven	75°C
Helium Pressure	30PSI
Helium Flow	20ml/min
O2 Pressure	20PSI
O2 Flow	40ml/min

Name	Weight /Vol	Beam Area	µg N	^{15}N (wrt Air)	Beam Area	µg C	^{13}C (PDB)
Blank	0.00001	2.53E-10	0.08	-9.61	6.90E-11	0.01	-59.65
TEST	0.0071	4.61E-09	1.51	0.81	1.41E-08	2.66	-26.56
AS_BEET_MIX	0.0071	4.57E-09	1.51	0.40	1.36E-08	2.56	-25.83
1	0.0071	4.62E-09	1.53	0.14	1.35E-08	2.54	-25.86
2	0.0071	4.46E-09	1.48	0.91	1.28E-08	2.41	-26.33
3	0.0071	4.61E-09	1.53	0.63	1.35E-08	2.54	-26.09
4	0.0071	4.39E-09	1.46	0.46	1.36E-08	2.55	-26.29
5	0.0071	4.58E-09	1.53	0.76	1.43E-08	2.68	-26.17
AS_BEET_MIX	0.0071	4.48E-09	1.51	0.40	1.36E-08	2.56	-25.83
			STDEV	0.29			0.19

Table 1 CN data from low volume samples on the Integra2

Name	Beam Area	µg N	15N (Air)
Blank	6.24E-11	0.01	-49.93
TEST	3.72E-08	4.99	-0.66
AS_BEET_MIX	3.73E-08	5.00	-0.77
10	7.52E-08	10.08	**-0.85**
5	3.43E-08	4.61	**-0.43**
3	1.91E-08	2.57	**-0.42**
1	5.52E-09	0.74	**0.04**
AS_BEET_MIX	3.72E-08	5.00	-0.77

Table 2 Linearity on low level CN

As shown in table 1, with this configuration excellent precision of 0.2 ‰ is achieved when analysing samples as low as 2.5 µg C and precision of 0.3 ‰ is achieved down to 1.5 µg N. As shown in Table 2, linearity on the system remains excellent even at sub-µg levels of N.

For more information on Sercon systems capable of analysing very small samples, please contact sales@sercongroup.com