

EXPLORANIUM
RADIATION DETECTION SYSTEMS

GR-320LAB System
with LabSPEC Analysis Software

SYSTEM MANUAL
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with LabSPEC Analysis Software

METALS LABORATORY

RADIATION MONITORING SYSTEM

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METALS LABORATORY

RADIATION MONITORING SYSTEM

1. GENERAL

The GR-320LAB system has been developed primarily to meet the special requirements of the metals Industry. Recent accidents in steel making facilities have produced significant volumes of radioactive contaminated steel material. In addition the increasing number of nuclear plants and nuclear weapons facilities being de-commissioned has added to the large volume of contaminated material. Various government organisations are starting to enforce radiation content regulations as a means of preventing the importation of contaminated material that could become a health hazard to the public.

The International Atomic Energy Agency (I.A.E.A.) have studied this matter and have produced a proposed regulatory guidance document titled "Clearance levels for radio nuclides in solid materials" IAEA-TECDOC-855 dated January 1996. In this document, some of the proposed control levels are shown in the following table :

Isotope	Min level	Prop. Level
Co-60, Cs-137, Ra-226, Th-232, U-238, Am-241	0.1 Bq/g	0.3 Bq/g
Co-58, Sr-90, Ir-192, I-131	1.0 Bq/g	3 Bq/g
Co-57, I-125, Pu-241	10 Bq/g	30 Bq/g
Cd-109, Sr-89, Tc-99	100 Bq/g	300 Bq/g

Note that most jurisdictions currently have NO ACCEPTABLE LEVEL and in the case of the USA - the NRC specifies that their acceptable level is 0.00 Bq/g and each contamination event will be treated on a case-by-case basis.

However many countries are starting to develop standards and early indications are that the very low IAEA minimum limit is currently a de-facto standard in many countries.

THUS CURRENT PRACTICE IS FOR LABORATORY SYSTEM TO MEASURE TO THE MINIMUM LEVEL TO ENSURE PROPOSED REGULATORY COMPLIANCE (also see section 1.3 below)

Common usage in Europe is :

STEEL samples = 0.1 Bq/g (2.7 pCi/g)

DUST/SLAG = 0.12 Bq/g (3.24 pCi/g)

1.1. STEEL INDUSTRY CONCERNS

The primary concerns to the metals industry are :

- a) Cobalt (Co-60) - this is the primary problem as it alloys directly with the STEEL.
 - b) Cesium (Cs-137) - this is an important isotope which would occur in the DUST from the furnace as the high temperatures cause the Cs-137 to flash-off as a gas that then gets mixed in the Dust extraction system and is deposited in the Dust collectors.
 - c) Radium (Ra-226) - this isotope would occur in the SLAG from the furnace.
-

1.2. SAMPLING and ANALYSIS PRIORITIES

- a) STEEL - The majority of users have decided that the primary use of lab systems are to check the incoming steel sample for COBALT contamination as soon as possible after the sample is taken, to ensure that corrective action can be taken in the event of the identification of a contaminated sample. For this reason the GR-320LAB is supplied normally with only STEEL calibration samples but Dust/ Slag samples are optionally available (see (b) below).

Any other contaminants in the steel sample OTHER than Cobalt are detected and UNIDENTIFY ISOTOP PRESENT message is shown.

- b) DUST - unlike Steel samples from a melt that have a fixed uniform size, Dust sampling is more difficult due to the huge volume of material. Exploranium supplies special calibration samples for Dust analysis and special containers for sample preparation. QUANTATIVE analysis is available currently for 6 isotopes - Cesium, Cobalt, Radium, Potassium, Thorium and Americium as these are the most common probabilities.

Users can adopt whatever monitoring system is practical for their plants but sampling Dust once/day is probably a minimum. Some users advocate sampling ALL Dust locations every HOUR, but the selected sampling system must be adapted to suit local plant requirements. One of the primary reasons for Dust analysis is to get early warning if a Cesium or other source is melted. A secondary requirement is to ensure that Cesium contaminated Dust is not shipped off site. In both these cases, early detection of the Cesium will ensure that any contamination will be restricted to a relatively low volume of Dust, whereas late detection will usually result in relatively large volumes of contaminated Dust with a huge disposal cost.

FOR THESE REASONS EXPLORANIUM RECOMMENDS 1/ HOUR SAMPLING OF DUST AT ALL SITES, HOWEVER THESE RECOMMENDATIONS MUST BE ADAPTED TO SUIT LOCAL CONDITIONS.

It is important that the supplied sample containers are used and that they are filled as much as possible to ensure that all samples for analysis have approximately the same volume. This is easy for Dust samples as Dust is already in powder form. Also note that if the sample containers are reused, great care should be taken in cleaning to avoid sample contamination.

- c) SLAG - this is an even more difficult problem than Dust, as Slag is usually not readily accessible as it is at a high temperature and when it IS accessible, the material has cooled into large slabs. It is necessary to break off some representative material and then crush it into small fragments suitable for placing in an appropriate sample container. Most of the comments above for Dust sampling also apply to Slag sampling.
-
-

1.3. DETECTION LEVELS

Most users adopt the I.A.E.A. lower limits for Steel and a lower level for Dust/Slag

STEEL samples = 0.1 Bq/g (2.7 pCi/g)

DUST/SLAG samples = 0.12 Bq/g (3.24 pCi/g)

However some jurisdictions accept higher levels. For simplicity the manual has been written assuming the above detection levels, if they do not suit the user please consult with Exploranium to select a parameter range more suitable to local conditions.

1.4. GR-320LAB SYSTEM

The GR-320LAB system comprises 2 primary parts :

- the shielded detector chassis (lead shielded sample system)
 - an external computer running the Exploranium LabSPEC software
-

a) GR-320LAB chassis

The chassis comprises a 3" x 3" (21 cu ins = 0.35 dm³) Sodium-Iodide detector enclosed in a 2" (5 cm) lead shield. A specially designed sliding lid permits very easy sample loading and removal. The large volume sample -chamber (110 x 80 mm) allows use of a wide variety of sample sizes/shapes.

Note that STEEL samples typically are small in volume and high in density, whereas the Dust/Slag samples are lower density and need a higher sample volume to achieve the required sensitivity limits.

The main sample unit system has a built-in spectrometer system that is operated under the control of the system computer (PC) via the RS-232 serial data port.

The chassis runs directly from 12V DC (via a special AC adapter) and all system power is generated internally.

b) External PC

The external user supplied PC requires a minimum configuration of :

- 486+
- 64Mb RAM
- Hard drive - minimum 100M
- minimum VGA display
- at least 1 RS-232 port
- external printer
- Windows 95, 98, Windows 2000 only

The Exploranium LabSPEC software has been specially optimised to suit metals laboratory usage.

The software controls the spectrometer system and advises users of system operation, analyses data and produces printouts in a format suitable for the application.

The Exploranium LabSPEC software has been optimised to the screen 800 x 600 points. The software is automatically adapted to another screens, but it may not be so precise.

1.5. SUPPLIED SYSTEM

- 320LAB system chassis
 - External AC power adapter (110 V AC to 12 V DC)
 - RS-232 cable (2 m) to connect system to the remote PC
 - LabSPEC software (1 CD-ROM)
 - LabSPEC system manual
-

- 1 Cesium-137 (0.25 μ Ci) Test Source - for Gain stabilisation
- 1 set of Cobalt-60 standards (5) and certification sheet

OPTIONAL ITEMS :

- 220 V AC adapter (replaces the standard 110 V version)
 - Dust/Slag Calibration samples - set of 7
 - Dust/Slag sample containers - (Qty 100 lots)
 - Scale (user supplied)
-

1.6. GR-320LAB - PRINCIPLES OF OPERATION

The GR-320LAB system has been designed to meet the special requirements of the Metals Processing Industry. Various advanced features optimise system performance to maximise system sensitivity while being very easy-to-use.

These features include :

a) THREE (3) ANALYSIS PERIODS

To meet the very high precision requirements of the IAEA specifications the system requires a 300 second Sample Period for optimum performance. However much shorter time periods can be reliably used when higher levels of contamination are being measured. It is very important to give the user as fast an analysis result as possible to speed up the emergency response.

This is achieved in the GR-320LAB system by the use of ERROR ANALYSIS. As the sample data is accumulating, the system computes the inherent ERROR in the data on a second-by-second basis. The system Configuration file specifies an Error Limit that is scientifically acceptable and this is usually set at 5 %.

b) STEEL SAMPLE ANALYSIS :

The Configuration File also sets the TIME of the 3 sample periods as - 30 seconds, 60 seconds and 300 seconds. In high-level contamination even a 10 second sample may be sufficient, however statistically a 30 second lower limit is preferred. The sample analysis sequence is as follows :

@ 30 seconds :

- ERROR analysis is checked
- if ERROR is less than a certain level then the data is statistically OK
- Alarm message is displayed
- user is allowed to exit the measurement and spectral analysis evaluation can be carried out
- if Alarm Analysis is below the set limit (0.1 Bq/g) sampling continues

@ 60 seconds :

- ERROR analysis is checked
- if ERROR is less than a certain level then the data is statistically OK
- Alarm message is displayed
- user is allowed to exit the measurement and spectral analysis evaluation can be carried out
- if Alarm Analysis is below the set limit (0.1 Bq/g) sampling continues

@ 300 secs :

- Alarm Analysis is carried out
- if Alarm Analysis is below the set limit (0.1 Bq/g) sampling is halted

After analysis the display shows one of the following messages :

SAMPLE OK - if no alarm is detected.

SAMPLE ABOVE THRESHOLD - if an Alarm is detected.

This specially designed procedure gives the user maximum sensitivity and minimum sample time.

- c) DUST/ SLAG ANALYSIS - for the common detection limits of 0.12 Bq/g, even though the data analysis is more complex, the 30, 100, 300 second limit levels described above are also used. However if a more precise analysis level of 0.08 Bq/g is required for these samples then 100, 200 and 300 seconds are required. In some applications if a large volume of samples need analysis, the 300 seconds maximum limit may cause analysis congestion. In this case use 30, 60, 180 as the limits as these settings will have a minor effect on system precision. These trade offs are a little complex as they are inter-active. When in doubt, discuss with Exploranium.
-
-

2. SYSTEM SETUP

The following steps advise the user how to set-up the system and install operating software.

2.1. PREPARATION

- B Unpack the main chassis and inspect for shipping damage.
- B Locate the chassis next to a desk on which the system PC is mounted. You can uncover four holes in the bottom part and use two metal tubes with diameter 50 mm for safe manipulation.

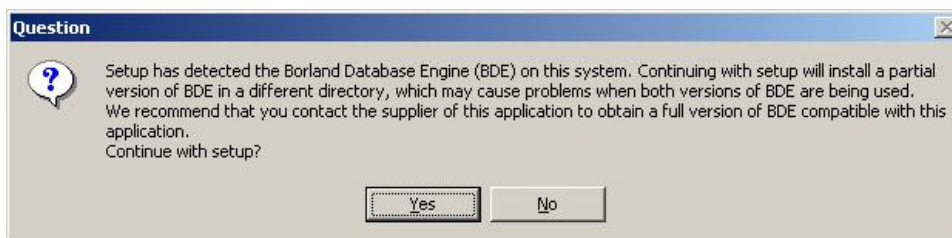
NOTE
THE UNIT IS VERY HEAVY
SO GREAT CARE SHOULD BE TAKEN
WHEN MOVING IT ABOUT
AS IT MAY UNBALANCE IF TRANSPORTED
INCORRECTLY

- B Connect the RS-232 cable from the rear of the main chassis to COM1 of the PC (other COM ports can be used if necessary – see Configuration)
 - B Ensure the printer is connected to the PC (if required)
 - B Connect the AC power adapter to the GR-320LAB chassis
 - B Power on the spectrometer, PC and printer
-

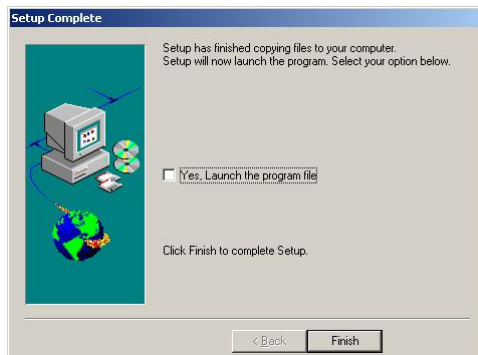
2.2. LOADING SOFTWARE

- B Load the supplied CD-ROM
- B Locate the "SETUP" Application file on this disk and double-click to start the installation process and follow screen prompts
- B Note the recommended subdirectory. Press <Enter> to accept this selection or change the sub-directory name as required, and system installation will start.

NOTE: it is possible that the following display will appear – if it does select "Yes".



Once all is complete the following screen appears:



Select "Finish" to complete the installation

Use Windows Explorer Setup to create an Icon on the Desktop to activate the GR320LAB system

2.3. RS-232 CONFIGURATION

Most users start to operate the system immediately but if they wish they could check that their RS-232 port conforms to the following requirements. RS-232 setup is available through Control Panel should be :

Baud rate =	9600
Data bits =	8
Stop bits =	1
Parity =	none
Flow control =	none

3. SYSTEM START - FIRST TIME USER

This section is for the first time user. Once these procedures have been followed then the user should refer to Section 5 = Daily Procedures or Section 6 = Monthly procedures.

3.1. POWER ON

Power the Computer and Printer ON then press the YELLOW button on the GR-320LAB chassis.

3.2. SYSTEM START

Click on the GR320LAB icon on the Desktop and the following screen is seen:



ig. 1

3.3. SETUP SYSTEM CONFIGURATION

The system uses various configuration files for correct parameter setup to suit the users application. It is important that the user setup all these parameters at the start of operation. Once set then usually they don't need adjustment.

From the startup screen – click "Options" and Figure 2 is displayed.

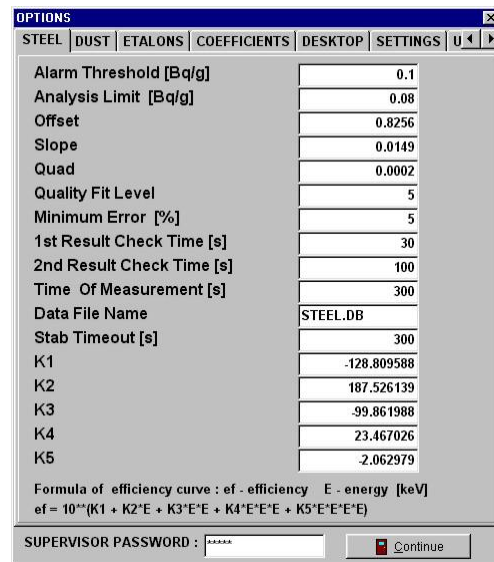


Fig. 2

3.3.1. PASSWORD

The system is setup with a default Password of "12345". Enter this where the displays shows "SUPERVISOR PASSWORD" and Parameter setup is enabled. Later in the parameter setup sequence the user can select his own Password for security reasons.

Once the Password is entered then the user can select the appropriate data entry page as detailed below.

NOTE : DO NOT ADJUST THESE FILES UNLESS SPECIFICALLY ADVISED IN THIS MANUAL AS INCORRECT PARAMETER ADJUSTMENT CAN SERIOUSLY AFFECT SYSTEM PERFORMANCE.

NOTE

USERS SHOULD CAREFULLY INSPECT ALL THE DATA PARAMETERS LISTED AND ENSURE THAT THE CORRECT DATA ARE ENTERED AS NOTED IN THE DETAILS BELOW

THE USER SHOULD PAY SPECIAL ATTENTION TO ALL PARAMETERS MARKED *** AS IT IS CRUCIAL THAT THESE PARAMETERS BE SET AS NOTED.

STEEL and DUST/ SLAG

In the parameter setup screens detailed below the user is requested to enter in various data. Users who are working ONLY with Steel samples can ignore the DUST parameter requirements. However if the user intends to work with STEEL and DUST/ SLAG samples then all relevant data must be entered.

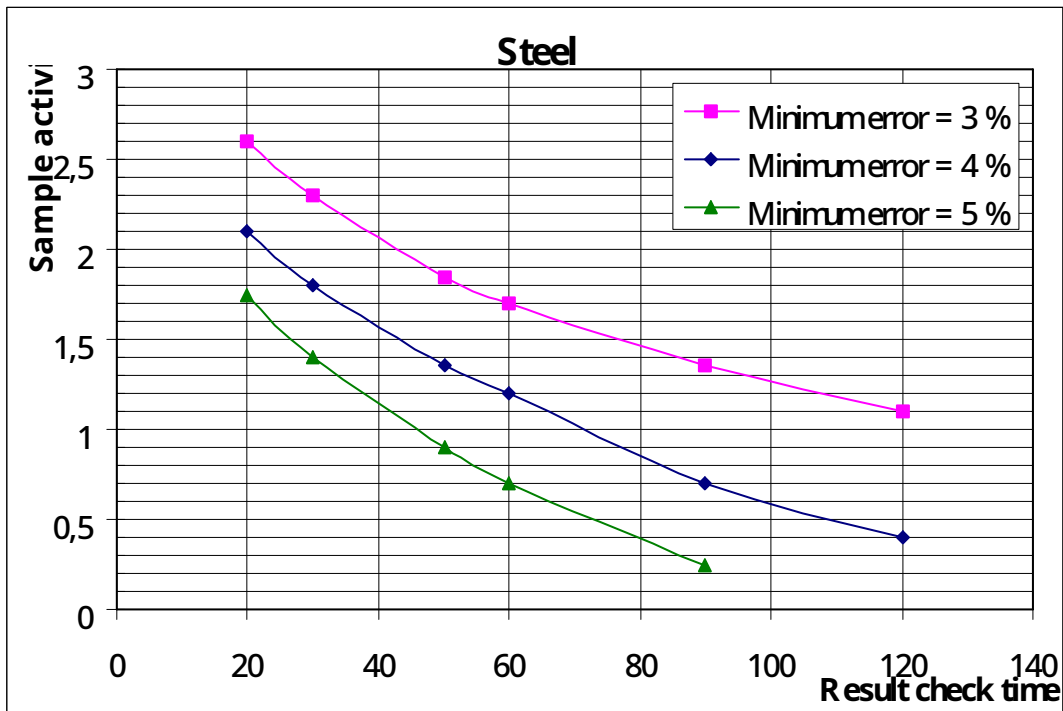
3.3.2. STEEL page

	Parameter	Range	Recommended setting	Comments
***	Alarm Threshold (Bq/g)	0.1-9.9	0.1	Note 1
***	Analysis Limit (Bq/g)		0.08	Do not change
***	Offset		0.8256	Do not change
***	Slope		0.0149	Do not change
***	Quad		0.0002	Do not change
***	Quality Fit Level		5	Do not change
	Minimum error (%)		5	Note 2
	1 st result check time [s]		30	Note 2
	2 nd result check time [s]		60	Note 2
	Time of measurement [s]		300	Note 2
	Data file name		STEEL.DB	Note 3
	Stab Timeout (s)		300	Do not change
***	K1		-128.809588	Do not change
***	K2		187.526139	Do not change
***	K3		-99.867988	Do not change
***	K4		23.467026	Do not change
***	K5		-2.062979	Do not change

The above settings have been selected to optimise the application – it is recommended that all parameters marked “***” be entered as shown. They should not be changed in any way without direct consultation with Exploranium or system performance may be degraded.

NOTES:

1. User can set this to suit their requirements but 0.1 Bq/g is the recommended level
2. Next graph can be used for setting of these parameters:



The combination of Minimum Error and Check Point time gives the Sample activity, which caused the warning screen.

3. The filename entered here is where the data from Steel samples are stored. The use of a simple name like STEEL.DB is recommended for simplicity but users can select any name they find appropriate. You can enter path and directory on another computer in the net if you want to save the results there. For example: \\Computer\Disk\Directory\file.db



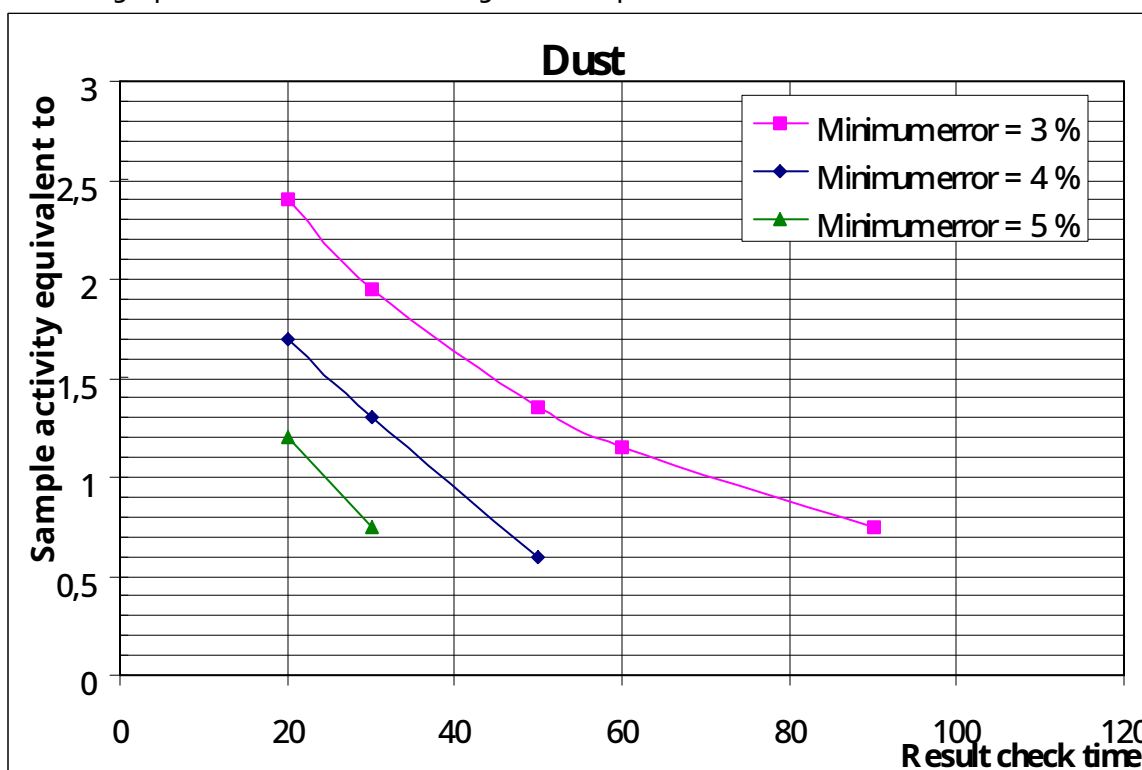
3.3.3. DUST page

	Parameter	Range	Recommended setting	Comments
***	Alarm Threshold (Bq/g)	0.1-9.9	0.12	Note 1
***	Analysis Limit (Bq/g)		0.05	Note 1
***	Offset		1	Do not change
***	Slope		0	Do not change
***	Quad		0	Do not change
***	Quality Fit Level		5	Do not change
	Minimum error (%)		1	Note 2
	1 st result check time [s]		30	Note 2
	2 nd result check time [s]		60	Note 2
	Time of measurement [s]		300	Note 2
	Data file name		DUST.DB	Note 3
	Stab Timeout (s)		300	Do not change
***	K1		-65.155243	Do not change
***	K2		106.500747	Do not change
***	K3		-62.793465	Do not change
***	K4		16.349436	Do not change
***	K5		-1.593933	Do not change

The above settings have been selected to optimise the application – it is recommended that all parameters marked “***” be entered as shown. They should not be changed in any way without direct consultation with Exploranium or system performance may be degraded.

NOTES:

1. User can set this to suit their requirements but 0.12 Bq/g is the recommended level for Alarm Threshold, 0.05 for Analysis Limit is the lowest suitable value – Analysis will be done when the activity of the sample is significantly above zero.
2. Next graph can be used for setting of these parameters:



The combination of Minimum Error and Check Point time gives the Sample activity, which caused the warning screen. The typical ratios between the Activity equivalent to Co-60 and Nuclide activity are given in next table:

Sample activity equivalent to Co-60 equal to Nuclide activity 1 Bq/g	Nuclide	Nuclide activity equal to Sample activity equivalent to Co-60 1 Bq/g
1,0 Bq/g	Co-60	1,0 Bq/g
0,6 Bq/g	Cs-137	1,8 Bq/g
1,6 Bq/g	Ra-226	0,6 Bq/g
0,13 Bq/g	K-40	7,8 Bq/g
1,8 Bq/g	Th-232	0,6 Bq/g
0,08 Bq/g	Am-241	12 Bq/g

3. The filename entered here is where the data from Steel samples are stored. The use of a simple name like DUST.DB is recommended for simplicity but users can select any name they find appropriate. You can enter path and directory on another computer in the net if you want to save the results there. For example: \\Computer\Disk\Directory\file.db
-

3.3.4. ETALONS page (Calibration samples)

This page is used to enter the data of the Calibration Samples - often referred to as Etalons. Exploranium normally supplies only STEEL samples unless the DUST/SLAG option is taken in which case a full set of these samples is also supplied. The user is supplied with a Calibration Sheet for each set of these samples. Some data from these Calibration sheets must be entered here. The user must click on the appropriate sample (STEEL or DUST) then enter some data as follows:

Id – the label of the sample – users should select the Steel sample number of approximately 8 Bq/g and enter those samples details

Etalon – the sample isotope – Am-241, Co-60 etc

Activity – the total activity of the sample in Bq = Weight x Bq/g

RefDate – the Reference Date of the sample – format is YYYY-MM-DD - note ENTER IN THE FULL FOUR DIGIT YEAR

All other parameters should stay unchanged

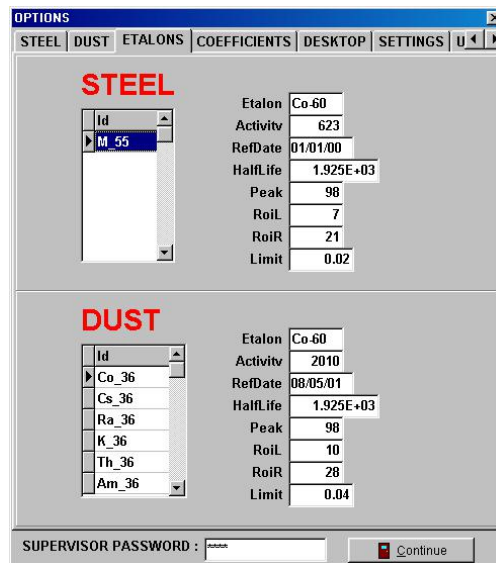


Fig. 3

3.3.5. COEFFICIENTS page

ADC Offset – set this parameter to the value shown on the system Calibration Sheet – this is a VERY IMPORTANT parameter so ensure it is correct.

Disc level – set to 4

A0/ 1/ 2 – leave at current settings

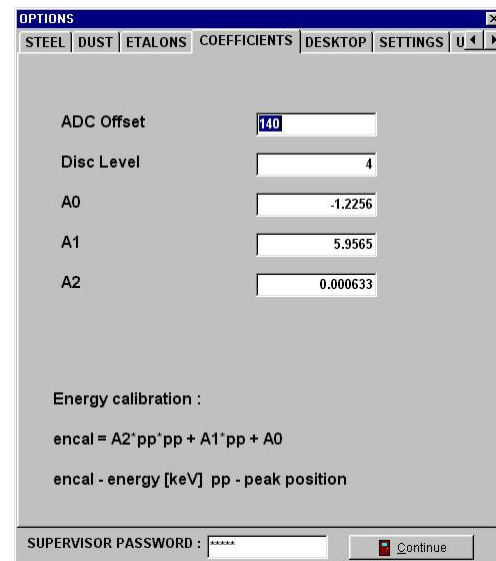


Fig. 4

3.3.6. DESKTOP page

– set as required to suit the user

3.3.7. SETTINGS page

– this page is used to select user settings

PRINTER – as required – Windows selects the appropriate printer driver – this selection is only to select color or not

GAIN – Exploranium supplies a Cesium test source with the system so CESIUM should be selected

LANGUAGE – as required by the user

GR-320 PORT – usually COM1 but user can select

BALANCES PORT – Currently two weigh scale models are supported – the OHAUS ADVENTURER and the Mettler Toledo PB602-SD usually COM2 but user can select

COMPAC PORT – for special applications see Appendix D

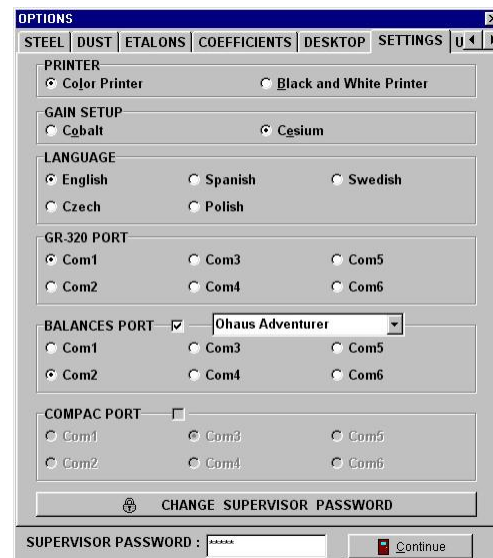


Fig. 5

3.3.8. USER LABELS page

In many applications the user wants to enter in a variety of data inputs for each sample measurement to be able to tie the data results into other database data. To permit this capability this setup page permits the user to select up to 10 data input labels – most users only use the default setting of 1 label. The user should decide what they want and with this experience change this setting as required.

Only the ID_1 is used for communication with this software so ID_1 is recommended to be definite for the sample.

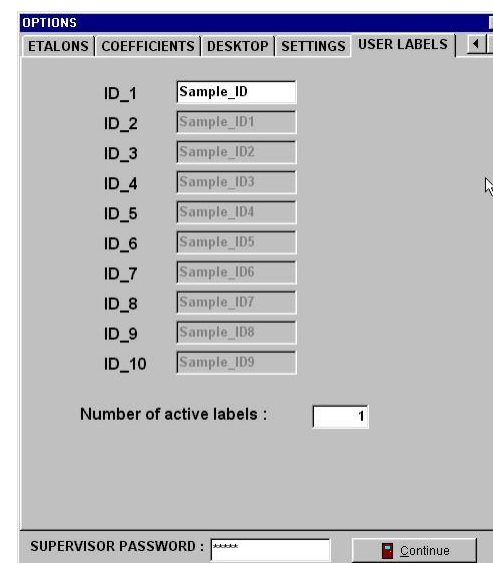


Fig. 6

As an example – if 2 data labels are selected then the user can enter 2 variable names for each sample. For example - Sample # and Operators #, Batch # etc. etc.

3.4. SYSTEM STABILITY

The GR-320LAB is designed for continuous operation, however it must be switched on for a period of time so that all internal electronic components can be “warmed-up”. It is assumed that the system is installed in a typical laboratory environment where the ambient temperature ranges no more than +/- 2 °C over any time period. Under these conditions, once the system has been “warmed-up” (and assuming power is NOT interrupted) then system calibration need only be repeated on a MONTHLY basis.

However if great variation of temperature occurs then more frequent calibration may be required. System operational requirements (detailed in Section 4 - Recommended Procedures) will

show up any drift problems caused by temperature and Exploranium can work with the user to change recommended procedures to suit the local conditions. However all current installations have shown no serious temperature problems, the following procedures assume "normal" temperature stability in the lab.

3.5. TOTAL CALIBRATION

The system must be fully calibrated before use (typically 1/month - see Section 4). If the user wants to measure STEEL and DUST/SLAG samples then TWO CALIBRATIONS ARE REQUIRED before sample measurements are started.

The calibration process is carried out as follows :

- a) GAIN SETUP - this procedure adjusts the system internal gain to match system calibration using the supplied CESIUM test sample (NOT THE CESIUM DUST CALIBRATION SAMPLE).
- b) CAL. SAMPLE(S) - in this procedure a calibrated sample is used to measure the response
- c) BACKGROUND - in this procedure the BACKGROUND sample is measured and used to remove background effects.

NOTE
FOR BEST ACCURACY AND REPEATABILITY OF ANALYSIS,
TRY TO POSITION THE SAMPLES APPROXIMATELY
CENTRAL IN THE SAMPLE CHAMBER.

3.5.1. STEEL CALIBRATION

To start the Calibration process click Calibration on the GR-320 LabSPEC desktop. Popup window will appear allowing the user to select either STEEL or DUST samples for calibration. Select the type of calibration to be done and click OK. The section below describes the steps to be taken to do a calibration to prepare the system for STEEL sample measurement. DUST calibration is described later.



Fig. 7

If the spectrometer system is not detected then a warning message will be seen (Fig. 8).

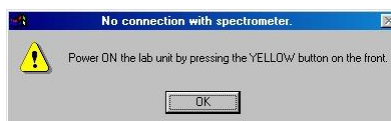


Fig. 8

Switch on the spectrometer and restart the procedure.

If communication with spectrometer is OK then the user is advised of system progress by a series of messages in the ANALYZER MESSAGE CENTER (top left of the display – see Fig. 10). Several

messages will be displayed while the software does additional checks and setups. When the system is ready to proceed the GAIN SETUP routine is started and the following display is seen:



Fig. 9

The user should now insert the Cs-137 Test Source (NOT the DUST Cs sample) into the measurement chamber of the GR320 Lab for gain adjustment. Place the supplied 0.25uCi Cs137 source into the center of the measurement chamber and close the lid, then click OK.

Fig. 10 appears and the system starts to measure the Cs-137 source and adjust the Gain of the system automatically for correct system operation.

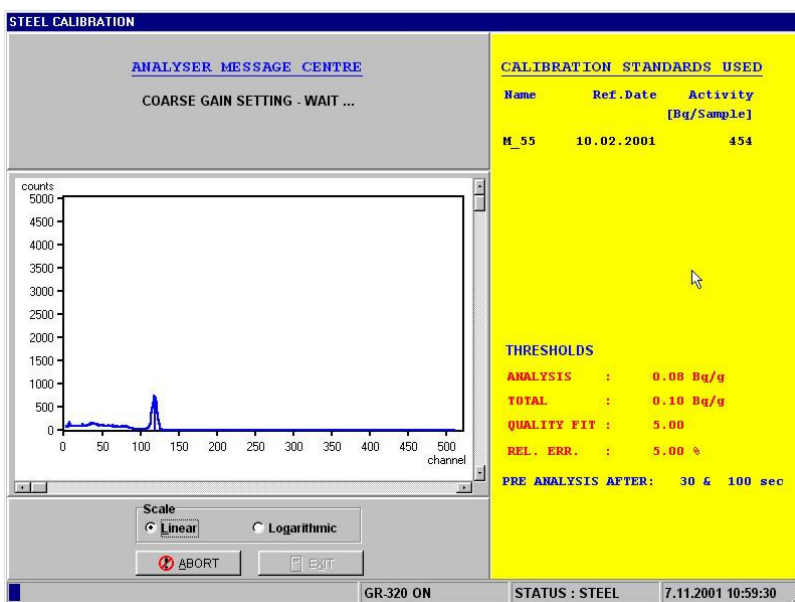


Fig. 10

Several MESSAGES will be displayed as the system goes through the various steps of stabilisation and gain adjustment – including "COARSE GAIN SETTING, FINE GAIN #1.., #2.., #3...

When the GAIN setup and auto-adjustment is complete, the following display is seen:

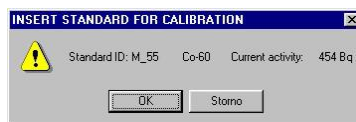


Fig.11

The user must now REMOVE the Cesium Test Source and place the specified Steel Calibration source in the chamber, close the lid – then click OK.

When the process is complete a new message appears:

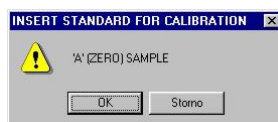


Fig. 12

REMOVE the previous sample and insert the new sample as specified then press OK to proceed.
NOTE: the ZERO sample refers to the steel calibration sample (labelled 'A') with zero radioactivity. ZERO standard is measured for a period twice longer than is set for the standards due to the enhancement of the measurement accuracy.

When the calibration process is finished this will be indicated in the Analyser Message Center by the "END OF CALIBRATION MESSAGE".

A new popup window will appear requesting permission to overwrite the old calibration file.

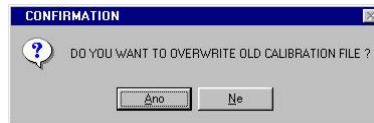


Fig. 13

Click Yes to store and use the new calibration coefficients and to proceed with measurement. Next message will ask the user to print a hard copy of the calibration results.

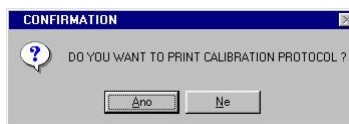


Fig. 14

Click YES, normally this is recommended procedure to keep a copy of all Calibration files especially in the first months of operation to become familiar with the system and track data for trouble analysis.

A typical printed calibration protocol is seen below.

EXPLORANIUM
RADIATION DETECTION SYSTEMS**Gr-320 labSPEC**

Version 2.7

STEEL CALIBRATION PROTOCOL 5.11.2001 12:52:59GAIN SETUP

Peak position : 109.9
FWHM : 6.6 %
GAIN : 360

ACTIVITIES OF STANDARDS

Name	Nuclide	ref.date	act.ref. [Bq/sam]	curr.act. [Bq/sam]	peak pos. [ch]
M_55	Co-60	10.02.2001	498	453	191.2

Time of measurement : 300 s

Background total : 493 cpm
Standard total : 10195 cpm
Calibration constant : 21.530

Name of the calibration file : STEEL.BIN
File date: 05.10.2001 File time: 12:35:04

Calibration file backed up as SOO11005.BIN
File date: 05.10.2001 File time: 12:35:04

ROIs: #1 177 - 233

CALIBRATION SUCCESSFULLY COMPLETED

Fig. 15

Click EXIT to exit the Calibration section.

3.5.2. DUST CALIBRATION

To prepare the GR320 Lab system for measurement of DUST samples, click on Calibration from the GR320 LabSPEC desktop and select DUST.



Fig. 16

Then click OK.

The Analyser Message Center will appear on the display and the system will perform several tests.

A Popup box will appear and the user is prompted to insert the Cs-137 sample into the measurement chamber of the GR320 Lab for gain adjustment (a repeat of the procedure for STEEL above). Place the supplied Cs137 Test Source (NOT the DUST Cs sample) into the center of the measurement chamber and close the lid, then OK.



Fig. 17

The following display will appear and the system will automatically setup system, Gain (the same as for STEEL above – the difference being that the screen shows the DUST calibration samples).

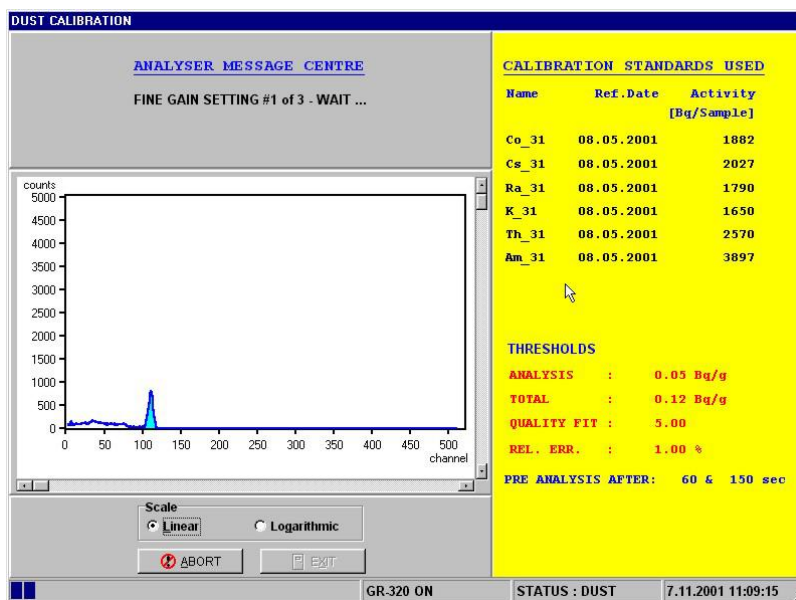


Fig. 18

When the gain adjustment has been completed the system will request several dust standards for calibration – this refers to the set of 7 dust standards supplied with the system and setup in the system in Section 3.3.4. The set is comprised of Cobalt, Cesium, Radium (Uranium), Potassium, Thorium, Americium and Background samples. Each will be measured in succession to properly calibrate the GR320 Lab system for dust sample analysis.

Follow the on screen prompts and be sure to insert the sample specified in the "INSERT STANDARD FOR CALIBRATION" popup window and remove the sample used previously – ONLY ONE SAMPLE AT A TIME IN THE CHAMBER.

During the calibration process a display similar to the figure will be visible.

Each sample will be measured for a preset period of time and after each measurement the user will be prompted to insert the next sample.

NOTE: that as the ZERO calibration sample an empty container is used because of every slag or dust has the activity higher than zero. You can verify it by measuring the "B" standard. It is slag used for preparing of the calibration standards. It's activities of Thorium and Radium is about the detection limit.

ZERO standard is measured for a period twice longer than is set for the standards due to the enhancement of the measurement accuracy.

When all the calibrated samples have been measured the Analyser Message Center will indicate "END OF CALIBRATION-PRESS EXIT".

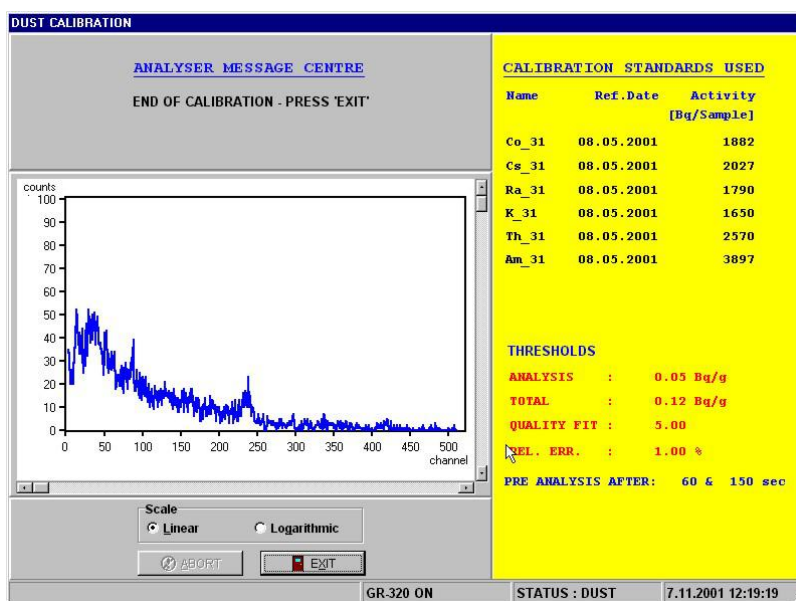


Fig. 19

As with the Steel calibration the user is asked for permission to overwrite the old calibration file - the user should answer YES.

The user is also given the option to print the calibration results – the user should select YES – and keep the printed record for future system analysis as required.

An example of a printed calibration protocol is seen below.

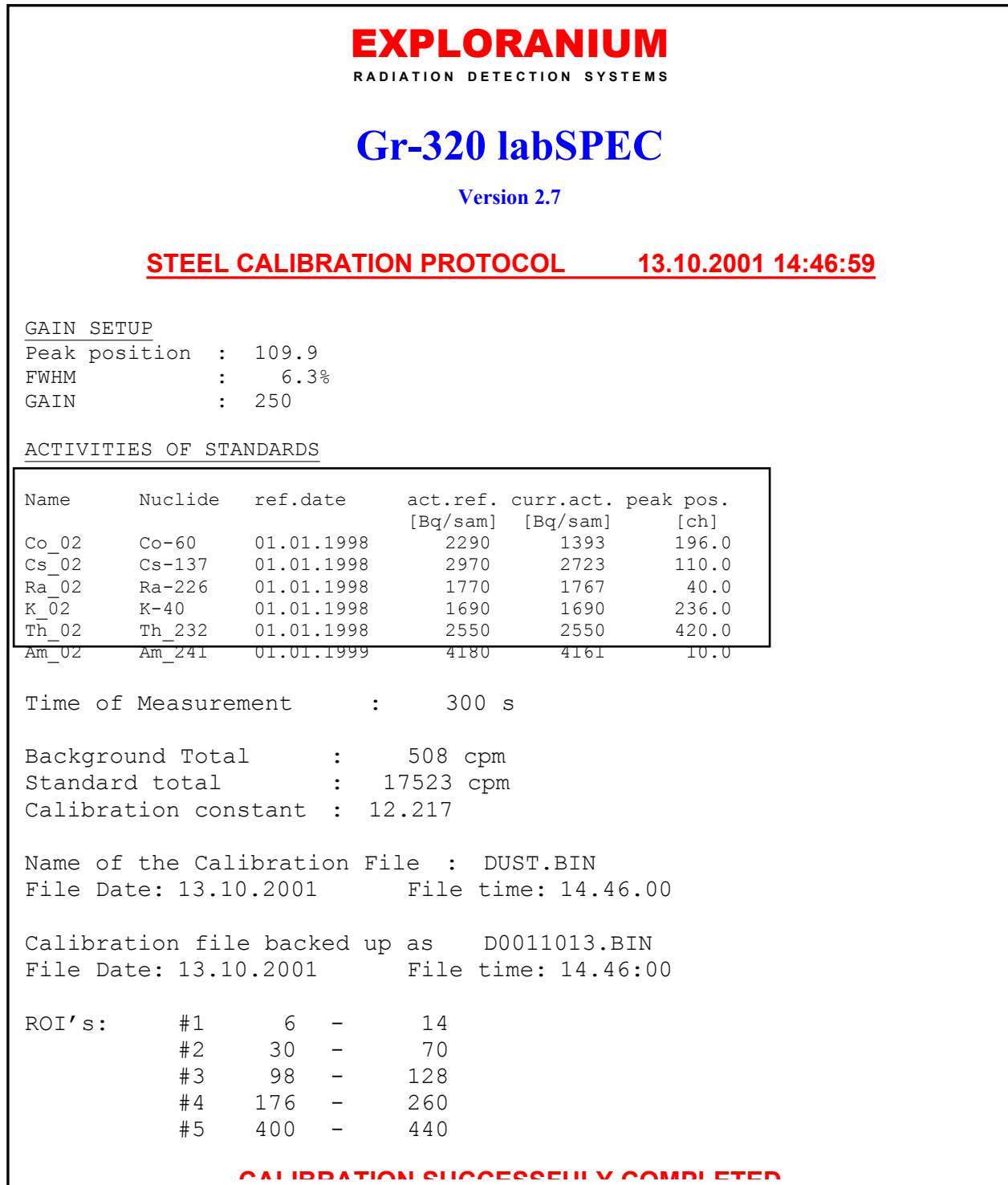


Fig. 20

3.6. GAIN SETUP

This process is used to check system Gain performance. It is only a Maintenance requirement as the GAIN setup is incorporated in the CALIBRATION carried out as described above. The procedure implemented is exactly the same as is described at the start of the STEEL calibration in section 3.5.1 above.

4. MEASUREMENT

The following steps advise the user how to provide current measurement.

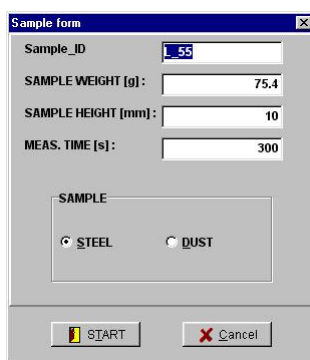
4.1. SAMPLE MEASUREMENT

Once the system is calibrated, normal sample measurements may be carried out using the procedure described below. As mentioned previously, try to always centre the sample on the bottom of the sample chamber for the best results.

Data results are stored in the data file specified in the Options file – normally STEEL.DB for Steel samples and DUST.DB for Dust samples

To measure a sample, proceed as follows from the Main Menu

Select MEASUREMENT and the SAMPLE FORM menu appears – Fig. 21:



The screenshot shows a 'Sample form' dialog box with the following fields and values:

Field	Value
Sample_ID	55
SAMPLE WEIGHT [g]	75.4
SAMPLE HEIGHT [mm]	10
MEAS. TIME [s]	300

Below the fields is a section labeled 'SAMPLE' with two radio buttons: 'STEEL' (selected) and 'DUST'. At the bottom are 'START' and 'Cancel' buttons.

Fig. 21

NOTE: DATA ENTRY

In this data form any data entered is retained for the next sample unless changed. Thus if a data field entry for weight is 120 g and the users procedures always use a 120 g sample weight, then when entered the first time it is "latched" so in future for a new data sample pressing Tab instead of entering data will retain the previously entered data for that field.

Select DUST or STEEL (the forms data entry requirements change) – once again if DUST is selected here then when a new sample is started the DUST designation will be retained.

Enter in the data required:

- Sample ID - the sample identification data is entered here. This display shows only a single sample identification entry requirement but as explained in section 3.3.3. the user can select up to 10 data labels. The purpose of the label is to ensure that the data results can be tied back to a specific sample and therefore a specific batch of steel. User should derive a coding system appropriate for their local methods of operation
- SAMPLE WEIGHT – since data analysis computes the data in Bq/g units, sample weight is an important requirement. If users have a digital scale formatted and connected, then a double-click on this data entry line will load the correct weight from the scale
- SAMPLE HEIGHT – not required for DUST samples but an important data entry for STEEL samples as the system, automatically corrects for self-absorption caused by sample height differences.

- MEAS. TIME – setup in the Options file previously. This entry can be overwritten if required or TAB to accept.

Once data entry is complete – click START

The following display appears:

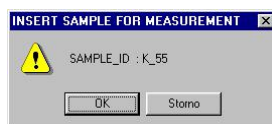


Fig. 22

This is a reminder to the user to insert the correct sample in the sample chamber and close the lid.

NOTE:

The sample must have the laboratory temperature! It is important especially for steel samples because of they are usually taken from the AAS spectrometry where the sample is heated to about 60°C. So warm sample can change the detector temperature and move the gain far from the correct region. Cooling in water or on the copper plate is recommended.

Then press OK.

Sample analysis starts and the following display is seen. The data analysis is fully automatic as described below:

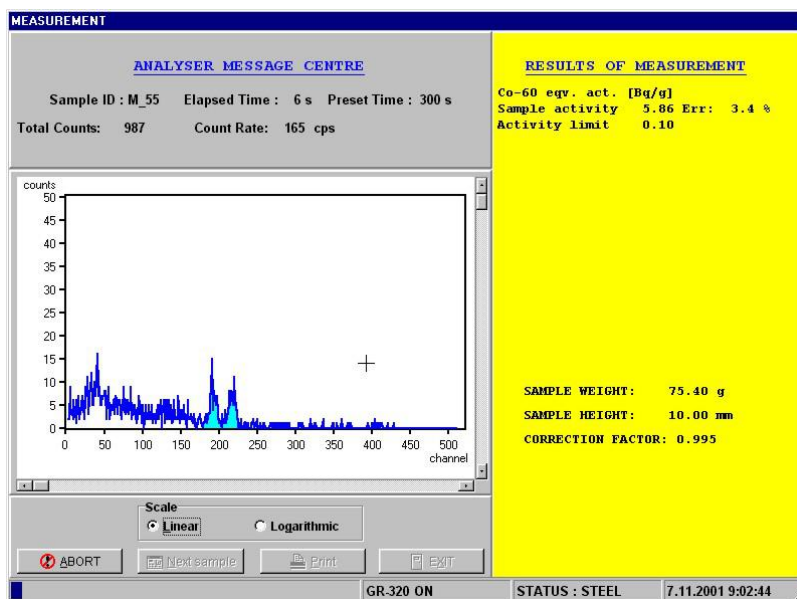


Fig. 23

This is an example of a STEEL sample analysis display. Various data are shown:

Sample ID - this is the sample ID data entered by the user as the sample label

Elapsed Time – the progress of the sample analysis - in this case the display shows the sample data accumulation after 8 seconds

Preset Time – 60 seconds is the preset time. Normally 300 seconds is the correct sample time

Total Counts – this is the sum of the detected counts in the entire spectrum at the 8-second data point

Count Rate – this is the data rate and is Total Counts/Elapsed Time

Display box - this is the accumulating spectrum with the vertical axis is counts and the horizontal axis is channel number. Since the 512 channels are spread over the full 0-3 MeV spectrum – 1 channel = 6 keV.

Scale – Linear or Logarithmic – the graphic display can be selected to be Logarithmic if required as this makes it easier to see the very low count peaks in the upper spectrum more clearly. Selection of this display has no effect on data analysis.

Co-60 eqv. act. – this is a data label for the next display line

Sample activity - the whole spectrum is assumed to be caused by Cobalt contamination so the algorithm computes the EQUIVALENT COBALT ACTIVITY in Bq/g from the accumulating data each second. Since the alarm analysis threshold is based on these data, the alarm threshold is tested for an alarm condition. This display shows that the current Sample Activity after 8 seconds of data analysis is 0.03 Bq/g for Cobalt-60 – also see comments below

Err - the error shown is the computed statistical error on the data. In the Options setup an error level of 5 % was entered. This means that if the data error, that is computed each second on the accumulating data spectrum, falls below the 5 % limit then the data is deemed to be statistically significant – see comments below

Activity limit – the limit in Bq/g that is set in the Options menu to suit the user requirements (usually 0.1 Bq/g for STEEL and 1.0 Bq/g for DUST). If the Sample activity is above the Activity limit then an alarm condition is flagged – see comments below

SAMPLE WEIGHT, SAMPLE HEIGHT – are labels from the data entry

CORRECTION FACTOR – is an automatic computation based on sample height for Steel samples

If no alarm is detected the following display appears:

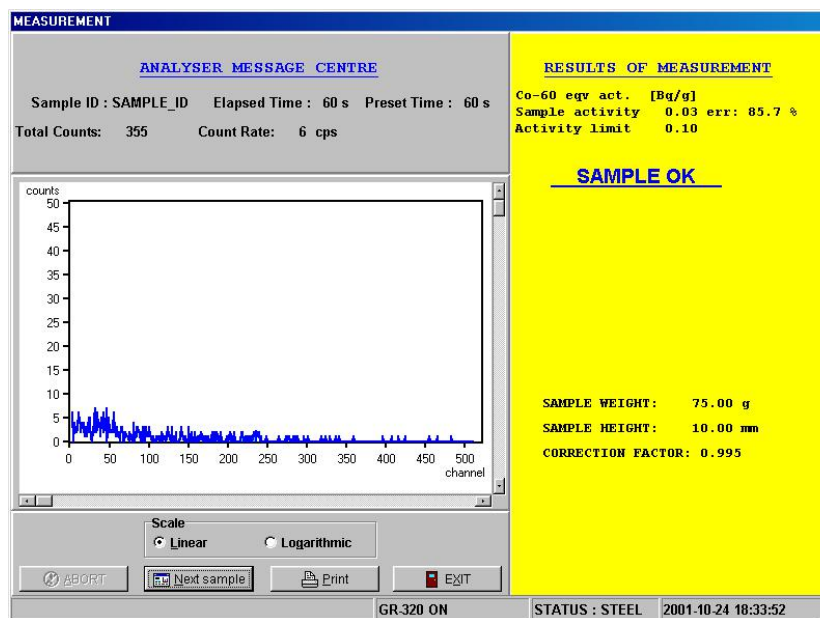


Fig. 24

SAMPLE OK – means that no alarm was detected on this sample

NEXT SAMPLE – if this is selected then the above sequence is repeated for a new sample

COMMENTS:

Data analysis is carried out as follows:

- in the Options menu 3 Alarm Testing times are defined – usually 30, 60 and 300 seconds.
- at the 30-second point the data results are inspected
- if the Sample activity is ABOVE the Activity limit AND if the Err % is BELOW the preset limit of 5 % , then an alarm is declared - see Fig. 25 – and user is allowed to exit the measurement and spectral analysis evaluation can be carried out
- however if either of these conditions is not met or user does not exit the measurement then sample accumulation continues and these limits are tested at the 60-second set point.
- If the alarm conditions are still not met at the 60-second point or user does not exit the measurement, the sample accumulation continues and is finally terminated at the final 300 second set point regardless of the limits – see Fig. 26

ALARM DISPLAYS:

a) ALARM AT A SET POINT

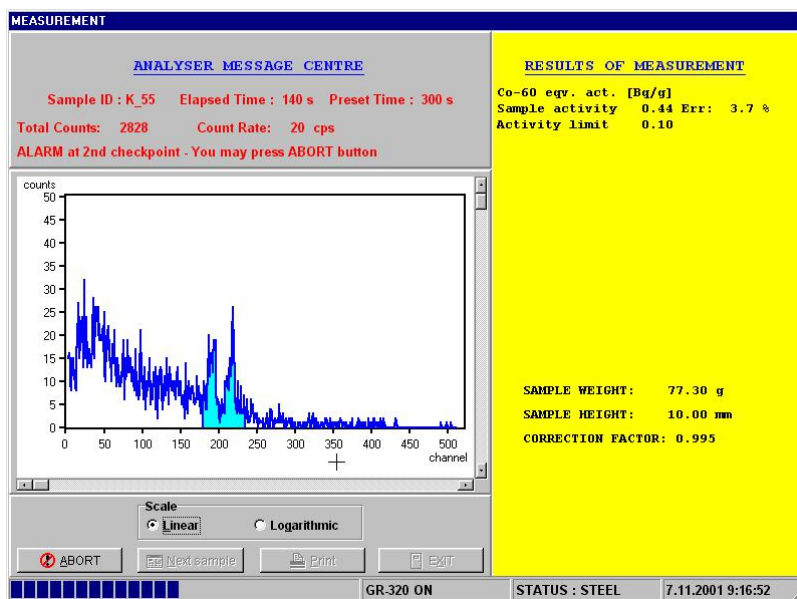


Fig. 25

b) ALARM AT THE END OF SAMPLE

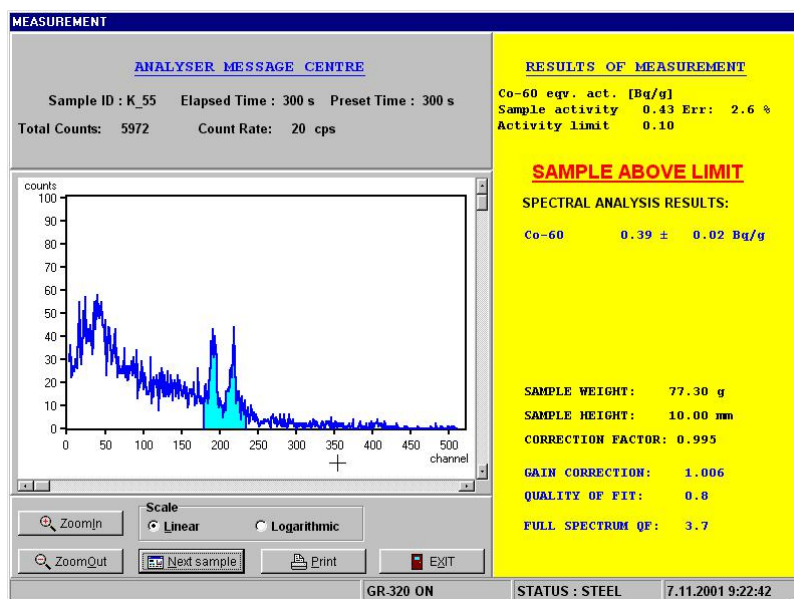


Fig. 26

4.2. DISCUSSION OF THE RESULTS

What you should check to be sure that the results are correct? See the flow chart at the end of this chapter.

4.2.1. SAMPLE OK

- The real time of measurement is the same as the set time, usually 300 seconds. If no repeat the measurement.
- The spectrum looks like on the figure 24 – it means no peaks are visible - you are sure sample is O.K. If some peak appears a nuclide is present, probably other nuclide than nuclides in the calibration set. Ask radiation safety officer (RSO) for help.

4.2.2. Steel SAMPLE ABOVE LIMIT

- The real time of measurement is the same as the set time, usually 300 seconds. If no repeat the measurement.
- There is no message UNIDENTIFY ISOTOPE PRESENT in the right down corner of the screen and the QUALITY OF FIT (QF) is near to 1.0, usually lower than 5. If the message is present check if conditions for measurement are O.K. (sample has sizes and thickness closed to the calibration standards, sample is in the center of the sample chamber, time of measurement is 300 seconds) and repeat the measurement. In case of good measurement conditions and message again ask RSO for help.
- The Co-60 equivalent activity is almost the same as the activity of Co-60 from spectral analysis. "Almost" means the difference is usually lower than 0.05 Bq/g.

Exactly: the region of reliability

$$(Co-60 \text{ equivalent activity}) \pm 3 * (Co-60 \text{ equivalent activity}) * (Err.) / 100$$

is particularly covered by region of reliability

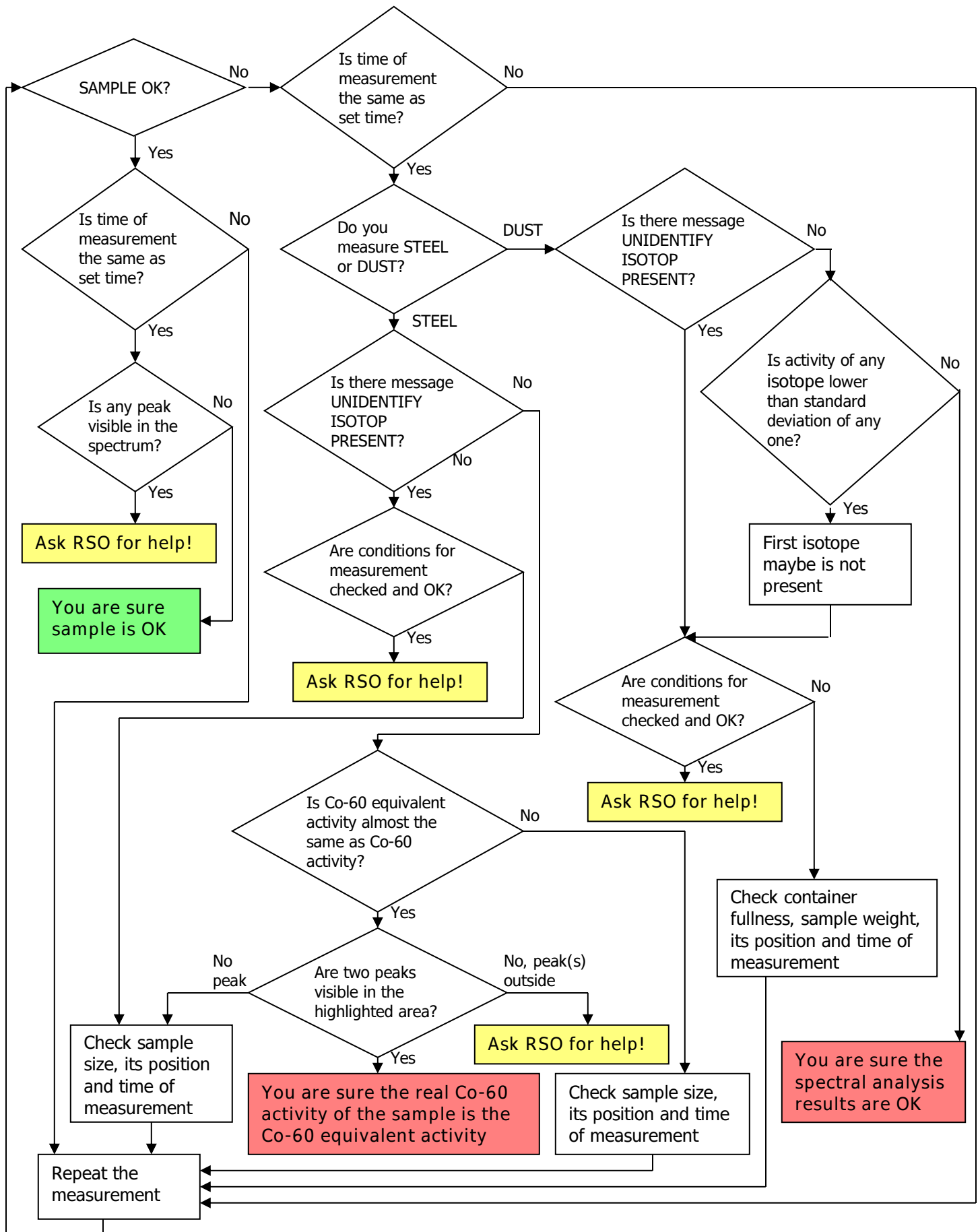
$$(Co-60 \text{ activity}) \pm 3 * (\text{standard deviation of Co-60 activity}).$$

If no check if conditions for measurement are O.K. (sample has sizes and thickness closed to the calibration standards, sample is in the center of the sample chamber, time of measurement is 300 seconds) and repeat the measurement.

- The spectrum looks like on the figure 26 – it means two peaks are visible in the highlighted region. You are sure the Co-60 activity of the sample is Co-60 equivalent activity. If no peak is in the highlighted area, no Co-60 is present or the time of measurement is too short. If some peak appears outside of the highlighted area, some other nuclide is present. Ask RSO for help.

4.2.3. Dust and Slag SAMPLE ABOVE LIMIT

- The real time of measurement is the same as set time, usually 300 seconds. If no repeat the measurement.
 - There is no message UNIDENTIFY ISOTOPE PRESENT in the right down corner of the screen and the QUALITY OF FIT (QF) is near to 1.0, usually lower than 5. If the message is present check if conditions for measurement are O.K. (the container is full of the sample, its weight is between 100 g and 140 g, sample is in the center of the sample chamber, time of measurement is 300 seconds) and repeat the measurement.
 - The activity of any nuclide is higher than its standard deviation. If no then this nuclide maybe is not present. Check if the conditions for measurement are O.K. (the container is full of the sample, its weight is between 100 g and 140 g, sample is in the center of sample chamber, and time of measurement is 300 seconds) and repeat the measurement. In case of good measurement conditions and message again ask RSO for help.
 - The activity of one nuclide is higher than the standard deviation of another one. In case of Am-241 the activity divided by 3 should be compared with the standard deviation of another nuclide. You are sure the spectral analysis results are O.K. If no than the first nuclide maybe is not present. Check if conditions for measurement are O.K. (the container is full of the sample, its weight is between 100 g and 140 g, sample is in the center of the sample chamber, and time of measurement is 300 seconds) and repeat the measurement. In case of good measurement conditions and message again ask RSO for help.
-



4.3. RESULT FILE

From the main GR-320 screen select Result File. A dialog box appears, prompting the user to select the file specified in Options setup (e.g. STEEL.DB or DUST.DB). Once selected the following figure is shown:

NRec	Sample_ID	Date	Time	MeasTime	Weight	Height	Act [Bq/g]	Err [%]	Q factor	Alarm
7	D07	2001-10-28	10:47:59	60	74.7	10	1.73	2.2	.7	YES
6	C07	2001-10-28	10:42:10	60	73.9	10	13.24	.7	2.3	YES
5	C07	2001-10-28	10:31:12	60	73.9	10	13.29	.7	1.6	YES
4	SAMPLE_ID	2001-10-26	08:23:51	60	73.9	10	20.67	.7	1.7	YES
3	SAMPLE_ID	2001-10-24	17:58:15	60	75	10	.03	85.7		NO
2	SAMPLE_ID	2001-10-24	17:57:02	39	75	10	.06	59.5		NO
1	SAMPLE_ID	2001-10-24	17:49:34	60	75	10	.52	6.9	.8	YES

Figure 27

Each line represents a data sample; highlighted entries are samples above the alarm limit.

Double-clicking any sample result shows the original display from the analysis:

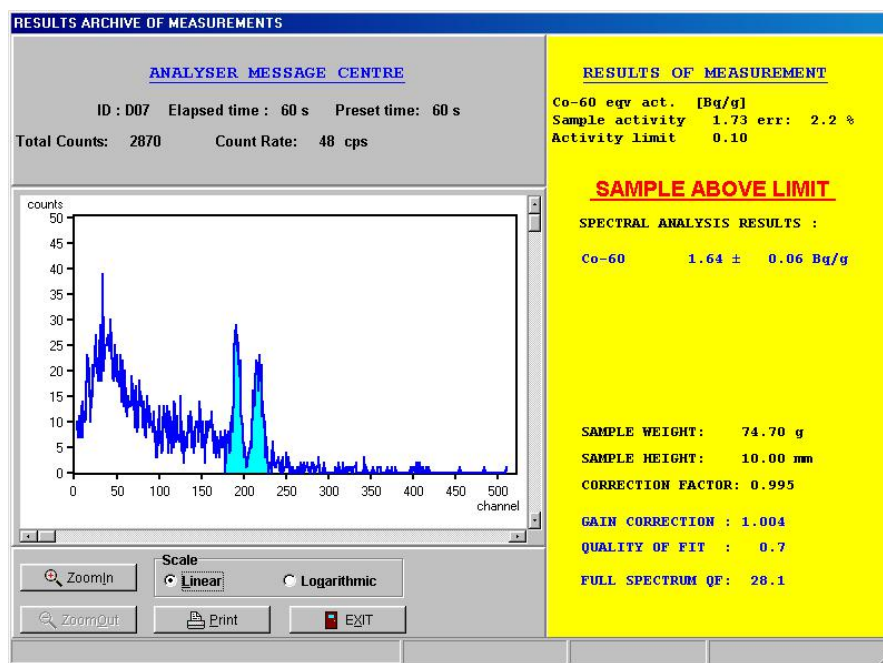


Figure 28

Click Continue to exit the Results screen.

4.4. Accuracy measurement verification

It is recommended to verify the calibration via measurement of the calibration standards. You must obtain the activity within toleration given in the MS Excel table "Actual Activity Calculation.xls" supplied with LabSPEC software after updating the date in appropriate cell. Activity of other nuclides must be lower than its detection limit.

The simplest way how to check the accuracy of measurement after some time, is measurement of calibration standard for the steel – usually "M xx" standard - for the period of 300 seconds. The result screen gives you all necessary information about the system.

1. The activity equivalent to Co-60 and Activity of Co-60 from the spectral analysis must be within toleration given in the MS Excel table "Actual Activity Calculation.xls" supplied with LabSPEC software. If no, you must make the Calibration.
 2. The QUALITY OF FIT must be lower than 4.0. If no, you must make the Calibration.
 3. The GAIN CORRECTION must be higher than 0,98 and lower than 1,02. If no, you must make the Gain setup.
-

5. RECOMMENDED DAILY PROCEDURES

Users will have to adjust the actual operating sequence to suit their local requirements but the following are recommended procedures. Normally the following procedures should be carried out at the START of every SHIFT to ensure that system performance is optimised.

- a) CLEAN - Clean the sample chamber to remove any dirt from the samples using alcohol or similar cleaner.
 - b) GAIN SETUP – Run the Gain setup procedure from the start up screen to ensure that spectrometer is working under the optimal conditions.
-

6. MONTHLY PROCEDURES

The user should carry out a more complete procedure each Month to check system overall performance. The recommended procedure is to carry out the TOTAL CALIBRATION as described in 3.4 above. Note if STEEL and DUST samples are both measured - then both calibrations are required.

It is recommended that the user keeps a record of the Total calibration results and compares to the previous periods to check overall system performance. The following data recording method is recommended – data extracted from Calibration printouts.

Date	Gain	Peak Pos	BG Total	Std Total	Cal Const
1/ 1/ 01	254	190.0	395	9847	21.346
2/ 1/ 01	265	189.8	384	9912	21.312
Etc					

Recommended limits are :

- a) GAIN - 100-400 - will change somewhat over time but from month/month would not expect a change of more than (say) 30 steps. As long as the actual number is within the range 100-400 the system is OK.
- b) Peak Pos - 186-194 - should stay in the range 186-194 for correct operation
- c) BG Total - +/- 10 % - this could change if the unit is moved or if any samples are stored near the unit but a change of more than +/- 10 % from the norm indicates that something is happening in the environment and this should be investigated. A change in this data could also mean that the internal electronics are degrading so discuss with Exploranium if no ready explanation occurs.
- d) Standard Total - +/- 10 % - since this data is corrected for background - this number should stay within a +/- 10 % range. If it exceeds this limit - contact Exploranium. There will of course be a slight degradation over time as the isotope decays (see Appendix A to compute the current value of the standard).
- e) Cal Constant - 17.0 - 25.0 - this number should typically stay within +/- 10 % limits of the number achieved on the first system calibration. Any significant change trend in this data should be discussed with Exploranium.

IF THESE DATA ARE APPROXIMATELY SIMILAR, THEN SYSTEM PERFORMANCE IS NORMAL. LOOK FOR ANY SUDDEN DATA CHANGES AS THIS MAY SIGNAL THE USER THAT THE SYSTEM IS BEGINNING TO MALFUNCTION.

7. APPENDIX A CALIBRATION SOURCES

7.1. STEEL

The supplied Calibration Standards are special steel samples specially contaminated with the isotope to provide an accurate calibration reference for the systems. The samples are supplied in a wooden container and comprise a set of 5 samples as follows. The sample sets are numbered with a Serial Number as shown in the following data (e.g. A55 = Sample A - Serial # 55 etc).

Source Type : Steel

Nuclide : Cobalt-60 (Co-60)

Half-Life : 1925 days

Reference date : 1/Jan/2000

of samples :5

Sample Activity:

Sample #	Activity Bq/g	Tolerance +/- Bq/g	Weight (g)	Height (mm)
A##	0	-	75	10
J##	0.20	+/- 0.05	75	10
K##	0.52	+/- 0.07	75	10
L##	3.45	+/- 0.2	75	10
M##	7.7	+/- 0.4	75	10

NOTE re Half-Life: The samples will gradually reduce in value with time due to radioactive decay. This is simply computed as follows.

Using Sample K## as an example:

Original value - 0.52 Bq/g

Sample date - 1/1/00

Current date - (say) 14 Jun 2001

Elapsed time in days - 530 days (1/1/00 to 6/14/01)

Half-life - 1925 days

Current value = Original * $(2^{-(\text{Elapsed}/\text{Half-life})}) = 0.52 * (2^{-(530/1925)}) = 0.43 \text{ Bq/g}$

7.2. CESIUM TEST SOURCE

A 9.25 kBq (0.25 μCi) Cesium-137 source is supplied for system calibration.

7.3. DUST SAMPLES

Source Type : DUST

Nuclide : Various

Half-Life : Various

Reference date : 1/Jan/1998

of samples :7

Sample Activity:

Sample #	Isotope	Activity kBq/ sample	Tolerance Bq/ sample +/-	Half life days
ETA Co	Co-60	1.93	0.08	1925
ETA Cs	Cs-137	2.67	0.12	11019
ETA Ra	Ra-226	1.83	0.08	584300
ETA K	K-40	1.65	0.10	4.66e11
ETA Th	Th-232	2.61	0.12	5.12e12
ETA Am	Am-241	4.19	0.4	10.2e15
ETA B	--	Ra 0.04 Th 0.03	0	

WEIGHT - 120 grams (actually 120 grams of material + 17g of container)

HEIGHT - 35

8. APPENDIX B OTHER DISPLAYS

Various Displays can be selected in the system. The following describes these options.

8.1. INFORMATION

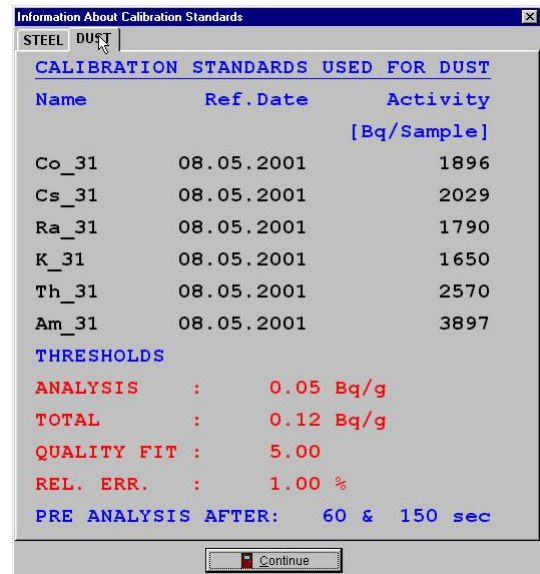
The 2 selections here are :

1. INFORMATION ABOUT CALIBRATION STANDARDS

This is the summary of system Option setup for the STEEL analysis

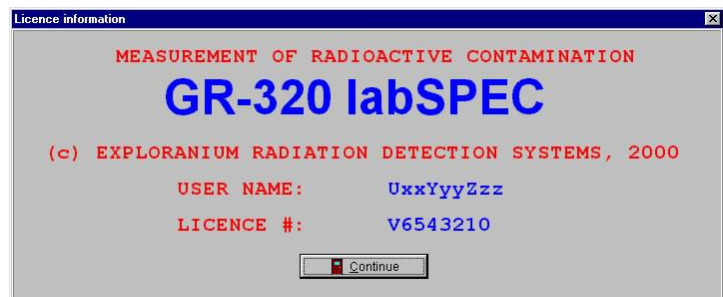


This is the summary of system Options setup for the DUST analysis



2. LICENSE INFORMATION

This shows the user Licensed information set in the system



8.2. ABOUT

This is the basic information about this software and some pictures of the spectrometer GR-320LAB.



9. APPENDIX C ERROR MESSAGES and TYPICAL PROBLEMS

1. GENERAL

Steel samples are often dirty and eventually dirt particles will build up in the sample chamber and perhaps distort the analysis. To prevent this - clean the sample chamber out on a regular basis (at least daily and preferably during each shift) with a clean cloth and a cleaner such as Alcohol.

2. GAIN TIMEOUT

The Gain Stabilisation process timed out before stabilisation was achieved.

- check calibration source is Cesium-137
 - check calibration source is the correct size
 - check calibration source is inserted
-

10. APPENDIX D COMPAC PROTOCOL

The COMPAC protocol is a language, which facilitates the efficient and secure transmission of results from an analytical computer system to a host system. Data is transmitted in ASCII and comprehensive checks are performed to ensure that correct information exchange occurs.

The transmission of analytical results is always started by the analytical system, which acts as the "master" in all transactions.

An ASCII ENQ (enquiry) character is sent to the host computer to indicate that the analytical system wishes to send a data message holding an analysis result.

The host computer returns an ACK (acknowledge) or NAK (negative acknowledge) character to indicate acceptance or non-acceptance of the ENQ.

If the request to transmit is accepted, the transmission of the data message is then performed.

The host computer returns ACK if the data message is accepted and NAK if any error occurred during the transmission.

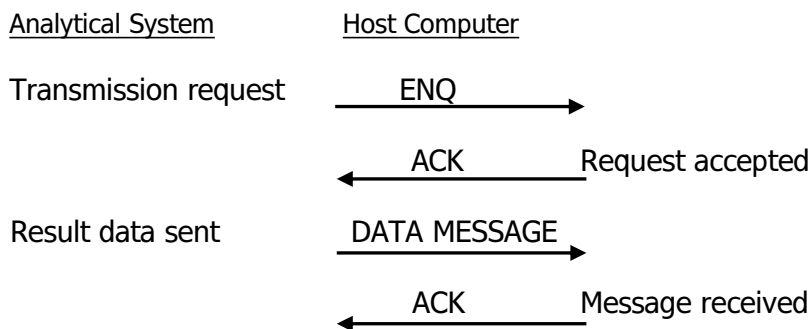
The analytical system will automatically try to send the whole message again (ENQ is set again) if the host computer fails to react within the timeout period (usually 10 seconds) or if an ASCII NAK is issued. A number of attempts (normally 3) are automatically performed in the case of:

- i. Time out
- or
- ii. NAK responses at the end of data transmission

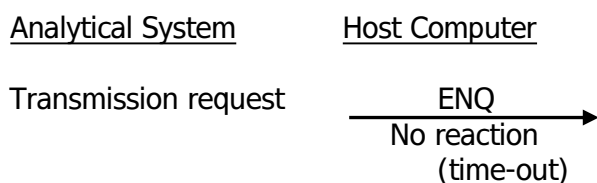
If they do not succeed, a warning message is printed on the system terminal.

NAK responses to ENQ characters do not affect the retry count since this does not indicate an error condition but that the host computer is not ready to receive the information.

Normal Sequence:



Sequence with time-out and transmission error:



11. APPENDIX Z SYSTEM SOFTWARE CHANGES

- Z.1 This is a new release of software entitled LabSPEC that runs under the Window operating system. This is such a radical software change that previous manual revisions are irrelevant so they have been removed. Manual changes in future will be recorded here.
-