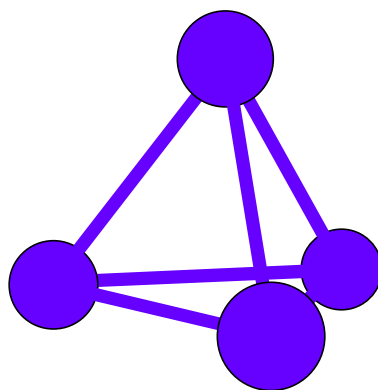


The N₄ project. Report for the first quarter of 2001.

O. Launila, H. Östmark, R. Tryman,
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Progress report



WEAPONS AND PROTECTION
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O. Launila, H. Östmark, R. Tryman,

S. Wallin, G. Petri, A. Pettersson

FOI, Swedish Defence Research Agency

Weapons and Protection

Department of Energetic Materials

Grindsjön, 147 25 Tumba, Sweden

Progress report

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Introduction

During the first quarter of 2001, the main efforts within the N₄ project have been concentrated to electric discharge excitation, ion-bombarding and microwave excitation experiments on cryogenic nitrogen-containing matrices. Several synthesis attempts have been made using long integration times in order to suppress random noise. Attempts to excite liquid nitrogen with a 193 nm ArF laser have been performed. Initial temperature-controlled experiments have been performed on products from microwave discharge condensed on a sapphire window in a small cryostat.

Synthesis experiments in cryogenic matrices

After the initial experiments with ion gun (VG Microtech AG5000 ION/8) for the production of N_2^+ , N^+ or positive rare gas ions for direct bombardment of cryogenic nitrogen-containing matrices, two additional means of matrix excitation have been tested. A microwave cavity of commercial design (SAIREM GMP 03 KE/D) and a laboratory-built small hollow cathode device. All three devices are mounted simultaneously to the same cryochamber and can be used in parallel. Figs. 1 and 2 show pictures of the cryostat with the microwave cavity running on pure neon.

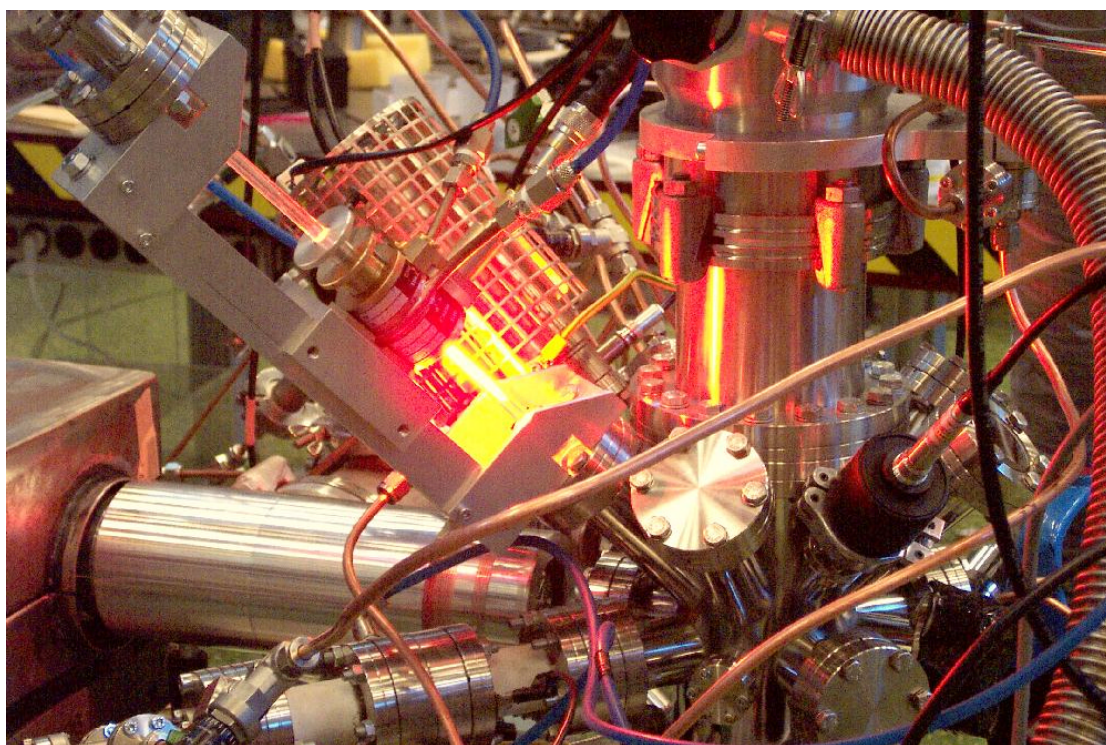


Fig. 1. Three matrix excitation devices, mounted on the same cryostat. The miniature hollow cathode is seen in the lower left. In the middle, the microwave cavity is seen running on pure neon. The protection shield of the ion gun is seen behind the discharge. The aluminum tube below the ion gun contains the lens system (cf. Fig. 3)

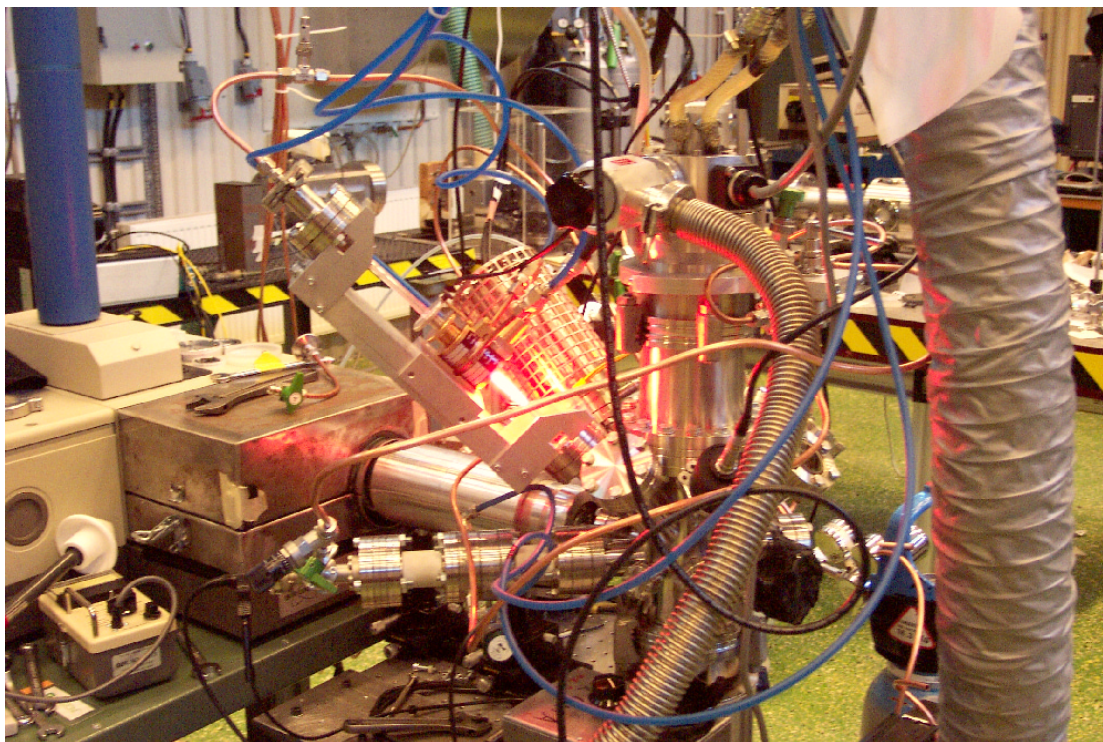


Fig. 2. Another view of the cryostat. The FT-Raman interferometer (Bruker IFS55/FRA106) is seen to the left.

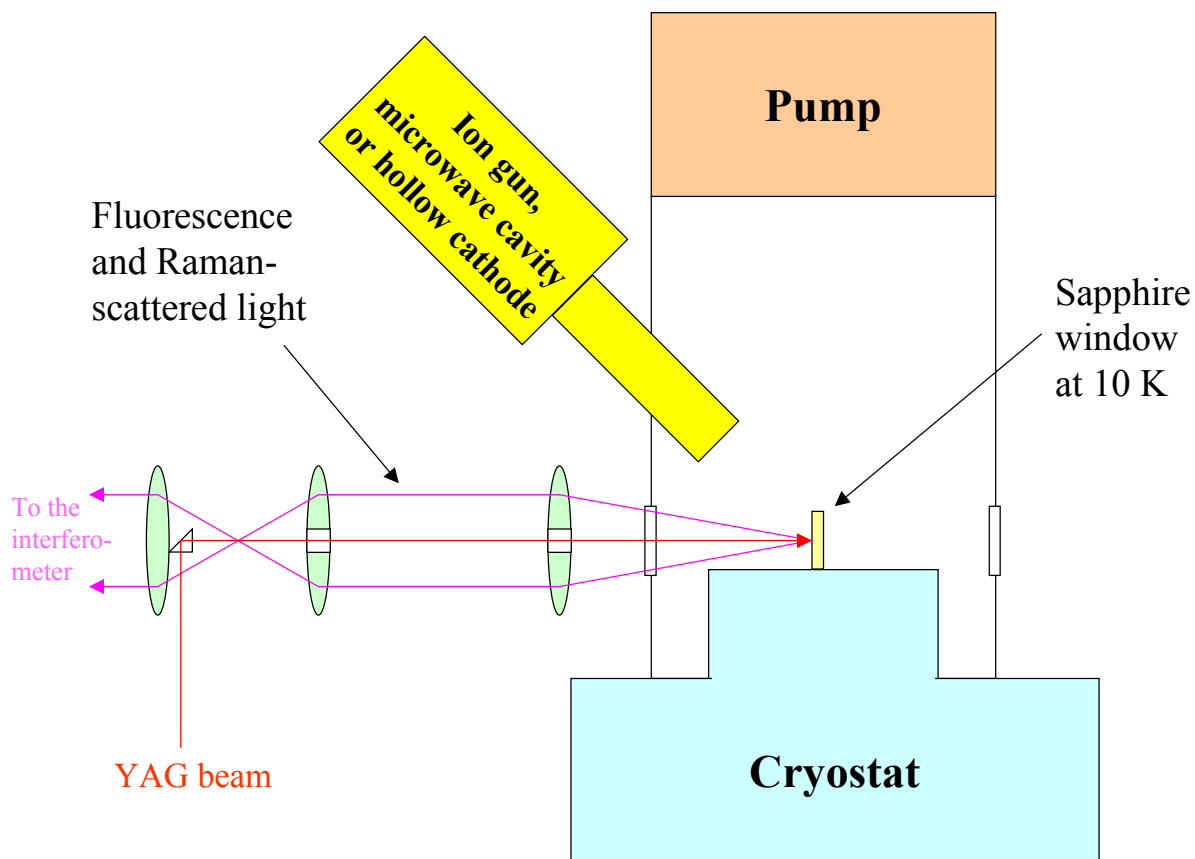


Fig. 3. Schematic arrangement of the matrix excitation experiments. A cryogenic matrix, consisting of pure nitrogen or a mixture of nitrogen with different nitrogen-containing species is first deposited on the sapphire window. During the deposition, the Raman signal is continuously monitored with a FT-Raman interferometer. When a desired matrix thickness has been reached, the matrix is bombarded with ions (N_2^+ , N^+ , Ar^+ , Ne^+ or He^+) or corresponding products from a microwave or a hollow cathode discharge. The resulting matrix fluorescence is monitored with an IFS55/FRA106 FT-Raman interferometer and a Mechelle 900 UV-VIS-NIR spectrometer.

When the cryostat cold finger is cooled and when the discharge contains enough nitrogen, the deposited nitrogen glows in bright green (Fig. 4)



Fig. 4. The cryostat sapphire window, photographed under nitrogen deposition from microwave discharge. The sapphire window is located in the circular aperture of the rectangular copper block, mounted on the cryotip. The transparent, very thin nitrogen matrix is photographed from behind. The magenta-colored orifice of the microwave capillary is seen to the upper left. The green color arises from a ${}^2D-{}^4S$ transition in nitrogen atoms in the deposited solid, while the magenta color of the capillary tube is due to the $C^3\Pi_u-B^3\Pi_g$ and $B^3\Pi_g-A^3\Sigma_u^+$ emissions in N_2 .

Numerous synthesis experiments have been carried out on excited, nitrogen-containing matrices during the first quarter of 2001. All three excitation devices mounted on the cryostat have been used in these experiments. Three different types of recordings have been made:

1. *Recordings of a matrix during the excitation phase (relatively short recordings during several hours of matrix build-up).*
2. *Long-time recordings of the matrix after excitation (FT-Raman)*
3. *Short time series during controlled warm-up of the excited matrix.*

Different types of information can be collected in these experiments. During the excitation phase, mixed spectra of gas-phase and matrix processes are obtained with the FT-Raman instrument either in Raman mode (laser on) or in infrared emission mode (laser nearly off). Simultaneous spectra are recorded with the Mechelle 900 UV-VIS-NIR spectrometer. Together, these instruments cover the wavelength range of 200-1800 nm. The gas phase spectra most often consist of well-known molecular N_2 or N_2^+ bands, together with rare gas atomic emission spectra in the cases where mixtures of rare gases and N_2 have been used. In the case of microwave discharges, the $B^3\Pi_g-A^3\Sigma_u^+$ system of N_2 usually dominates, while the hollow cathode device also gives N_2^+ , as well as higher-lying excited states of N_2 . An example can be seen in Fig. 5. The matrix emission mostly consists of atomic N spectra ($2P-2D$ and $2D-4S$), bearing witness of atom-matrix interactions through the occurrence of characteristic vibron- and phonon-induced sidebands.

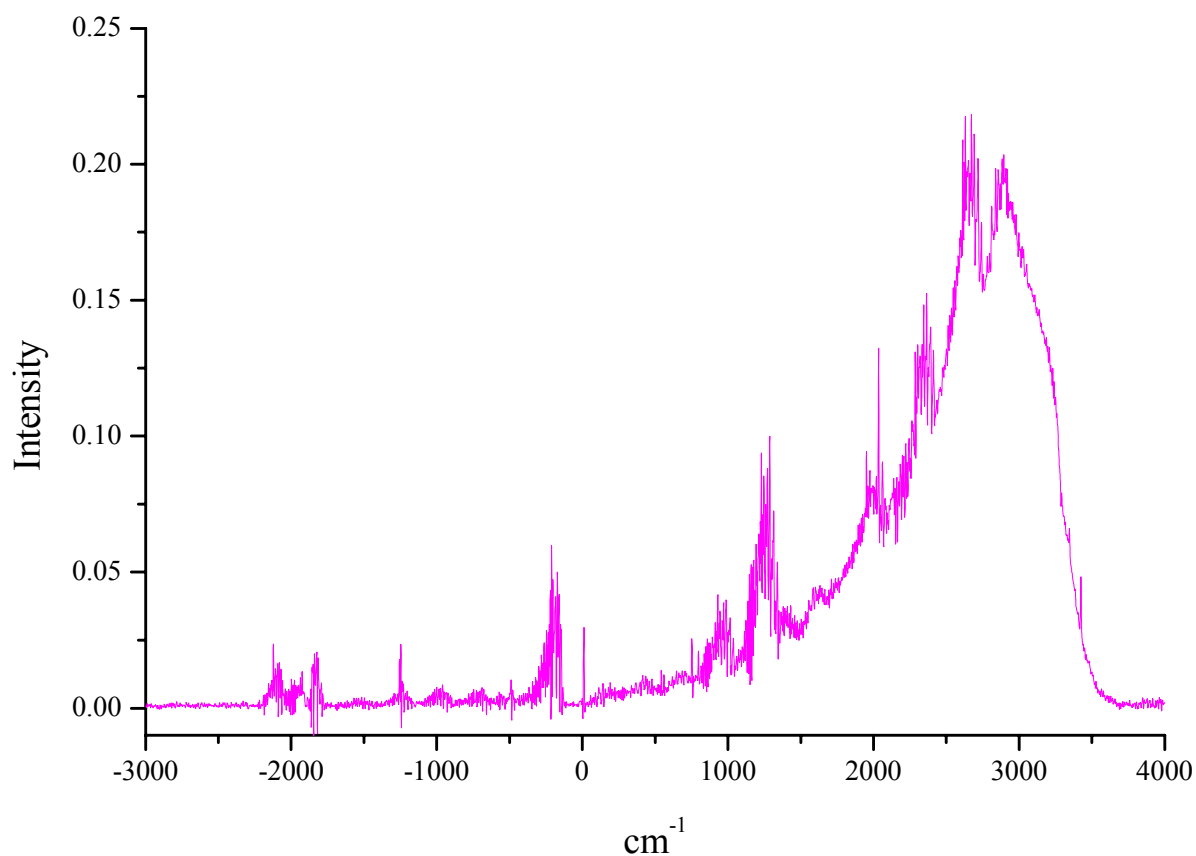


Fig. 5. A short (100 scans) FT-Raman recording of a N₂ matrix during deposition from a microwave discharge (130 W). The molecular bands are due to the $B^3\Pi_g-A^3\Sigma_u^+$ system of N₂. The YAG power was 350 mW and resolution 4 cm⁻¹.

In the long-time recordings (exceeding 14 hours), only the FT-Raman instrument has been used. These recordings give highly puzzling spectra, where the most dominating features are still unassigned. One of the unassigned features is a characteristic triplet, consisting of two close-lying strong lines and a weaker one, shown in Fig. 6.

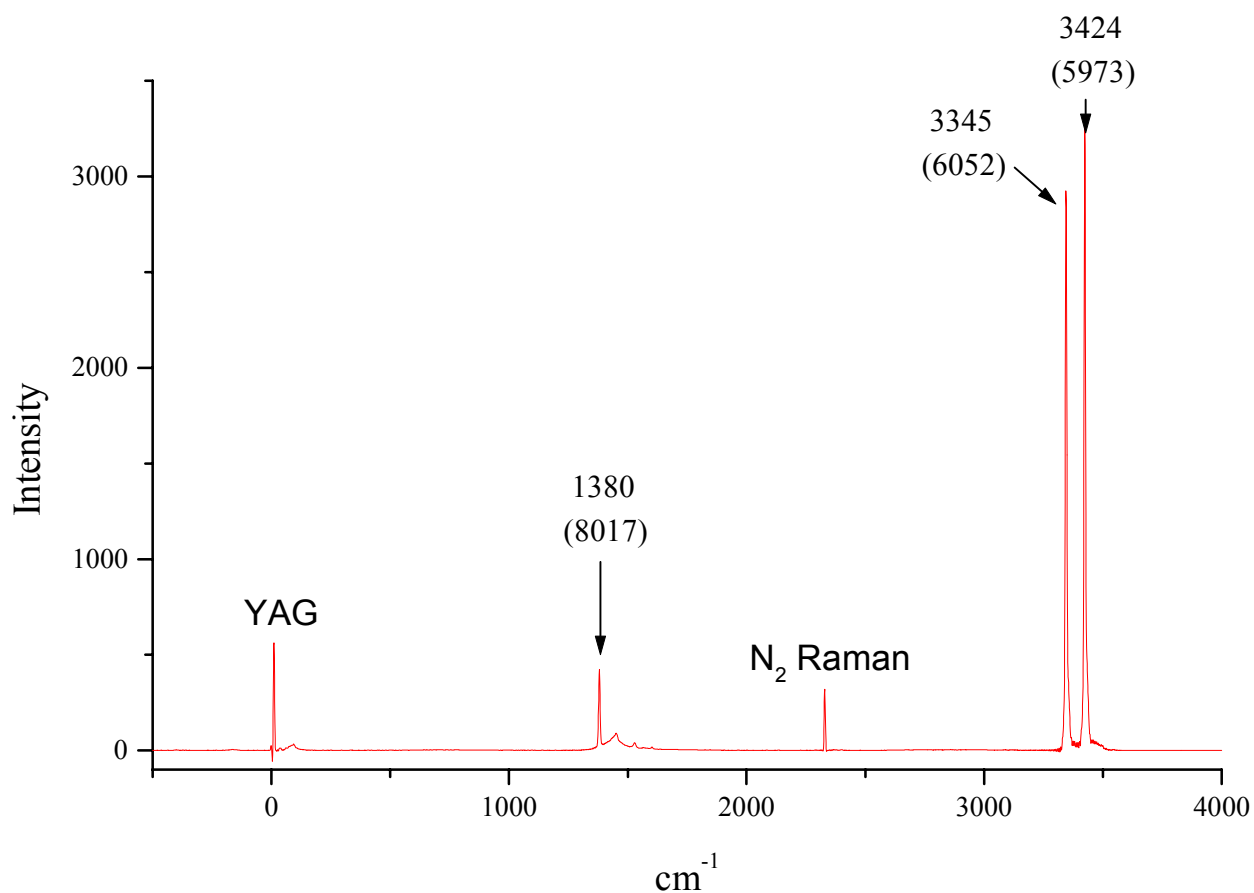


Fig. 6. A long-time FT-Raman recording (28000 scans in 14 hours) of a microwave discharge-excited N_2 matrix. The apparent wavenumbers of the triplet are shown, as given by the interferometer software, together with the true fluorescence wavenumbers in parenthesis. The YAG power was 500 mW and the resolution 4 cm^{-1} .

This triplet is laser-induced, and its intensity is linearly dependent on the YAG power (Fig. 7).

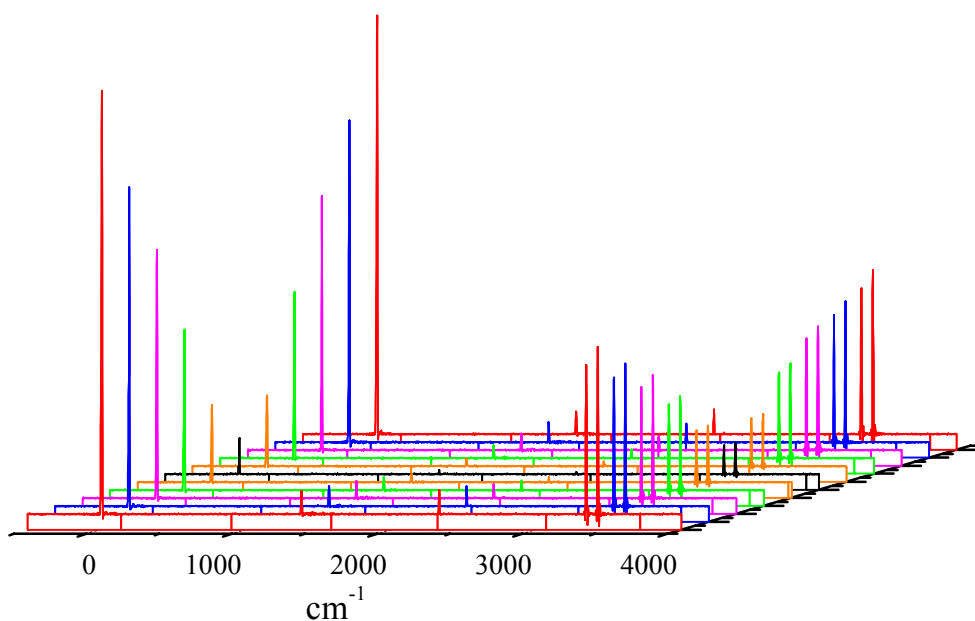


Fig. 7. The laser power dependence of the unassigned triplet. The YAG power was varied between 50 mW and 500 mW. As can be seen, the two strong lines of the triplet follow the laser intensity linearly. The sample is the same as in Fig. 6, but the imaging was inferior, the signal containing more scattered laser light from the lens system. Very slight saturation of the fluorescence signal occurs at the higher power levels.

This behavior suggests that the excitation is due to a one-photon process. However, in matrix environment this conclusion might be erroneous. Clearly, the triplet is not due to a Raman process, as has been shown in isotopic substitution experiments (Fig. 8).

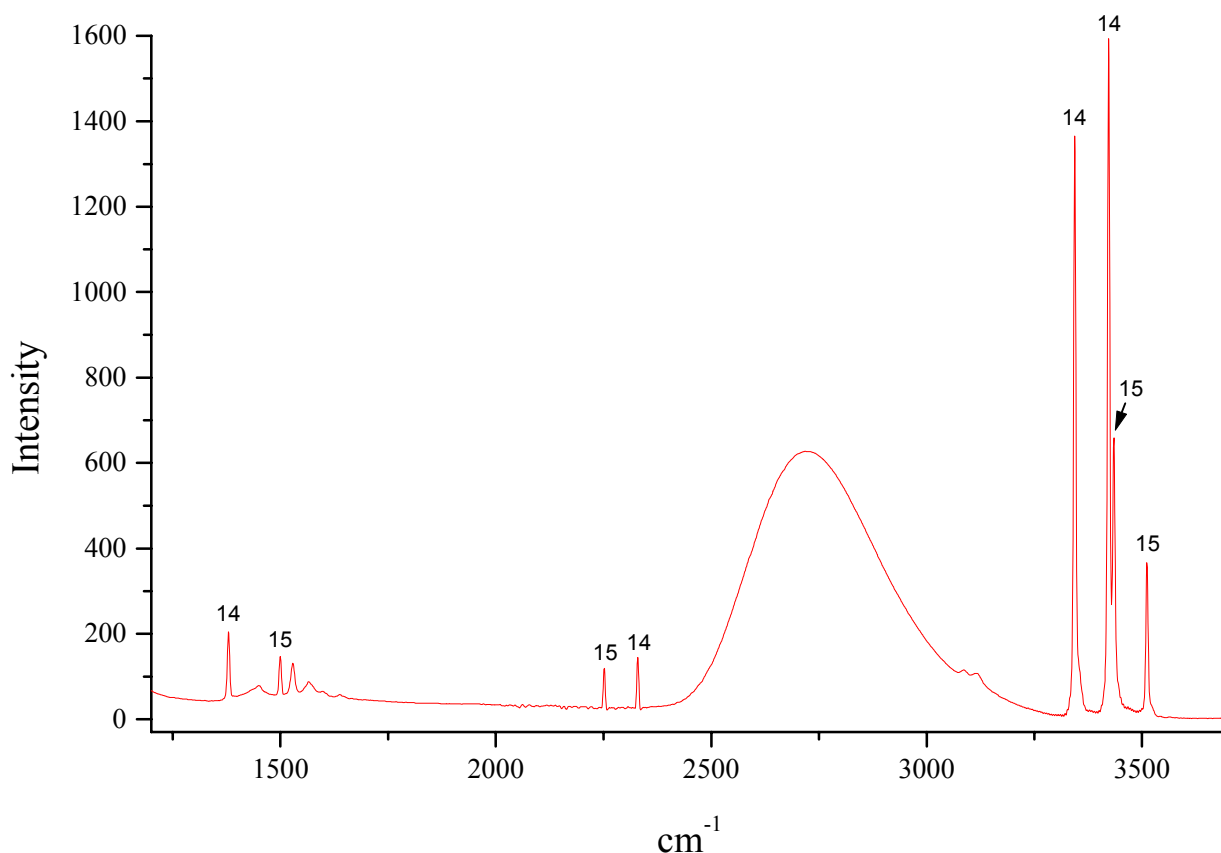


Fig. 8. Long-time FT-Raman recording (33000 scans in 16½ hours) of a $^{14}\text{N}_2+^{15}\text{N}_2$ matrix, excited by ion gun, running on pure $^{14}\text{N}_2$, pure $^{15}\text{N}_2$ and on a mixture of these. The two lines in the 2300 cm^{-1} region are the N_2 Raman lines for both isotopomers. No sign of $^{14}\text{N}^{15}\text{N}$ was seen. The fact that the triplet lines move to higher (apparent) wavenumbers, means that they cannot be Raman lines. The YAG power was 500 mW and the resolution 4 cm^{-1} .

The triplet occurs in all three types of excitation, being strongest in hollow cathode- and microwave-excited matrices. The relative intensities within the triplet are 5, 44 and 51 percent of the total intensity carried by the triplet. As a comparison, we can note that the N_2 Raman line carries only about 3 per cent of this intensity. Unfortunately, we cannot convert these values to concentration information for the

unknown species, but it does not appear to be very bold to suggest that we are dealing with concentrations clearly exceeding ppm-levels.

The triplet has been investigated more closely in an isotopic replacement experiment. The ion gun was first run with pure $^{14}\text{N}_2$ in order to establish proper conditions for the triplet build-up. When these conditions were found, pure $^{15}\text{N}_2$ was introduced in the ion gun. We observed that the N_2 Raman line moved as it should (towards lower Raman wavenumbers), while the triplet lines moved to the opposite direction (Fig. 8). This behavior is consistent with the assumed fluorescence origin of the triplet. In the following table, the true wavenumbers are used for these lines.

σ_{14}	σ_{15}	σ_{14}/σ_{15}	Commentary
2329.29	2251.59	1.0345	N_2 Raman
8016.95	7897.07	1.0152	LIF (weak)
6052.33	5961.25	1.0153	LIF (strong)
5973.26	5884.51	1.0151	LIF (strong)

What could be considered as intriguing, is that only two different triplet spectra are observed, despite the fact that the matrix contains $^{14}\text{N}_2$ and $^{15}\text{N}_2$ in approximately equal concentrations, as can be seen by looking at the intensities of the N_2 Raman lines. According to our opinion, this clearly excludes N_4 as a carrier (rectangular or tetrahedral), since such a molecule should occur in at least three different isotopic configurations in the matrix ($^{14}\text{N}_2+^{14}\text{N}_2$, $^{14}\text{N}_2+^{15}\text{N}_2$ and $^{15}\text{N}_2+^{15}\text{N}_2$), which should result in three distinct spectra. On the other hand, the carrier must contain nitrogen in a rather tightly bound position, because the isotopic shift is quite substantial. A comparison with diatomics shows that the CN radical has a similar isotopic shift (1.5 per cent). There is always a possibility for the occurrence of CN in ion gun spectra, since the gun contains a carbon disk in the discharge cell. The CN assignment, however, is not supported by data from carbon-free experiments (microwave- and hollow cathode-discharge), where the triplet is actually even stronger than in the ion gun experiments.

In this context, we should mention that the weak LIF line at 8016.95 cm^{-1} is always accompanied by a broad satellite on the low wavenumber side at 7946.53 cm^{-1} . It might be tempting to assume that this satellite is simply predissociated or otherwise broadened, and that we are actually having a quadruplet and not a triplet of LIF lines. However, a severe objection to this conclusion is that the intensity of this broad feature varies between different experiments, while the relative intensities of the remaining three lines remain unaffected, corrections having been made for the different sensitivity of the Ge detector in different temperatures (see below).

We have considered the possibility of the linear N_3 radical, where spectroscopic data are abundant, as well as the hitherto not reported triangular N_3 radical. Calculations on this second species are underway - preliminary results suggest that it might be bound. If so, we could have a new scheme for the $\text{N}_4(T_d)$ synthesis: $\text{N}_3 + \text{N} \rightarrow \text{N}_4$.

At this stage, any conclusions must be supported by additional ^{15}N -data. For instance, experiments with $^{14}\text{N}^{15}\text{N}$ -enriched nitrogen samples must be made.

In a previous report, we reported anomalous intensity behavior for the two strong lines of the triplet (Fig. 28 in Ref. [1]). This behavior has now been shown to be due to increasing sensitivity of the Ge-detector at temperatures slightly above 77 K. The anomalous intensities always occurred towards the end of long-time recordings, when the liquid nitrogen level in the detector Dewar had decreased below a certain level. This usually happened after some 12 hours of continuous recording, i.e. during early morning hours. Interestingly, the sensitivity enhancement seems to be limited to a rather narrow band around $1.66\text{ }\mu$. The enhancement is substantial, as can be seen in Fig. 9. We have checked that the behavior is reversible by pouring in more liquid nitrogen in the Dewar, and noticing a corresponding decrease in sensitivity.

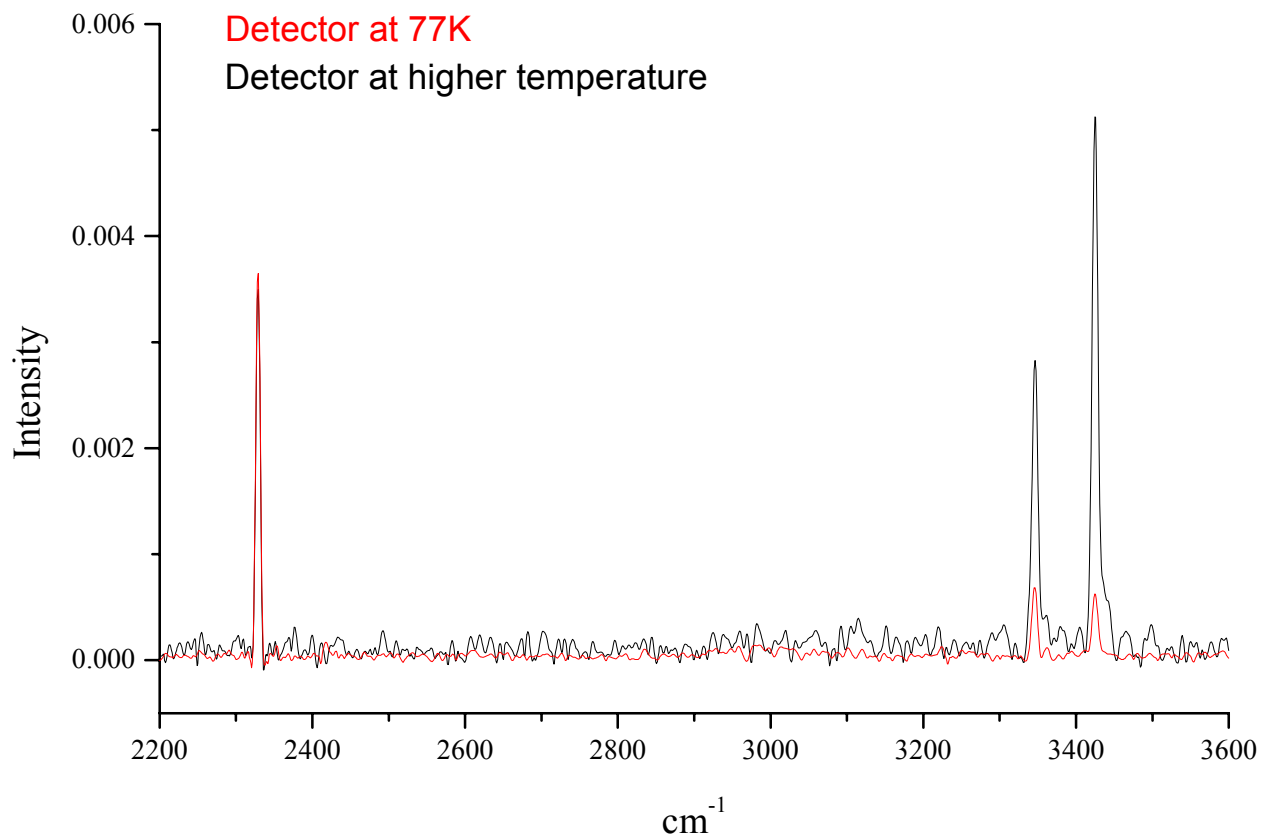


Fig. 9. Sensitivity enhancement of the Ge detector in the 1.66 μ region, manifested by the dramatic increase of the doublet intensity, while the N₂ Raman signal, corresponding to 1.4 μ , remains unaffected.

The third type of experiment consists of heating the matrix from 10 K to 30 K by gradually applying an increasing current to a resistor, in thermal contact with the matrix. These experiments often give characteristic structured emissions in the infrared and in the visible. These processes can largely be explained by recombination schemes, where free nitrogen atoms are involved. A pair of $4S$ nitrogen atoms lies at an energy of 9.8 eV, compared with a N_2 molecule in the ground state. A process like $N(4S) + N(4S) \rightarrow N_2(A^3\Sigma_u^+)$ is exothermal by several eV. The 6.2 eV energy stored in $N_2(A^3\Sigma_u^+)$ is subsequently used to pump up N atoms to the $2P$ or $2D$ states at 2.4 and 3.6 eV, respectively, causing the intensive green ($2D \rightarrow 4S$) and IR ($2P \rightarrow 2D$) emissions, which persist as long as fresh $N(4S)$ supply is guaranteed. Another aspect of this process, dealing with α -particle excitation of N_2 , has been described in Ref. [1]. Many features in the warm-up spectra coincide with the spectra recorded under excitation, although the relative intensities differ considerably.

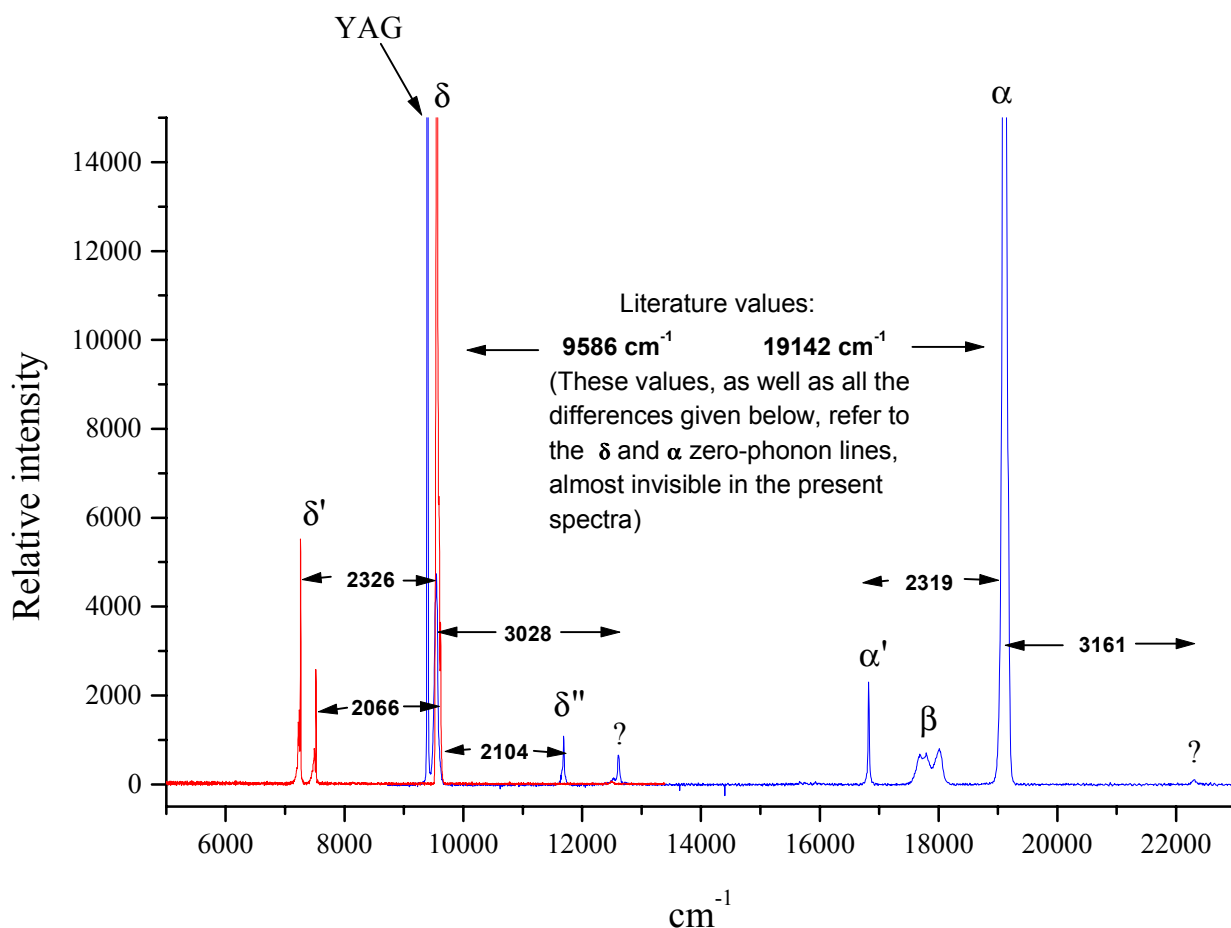


Fig. 10. Combined FT- and mechelle-spectra during the warm-up of a nitrogen matrix. The α and δ lines correspond to the atomic nitrogen $^2D-^4S$ and $^2P-^2D$ transitions, respectively. The α' and δ' lines are corresponding vibrational satellites. Several weaker lines near the δ line have not been assigned. The β line has been assigned as a $^1S-^1D$ transition in atomic oxygen. The oxygen atoms are presumably released from the quartz capillary through the influence of the discharge. The YAG power was very low (8 mW). The integration time was 30 s.

In Figs. 11 and 12a, another type of experiment is shown. This warming-up spectrum was recorded using a three-layer N_2 matrix, deposited with neutral, hollow-cathode excited and neutral layers, respectively. This experiment was performed in order to investigate the possibility of surface phenomena as being the cause of the occurrence of the triplet.

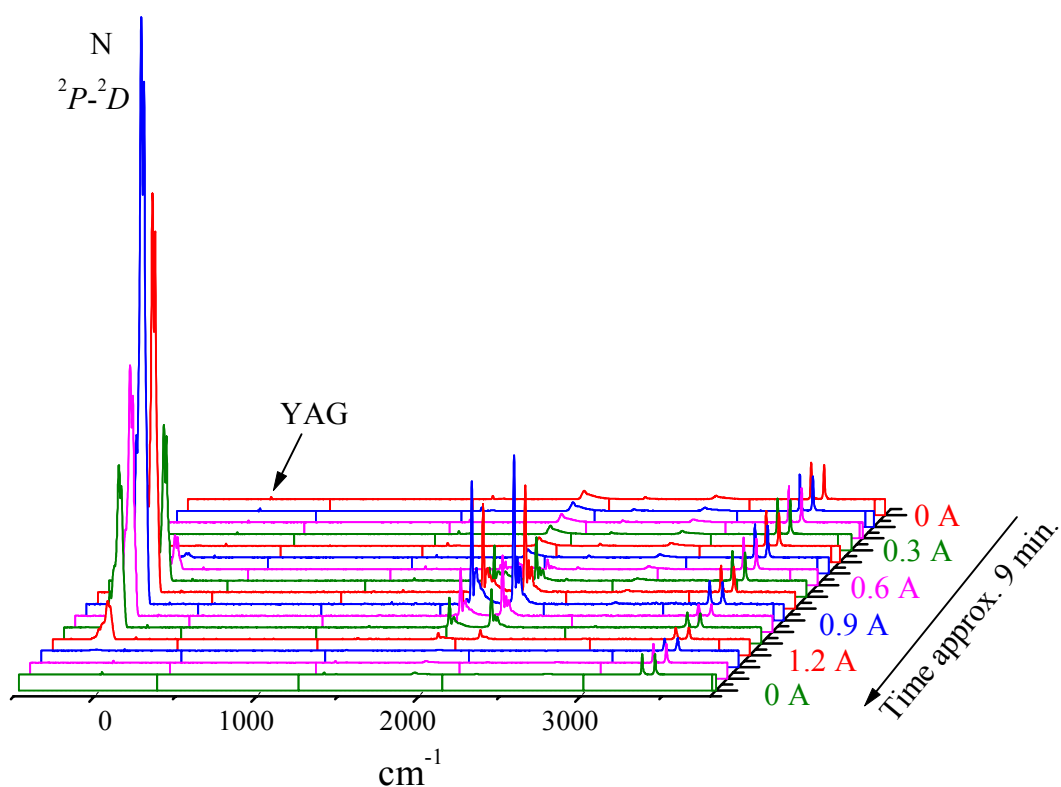


Fig. 11. A warming-up series of a hollow-cathode excited, layered N_2 matrix. The matrix was constructed by deposition of consecutive neutral, excited and neutral N_2 layers, respectively. The atomic N signal was unusually strong in this recording, as can be seen upon comparison with the residual YAG line. The laser power was 500 mW and resolution 4 cm^{-1} .

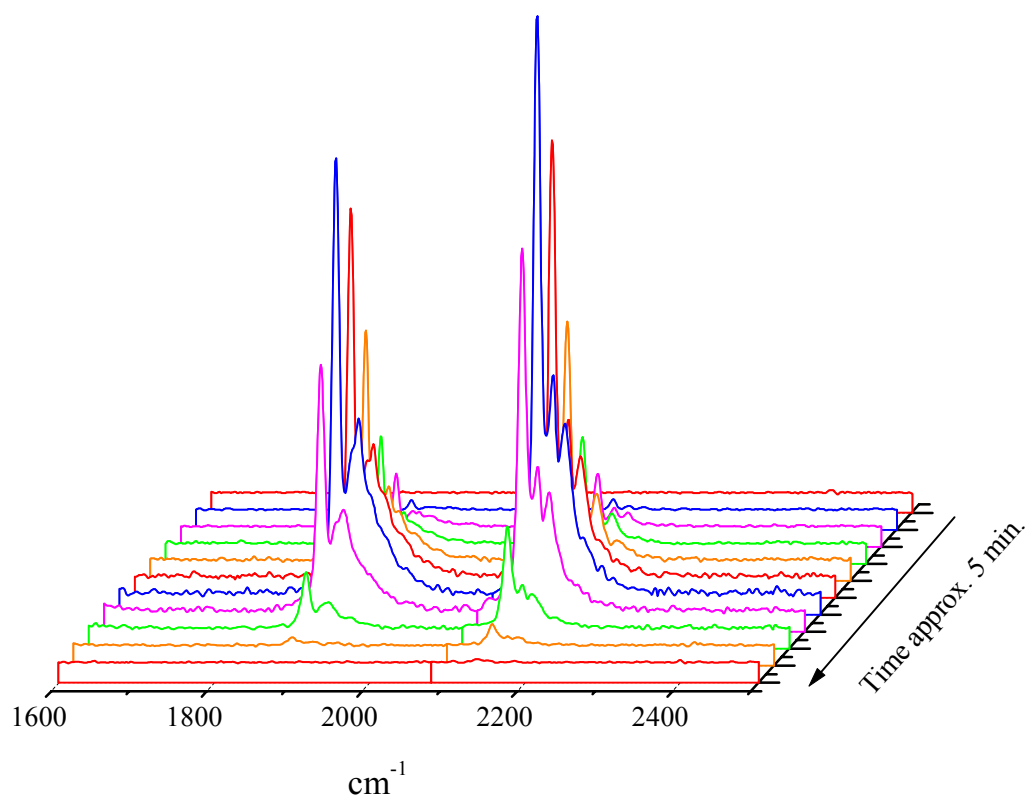


Fig. 12a. Portion of a warming-up spectrum of a microwave-excited N₂ matrix. Compare the relative intensities of the two bands with those of Fig. 12b.

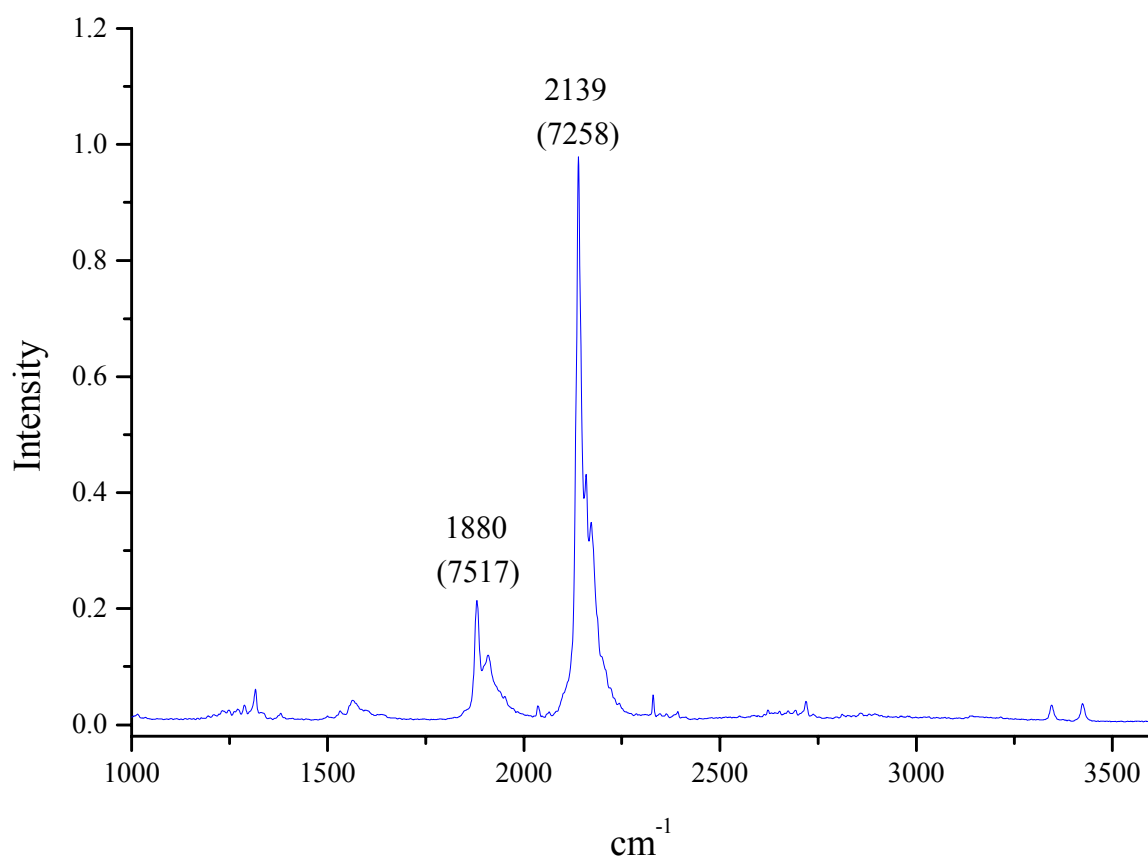


Fig. 12b. Spectrum of an ion-gun excited N₂ matrix during the excitation phase. Compare the relative intensities of the two bands with those of Fig. 12a.

Yet another type of experiment consists of switching off the excitation source and recording time series (Fig. 13). Long integration times can be used since some of the de-excitation processes are very slow (30-100 s). An average of two of the spectra of the time series of Fig. 13 is shown in Fig. 14. The unassigned features in these spectra are probably due to different species.

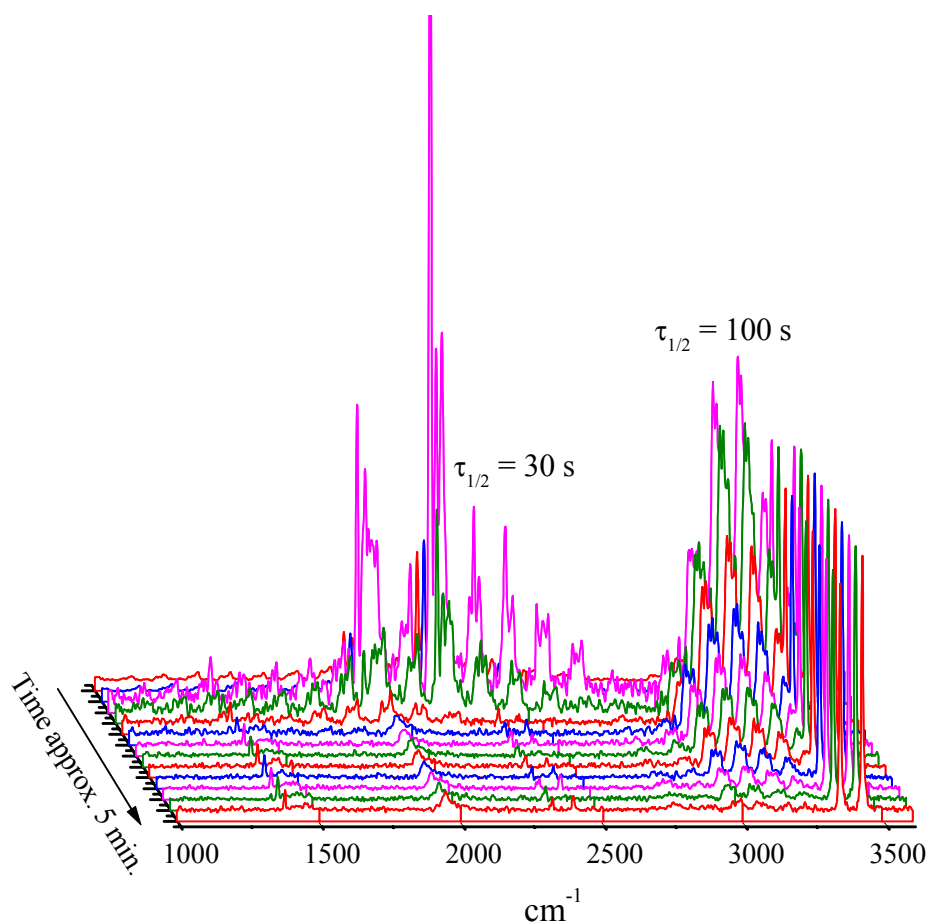


Fig. 13. A time series of excited N_2 matrix at 10 K, recorded after sudden switching-off of the hollow cathode excitation discharge. The spectra represent consecutive 20-scan FT-Raman recordings with a YAG power of 500 mW and resolution 4 cm^{-1} . Two characteristic decay times are observed for the two different (unassigned) band systems in the 2000 cm^{-1} and 3200 cm^{-1} regions, respectively. The conclusion is that there are two different carriers involved.

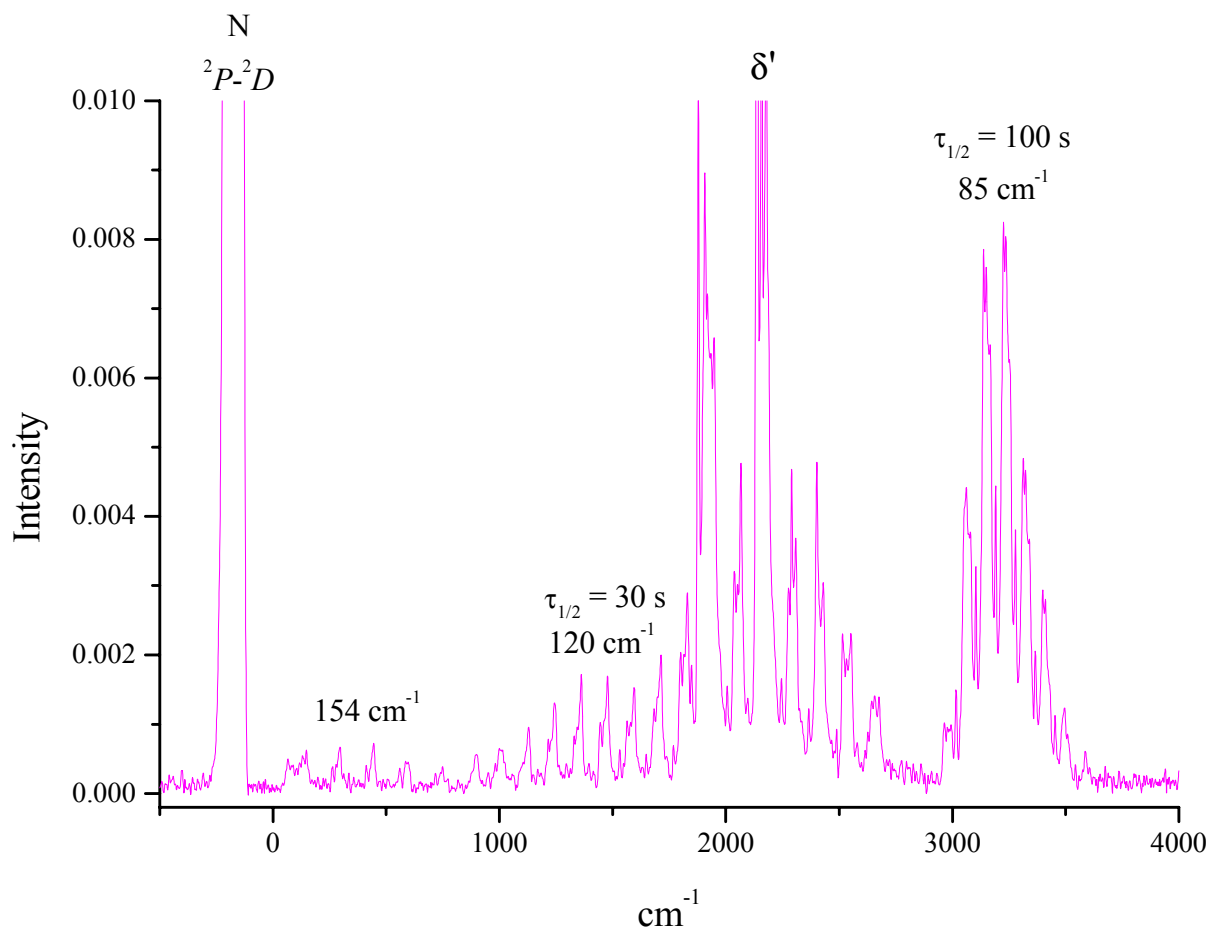


Fig. 14. An average of two of the spectra in the time series of Fig. 13. The wavenumber designations in the spectra refer to the characteristic sub-band separations within the corresponding band systems. In the case of the 3200 cm^{-1} band system, the sub-bands can be fitted to a first-degree polynomial, while a second-degree polynomial is needed for the 2000 cm^{-1} system.

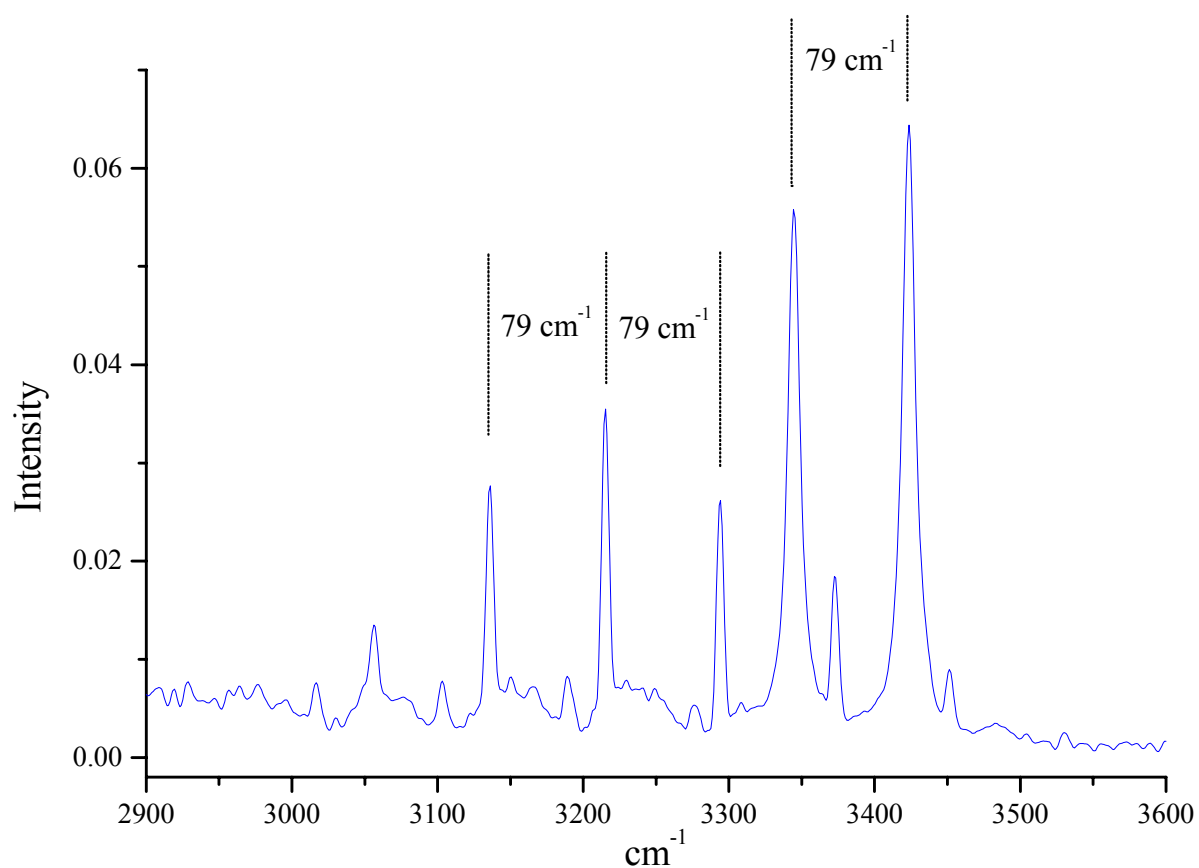


Fig. 15. Unassigned structures in a FT-Raman recording during matrix build-up from a microwave discharge. The spectrum is an average of 900 scans, recorded during approximately 30 minutes at a resolution of 4 cm⁻¹ and slightly varying conditions. The broad and narrow lines do not appear to be related, despite the identical 79 cm⁻¹ separations. See further in Fig. 16.

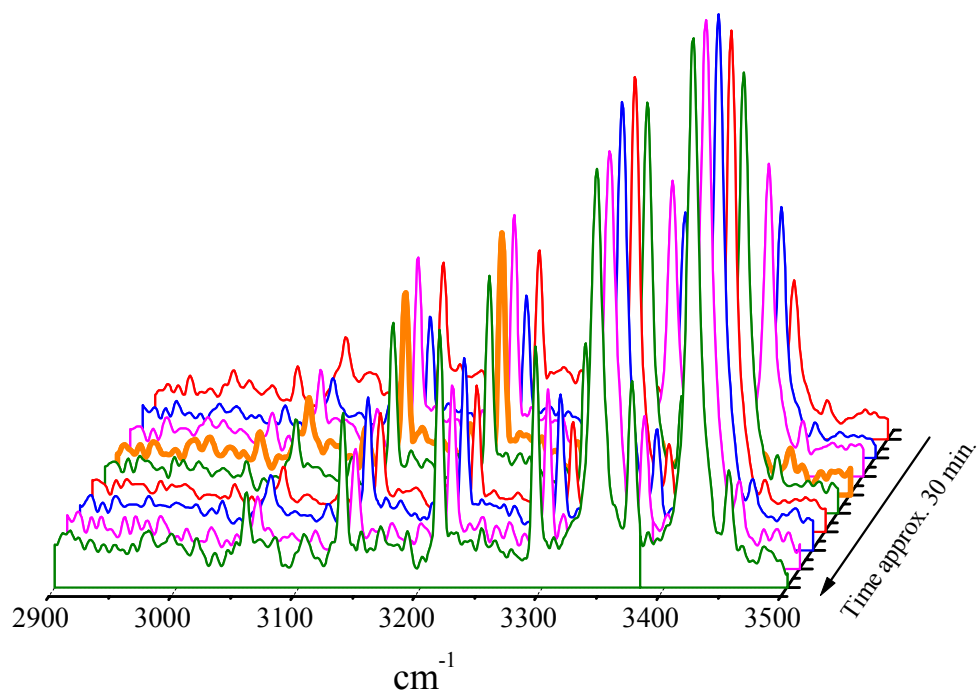


Fig. 16. A series of FT-Raman recordings (9x100 scans) of a microwave discharge-excited N_2 matrix during the matrix build-up phase. As can be seen, the time behaviors of the 3400 cm^{-1} doublet and the narrow lines in the 3200 cm^{-1} band are different. In fact, the intensity fluctuations of the narrow lines mirror exactly the fluctuations of the atomic $^2P-^2D$ line of nitrogen, not shown in this figure. The bold orange spectrum in the middle was recorded with low YAG power (5 mW), while the remaining spectra were taken at 500 mW laser power. The narrow lines are clearly not laser-induced.

Hollow-cathode discharge excitation of N_2 matrices gives rise to somewhat confusing spectra. In addition to the features occurring in microwave- and ion gun-excited matrices, several additional bands appear (Fig. 17). These bands are also laser-induced.

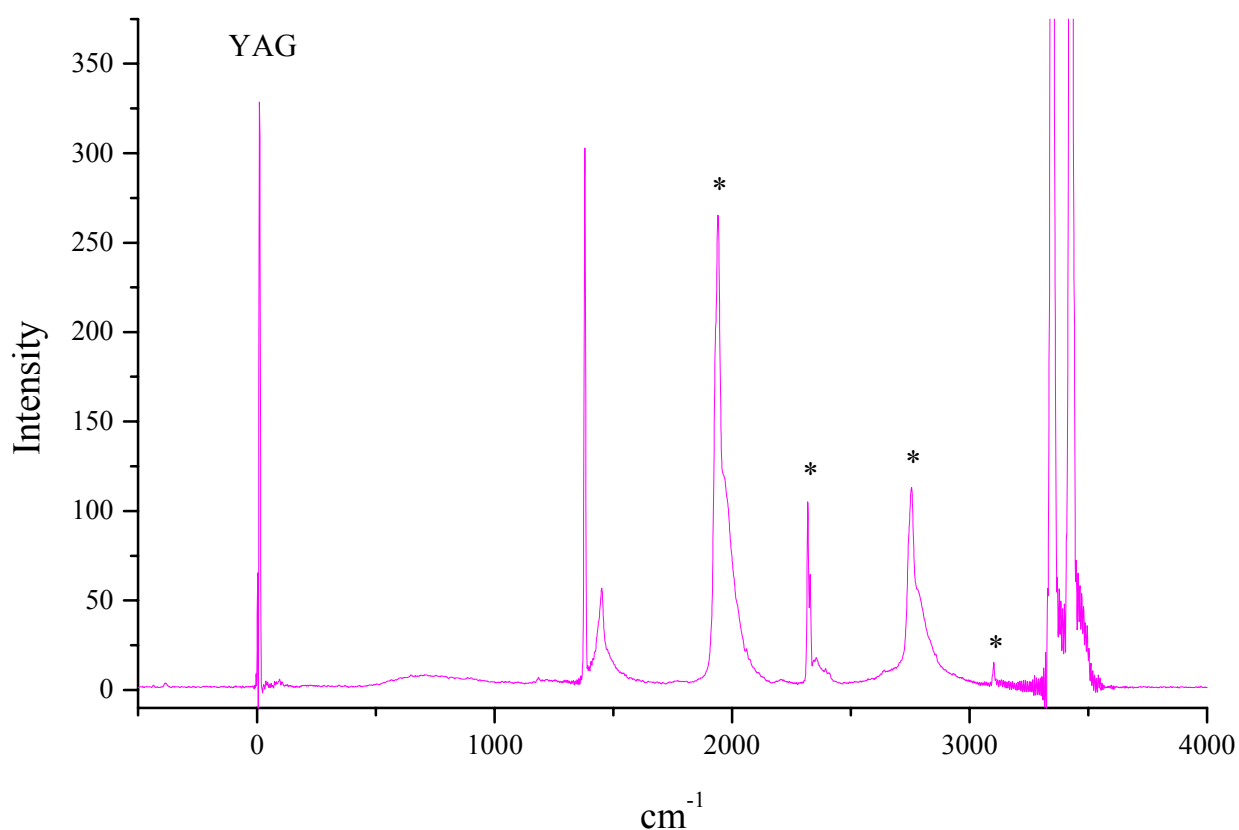


Fig. 17. New bands, marked with asterisks, appearing in hollow-cathode excited nitrogen matrices. The spectrum is an average of 36000 scans, recorded during 18 hours with a laser power of 500 mW at a resolution of 4 cm^{-1} . The spectrum has been expanded with a factor of 10 in the vertical direction.

Furthermore, the bands show a slow decay in long-time experiments, possibly related to diffusion-induced phenomena (Fig. 18).

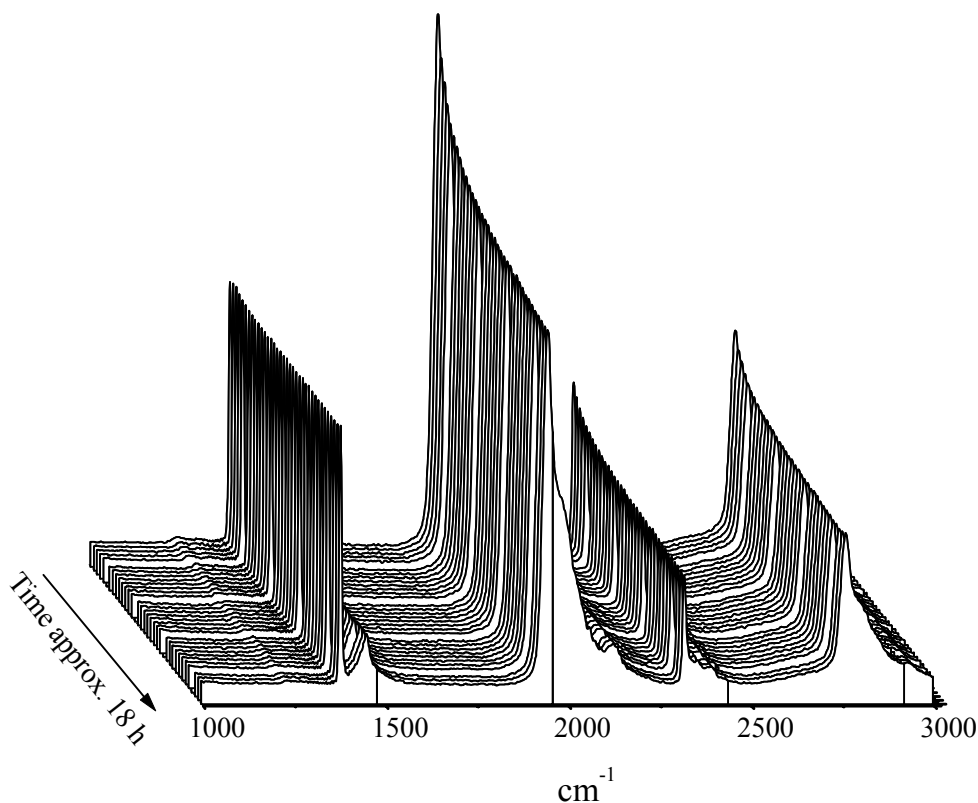


Fig. 18. Long time behavior of the hollow-cathode bands in N_2 matrix. Each spectrum is an average of 1000 scans, collected during 30 minutes. The laser power was 500 mW and resolution 4 cm^{-1} .

Upon annealing, the new bands disappear more readily than the triplet, which shows a temporary decrease, climbing up again to approximately 80 per cent of the original strength when the temperature is lowered (Figs. 19 and 20). The new bands, however, lose most of their intensity upon warming-up to 30 K. The triplet vanishes irreversibly at approximately 37 K, as can be seen in Fig. 21. Interestingly, this is approximately the temperature at which the α -nitrogen ceases to exist.

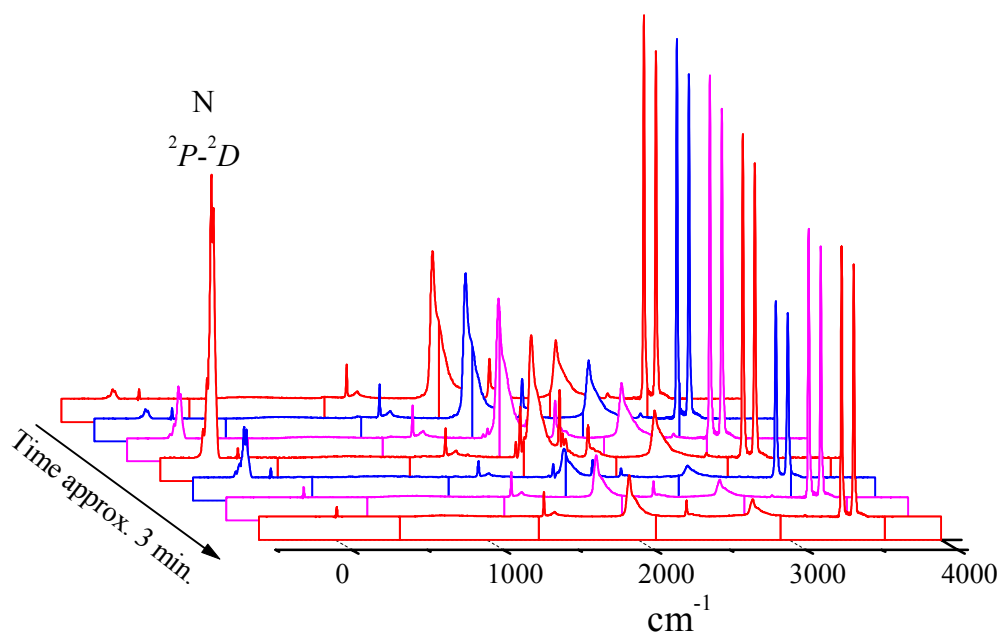


Fig. 19. A warming-up series ($10\text{ K} \rightarrow 30\text{ K} \rightarrow 10\text{ K}$) of a hollow-cathode excited N_2 matrix. The two broad bands at 2000 and 2800 cm^{-1} vanish more easily than the triplet lines. The laser power was 500 mW and resolution 4 cm^{-1} .

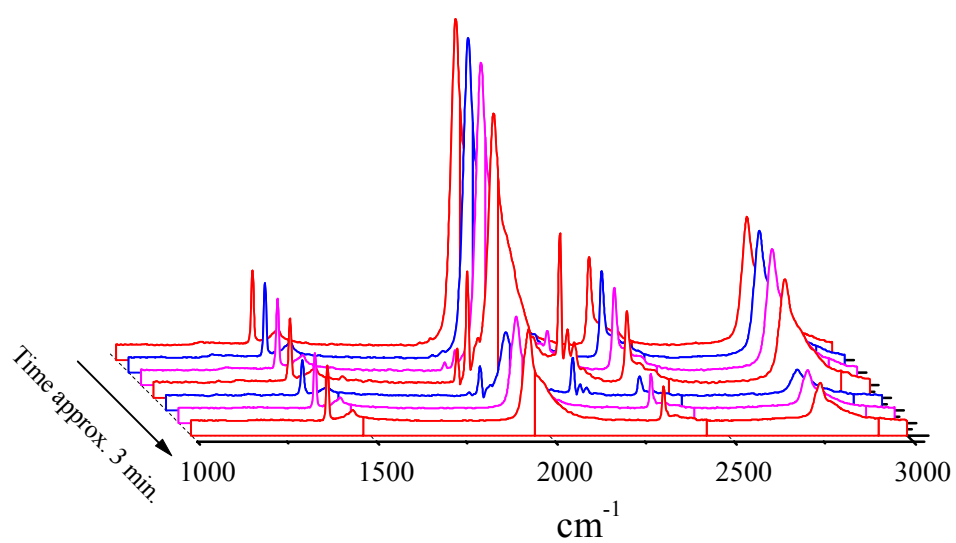


Fig. 20. A warming-up series ($10\text{ K} \rightarrow 30\text{ K} \rightarrow 10\text{ K}$) of a hollow-cathode excited N_2 matrix. The two broad bands at 2000 and 2800 cm^{-1} vanish more readily than the triplet line at 1380 cm^{-1} . The laser power was 500 mW and resolution 4 cm^{-1} .

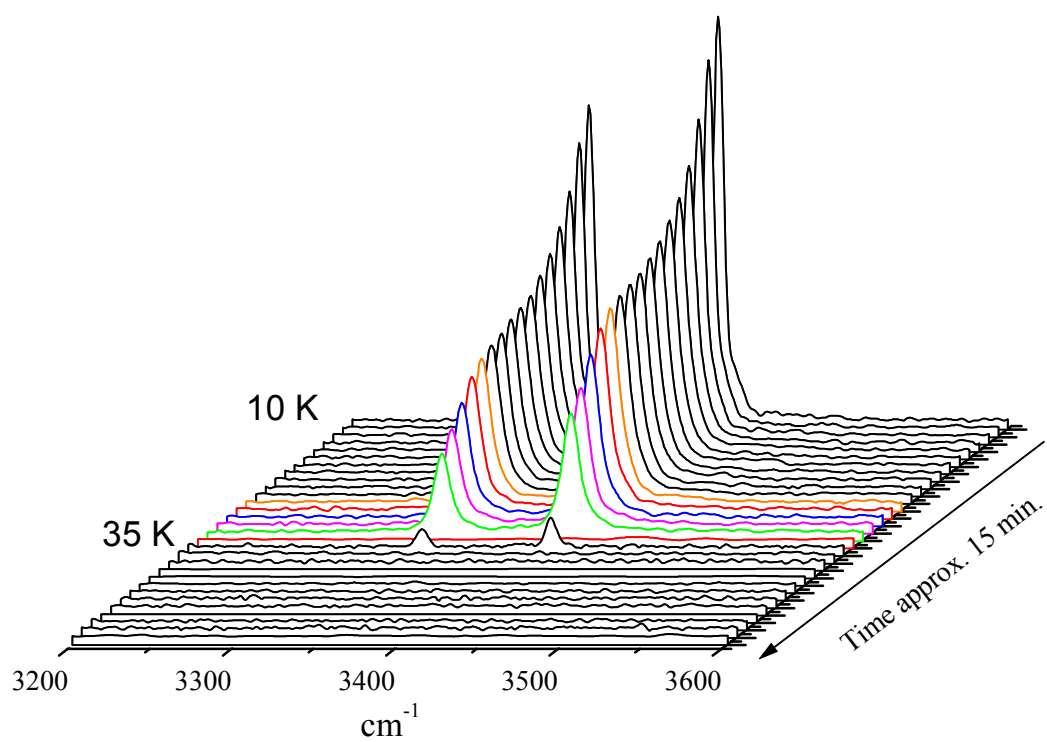


Fig. 21. The 3400 cm^{-1} doublet, recorded during 15 minutes after compressor shut-off. Each individual spectrum represents an average of 20 scans. The laser power was 500 mW and resolution 4 cm^{-1} .

Laser-excitation of liquid nitrogen

The experiments using multiphoton excitation of liquid nitrogen have continued with experiments with ArF excimer laser, operating at 193 nm. Although there are no allowed excitation schemes, the high power of this laser might be used in order to cause electric breakdown in liquid nitrogen. This process could subsequently have the potential of generating N_4 . Obviously, the energy is not a problem here, since the breakdown processes easily ionize N_2 . We have verified this by recording breakdown spectra in air (Fig. 22).

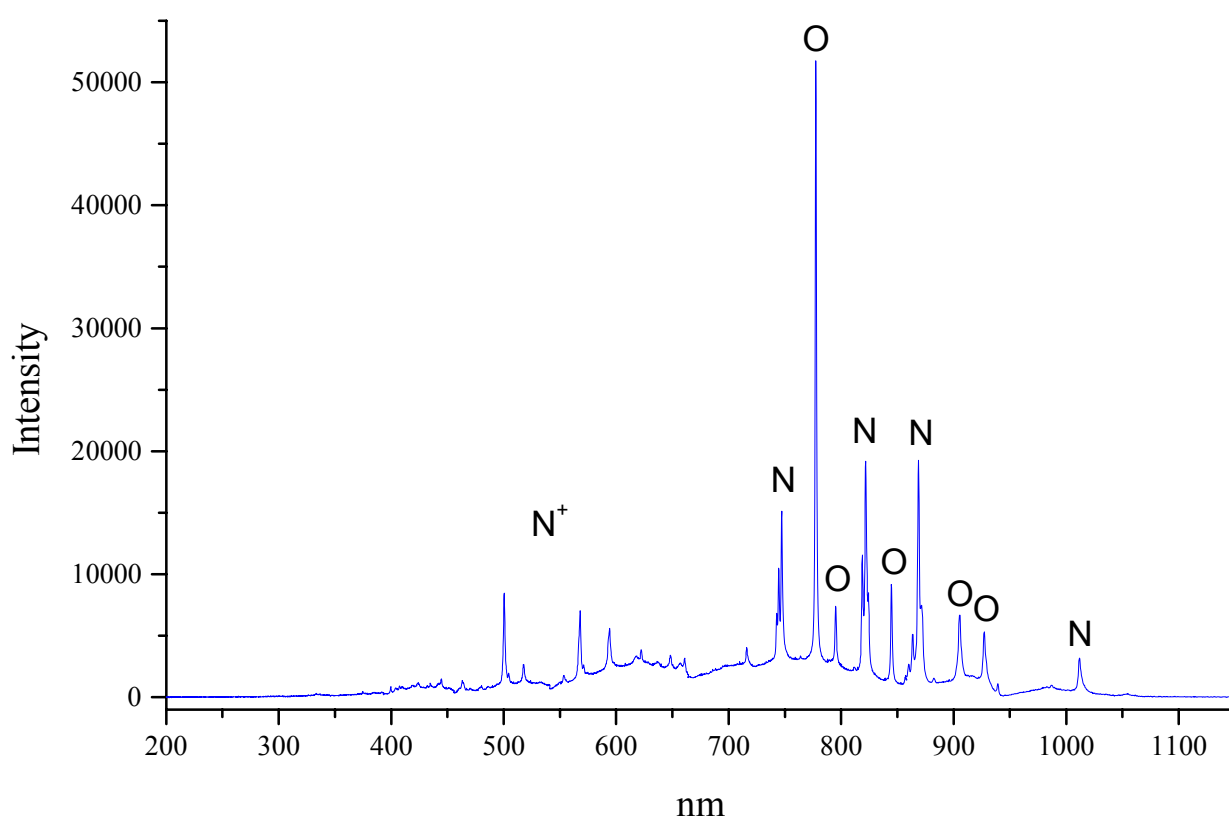


Fig. 22. Spectrum of laser-induced electric breakdown in air, using an ArF laser at 193 nm. The available energy at the focus was in the order of 100 mJ/pulse. The focal length of the lens was 7.5 cm. The exposure time was 100 s using a Mechelle 900 spectrometer. No molecular emission features are seen in this spectrum.

The experimental set-up for the liquid nitrogen breakdown experiments consists of a modified liquid nitrogen cryostat (JANIS), where a vertical, evacuated tube was inserted in order to minimize energy losses. A second reason for having the tube was the possibility of using short focal length lenses, necessary for small focal diameters. An overview of the experimental apparatus is shown in Fig. 23.

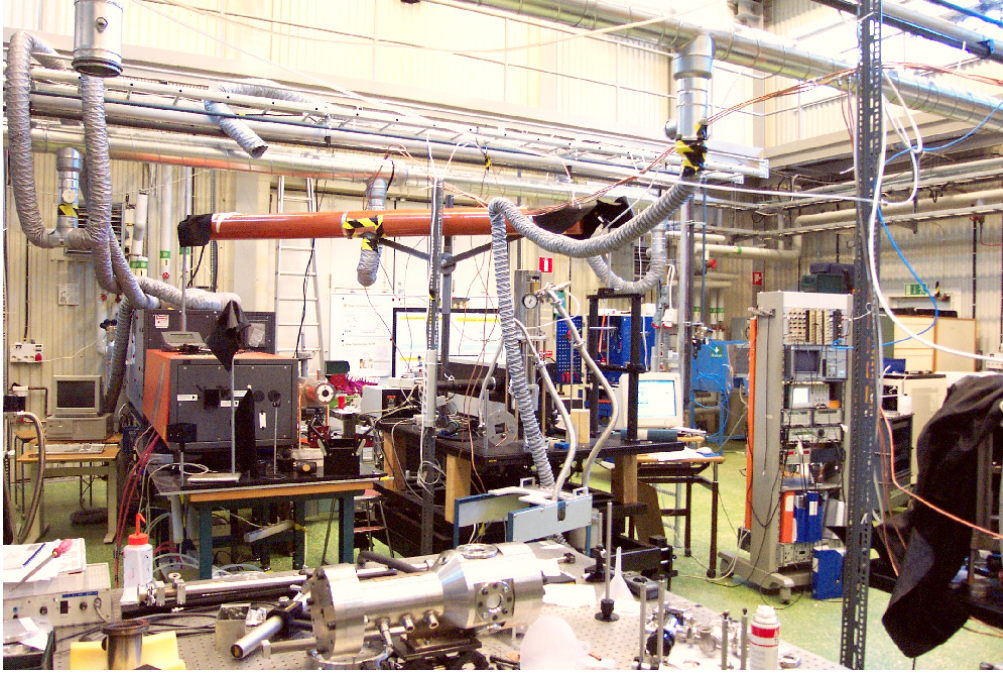


Fig. 23. The experimental equipment used in the liquid nitrogen experiments. *To the left:* The excimer laser in the background, the dye laser with orange cover in the foreground. *In the middle:* The cryostat and the SPEX 1000M spectrometer. *To the right:* The data collection equipment.

In the ArF liquid nitrogen experiments, we have not been able to see any electric breakdown in the liquid. The next step is to try to record molecular emission in liquid nitrogen using photon-counting techniques.

Experiments in a temperature-controlled cryostat

A CRYOCOOLER cryostat has been set up for temperature-controlled Raman experiments on matrices, built from microwave discharge products. The basic idea for these experiments is to avoid dilution of possible N_4 products through the excessive condensation of nitrogen, as is the case for the LEYBOLD cryostat experiments. The laser excitation experiments on solid nitrogen, mentioned in Ref. [1], can also be carried out using this set-up. The experimental equipment is shown in Fig. 24.

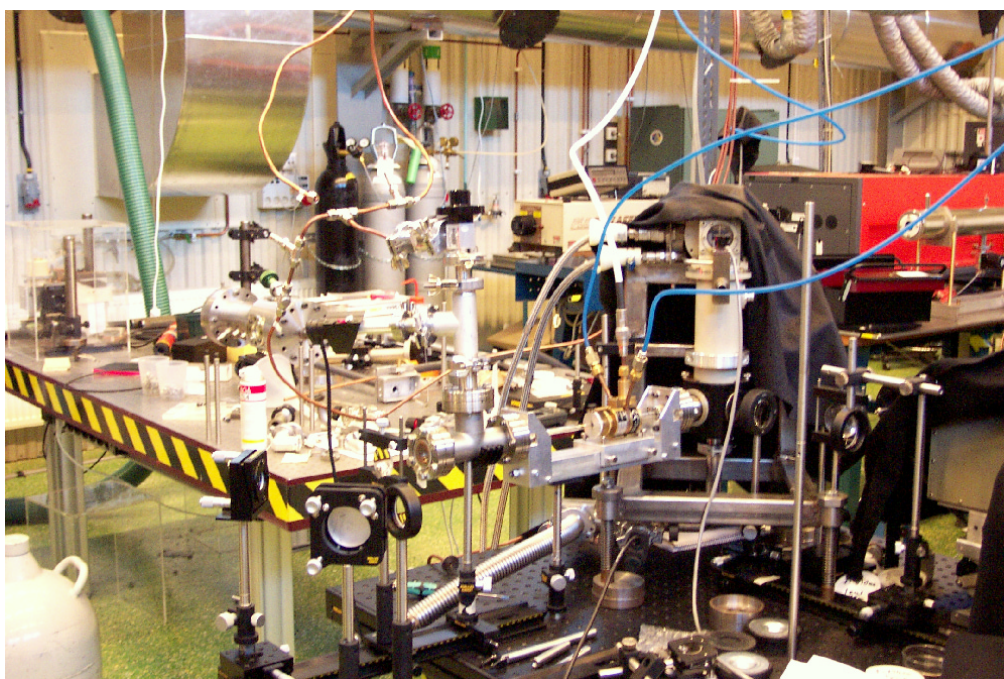


Fig. 24. The experimental set-up for the temperature-controlled experiments on cryogenic matrices. The cryostat and the microwave cavity are in the middle. The dye laser with the orange cover is to the right. The SPEX 500M monochromator is not shown.

The preparations for the temperature-controlled experiments have now been made. In the first experiments, the microwave discharge will be run on different nitrogen-containing mixtures during extended periods of time, while keeping the sapphire window of the cryostat at a temperature above 77K, in order to avoid condensation of nitrogen. The deposited substances are subsequently analyzed using Raman spectroscopy with the 514.5 nm line of an Ar⁺ laser, in combination with a notch filter with extinction of the laser light in the order of 10⁻⁶. Several preparatory experiments have shown that the equipment is capable of running during extended periods of time (several days) without intervention.

Plan for the rest of the year 2001

The main emphasis during the rest of the year 2001 will be on continued N_4 synthesis experiments with long integration times. The experiments will be carried out on liquid nitrogen samples, solid nitrogen matrices and samples containing heterocyclic high-nitrogen compounds at cryogenic and room temperatures.

The N_4 synthesis experiments using laser excitation of liquid nitrogen in capillary tubes will be set up starting from April 2001. We will use three excitation schemes (5, 7 and 9 in Ref [2]) with excitations at 208.8, 222.75 and 202.3 nm, respectively.

The cryogenic matrix bombarding experiments in the LEYBOLD cryostat will continue. During the rest of 2001, we will perform a number of long time experiments (integration times exceeding 14 hours) on ion-bombarded, microwave- and hollow cathode-irradiated matrices.

The temperature-controlled cryostat experiments will start in April 2001. The experiment will utilize the CRYOCOOLER cryostat in combination with a SPEX 500M monochromator, an Ar^+ laser and an optical multichannel analyzer.

Summary

Activities:

- Ion bombardment, microwave-excitation and hollow-cathode excitation experiments of solid N₂ have been performed in the 16-port cryochamber. Several synthesis experiments with long integration times have been carried out.
- A second cryostat has been set up for laser excitation experiments and preparations have been made for temperature-controlled matrix excitation experiments with Raman detection using the 514.5 nm line of an Ar⁺ laser.
- Initial tests have been made on excitation of liquid nitrogen with a 193 nm ArF laser.

Progress:

- A number of unassigned features have been observed in ion bombarding, microwave-excitation and hollow-cathode excitation experiments on nitrogen-containing matrices.

Expectations for the rest of the year 2001:

- Continued long-time experiments on different nitrogen-containing cryogenic matrices will hopefully reveal the carriers of the unassigned features observed so far.
- Further laser excitation experiments of solid nitrogen in the small cryostat will be carried out, envisaging the possibility of two-laser experiments.
- Capillary experiments will hopefully answer some of the questions in the laser excitation experiments on liquid nitrogen.
- A continuation of the experiments on the heterocyclic nitrogen-rich compounds is envisaged.
- And of course our ultimate goal: The successful synthesis and detection of N₄.

Reports:

- One report has been written (Ref. 1 in the reference list).
- Two articles have been published. (Refs 3-4 in the reference list).

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Issuing organization FOI – Swedish Defence Research Agency	Report number, ISRN FOI-R--0127--SE	Report type Progress report
	Research area code	
	Month year June 2001	Project no. E2968, E2004
	Customers code	
	Sub area code	
Author/s (editor/s) Olli Launila Henric Östmark Rolf Tryman Sara Wallin Gunnel Petri Anna Pettersson	Project manager Henric Östmark	
	Approved by	
	Scientifically and technically responsible Olli Launila	
Report title The N ₄ project. Report for the first quarter of 2001.		
Abstract (not more than 200 words) A summary of activities and achievements within the N ₄ project during the first quarter of year 2001 is presented.		
Keywords DARPA, N ₄ , HEDM, Energetic materials, Solid nitrogen, Cryogenic matrices, Liquid nitrogen		
Further bibliographic information	Language English	
ISSN 1650-1942	Pages 39 p.	
Price acc. to pricelist Security classification		

Utgivare Totalförsvarets Forskningsinstitut - FOI	Rapportnummer, ISRN FOI-R--0127--SE	Klassificering Verksamhetsrapport
	Forskningsområde	
	Månad, år Juni 2001	Projektnummer E2968, E2004
	Verksamhetsgren	
	Delområde	
Författare/redaktör Olli Launila Henric Östmark Rolf Tryman Sara Wallin Gunnel Petri Anna Pettersson	Projektledare Henric Östmark	
	Godkänd av	
	Tekniskt och/eller vetenskapligt ansvarig Olli Launila	
Rapportens titel (i översättning) N ₄ projektet. Rapport för det första kvartalet av år 2001.		
Sammanfattning (högst 200 ord) En sammanfattning över aktiviteter och resultat inom N ₄ projektet under det första kvartalet av år 2001 presenteras.		
Nyckelord DARPA, N ₄ , HEDM, Energetiska material, Fast kväve, Kryogeniska matriser, Flytande kväve		
Övriga bibliografiska uppgifter	Språk Engelska	
ISSN 1650-1942	Antal sidor: 39 s.	
Distribution enligt missiv	Pris: Enligt prislista Sekretess	

