

Europlanet TNA Report

PROJECT LEADER

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Date of TNA visit:	19th February – 20th March
Host laboratory:	Molecular physics laboratory, The Open University, Milton Keynes, United Kingdom

Project Title – Synthesis and analysis of organics generated in glow discharge fed by in CH₄-N₂ mixture at reduced pressures

- Report on the outcomes of the TNA visit (approx 1 page)

My TNA was focused on design and building an equipment for measuring characteristic emission bands, and measuring FTIR spectra occurred in a N₂-CH₄ glow discharge plasma ignited at reduced pressure (0.7-0.8 mbar) and ambient temperature. We used XPS diagnostic method for analyze solid products formed in the discharge reactor. The experimental equipments used in our experiments are described in Figure 1. Experiments have been carried out in a gas flow regime at reduced pressure of 0.7-0.8mbar. The required pressure level was set using a rotary pump. The mixture ratios for both CH₄ (purity 99.9995%) and N₂ (purity 99.9999%) gas stream were regulated using MKS mass flow controllers. The content of methane in nitrogen was about in range (1:6). The electrode configuration consisted of a central HV rod electrode and an outer cylinder reactor body. The central electrode was connected to a DC HV positive power supply, the outer electrode was grounded. The ignition voltage between the electrodes was in range between 350-360 V, meanwhile the discharge current was regulated in range 10-40mA. Temperature of the reactor body did not exceed 320 K.

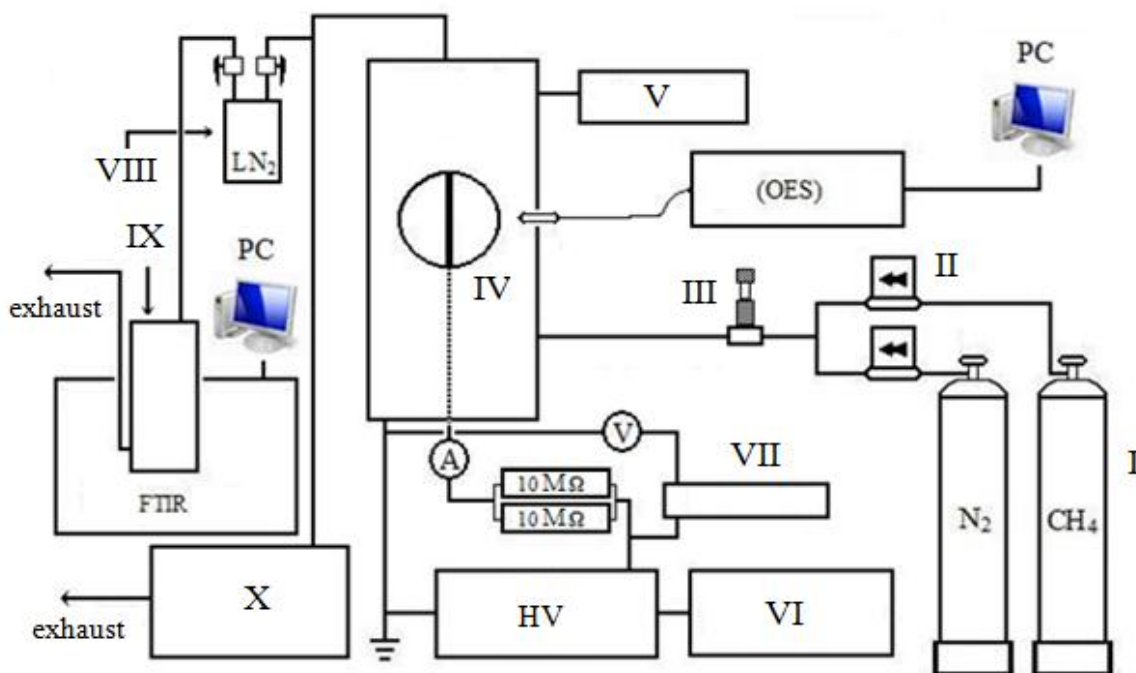


Fig. 1: A simplified scheme of the experimental setup: I. Nitrogen and methane gas bottles; II. mass flow controllers for setting the required CH₄-N₂ mixture ratio; III. needle valve IV. coaxial discharge reactor with a HV rod electrode; V. vacuumeter; VI. transformer; VII. HV probe; VIII. kryotrap with liquid nitrogen; IX. InfraRed cell; X. rotary oil pump with gauge.

A Quartz side port view was built at the reactor body allowing spectral analysis of the generated glow plasma using a Jobin Yvon Triax 550 spectrograph equipped with iCCD detector. The emitted light from the plasma was led through the viewport to the entrance slit of spectrograph by a multi-core quartz optical fiber. Optical emission spectra were recorded at groove settings 300 and 1800 grooves/mm.

For effective analysis of gaseous products using FTIR spectroscopy kryotrap was used to collect a sufficient content of condensed gaseous products, which was attached to the exhausts and cooled with liquid nitrogen (LN₂). The time needed for collection was 50 hours.

The diagnostic method XPS was used to analyze solid products forming in the discharge chamber. A sticky carbon sampling films was placed inside the discharge zone which adsorbed solid/dust products.

- **Summary of the main results obtained**

OES spectrometric study of products formed in our glow discharge fed by an admixture of CH₄ in N₂ was carried out in a flowing regime at room temperature and reduced pressure 0.7-0.8 mbar. As it is shown in Fig. 2, the spectra of CN violet and red, furthermore the first/second positive and first negative systems of nitrogen bands have been found to be the most dominant.

Increasing of discharge current caused an increase in the band intensities thus indicating the increasing density of electron induced ionization processes and population of excited species. However, calculation of electron density and temperature, vibrational and rotational temperatures of nitrogen excited molecules need a deeper analysis. Since the glow plasma is strongly non-thermal and the mean free path of electrons is few order of magnitude higher than in atmospheric pressure discharges, it can be concluded the ionization processes are

primarily caused by electron impact processes. However, detection of N₂ first positive system indicates the presence of long-life nitrogen metastables, which act as an energy reservoir in the plasmachemical processes. In the higher altitudes of Titan's atmosphere such metastable molecules have an important role because the long mean free path allows them to be diffused to higher or lower spheres thus inducing an organic chemistry. Absence of atomic hydrogen emission lines suggests a fast consumption of hydrogen atoms/molecules during the product formation.

Results will have a good contribution in numerical modeling of reaction kinetics.

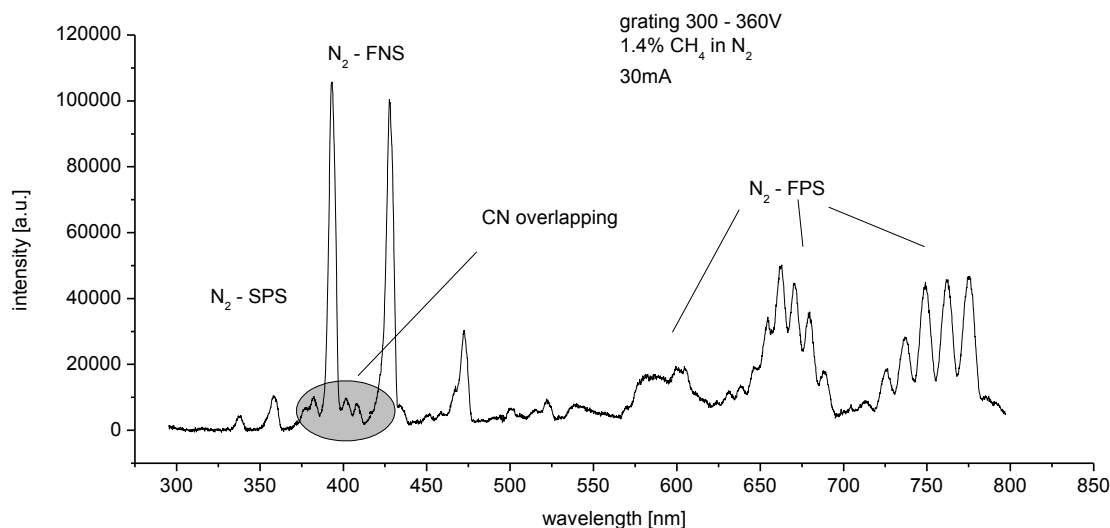


Fig. 2: Example of a N₂-CH₄ glow discharge spectra. FPS – first positive system of N₂; FNS – first negative system of N₂; SPS – second negative system of N₂.

To be condensed enough products in kryotrap, it was necessary to wait several tens of hours, in this case 30 to 50 hours. After the experiment kryotrap was overheated gradually to room temperature and the condensed products reaching the gas phase temperature were penetrating to the infrared cell which has been placed directly in the FTIR spectrometer. A typical FTIR spectrum of desorbed products is shown in figure 3. After a detailed analysis of FTIR spectra it shows the presence of following discharge products: NH₃, HCN, C₂H₄, and HC₃N.

After 35 hours a thin layer of brown deposits was formed on the surface of carbon film. As shown in Figure 4, XPS analysis confirmed the presence of several metals such as iron - Fe, nickel - Ni, aluminum – Al, and platinum - Pt, but also elements like carbon - C, nitrogen - N and oxygen - O. The presence of metals in the carbon films is due to sputtering of aluminium/steel components in the discharge gap.

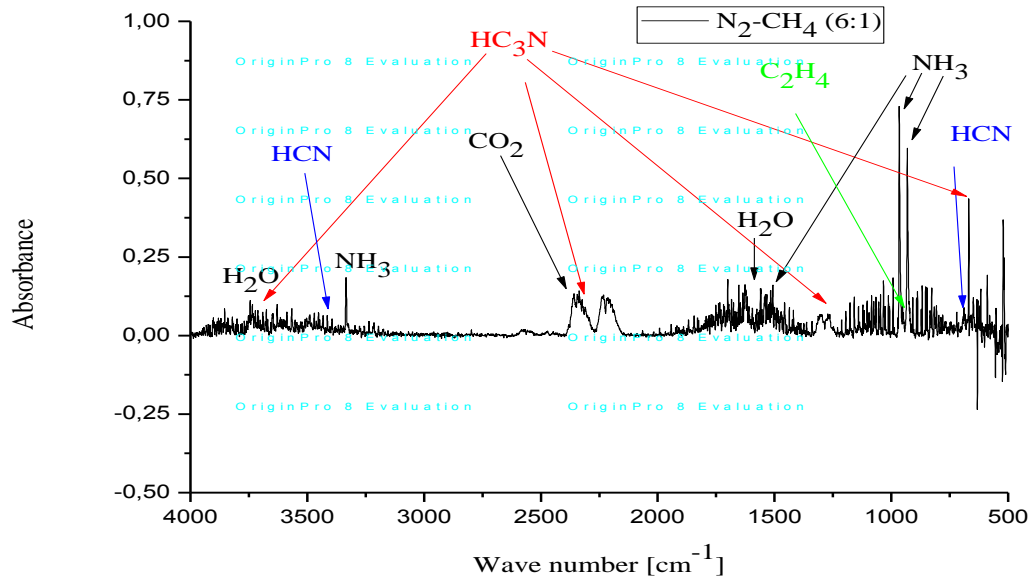


Fig. 3: FTIR spectra of the discharge products in N₂-CH₄ (6:1) gas mixture at pressure 0.7 mbar and electric current 40 mA.

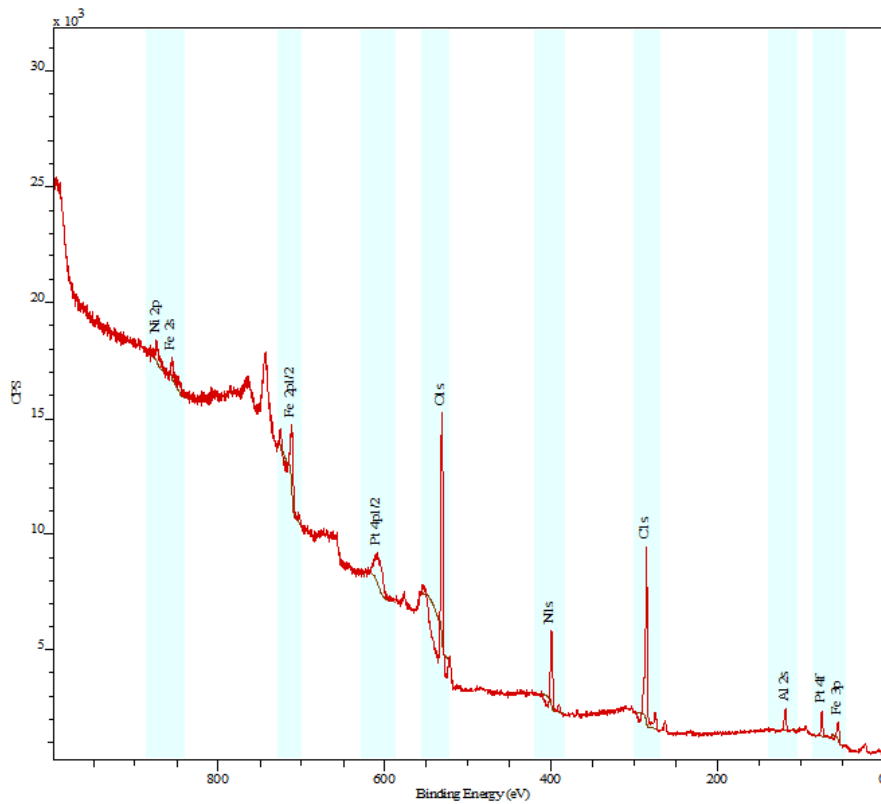


Fig. 4: The measured XPS spectrum of deposits in glow discharge in a mixture of CH₄-N₂.

Please include:

- Publications arising/planned (include conference abstracts etc)

The results obtained in glow gliding arc discharge are planned to be published in ICARUS, PSST and EPJ-D journals.

- Host approval The host is required to approve the report agreeing it is an accurate account of the research performed.