

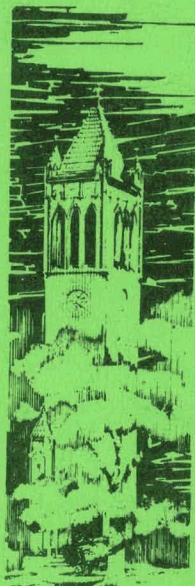
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SPECIFICATIONS FOR AN
INDUCTIVELY COUPLED
PLASMA SIMULTANEOUS
MULTIELEMENT ANALYSIS
SYSTEM

W.J. Haas, Jr., R.K. Winge
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FOREWORD

The Spectrochemistry Group of the Ames Laboratory has been a pioneer in the development of inductively coupled plasma - atomic emission spectrometry (ICP-AES) for simultaneous multielement analyses (SMA). Today ICP-AES is recognized as one of the most important and rapidly growing fields of analytical endeavor. At least nine scientific instrument manufacturers are now supplying complete ICP-SMA systems for the commercial market.

Because an ICP-SMA system is a package of relatively sophisticated software and hardware whose analytical capabilities and usefulness are quite fixed once the instrument is purchased, the writing of appropriate specifications is both a very important and non-trivial undertaking. The Ames Laboratory has received numerous requests for advice and assistance in this area.

The specifications contained in this document represent our best efforts, to date, to specify those items of hardware, software, and overall system performance which are of known importance in ICP-SMA instrument systems. Particular attention is directed to two important aspects of desired ICP-SMA system capability which, to the best of our knowledge, have not been adequately provided in previous systems. These are:

- 1) the specified data reporting and quality assurance features which are required for improving the validity and interpretability of the analytical results, and
- 2) the specified background correction procedures which are necessary for accurate quantitative determinations near the limit of detection.

Our objective in preparing this specification package has been to specify and purchase an ICP-SMA system for our own use which will provide maximum value and utility for the simultaneous determination of major, minor and trace quantities of the elements in a wide variety of sample materials. Modifications of the analytical line array specified here may be appropriate for other, more specific, analytical tasks. Budgetary realities may necessitate additional, appropriate modifications. It is anticipated, however, that the specification package presented here will have general utility as a guide in the preparation of ICP-SMA procurement packages for other laboratories and other analytical applications.

SPECIFICATIONS FOR AN INDUCTIVELY COUPLED PLASMA
SIMULTANEOUS MULTIELEMENT ANALYSIS SYSTEM

This specification package, including Table 1, and Figures 1 and 2, contains specifications for an inductively coupled plasma--simultaneous multielement analysis system. The complete system shall include the components listed in Items 1 through 6, below. The complete system and the services provided by the vendor shall conform to the complete set of specifications given in Sections 0.0 through 8.3 of this package.

1. Polychromator with entrance slit and appropriate exit slits.
2. Inductively-coupled plasma excitation source with high-frequency power supply, torch assembly, gas flow regulating system, and sample nebulizing system.
3. Photomultipliers and necessary operating and measurement electronics.
4. Analytical program, data acquisition, processing, and recording system.
5. All power supplies, control circuitry, and interlocks necessary for safe operation of the above items.
6. Electrical performance, installation, hardware and software documentation, and training of customer personnel.
7. Specific performance tests.
8. Delivery, installation, acceptance, and warranty specifications.

SPECIFICATIONS:
INDUCTIVELY COUPLED PLASMA
SIMULTANEOUS MULTIELEMENT ANALYSIS SYSTEM

0.0 General

- 0.1 The fulfillment of all of the specifications contained in this specification package shall be guaranteed by the vendor.
- 0.2 Items 1 through 6 on the purchase order shall constitute a complete, fully operative, simultaneous multielement analysis system.
- 0.3 The complete analytical instrument system shall be capable of detecting the elements listed in Table 1 of these specifications when the elements are present in aqueous solution at the respective concentration levels specified in Table 1. The complete analytical instrument shall also be capable of quantitative determination of the same elements with a precision of plus or minus ten percent (for 10 consecutive determinations) when the concentration levels are equal to ten times the respective detection limit concentrations stated in Table 1; with a precision of plus or minus one percent for concentrations equal to or greater than 100 times the specified detection limit concentrations.
- 0.3.1 Option: A quotation is also requested for a complete analytical system which will only provide analytical capability for those elements/lines which are listed in Part 1 of Table 1. This optional system shall conform in all other respects to all of the specifications contained in this specification package.
- 0.4 The vendor is to demonstrate to a representative of the buyer in the vendor's laboratory before delivery, and in the buyer's laboratory after delivery, that the instrument conforms to the specifications detailed here and to other tests approved by the buyer. The necessary test solutions will be provided by the buyer.
- 0.5 The bidder is to quote on the cost of field and factory installation of all necessary components for the addition of one or more additional channels to the polychromator. The components shall include all optical parts, all necessary

electronic components, and any necessary software modifications for the additional channel(s), as well as the installation.

- 0.6 The bidder is to quote on the cost of field and factory conversion of one or more existing exit slits to new wavelengths.
- 0.7 The following sections, corresponding in number with Items 1 through 8 on the purchase order, give detailed specifications for the individual components of the system. Changes or exceptions to these specifications must be noted, and differences between the specified instrument and the one bid on shall be detailed clearly. The vendor is asked to describe and provide quotes for standard instrument options which may be available.
- 0.8 The vendor shall provide alternate specifications for any specifications in this package which the vendor's instrument cannot meet. If the specifications can be met only at additional cost above that charged for a standard instrument the additional cost should be noted.
- 0.9 The vendor is asked to provide quotations for two purchase options:
- 1) Outright purchase;
 - 2) Lease with purchase option.
- (A five year term is preferred for the lease-purchase case.)

1.0 Polychromator.

- 1.1 The polychromator shall be equipped with a holographically recorded grating.
- 1.2 The polychromator is to be equipped with exit slits for the elements and lines listed in Table 1 of this specification package.
- 1.3 The exit slits for the elements listed shall be aligned on their appropriate wavelengths by the vendor.
- 1.4 Effect of Stray Light and Recombination Spectra. The vendor shall demonstrate that the total effect of the stray light level of the polychromator and of the spectral background due to recombination spectra is equivalent to no more than two times the detection limit concentrations listed in Table 1 of these specifications, for each readout channel, when the solutions listed below are nebulized.

Ca	5 mg/ml
Mg	5 mg/ml
C	5 mg/ml
Na	5 mg/ml
K	5 mg/ml
Fe	5 mg/ml
P	5 mg/ml
Al	5 mg/ml

- 1.5 Isolation of Secondary Optics and Readout Electronics. The vendor shall demonstrate that optical and electrical crosstalk which may occur in the post exit slit region of the system are equivalent to no more than two times the detection limit concentrations listed in Table 1 of these specifications for all of the exit slit-readout channels when solutions of the following concentrations are nebulized.

Ca	5 mg/ml
Mg	5 mg/ml
C	5 mg/ml
Na	5 mg/ml
K	5 mg/ml
Fe	5 mg/ml
P	5 mg/ml

- 1.6 By means of a thermostatted compartment or otherwise the polychromator shall remain in

optical alignment, that is, all exit slits shall remain aligned on their respective wavelengths over the ambient temperature range from 18-30 C (65-85 F).

- 1.7 The polychromator shall have provision for computer-controlled wavelength scanning of a 0.5 nm region past any exit slit. The scanning range for each exit slit shall include the programmed analytical line wavelength plus or minus 0.25 nm. Provision for computer controlled integration or time averaging of the intensity data at operator selected wavelengths within the scanning range shall be provided.
- 1.8 The vendor shall state the exact wavelength ranges of the polychromator for which quotations are given.
- 1.9 The spectral bandpass of the polychromator shall be less than 0.025 nm.
- 1.10 The complete analytical instrument system shall be constructed in a way that permits simultaneous observation of the plasma by a second spectrometric instrument. The orientation of the optical path for the second spectrometer system shall be planned so that, when installed, the second spectrometer will not block any of the operating controls for the polychromator or plasma system, and so that light falling on either the polychromator or the spectrometer cannot be reflected into the optical system of the other. The operating controls for the additional spectrometer system shall be conveniently accessible to the instrument operator from his normal position for operation of the (main) plasma-polychromator system.
- 1.11 A lens or mirror is to be supplied for the entrance optics of the polychromator. The transfer efficiency of the lens or mirror which is supplied shall be 70% or greater for radiation with a wavelength of 185.0 nm.
- 1.12 The average first order reciprocal linear dispersion of the polychromator shall be less than 0.55 nm/mm.

- 2.0 Inductively-coupled plasma excitation source with high-frequency power supply, torch assembly, gas flow regulating system, and sample nebulizing system.
- 2.1 The plasma excitation source, in conjunction with the other parts of the facility, shall be capable of achieving the detection limits listed in Table 1 of these specifications without pretreatment or concentration of the sample. Addition of moderate amounts of acid to the sample for preservation purposes is acceptable.
- 2.2 The output power of the rf generator shall be at least 2.5 kW at 27.12 MHz. However, the vendor is asked to provide alternate quotations for both 2.5 kW and 5. kW rf generators (and appropriate impedance matching networks and programmable, automatic power level controllers as specified in Sections 2.3 and 2.4).
- 2.3 The high frequency power supply shall provide automatic, programmable, power level control to within plus or minus 0.1% when used in actual plasma operation.
- 2.4 The impedance matching network must match the plasma to the 50 ohm high frequency power input line so that the ratio of reflected rf power to forward rf power is maintained at a value equal to or less than 0.005, for forward power levels from 800 to 2000 watts (800 to 4000 watts for the optional 5kW generator).
- 2.5 Meters shall be provided to show forward and reflected power measured at the input to the impedance matching network.
- 2.6 If automatic, electronic control of the plasma impedance matching network is not standard on the instrument, the vendor shall provide a separate quotation for this option.
- 2.7 The nebulizer and the plasma must be operable at nebulizer argon flowrates within the range of 0.75 to 2.0 liters of argon per minute.
- 2.8 The nebulizer and sample aerosol system shall be capable of nebulizing and conducting the aerosol of sample solutions containing up to 50% nitric acid without damage to either the nebulizer or to the aerosol transport facilities.

- 2.9 It shall be demonstrated that memory effects in the nebulizer-sample delivery system are less than 1%. That is, the calcium signal from a test solution of 10 mg/l Ca, when nebulized into the plasma for ten minutes shall, upon substituting deionized water, drop to less than 1% of its original value within 30 seconds.
- 2.10 Lateral and vertical adjustment of the operational plasma torch assembly with respect to the optical axis of the polychromator shall be provided as follows:
- 2.10.1 The vertical range of adjustment of the plasma torch assembly shall allow the traverse of the region (by the optical axis) from 0 mm to 40 mm above the load coil of the torch.
- 2.10.2 The horizontal range of adjustment of the plasma torch assembly shall allow the torch to traverse a 30 mm region (plus or minus 15 mm from the optical axis) perpendicular to the optical axis of the polychromator.
- 2.10.3 Adjustment of the plasma torch facility over the ranges specified in Sections 2.10.1 and 2.10.2 shall not affect the impedance match between the plasma and the 50 ohm high frequency power input line. That is, if the optimum standing wave ratio is set by adjustment of the impedance matching network, the same standing wave ratio should be maintained after adjustment of the plasma torch assembly, without readjustment of the impedance matching network controls.
- 2.11 The vendor shall provide at least one nebulizer, nebulization chamber, and torch assembly for the instrument system described herein, and shall provide price and delivery information for replacement of these items. The replacement components shall be completely interchangeable with the components originally supplied, without loss of system performance.
- 2.12 A flow control system is to be provided for the nebulizer argon flow stream and for each argon flow stream which is required for normal operation of the plasma as part of the complete analytical

instrument system. The flow rate in the nebulizer argon stream shall be controlled automatically, so that the sample solution uptake rate remains constant to within plus or minus one percent for main argon supply pressures between 2400 and 100 psi. The flow rates for the other argon streams which are required for analytical operation of the plasma shall be maintained, automatically, to within plus or minus one percent for the same argon supply pressure range as specified above, and for all flow rate settings which are appropriate for analytical use of the plasma.

- 2.13 For the case in which the main argon supply is maintained at a constant pressure of 100 psi, and for argon flow rates which are appropriate for use of the complete analytical system, the argon flow control systems shall maintain those flow rates at values which are constant to within plus or minus 0.1 percent.
- 2.14 (Please quote on this item as an option.)
The complete analytical system shall be capable of automatic, unattended introduction and analysis of a series of at least 50 "unknown" sample solutions. The necessary "wash," "introduce blank," and "introduce sample," operations shall be fully and conveniently programmable by the operator. Separate bulk reservoirs shall be provided for "wash" and blank solutions, and the blank and all sample solutions shall be protected against environmental contamination except during the actual time required for sample changing.

3.0 Photomultipliers and Related Operating and Measurement Electronics.

- 3.1 A photomultiplier and appropriate measurement electronics shall be provided for each exit slit listed in Table 1 of these specifications. The measurement electronics shall be designed to allow measurements to be made for all channels, i.e., all analysis wavelengths, simultaneously.
- 3.2 The value of the high voltage supplied to the photomultiplier tubes (measured at the photomultipliers) shall be constant to within plus or minus 0.01 percent over an eight hour period, and for ambient temperatures in the range 18 - 30 C. This stability shall be attained for all the photomultipliers, under the normal operating conditions for the complete analytical instrument system, and over the entire range of high voltage for which the photomultipliers were designed.
- 3.3 For input signals within the range $1.0E-10$ Ampere to $1.0E-6$ Ampere, supplied from a constant current source, the measurement electronics shall supply output signals which are accurate to within plus or minus one percent, and stable to within plus or minus 0.1 percent over an eight hour period. (Please see Section 4.3)
- 3.4 The above stability (Section 3.3) shall be maintained over the ambient temperature range 18-30 C and for an ambient relative humidity up to 65 percent.
- 3.5 The vendor shall provide quotations for the option of magnetic shielding of the photomultiplier tubes, and for the option of rf shielding of the photomultiplier tubes.

4.0 Analytical Program, Data Acquisition, Processing, and Recording System.

- 4.1 The data acquisition, processing, and recording system shall consist of a dedicated computer and dual floppy-disk system, hard copy input/output terminal, and all software and interfacing necessary to control the analytical facility and to receive and process data from the analytical facility. The computer system shall be hardware and software compatible with the DEC PDP-11/03 system and it shall have sufficient backplane space to accommodate an additional serial input/output unit for a (customer supplied) video display unit. A complete computer diagnostic software package shall also be provided. (Please see also Section 6.10 of these specifications.)
- 4.2 The hard copy input/output terminal shall print at a minimum speed of 30 characters per second. However, the vendor shall also quote on the option of providing a hard-copy input/output terminal which will print at a minimum speed of 120 characters per second.
- 4.3 All results of intensity measurements shall be reported in terms of photomultiplier output current (Amperes).
- 4.4 The analytical program shall provide a hard copy, real-time log of all solutions which are run, all analytical program commands and parameter values entered by the operator, and any other comment-type information which the operator may wish to enter into the permanent record. The real time and date shall be printed for each log entry.
- 4.5 The data acquisition system shall have provision for operator specification of both computer-controlled "wait," and "take data" time periods. The "wait" period is the time which is to be allowed (for sample cleanout and/or plasma stabilization) between introduction of each new sample or reference solution and the initiation of intensity measurements for that solution; the "take data" time is the time period throughout which the intensity measurements shall be conducted. The "take data" time is also referred to in these specifications as the integration time.

- 4.6 At the operator's choice the measurement data shall be printed in any one or all of the following forms:
- 4.6.1 Total sample and reference blank line intensities (i. e., analyte line plus spectral background intensity for sample and reference blank solutions).
 - 4.6.2 Net sample intensities, i.e., that part of the total intensity observed at each analytical wavelength which is due to the analyte element of interest in that sample.
 - 4.6.3 Net sample intensities as in 4.6.2, but expressed in concentration units as obtained from the appropriate calibration data.
- 4.7 Two modes of operation shall be provided for background correction, i. e., for the determination of the net sample intensities as defined in Section 4.6.2. The necessary calculations and control operations for both modes shall be performed, automatically, by the analytical program. The two modes will be referred to in the following as
- 1) the blank subtraction mode, and
 - 2) the sample background correction mode.

In the first mode, intensities shall be measured (at the analysis line wavelengths only) for an appropriate blank solution and for each sample solution. The net sample intensity for each analysis line wavelength shall be computed by simply subtracting the "blank" intensity values from the corresponding intensity values obtained for the sample. (This is the most commonly used background correction approach for inductively coupled plasma spectrometry.)

For the second background correction mode, provision shall be made for automatic, analytical program controlled measurement of the intensities at the analysis line wavelengths and at one or more operator specified wavelengths in the immediate vicinity of each of the analysis lines. The intensity data which are obtained are to be treated as described below, to obtain the net sample intensity data as defined in Section 4.6.2. Suppose that

I_{Ai} is the measured intensity at the i^{th} analysis

line wavelength, and that

I_{ji} is the intensity (spectral background) at the j^{th} (non-analyte) wavelength in the vicinity of the i^{th} analysis line.

Then an array of relative intensity values,

$$r_{ji} = I_{Ai} - I_{ji},$$

and, similarly, an array of net intensity values,

$$N_{ji} = r_{ji}(\text{sample}) - r_{ji}(\text{blank}),$$

can be defined for each analyte line (i) and for each sample. One or more (but perhaps not all) of the values, N_{ji} , correspond to valid measurements of the net sample intensity at the i^{th} analysis wavelength. Provision shall be made, therefore, for operator selection of the appropriate value or values which are to be reported and/or used in subsequent operations which require net sample intensity data. If the operator specifies the use of more than one element of the net intensity array for any analysis line, then the average of the selected elements shall be used for subsequent reporting and/or use of the net sample intensity data.

For both background correction modes, the operator shall have the option of either performing new blank intensity measurements as he desires or of specifying that a blank is to be run before and after each sample solution. In the first case the most recently determined values shall be used for background correction calculations. In the second case, the intensities to be used for background correction shall be the computed average of the intensities for the blank run immediately preceding each sample and for the blank run immediately after the sample.

- 4.8 The analytical program shall also allow, at the operator's choice, the ratioing of the net intensity of any line to that of any other analytical line. In this mode, referred to below as the internal reference mode, the analytical calibrations and concentration calculations shall be based upon these ratios. The measurement data shall be printed, at the operator's choice, in any one or all of the following forms:

- 4.8.1 Intensities for sample with internal standard(s).
 - 4.8.2 Intensities for reference blank.
 - 4.8.3 Intensities for reference blank with internal standard(s).
 - 4.8.4 Net analyte/net internal standard line intensity ratios.
 - 4.8.5 Analyte intensity ratios expressed in concentration units as obtained from the appropriate calibration data for internal reference mode operation.
- 4.9 Computer-controlled, repetitive measurement of intensities, intensity ratios, or analyte concentrations shall also be an operator selectable mode of operation. The operator shall have the option of choosing printout of the individual results of the measurements and/or a printout of the average values and precision data for the channels selected.
- 4.10 The analytical program shall have provision for the determination of detection limit concentrations as defined in Section 7.1 of these specifications. The operator shall have the option of specifying both the number of integrations and their duration. It is assumed that the operator will enter the known concentrations of the analytes for the first detection limit determination. For subsequent determinations, however, he shall have the option of using the previously entered concentrations, a multiple of the previous concentrations, or entering new concentration data.
- 4.11 A linear relationship between net analyte line intensities (or ratios) and analyte concentrations may not be assumed for the purpose of calibration.
- 4.11.1 Provision shall be made for operator specification of the polynomial types which are to be used for calibration.
 - 4.11.2 The analytical calibration program shall have provision for multiple runs of the calibrating solutions, and for printout of the observed intensity and concentration precision information for those cases in

which three or more runs are made for any of the calibrating solutions.

4.11.3 For determination of the analytical calibration parameters, the calibrating data points shall either be weighted equally, or, at the operator's choice, by factors which are inversely proportional to the squares of the calibrating concentrations, or by factors which are inversely proportional to the respective variances of the measured net intensities for the calibrating solutions.

4.12 The analytical calibration data and calibration parameters shall be stored on floppy disk and shall be available for printout upon operator command. In addition, for each line calibration, the following information shall be stored on the floppy disk and shall be available for printout upon operator command.

4.12.1 Slopes and relative slope ranges, RSR, where

$$RSR = 2*(S_g - S_l)/(S_g + S_l).$$

Here S_g = slope at the greatest calibration concentration, and

S_l = slope at the lowest calibration concentration.

4.12.2 Concentration axis intercept values and corresponding uncertainties, when sufficient data is available for the determination of the latter quantities.

4.12.3 Root mean square relative deviation of the fit of the calibration data, R, where

$$R = 100 \sqrt{\frac{1}{n} \sum_{i=1}^n \left(\frac{c_{if} - c_i}{c_i} \right)^2}$$

Here n = the number of calibrating concentrations which were used.

c_i = the i^{th} calibrating concentration, and

c_{if} = the concentration found from the calibrating function for the i^{th}

calibrating solution.

- 4.13 The analytical program shall include provision for automatic application of spectral interference corrections, as an operator option. The interferences may not be assumed to be linear functions of the interferent concentrations. The interference corrections shall be based upon known and measured interferent concentrations, and the analytical program shall permit a choice between direct, on-line determination of the necessary interference correction parameters by the analytical program, or use of previously determined parameter values recalled from disk storage.
- 4.14 The accuracy of the interference correction procedure shall be such that the (interference corrected) concentration results for solutions containing fixed concentrations of analytes, but varying concentrations of interferents, shall agree with each other to within plus or minus five percent when the interferent/analyte net intensity ratio (as measured at the appropriate analyte wavelength) is less than or equal to ten, and to within plus or minus two percent when the interferent/analyte intensity ratio is less than or equal to one.
- 4.15 The analytical program printout shall identify those analyte concentration results which lie outside the actual range of analyte concentrations which was employed for calibration of the instrument. For example, by printing * following analyte concentration results which are less than the experimental detection limit for that analyte, by printing < following analyte results which are less than the lowest concentration of that analyte which was used in the calibration, and by printing > following analyte concentration results which are greater than the highest concentration of that analyte which was used in the calibration.
- 4.16 (Please quote on this item as an optional additional feature.)
The analytical program shall have provision for optional monitoring of observed blank solution intensities. The purpose of the monitoring is to guarantee that the observed blank intensity for each analysis wavelength remains within predetermined control limits throughout the entire course of a series of determinations. Standard

procedure upon the occurrence of an out of limit condition shall be to print a warning message to the operator, to identify the channel(s) and analyte(s) whose blank intensities are out of limits, and to repeat the blank measurement. If any of the second set of blank intensities are also out of limits, the operator shall be instructed to change (renew) the blank solution. If the out of limit condition still persists, it shall be assumed that the current system performance warrants some remedial action and that (at least) the system should be recalibrated and new control values established.

One procedure for setting the blank control values is suggested in the following. Suppose that 25 consecutive ten-second measurements of the blank intensities were performed to establish an average value, \bar{B}_i , and standard deviation, s_i , for each of the i analytical channels. Subsequent blank intensity measurements, B_i , could then be required to satisfy the relationship

$$\bar{B}_i - Xs_i \leq B_i \leq \bar{B}_i + Xs_i,$$

or the equivalent relationship

$$|B_i - \bar{B}_i| \leq Xs_i.$$

A reasonable value for X (to be specified by the operator) would be $X = 3$, for example.

The average values and standard deviations of the blank intensity values required for determination of the blank intensity control limits shall be performed automatically under analytical program control (Please see Section 4.9). The necessary data shall therefore be available from disk storage and shall not have to be reentered by the operator.

- 4.17 (Please provide a quotation for this item as an optional additional feature.)
The analytical program shall also have provision for optional monitoring of the measured concentrations of the analyte elements in one or more solutions designated as check standards. The purpose of this monitoring is to guarantee that the analytical performance of the complete instrument system remains within predetermined control limits throughout the entire course of an extended series of concentration determinations

for "unknown" samples. The suggested procedure for establishment of the control limits for the check standards is similar to that suggested above for blank intensity monitoring. The check standard solutions actually could be one or more of the solutions used for system calibration; the average values of the measured concentrations and their corresponding standard deviations could be determined either during the course of the calibration procedure, to save time, or they could be determined from a series of consecutive, repeat measurements at any time after calibration.

Suppose that \bar{c}_{ij} and p_{ij} are the average value and the percent relative standard deviation, respectively, of the measured concentrations of the i^{th} analyte in the j^{th} check standard solution. Concentration results, c_{ij} , for all subsequent runs of the check standards could then be expected (required) to satisfy the condition

$$|p_{ij}| \leq X p_{ij}.$$

Here, p_{ij} , defined by

$$p_{ij} = 100 * (c_{ij} - \bar{c}_{ij}) / \bar{c}_{ij}$$

is the observed difference, in percent, between the measured concentration, c_{ij} , and the previously determined (and expected) concentration result, \bar{c}_{ij} , for the i^{th} analyte in the j^{th} check standard. As before, a reasonable value for the parameter X might be $X = 3$.

As in Section 4.16, the experimental data which are required for setting of the check standard control limits shall be determined under analytical program control. The data shall therefore be available from disk storage, and shall not have to be reentered by the operator.

Standard procedure upon the occurrence of an out of limit condition shall be to print a warning message to the operator, to identify the channel(s) and analyte(s) whose measured check standard concentration(s) are out of limits, and to repeat the check standard concentration determination. If any analyte concentration is also out of limits for the repeat determination, the operator shall be instructed that some remedial action is required. The analytical program shall continue to monitor results from

additional runnings of the check standard
solution(s) for verification of the progress of
the remedial work

- 5.0 All power supplies, control circuitry, and interlocks necessary for safe operation of the above items.
- 5.1 The instrument and all components shall conform to OSHA regulations.
 - 5.2 The high-frequency power supply shall conform to Federal Communications Commission regulations.
 - 5.3 The instrument shall be fully interlocked for operational simplicity, complete operator safety and full protection of the instrument against damage.
 - 5.4 All meters, controls, control circuits and interlocks necessary for the operation, adjustment and maintenance of the instrument shall be provided.
 - 5.5 The instrument controls shall not interact with one another so that the adjustment of any control affects the proper setting of any other control, except as may be required for alignment of the instrument, and except for those controls which have a normal dependence upon one another.

- 6.0 Electrical performance, installation, hardware and software documentation, and training of customer personnel.
- 6.1 The instrument shall be fully operative over a gradual line voltage shift from 90% to 110% of the nominal voltage and over a gradual frequency deviation of plus or minus two percent from the normal frequency.
 - 6.2 The regulators shall handle voltage transients of plus or minus 10% with a recovery time of less than 100 milliseconds.
 - 6.3 The instrument shall be of sturdy construction and all necessary supporting structures shall be supplied to make the instrument suitable for floor mounting.
 - 6.4 The instrument shall be supplied with two complete sets of instruction manuals. These manuals shall cover operation, trouble-shooting, and maintenance of the instrument, and shall contain the appropriate illustrations for this purpose.
 - 6.5 Two complete sets of equipment diagrams, including circuit schematics, wiring diagrams and mechanical diagrams, shall be furnished. These shall have all points clearly identified and shall agree with each other and with the equipment as supplied.
 - 6.6 The vendor shall train the buyer's personnel in the proper operation of the instrument.
 - 6.7 The vendor shall demonstrate that the instrument, installed in the buyer's laboratory, fulfills all of the specifications in this document. Final acceptance will be made only after successful completion of these tests. Installation and final acceptance testing must be completed within one month after delivery of the instrument.
 - 6.8 The operation of the instrument shall not be affected by normal laboratory electrical noise such as that from fluorescent lights, motors, and laboratory furnaces.
 - 6.9 A complete instrument software package shall be supplied with the instrument. This package shall include two copies of the complete computer operating software system, and of all analytical program software which is required for operation

supplied with the instrument. This package shall include two copies of the complete computer operating software system, and of all analytical program software which is required for operation of the instrument as described in these specifications. It shall also contain all source language files and complete software documentation, i. e., a listing of the names, locations, and functions of all important software parameters and sections.

- 7.0 The conformance of the instrument to the above specifications must be demonstrated by the vendor, using tests which are approved by the buyer, except that the performance tests specified in this section (Section 7) shall be fulfilled.
- 7.1 Detection limits. For the purpose of these specifications, the detection limit is defined as the analyte concentration which corresponds to a net analyte signal of three times the standard deviation of the background beneath the analyte line. The standard deviation of the background is to be determined from ten consecutive measurements of the background intensity, each measurement to be for a period of no longer than 10 seconds.
- The complete analytical instrument system shall be capable of attaining simultaneous multielement detection limits which are equal to or less than the values listed in Table 1 of these specifications.
- If exceptions are taken to the specified detection limits, then the vendor shall specify detection limits that can be guaranteed for the system.
- 7.2 The slopes of analytical curves obtained from standard solutions containing zinc at five concentrations (0.03, 0.1, 0.3, 1.0, 3.0 mg/l) shall remain stable to within plus or minus 2.5 percent over an eight hour period. The standard solutions shall be run at least once each hour. A 30 minute warm-up is permissible.
- 7.3 The background under the Cr 205.55 nm line, under the same conditions as indicated in Section 7.2, shall remain constant to within plus or minus 1.5% over an eight hour period while deionized water is nebulized into the plasma.
- 7.4 The maximum permissible dark current for any photomultiplier tube shall not exceed 0.1 nA and the maximum permissible relative standard deviation of the measured dark current values shall not exceed plus or minus 3.0% of the respective average dark current values for 200 ten-second integrations.
- 7.5 The complete analytical instrument system shall exhibit freedom from interference in the Al/Ca system equivalent to that illustrated in Figure 1

of these specifications. That is, for an observation height of approximately 20 mm, the depression of the Ca line intensity for Al/Ca mole ratios of up to 100 shall not exceed five percent of the net Ca line intensity for the case in which Al is not present.

- 7.6 The complete analytical instrument system shall exhibit a freedom of interference from Na which is equivalent to that illustrated in Figure 2 of these specifications. That is, at an observation height of approximately 20 mm above the load coil, a Na/Ca mole ratio of 100 shall cause no more than a five percent change in the net intensity of the Ca line.
- 7.7 The complete analytical instrument system shall provide analytical calibration curves which, for each analytical line, shall be linear over a range of concentrations which covers four orders of magnitude, i. e., over a concentration range from X to 10,000X, where X is a "base" concentration which is of the order of 10 times the detection limit concentration. The criterion for acceptable linearity shall be that the root mean square of the relative deviations of the measured concentrations from a linear least squares fit of the concentration versus intensity data for at least five geometrically progressed concentration steps shall not exceed one percent.
- 7.8 The polychromator shall remain optically aligned over the temperature range 18-30 C. That is, the measured intensities of test emission lines from appropriate hollow cathode light sources shall not change by more than three percent over the specified temperature range.
- 7.9 For solutions in which methyl isobutyl ketone (MIBK) is the solvent material, the overall performance of the complete analytical system shall be equivalent to that specified herein for aqueous solutions.

8.0 Delivery and installation date specifications

- 8.1 Vendor must state guaranteed delivery date and a firm date for completion of installation and final performance testing.
- 8.2 The complete analytical instrument system specified herein shall constitute a fully operative simultaneous multielement analysis system. As such it shall conform to all of the specifications contained in this package. The instrument shall be installed in the buyer's laboratory by qualified personnel in the employ of the vendor and shall be fully warranted for a period of one year, beginning on the date of formal acceptance. This warranty shall cover the costs of all labor and parts, and all transportation costs for materials or personnel required for the repair of the instrument, provided only that the defects developed within the warranty period, and under normal and proper use.
- 8.3 Fulfillment of the specifications contained in this specification package shall be demonstrated on the instrument, installed in the buyer's laboratory, before final acceptance will be made.

Table 1. Element, Analysis Line, and Detection Limit Specifications for Multielement Analysis System.

PART 1		
Element	Analysis Line Wavelength(nm)	Detection Limit (ng/ml)
Ag	328.068	1.
Al	308.215	5.
As	189.042	15.
As	193.759	5.
B	208.95	1.
Ba	455.403	0.1
Be	313.042	0.05
Ca	315.887	2.
Cd	226.502	0.5
Co	231.160	2.
Cr	205.552	0.6
Cr	357.869	2.
Cu	324.75	0.5
Fe	238.204	0.5
Mg	279.806	2.
Mn	257.610	0.1
Mo	281.615	2.
Ni	231.604	2.
P	214.914	8.
Pb	220.353	4.
Pb	283.307	15.
S	180.731	0.2
Sb	206.833	3.
Se	196.026	8.
Si	251.612	2.
Sn	189.909	3.
Sr	407.771	0.05
Te	214.281	5.
Ti	334.94	0.5
Tl	190.864	5.
V	310.230	1.
Y	371.030	0.3
Zn	213.856	0.1

PART 2		
Element	Analysis Line Wavelength(nm)	Detection Limit (ng/ml)
Bi	223.068	5.
Ga	294.364	5.
Ge	199.824	10.
K	766.49	1.
Na	589.592	7.
Nb	271.662	10.
P	178.287	5.
Sc	255.237	0.4
Th	401.913	8.
U	385.958	25.
W	207.911	3.
Zr	339.198	0.7

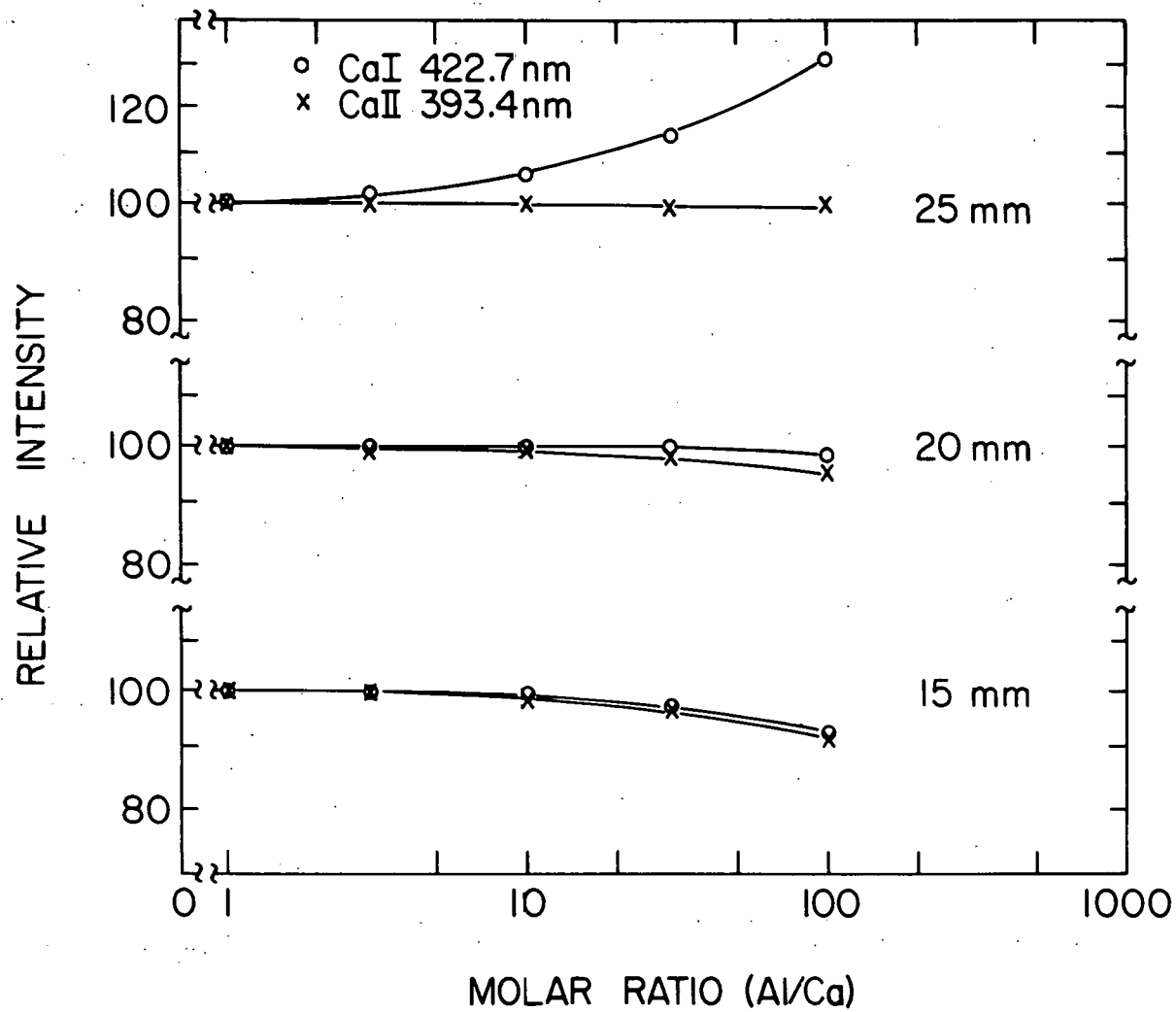


Figure 1. Effect of Al on Ca emission intensity at three heights of observation above load coil (0.5 $\mu\text{mol/ml}$ Ca)

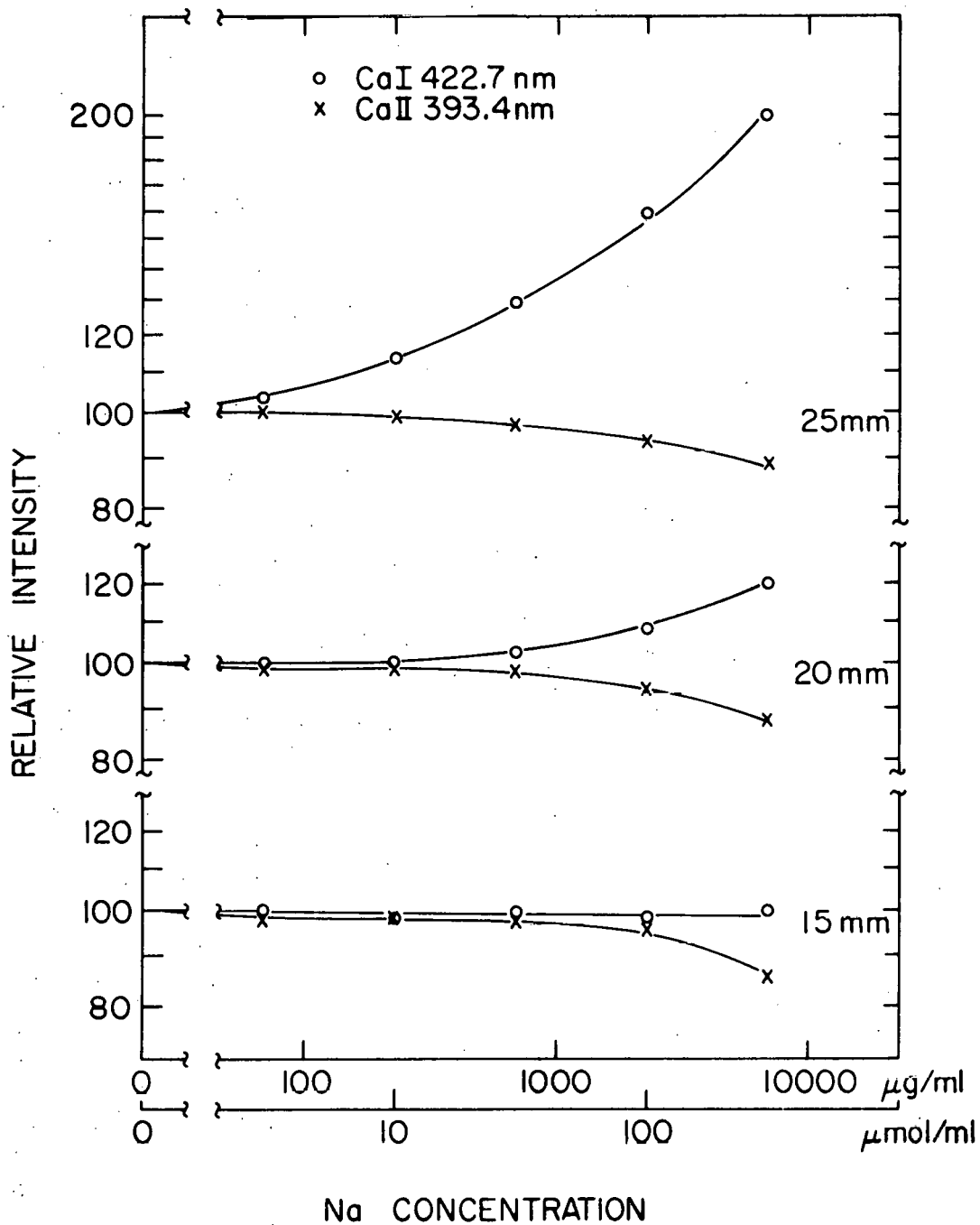


Figure 8. Effect of Na on Ca emission intensity at three heights of observation above load coil ($0.5 \mu\text{mol/ml}$ Ca)

Distribution List

Haas	10
Winge	10
Kniseley	5
Fassel	15
USDOE-TIC	27
Ames Lab. Library	13