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NATURAL ENVIRONMENT RESEARCH COUNCIL

From LIMS to Geochemistry Database: G-BASE Samples Analytical Data

Information Management Programme

Internal Report IR/06/075

BRITISH GEOLOGICAL SURVEY

INFORMATION MANAGEMENT PROGRAMME

INTERNAL REPORT IR/06/075

From LIMS to Geochemistry Database: G-BASE Samples Analytical Data

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Foreword

The BGS is involved in automation of its data handling activities. This report describes work carried out under the joint projects of laboratory technique development and corporate information management.

The BGS Analytical Geochemistry Laboratories in Keyworth routinely analyse samples for the Geochemistry Baseline Survey of the Environment, G-BASE. The aim of this project was to automate data collection and transfer of results of analysis of solid samples from the laboratories to the corporate geochemistry database.

The first part of the report describes the processes involved. The appendices contain more technical detail.

Acknowledgements

A number of individuals in Analytical Geochemistry have contributed to the project. This assistance has been received at all stages of the work. Of the many individuals who have contributed to the project we would particularly like to acknowledge the support of the following:

Mr T S Barlow

Miss V L Moss-Hayes

Mr N D Eatherington

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Summary

The BGS analytical geochemistry laboratories in Keyworth analyse samples for G-BASE. This report describes the collection, collation, verification and storage of the analytical results for the solid samples.

The laboratories analytical records are stored in a Laboratory Information Management System (LIMS) data checking, collation and verification are carried out in this system. A Microsoft Access routine is used to extract information for specified samples and transfer them to tables in the corporate Geochemistry Database. These tables hold the data in its “raw” state, i.e. as delivered by the laboratories, before any further G-BASE processing takes place. This allows the analytical data to become more widely available in a much shorter timescale than previously and facilitates secure permanent storage in the corporate system.

1 Introduction

The Geochemistry Database is the BGS corporate repository for geochemical data for the UK land mass. It is described in *Harris and Coats, 1992*. Data from G-BASE work should ultimately reside here, previously in the finished state after data conditioning, *Lister and Johnson, 2005.* No corporate provision had been made for a central store of "raw" G-BASE data, i.e. data as received from the analytical laboratories. These data had previously been held in the G-BASE data archive managed by the G-BASE data manager (T.R Lister).

The Laboratory Information Management System (LIMS) is a commercial product supplied by LabVantage Solutions, currently Sapphire V1.5 (Build 010). This consists of a number of tools and a core relational database in SQL Server that can be added to, developed and customised to suit laboratory needs.

This report covers the work to provide a direct link from the analytical laboratories' LIMS to "raw" geochemistry database tables, i.e. tables designed to hold the data as received. The report covers the analysis of solid samples only. For G-BASE, solid samples' analytical data consists of major and trace elements by XRFS together with pH and LOI (Loss On Ignition) data (soils only) from General and Organic Chemistry Section.

1.1 XRFS

Pressed powder pellets are geological, environmental and related materials ground to ideally <30 µm with 25% (by original sample weight) of binder, a styrene co-polymer with a polyethylene wax, and free pressed into a 40 mm diameter pellet at 25 tons load.

For XRFWD the pellets are analysed by wavelength dispersive X-ray fluorescence for the determination of major and trace elements, as two separate programs coded as XRDWDM and XRFWDT in the Geochemistry Database.

For XRFED the pellets are analysed by energy dispersive X-ray fluorescence for the determination of trace elements.

1.2 LOI AT 450°C

Samples are usually pre-dried at 105°C prior to heating at 450°C. The method is to weigh a portion of sample, usually between 1-2g, into a pre-weighed beaker and then dry overnight at 105°C. After cooling, the sample and beaker are weighed, placed in a furnace at 450°C for a minimum of 4 hours and then re-weighed after cooling. The loss in weight before and after heating at 450°C is calculated as a proportion of the pre-dried sample weight and expressed as a percentage.

1.3 pH

A portion of sample is mixed with 0.01M calcium chloride solution to produce a slurry (typically, 25ml of 0.01M $CaCl_2$ is added to 10.0g of sample). The pH is measured using a solid body combination pH electrode attached to a bench pH meter. The BGS Analytical Geochemistry Laboratory method (AGN 2.5.1) is validated between pH 3 & 10 and has UKAS (United Kingdom Accreditation Service) and MCERTS (the Environment Agency Monitoring Certification Scheme, specifically the performance standard for laboratories undertaking chemical testing of soil) accreditation, *Taylor, Vickers and Moss-Hayes.* 2005.

2 Analysis Definition

In the LIMS, analytical requirements are recorded as Parameter Lists where Parameters are the items to be measured or recorded such as element concentrations, sample weights or analyst name. Individual parameters can be assigned limits (detection, over range, reporting etc.) or restricted to a given range or set of values. What happens when values fall outside the limits or range of values can be defined by a set of rules. Units for the parameters (those which have them) are also defined within the Parameter List and values can be rounded to the correct number of decimal places (see appendix 1).

In short, the analytical requirement is defined as a parameter list and the parameter list is attached to the sample. There may be several different requirements, e.g. XRFS analysis plus pH and LOI. Several parameter lists can be attached to a sample, one for each type or suite of analysis.

2.1 PARAMETER LISTS

An example, one of the XRFS parameter lists, is given below to demonstrate how the system works.

P-Na-Fe is a Parameter List for XRFWDM. It contains the following parameters:

Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, SO_3, K_2O, CaO, TiO_2, MnO, Fe_2O_3, Ba, Cl, Instrument, DateTime, Analyst. For every sample that has the P-Na-Fe Parameter List attached there is a requirement for this analytical data. Values for the listed parameters will be entered into the LIMS, concentrations for the element parameters, instrument name for the Instrument parameter, date and time of analysis for the DateTime parameter and analyst identifier for the Analyst parameter e.g.

Parameter	Value
Na_2O	0.5
MgO	0.8
Al_2O_3	10.5
SiO_2	68.4
P_2O_5	0.16
SO_3	-97
K_2O	2
CaO	1.32
TiO_2	0.629
MnO	0.064
Fe_2O_3	3.41
Ba	0.03
Cl	-97
Instrument	MagiX PRO-1
DateTime	21/10/2005 11:52
Analyst.	NDE

2.1.1 Parameter Limits and Limit Rules

For this parameter list each element parameter has limits defined e.g. for Na_2O:

LLD	NDL	ULC	ULR
0.3	-90 to 0.049	10	10

LLD is the Lower Limit of Detection. Values equal to and above this are within the validated concentration range of the instrument.

NDL is Not Detected because of interference but probably of low concentration. Values within this range are not to be regarded as actual concentration values.

ULC is the Upper Limit of Calibration. Values equal to or below this are within the validated concentration range of the instrument.

ULR is the Upper Limit of Reporting. Values equal to and below this but above the ULC are still acceptable as concentrations but are not as accurate as values within the validated concentration range of the instrument. Values above this limit are not to be regarded as actual concentration values and are flagged as not determined because of interference; probably of high concentration.

In order to ensure that values in the NDL range and values above the ULR are not used as concentration values they are converted to a limit code by the Parameter List rules. This code will indicate that the parameter could not be measured at the level it was present in the sample:

The code for NDL values is –97 signifying “not determined because of interference; probably of low concentration”.

The code for ULR values is –95 signifying “not determined because of interference; probably of high concentration”.

3 Data Entry

3.1 XRFS

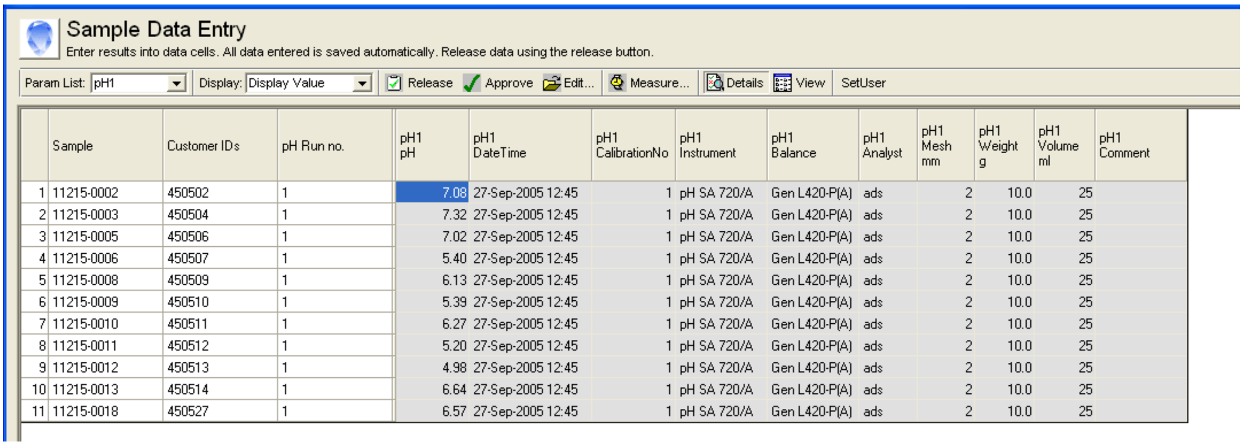
Data entry for XRFS is semi-automatic. Files are manually saved to a special loading directory and fed directly into the LIMS tables. This is achieved by a LIMS “action”, a specially written piece of code within the LIMS. The action is initiated by staff whenever verified files are positioned ready for upload.

Files that load successfully are copied as an attachment on to the relevant batch within the LIMS. The original file is deleted from the loading folder. Files that fail to load correctly are moved to a “failed” folder. See appendix 2 for a more detailed overview.

3.2 pH AND LOI

Data entry for pH and LOI is manual; data being entered into a data entry spreadsheet within the LIMS, see figure 1. Data can be entered by typing in the value, choosing from a drop down list or weights can be entered directly from the balance by electronic transfer once the correct cell is highlighted in the entry page.

Figure 1 LIMS data entry page



Sample Data Entry

Enter results into data cells. All data entered is saved automatically. Release data using the release button.

Param List: pH1

Display: Display Value

	Sample	Customer IDs	pH Run no.	pH1 pH	pH1 DateTime	pH1 CalibrationNo	pH1 Instrument	pH1 Balance	pH1 Analyst	pH1 Mesh mm	pH1 Weight g	pH1 Volume ml	pH1 Comment
1	11215-0002	450502	1	7.08	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
2	11215-0003	450504	1	7.32	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
3	11215-0005	450506	1	7.02	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
4	11215-0006	450507	1	5.40	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
5	11215-0008	450509	1	6.13	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
6	11215-0009	450510	1	5.39	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
7	11215-0010	450511	1	6.27	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
8	11215-0011	450512	1	5.20	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
9	11215-0012	450513	1	4.98	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
10	11215-0013	450514	1	6.64	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	
11	11215-0018	450527	1	6.57	27-Sep-2005 12:45	1	pH SA 720/A	Gen L420-P(A)	ads	2	10.0	25	

4 Data Verification

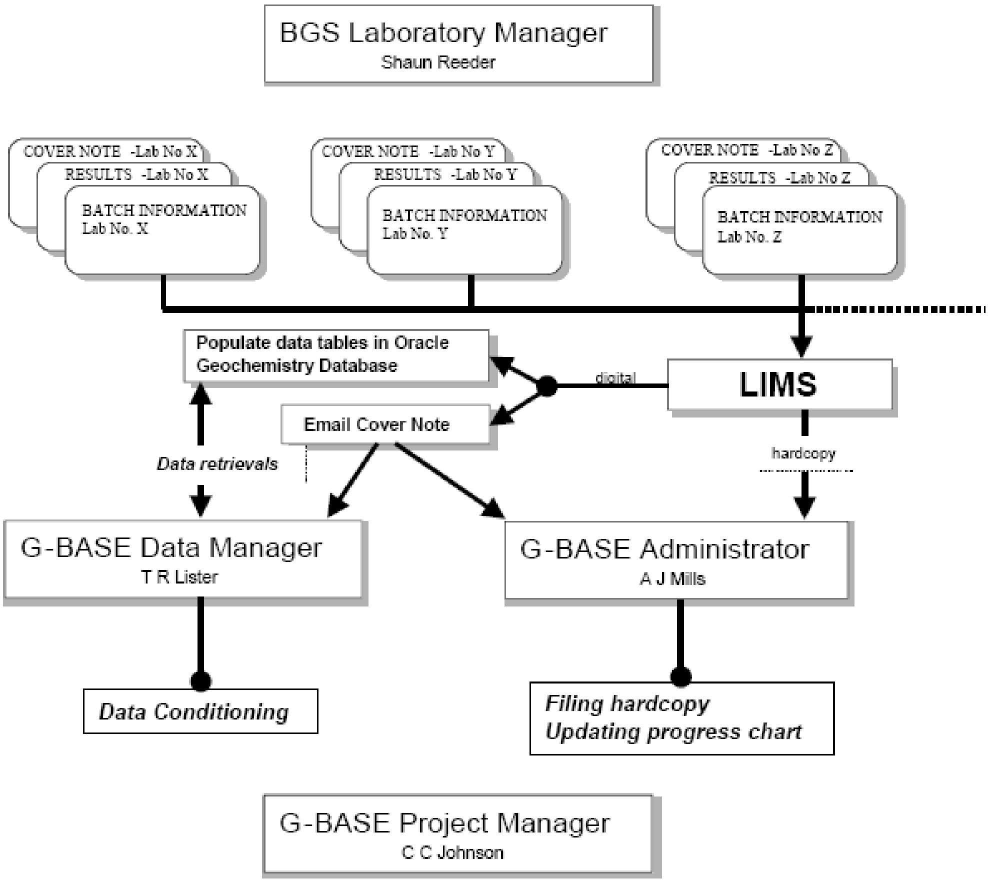
Data can be exported to Excel for printing out and verification by the relevant section head. After verification, data are given ‘released’ status within the LIMS, effectively locking the data so that no further changes can be made. Released status is achieved by highlighting relevant data in the data entry page and clicking on the “Release” icon on the button bar. It is worth noting that although released data cannot be edited manually they can still be overwritten by automatic loading scripts.

Security is set so that only certain individuals have the privileges that allow release and unrelease of data.

5 Data Reporting

Once all data have been released as verified, data can be reported to the client. The flow of procedures when results are sent from the laboratories is illustrated in figure 2 taken from *Lister and Johnson, 2005.*

Figure 2 Reporting procedures



5.1 TEST REPORTS AND COVER NOTES IN MS EXCEL AND MS WORD

A LIMS action is used to transfer analytical results from the LIMS tables into an Excel spreadsheet, and a further action is used to produce the cover note that accompanies the data. These actions produce output similar to that produced by earlier manual methods in Excel and Word.

A LIMS action is a small program written within the LIMS in (this case) Visual Basic Script, a cut-down version of Visual Basic. The LIMS provides some ready written actions that can be incorporated into the code to help with system calls and routine functions.

The action that produces the Test Report is comprised of a number of queries of the LIMS tables, the data retrieved being written into a MS Excel spreadsheet to produce a report similar to those produced in Excel for the normal manual reporting, as in figure 3.

Figure 3 Example Test Report

		XRFED	XRFED	XRFED	XRFED	XRFED	XRFED	XRFED	XRFED
Customer ID	LIMS ID	Ag	Cd	In	Sn	Sb	Te	I	Cs
		ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
450501	11215-0001	0.1	0.4	-97.0	2.4	0.9	-97.0	4.6	4
450502	11215-0002	-97.0	-97.0	-97.0	2.7	0.8	-97.0	3.3	4
450504	11215-0003	-97.0	0.1	-97.0	1.9	0.7	-97.0	4.9	4
450505	11215-0004	-97.0	-97.0	-97.0	7.3	1.4	-97.0	4.1	3
450506	11215-0005	-97.0	0.3	0.1	3.2	0.5	0.2	3.8	4
450507	11215-0006	-97.0	0.1	0.2	3.0	0.6	-97.0	2.1	2
450508	11215-0007	-97.0	0.4	-97.0	2.7	0.5	-97.0	2.5	4
450509	11215-0008	-97.0	0.6	-97.0	2.8	0.4	0.3	5.3	4
450510	11215-0009	-97.0	-97.0	0.3	2.5	0.7	-97.0	2.3	3
450511	11215-0010	-97.0	-97.0	-97.0	2.4	0.6	0.1	4.3	4
450512	11215-0011	-97.0	0.2	-97.0	3.1	0.9	-97.0	3.7	3
450513	11215-0012	-97.0	0.7	0.1	5.1	0.6	0.1	2.8	4

The action that produces the cover note queries the LIMS tables for the necessary information and inserts it into a pre-defined MS Word template. The resulting document resembles the cover notes produced by manual means. See appendix 3.

5.2 DIRECT TO GEOCHEMISTRY DATABASE RAW DATA TABLES

Data are transferred from the LIMS to the raw geochemistry database data tables by means of a Microsoft Access application. The application queries the LIMS tables for the required information and loads the information into the relevant tables in the Geochemistry Database. The raw geochemistry database tables and the LIMS tables are linked to the application using ODBC.

Nominated LIMS staff transfer analytical results and related ancillary data for batches of samples directly to the BGS database tables which comprise the “raw” geochemical analyses database. The “raw” database stores the data values exactly as received from the LIMS, with no changes at all. This is different from the “modified” geochemistry database that holds “conditioned” data, *Lister and Johnson, 2005.* This method of transfer gives staff immediate access to raw data whereas the “conditioned” data may take several years to appear in the Geochemistry Database.

The design of the “raw data” database tables differs from the “modified data” table design in that several results for the same element but from different calibrations by the same method on the same sample can be stored. Details of which calibration is best for each parameter are stored in the RAW_ANALYTE_LIMITS table (see appendix 5). This choice typically would depend on concentration level. The abundances are stored in their original units unlike the modified data table where data are converted to mg/kg where possible.

Users will be prompted for both Oracle (Geochemistry Database) and Microsoft SQL Server (LIMS) IDs and this should provide adequate security. Only nominated staff will have access to the application directory.

To enable verification of the data transferred, the program allows viewing of the data, in the form that it will be transferred to the geochemistry database, before transfer is initiated.

Transfer staff make a quick visual check to verify the data are correct, e.g. correct analytes, limits, etc. before initiating the transfer. The program runs queries to populate each table in the raw database in turn. In addition, automatic checks are made before transfer to the raw analytes table to:

1. list any analytes for which there are no detection limits entered in the LIMS, and

2. list any results that have no display units entered.

If the checks fail the application will not continue to append data to the geochemistry raw database tables.

The LIMS administrator and the G-BASE data manager and administrator (see figure 2) are notified by e-mail when the data transfer is successful. If the transfer is stopped at any stage due to data errors, the errors must be corrected and the application restarted from the stage it had reached. This is done rather than from the start at stage 1, since the data in the previous stages will already have been loaded. The stages must be run in order; it is not possible to load data at any stage if a previous stage has been missed out. An Oracle error will be displayed if this is attempted.

A summary of what has been loaded to the raw geochemistry database analyte details table is stored in the access table called LOADING_LOG linked to the application and also visible in the bulk G-BASE data loader application.

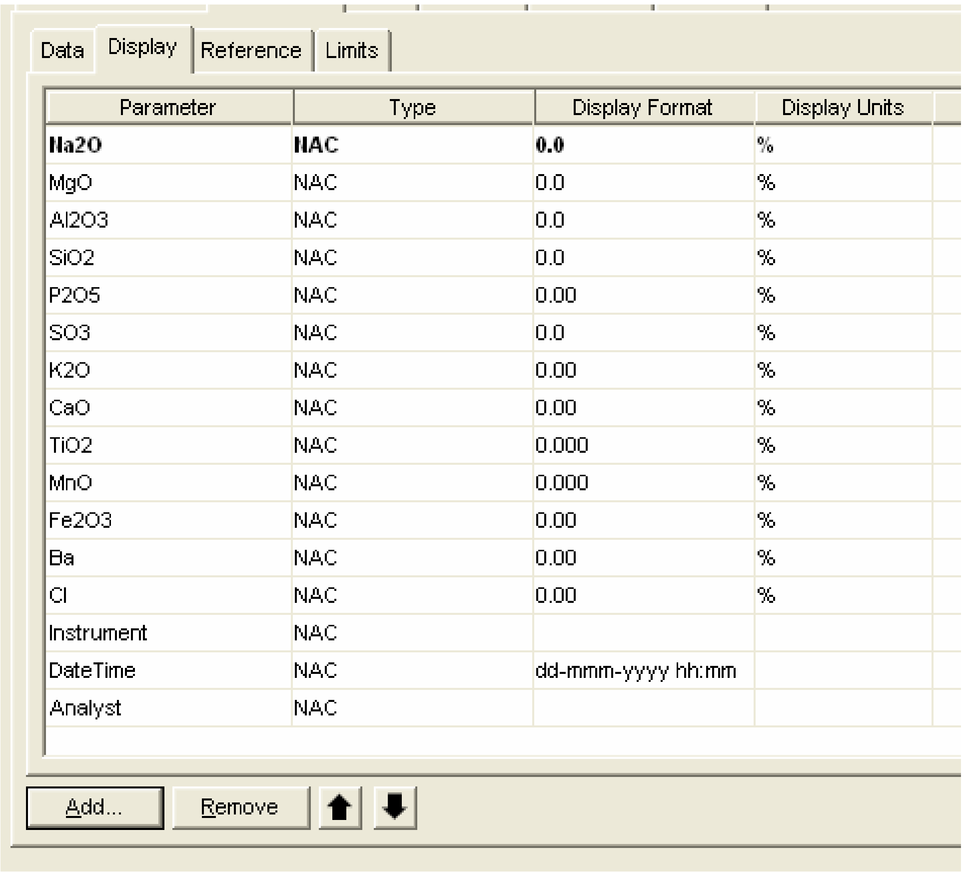
See appendix 4 for a more detailed description of the workings of the application and appendix 5 for a diagram of the tables.

6 Final Summary

There are now systems in place for analytical data for G-BASE solid samples to be recorded in a LIMS. Data values from various analytical methods can be automatically collated into the usual hardcopy reports. In addition data can be automatically transferred to Oracle tables designed to hold raw unconditioned data. G-BASE staff will have immediate access to the data, no longer having to wait for the conditioned data to appear in the usual geochemistry data base tables.

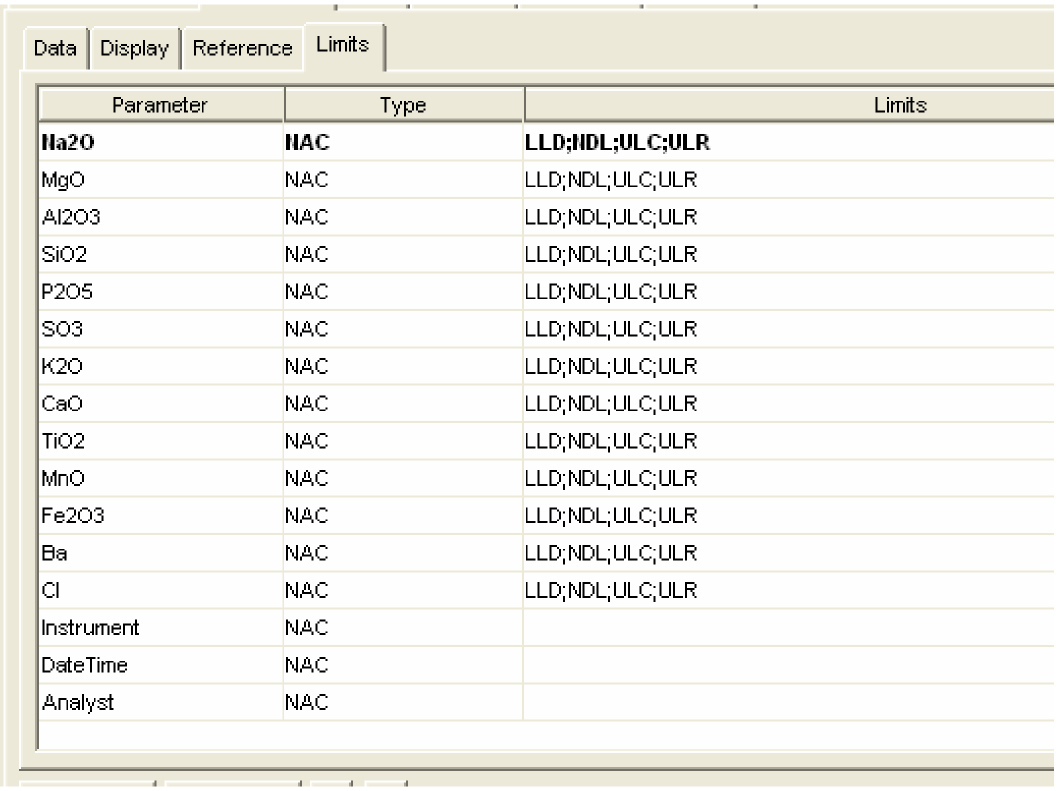
Appendix 1 Parameter Lists

Entering units and display formats for parameters in the P-Na-Fe Parameter List.



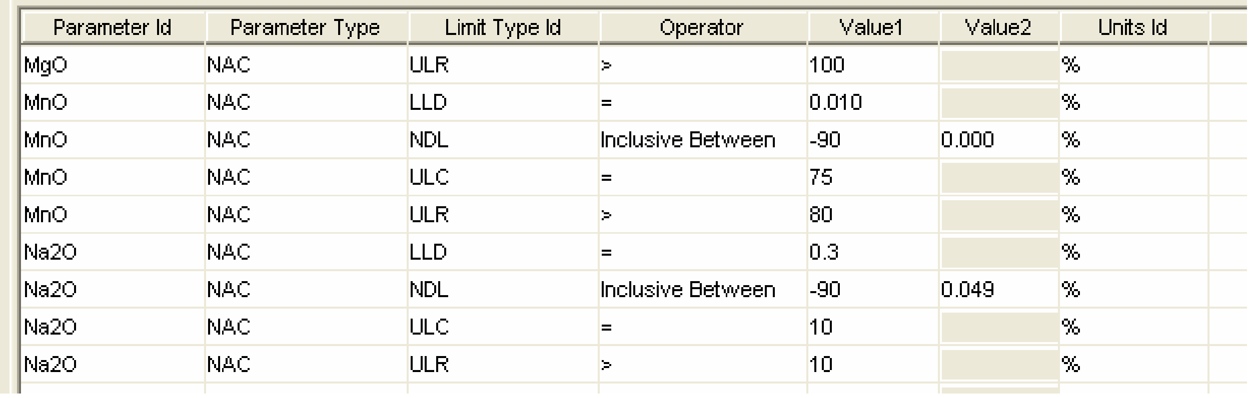
Parameter	Type	Display Format	Display Units
Na2O	**NAC**	**0.0**	%
MgO	NAC	0.0	%
Al2O3	NAC	0.0	%
SiO2	NAC	0.0	%
P2O5	NAC	0.00	%
SO3	NAC	0.0	%
K2O	NAC	0.00	%
CaO	NAC	0.00	%
TiO2	NAC	0.000	%
MnO	NAC	0.000	%
Fe2O3	NAC	0.00	%
Ba	NAC	0.00	%
Cl	NAC	0.00	%
Instrument	NAC		
DateTime	NAC	dd-mmm-yyyy hh:mm	
Analyst	NAC		

Assigning limits to parameters in the P-Na-Fe Parameter List.



Parameter	Type	Limits
Na2O	**NAC**	**LLD;NDL;ULC;ULR**
MgO	NAC	LLD;NDL;ULC;ULR
Al2O3	NAC	LLD;NDL;ULC;ULR
SiO2	NAC	LLD;NDL;ULC;ULR
P2O5	NAC	LLD;NDL;ULC;ULR
SO3	NAC	LLD;NDL;ULC;ULR
K2O	NAC	LLD;NDL;ULC;ULR
CaO	NAC	LLD;NDL;ULC;ULR
TiO2	NAC	LLD;NDL;ULC;ULR
MnO	NAC	LLD;NDL;ULC;ULR
Fe2O3	NAC	LLD;NDL;ULC;ULR
Ba	NAC	LLD;NDL;ULC;ULR
Cl	NAC	LLD;NDL;ULC;ULR
Instrument	NAC	
DateTime	NAC	
Analyst	NAC	

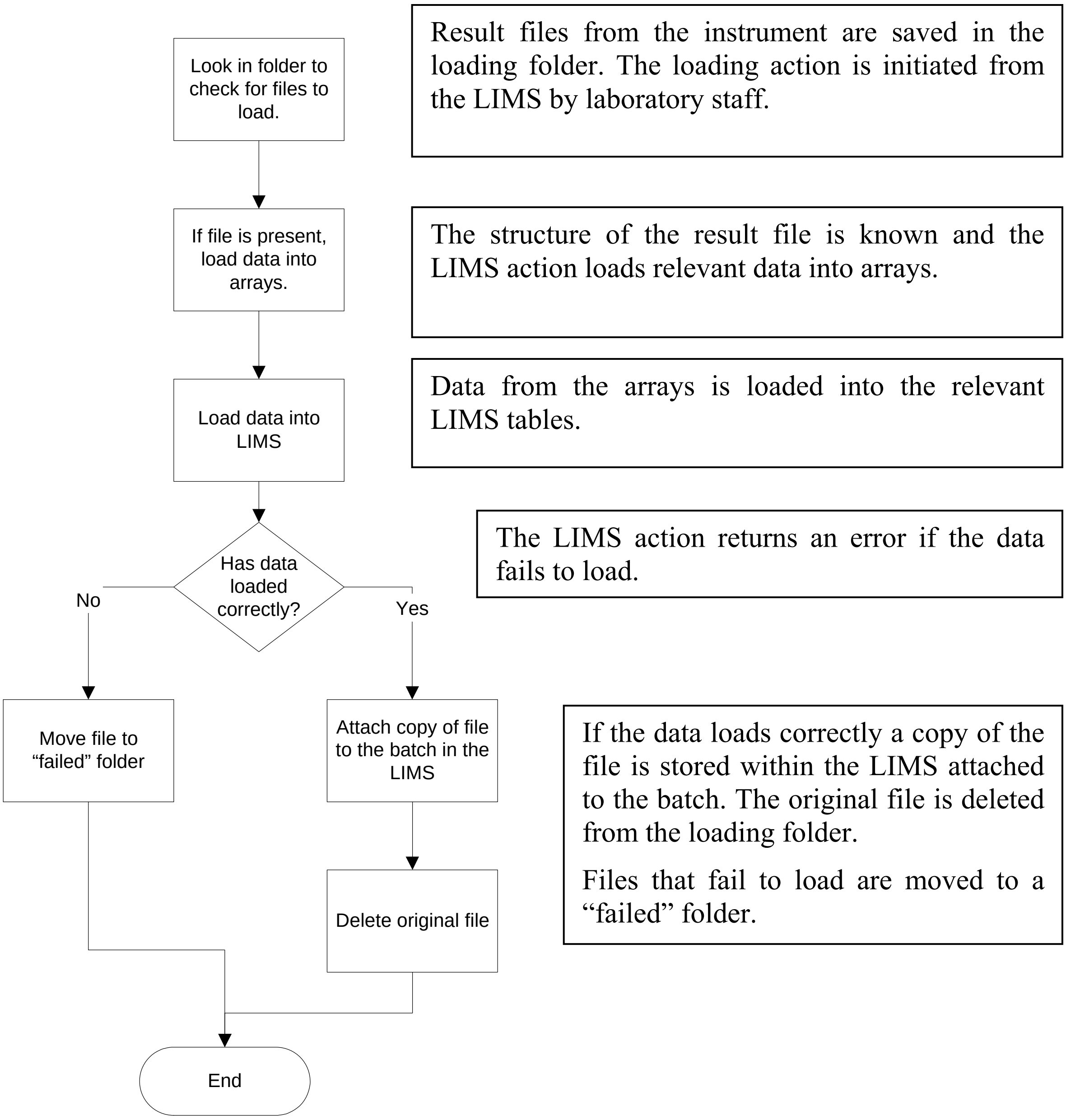
Setting the values for the limits



Parameter Id	Parameter Type	Limit Type Id	Operator	Value1	Value2	Units Id
MgO	NAC	ULR	>	100		%
MnO	NAC	LLD	=	0.010		%
MnO	NAC	NDL	Inclusive Between	-90	0.000	%
MnO	NAC	ULC	=	75		%
MnO	NAC	ULR	>	80		%
Na2O	NAC	LLD	=	0.3		%
Na2O	NAC	NDL	Inclusive Between	-90	0.049	%
Na2O	NAC	ULC	=	10		%
Na2O	NAC	ULR	>	10		%

Appendix 2 Semi automatic data loading

XRFS Data Loading



Result files from the instrument are saved in the loading folder. The loading action is initiated from the LIMS by laboratory staff.

The structure of the result file is known and the LIMS action loads relevant data into arrays.

Data from the arrays is loaded into the relevant LIMS tables.

The LIMS action returns an error if the data fails to load.

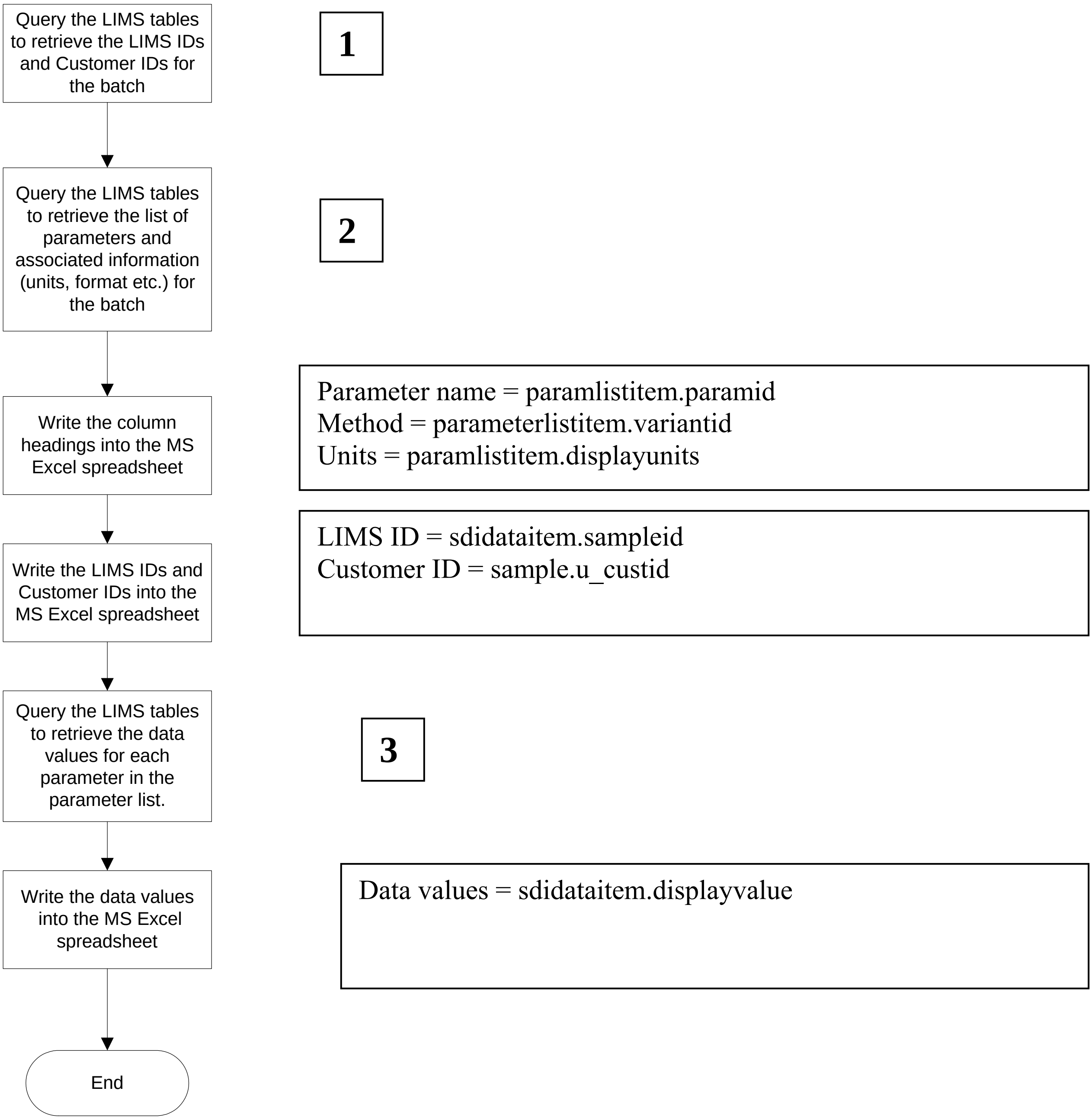
If the data loads correctly a copy of the file is stored within the LIMS attached to the batch. The original file is deleted from the loading folder.

Files that fail to load are moved to a “failed” folder.

Appendix 3 Test Reports and Cover Notes

TEST REPORTS

The code that produces the Test Report is comprised of a number of queries of the LIMS tables; the data retrieved being written into a MS Excel spreadsheet to produce a report similar to those produced in Excel for the normal manual reporting. A simplified flow is given below:



Note This diagram is a simplified account of the process. The program retrieves data for each parameter list in turn and enters them into the spreadsheet until all the data are retrieved. There are error trapping routines which are also not displayed.

1 Query for LIMS IDs and Customer Ids

```
select distinct sampleid, u_custid from sample, sdidataitem, u_batch where left(sampleid,5) like
strBatch and (ISNUMERIC(substring(sampleid,7,1))=1) and (sdidataitem.keyid1=sample.sampleid)
order by sample.sampleid
```

Notes

strBatch is a variable holding the batch number

The query checks to see that the 7th character of the LIMS ID is numeric to filter out any reference samples where the 7th character would be a letter.

2 Queries for parameter names and associated information

```
select paramid from paramlistitem where paramlistid = aryPlists(i)
```

Note aryPlists is an array of parameter lists for the batch.

```
select VariantId, NumReplicates, DisplayUnits, DisplayFormat, MandatoryFlag from paramlistitem
where ParamlistId = aryPlist(i) and Paramid = aryParams(m)
```

Notes

aryParams is an array of parameters contained in the parameter list

VariantId is the method name, e.g. XRFED, XRFWD, LOI450.

NumReplicates is a number representing the instance of the data value for the parameter within the parameter list, i.e. some parameters may be measured a number of times giving, for example three values.

DisplayUnits, are the units the parameter is measured in, e.g. %, ppm etc.

DisplayFormat is the format in which the data are to be reported, e.g. DD MMM YYYY for dates or 0.00 for numbers with two decimal places.

MandatoryFlag, this is set to Y if the parameter is mandatory, i.e. to be reported to the customer or N if the parameter is not Mandatory, i.e. it is not to be reported to the customer, it was measured for analytical reasons, e.g. an interference correction.

3 Query for data values

```
select displayvalue from sdidataitem where keyid1 = arySids(j) and ParamListId = aryPlists(i) and
(ParamId =xxx) and DataSet =(k)
```

Notes

This query is run in a loop to retrieve all parameters for all parameter lists for all samples within the batch.

arySids(i) is the array of LIMS Ids (sample numbers) for the batch

(ParamId = xxxx) this represents a list of all available parameters measured/recorded for the batch

DataSet is a number representing the instance of the parameter list for the sample, i.e. the parameter list (as a whole, the just the parameter within the parameter list as for NumReplicate) can be measured several times for the same sample.

COVER NOTES

The Cover Notes are produced by inserting relevant information from the LIMS and relevant files into a Cover Note template like the one below as directed by the Visual Basic routine:

Kingsley Dunham Centre
Keyworth
Nottingham
United Kingdom
NG12 5GG

To:
[u_report_to]
British Geological Survey
Kingsley Dunham Centre
Keyworth
Nottingham
NG12 5GG

Telephone	+44 (0)115 936 3100
Direct line	+44 (0)115 936 3341
Fax	+44 (0)115 936 3200
E-mail	mni@bgs.ac.uk

ANALYTICAL GEOCHEMISTRY LABORATORIES

ANALYSIS REPORT COVER NOTE

This report consists of a 35 page Analysis Report Cover Note and 2 pages of test data

---

Report Number:	[Batch]/1	Customer Ref/Order No:	[u_order]
Report Date:	[RepDate]	Sample(s) received on:	[u_samrec_on]
Issue Status:	Complete	Analysis commenced on:	[Analysis_on]
No of samples:	[inosams]	Analysis finished on:	[Analysis_end]

Sample Details

All samples were received in good condition.

[lightweights]

[OUTMAK]

[OUTpHs]

[nosample1]
[nosample2]
[nosample3]
[nosample4]
[nosample5]
[nosample6]

Analysis Details

Determinands	Test Method	No of Samples	Notes
[params1]	[plist1]	[inosams1]	
[params2]	[plist2]	[inosams2]	
[params3]	[plist3]	[inosam	

		s3]	
[params4]	[plist4]	[inosams4]	
[params5]	[plist5]	[inosams5]	
[params6]	[plist6]	[inosams6]	

QC Determinands	QC Test Method	No of QC Samples	Notes
[qcparams1]	[qcplist1]	[inoQCs1]	
[qcparams2]	[qcplist2]	[inoQCs2]	
[qcparams3]	[qcplist3]	[inoQCs3]	
[qcparams4]	[qcplist4]	[inoQCs4]	
[qcparams5]	[qcplist5]	[inoQCs5]	
[qcparams6]	[qcplist6]	[inoQCs6]	
[qcparams7]	[qcplist7]	[inoQCs7]	
[qcparams8]	[qcplist8]	[inoQCs8]	
[qcparams9]	[qcplist9]	[inoQCs9]	
[qcparams10]	[qcplist10]	[inoQCs10]	

Tests marked N in the above table are not included in the UKAS Accreditation Schedule for this Laboratory.

Data for some elements are released beneath the usual reporting limits and to additional decimal places at the Client's request.

The $CaCl_2$ slurry method was used to determine pH. Due to the nature of the material of some peat rich samples, it might be necessary to change the usual sample to solution ratio in order to obtain a suitable slurry from which a pH measurement can be obtained. Brief experiments have shown that this has no significant effect on the pH measurement. The scope of the pH method is between pH 3 and 10. Values outside the scope should be used only as a guide.

The aliquot for LOI was dried overnight at 105ºC prior to heating at 450ºC for a minimum of 4 hours.

The XRF QC standards do not contain In, I or Sm. The QC status of these elements is inferred from a spectrographically adjacent element.

[ULCs]

Data for some analytes may have been reported with the value –94 to –99. These codes are defined in the list below:

Code	Explanation
-94	Insufficient sample (e.g. sample collected but not enough to analyse).
-95	Not determined because of high concentration; but exceeds the calibration limit (XRFS cannot provide a value but the analyte concentration is over the ULR).
-96	Not determined because of interference; probably of high concentration (e.g. spectral overlap from a trace element present at high concentration but we can see the analyte concentration is high).
-97	Not determined, possibly because of interference; probably of low concentration (e.g. matrix effects or severe line overlap on trace element, so value is very negative i.e. <NDL or ≤0 for elements determined by ED(P)XRFS).
-98	Not determined because of interference; no estimate (e.g. spectral overlap from a trace element present at high concentration and XRFS cannot provide a value).

-99	Absent data (e.g. analyte not requested).

Because of limitations with the current software used for reporting data, the number of significant figures quoted in the attached table may not be representative of the actual uncertainty. Data should be considered accurate to no more than three significant figures.

This report is issued under complete status. All analyses requested have been completed and results are issued with full compliance of data verification subject to the statements above.

Report authorised by: ………………………………………. Date: ……………………….

Mark N Ingham
X-Ray Fluorescence Spectrometry Section Manager

Notes

Text highlighted in pink are to be checked and edited manually.

Text in square brackets is to be replaced by information from the LIMS or associated files.

The resulting Cover Note will look something like this:

Kingsley Dunham Centre
Keyworth
Nottingham
United Kingdom
NG12 5GG

To:
T R Lister
British Geological Survey
Kingsley Dunham Centre
Keyworth
Nottingham
NG12 5GG

Telephone +44 (0)115 936 3100
Direct line +44 (0)115 936 3341
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E-mail mni@bgs.ac.uk

ANALYTICAL GEOCHEMISTRY LABORATORIES

ANALYSIS REPORT COVER NOTE

This report consists of a 35 page Analysis Report Cover Note and 89 pages of test data

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Report Number:	11231/1	Customer Ref/Order No:	IR 02056/57/58
Report Date:	28 February 2006	Sample(s) received on:	08 August 2005
Issue Status:	Complete	Analysis commenced on:	22 October 2005
No of samples:	545	Analysis finished on:	16 February 2006

Sample Details

All samples were received in good condition.

Analysis Details

Determinands	Test Method	No of Samples	Notes
LOI	Physical measurement	535	N
pH	pH electrode AGN 2.5.1	535	
Ag, Cd, In, Sn, Sb, Te, I, Cs, Ba, La, Ce	ED-XRF Slow Speed Pressed Powder Pellets Ag-Ce AGN 2.1.6	545	
Na2O, MgO, Al2O3, SiO2, P2O5, SO3, CaO, TiO2, Fe2O3, Ba, Cl	WD-XRF G-BASE pellets Na-Fe	545	N
K2O, CaO, S, Cl	WD-XRF G-BASE pellets Pro-Trace	545	N
TiO2, MnO, Fe2O3, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Nd, Sm, Yb, Hf, Ta, W, Tl, Pb, Bi, Th, U	WD-XRF G-BASE pellets Pro-Trace AGN 2.1.5	545	

QC Determinands	QC Test Method	No of QC Samples	Notes
Ag, Cd, In, Sn, Sb, Te, I, Cs, Ba, La, Ce	ED-XRF Slow Speed Pressed Powder Pellets Ag-Ce AGN 2.1.6	8	

Na2O, MgO, Al2O3, SiO2, P2O5, SO3, CaO, TiO2, Fe2O3, Ba, Cl	WD-XRF G-BASE pellets Na-Fe	8	N
K2O, CaO, S, Cl	WD-XRF G-BASE pellets Pro-Trace	8	N
TiO2, MnO, Fe2O3, Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Nd, Sm, Yb, Hf, Ta, W, Tl, Pb, Bi, Th, U	WD-XRF G-BASE pellets Pro-Trace AGN 2.1.5	8	

Tests marked N in the above table are not included in the UKAS Accreditation Schedule for this Laboratory.

Data for some elements are released beneath the usual reporting limits and to additional decimal places at the Client's request.

The $CaCl_2$ slurry method was used to determine pH. Due to the nature of the material of some peat rich samples, it might be necessary to change the usual sample to solution ratio in order to obtain a suitable slurry from which a pH measurement can be obtained. Brief experiments have shown that this has no significant effect on the pH measurement. The scope of the pH method is between pH 3 and 10. Values outside the scope should be used only as a guide.

The aliquot for LOI was dried overnight at 105ºC prior to heating at 450ºC for a minimum of 4 hours.

The XRF QC standards do not contain In, I or Sm. The QC status of these elements is inferred from a spectrographically adjacent element.

Samples with Cu >1000ppm will affect Sc, V, Cr, Co, Ni, Cu, Zn, Nd, Sm, Yb, Hf, Ta, W data and possibly other elements. The specified trace element data have not been corrected for this effect.

Samples with Pb >1000ppm will affect Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nd, Sm, Yb, Hf, Ta, W, Tl, Pb, Bi, Th, U data and possibly other elements. The specified trace element data have not been corrected for this effect.

Samples with Zn >1000ppm will affect Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Nd, Sm, Yb, Hf, Ta, W data and possibly other elements. The specified trace element data have not been corrected for this effect.

Samples with Zr >1000ppm will affect Sc, V, Cr, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br, Rb, Sr, Y, Zr, Nb, Mo, Nd, Sm, Yb, Hf, Ta, W, Tl, Pb, Bi, Th, U data and possibly other elements. The specified trace element data have not been corrected for this effect.

Samples with Ba >1000ppm will affect Ag, In, Sn, Sb, I, Cs and possibly other elements. The specified trace element data have not been corrected for this effect.

Data for some analytes may have been reported with the value –94 to –99. These codes are defined in the list below:

Code	Explanation
-94	Insufficient sample (e.g. sample collected but not enough to analyse).
-95	Not determined because of high concentration; but exceeds the calibration limit (XRFS cannot provide a value but the analyte concentration is over the ULR).
-96	Not determined because of interference; probably of high concentration (e.g. spectral overlap from a trace element present at high concentration but we can see the analyte concentration is high).
-97	Not determined, possibly because of interference; probably of low concentration (e.g. matrix effects or severe line overlap on trace element, so value is very negative i.e. <NDL or ≤0 for elements determined by ED(P)XRFS).
-98	Not determined because of interference; no estimate (e.g. spectral overlap from a trace element present at high concentration and XRFS cannot provide a value).
-99	Absent data (e.g. analyte not requested).

Because of limitations with the current software used for reporting data, the number of significant figures quoted in the attached table may not be representative of the actual uncertainty. Data should be considered accurate to no more than three significant figures.

This report is issued under complete status. All analyses requested have been completed and results are issued with full compliance of data verification subject to the statements above.

Report authorised by: ………………………………………. Date: ……………………….

Mark N Ingham
X-Ray Fluorescence Spectrometry Section Manager

Appendix 4 Data Transfer. LIMS to Geochemistry Database

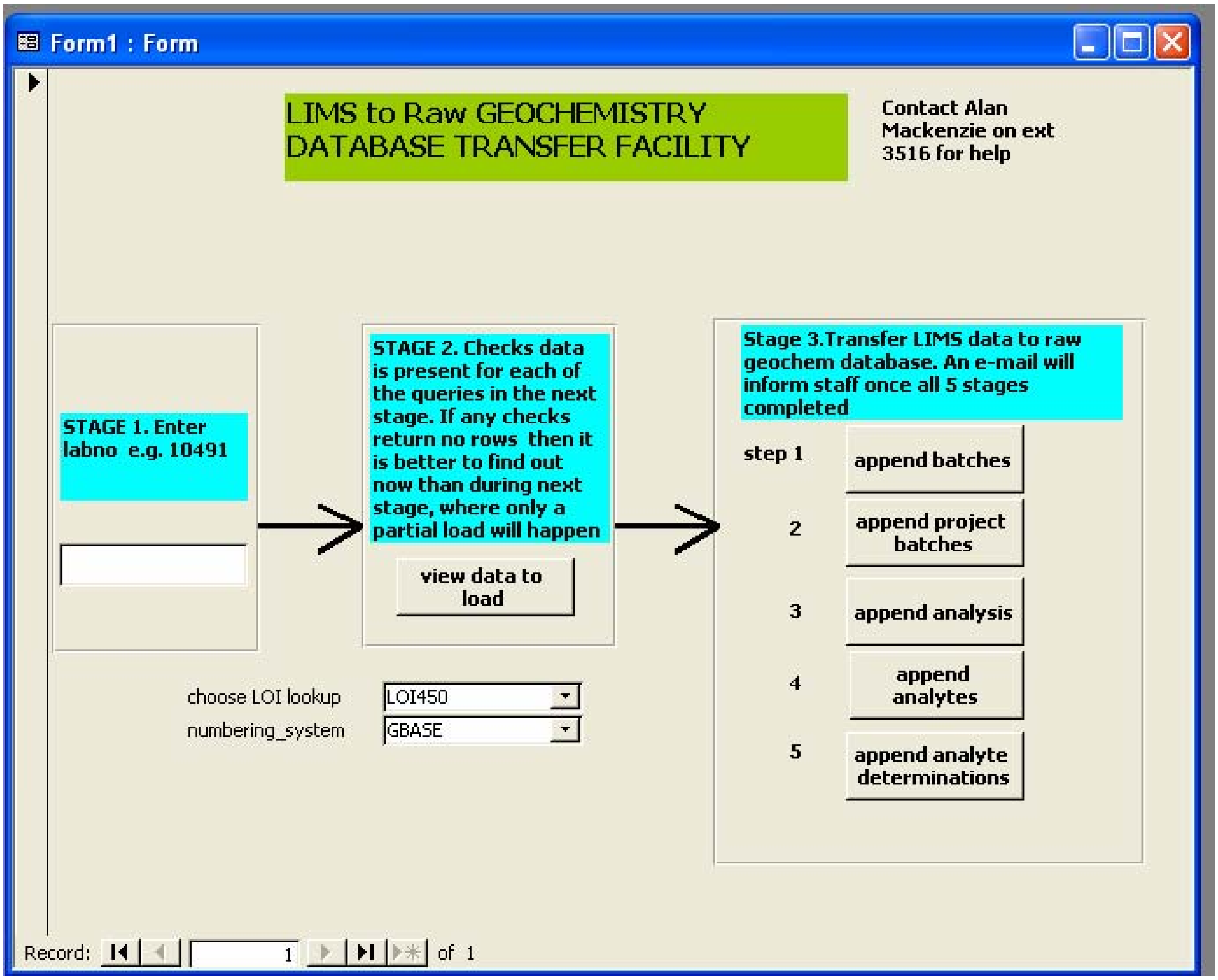
Transfer from LIMS to Geochemistry Raw Database Tables is achieved via a Microsoft Access application.

This application is not designed for multi users. The program checks if someone else is using the application and will stop and display a message if more than one person tries to use it at the same time.

When a user exits the application, all temporary tables have their contents removed. If another user tries to use the application whilst there are rows in these tables then they cannot proceed.

If there is a data loading error at stage 3 for whatever reason, e.g. attempting to load an analyte not recognized by the Geochemistry Database, or errors in the original data for loading, the application must be run again from stage 1 after any amendments to data have been made.

The form below is used to initiate the transfer:



LIMS to Raw GEOCHEMISTRY DATABASE TRANSFER FACILITY

Contact Alan Mackenzie on ext 3516 for help

STAGE 1. Enter labno e.g. 10491

STAGE 2. Checks data is present for each of the queries in the next stage. If any checks return no rows then it is better to find out now than during next stage, where only a partial load will happen

Stage 3.Transfer LIMS data to raw geochem database. An e-mail will inform staff once all 5 stages completed

choose LOI lookup: LOI450

numbering_system: GBASE

There are three stages with stages 2 and 3 containing several steps:

STAGE 1

The number of the batch is entered into the system. Default codes for LOI method (if applicable) and the numbering system are changed if needed.

STAGE 2

The application retrieves the information required for the Geochemistry Raw Database Tables from the LIMS. It performs queries in the following order:

1. The **sdidataitem_view Select** query retrieves information to create a view of the LIMS tables.

The query selects fields from the table sdidataitem. This table holds the result data. Information is retrieved where:

> The field KeyId1 (the sample number) begins with the batch number entered from the form, e.g. batch number = 11215, KeyId1 = 11215-0001 to 11215-0450.
>
> The MandatoryFlag field is equal to "Y", i.e. only retrieves data it is mandatory to report, does not retrieve data collected for interference correction etc.
>
> The SDCId field is equal to "SAMPLE"
>
> The field ParamId is not equal to DateTime, Instrument or Analyst
>
> VariantId is equal to XRFED, XRFWD; LOI or pH, *i.e. only retrieves data for selected methods, e.g.*
>
> *variantId in ('XRFWD','XRFED','LOI','pH').*

2. The **batches** query information about the batch

Geochemistry	**LIMS**	**Comment**
lab .	Not present.	The analytical laboratory responsible for issuing the analysis batch number Not retrieved from LIMS, always “BGSL”.
batch_id	u_batch.u_batchid	The batch number, taken from the form in the application, entered manually by the lab staff transferring the data.
registered	u_batch.u_reg_date	Date of registration of the batch, taken as the date the samples were registered (by G-BASE)
reg_date_accuracy	Not present.	Accuracy of the date of registration of the batch. Not retrieved from LIMS, always “D”.
geographical_area	u_batch.u_geogarea	Geographical area from which the samples were collected.
locality	u_batch.u_locality	Geographical locality from which the samples were collected.
notes	u_batch.batchdesc	Notes on the samples in the batch which may be of use to the analytical or sample preparation staff.
numbering_system	Not present.	From drop down list on the form.

3. The **analysis query** retrieves information about which analysis were requested.

Geochemistry	**LIMS**	**Comment**
lab	Not present.	The analytical laboratory responsible for issuing the analysis batch number. Not retrieved from LIMS, always "BGSL".
batch_id	u_batch.u_batchid	The batch number, taken from the form in the application, entered manually by the lab staff transferring the data.
method	sdidataitem_view.variantId*	The method of analysis carried out on the samples, e.g. XRFED or pH.*
requested_by	u_batch.date_reg	Name of the person who requested the analysis, e.g. Chris Johnson.
requested	u_batch.u_reg_date	Date on which the analysis was requested, see registered above.
date_accuracy	Not present.	Accuracy of the date of registration of the batch. Not retrieved from LIMS, always "D"
requested_for	u_batch.date_req	Date by which the analysis is to be reported.
req_for_date_accuracy	Not present.	Accuracy of the requested for date. Not retrieved from LIMS, always "D"
received	u_batch.u_samrec_on	Date on which the batch of samples was received by the analytical laboratory.
rec_date_accuracy	Not present.	Accuracy of "received on" date. Not retrieved from LIMS, always "D"
reported	Not present.	Taken as "Now()" the date the results are transferred to the raw database tables.
rep_date_accuracy	Not present.	Accuracy of the reported date. Not retrieved from LIMS, always "D"

* This is the varianId from the LIMS unless the parameter is LOI when the user chosen value, LOI450 (LOI at 450°C) or LOI1050 (LOI at 1050°C) from the drop-down is used.

4. The **project_batches** query retrieves information about the samples within the batch.

Geochemistry	**LIMS**	**Comment**
lab	Not present.	The analytical laboratory responsible for issuing the analysis batch number. Not retrieved from LIMS, always "BGSL".
batch_id	u_batch.u_batchid	The batch number, taken from the form in the application, entered manually by the lab staff transferring the data.
sample_type	u_batch.u_samtype	Classification of the sample type of the batch. A code letter, e.g. "A" or "C"
numbering_system	Not present.	From drop down list on the form.
project	The first 2 characters of u_batch.u_custid	Geochemistry project code referring to a collector or an area.
min_siteno	u_batch.u_minno	Lowest site number for the batch.
max_siteno	u_batch.u_maxno	Highest site number for the batch.
numb_of_sample	u_num_sam	Total number of samples in the project batch including controls as entered by G-BASE staff at registration.

5. The **analytes** query retrieves information about the parameters measured/recorded.

Geochemistry	**LIMS**	**Comment**
lab	Not present.	The analytical laboratory responsible for issuing the analysis batch number. Not retrieved from LIMS, always "BGSL".
u_batchid	u_batch.u_batchid	The batch number, taken from the form in the application, entered manually by the lab staff transferring the data.
calibration_name	sdidataitem_view.paramListId	The specific analytical method task, a name for a suite of elements determined by a particular analytical method under a specific set of rules and conditions.
method	sdidataitem_view.variantId*	The method code of analysis carried out on the samples, e.g. XRFED or pH.*
analyte	sdidataitem_view.paramId	Name of analyte determined, e.g. LOI, Ag, Fe2O3.
units	sdidataitemview.displayUnits	Units for the analyte, e.g. mg/kg.
Limit_Type	SDIDataItemLimits.limitTypeID	Limit type, e.g. ULC (Upper Limit of Calibration), ULR (Upper Limit of Reporting).
Operator	SDIDataItemLimits.operator	Operator on limit value, e.g. "=" or "<".
limit_Value	SDIDataItemLimits.value1	Limit value, a number, e.g. a lower range limit value.
limit_Value2	SDIDataItemLimits.value2	Limit value, a number, e.g. an upper range limit value.

* This is the variantId from the LIMS unless the parameter is LOI when the user chosen value, LOI450 (LOI at 450°C) or LOI1050 (LOI at 1050°C) from the drop-down is used.

The retrieved information is used to populate the dta_raw_analytes table.

6. The **analyte_determinations** query retrieves the result values.

Geochemistry	**LIMS**	**Comment**
numbering_system	Not present.	From drop down list on the form.
project	The first 2 characters of u_batch.u_custid	Geochemistry project code referring to a collector or an area.
siteno	The last 4 characters of u_batch.u_custid	The number given to the site, the sample number within the project.
lab	Not present.	Not retrieved from LIMS, always “BGSL”.
method	sdidataitem_view.variantId*	The method of analysis carried out on the samples, e.g. XRFED or pH.*.
analyte	sdidataitem_view.paramId	The analyte determined by the analysis, the analyte name, e.g. LOI, Ag, Fe2O3.
abundance	sdidataitem_view.displayValue	The determined abundance of the analyte.
units	sdidataitem_view.displayUnits**	Units for the analyte, e.g. mg/kg.
duplicate	Not present.	Not retrieved from LIMS, always “+”
batch_id	u_batch.u_batchid	The batch number, taken from the form in the application, entered manually by the lab staff transferring the data.
sample_type	batch.u_samtype	Code to denote sample type. e.g. “A” or “C”
calibration_name	batchg.paramListId	The parameter list name.
date_analysed	sdidataitem_view.displayValue***	The date of analysis for the parameter (often just one date for the parameter list)

* This is the variantId from the LIMS unless the parameter is LOI when the user chosen value, LOI450 (LOI at 450°C) or LOI1050 (LOI at 1050°C) from the drop-down is used.

** Some analytes have no units, e.g. pH and are assigned a value’pH’ as a unit.

*** The displayValue where the parameterListItem equals “DateTime”.

Note The non reference analyte determinations from LIMS are shown in one query and those from reference materials in another. The rows which go into each table are determined by the result of the expression... IsNumeric(Mid([dbo_Sample].[sampleid],7,5))

The data retrieved from these queries are displayed on screen and the view is used to verify that the queries have worked correctly.

The data retrieved are stored in temporary data tables ready for transfer at stage 3.

STAGE 3

The results from the queries at stage 2 are appended from the temporary data tables to the relevant Geochemistry Raw Database Tables. It is not possible to append data without first running stage 2 to run the queries. The data must be appended in the order, step 1 to step 5. The raw data tables are designed to correspond with the similarly named tables from the Geochemistry "amended" Database Tables and various codes used are defined in dictionary tables (*Harris and Coats 1992).*

Appendix 5 Raw Geochemistry Database Tables

Glossary

LIMS Laboratory Information Management System. A system of tools based around a relational database to provide the functionality of an automatic electronic filing system.

LOI Loss On Ignition. A method of determining the weight loss on heating of a sample. The heating temperature used will determine the likely sample fraction lost on heating. Loss at 450°C gives an indication of the amount of organic matter present.

pH A measure of acidity.

XRFS X-Ray Fluorescence Spectrometry. An analytical technique utilising x-rays. There are two types of XRFS spectrometer currently in use within BGS, wavelength dispersive, used in XRFWD methods and energy dispersive, used in XRFED methods.

References

Harris, J R and Coats, J. S, 1992, Geochemistry database: data analysis and proposed design, BGS WF/92/5

Lister, T R and Johnson, C C, 2005, G-Base data conditioning procedures for stream sediment and soil chemical analysis, BGS IR/05/150

Taylor, H, Vickers, B P and Moss-Hayes, V, 2005, Validation of the procedure for the determination of soil pH, BGS IR/05/080.