特別寄稿

Introduce of KIGAM AMS laboratory for Natural Science Research

Hong, Wan[1]*

[1]Korea Institute of Geoscience and Mineral Resources (KIGAM), Korea

*Correspondence author. E-mail: whong@kigam.re.kr

KIGAM introduced an AMS machine in 2007 on purpose to dedicate to research on earth science. Before the installation, renovation of the laboratory was performed. The main goals of the renovation were 1) to strengthen floor because of heavy weight of accelerator and magnet (see the Fig. 1), 2) to make trench to hide wires and tubes of AMS machine (see the Fig. 2), 3) to make air stream between laboratories to keep the air in the laboratories clean and dry, 4) to separate electricity supply to the AMS lab from other laboratories in the same building for safety, 5) to provide first-grade ground connection for accelerator and precise measurement system, and 6) to set the electricity outlet at proper sites to make maintenance of accelerator easy. The result of the renovation was very satisfactory because it was greatly helpful during the installation of the accelerator. And misalignment troubles of beam line and the troubles with water or compressed air system have never happened in this laboratory for almost a decade.

More than 25,000 radiocarbon dating targets have been measured by the AMS during the last 9 years. 4,664 targets and 3,445 targets were measured in 2015 and 2016, respectively (Fig. 3). Around 38% of the targets were control targets (blank and known samples including oxalic standards) to guarantee the quality of analysis. KIGAM provides diverse chemical and physical procedures for sample preparation of various samples, such as wood and charcoal (including alpha cellulose extraction), carbonate (wet and dry), sediment (humic and humin), bone, protein (organs and silk), air, biomass, iron artifact and pottery. Materials measured for last 9 years were presented in Fig. 4.

Fig. 1 Floor renovation with dense rebar mesh and supply tubes.

Fig. 2　Accelerator room ready for installation.

本原稿は、第29回（2016年度）名古屋大学宇宙地球環境研究所年代測定研究シンポジウム・特別講演で話す予定の内容を主体にまとめたものです。

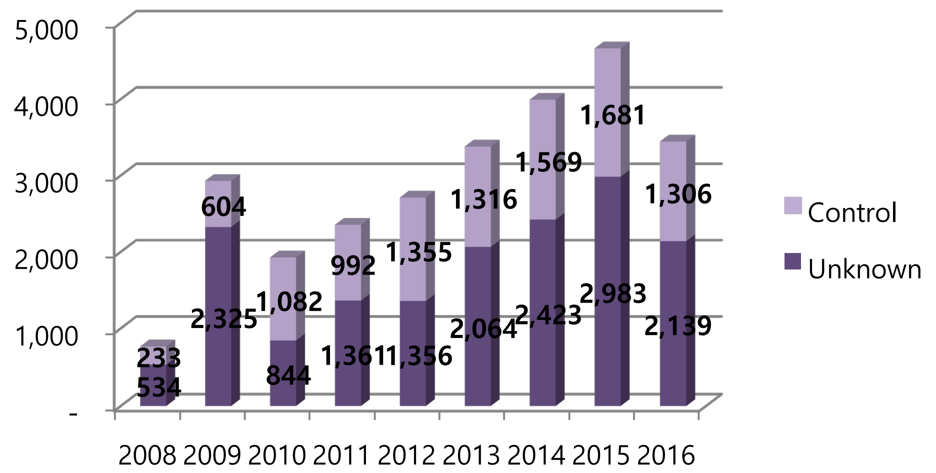


Fig. 3 Number of radiocarbon targets for 9 years.

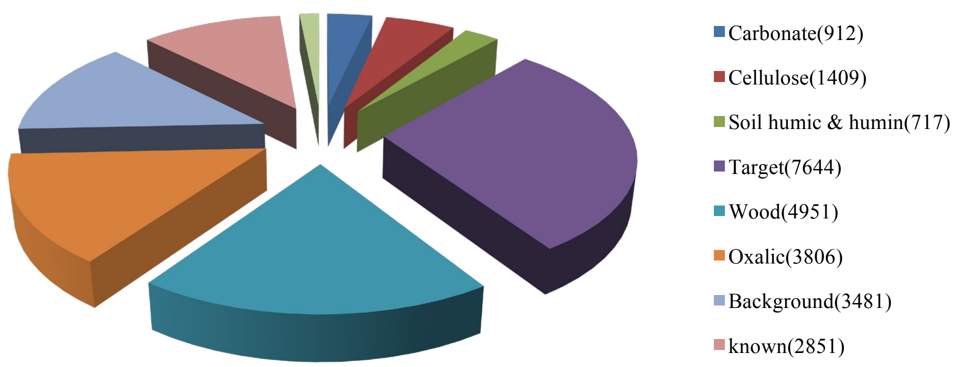


Fig. 4 Targets of KIGAM AMS for last 9 years.

Every data produced during the preparation procedures are collected in a database server (Fig. 5). The database system was designed in order to centralize and inquire data very easily. It was written in Oracle, NET and Excel script. This data base system helps analysis work. To make data collection easy, all the weighing system, reduction system, and AMS system are connected to PC, and data from the systems can be transferred to the data base by mouse click. Apply form for age dating is also written Excel script, and the sample information from the customer including sample type can be transferred to the data base. Then the proper chemical procedures are recommended by it. Receptionist can select one of the procedures. This data base system also plays a role of information center of samples. For example, in every step of sample preparation, an analyst can refer all the data of previous step easily. When we review the age dating results of the sample, we can access to history of a sample easily because this system operates on web base in order to access to it everywhere.

A 24-fold automatic reduction system, shown in Figure 6, was developed, and has been dedicate to routine combustion and reduction processes of samples. This system is coupled with an elemental analyzer to combust samples sequentially, and controlled by a PC, which collects all the data from reduction process and sends them to the database server. The average yield of the reduction system is higher than 95% with typical reaction time of 3 hours. High reduction yield is preferred not only for small sample size but also for the minimization of isotopic fractionation.

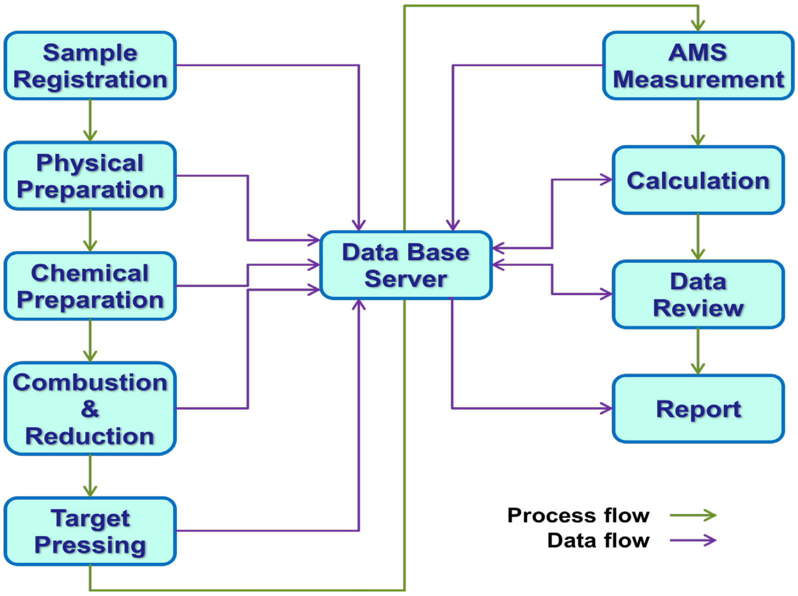
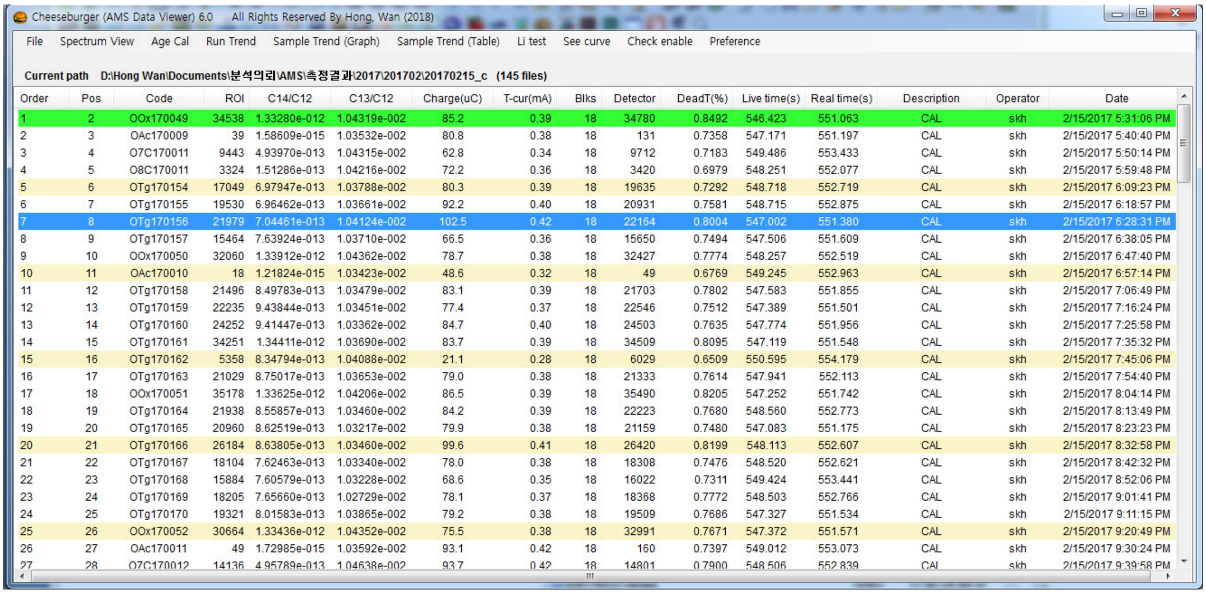


Fig. 2　Diagram of process and data flows for radiocarbon dating at KIGAM AMS laboratory.

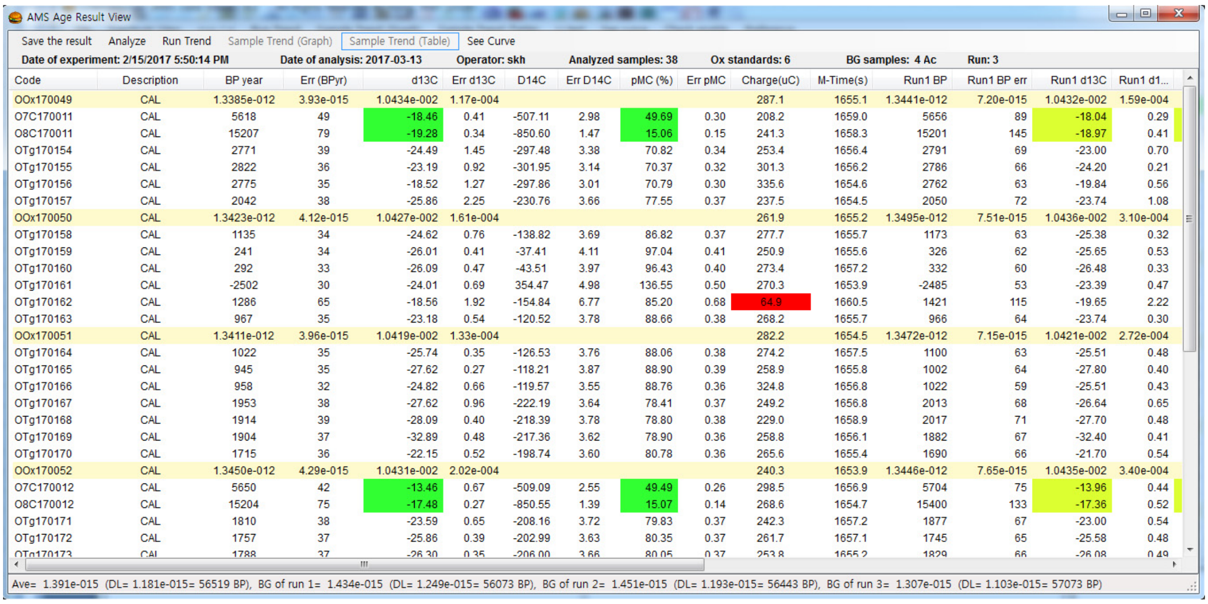
Fig. 3　A 24-fold automatic reduction system developed by KIGAM.

AMS measurement is consisted of several times of runs in which batch of samples are sequentially measured to avoid a risk of long-term variation of AMS condition. In a run, all the samples are measured for 9 minutes in routine work, but data are stored every 30 seconds. Hence each run has 18 data blocks per sample. Generally, three runs go for normal sample. For 50 targets (one carrousel), one runs takes 450 minutes, and three runs takes 1350 minutes (around 22 hours). For overnight measurement, Unmanned Operation System (UOS) was developed. A CCD camera watches the control PC of AMS system during the operation, and UOS calls to operator's mobile phone when it detects activity of an interlock system of AMS.

All the age data of samples are reviewed at revision meeting to verify their validities. The meeting is the last step before issuing the age report. The data tables for the review meeting are shown in Figs. 7 and 8, which are provided by a tiny and fast data view and analysis tool, called "Cheeseburger", which was developed at KIGAM AMS laboratory. The raw data table (Fig. 7a) is extracted from raw data files created by AMS system, and the age table (Fig. 7b) is created by calculation of sample ages. Cheeseburger can recognize prefixes of several special sample codes of KIGAM Sample Code Protocol (KSCP), such as oxalic sample (“Ox”), several different types of blank samples (“Bg”, “As”, “Ac”, “Aw”, “1C”) and known samples (“St”, “2C”, “4C”, “7C”, “8C”). The program performs blank subtraction, evaluation of $\delta^{13}C$ from ^{13}C value of each sample, correction for isotopic fractionation using the $\delta^{13}C$, averaging the data from one sample, age and error calculation, and statistics of the results.



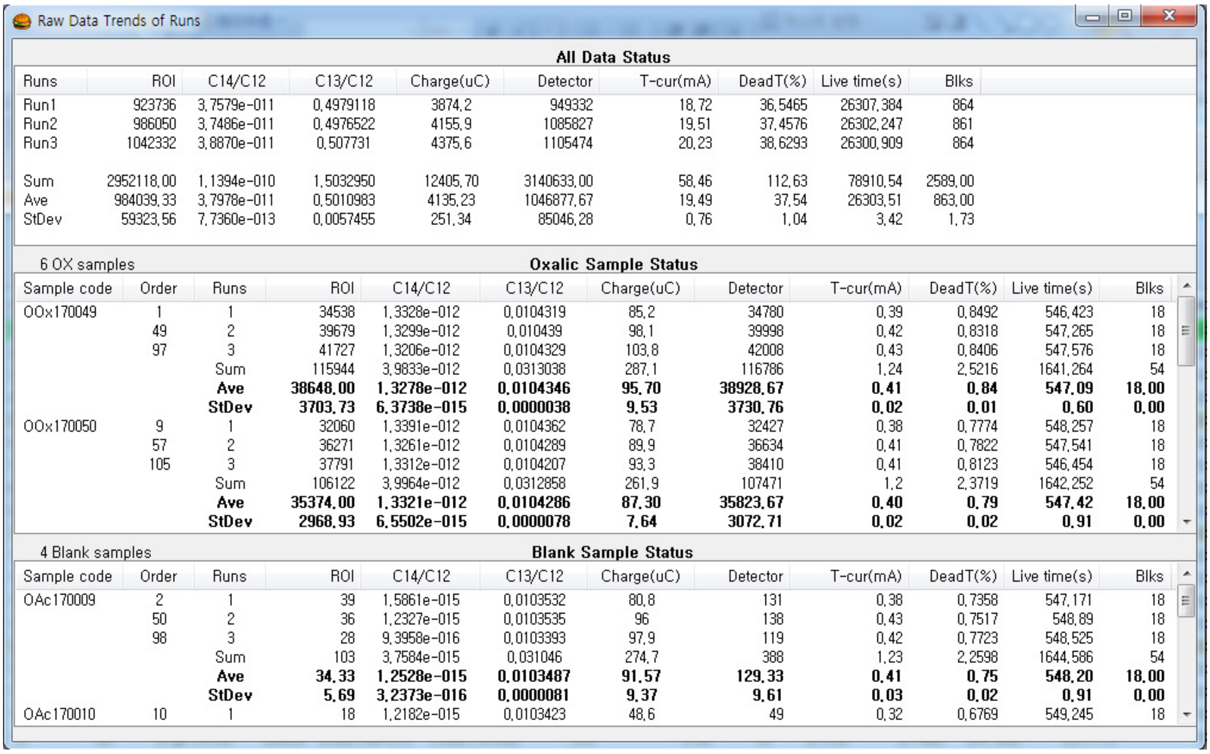
(a)



(b)

Fig. 4 (a) Raw data table and (b) age data table provided by Cheeseburger, the data view and analysis tool, developed at KIGAM AMS laboratory. Green colored numbers in (b) are representing that they are correct within the error ranges of consensus values of the known samples. Red colored number means that the number is out of criteria. In this example, the accumulated charge is not sufficient for good statistics.

In the age table, ages of known samples are compared with their consensus values, and the results are marked by colors. Measurement results (BG) of blank samples are also evaluated and given at the bottom of in the table. Detection limit (DL) for the measurements is calculated from deviation of the blank values.



Raw Data Trends of Runs

All Data Status

Runs	ROI	C14/C12	C13/C12	Charge(uC)	Detector	T-cur(mA)	DeadT(%)	Live time(s)	Blks
Run1	923736	3,7579e-011	0,4979118	3874,2	949332	18,72	36,5465	26307,384	864
Run2	986050	3,7486e-011	0,4976522	4155,9	1085827	19,51	37,4576	26302,247	861
Run3	1042332	3,8870e-011	0,507731	4375,6	1105474	20,23	38,6293	26300,909	864
Sum	2952118,00	1,1394e-010	1,5032950	12405,70	3140633,00	58,46	112,63	78910,54	2589,00
Ave	984039,33	3,7978e-011	0,5010983	4135,23	1046877,67	19,49	37,54	26303,51	863,00
StDev	59323,56	7,7360e-013	0,0057455	251,34	85046,28	0,76	1,04	3,42	1,73

6 OX samples

Oxalic Sample Status

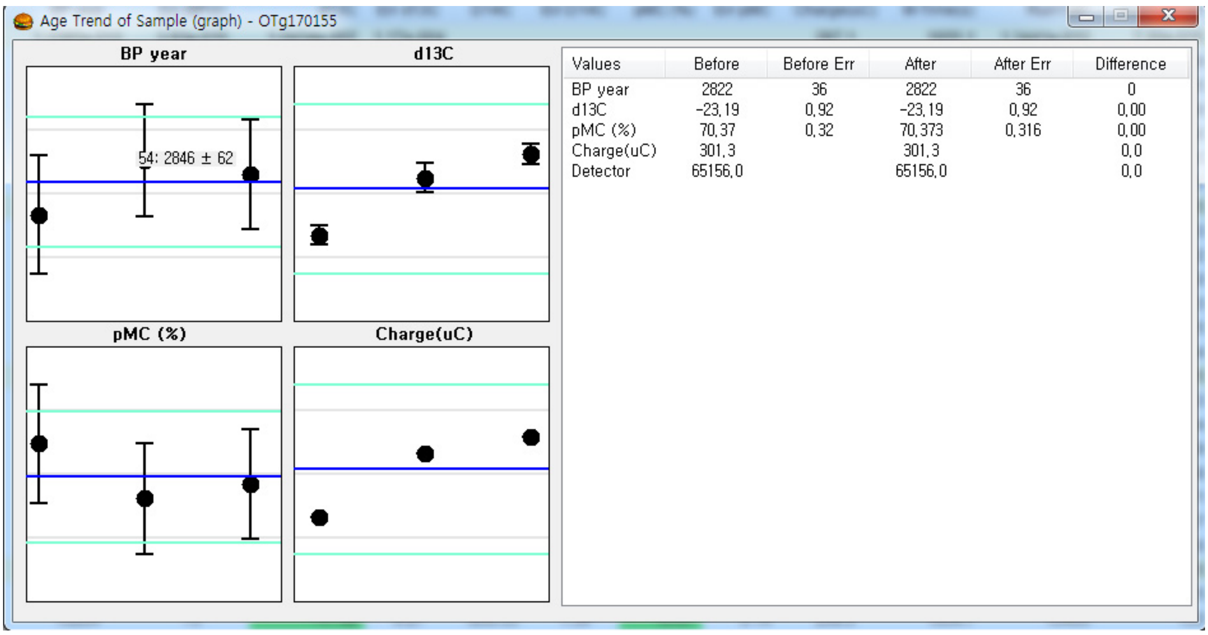
Sample code	Order	Runs	ROI	C14/C12	C13/C12	Charge(uC)	Detector	T-cur(mA)	DeadT(%)	Live time(s)	Blks
OOx170049	1	1	34538	1,3328e-012	0,0104319	85,2	34780	0,39	0,8492	546,423	18
	49	2	39679	1,3299e-012	0,010439	98,1	39998	0,42	0,8318	547,265	18
	97	3	41727	1,3206e-012	0,0104329	103,8	42008	0,43	0,8406	547,576	18
		Sum	115944	3,9833e-012	0,0313038	287,1	116786	1,24	2,5216	1641,264	54
		Ave	**38648,00**	**1,3278e-012**	**0,0104346**	**95,70**	**38928,67**	**0,41**	**0,84**	**547,09**	**18,00**
		StDev	**3703,73**	**6,3738e-015**	**0,0000038**	**9,53**	**3730,76**	**0,02**	**0,01**	**0,60**	**0,00**
OOx170050	9	1	32060	1,3391e-012	0,0104362	78,7	32427	0,38	0,7774	548,257	18
	57	2	36271	1,3261e-012	0,0104289	89,9	36634	0,41	0,7822	547,541	18
	105	3	37791	1,3312e-012	0,0104207	93,3	38410	0,41	0,8123	546,454	18
		Sum	106122	3,9964e-012	0,0312858	261,9	107471	1,2	2,3719	1642,252	54
		Ave	**35374,00**	**1,3321e-012**	**0,0104286**	**87,30**	**35823,67**	**0,40**	**0,79**	**547,42**	**18,00**
		StDev	**2968,93**	**6,5502e-015**	**0,0000078**	**7,64**	**3072,71**	**0,02**	**0,02**	**0,91**	**0,00**

4 Blank samples

Blank Sample Status

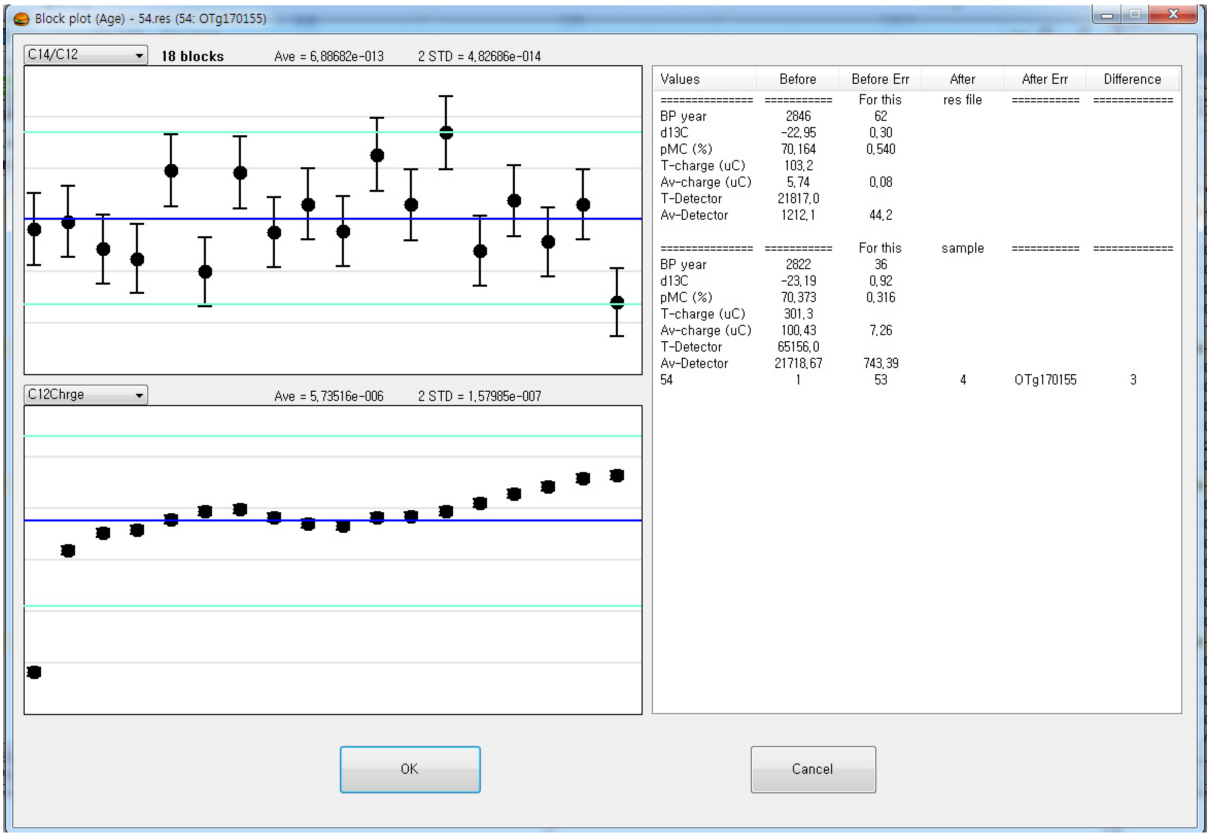
Sample code	Order	Runs	ROI	C14/C12	C13/C12	Charge(uC)	Detector	T-cur(mA)	DeadT(%)	Live time(s)	Blks
OAc170009	2	1	39	1,5861e-015	0,0103532	80,8	131	0,38	0,7358	547,171	18
	50	2	36	1,2327e-015	0,0103535	96	138	0,43	0,7517	548,89	18
	98	3	28	9,3958e-016	0,0103393	97,9	119	0,42	0,7723	548,525	18
		Sum	103	3,7584e-015	0,031046	274,7	388	1,23	2,2598	1644,586	54
		Ave	**34,33**	**1,2528e-015**	**0,0103487**	**91,57**	**129,33**	**0,41**	**0,75**	**548,20**	**18,00**
		StDev	**5,69**	**3,2373e-016**	**0,0000081**	**9,37**	**9,61**	**0,03**	**0,02**	**0,91**	**0,00**
OAc170010	10	1	18	1,2182e-015	0,0103423	48,6	49	0,32	0,6769	549,245	18

(a)



Values	Before	Before Err	After	After Err	Difference
BP year	2822	36	2822	36	0
d13C	-23,19	0,92	-23,19	0,92	0,00
pMC (%)	70,37	0,32	70,373	0,316	0,00
Charge(uC)	301,3		301,3		0,0
Detector	65156,0		65156,0		0,0

(b)



(c)

Fig. 5 Analysis results of Cheeseburger. (a) A trend table between runs (a sequence of multi-sample measurements). (b) Comparison of data of a sample between runs. In KIGAM AMS Lab, a sample is measured 3 times (runs) in routine work. (c) Block data plot with calculation results for a single measurement. In the plots (b) and (c), each dot can be removed by double-clicking and the calculation results in right-hand side table are changed immediately. Users can un-do deleting of a dot by double-clicking again.

Figure 8 presents analysis results of Cheeseburger. Long-term change of components in AMS system, such as magnets, can sometimes cause slow but large variation during measurement, which usually takes several days. Run trend table (Fig. 8a) is very useful to confirm the AMS machine stability during the measurements. It also gives information of oxalic standards and blank targets. This kind of long-term condition change of AMS spectrometer makes age discrepancy between runs of a sample. Sample trend table (Fig. 8b) shows plots of several parameter changes between runs so that the long-term variation can be caught easily. When the variation of a run data is larger than two sigma range over all runs, then user can remove the run data from result calculation.

In the AMS measurement, sudden spark at terminal or ion source sometimes occurs, and this kind of event causes drastic condition change within very short time, which may be presented as a block data fluctuation of a measurement. The block plot (Fig. 8c) provides powerful tool to pick up such outliers from blocks of the measurement. With the program, it becomes very easy and effective to review AMS data.