

Practical Use of SpectrAA Series for Multielement Analysis

Application Note

Atomic Absorption

Introduction

The SpectrAA system is designed for automatic multi-element analysis in flame furnace and hydride modes, up to 12 elements in 67 samples (45 in furnace mode) with minimum operator supervision. When operating in the flame mode an operator should be near at hand but can be carrying out other tasks.

When operating in the furnace mode the system can be left unattended as there are no flammable gases and no exposed ignition source hence it can be left to operate overnight.

A scenario is described for analysis by flame and shows how even a very complex analytical program is easily handled by the SpectrAA.

The components of the system are:

SpectrAA-30 or SpectrAA-40 Atomic Absorption Spectrophotometer PSC-56 Programmable Sample Changer GTA-96 Graphite Tube Atomizer VGA-76 Vapor Generator Accessory DS-15 Data Station

The above units except for the DS-15, are only fitted with an on/off power switch. Programming and control of each unit in the system is through the DS-15 keyboard but no computer programming knowledge is required by the operator. In fact the system is designed for one finger operation using the "fill in the form" (F.I.T.F) method developed by Agilent Technologies.



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Versatility and ease of use has been achieved in the system design. The user friendly programming is designed to lead the operator through the correct procedures.

At power up the DS-15 screen displays 3 modes of operation. The operator selects one mode using the soft keys labelled:

DEVELOP METHOD MODIFY METHOD AUTOMATIC RUN

DEVELOP METHOD used by a chemist to set up and store methods for the use of other staff.

MODIFY METHOD used in a similar manner.

AUTOMATIC RUN used for routine automatic analysis.

Flame Analytical Procedure

The following scenario shows how all the facilities of the system may be used to perform a complex analytical program.

A laboratory has the following samples for analysis:

- 20 steel samples for the determination of chromium, copper, lead, manganese, molybdenum, nickel and silicon.
- 10 water samples for the determination of iron, sodium and arsenic.
- 10 slag samples for the determination of calcium, iron and silicon.

Steel Samples

Analytical method development has shown that most of the elements in the steel samples can be determined by normal calibration using mixed standards containing iron. The SpectrAA- will be calibrated in the concentration mode using the integration measurement mode for these elements.

Chromium and nickel concentrations must be determined accurately over a narrow range and bracketing standards calibration will be used for these determinations. High precision of measurement is also required thus PROMT mode will be used.

PROMT is Precision Optimised Measurement Time. This allows the operator to specify the precision of measurement

required (from 0.1% to 99.9%) and set a default time (1 to 500 seconds) for the system to achieve this. If the system is unable to achieve the precision within the default time specified, measurement will stop and the precision achieved printed out with the result. (The smaller the absorbance signal the longer the measurement time required to obtain a given precision).

This facility is extremely useful when high precision is required but the element concentration (and thus absorbance) is expected to vary widely. The operator can set the default time for the lowest expected absorbance knowing that higher absorbances will be read in a shorter time with no loss of precision or waste of analysis time.

The concentrations of lead are expected to be low thus the air-acetylene burner will be used to obtain higher sensitivity. (NOTE When high sensitivity is not required a burner change can be avoided by using a nitrous oxide-acetylene burner for an air-acetylene flame. This eliminates programming a "Pause" to change burners). PROMT mode could be used for measurement but in this case the mean of three replicate readings of 7-second integration is sufficient for the analytical requirements.

Chromium forms ferro-chrome compounds in the air-acetylene flame and will therefore be determined using the nitrous oxide-acetylene flame. This flame will also be used to determine molybdenum and silicon. All three elements will be determined using five second integration and three replicates.

A delay of five seconds is programmed when using continuous aspiration so that each solution will be aspirated for five seconds before any readings are taken. This ensures that the previous solution is washed out of the nebulizer/burner system. Cross contamination is prevented and the determinations will be more accurate. In addition a RINSE RATE and RINSE TIME may be programmed if required (where sample concentrations cover a wide range).

RECALIBRATION RATE and/or RESLOPE RATE are selected on previous experience with the samples and elements. Typical values could be reslope 10, recalibration 20.

Up to five standards may be used for each element calibration. 5 mixed standards will be loaded into the sampler carousel, but the number used will vary from element to element (see Table 1).

Program	Element	Delay		Sta	nda	rds		Sar	nple	Reslope	Recal	Flame	Notes
-			1	2	3	4	5	First	Last				
15	Fe	2	*	*				1	20		10	AA) Std
17	Ca	1	•	٠				11	20			NA) Addn
18	Si	-	•	*				11	20		—	NA	j
					P	AUS	SE (change (carousel)				
13	Cr			*				31	50		10	NA	ļ
19	Si		•	•	٠	•	•	21	45	14	—	NA	
23	Mo			*	*	*		41	50		-	NA	
9	Na	3	*	*	٠			1	10		_	AA	
1	Cu	_	•		*		•	21	50	15	30	AA	Reslope
7	Ni	_	*	*				31	50		10	AA	
8	Mn				•	• •	•	27	45		—	AA	
					F	PAU	SE	(change	burner)				
2	РЬ	2	*	*	*			31	50	_	10	AA	
			PA	USE	i (cl	hang	ge c	arousel,	connect	VGA-76)			
10	As		•	*] 1	10	I — 1	-	AA	Std Addn

Although RECAL will not be used for program 1, it is included here as it is part of the stored program. AA = Air-Acetylene

NA = Nitrous Oxide-Acetyleen

Water Samples

EPA methods specify standard additions calibration for water analysis. Iron will be determined by this method using the airacetylene flame burning on a nitrous oxide-acetylene burner. Continuous aspiration would normally be used for water samples but in this case the program "Fe in Slag" will be used which uses the micro-sampling technique. Thus two different sample types can be run on one program, the recalibration rate being defined by the number of water samples.

Arsenic will also be determined by standard additions calibration but using hydride generation. Sodium will be determined by EMISSION using normal calibration.

Two addition standards will be prepared from sample 1 and used to produce the calibration graph. The other 9 samples will be measured against this calibration because the matrices will be very similar.

Slag Samples

As the matrices will be complex and variable all elements will be determined using STANDARD ADDITIONS calibration. The dissolved solids concentration will be very high therefore continuous aspiration will not be used. Instead, the MICRO-SAM-PLING feature of the PSC-56 will be used and measurements made in peak height mode. In micro-sampling the sampling probe dips into the solution for a programmed time. During this time a certain volume of liquid is drawn into the flame, the longer the time the greater the volume (for a one second dip about 350 μ L). An integration "window" of eight seconds is set on the system. This allows enough time for the solution to travel through the nebulizer/burner system into the optical path to be measured. A volume of 200 μ L or more will give the same signal as continuous aspiration, 100 μ L will give about 80% and 50 μ L about 50% of the continuous aspiration signal. Micro-sampling is also useful when only small volumes of sample are available [1,2,3,4].

A RINSE between each sample and a SAMPLE FLUSH will also be programmed. Using sample flush the probe dips into a solution for half a second, the slug of liquid is drawn through the capillary "flushing out" any traces of the previous solution aspirated: no measurement is made on this solution. After about 10 seconds the probe dips into the solution for the specified dip time and the signal produced is measured.

Iron will be determined using the air-acetylene flame, silicon and calcium using the nitrous oxide-acetylene flame, both flames being run on the nitrous oxide-acetylene burner.

Typical system parameters for some elements are shown in Figures 1 to 6.

PROGRAM 15 Fe in Slag

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE LAMP POSITION LAMP CURRENT (mA) SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FLAME SAMPLE INTRODUCTION TIME CONSTANT MEASUREMENT TIME (sec) REPLICATES BACKGROUND CORRECTION AIR FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) DIP TIME (sec) SAMPLE FLUSH RECALIBRATION RATE

ABSORBANCE

PEAK HEIGHT

AIR-ACETYLENE

AUTO MICRO

5

0.2

NORMAL.

248.3

0.05

8.0

3

1

ΟN

13.5

2.00

5.0

0.0

YES

ABSORBANCE

AIR-ACETYLENE

FLAME EMISSION

CONCENTRATION

AIR-ACETYLENE

AUTO NORMAL

INTEGRATION

AUTO NORMAL

PROMT

8

Д,

5

0.05

10.0

0.2

DEE

13.5

2.00

5.0

0.5

5

3

1

0

0

0.05

13.5

2.00

5.0

3.0

NORMAL

589.0

10

1

0.5

NORMAL

351.5

BRACKETING STANDARDS

10

STANDARD ADDITIONS

Figure 1.

PROGRAM 7 Ni in Steel

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE LAMP POSITION LAMP CURRENT (mA) SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FI AME SAMPLE INTRODUCTION DELAY TIME TIME CONSTANT DEFAULT TIME (sec) PRECISION BACKGROUND CORRECTION AIR FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) RECAL TRRATION RATE

Figure 2.

PROGRAM 9 Na in Water

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FLAME SAMPLE INTRODUCTION DELAY TIME TIME CONSTANT MEASUREMENT TIME (sec) REPLICATES AIR FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) RECALIBRATION RATE RESLOPE RATE

Figure 3.

PROGRAM 10 As in Water

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE LAMP POSITION LAMP CURRENT (mA) SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FLAME SAMPLE INTRODUCTION DELAY TIME TIME CONSTANT MEASUREMENT TIME (sec) REPLICATES BACKGROUND CORRECTION AIR FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) RECALIBRATION RATE

ABSORBANCE STANDARD ADDITIONS INTEGRATION 10 0.5 NORMAL 193.7 AIR-ACETYLENE AUTO NORMAL 35 0.05 5.0 * OFF 13.5 2.00 5.0

0

Figure 4.

PROGRAM 13 Cr in Steel

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE LAMP POSITION LAMP CURRENT (mA) SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FLAME SAMPLE INTRODUCTION DELAY TIME TIME CONSTANT DEFAULT TIME (sec) PRECISION BACKGROUND CORRECTION N2O FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) RECALIBRATION RATE

ABSORBANCE BRACKETING STANDARDS PROMT R 7 0.2 NORMAL 520.8 N20-ACETYLENE AUTO NORMAL 5 0.05 10.0 0.2 OFF 11.0 6.00 1 5.0 10

Figure 5.

PROGRAM 18 Si in Slag

INSTRUMENT MODE CALIBRATION MODE MEASUREMENT MODE LAMP POSITION LAMP CURRENT (mA) SLIT WIDTH (nm) SLIT HEIGHT WAVELENGTH (nm) FLAME SAMPLE INTRODUCTION TIME CONSTANT MEASUREMENT TIME (sec) REPLICATES BACKGROUND CORRECTION N2O FLOW (L/min) ACETYLENE FLOW (L/min) RINSE RATE RINSE TIME (sec) DIP TIME (sec) SAMPLE FLUSH RECALIBRATION RATE Figure 6

ABSORBANCE STANDARD ADDITIONS PEAK HEIGHT 2 10 0.2 NORMAL 251.6 N2D-ACETYLENE AUTO MICRO 0.05 8.0 3 ΟN 13.5 9.00 2 5.0 0.5 YES Ö

Programming the Automatic Run

It will be useful to draw up a table (Table 1) when planning the analysis. This can then be used when compiling the sequence control page on the DS-15 (Figure 8).

Elements requiring standard additions calibration will be determined first.

AUTOMATIC RUN soft key is pressed and SEQUENCE SELEC-TION page (Figure 7) is displayed. The relevant programs are selected on this page and the soft key for SEQUENCE CON-TROL pressed. The programs selected are displayed by number and title on the left side of the screen (Figure 8).

	PROGRAM 15	Fe in Slag
	NO. 1 BLANK	REPLICATE 1
	ABSORBANCE	STANDARD ADDN'S BC ON
	PEAK HEIGHT	.8.0 (sec) AIR-ACETYLENE
PRINTING		
	SEQUENCE	SELECTION
1. Cu in Steel		16. Si ORE SAMPLES
2. Pb in Steel		17. Ca in Slag
3. Pb in Water		18. Si in Slag
4. Cu 820		19. Si in Steel
5.		29. A) Water Treatment
6.		21.
7. Ni in Steel		22. AL ORE SAMPLES
8. Mn in Steel		27 Mo in Steal
9 No in Water		24
18 Ac in Nater		25 TE OPE COMPLES
11 A- 0000		32
11. HS UKED		20.
17 fr in Staal		20
13. Cr In Dieet		29.
15 E. is Clas		78 N NATED
crourner is	17 10 17 10	30, NI IN WHICK
SEQUENCE 15	17 18 13 19	23 9 1 7 8 2 10
ULEAK		: : : : : : : : : : : : : : : : : : :
SEWUENCE		
Figure 7.		

	PROGRAM 15	Fe in Sla	g	
,	NO. 1 BLAN	K REPLICATE	1	
	ABSORBANCE	STANDARD A	ODN'S BC	ON
	PEAK HEIGHT	8. 0 (s	ec) AIR	-ACETYLENE
PRINTING				
	SEQ	VENCE CONTROL		
PROGRAM	PROGRAM	BELAY FIRST	LAST	FIRST
	10			WEASHBRUCKER.

ND. ID.		(nin)	SAMPLE	SAMPLE	MEASUREMENT
15. Fe in Slag		2	1	28	CALIBRATION
17. Ca in Slag		1	11	28	CALIBRATION
18. Si in Slag		0	11	20	CALIBRATION
13. Cr in Steel		8	31	58	CALIBRATION
19. Si in Steel		0	21	45	CALIBRATION
23. Ho in Steel		0	41	50	CALIBRATION
9. Na in Water		3	1	10	CALIBRATION
1. Cu in Steel		Ó	21	50	RESLOPE
7. Hi in Steel		0	31	50	CALIBRATION
8. Mn in Steel		0	27	45	CALIBRATION
2. Pb in Steel		2	31	50	CALIBRATION
10. As in Water		0	1	18	CALIBRATION
START AT PROGRAM NO.	15	SAMPLE	NO. 1	WITH	CALIBRATION
PAUSE AFTER PROGRAM	18	AUTO FI	AME OFF	YES	

Figure 8.

A DELAY of two minutes is programmed at the start of the first program (15 iron) to ensure the burner is stabilized at its operating temperature. FIRST and LAST sample numbers are then entered, in this example 1 and 20.

The FIRST MEASUREMENT can be a calibration, a reslope or sample.

If CALIBRATION is selected the system will calibrate with the number of standards specified. If RESLOPE is selected this will be applied to the stored calibration for that element. If SAMPLE is selected the concentration values will be calculated from the stored calibration data. If there is no stored calibration then only the absorbance is measured. (Only copper will be determined using a previously stored calibration and thus the first measurement will be reslope.)

For the next program (17, calcium) a delay of 1 minute has been entered to allow the burner to reach its operating temperature with the nitrous oxide-acetylene flame. First and last sample numbers are entered as before. This procedure is followed for programs 18 (silicon), 13 (chromium), 19 (silicon) and 23 (molybdenum) but without the delay time.

A delay of three minutes is entered for program 9 (sodium) which will allow the burner to cool down to its operating temperature; first and last sample numbers being entered as before.

Sample numbers are entered for program 1 (copper) and first measurement is altered to RESLOPE. Sample numbers are entered for programs 7 (nickel) and 8 (manganese).

A delay of two minutes is entered for program 2 (lead) to allow the burner to reach its operating temperature as it will be changed before this determination. The relevant sample numbers are then entered for program 2 and 10 (arsenic).

The final entry to be made is PAUSE AFTER PROGRAM which appears at the bottom of the screen. This PAUSE facility can be used several times in a sequence, enabling carousels, standards and reslope standards to be replaced; burners to be exchanged, rotated, or their height adjusted; the VGA-76 to be fitted. Lamps can also be exchanged if nine or more elements are being determined and multi-element lamps are not being used.

The first PAUSE entry will be 18 which will enable the carousels to be exchanged. When the system automatically pauses after the completion of program 18, the operator can insert the next PAUSE request, in our example 8, to change the burner. Finally when this second pause command is acted upon during operation the operator can insert the last PAUSE request, in our example 2, to enable the VGA-76 to be installed.

Having completed the sequence control page the NOTES soft key is pressed then the REPORT FORMAT soft key. This screen enables the following to be displayed and printed on the report:

Operator name Date Batch name

and the report parameters selected:

Format	Sequential* or Multi-element
Data printed	Mean Concentration only [*] or Mean Concentration and %RSDor Mean Concentration, %RSD & Mean Absorbance or Each Reading (Mean Concentration, %RSD, Mean Absorbance and Every Absorbance Reading).
Print	During Run* or After Run or Manual
Instrument status	Yes or No*
Notes	Yes or No*
Calibration results	Yes* or No
Calibration graph	Yes or No*
Sample labels	Yes* or No
Lines per page	Select for page length used (usually 66)

See Figure 9. Parameters normally used are indicated by *.

PROGRAM 1S NO. 1 BLANK Absorbance Integration	Fe in Water REPLICATE 1 STANDARD ADDN'S BC DN <u>5.0 (sec) AIR-ACETYLENE</u>
PRINTING	
REPORT	FORMAT
OPERATOR Date Batch Name	B.D.FRARY 22 APRIL 1985 ST907;W113;SL536
FORMAT DATA PRINTED PRINT INSTRUMENT STATUS HOTES Calibration Results Calibration Graph Sample Labels Lines PER Report Page	SEQUENTIAL EACH READING DURING RUN NO YES NO YES 72
	1942 F

Figure 9.

If PRINT DURING RUN is selected then all the other features selected will be printed during the run. All results obtained after the start of the run can be inspected on the print-out. However when using PRINT MANUAL or PRINT AFTER RUN, only the results currently displayed on the screen are available for inspection. The analytical run can be completed in a slightly shorter time if PRINT MANUAL is selected.

The absorbance values of all solutions are stored on the program disc as they are read (no matter which PRINT status is selected). If a power failure occurs during the analysis the only data lost will be that for the solution under analysis. No other data is lost or corrupted.

At the end of the analytical run all the data may be archived and a multi-element report produced. It is recommended that the data is archived before another run is performed as the program disc is cleared of data when START is pressed.

If SAMPLE LABELS is selected then a screen as shown in Figure 10 is displayed (up to 12 alphanumeric characters can be used). INCREMENT LABELS is a time saving facility. In Figure 10, W113 is the first water sample and is entered as sample 1. INCREMENT LABELS is pressed and samples 2 to 10 are automatically labelled W114 to W122. The cursor is moved to sample 11, relabelled SL536 and INCREMENT LABELS pressed. Samples 12 to 20 are automatically labelled SL537 to SL545. Finally the cursor is moved to sample 21, relabelled S907 and INCREMENT LABELS pressed. Samples 22 to 42 are automatically labelled S908 to S928. MORE LABELS is pressed, samples 43 to 67 are displayed and are automatically labelled S929 to S953.

FRINTING SAMPLE LABELS 1. W 113 2. W 114 3. W 115 4. W 116 5. W 117 6. W 118 7. W 116 5. W 117 6. W 118 7. W 115 8. W 128 9. W 121 18. W 122 11. SL 536 12. SL 537 13. SL 539 14. SL 539 15. SL 540 16. SL 541 17. SL 542 18. SL 543 19. SL 544 26. SL 545 21. ST 907 22. ST 908 23. ST 9199 24. ST 916 25. ST 911 26. ST 912 27. ST 913 28. ST 914 29. ST 915 36. ST 916 31. ST 917 32. ST 918 33. ST 919 34. ST 920 35. ST 921 36. ST 922 37. ST 923 38. ST 924 39. ST 925 40. ST 926 41. ST 927 42. ST 928 MORE INCREMENT 10. ST 928	PR00 ND ABS0 PEA1	RAM 15 Fe in 1 Blank Replic Drbance Standa (Height 8.0	Stag ATE 1 IRD ADDN'S BC ON (sec) AIR-ACETYLENE
SAMPLE LABELS 1. W 113 2. W 114 3. W 115 4. W 116 5. H 117 6. H 118 7. W 115 8. W 120 9. W 121 10. W 122 11. SL 536 12. SL 537 13. SL 539 14. SL 539 15. SL 540 16. W 122 11. SL 536 12. SL 543 19. SL 544 20. SL 545 21. ST 907 22. ST 908 23. ST 909 24. ST 910 25. ST 911 26. ST 912 27. ST 913 28. ST 914 29. ST 915 36. ST 916 31. ST 917 32. ST 918 33. ST 919 34. ST 920 35. ST 921 36. ST 922 37. ST 923 38. ST 924 39. ST 925 46. ST 926 41. ST 927 42. ST 928	PRINTING		
1. W 113 2. W 114 3. W 115 4. W 116 5. W 117 6. W 118 7. W 119 8. W 128 9. W 121 10. W 122 11. SL 536 12. SL 537 13. SL 538 14. SL 539 15. SL 537 16. SL 541 17. SL 542 18. SL 543 19. SL 544 26. SL 545 21. ST 907 22. ST 908 23. ST 912 27. ST 913 28. ST 911 26. ST 915 36. ST 916 31. ST 917 32. ST 918 33. ST 919 34. ST 920 35. ST 921 36. ST 922 37. ST 923 38. ST 924 39. ST 925 48. ST 926 41. ST 927 42. ST 928 MORE INCREMENT 1000000000000000000000000000000000000		SAMPLE LABELS	i i i i i i i i i i i i i i i i i i i
MORE INCREMENT	1. W 113 4. W 116 7. W 119 10. M 122 13. SL 533 16. SL 541 19. SL 544 22. ST 908 25. ST 911 28. ST 914 31. ST 917 34. ST 928 37. ST 923 40. ST 926	2. W 114 5. W 117 8. W 120 11. SL 536 14. SL 539 17. SL 542 20. SL 542 23. ST 989 26. ST 912 32. ST 915 32. ST 918 35. ST 921 38. ST 924 41. ST 927	3. W 115 6. W 118 9. W 121 12. SL 537 15. SL 540 18. SL 543 21. ST 907 24. ST 916 36. ST 916 37. ST 919 36. ST 922 39. ST 928
	MORE INCREM	ENT	

Figure 10.

Loading Off Carousels

Two carousels will be used, one for STANDARD ADDITIONS calibration (water samples 1 to 10, slag samples 11 to 20), the other for NORMAL calibration (steel samples 21 to 50).

The standard additions carousel will be loaded as follows:

Sample position 1 Blank 2 Addition 1 3 Addition 2 4 Addition 0 (sample 1) 5 to 13 Addition 0 (samples 2 to 10) 14 Blank 15 Addition 1 16 Addition 2 17 Addition 0 (sample 11) 18 to 26 Addition 0 (samples 12 to 20)

The second carousel, for normal calibration will be loaded:

Multi-element standards in standard positions 1 to 5. Blank and reslope solutions in their respective positions. Water samples in sample positions 1 to 10 and steel samples in positions 21 to 50.

Operation

When the operator has fitted the required burner and carousel the flame is lit and START key pressed.

Operation proceeds as follows:

The system recalls the first program in the sequence (15) resets the sampler and checks all system parameters are correctly set. It waits 2 minutes for the burner to warm up while aspirating distilled water. It then carries out the calibration of the system with the addition standards for waters and measures the first 10 samples (waters). It then recalibrates the system with the addition standards for slags and measures samples 11 to 20 (slags).

The next program (17) is recalled and the system set up to these parameters. The burner is allowed to warm up for the programmed 1 minute before calibration proceeds. Samples 1 to 10 are then measured.

The next program (18) in the sequence is recalled and the system set up on these parameters. Calibration proceeds immediately as no delay has been programmed (the burner is already at its operating temperature). Samples 1 to 10 are measured for this element.

After sample 10 is measured the next program (13) is recalled and the system "beeps" to remind the operator that a programmed PAUSE has been reached. The operator presses STOP, changes carousels, alters PAUSE AFTER PROGRAM to 8 and presses START. As no delay has been programmed the system proceeds to calibrate and then measures samples 31 to 40. A RECALIBRATION is then carried out and samples 41 to 50 are measured. Program 19 is then recalled, the system set up, calibration is performed and samples 21 to 35 are measured. RESLOPE is performed and samples 36 to 45 measured.

Program 23 is recalled, the system set up, calibration is performed and samples 41 to 50 measured.

Program 9 is recalled, the system is set up and changes to the air-acetylene gas mixture waiting for the 3 minutes programmed delay while the burner cools down to its operating temperature. Calibration is performed and samples 1 to 10 are measured.

Program 1 is recalled, the system set up, a RESLOPE is performed and applied to the stored calibration. If the reslope is correct, samples 21 to 36 are measured. A RESLOPE is performed and samples 37 to 50 measured.

Program 7 is recalled, the system set up, calibration is performed and samples 31 to 40 measured. A RECALIBRATION is performed and samples 41 to 50 measured.

Program 8 is recalled, the system set up, calibration is performed and samples 27 to 45 measured. Program 2 is recalled and the system "beeps" to remind the operator that a programmed PAUSE has been reached. The operator presses STOP, presses FLAME OFF, changes burners, alters PAUSE AFTER PROGRAM to 2 then presses IGNITE and START. The system waits 2 minutes for the burner to warm up then calibrates and measures samples 31 to 40. A RECALIBRATION is then carried out and samples 41 to 50 are measured.

Program 10 is recalled and the system "beeps" to remind the operator that a programmed PAUSE has been reached. The operator presses STOP and FLAME OFF, connects the VGA-76 hydride generator, replaces the carousel in use by the one for standard addition calibration and presses IGNITE. START is pressed, the system performs the calibration procedure and determines the arsenic concentrations of samples 1 to 10. After the last sample is determined the flame is automatically extinguished and the system "beeps" indicating the analytical run has been completed.

Archiving and multi-element report

The INDEX key is pressed and page 19 UTILITIES selected. From the display ARCHIVE is selected followed by the file (on the utilities disc) for archiving. The ARCHIVE soft key is pressed and the data is permanently stored.

After returning to the UTILITIES index, PRINT REPORTS is selected. From the next display the data set required is chosen and then the elements required. REPORT FORMAT soft key is pressed and the following is displayed:

Format	Sequential or multi-element*
Data printed	Mean concentration only [*] or Mean concentration and %RSDor Mean concentration, %RSD and mean absorbance or Each Reading (Mean Concentration, %RSD, Mean Absorbance and Every Absorbance Reading).
Instrument status	Yes or No*
Notes	Yes or No*
Calibration results	Yes* or No
Calibration graph	Yes or No*
Sample labels	Yes* or No
Weight correction	Yes* or No
Lines per page	Select for page length used (usually 66)

Parameters normally used are indicated by *

If WEIGHT CORRECTION is selected the measured concentration of the elements in the samples is automatically calculated before print out. This is a time (and error) saving facility.

PRINT REPORT is pressed and the multi-element report is duly obtained (Figure 11).

Applications Laboratory, VARIAN TECHTRON, 679 Springvale Road, MULGRAVE, VICTORIA, AUSTRALIA 3170. Telex: AA 31229 Telephone: (03) 560 7133									
Operator		B.D.FRARY							
Date		22 April 1985							
Batch	1	ST 907/	953; W	/ 113/12	22;	1			
		SL 536/	545						
Program 15		Fe in Slag							
Program 17		Ca in Si	ag						
Program 18		Si in Sla	g						
Program 13		Cr in Steel							
Program 19		SI in Steel							
Program 23		Mo in S	teel						
Program 9		NainW	ater						
Program 1		Cuin St	eel						
Program 7		NI IN STE	eel						
Program 8		Millin Si Dhào St	(88) 201						
Program 2	}	rom⊃t AsiaW	əter						
Flogram to		-Co	त(छ। ० :	0-	- D:				
	re *	l Ca	୍ର %	%	୍ର %	36			
BI ANK			~~						
CONC	0.00	0.0	0.0	0.00	0.00	0.00			
STANDARD 1									
CONC			{	18.00	0.10	l i			
STANDARD 2				20.00		0.10			
STANDARD 3				20.00	0.25	0.25			
STANDARD 4		l r			0.20	0.20			
						U.50			
STANDARD 5 CONC					0.50				
ADDITION 1									
CONC	1.00	10.0	5.0						
ADDITION 2									
CONC	2.00	20.0	10.0						
W113									
CONC	1.12								
W114						l i			
CONC	0.98					i i			
• •	[.			[
SL 542 CONC	3.63	41.6	12.4						
SL 543 CONC	2.54	45.3	19.2						
51910		i		18.3	0.09	0.013			
ST 919				17.9	0.11	0.019			
* Concentration samples and %	n Units 5 in Sla	a not sha ag samd	own as oles.	will be	ppm in	Water			
sampios and vent orag sampios.									

Figure 11.

As all the analytical data is now archived other reports can be generated at any time if required. For example a report showing all data for arsenic (program 25) is required. This is easily produced by:

- Accessing the utilities disc, selecting the appropriate archive file, then selecting the element to be printed (in this case arsenic). Proceed to report format via the soft key and select the options required, then print report.
- The SpectrAA- system can be used for an automatic run in the same way. However as it has a manually operated turret the operator must bring the next lamp into the operating position when prompted by the DS-15. If multielement lamps are used then this requirement is reduced or even eliminated.

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