Mars Exploration Rover (MER) Project

APXS Calibration Plan

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CHANGE LOG

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1 INTRODUCTION

1.1 Description of Instrument

The Athena Alpha Particle X-ray Spectrometer (APXS) has a set of six curium-244 alpha sources. These sources are optimized for very little spread of the energy of the emitted alpha particles. Irradiating the surface of a sample, two different physical methods for the analysis of chemical elements are applied.

A schematic view of the new APXS is shown in Figure 1.

1.1.1 X-ray Mode

The alpha particles interact with the electron shells of the atoms of the sample. Characteristic x rays are emitted when low electron orbit vacancies (in K and L shells) produced by the bombardment of alpha particles are filled by electrons from higher orbits. Mainly elements with low Z (atomic number) are excited by alpha particle. This mode is known as particles induced x-ray emission (PIXE). Also x ray emitted by the Cm-244 source, excite elements mainly with higher Z and x rays are produced (x-ray fluorescence). The x rays are measured with a Si x-ray detector.

Sample x-ray spectra taken by Mars Pathfinder APXS see Figures 2 and 3.

1.1.2 Alpha Mode

Using the almost monochromatic alpha particles of 5 MeV emitted by the special Cm-244 sources, alpha-backscattering or Rutherford scattering is applied. Alpha particles irradiate the sample to be analyzed. Those alphas scattered back by the sample nuclei close to an angle of 180° are measured by Si detectors. The spectral response of backscattered alphas is a step function with a steep slope at the step. The position of the step is diagnostic of the type of element, while the height of the step reflects the concentration of the element.

A detector thickness of about 35 μm stops completely all alpha particles. At the same time, this thickness is thin enough not to detect charged particles from cosmic-rays, because their energy deposition is very low in 35-μm thin material and is therefore below the energy threshold.

Sample alpha spectra taken by Mars Pathfinder APXS see Figures 4 to 6.

1.2 Rationale for X-Ray Calibration

The following elements are usually measured depending on their concentrations in the sample and will be calibrated:

- Na, Mg, Al, Si, P, S, Cl, K, Ca, Ti, Cr, Mn, Fe, and Ni

The concentration of the following elements are usually too low to be measured in samples from evolved planets:

- Sc, V, Co, Cu, Zn, Ga, Ge, As, Se and Br

These elements are likely to be present at levels below a few hundred PPM which is roughly an order of magnitude lower than the expected detection limit. Their detection limits will be estimated or determined provided samples with sufficiently high concentrations will be available.

Argon is found in the martian atmosphere, but not in solid surface samples.

Elements whose Z is below Na usually cannot be detected by the x-ray detector because of the thickness of its beryllium cover window of 5 μm. The attenuation of their low-energy lines is very strong. An exception might be fluorine (see section 3.23.2)

In order to minimize matrix effects of different "simple compounds" (see section 5.1), complex standards (siliceous material) are chosen to establish calibration curves (see section 6).

To obtain the required accuracy, the following set of complex standards are used.

- Certified rocks
- Validated rocks
- Validated meteorites
- Validated other samples, such as sediments, oxides, evaporites etc.

Certified and validated samples are referred to as "geostandards".

The elemental concentrations of some geostandards are listed in section 7.

Certified rocks are standard geochemical reference materials that have well-determined compositions and that are used by geochemical laboratories worldwide. Validation in this context means that two or more independent analytical methods were applied to determine the elemental composition of other chosen natural samples, and that their results agree within the errors. The validated rocks and meteorites used for the Mars Pathfinder (MPF) calibration were measured in the Mainz laboratory (MPICh), the laboratory of the Institut für Geochemie of the University of Frankfurt, Germany, and other quoted labs.

The following analytical methods were used to determine the concentration of geostandards:

Mainz:

- Instrumental Neutron Activation Analysis (INAA)
- Lecoautomat RC-412 for Carbon and H_2O
- Carbon-Sulfur-Analyzer 5003

Frankfurt:

- X-Ray Fluorescence analysis (XRFA)

For most of the measured samples, it was found that the concentrations obtained by different analytical techniques agree within the error for most of the elements. In these cases, the obtained measured concentration values were taken as “true values”.

As an additional check, some certified rock standards were also analyzed by the Frankfurt lab. Their concentrations agreed very well within the error with the certified values.

1.3 Rationale for Alpha Calibration

To fit the alpha spectra, a library of simple compound spectra are needed for each element, preferentially oxides. Oxygen is the most abundant element in rock samples. If suitable oxides cannot be found, other compounds are used.

See section 5.1 for listings of oxides and other simple compounds used.

The martian atmosphere consists of 95 % CO_2. Carbon and oxygen in the atmosphere contribute to the sample spectrum and produce interferences (see Figures 4 to 6). The C interference is especially high because of a resonance cross section of alpha scattering on C atoms. Because the atmosphere is thin for the alphas (in contrast to the sample that is infinitely thick) asymmetric peaks rather than steps are produced in the spectra.

Because the alpha mode is very important for the determination of C and O in martian rock and soil samples, atmospheric interference must be measured very precisely. Therefore, most of the simple compounds will be measured both in vacuum (no atmospheric interferences) and in CO_2 gas (about 10 mbar). By comparing vacuum and low-pressure spectra, the interference corrections can be best performed.

2 OVERVIEW

2.1 Purpose

The intent of this Calibration Plan is to define a consistent method to test and calibrate the Athena APXS instrument and to define the methods and types of tests used to validate the performance against the functional requirements outlined in the MER Project requirements documents described below. This plan also describes a sequence of calibration steps, so that calibration measurements with the flight instruments and with an identical duplicate will be clearly established. The calibration procedure according to the plan will provide fully calibrated instruments that meets the functional requirements.

2.2 Requirements

The primary goal of calibration and testing of the APXS is to verify that the instrument will meet or exceed all of the MER Project requirements relevant to x-ray and alpha spectral measurements on Mars. The relevant requirements are summarized in Tables 2.2.1 and 2.2.2.

Table 2.2.1: MER Mission Requirements relevant to APXS Calibration and Testing

Level	ID #	Requirement
2	329	The PS shall ensure that the quality of the science measurements be sufficient to satisfy the requirements and objectives in the SRD and the level 1 science requirements.
2	922	The Project System shall ensure that the quality of the calibration of the science instruments be sufficient to satisfy the requirements and objectives in the Science Requirements Document and the Level 1 science requirements.
4	28	The Alpha-Particle-X-Ray Spectrometer (APXS) shall measure the energy spectrum of backscattered alpha particles, determining the abundances of light elements, including [TBS], with detection limits [TBS].
4	29	The Alpha-Particle-X-Ray Spectrometer (APXS) shall measure the energy spectrum of emitted x-rays, determining the abundances of major and minor elements heavier than sodium, including the elements [TBS], with detection limits [TBS].
4	31	The full width at half maximum (FWHM) for the alpha mode of a 244Cm peak at 5.8 MeV shall be less than 100 keV.
4	32	The FWHM for an 55Fe x-ray source at 5.89 keV for the x-ray mode shall be less than 280 eV.
4	33	The noise level in the X-ray mode shall be less than 600 eV at temperatures below –30 degrees C.
4	34	The efficiency of the x-ray mode at the 1.24 keV line of Mg shall be at least 20%.
4	36	The APXS FOV shall be 40 mm at the contacting surface of the APXS.

Table 2.2.2.: MER Requirements relevant to APXS System Level and ATLO Testing		
Level	**ID #**	**Requirement**
3	600	The Flight System shall be capable of operation of the Mössbauer or APXS while cameras or Mini-TES are operating
3	718	The IDD shall be capable of providing the necessary level of control to actuate the APXS instrument doors.
4	55	It shall be possible to verify the nominal performance of the APXS after installation in and integration with the rover.

2.3 Scope

This plan covers all calibration-related APXS "Deliverables" as described in the MER Project Implementation Plan (JPL D-19620, MER 240-1-101).

2.4 Applicable Documents

- MER Project System Level 2 Requirements Document (JPL D-19650, MER 420-2-120)
- MER Flight System Level 3 Requirements Document (JPL D-19692, MER 420-2-401)
- MER Alpha Particle X-Ray Spectrometer Payload Level-4 Requirements Document (JPL D-19705, MER 420-2-412)
- MER Science Requirements Document (JPL D-19638; MER 420-2-128)
- APXS IICD (JPL D-20251, MER 420-3-480.c)
- MER Archive Generation, Validation, and Transfer Plan (JPL D-19658, MER 420-1-200)

2.5 Calibration Plan

2.5.1 Objectives

- To validate the design and implementation of the APXS instrument;

- To maintain the APXS Planetary Protection Control Plan's specified standards of cleanliness in all test phases and activities, including the preparation and maintenance of support facilities and equipment;

- To collect baseline data sets of engineering data in the various instrument states. These data sets shall be used during the I,T&C Program phases at JPL and elsewhere to assure correct instrument operation. These data sets shall also be used for trend analysis to detect any long-term change or degradation in instrument performance

- To monitor and document hours of use and/or cycles of limited life items, such as mechanisms and electronics;

- To implement procedures developed by the APXS calibration team to perform thorough calibration and performance verification.

2.5.2 Performance Verification

This Calibration Plan shall comply with the test requirements outlined in the MER Environmental Verification Matrix. Instrument compliance to this matrix shall be the responsibility of the Integration, Test, and Calibration Lead. Measuring and test equipment shall be used in a manner that ensures that the measurement uncertainty is known and is consistent with the required measurement capability.

Accuracy of the required measurements shall be known and appropriate equipment shall be selected to perform the measurements. The equipment shall be maintained and its placement and use shall be controlled. All measurements shall be traceable to national standards, and a log of all test equipment calibration shall be maintained.

3 CALIBRATION PLAN DETAILS

3.1 Instrument Performance and Calibration

3.1.1 X-Ray Mode

The APXS sensor head consists of detectors, electronics, and housing. Because good energy resolution is the major determinant of the quality of an x-ray spectrum, the best available x-ray detectors were selected. The silicon drift detectors, developed by the MPI Halbleiterlabor, Germany, offer high energy resolution combined with excellent count rate stability (Lechner *et al*., 1996, *Nucl. Instr. & Meth. Phys. Res*., **A 377**, 346-351). The latter is of no importance for the APXS, because the excitation source, the curium-244 alpha sources are rather weak. At –50°C, an energy resolution of 160 eV, full-width-at half-maximum measured at 6.4 eV, is obtained, while at +5°C a resolution of about 200 eV can be reached (measured with the flight electronics). These values surpass the performance of the Mars Pathfinder APXS that achieved a resolution of 260 eV (6.4 keV) at temperatures below –60°C. The Athena APXS electronics are designed to preserve the low-noise performance of the x-ray detector and to produce high-resolution spectra.

An aim of calibration is to make x-ray measurements that show at all times an energy resolution of about 160 eV, so low temperatures will normally be used. Periodic measurements under reproducible conditions will check the performance of the detector (see section 3.15). During the extended calibration (section 3.14), measurements at higher temperatures will be performed. It is mathematically possible to degrade spectra with high resolution to match those with lower resolution.

Calibration of the x-ray detector will consist of several types of measurements. Measurements of single elements or simple compounds will be performed to record the instrument response to different x-ray energies. (For a listing of the x-ray line energies produced by emission from various elements, see http://xdb.lbl.gov). Measurements of complex geostandards will be made to obtain spectra with many peaks. The peak areas will be evaluated and the relationship of peak area versus concentration will be determined for each element. From these data, element calibration curves (polynomials) will be derived, characterizing the degree of dependence over a broad range of concentration.

Samples with siliceous matrixes will be used to minimize the matrix effect of substantially different sample compositions. On Mars, siliceous matrixes are expected as the most common type of sample. However, other types of samples, such as carbonates, oxides (hematite), and evaporites, will also be measured to cover a large range of different matrixes.

3.1.2 Alpha Mode

The performance of the alpha mode depends on several factors that influence the shape of the backscattered step function: the energy spread of the emitted alpha particles, the range of the backscatter angles, and the detector performance. The limiting factors are the energy spread, the backscatter angle, and (related to these two) the source strength of the alpha source. In order to minimize the energy spread due to energy loss in the sample, the source layer must be

sufficiently thin. The source area cannot be enlarged sufficiently to compensate for the losses because a larger area would results in a wider range of backscatter angles, which deteriorate the shape (resolution) of the step spectra. To maintain a reasonable spectral shape, rather long measurements are required to compensate for the weak source strength and to obtain sufficient counting statistics.

The alpha mode will be calibrated by measuring the spectral shape of each element that might be contained in the sample. A relationship of the step height to the concentration of the element will be established. To determine the concentrations of several elements, a linear superposition must be determined that best fits the envelope of the total spectral shape of the complex sample.

The resulting measurements are rather long, as described in section 3.1.3. Because of time limitations, it may turn out that the measurement times are the limiting factor for the quality of the alpha calibration. The quality of the alpha mode calibration is also a direct function of the quality of the x-ray mode calibration, as explained in section 3.21, and the interferences of the martian atmosphere, as described in section 1.3.

3.1.3 Measurement Time Periods

The alpha mode needs much longer counting times to obtain reasonable counting statistics in the spectra compared to the x-ray mode. Because x-ray and alpha mode are measured simultaneously, the length of each measurement is mainly determined by the needs of the alpha mode.

The length of each integration for calibration measurements is selected to obtain substantially better counting statistics compared to the expected measurement times on Mars. Typical "long measurements" on Mars will last about 8 hours each. To obtain spectra of about three times better quality during calibration, the square of the improvement is required for the counting time, *i.e.* $8 \times 3^2 = 72$ hours, which are 3 days of measurements. Usually a weekend will be used for such measurements. These long measurements will be broken up into many shorter integration periods of 2 hours duration, for each of which the data will be stored separately. Having many bundles of 2-hour statistically independent spectra at the end, any counting statistics can be produced by summing the appropriate number of spectra. Such information will be useful, for example, in examining the difference in sensitivity between short "touch-and-go" spectra (time period of 1 to 2 hours) and long overnight spectra, see section 3.19).

3.1.4 Standard High-Precision Laboratory Practices

To control high-precision work in the laboratory, recurring checks of instrument performance are mandatory. For the APXS two major degradations in performance can occur:

- Degradation of energy resolution
- Degradation of efficiency

Degradation of energy resolution can be the result of increased noise level, produced either in the detector or the signal electronics chain. To assess energy resolution changes for the x-ray mode,

a reference peak is selected whose energy resolution is examined as a function of time. For the alpha mode, changes in the steepness of the step function are assessed.

Degradation of efficiency can occur if the signal strength is weakened by contamination of the exit windows of the alpha sources, the beryllium windows of the x-ray detector, or the surface area of the alpha detector. Such contamination can be causes, for example, by trace amounts of oil in the gases used to simulate the martian atmosphere. For the x-ray mode, efficiency will be assessed by examining the intensity ratio of a pair of high- and low-energy lines, knowing that a contaminating film absorbs the low-energy line more than the high-energy one. In case of alpha mode, the area of the step spectrum is examined.

A test sample (either a Ta plate or the insides of the APXS doors) will be measured periodically, typically once per week, to assess the ongoing performance of the instrument under replicate conditions. The parameters of two reference x-ray peaks (position and area) and one/two alpha edges (position and area) will be determined. The time series of these parameters will be used to demonstrate the precision of the instrument over the whole time period of the calibration. Details are described in section 3.15.

Accuracy will be tested by using known geostandards (section 3.2), which serve as "true" values. Blind samples, provided by JPL, will be measured at the end of the calibration (section 3.4) and the obtained concentrations will be compared with other analytical methods.

3.2 Compositional Range of Geostandards

Geostandards have been selected for calibration purposes that span the plausible range of compositions of martian igneous rocks. This basic set of geostandards is incorporated into the "incompressible" portion of the APXS calibration plan (see section 3.6)

A listing of the basic geostandard set is provided in section 7. The oxide or elemental concentrations for each element are listed in sections 7.3 and 7.4, respectively. The values of the highest and lowest concentration, which bind the concentration span, are color coded. These samples will be measured during the "Basic Calibration" period (section 9.1). They are part of the incompressible calibration plan.

This set of basic geostandards will be expanded by adding more samples to the list that are non-igneous rocks. These samples will include sediments, carbonates, clays, iron oxyhydroxides (hematite), epsomites, and evaporites (see 3.3). Other samples may be included to further extend the obtained span of concentrations.

3.3 Measurements of Carbonates, Hematite, Epsomites, and Evaporites

To account for possible non-igneous materials of Mars, we will expand our list of geostandards. At least one sample that represents each of carbonates, hematite, epsomite, and evaporites will be included in the plan. Some of these samples will be provided by Athena co-I Dick Morris. These

non-igneous samples will be measured either at the end of the "Normal Calibration" period or during the "Extended Calibration" period (section 9.1).

3.4 Blind Samples

JPL, through instructions from Joy Crisp, has provided five blind samples for measurements with the calibrated APXS. They will be measured during the "Normal Calibration" period, and evaluation of the spectra will take place once normal calibration has been completed. The calibration curves that have been obtained will be used to derive the elemental composition of the blind samples. Error propagation will be applied. Derived compositions will then be compared to the known compositions after the blind controls have been lifted.

3.5 Sample Preparation

A major constraint on all APXS measurements is the very shallow penetration depth of the exciting radiation: alpha particles penetrate a few micrometers and x rays up to ~10 μm. As a result, it is crucial that the surfaces of all samples used in the calibration process be representative of the whole sample. For some materials, particularly oxides, we can use ultra-pure sputtering targets of the sort used in various electronics manufacturing processes. Powdered samples are more complicated, as they must be homogeneously mixed, gravitational sedimentation must be minimized, and adsorption of moisture must be avoided. The latter is of particular importance because it can disturb the measurements considerably. Special care will be taken during powdered sample preparation process to minimize all these effects. Powdered sample materials will be mixed carefully (section 3.5.2), and baked out (section 3.5.3) before being transferred to the Mars simulation chamber.

3.5.1 Mars Simulation Chamber

The samples are measured in a Mars simulation chamber. In this chamber, the alpha and x-ray detectors can be cooled to temperatures of –60 to –70°C. The gas pressure in the chamber can be lowered to any value above 1 mbar or below 10^{-3} mbar, depending on the type of oil-free pump used (membrane pump or turbo-molecular pump). Applying a steady flow of gas at the required rate provides a simulated martian atmosphere. A feedback system guarantees a constant pressure over a time period of several days. Because the chamber and the gas in it are at room temperature, the pressure used is somewhat higher (9 mbar) than the 6.5 mbar pressure found on Mars, providing the appropriate number density of gas molecules.

The environment of the chamber will be adjusted to simulate martian night operations. During the night, measurement times will be long enough to obtain alpha spectra with good enough counting statistics. The main objective of using an atmosphere is to simulate the atmospheric interferences on the alpha spectra. During the day, only short-term measurements (touch-and go) will be carried out to obtain statistically acceptable x-ray spectra, but, no suitable alpha spectra.

Two types of atmospheres will be used:

- Ultra-clean CO_2 gas for most of the measurements

- Simulated martian atmosphere: CO_2 (95%); N_2 (3%), and Ar (2%) for selected measurements

The reason for using ultra-pure CO_2 gas for most measurements is that it is much less prone to oil contamination than the other relevant gases, protecting the detectors from slow hydrocarbon film buildup that could degrade their efficiency.

The CO_2-N_2-Ar atmosphere seems to be a good enough approximation of the real martian atmosphere, because already N_2 and Ar are barely detected in the alpha spectra. Because the backscatter cross section is an attribute of the nucleus, it is not affected by the speciation of the atom, *i.e.* it doesn't matter if there is CO_2, CO, O_2, or O.

In operations on Mars, there are two extreme geometric cases possible: the sensor head is in direct contact with a sample, or the sensor head is pointing at nothing but atmosphere. The former case means that a minimum-thickness layer of gas is between detector and sample, while in the latter case an infinitely thick gas sample is seen. Measurements in simulated martian atmosphere will be carried out for both cases, although no detailed Ar and N concentration calibration will be performed. However, the change of the Ar x-ray peak area as function of sample distance under the same pressure will be determined.

3.5.2 Sample Treatment

Treatment of each powdered sample begins with thorough grinding, followed by sieving through a 50-µm mesh. The sieved powder is then subjected to a long-duration mechanical mixing process before extracting an aliquot for measurements. The powdered sample is filled into the hollow of a copper plate that provides a sufficiently large surface diameter, and the surface is smoothed to avoid geometric effects. During these processes, water vapor is adsorbed by the sample grains depending on the moisture content of the air.

3.5.3 Vacuum Oven

To remove the adsorbed water before the sample measurement, the following procedure is applied. The copper plate with the sample is placed into a vacuum oven. Very gradually, the gas pressure is reduced by slowly pumping. This process must be monitored closely, because sudden gas releases of the sample can occur and powder can be spilled into the oven. When a pressure of 10^{-3} mbar is reached, a heater is switched on to heat the sample. A temperature of 110°C in vacuum is maintained for several hours to drive off all moisture from the sample. Temperature and vacuum are maintained until the Mars simulation chamber is ready for the measurement.

3.5.4 Sample Transfer

When the Mars simulation chamber is ready, the hot copper plate with the hot sample is quickly transferred from the vacuum oven into the chamber. In case some new moisture has been adsorbed on the warm sample surface during the transfer, the following procedure is applied. When the sample is properly positioned in the simulation chamber, the gas pressure is slowly reduced (to preserve the integrity of the sample surface) until about 3 mbar is reached in the

chamber. Then, a turbo pump is switched on and the chamber is pumped until a pressure below 10^{-4} mbar is reached. This ensures that almost all adsorbed water on the sample surface is removed. Afterwards, dry martian gas is filled into the chamber and a steady flow of gas is maintained. While this whole process of sample preparation is time-consuming and laborious, it has been found necessary to minimize moisture adsorption on the sample surface.

3.6 Calibration Plan Overview

The complete calibration plan consists of two major sections, called "Full Calibration" and "Extended Calibration". Full Calibration consists of the following steps:

- Baseline Characterization of the flight instruments (section 3.10)
- Cross Calibration (section 3.11): Cross calibration allows comparison of each flight sensor head with its flight spare. (The flight spare sensor heads will be delivered to ATLO initially, and subsequently will be exchanged for the fully calibrated flight units.)
- Basic Calibration (section 3.12): This follows Cross Calibration, and includes measurements of the set of basic geostandards.
- Normal Calibration (section 3.13)

Together, Baseline Characterization, Cross Calibration, and Basic Calibration comprise what we call "Incompressible Calibration", which is the minimum calibration effort required to yield an adequately calibrated (as opposed to properly calibrated) instrument.

Extended Calibration follows Full Calibration. It is performed with non-flight sensor heads, after delivery of the flight sensor heads.

An overview of the complete calibration schedule, showing the interrelationships among these various stages in the calibration process, is given in section 9.1. Details are given in section 9.2.

3.7 Calibration Unit Strategy

Ideally, we would like to perform all phases of APXS calibration with the full flight units, including sources, detectors, and electronics. However, the tight MER Project schedule and the long duration of the APXS calibration measurements dictate use of a more flexible approach that takes into account which components of the APXS instruments show significant unit-to-unit variability, and which do not. The items that vary the most from instrument to instrument are the alpha sources, due to variations in both the source manufacturing process and the thickness of the foils that cover the sources. Detectors vary less, and electronics show negligible variations.

Taking these characteristics into account, the various phases in the calibration plan will be carried out with the following components:

- Full Calibration:
    - Cross Calibration: **flight** sources plus **flight** sensor heads plus **flight** electronics
    - Basic Calibration: **flight** sources plus **flight** sensor heads plus **flight-like** electronics
    - Normal Calibration: **flight** sources plus **flight** sensor heads plus **flight-like** electronics
- Extended Calibration: **Flight** sources plus **flight-like** sensor heads plus **flight-like** electronics
- Post launch calibration: **flight-like** sources with **flight-like** lab modules

After launch, further measurements can be made as necessary with flight-like sources, sensor heads, and electronics.

3.8 Calibration Configuration

All Full Calibration measurements that occur after Cross Calibration will be carried out in the ground support equipment (GSE) configuration plus the Mars simulation chamber. That means use of:

- Flight sensor head, including first stage electronics and preamplifiers
- Exact duplicates of the main flight electronics, containing signal shaper, analog to digital converter, microprocessor, and interface. (The main flight electronics will be delivered to the rover after completion of Cross Calibration.)
- Control computer (PC) to command the instrument via the interface simulating avionics of the rover.

The GSE configuration will be as close to the real flight configuration as possible.

3.9 Turnaround Times of Sample Measurements

The turnaround times of sample measurements are governed by the counting statistics that must be obtained, and also by the lead time, *i.e*. time that is required between sample insertion into the chamber and the start of the measurement. Lead time is a compromise between the need to pump slowly enough not to disperse powdered sample material in the chamber and the wish to minimize the pumping time. After a good vacuum is reached, a low-pressure simulated martian atmosphere is established by a controlled gas flow mechanism. Because of considerable lead times before and after the measurement, it is not wise to make measurements of powder samples shorter than half a day, preferentially a whole day.

The turnaround time for the shorter measurements (0.5, 1 or 2 days) takes place from Monday to Thursday, while the turnaround time for the longer measurements (3 days for geostandards) is over the weekend (Friday to Sunday). Start of the turnaround time is the morning of the starting day, while stop is the next morning after the ending day, so “Friday to Sunday” means Friday late morning to Monday early morning.

The dimensions of available chambers place practical limits on the number of instruments that can be calibrated per chamber, and on the numbers of samples that can be placed in a chamber. These practical limits are offset somewhat from a personnel standpoint by the fact that long-duration measurements can be run largely unattended.

The calibration schedule is structured such that the starting day can be any Monday. It is unrealistic to assume that such an extensive measurement schedule will be carried out without any interruptions either by breakdown of ground support equipment or absence of personnel due to meetings, *etc*. Time margins are included in the schedule.

3.10 Baseline Characterization of Flight Instruments

Baseline Characterization is a subset of the Cross Calibration, and provides the response of the detectors to incoming radiation. The former term was proposed by the calibration review committee. In the project, the latter term Cross Calibration was used in a somewhat broader sense. To be in line with both recommendations, both terms are presented, here.

During the Baseline Characterization, samples that provide only one major K_α and K_β x-ray line (originating from one element) are measured in vacuum. At the same time, the spectral shape of alpha step-spectra is measured for very simple compounds (mostly oxides). Vacuum is used at this stage to avoid gas interference.

The following elements will be measured (a listing of simple samples can be found in section 5.3.):

- C, Na, Mg, Al, Si, Ca, Ti, Fe, and Ta

These spectra will be used to characterize the spectral shape of the most important x-ray lines and alpha steps.

3.11 Cross Calibration

Cross Calibration is a procedure to characterize the response functions of two or three sensor heads. In addition, several geostandards will be measured. The measured data will allow us to compare the spectra from the two flight units, the flight spare, and a duplicate laboratory unit. To achieve this within the given time, two separate full-functional simulation chambers with ground support equipment will be available, so that parallel measurements of two units can be carried out.

One flight spare instrument will be cross calibrated in chamber one with flight alpha source set one first, because it has to be delivered first. Somewhat later, flight instrument two will begin Cross Calibration with flight sources two in chamber two. When the flight spare is delivered, flight instrument one with the same sources will undergo Cross Calibration in chamber one. Flight electronics can be used until these have to be delivered, too. Then, flight-like electronics that are almost identical to the flight electronics will be used. After the delivery of the flight

sensor heads, an exact duplicate of the sensor head (flight-like model) will be used to continue with flight source set one, and flight-like electronics.

Cross calibration is needed if, for example, two x-ray spectra measured by two different units must be compared. One can degrade the one spectrum with higher energy resolution to match the one with lower resolution. Or for the measurements of the same sample by two different units, the conversion factors for the efficiency as function of energy will be determined. In general a set of parameters, such as energy resolution, efficiency, alpha source strength, and alpha energy spread, will be determined under identical conditions.

Measurements with single element and simple compound samples are performed in vacuum, undisturbed by atmospheric interferences. Three geostandards are also measured to derive a basic set of calibration curves. The effects of the atmosphere on the spectral shapes, especially the alpha spectra, will be studied in a subsequent calibration phases. All measurements are made at a single constant temperature.

3.12 Basic Calibration

Basic Calibration will be done mainly in atmosphere to simulate martian conditions. All single element and simple compound samples that are necessary for the calibration will be measured sufficiently long that the alpha spectra can be used for data evaluation. (For details see section 9.2.) However, their counting statistics will not yet be optimal (section 3.1.3).

The basic set of geostandards will be measured to derive calibration curves (section 3.17). The counting times are long enough to achieve good counting statistics for the alpha spectra.

A copy of the rovers' magnetic targets will be measured to determine the elemental concentration signature of the clean target. This will ensure proper identification of wind-blown dust that adheres to the magnet on the martian surface. If appropriate a more comprehensive study of magnets with adherent dust can be done during the Extended Calibration.

Cross Calibration and Basic Calibration compose the Incompressible Calibration that is essential to consider the instrument basically calibrated. If schedule pressures were to force delivery of the instrument at this point in the process, adequate APXS measurements could be performed on Mars. (The calibration plan is structured the way it is to allow for this eventuality.) However, the calibration can be improved by adding more valuable measurements, which is described in the Normal Calibration (section 3.13)

3.13 Normal Calibration

For the x-ray mode, the counting statistics obtained during Basic Calibration are sufficient for most purposes. For the alpha mode, however, this may not be true. Measurements during Normal Calibration will rectify this situation. Certain samples, such as the simple samples, will be measured for a second time to improve the counting statistics of the spectra. Re-measurement of the simple samples also provides duplicate alpha and x-ray spectra measured after some time period had elapsed to check the reproducibility of the instrument, details see section 9.2.

During Normal Calibration, the following samples will be measured:

- Additional geostandards
- Rocks provides by Dick Morris for round-robin studies
- Blind samples provided by JPL
- Cal target
- Simple samples

The rock samples to be provided by Dick Morris will be measured by all of the Athena instruments. These replicate measurements will be assessed and the obtained measurement errors compared. The round-robin exercise is a preparation of the type of investigations to be carried out on Mars.

3.14 Extended calibration

During the Extended Calibration, flight sources and flight-like sensor heads and electronics will be used to study the effect of various parameters, such as distance, temperature, pressure, and to measure additional samples to address various issues. Measurements will be made of some exotic samples, like the ones mentioned in section 3.3.

The Extended Calibration plan is largely open at the present, so that it can be used to deal with questions that have arisen during the Full Calibration. A Ph.D. student in Mars studies at the Max-Planck-Institut for Chemistry in Mainz will participate in APXS calibration, and his or her ideas will be incorporated into the plan for Extended Calibration based on the results obtained during the Full Calibration.

3.15 Monitoring

Once per week (usually on Monday) a monitor sample will be measured to provide a time baseline of instrument performance. In the past, a metal plate of Ta was used providing two diagnostics lines: Ta L_α line and Ta M_α line. For the Athena instrument, the inner side of the doors will be used as calibration target. The basic CuBe alloy door surface will be partly covered with additional elements. The same doors will also be used as the monitor target.

The following elements will be available for monitoring: Al, Ni, Cu, and Au. They provide suitable low and high energy x-ray lines and low and high energy alpha edges.

Some important parameters will be monitored as function of time:

3.15.1 X-Ray Mode

- Peak position of at least two x-ray lines (low and high energy) → energy calibration → check of overall gain stability
- Peak area of two x-ray lines (low and high energy) → check of contamination of x-ray detector entrance window

- Energy resolution → performance of detector and electronics

3.15.2 Alpha Mode

- Height of step and area of two elements (low and high energy) → check of contamination of alpha-ray detector surface and alpha source surface
- Position of two alpha edges (low and high energy) → performance of detectors and electronics

3.16 Spectrum Evaluation

Spectrum evaluation is an important step for the production of higher-order data products. The shapes of x-ray and alpha spectra are substantially different from one another. For both types of spectra, spectrum evaluation codes have been developed and were successfully used for the Mars Pathfinder APXS spectra. The improvements incorporated in the Athena APXS design will permit use of improved codes, to be optimized once we have started the detailed calibration measurements and have new sets of spectra obtained under flight like conditions.

3.16.1 X-Ray Mode

Codes that are suitable for x-ray spectrum fitting produce:

- Peak positions
- Peak areas
- Energy resolution

Codes to be used are:

- "XRGANY" (x-ray derivative of GANYMED gamma-ray peak-fitting code developed in Mainz, proprietary)
- "ALP" (developed in Mainz)

3.16.2 Alpha Mode

A library of alpha spectra (simple compounds) is used to fit the measured spectra and to determine step areas. The code used is "ALPHA", developed in Mainz.

All Mainz-developed codes (x-ray and alpha) are written in FORTRAN 77 for MS-DOS v. 3.5 or higher.

3.17 Calibration curves

The known concentrations of each element for different samples are plotted versus measured peak areas. The fit of the data points is called a "calibration curve". In most cases, the curve is a straight line that can be extrapolated close by the origin of the coordinate system. If the curve is very close by the origin, the fit of the straight line can be forced to pass through the origin expressing the fact that an element with zero concentration produces no signal. These curves cannot be extrapolated to higher-than-measured concentrations, because non-linearities will occur. The siliceous composition of the geostandards (about 40 to 50 weight percent oxygen) and

the excitation of alpha particles and x ray are the reason for non-matrix effects for most of the calibration curves, as demonstrated by Mars Pathfinder APXS measurements (Figures 7 to 10). However for Si, the matrix effect is strong enough to require a calibration curve that is a parabola forced to go through the origin.

3.18 Accuracy of Calibration

Measurement accuracy will be derived using the certified and validated geostandards. The spread of the different analytical methods used and the replicate measurements obtained are given in section 7.2. The very good agreement of different analysis methods can be recognized clearly. The preferred values of the oxide and elemental concentrations for each element are listed in 7.3 and 7.4, respectively.

The basic geostandards provide a set of 'true elemental concentrations' and are the most important source of information for determining the accuracy of APXS measurements.

3.19 Measurement Repeatability

As described in section 3.1.3, two-hour accumulated spectra are saved in memory of the instrument, the spectral content of the multi-channel memory unit is cleared, and a new integration is started. From this data base, spectra with multiple counting times can be obtained by summing appropriately. Using data collected in this manner, the repeatability of the instrument's measurements will be investigated. The variation of certain parameters and spectra measured repeatedly over a period of three days will reveal the short-term stability of the instrument. Because the samples will not be touched during these measurements, this will provide a measure of the stability of the detectors and electronics.

The weekly monitor target measurements (section 3.15) will document any long-term drift of parameters over the whole calibration, including detector, electronics, and mechanical structure (distance between sensor head and sample surface inside the simulation chamber). In addition, replicate measurements obtained at several different points in the calibration plan for several powdered and solid samples (section 3.13) will provide a further measure of repeatability over long time baselines.

3.20 Sources of Calibration Errors

There are several potential sources of calibration errors. First, the accuracy of the analysis can be biased by a systematic errors. To minimize systematic errors, certified and validated geostandards are measured and calibration curves are established. These curves provide the foundation of accuracy. Continuous checks of precision will then be used to assure that the obtained accuracy is maintained over time.

The areas of x-ray peaks or alpha steps have a counting error that is usually rather small for laboratory measurements (less than 2 %). The calibration curve parameters have an error according to the quality of the data fitting. Proper error propagation will be carried in all

calculations and will be factored properly into the final error of elemental concentrations. The propagated errors will be assessed with measurements of samples that are treated as unknowns, but whose composition is known.

3.21 Calibration Procedure for the Alpha Mode

Alpha measurements are fitted by single-element library spectra. Due to the limited energy resolution of the alpha particles and the resulting edges, elements must be grouped together because they cannot be resolved in the spectra:

- C plateau and edge (if present)
- O plateau and edge
- Si group (from Na to Cl)
- Ca group (from K to Ti)
- Fe group (from Cr to Ni)

The main emphasis of the analysis is on C and O (because the other elements are determined much better by the x-ray mode). The ratios of the elements within the groups must be taken from the x-ray mode, and are used as input for the alpha spectrum fitting. The heights of the groups are fitted by the properly-composed library spectra groups.

Usually, the alpha group ratios agree within their statistical errors with the x-ray derived group ratios. A larger deviation between the two methods would indicate that the backscattered alphas see a different composition compared to the emitted x ray, an effect that would be largest for Fe (excited by high-energy x ray and emitting 6.4 keV x rays). One possible explanation of such an effect could be a depth concentration profile.

The alpha results are compared with the known concentrations of geostandards.

3.22 Effect of Distance on Alpha Spectra

All alpha calibration measurements are normally carried out using one common distance. If the distance for a sample of unknown shape varies by no more than 1 to 2 mm, the concentration can be normalized to 100 %. However, for rough rock surfaces we can expect distance variations greater than this. Because of the martian atmosphere, increased distances cause the alpha particles to loose energy relative to the nominal distance. Backscatter cross sections of carbon and oxygen are energy dependent, so the spectral shape of oxygen and carbon step functions may change as function of distance. Therefore, we will make measurements of oxygen compound and carbon samples to study the magnitude of the effect.

These measurements will be performed in artificial martian atmosphere as function of distance. A corrected set of O and C library alpha spectra may have to be established based on the size of the distance effect or a correction method will have to be introduced that takes care of the effect.

3.23 Minimum detection limits

One common definition of detection limit for an x-ray line is that the error of the peak area is 50 %. The error takes the height of the background into consideration. From the knowledge of the general background in the spectrum at the peak position, a peak area with an error of 50 % can be calculated and the detection limit determined using the calibration curve. Such a detection limit is somewhat independent of the measurement time, as long as the time is not too short. The reason is that a known peak area (counts per unit time) is measured long enough and then scaled down according to the background. However, to measure a very low concentration requires long counting times in order to get sufficient counting statistics.

The detection limit will be determined for all elements that are contained in geostandards and are detectable by the instrument in a given timeframe. For some trace elements (listed in section 1.2) neighboring elements may be used to estimate their detection limit, because none of the geostandards may have them in a high enough concentration to obtain a simple two-point calibration curve.

3.23.1 Minimum Detection Limits for Carbon and Chlorine

The detection limit of carbon is based on a similar definition as for the x-ray peaks: the peak area of the C step area should not exceed an error of 50 %. However, to estimate such an area is somewhat more elaborate compared to x-ray mode. One needs two or three samples with different C concentrations (all above the probable detection limit). Fitting of all element groups including C will yield C areas with errors according to the C concentrations. Extrapolation to areas with just an error of 50 percent will provide an estimation of the detection limit. For carbon, carbonaceous meteorites and TBD terrestrial samples will be used. For chlorine, geostandards and TBD terrestrial samples will be measured.

3.23.2 Detection Sensitivity for Bromine and Fluorine

The energy of the bromine x-ray line is 11.9 keV. The upper energy threshold of the instrument will be around 15 keV. However, there is an increased background in the high-energy part of the spectrum. This background results from scattered x ray of the alpha sources and increases the detection limit of all high-energy lines. The Br detection limit will be determined during the extended calibration.

The energy of the fluorine x-ray line is at 0.67 keV. The lower threshold of the instrument will be between 0.4 and 0.6 keV depending on the low-energy noise component. The x-ray detector is covered by a 5 µm thin window, which absorbs most of the radiation below 1 keV. Any contamination of the window will increase absorption of lines below 1 keV in an unknown amount. Therefore, the presence of fluorine may be detected, but the derived concentration will have an unpredictable error. The F detection limit will be estimated during the extended calibration.

3.23.3 Detection limits and schedule

Because no detection limits can be estimated *a priori*, sample measurements during the full calibration will show if concentration ranges are sufficient. If not, additional samples will be acquired and measured during the Extended Calibration. In most cases, rough estimates should be available after the Full Calibration.

4 CALIBRATION AND CHECKOUT AFTER LAUNCH

4.1 Cruise Health Check

The inner surfaces of the APXS sensor head doors serve as a calibration reference for the instrument during surface operations. The doors will be open during cruise, so calibration measurements cannot be made during this period. However, brief instrument health checks will be performed. The electronics will be switched on and some commands will be executed to check the functionality of the microprocessor. Because the APXS sensor head will be pointing into vacuum during cruise, no meaningful spectra can be accumulated. Only noise spectra can be measured, which do not reveal the performance of the detectors. Therefore, the cruise health check is limited to verifying proper performance of the electronics.

4.2 Post-Landing Calibration

The calibration reference surfaces on the APXS doors are covered with several elements (Al, Ni, Cu, and Au) to provide low- and high-energy x-ray lines and alpha steps in the spectra. In order to properly sustain launch and landing loads, the doors must be held open from launch to landing. As a safety precaution, the APXS doors will not be closed for the first time until at least one measurement has been obtained on martian surface materials. Therefore, post-launch observations of the APXS calibration reference will not be made until after the first measurements of martian surface materials have taken place.

In order to provide some basic calibration information shortly after landing, two observations can be made prior to the first surface measurement. One would be simply to observe the martian atmosphere. The other would be to observe the Compositional Calibration Target (CCT). The CCT is intended primarily for Mössbauer calibration, and has a composition that is nearly pure magnetite. Because they require use of the IDD, measurements of the CCT would have to be performed after egress. Measurements of air could be performed while the rover is still on the lander. These early measurements should be carried out during the night to test the energy resolution of the x-ray detector, and should last several hours (minimum 5 hours, preferentially 10 hours) to test the performance of the alpha detectors.

Once the first measurement has been made on martian surface materials, the doors can be closed and the first calibration reference measurement can be performed. Such measurements will be made periodically over the course of the surface mission to monitor the performance of x-ray and alpha detectors. After the first calibration reference measurement has been made, the doors will normally be kept closed except when the instrument is in use on a surface target. The doors must always be closed during RAT operations.

5 SIMPLE COMPOUNDS

5.1 Listings of Simple Compounds

Note: Times with plus sign indicate that these measurements will be carried out at different time periods of the schedule.

	Elt.	Simple Standards	Measurement Times [d]	Pressure	Type & Size [mm x mm]
	Al	Al	1	vacuum	plate
		Al	1	9 mbar	plate
		Al2O3	1	vacuum	sputter
		Al2O3	2	9 mbar	sputter
	Be	Be	1	vacuum	plate 60x60
		Be	1	9 mbar	plate 60x60
		Be	2+3	at diff. pressures	plate 60x60
	C	C	5	vacuum	plate 60x60 & 100x100
		C	4	9 mbar	plate 60x60 & 100x100
	Ca	Ca3(PO4)2	1	9 mbar	powder
		CaCO3	3	vacuum	powder
		CaCO3	2+2	9 mbar	powder
		CaO	1+2	vacuum	powder
		CaO	3+1	9 mbar	powder
	Cl	NaCl	1	vacuum	powder
		NaCl	1+2	9 mbar	powder
	Cr	Cr	1+1	9 mbar	plate diam. 50
	Fe	Fe	1+2	vacuum	plate 100x100
		Fe	2	9 mbar	plate 100x100
		Fe2O3	1+2	vacuum	powder
		Fe2O3	2+2	9 mbar	powder
		Fe2O3	2	9 mbar & diff. geo	powder
	K	K2CO3	2+2	9 mbar	powder
	Mg	MgO	1	vacuum	sputter
		MgO	2+2	9 mbar	sputter
		MgO	1	9 mbar	powder
		MgSO4	2+2	9 mbar	powder

	Mn	MnO	1	9 mbar	powder
	Na	Na2CO3	2+1	9 mbar	powder
		NaCl	1	vacuum	powder
		NaCl	1+2	9 mbar	powder
	Ni	Ni	1	9 mbar	plate 60x60 & 100x100
	O	calculated			
	P	Ca3(PO4)2	1	9 mbar	powder
	S	MgSO4	2+2	9 mbar	powder
	Si	Si	1	vacuum	plate 60x60
		Si	1	9 mbar	plate 60x60
		SiO	1+2	vacuum	sputter
		SiO	3+1	9 mbar	sputter
		SiO2	1+3	vacuum	sputter
		SiO2	2+2	9 mbar	sputter
		SiO2	3	9 mbar & diff. geo.	sputter
		SiO2	3	9 mbar	powder
	Ta	Ta	5	vacuum	plate
		Ta	5	9 mbar	pla+F11te
	Ti	Ti	1	vacuum	plate 60x60 & 100x100
		Ti	1	9 mbar	plate 60x60 & 100x100
		TiO2	1	vacuum	powder
		TiO2	2+1	9 mbar	powder
	V	V	1	9 mbar	plate

File: Calibration_standards_times_ath_2.xls\Simple compounds

5.2 Derived Standards for Alpha Mode

Alpha mode			
	CO_2	CaCO3	CaCO3-CaO=CO2
		MgCO3	MgCO3-MgO=CO2
		MnCO3	MnCO3-MnO=CO2
	Na	Na2CO3	Na2CO3-CO2=Na2O
	O	SiO2	SiO2-SiO=O

5.3 Overview of Simple Compounds and Their Relationship to the Plan

Elt	Simple standards	Vacuum					Gas					Gas diff. mbar					Geom. diff.			
Be	Be metal			o			o					o	o	o	o	o				
C	C	o	o	o	o	o	o	o	o	o										
O	*calculated*																			
Na	Na2CO3						o	o	o											
Na	NaCl	o					o		o	o										
Mg	MgO powder								o											
Mg	MgO sputter	o					o	o	o	o										
Mg	MgSO4						o	o	o	o										
Al	Al plate	o					o													
Al	Al2O3 sputter	o					o	o	o	o										
Si	Si plate	o							o											
Si	SiO2 powder						o	o	o											
Si	SiO2 sputter	o		o	o		o	o	o	o							o	o	o	
Si	SiO sputter	o		o	o		o	o	o	o										
P	Ca3(PO4)2						o													
S	MgSO4						o	o	o	o										
Cl	NaCl	o					o		o	o										
K	K2CO3						o	o	o	o										
Ca	CaO	o		o	o		o	o	o	o										
Ca	CaCO3			o	o	o	o	o	o	o										
Ti	Ti plate	o					o													
Ti	TiO2	o					o	o	o											
V	V me						o													
Cr	Cr me						o													
Mn	MnO						o													
Fe	Fe plate	o		o	o		o		o											
Fe	Fe2O3	o		o	o		o	o	o								o	o		
Ni	Ni plate								o											
Ta	Ta plate	o	o	o	o	o	o	o	o	o	o									

Key:

To be done	Done
O Cross calibration	X Cross calibration
O Basic calibration	X Basic calibration
O Normal calibration	X Normal calibration

1 symbol corresponds to 1 day

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6 GEOSTANDARDS AND ROCK SAMPLES

6.1 Basic Geostandards

		Geostan-dards, meteorites & rocks	Measure-ment time [d]	Pressure	Type
		Allende	3	9 mbar	meteorite powder
		AN-G	3	9 mbar	geostandard powder
		BE-N	3	9 mbar	geostandard powder
		I555	3	9 mbar	andesite powder
		I555	TBD	measurements at different distances	
		I555	TBD	measurements at different pressures	
		I555	2	vacuum	andesite plate
		I555	3	9 mbar	andesite plate
		Mica-Fe	3	9 mbar	geostandard powder
		Millbillillie	3	9 mbar	meteorite powder
		Murchison	3	9 mbar	meteorite powder
		SSK1.1	1	vacuum	andesite powder
		SSK1.1	3	9 mbar	andesite powder

6.2 Additional Geostandards

Geostandards, meteorites & rocks	Measure-ment time [d]	Pressure	Type
"Morris rock 1"	3	9 mbar	TBD
"Morris rock 2"	3	9 mbar	TBD
"Morris rock 3"	3	9 mbar	TBD
"Morris rock 4"	3	9 mbar	TBD

6.3 Overview of Rock Samples and Their Relationship to the Plan

Rock standard	Vacuum	Gas	Gas
Allende		o	
AN-G	o	o	
BE-N	o	o	
Mica-Fe		o	
Millbillillie		o	
Murchison		o	
SSK1.1	o	o	
I555 powder		o	
I555 plate			o
Cal Target	o	o	o
Magnet		o	
Morris Rock-1			o
Morris Rock-2			o
Morris Rock-3			o
Morris Rock-4			o
Blind-1			o
Blind-2			o
Blind-3			o
Blind-4			o
Blind-5			o

To be done		Done	
o	Cross calibration	x	Cross calibration
o	Basic calibration	x	Basic calibration
o	Normal calibration	x	Normal calibration

7 ELEMENT CONCENTRATIONS OF GEOSTANDARDS

Concentrations are given in weight %.
For each element, the highest (red) and lowest (blue) concentration values are marked by color.
In Table 7.2 results from several analytical techniques are given. The selected used value is marked with gree color.
Note: sometimes only the lowest value above the detection limit is indicated.

Keys:

H2O+	Combined (structural) water, measured above 110ºC
H2O-	Moisture (lost by drying at 105 – 110ºC)
Fe2O3T	Total iron calculated as Fe_2O_3
LOI	Loss on Ignition (usually at 1000ºC)

Color key for tables:

Used value	Lowest value	Highest value

7.1 References of Basic Geostandards

$	Geostandards: Geostandards Newsletters
#	Allende & Murch.: Jarosevitch (1990), Meteoritics 25,323; Millbi.: Mason et al., (1979), Smithson. Earth Sci. 22, 27-45
@	SSK1.1: Pearce et al. 1995, J Petr. 36,1073
*	Analysis made in Mainz or Frankfurt, Germany
INAA	Instrumental Neutron Activation Analysis, MPICh Mainz
XRF	X-Ray Fluorescence, Univ of Frankfurt, Germany
RC	Lecoautomat RC-412 for C & H_2O, MPICh Mainz
CSA	C-S-Analyzer 5003, MPICh Mainz

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7.2 Comparison of Analytical Methods for Oxide Concentration of Geostandards

Sample	SiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	SO_3	Cl	Cr	Ni	Zn
Allende#*	34.2	3.27		30.68	0.18	24.62	2.61	0.45	0.03	0.15	0.23	3.70		0.356		0.011
INAA		3.4		29.8	0.17	25.36	2.80	0.42	0.035	0.18			0.023	0.3503	1.42	0.012
XRF																
RC/CSA												5.16				
AN-G$	46.3	29.8	0.87	2.24	0.04	1.8	15.9	1.63	0.13	0.22	0.01	0.035		0.005	0.0035	
INAA			3.36		0.045		16.7	1.65	0.124					0.005		
XRF	45.81	29.85	3.30		0.045	1.79	15.87	1.59	0.13	0.22	0.01			0.005		
RC/CSA												0.033				
BE-N$	38.2	10.07	5.34	6.74	0.2	13.15	13.87	3.18	1.39	2.61	1.05	0.075		0.0360	0.0026	
INAA			12.67		0.199		13.59	3.13	1.33					0.0363	0.0029	0.012
XRF	38.21	10.12	12.68		0.20	13.15	13.90	3.14	1.40	2.66	1.06			0.0355		
RC/CSA												0.080				
Mica-Fe$	34.4	19.50	4.61	18.91	0.35	4.55	0.43	0.30	8.75	2.50	0.45	0.017		0.0090	0.0035	0.13
INAA																
XRF	34.23	19.26	25.52		0.35	4.57	0.39	0.22	8.78	2.49	0.40			0.0077		
RC/CSA												0.019				
Millbillillie#	52	12.8		18.3	0.61	6.76	10.2	0.36								
INAA	(49.2)	12.85		18.62	0.53	6.40	10.00	0.41	0.042	0.65				0.227	< 0.001	< 0.003
XRF	47.92	12.61		18.88	0.56	6.50	10.32	0.35	0.040	0.65	0.05					
RC/CSA												0.639				
Murchison#*	29.07	2.15		28.47	0.20	19.94	1.89	0.24	0.04	0.13	0.23	6.60		0.330		0.018
INAA		2.29		27.33	0.20	20.62	1.83	0.23	0.037	0.13			0.018	0.302	1.30	0.02
RC/CSA												7.66				
SSK1.1@*	60.08	13.94	11.93		0.19	1.98	5.92	3.87	1.13	1.17	0.22			9.0E-04		
INAA		14.6	11.48		0.19	2.30	5.86	3.6	1.10	1.17			0.148	1.5E-03		
XRF	60.5	14.11	11.94		0.20	1.96	6.12	3.57	1.13	1.16	0.22			1.1E-03		0.012
RC/CSA												0.008				
l555*																
INAA																
XRF	58.92	13.91	10.04		0.23	2.69	6.29	3.31	0.44	0.90	0.12					
RC/CSA																

Sample	H_2O+	H_2O-	CO_2	H_2O_{tot}	Fe_2O_3T	LOI
Allende#*			1.06			
INAA					34.6	
XRF						
RC/CSA	0.43	0.08	1.06	0.51		
AN-G$	0.61	0.11	0.13	0.72	3.36	0.65
INAA					3.36	
XRF					3.30	0.59
RC/CSA	0.65	0.087	0.2	0.74		
BE-N$	2.24	0.5	0.74	2.74	12.84	2.45
INAA					12.67	
XRF						2.27
RC/CSA	2.01	0.41	1.22	2.42		
Mica-Fe$	2.91	0.43	0.19	2.40	25.65	2.00
XRF					23.52	2.02
RC/CSA	3.49	0.32	0.35	3.81		
Millbillillie#						
INAA					20.69	
XRF						
RC/CSA	0.19	0.05	0.09	0.24		
Murchison#*	8.95	1.14	6.78			
INAA					30.37	
RC/CSA	9.32	2.08	6.26	11.4		
SSK1.1@*			0.07		11.93	
INAA					11.48	
XRF					11.94	-0.42
RC/CSA	0.56	0.05	0.082	0.61		
I555*						
INAA						
XRF			0.083		10.04	3.54
RC/CSA						

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7.3 Preferred Oxide Concentration Values for Geostandards

Probe	SiO_2	Al_2O_3	Fe_2O_3	FeO	MnO	MgO	CaO	Na_2O	K_2O	TiO_2	P_2O_5	SO_3	Cl	Cr	Ni	Zn
Allende#*	34.20	3.40		29.80	0.17	25.36	2.80	0.42	0.04	0.18		5.16	0.023	0.350	1.42	0.012
AN-G$	45.81	29.85	3.30		0.05	1.79	15.87	1.59	0.13	0.22	0.01	0.033		0	0.004	
BE-N$	38.21	10.12	12.68		0.20	13.15	13.90	3.14	1.40	2.66	1.06	0.080		0	0.003	0.012
Mica-Fe$	34.23	19.26	25.52		0.35	4.57	0.39	0.22	8.78	2.49	0.40	0.019	0.05	0.077	0.004	0.13
Millbillillie*	47.92	12.61		18.88	0.56	6.50	10.32	0.35	0.04	0.65	0.05	0.639				
Murchison#*	29.07	2.29		27.33	0.20	20.62	1.83	0.23	0.04	0.13	0.23	7.66	0.018	0.302	1.3	0.02
SSK1.1@*	60.50	14.11	11.94		0.20	1.96	6.12	3.57	1.13	1.16	0.22	0.008	0.148	0.001		0.012
I555*	58.92	13.91	10.04		0.23	2.69	6.29	3.31	0.44	0.90	0.12					

Probe	H_2O+	H_2O-	CO_2	H_2O-tot	LOI	Sum
Allende#*			1.06	0.51		104.4
AN-G$	0.65	0.087	0.2	0.74	0.59	99.6
BE-N$	2.01	0.41	1.22	2.42	2.27	100.3
Mica-Fe$	3.49	0.32	0.35	3.81	2.02	100.6
Millbillillie*	0.19	0.05	0.09	0.24		98.8
Murchison#*	9.32	2.08	6.26	11.4		108.9
SSK1.1@*	0.56	0.05	0.082	0.61	-0.42	101.8
I555*			0.083		3.54	96.9

File: Lit_geostandards_ath_1.xls\Geo_Stnds_ox_3

7.4 Preferred Elemental Concentration Values for Geostandards

Sample	Si	Al	Fe	Mn	Mg	Ca	Na	K	Ti	P	S	Cl	Cr	Ni	Zn	C
Allende#*	15.99	1.80	23.16	0.13	15.30	2.00	0.31	0.03	0.11	0.00	2.07	0.02	0.35	1.42	0.012	0.29
AN-G$	21.42	15.80	2.31	0.03	1.08	11.34	1.18	0.11	0.13	0.00	0.01	0.00	0	0.0035		0.05
BE-N$	17.86	5.36	8.87	0.15	7.93	9.93	2.33	1.16	1.59	0.46	0.03	0.00	0	0.0029	0.012	0.33
Mica-Fe$	16.00	10.19	17.85	0.27	2.76	0.28	0.16	7.29	1.49	0.17	0.01	0.05	0.008	0.0035	0.13	0.10
Millbillillie*	22.40	6.67	14.68	0.43	3.92	7.38	0.26	0.03	0.39	0.02	0.26	0.00				0.02
Murchison#*	13.59	1.21	21.24	0.16	12.44	1.31	0.17	0.0307	0.08	0.10	3.07	0.02	0.302	1.3	0.02	1.71
SSK1.1@*	28.28	7.47	8.35	0.15	1.18	4.37	2.65	0.94	0.70	0.10	0.00	0.15	0.001		0.012	0.02
I555*	27.55	7.36	7.02	0.18	1.62	4.50	2.46	0.37	0.54	0.052						0.023

File: Lit_geostandards_ath_1.xls\Geo_Standards_elt

8 FIGURES

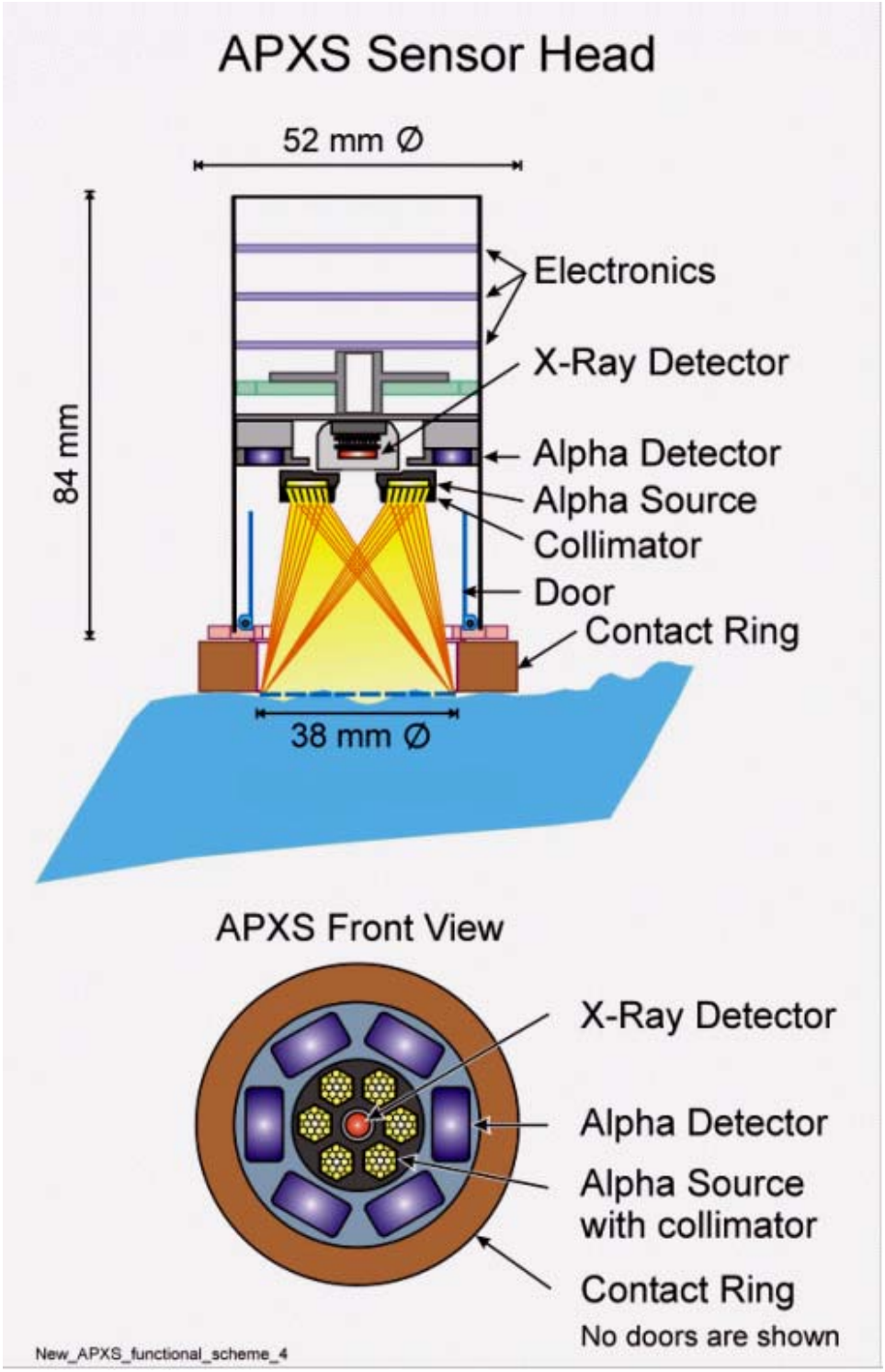


Figure 1 Schematic diagram of Athena APXS

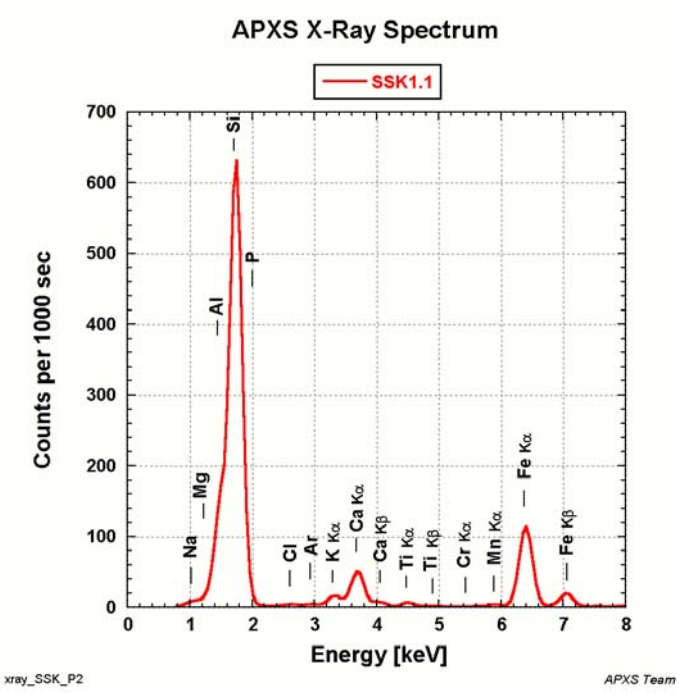


Figure 2 X-ray spectrum of rock 'SSK1' in linear scale measured by MPF type APXS

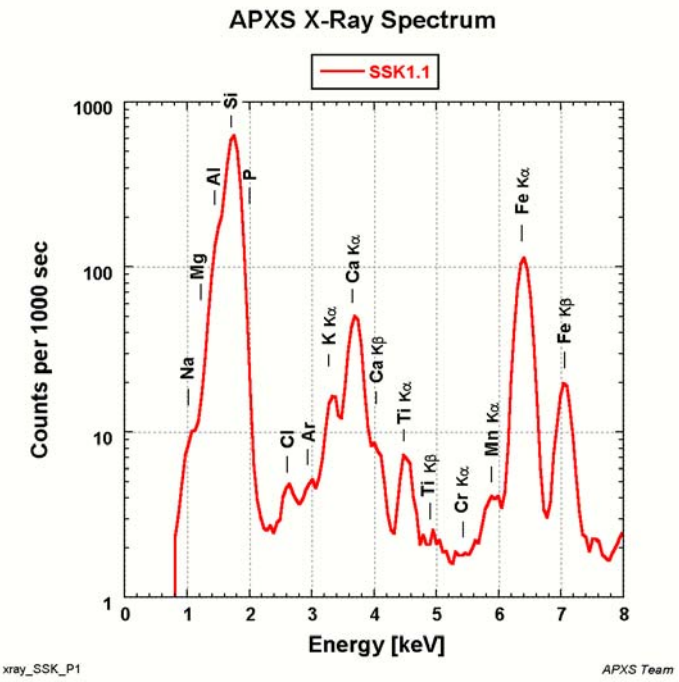


Figure 3 X-ray spectrum of rock 'SSK1' in logarithmic scale

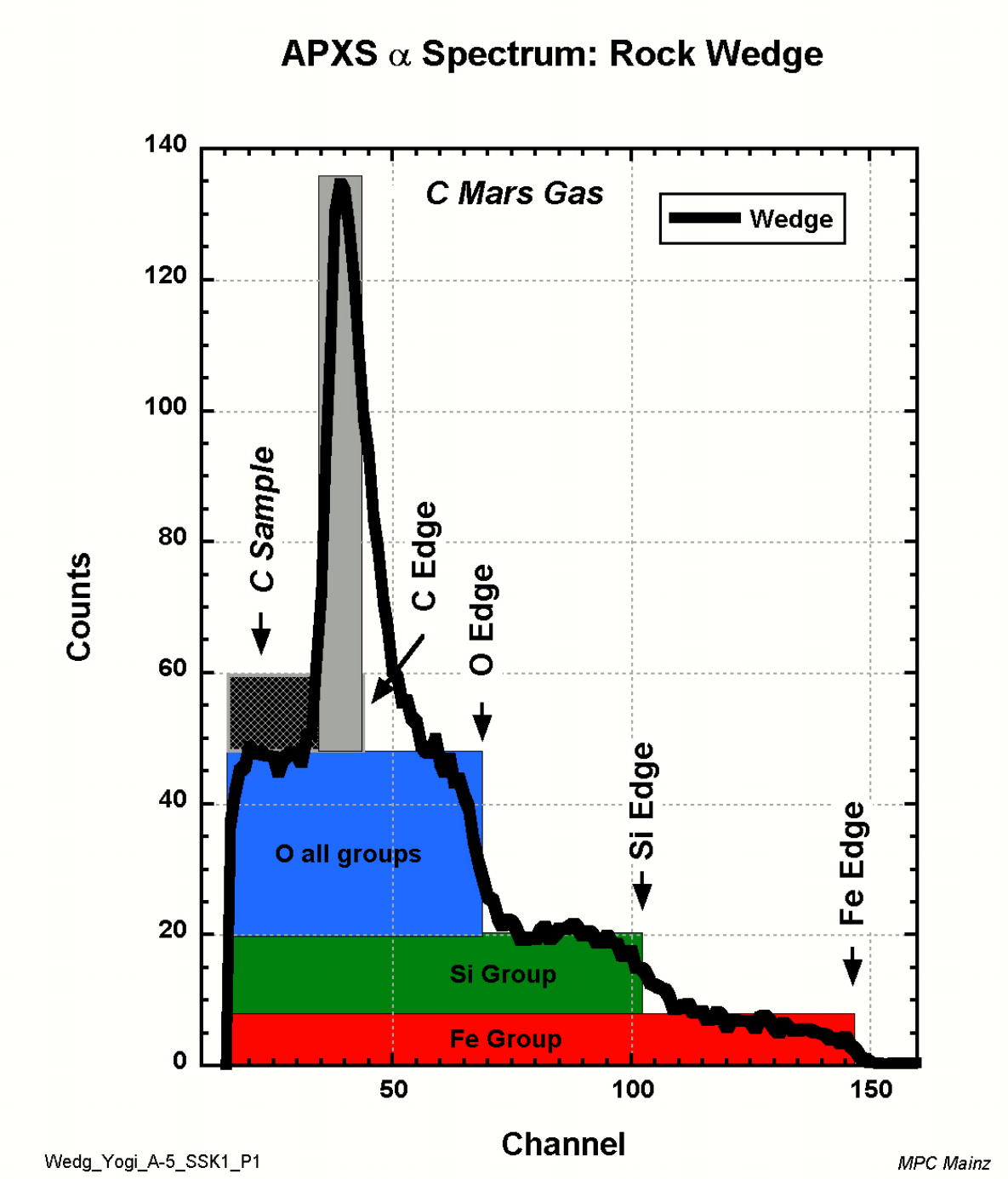


Figure 4 Alpha spectrum of MPF rock 'Wedge'

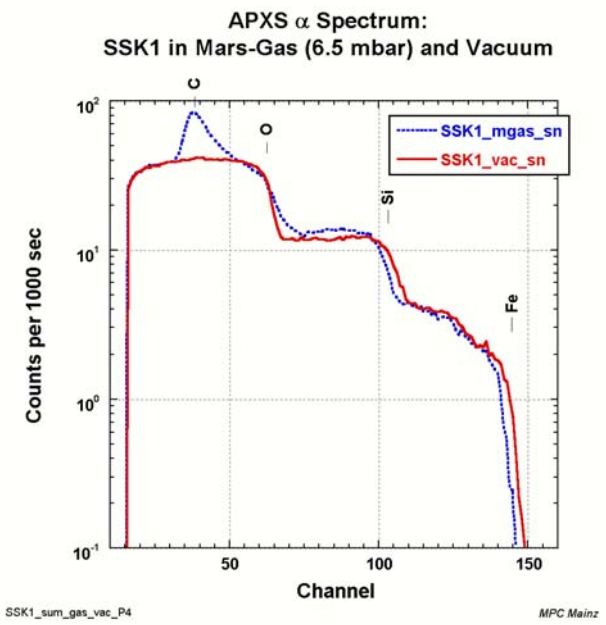


Figure 5 Alpha spectra of rock SSK1 in Martian atmosphere (blue) and vacuum (red), logarithmic scale

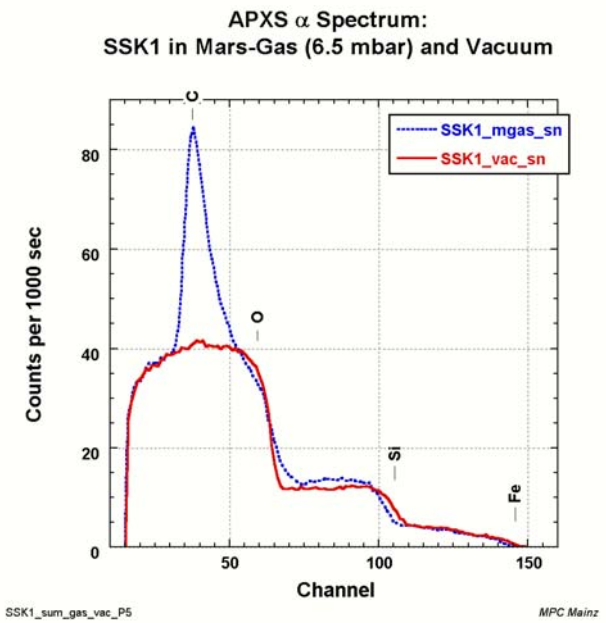


Figure 6 Alpha spectra of rock SSK1 in Martian atmosphere (blue) and vacuum (red), linear scale

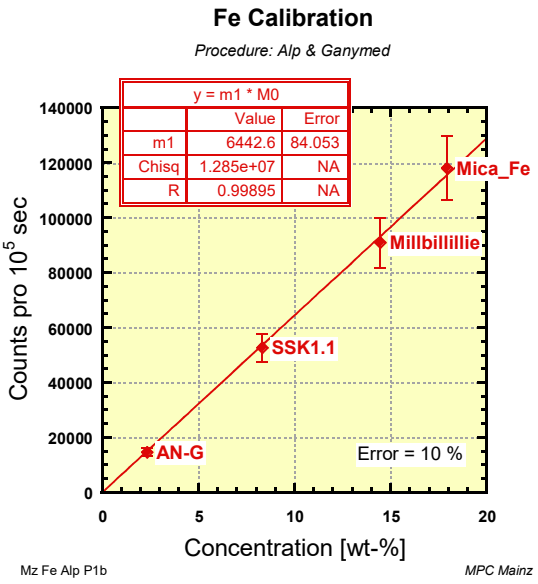


Figure 7 Iron calibration curve for Mars Pathfinder APXS

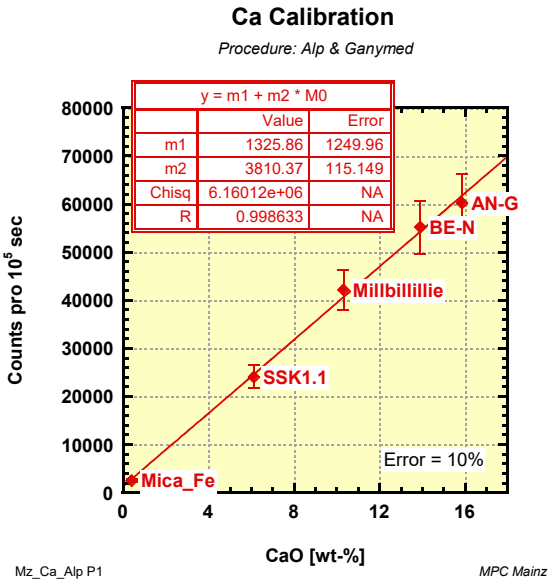


Figure 8 Calcium calibration curve for Mars Pathfinder APXS

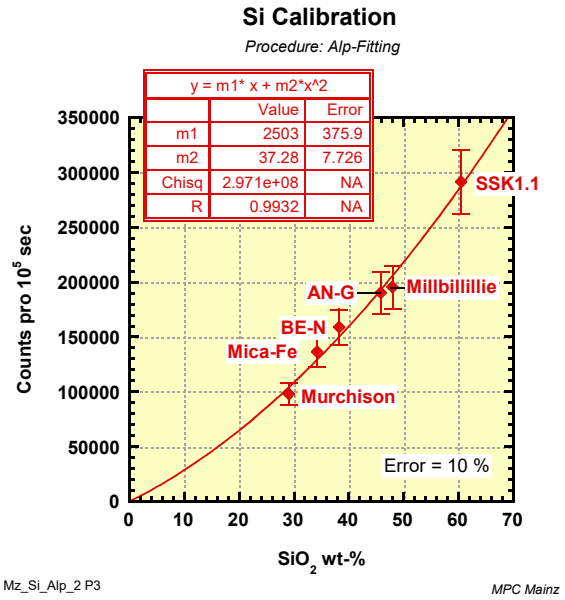


Figure 9 Silicon calibration curve for Mars Pathfinder APXS. Si is the only element whose data points have to be fitted with a polynomial of degree two whose fit is forced through zero.

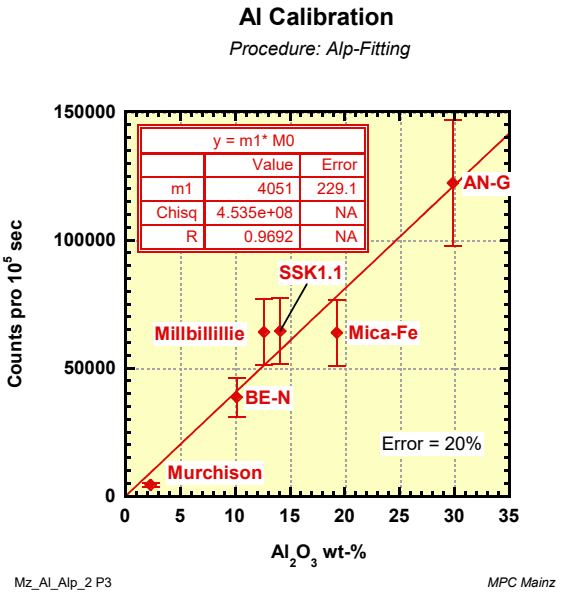


Figure 10 Aluminum calibration curve. Due to the limited energy resolution, there is more scatter of the data points compared to the Fe and Ca curves.

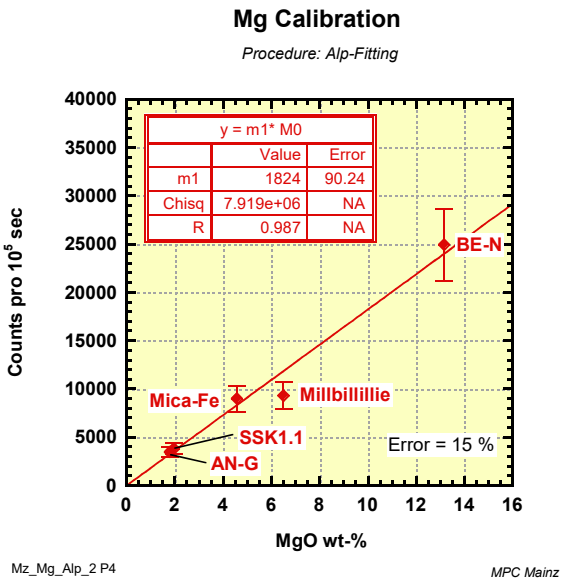
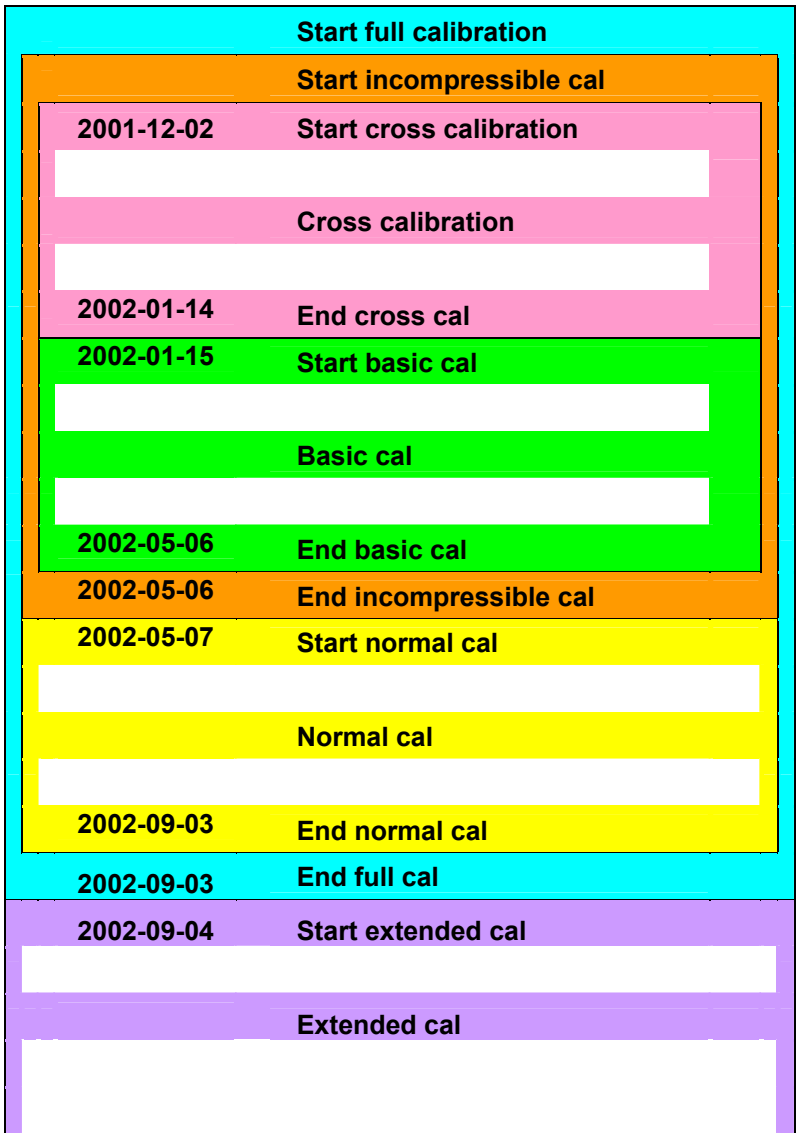


Figure 11 Magnesium calibration curve. Due to the limited energy resolution, there is more scatter of the data points compared to the Fe and Ca curves.

9 APPENDIX: APXS CALIBRATION SCHEDULE

The tables in the following sections detail the APXS calibration schedule. Most of the columns in the tables are self-explanatory. “Spare” means that if the “Margin” is not needed that a spare sample will be measured. Grey shading denotes a weekend, starting during Friday and lasting until Monday morning. Holidays are given in italics (and light blue). Usually long-term measurements are carried out over weekends or holidays. All calibration measurements will be performed at the Max Planck Institut für Chemie in Mainz.

9.1 Calibration Schedule Overview



9.2 APXS Full Calibration Schedule

Athena APXS Calibration				Simple Standards		Rock Standards		
	Date	Day of Week (1=Su.)	Measure-ment time [d]	Standards for x-ray & alpha	Pressure	Geostandards & meteorites	Comments	Spare
			Full calibration (= incompressible cal plus normal cal)					
			Incompressible cal (= cross cal plus basic cal)					
	2001-11-26	2	5	**Setup+ Testing**				
	2001-12-01	7	1	Margin				
	2001-12-02	1	1	**Start Cross Calibration**				
	2001-12-03	2	0.5	**Monitor**	performance check		APXS Doors	
	2001-12-03	2	0.5	Margin				
	2001-12-04	3	1	**Fe**	vacuum			
	2001-12-05	4	1	**SiO2**	vacuum		sputter	
	2001-12-06	5	1	Margin				**SiO2**
	2001-12-07	6	**3**		vacuum	**SSK1.1**	andesite (powder)	
	2001-12-10	2	0.5	**Monitor**	performance check			
	2001-12-10	2	0.5	Margin				**Be**
	2001-12-11	3	1	**MgO**	vacuum		sputter	
	2001-12-12	4	1	**CaO**	vacuum			
	2001-12-13	5	1	**Al_2O_3**	vacuum		sputter	
	2001-12-14	6	**3**		vacuum	**AN-G**	geostandard (powder)	
	2001-12-17	2	0.5	**Monitor**	performance check			
	2001-12-17	2	0.5	Margin				**Be**
	2001-12-18	3	1	**Fe_2O_3**	vacuum			
	2001-12-19	4	1	**Si**	vacuum			
	2001-12-20	5	1	**SiO**	vacuum		sputter	
	2001-12-21	6	**3**		vacuum	**BE-N**	geostandard (powder)	
	2001-12-24	2	0.5	Margin				**C**
	2001-12-24	2	0.5	Margin				**C**
	2001-12-25	3	1	Margin			*Xmas*	**C**
	2001-12-26	4	1	Margin			*Xmas*	**C**
	2001-12-27	5	1	Margin			*Xmas*	**C**
	2001-12-28	6	**3**	**Ta**	vacuum	**Ta**		
	2001-12-31	2	1	**Ta**	vacuum			
	2002-01-01	3	1	**Ta**	vacuum		*New Year*	
	2002-01-02	4	1	**Al**	vacuum			
	2002-01-03	5	1	**Ti**	vacuum			
	2002-01-04	6	**3**	**C**	vacuum	**C**		**Fe_2O_3**
	2002-01-07	2	1	**C**				**Fe**
	2002-01-08	3	1	**C**				**Fe**

	2002-01-09	4	0.5	**Monitor**	performance check			
	2002-01-09	4	0.5	Margin				**$MgSO_4$**
	2002-01-10	5	1	**TiO2**	vacuum			
	2002-01-11	6	1	**NaCl**	vacuum			
	2002-01-12	7	1	Margin				
	2002-01-13	1	1	Margin		**Margin**		
	2002-01-14	2	0.5	**Monitor**	performance check			
	2002-01-14	2	0.5	**End cross cal**				**Be**
	2002-01-15	3	1	**Start basic cal**				
	2002-01-16	4	1	**Ta**	9 mbar			
	2002-01-17	5	1	**Fe**	9 mbar			
	2002-01-18	6	**3**	**Ta**	9 mbar	**Ta**		
	2002-01-21	2	1	**Ta**	9 mbar			
	2002-01-22	3	1	**Fe_2O_3**	9 mbar			
	2002-01-23	4	1	**Fe_2O_3**	9 mbar			
	2002-01-24	5	1	Margin	9 mbar			**Fe_2O_3**
	2002-01-25	6	**3**	**C**	9 mbar	**C**		
	2002-01-28	2	1	**C**	9 mbar			
	2002-01-29	3	0.5	**Monitor**	performance check			
	2002-01-29	3	0.5	Margin				**Si**
	2002-01-30	4	1	**SiO_2**	9 mbar		sputter	
	2002-01-31	5	1	**SiO_2**	9 mbar		sputter	
	2002-02-01	6	**3**		9 mbar	**SSK1.1**	andesite (powder)	
	2002-02-04	2	0.5	**Monitor**	performance check			
	2002-02-04	2	0.5	Margin				**SiO_2**
	2002-02-05	3	1	**SiO_2**	vacuum		sputter	
	2002-02-06	4	1	**SiO_2**	vacuum		sputter	
	2002-02-07	5	1	Margin				**Si**
	2002-02-08	6	**3**			**AN-G**	geostandard (powder)	
	2002-02-11	2	1			**AN-G**	*Holidays*	
	2002-02-12	3	1			**AN-G**	*Holidays*	
	2002-02-13	4	0.5	**Monitor**	performance check			
	2002-02-13	4	0.5	Margin				**Al**
	2002-02-14	5	1	**Al**	9 mbar			
	2002-02-15	6	**3**		9 mbar	**BE-N**	geostandard (powder)	
	2002-02-18	2	0.5	**Monitor**	performance check			
	2002-02-18	2	0.5	Margin				
	2002-02-19	3	1	**CaO**	vacuum			
	2002-02-20	4	1	**CaO**	vacuum			
	2002-02-21	5	1	Margin	vacuum			**CaO**
	2002-02-22	6	**3**	**$CaCO_3$**	vacuum	**$CaCO_3$**		
	2002-02-25	2	0.5	**Monitor**	performance check			
	2002-02-25	2	0.5	Margin				
	2002-02-26	3	1	**Al_2O_3**	9 mbar		sputter	

	2002-02-27	4	1	**Al_2O_3**	9 mbar		sputter	
	2002-02-28	5	1	Margin				**Al_2O_3**
	2002-03-01	6	**3**		9 mbar	**Mica-Fe**	geostandard (powder)	
	2002-03-04	2	0.5	**Monitor**	performance check		*LPSC*	
	2002-03-04	2	0.5	Margin				
	2002-03-05	3	1	**MgO**	9 mbar		sputter	
	2002-03-06	4	1	**MgO**	9 mbar		sputter	
	2002-03-07	5	1	Margin				**MgO**
	2002-03-08	6	**3**	**SiO**	9 mbar	**SiO**	sputter	
	2002-03-11	2	0.5	**Monitor**	performance check			
	2002-03-11	2	0.5	Margin				**Be**
	2002-03-12	3	1	**K_2CO_3**	9 mbar			
	2002-03-13	4	1	**K_2CO_3**	9 mbar			
	2002-03-14	5	1	Margin				**K_2CO_3**
	2002-03-15	6	**3**		9 mbar	**Millbillillie**	meteorite (powder)	
	2002-03-18	2	0.5	**Monitor**	performance check			
	2002-03-18	2	0.5	Margin				**Ti**
	2002-03-19	3	1	**Ti**	9 mbar			
	2002-03-20	4	1	**TiO_2**	9 mbar			
	2002-03-21	5	1	**TiO_2**	9 mbar			
	2002-03-22	6	**3**	**CaO**	9 mbar	**CaO**		
	2002-03-25	2	0.5	**Monitor**	performance check			
	2002-03-25	2	0.5	Margin				**Be**
	2002-03-26	3	1	**NaCl**	9 mbar			
	2002-03-27	4	1	Margin	9 mbar			**NaCl**
	2002-03-28	5	1		9 mbar	**I555**	andesite, powder	
	2002-03-29	6	**3**		9 mbar	**I555**	*Eastern*	
	2002-04-01	2	1		9 mbar	**I555**	*Eastern*	
	2002-04-02	3	0.5	**Monitor**	performance check			
	2002-04-02	3	0.5	Margin				**Na_2CO_3**
	2002-04-03	4	1	**Na_2CO_3**	9 mbar			
	2002-04-04	5	1	**Na_2CO_3**	9 mbar			
	2002-04-05	6	**3**		9 mbar	**Murchison**	meteorite (powder)	
	2002-04-08	2	0.5	**Monitor**	performance check			
	2002-04-08	2	0.5	Margin				**MnO**
	2002-04-09	3	1	**MnO**	9 mbar			
	2002-04-10	4	1	**Cr**	9 mbar			
	2002-04-11	5	1	Margin				**Be**
	2002-04-12	6	**3**		9 mbar	**Allende**	meteorite (powder)	
	2002-04-15	2	0.5	**Monitor**	performance check			
	2002-04-15	2	0.5	Margin				**Be**
	2002-04-16	3	1	**$CaCO_3$**	9 mbar			
	2002-04-17	4	1	**$CaCO_3$**	9 mbar			
	2002-04-18	5	1	Margin				**$CaCO_3$**

	2002-04-19	6	**3**		9 mbar	**Cal Target**		
	2002-04-22	2	0.5	**Monitor**	performance check			
	2002-04-22	2	0.5	Margin				**Be**
	2002-04-23	3	1	**Be**	vacuum			
	2002-04-24	4	1	**Be**	9 mbar			
	2002-04-25	5	1	**$Ca_3(PO_4)_2$**	9 mbar			
	2002-04-26	6	**3**		9 mbar	**Magnet**		
	2002-04-29	2	0.5	**Monitor**	performance check			
	2002-04-29	2	0.5	Margin				
	2002-04-30	3	1	**MgSO4**	9 mbar			
	2002-05-01	4	1	**MgSO4**	9 mbar		*First of May*	
	2002-05-02	5	1	Margin				**Si**
	2002-05-03	6	**3**	**SiO_2**	9 mbar	**SiO_2**	powder	
	2002-05-06	2	0.5	**Monitor**	performance check			
	2002-05-06	2	0.25	**End basic cal**		**End incompressible cal**		
	2002-05-06	2	0.25	**Start normal cal**				
	2002-05-07	3	1	**SiO**	vacuum			
	2002-05-08	4	1	**SiO**	vacuum			
	2002-05-09	5	1			**Morris rock-1**	*Holiday*	
	2002-05-10	6	**3**			**Morris rock-1**	provided by D. M.	
	2002-05-13	2	0.5	**Monitor**	performance check			
	2002-05-13	2	0.5	Margin				
	2002-05-14	3	1	**Be**	diff. pressures			
	2002-05-15	4	1	**Be**	diff. pressures			
	2002-05-16	5	1		vacuum	**Cal Target**		
	2002-05-17	6	**3**		9 mbar	**Morris rock-2**	provided by D. M.	
	2002-05-20	2	1		9 mbar	**Morris rock-2**	*Holiday*	
	2002-05-21	3	0.5	**Monitor**	performance check			
	2002-05-21	3	0.5	Margin				**Si**
	2002-05-22	4	1	**Si**	9 mbar			
	2002-05-23	5	1	**MgO**	9 mbar		powder	
	2002-05-24	6	**3**		9 mbar	**Morris rock-3**	provided by D. M.	
	2002-05-27	2	0.5	**Monitor**	performance check			
	2002-05-27	2	0.5	Margin				**Ni**
	2002-05-28	3	1	**Ni**	9 mbar			
	2002-05-29	4	1	**V**	9 mbar			
	2002-05-30	5	1		9 mbar	**I555**	*Holiday*	
	2002-05-31	6	**3**		9 mbar	**I555**	plate	
	2002-06-03	2	0.5	**Monitor**	performance check			
	2002-06-03	2	0.5	Margin				**Fe_2O_3**
	2002-06-04	3	1	**Fe_2O_3**	vacuum			

	2002-06-05	4	1	**Fe_2O_3**	vacuum			
	2002-06-06	5	1	**Fe_2O_3**	9 mbar			
	2002-06-07	6	**3**		9 mbar	**Morris rock-4**	provided by D. M.	
	2002-06-10	2	0.5	**Monitor**	performance check			
	2002-06-10	2	0.5	Margin				**Fe_2O_3**
	2002-06-11	3	1	**Fe_2O_3**	9 mbar		diff. geo	
	2002-06-12	4	1	**Fe_2O_3**	9 mbar		diff. geo	
	2002-06-13	5	1	Margin				**Fe_2O_3**
	2002-06-14	6	**3**		9 mbar	**Blind-1**	provided by JPL	
	2002-06-17	2	0.5	**Monitor**	performance check			
	2002-06-17	2	0.5	Margin				**Fe**
	2002-06-18	3	1	**Fe**	vacuum			
	2002-06-19	4	1	**Fe**	vacuum			
	2002-06-20	5	1	**Fe**	9 mbar			
	2002-06-21	6	**3**		9 mbar	**Blind-2**	provided by JPL	
	2002-06-24	2	0.5	**Monitor**	performance check			
	2002-06-24	2	0.5	Margin				**SiO_2**
	2002-06-25	3	1	**SiO_2**	9 mbar		sputter	
	2002-06-26	4	1	**SiO_2**	9 mbar		sputter	
	2002-06-27	5	1	Margin				**SiO_2**
	2002-06-28	6	**3**		9 mbar	**Blind-3**	provided by JPL	
	2002-07-01	2	0.5	**Monitor**	performance check			
	2002-07-01	2	0.5	Margin				**SiO_2**
	2002-07-02	3	1	**SiO_2**	9 mbar		diff. geo	
	2002-07-03	4	1	**SiO_2**	9 mbar		diff. geo	
	2002-07-04	5	1	**SiO_2**	9 mbar		diff. geo	
	2002-07-05	6	**3**		9 mbar	**Cal Target**		
	2002-07-08	2	0.5	**Monitor**	performance check			
	2002-07-08	2	0.5	Margin				**MgO**
	2002-07-09	3	1	**MgO**	9 mbar		sputter	
	2002-07-10	4	1	**MgO**	9 mbar		sputter	
	2002-07-11	5	1	Margin				
	2002-07-12	6	**3**			**Bilnd-4**	provided by JPL	
	2002-07-15	2	0.5	**Monitor**	performance check			
	2002-07-15	2	0.5	Margin				
	2002-07-16	3	1	**NaCl**	9 mbar			
	2002-07-17	4	1	**NaCl**	9 mbar			
	2002-07-18	5	1	**SiO**	9 mbar		sputter	
	2002-07-19	6	**3**			**Bilnd-5**	provided by	

							JPL	
	2002-07-22	2	0.5	**Monitor**	performance check			
	2002-07-22	2	0.5	Margin				**$MgSO_4$**
	2002-07-23	3	1	**$MgSO_4$**	9 mbar			
	2002-07-24	4	1	**$MgSO_4$**	9 mbar			
	2002-07-25	5	1	Margin				**$MgSO_4$**
	2002-07-26	6	3			**Geostandard**		
	2002-07-29	2	0.5	**Monitor**	performance check			
	2002-07-29	2	0.5	Margin				**K_2CO_3**
	2002-07-30	3	1	**K_2CO_3**	9 mbar			
	2002-07-31	4	1	**K_2CO_3**	9 mbar			
	2002-08-01	5	1	**TiO_2**	9 mbar			
	2002-08-02	6	3			**Geostandard**		
	2002-08-05	2	0.5	**Monitor**	performance check			
	2002-08-05	2	0.5	Margin				**CaO**
	2002-08-06	3	1	**CaO**	9 mbar			
	2002-08-07	4	1	**Na_2CO_3**	9 mbar			
	2002-08-08	5	1	Margin				**Na_2CO_3**
	2002-08-09	6	3			**TBD**		
	2002-08-12	2	0.5	**Monitor**	performance check			
	2002-08-12	2	0.5	Margin				**Al_2O_3**
	2002-08-13	3	1	**Al_2O_3**	9 mbar		sputter	
	2002-08-14	4	1	**Al_2O_3**	9 mbar		sputter	
	2002-08-15	5	1	Margin				**Al_2O_3**
	2002-08-16	6	3			**TBD**		
	2002-08-19	2	0.5	**Monitor**	performance check			
	2002-08-19	2	0.5	Margin				**Be**
	2002-08-20	3	1	**Be**	diff. pressures			
	2002-08-21	4	1	**Be**	diff. pressures			
	2002-08-22	5	1	**Be**	diff. pressures			
	2002-08-23	6	3			**TBD**		
	2002-08-26	2	0.5	**Monitor**	performance check			
	2002-08-26	2	0.5	Margin				
	2002-08-27	3	1	**$CaCO_3$**	9 mbar			
	2002-08-28	4	1	**$CaCO_3$**	9 mbar			
	2002-08-29	5	1					
	2002-08-30	6	3			**TBD**		
	2002-09-02	2	0.5	**Monitor**	performance check			
	2002-09-02	2	0.5	Margin				
	2002-09-03	3	0.5		**End normal cal**			
	2002-09-03	3	0.5		**End full cal**			
	2002-09-04	4	1		**Start extended**			

					cal			
	2002-09-05	5	1					

To be continued

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