THE CHAMPOLLION COMETARY MOLECULAR ANALYSIS EXPERIMENT

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ABSTRACT

The Chemical Analysis of Released Gas Experiment (CHARGE), is one of several investigations selected for the Champollion New Millennium DS4 Mission. CHARGE is presently being designed to carry out a detailed molecular and isotopic analysis of material collected from the surface and several centimeters below the surface of comet Tempel 1. The highest priority scientific issues addressed by this investigation include: the chemical conditions present in the region of cometary formation; the chemical changes during cometary formation and over the lifetime of the comet; the relationship of comets to other primitive and more evolved bodies in the solar system and to the parent interstellar cloud; the contribution of cometary material to the atmospheres and oceans of planets; and the nature of the mixture of ices and dust grains which give rise to the coma and extended sources of gas as a comet approaches perihelion. CHARGE will be designed to thermally process samples of solid phase material from near the ambient temperature to approximately 900 K. Gases evolved from the frozen ices will be continuously analyzed as a function of sample temperature by a quadrupole mass spectrometer with a mass range of 2 to 300 amu. A broad range of major and trace species, both organic and inorganic, from the gases evolved from the solid samples will undergo both chemical and cold trapping for subsequent analysis by gas chromatograph mass spectrometer (GCMS) analysis. CHARGE technology heritage includes the Galileo Probe Mass Spectrometer (Niemann et al., 1996) that successfully
measured the composition of Jupiter’s atmosphere in December of 1995. The landed portion of the mission will enable analysis of subsurface materials and allow identification of organic species present at sub-parts per million mole-fraction in the nucleus. Prior to the landed operations, CHARGE will carry out measurements from orbit for a period of several weeks.

INTRODUCTION

The Champollion Deep Space 4 (DS4) Mission (Muirhead et al., 1997) is one of several spacecraft missions to comets planned within the next decade. These include: the Stardust Mission (Brownlee et al., 1997) supported by the Discovery Program of the National Aeronautics and Space Administration (NASA) and designed to return cometary dust particles to Earth for a detailed chemical analysis; another Discovery Missions, Comet Nucleus Tour (CONTOUR) designed to fly past three comets over a period of 5 years (Veverka, 1997); and the Rosetta Mission (Bar-Nun et al., 1993) of the European Space Agency (ESA) designed to provide both an orbital survey and an in situ characterization of the nucleus of comet P/Wirtanen with a surface science package. NASA’s Champollion Lander was originally designed as a comet nucleus lander for the Rosetta Mission. Present plans call for Champollion to provide the major scientific investigations of the DS4 Mission of NASA’s New Millennium Program with both an orbital and landed measurement phase. While the understanding of the chemical characteristics of comets has increased substantially over the past decade with the Halley flyby missions (Mason, 1990) and intensive remote sensing observations of recent comets such as Comet C/1995 B2 Hyakutake (Mumma et al., 1996) and Hale Bopp (Crukshank, 1997) in several spectral regions with powerful new detectors, the observations planned from the Champollion instruments located in close proximity to the nucleus of Tempel 1 are expected to provide a still more detailed set of data. In keeping with the focus of the Champollion Mission on the chemical and physical nature of the nucleus a surface landing is planned with chemical analysis of samples collected from up to one meter below the nucleus surface.

The study of the chemical composition and physical characteristics of comets is considered fundamental to a better understanding of solar system origins (Van Dishoeck et al., 1993), the origins of comets themselves (Mumma et al., 1993), the evolution of planetary atmospheres, and the eventual transformation of short period comets into less volatile bodies. The prime scientific objective developed for the Champollion Lander is:

- the determination of the elemental and molecular composition of cometary surface layers.

Other high priority scientific objectives include:

- a mineralogical and isotopic determination of surface layers
- a study of the physical properties such as ice phases, texture, porosity, mean density, as well as thermal and dielectric properties
- an optical characterization of the cometary surface
- an analysis of the internal structure of the cometary nucleus.

The surface lander experiments enables a range of unique measurements that can be satisfactorily implemented only from a nucleus lander. The scientific investigation defined for the Champollion molecular analysis experiment CHARGE is given below, followed by a description of the instrument proposed for this application.

THE SCIENTIFIC AND MEASUREMENT OBJECTIVES OF THE CHARGE INVESTIGATION

Principal Scientific Objectives

The gases contained in the ices of the cometary nucleus are considered to have originated either in the interstellar cloud from which the solar system formed, or through condensation in the outer part of the protosolar nebula. Thus, the detailed examination of the molecular characteristics of a comet’s nucleus can provide information on the relationship between the state and composition of the solar nebula to the parent interstellar cloud. In addition, comets are considered to be representatives of the icy planetesimals that through aggregation or collisions contributed to the cores of giant planets, to the atmospheres of inner and outer planets, and to other smaller bodies in the solar system. In order to more fully understand these chemical links, key measurement objectives for an isotopic and molecular analysis experiment, are the carbon oxidation state, the isotopic ratios of many elements and the noble gases including O, C, N, S, H, He, and Ar and the ratio of N₂/COR/Ar/other inert gas species. The inert gas ratios provide tracers of the
formation temperature of comets (Owen et al., 1991) and also clarify the extent of their delivery of volatiles to the inner solar system. The abundance ratios in homologous molecular series such as alcohols, aldehydes, and hydrocarbons can provide information on the formation conditions of molecules either in the gas phase or in heterogeneous processes and the relative importance of kinetic or equilibrium processes. The recent measurements (Mumma, 1996) of the ethane to methane ratio in Comet C/1996 B2 Hyakutake, for example, led these authors to conclude that the ices from this comet were not produced in conditions of thermochemical equilibrium.

**Unique Capabilities Provided by an in situ Molecular Composition Experiment**

There will be rapid and extensive chemical and photochemical processing of many possible cometary species after their evolution from the ices of the nucleus (Crifo, 1994). In the case of results from the Giotto mass spectrometer (Schmidt et al., 1988, and Geiss et al., 1991), where instruments were located many kilometers from the nucleus, the large number of possible chemical pathways and the short scale lengths for some species made it difficult or impossible to uniquely identify the parent molecular species present in the nucleus. Elaborate and detailed chemical models were necessary to demonstrate the presence of several major species and numerous trace species present in the spectra remain unidentified. Furthermore, many of the species remain trapped in the nucleus. Thus the both orbital and surface data sets are desirable and together are likely to provide detection of both a broad range of parent molecular species and of the transformations of these species after release into space. On the surface of the comet nucleus far from perihelion the sample volume will be determined primarily by the analytical requirements and also by the subsurface sampling mechanisms, and consequently a relatively large quantity of sample can be processed to isolate detectable quantities of very trace species.

**Measurement Objectives**

The primary measurement objective for CHARGE is to analyze the molecular and isotopic composition of solid phase components (both organic and inorganic) of the nucleus of Comet Tempel I. The organic fraction is expected to include:

- low molecular weight, highly volatile organic molecules
- more complex organic molecules such as amino acids, and
- high molecular weight, refractory macromolecular organics.

The inorganic fraction detectable by CHARGE is expected to include:

- a volatile component in frozen ices of species such as H₂O and CO² and gases trapped in the ice
- less volatile minerals such as hydrates, ammonia salts, and carbonates.

Each component or mineral phase of the solid phase material will evolve gas over a characteristic temperature range by processes such as sublimation, decomposition, or chemical reaction. The controlled thermal processing of a sample of the solid phase material, together with the planned continuous gas phase analysis, will allow the physicochemical characteristics of the nucleus material to be studied.

A secondary objective in the experiment is to analyze the coma neutral and ion composition from orbit and the neutral gas environment at the surface after landing.

**Overview of Measurement Technique**

The approach to achieving the primary objective of the CHARGE Investigation are based on both direct mass spectrometer and GCMS analysis of gaseous species evolved from a solid sample of the comet nucleus. This sample delivered by the Sample Acquisition and Transport Mechanism (SATM) is sealed in a pyrolysis cell and heated in a controlled manner. CHARGE will analyze both surface and subsurface samples provided by this facility sampling system. The measurement goal is to establish the relative abundance of the major volatile species present in the samples to an accuracy of better than ±10% and to detect many trace species with better than 1 part per million sensitivity. A very simple backup sampler system will provide a near surface sample for analysis in the event of a failure in the facility sampler.
Dust and a Representative Sample of Cometary Material

The dust impact experiments PIA and PUMA on Giotto and Vega 1 and 2 missions to Halley provided fundamental new information on comet dust heterogeneity and composition of individual grains (Jessberger and Kissel, 1991, McDonnell et al., 1991). Particle sizes sampled were within the $10^6$ to $10^{11}$ g and these individual grains were found to be either a Mg-rich silicate phase or a mixture of this phase with an organic phase (CHON). The observed grain heterogeneity, together with its enrichment in H, C, and N compared to CI-chondrites, suggests that the Halley dust is indeed primitive material. The dust particles sampled by these experiments, however, were entirely the very small (i.e. micron sized) particles while, in fact, much of the released solid phase mass in comets likely resides in larger particles (Sykes et al., 1990). The sample volumes which will be delivered to the CHARGE oven for chemical analysis will be on the order of $0.1 \text{ cm}^3$ and the analysis of the gases released from dust more representative of the bulk composition of the nucleus. The microscopic imager of the Champollion Infrared and Camera Lander Experiment (CIRCLE) will examine the composition and morphology of individual grains to establish heterogeneity. The temperatures achieved in the CHARGE oven will mimic the maximum temperatures achieved by the cometary dust grains that can be sampled from an orbiting or flyby spacecraft. However, the gases released from the CHARGE oven can be sampled in this experiment without the complication of the subsequent photochemical alteration that would be present in the coma measurements.

Parent Molecular Species

In the past 11 years the list of directly detected cometary parent volatile species has grown from CO to now include H$_2$O, CO$_2$, H$_2$CO, CH$_3$OH, CH$_4$, C$_2$H$_6$, C$_3$H$_8$, HCN, HC$_3$N, and H$_2$S (Mumma et al., 1993). However, monocarboxylic acids and higher homologues of alcohols and aldehydes have not yet been detected. The variety of interstellar molecules which have been discovered by radio techniques are prime candidates for detection by CHARGE. The high mass range of the CHARGE mass spectrometer combined with the use of a nonspecific column for the GC analysis is intended to allow a broad survey of the molecular composition of Comet Tempel I.

It has been suggested that carbon suboxide (C$_3$O$_2$) could be responsible for the observed extended emission of CO molecules in the coma of comet Halley (Huntress et al., 1991; Allen 1991; see also Crovisier et al., 1991). Carbon suboxide could be synthesized, in CO-rich pre-cometary interstellar ices, by energetic processing as demonstrated in the laboratory, by UV irradiation of frozen CO (Gerakines et al., 1996) and ion bombardment of CO, CO/O$_2$, and CO/N$_2$ frozen mixtures (Strazzulla et al., 1996). It was also found that, after ion irradiation at low T (10K) and warming up (170K) the deposited CO, and the produced CO$_2$ sublimated and a residue made of suboxides was left over. These experiments provide another set of candidate species for detection by CHARGE.

The European IS0 satellite, successfully launched in November 1995 led to fundamental new discoveries regarding the composition of interstellar grains, their ice mantles and their interaction with the gas phase. Solid CO$_2$ has been previously detected by d'Hendecourt and de Muizon (1989) in IRAS-LRS spectra of 3 sources through its bending mode at 15.2 micron. IS0 results (Van Dishoeck et al., 1996) show the ubiquitous presence of CO$_2$ ice in the interstellar medium. The abundance of solid CO$_2$ in some protostars is 20% greater than water ice (de Graauw et al. 1996, d'Hendecourt et al. 1996, Whittet et al. 1996). Solid CH$_4$ has been identified in several sources at a few percent of the water ice level (Boogert et al. 1996, d'Hendecourt et al. 1996). Current results of interest to the cometary studies reveal the presence of several molecules such as H$_2$CO and OCS in embedded protostars (d'Hendecourt et al. 1996). Future IS0 observations will provide important constraints on the composition and thermal history of precometary ices and organics.

Isotopic Abundances

The unit mass resolution of the CHARGE mass spectrometer and the use of advanced pulse-counting detectors should allow isotopic ratios to be obtained for many species. The D/H ratios measured in comet Halley water (Eberhardt et al., 1995, Balsiger et al., 1995), Hale Bopp water (Meier et al., 1998), and in Comet Hyakutake (Bockele-Morvan et al., 1998) were found to be much higher than the D/H ratio in Jupiter (Mahaffy et al., 1998, Lellouch et al., 1996), Saturn, Uranus and Neptune and the proto-sun indicating that this water did not equilibrate with H$_2$ in the solar nebula and suggesting also that a contribution may have been made to the Earth's oceans by cometary impact. The Halley value is twice the ratio of D/H in the Earth's oceans, suggesting that if comets contributed to the terrestrial water inventory, other comets must have lower values of D/H in their ices than Halley does, or the Earth's oceans consist of...
a blend of water from comets and water from another source, perhaps from the rocks that form the bulk of the planet’s mass (Owen and Bar-Nun 1995). Given the possible importance of comets as contributors of the biogenic elements to the early Earth, it is clearly important to determine just how massive the early cometary contribution was. The value of D/H in water is an obvious key. To make this analysis more compelling, it is important to determine this ratio in other cometary molecules as well, as we know that there are substantial variations in the value of D/H in interstellar molecules (Van Dishoek et al. 1993). This study would also help to establish just how pristine the material in the comets really is.

It is thus of considerable interest to obtain D/H ratios in several molecules in another comet through measurements by CHARGE and by experiments on other missions such as Rosetta. The D/H ratio even in the same molecule may, in fact, vary for different phases reflecting a different origin and the slow thermal processing provided by CHARGE will be designed to reveal these differences.

The success of the Galileo MS in detecting the isotopes of xenon in mixing ratios at sub parts per billion illustrates the potential of this technique for a determination of isotope ratios for this extremely instructive gas in Comet Tempel 1. The Xe isotopes in the solar wind, in the carbonaceous chondrites, and in the atmospheres of Mars and Earth are all different (Pepin, 1989) and it is not clear how these three distinctly different patterns of isotopes relate to one another. Under appropriate conditions, the escape of massive amounts of hydrogen from early atmospheres of Mars and Earth could convert an originally solar or meteoritic pattern to the present atmospheric values (Pepin, 1991), but what was the original isotopic pattern? Finding this pattern in the comets will provide a sensitive tool for determining just what fraction of the Earth’s volatile inventory and that of Mars was contributed by comets.

**Noble Gases and Chemical Thermometers**

Species such as CO, CH₄, H₂CO, and particularly the noble gases because of their chemical inertness are sensitive thermometers of the formation temperature and thermal history of comets. The temperatures derived (Owen, et al., 1991) depend on the trapping efficiency of gases in the amorphous ice, and a number of laboratory studies have been carried out to obtain this information (Bar-Nun et al., 1988). Once noble gas abundances and their isotopic distributions have been established for comets the importance of comets in bringing volatiles to the inner planets can more easily be established. In situ measurements at Halley were not able to derive noble gas abundances because of the extreme measurement conditions of the fast flyby, but upper limits have been obtained from EUV spectra of recent comets (e.g., Feldman et al. 1991). The CHARGE experiment will incorporate a mode designed to enhance the accuracy of the noble gas measurements. Helium, however, will not be measured by CHARGE since it is to be used as a carrier gas for the pyrolysis cell. This is not, however, expected to be significant, as the laboratory experiments have demonstrated that even neon is not trapped in ice forming at temperatures greater than approximately 25K (Bar-Nun et al. 1985).

**Complex Organic Species**

More than 500 organic molecules including numerous amino acids (Cronin, et al., 1988) have been identified in a somewhat more processed but still primitive solar system body, the Murchison meteorite a type I carbonaceous chondrite. Comets together with Mars and Titan are important solar system objects for the study of extraterrestrial organic chemistry. Comets are also possible sources of transport to earth of complex organic molecules. From the point of view of these studies it is important to analyze large macromolecular species. This will be done in the CHARGE experiment through a chemical analysis of directly desorbed species or of the pyrolysis products of the decomposition products of these macromolecules. Pyrolysis products of various amino acids have been studied in the laboratory (Raulin, 1995) and as illustrated in Figure 1, typical chemical reaction pathways identified which produce the smaller more stable molecules which will be analyzed by the CHARGE GCMS.

**Measurement Strategy**

The CHARGE measurement strategy is based on use of 3 major elements

- a mass spectrometer
Mass spectrometry is a general molecular analysis technique that does not discriminate against any sufficiently volatile and stable species. Use of the mass spectrometer detection also allows the determination of isotopic abundances for many elements. An enhancement to the direct mass spectrometer analysis is the gas chromatograph mass spectrometer. Helium is used as the carrier gas. For a complex mixture of molecules use of the gas chromatograph technique avoids ambiguities in interpretation which can otherwise arise when fragments in the mass spectra contain substantial levels of overlap. This is always the case when mixtures of hydrocarbons are analyzed. The gas chromatograph allows individual species to be eluted at different times into the mass spectrometer where an unambiguous identification can be carried out by matching the mass spectrum to a library of tens of thousands of spectra.

![Diagram of reaction pathways](image)

**Figure 1** An example of the reaction pathways for the decomposition of the amino acid L-Phenylalanine as inferred from a GCMS analysis. Molecules are labeled with an identifying peak number from the gas chromatogram.
INVESTIGATIVE APPROACH AND EXPERIMENT DESCRIPTION

Thermal processing of the solid phase comet sample in an oven allows the volatile species, or in the case of complex and fragile molecules, the pyrolysis products of these molecules, to be transferred to the gas phase for GCMS analysis. Since the CHARGE oven is located in a cold region with a low thermal conductance to the GCMS, the thermal processing can start near the temperature of the delivered sample. The temperature of the sample will then raised in a carefully controlled sequence over a period of several minutes to a final temperature of approximately 900 K. During this thermal processing the gaseous species evolved from the sample will continuously be removed from the sample area by a helium gas flow. Through the entire heating cycle another fraction of the gas stream will be diverted into a chemical trap in series with a cold trap located in the cold region. All except the lightest molecules will be trapped in one of these two traps and stored for subsequent GCMS analysis. A system of valves and capillary leaks into the ion source of the mass spectrometer will allow the 2 gas streams to be alternately sampled. That portion of the gas stream not introduced into the ion source will be vented to an exhaust port on the exterior of the Champollion lander. Although CHARGE will not be designed to measure heat flow into the solid sample, many solid phase transitions of interest will produce evolved gas at characteristic sample temperatures that will give information on the chemical and physical properties of these ices.

Experiment Description

Experiment Schematic. Figure 2 illustrates the transport of material within the instrument.

Overview of Measurement Sequence. Upon rendezvous with the Tempel I, CHARGE will begin operation. The first step is the breakoff of a metal cover that has kept the instrument in ultra high vacuum conditions during cruise. This is achieved by pyrotechnically impacting a ceramic to metal seal. This exposes apertures that can directly sample gas released from the cometary nucleus. Gas will be sampled in the 2 to 300 amu mass range of the instrument for a period of many weeks while the DS4 carrier vehicle orbits the nucleus to identify an optimal landing site. Just prior to the landing operation the mass spectrometer will be turned off and all apertures closed to protect the instrument from contamination.

Shortly after landing on the surface of the nucleus, two 24-hour measurement periods are planned for the Champollion instrument set separated by a 36 hour time period for a rapid evaluation of the first data set. The Lander Sample Acquisition and Transfer Mechanism will deliver a minimum of three samples to CHARGE during the first 24-hour period. Shortly after landing, all CHARGE microvalves that have been kept open during cruise will be closed. The two miniaturized pyrotechnically actuated ceramic seal breakoff caps that have kept the mass spectrometer and the volume connected to the pyrolysis cell contamination free (under hard vacuum) will be activated to expose the CHARGE pyrolysis system. At this point CHARGE is ready to receive samples and begin chemical analysis. The time required to carry out each thermal and GCMS analysis cycle after transfer of a solid sample from the SATM will be approximately 1 hour. The pyrolysis oven shown in Figure 2 in the cold region will be located on the baseplate of the Champollion Lander.

The measurement sequence for each sample is expected to include the following steps:

- obtain background mass spectra over the entire mass range
- accept a sample from SATM in the cooled pyrolysis cell, seal the oven door, and initiate the flow of the helium carrier gas.
- ramp the temperature of the pyrolysis cell from ambient to 900 K over a time period of several minutes. Through valve operations alternately obtain mass scans during this heating cycle from the gas stream directly transported from the sample cell and the gas stream directed through the two trap systems. The former will allow a nearly continuous detection of evolved species as a function of the sample temperature while the latter sampling scheme will enhance the accuracy of the noble gas measurements since nearly all other species that might create spectral interference will be retained by the two traps. During the temperature ramp of the oven, evolution of the most volatile gaseous species will first occur from sublimation of their frozen ices, then evolution of more complex molecules and simple molecules chemically bound in more complex ices or other solid materials, and finally at the
highest temperatures characteristic pyrolysis products of very complex organic molecules and macromolecules.

- after the pyrolysis cycle is complete, release gas from the cold trap by rapid heating and introduce a sample of this gas into the GC for GCMS analysis.
- release gas from the chemical trap while maintaining the cold trap temperature sufficiently high to transmit all species and repeat the GCMS analysis.
- obtain background spectra and open the oven door to allow it to begin cooling for delivery of the next sample.

The baseline plan for the second 24-hour analysis period will be to repeat the operational sequence summarized above. However, if analysis of the data from the first 24-hour sequence shows a failure in the Champollion sample delivery system, then the backup instrument sampler system will be deployed. The backup sampler acquires a small volume of the nucleus material in a collection tube and then docks the tube together with the collected material to a heated inlet port. A portion of the vapor evolved by this thermal processing will diffuse through the inlet tube connected to valve V3 and will be analyzed by the mass spectrometer. The backup sampler system has been kept very simple because of mass constraints and no GCMS analysis will be carried out of this sample of evolved gas.

Figure 2. The gas flow path in charge is illustrated with valves (V1 thru V9), an injection valve (IV), pressure sensors (PS1 and PS2) and flow regulators (FR).
Instrument Elements and their Heritage. The mass spectrometer quadrupole analyzer assembly will be similar to that used on the Galileo Probe Neutral Mass Spectrometer (Niemann et al., 1992). It consists of 4 precision ground hyperbolic countered rods mounted in a rigid assembly. Combinations of RF and DC voltages applied to opposite rod pairs control the transmitted mass. Two frequency steps are used to allow the maximum value of the RF voltage to remain within a level that does not consume an excessive amount of power. Ions transmitted through the analyzer are detected by a pulse counting detector. The ion source will incorporate dual electron guns for the sake of redundancy. Should the filament in one gun break or burn out, the other filament is automatically activated. The source filament material will be 0.075 mm diameter 97%W-3%Re wire. The ion source electronics will allow electron beam energies of 75 or 20 eV to be selected. 20 eV is below the ionization potential of the carrier gas which will reduce background effects related to the number of total ions in the source as well as providing an alternate set of cracking patterns for complex molecules. The gas chromatograph column baselined is a Wall Coated Open Tubular such as a metal fused silica film MXT capillary column. The wide number of chemical species expected, including both simple molecules and complex species such as organic macromolecules, suggests the use of a non specific column. There are several options for coating such as dimethylpolysiloxane (apolar) and cyanopropylphenyl-methy polysiloxane (slightly polar) crossbond stationary phases. These columns have been extensively studied in our laboratories for the Cassini Huygens application (Niemann et al., 1992) and have been flight qualified. The chemical trap consists of a high surface area material generated from a porous carbon structure. The less volatile component of the gas will be retained on this material at the operating temperature of the chemical trap while lighter molecules will pass through this region and be collected in the cold trap.

SUMMARY

CHARGE will be designed to carry out a broad survey of the molecular state of the nucleus of comet Temple I. The scientific investigation is designed to contribute to the Champollion study of the nature of the formation and evolution of comets and to address questions of the relationship of these primitive objects to more evolved solar system bodies and to the parent interstellar cloud. The technical challenge will be to obtain these measurements in the extreme conditions encountered at the cometary surface and within the limited mass and power resources allocated to the Champollion Lander.

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