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Final Report on the Operation of the Experimental Electrospray Source for Nanoparticles

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Contract Scientific Authority: D. Pedersen, DRDC Suffield

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Final Report on the Operation of the Experimental Electrospray Source for Nanoparticles
Prepared by Adam Malcolm (June 23, 2007)

Current Progress with Operation

The following section contains all of the academic reports made on the testing and operation of the apparatus. Research and reports were performed and written in the laboratory of Dr. J. Mark Parnis at Trent University.

General Repair for Operability (March 2009)

Work on the electrospray began with placement of a flat electrode plate in front of the first skimmer cone to correct for the electric field generated by the cone, which is common practice in typical electrospray. The second change made to the electrospray interface was the capillary itself. The previous design had a small flaw where the Teflon capillary mount was not aimed directly at the centre of the electrode plate. To fix this, the mount was filed flat, and extra washers were added to maintain a level surface for the capillary to rest. However this didn't fully correct the flaw, so a small piece of vanadium foil was used to shim the capillary such that it pointed directly at the orifice. These alterations allowed for the successful reproducible formation of a Taylor cone. In order to get eyes on the capillary and the formed cone, a flat surface was ground into the side of the casing, however this proved to cause the magnification of the capillary to decrease. Consequently minor repairs were made to the solder points on the capillary, as well the walls of the casing had to be polished and buffed so that we could get a clear image of the capillary tip with our microscope camera. This was necessary as there is currently no diagnostic other than visual confirmation that a Taylor cone has formed.

After these repairs and modifications, the conditions at which Taylor cone formation for methanol occurs were documented and a test spray into an Ar matrix was attempted. Two separate experiments were performed. The first was to pump solvent through the capillary, but without applying any electric field such that no Taylor cone formed. The second was to repeat the experiment with the potentials applied to form a Taylor cone. These were done to determine if methanol would reach the window in the form of gaseous ions (via successful electrospray) or simply as methanol vapour from the liquid. Analysis was done using IR spectroscopy, and the two measurements were very different. Characteristic methanol absorptions were observed in the experiment where no electrospray was used. For the experiment with applied potentials and Taylor cone formation, no absorptions for methanol were observed above the signal to noise, however many unassigned absorptions were observed. This was confirmation that the electrospray was having an effect on the methanol, however due to the large amount of air trapped inside the matrix isolation apparatus, the spectra were noisy and contained many broad features, making elucidation of what was happening difficult. Furthermore, the pressure of the system increased from a significant air leak (since part the bulk of the apparatus is at 1 atm) such that whether or not the species were being isolated in a pure Ar matrix was uncertain.

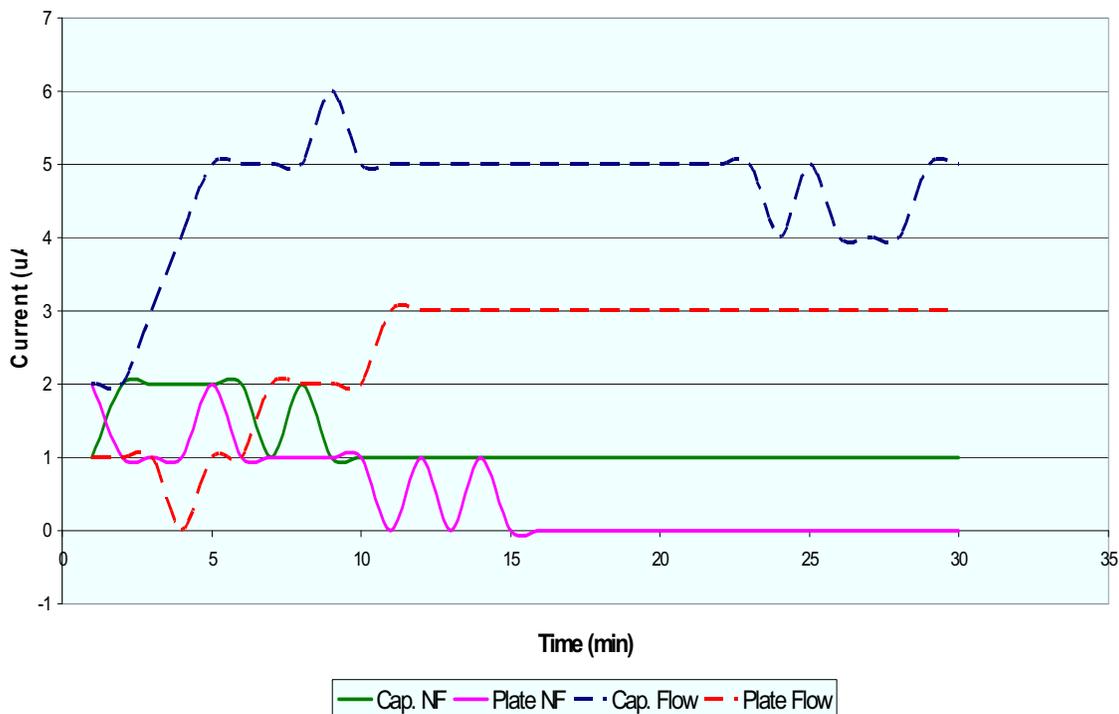
In spite of the large pressure increase, an electrospray of nanoparticles was done since analysis would have been done with UV, which would run unhindered by excess air. After finding a replacement computer that could interface with the UV-Vis spectrometer, this experiment was performed, however no characteristic surface plasmon absorptions were observed, even for extended depositions. It was observed that the pink/red nanoparticle solution was collecting on the electrode plate, implicating an off-axis Taylor cone. It is currently unknown what caused this, but further attempts will be made.

It was observed that the electrospray casing was missing an o-ring, which would have contributed to the significant air leak inside the apparatus, and thus this was replaced and re-greased to form a good seal. This was then put back onto the apparatus to check to see if the pressure inside the matrix apparatus would remain nominal instead of spiking to the 1×10^0 region. A pressure of $7 - 9 \times 10^{-2}$ Torr was observed, which was a significant improvement upon the previous pressure, however still very high compared to what the matrix apparatus can handle. We have proposed flowing an Ar drying gas between the front plate electrode and the first skimmer cone to prevent air from leaking into the differential pumping region. Instead Ar will leak into the system, which may actually benefit the experiment by contributing to the matrix formed on the cold window.

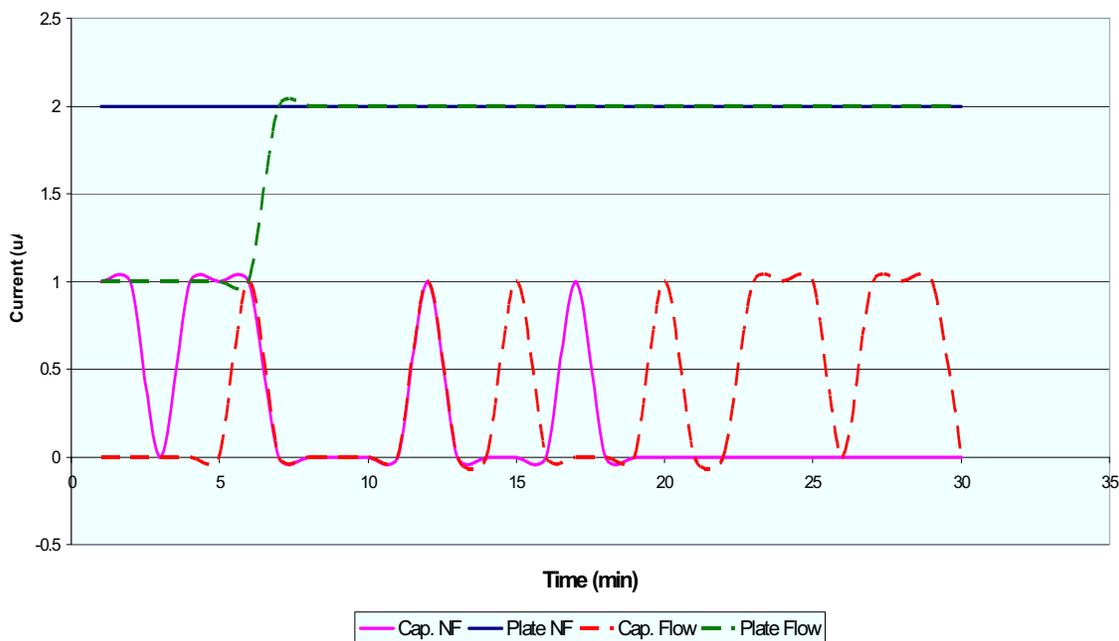
Testing for Ion Current

Summary Report of Ion Current Tests in Nanoparticle Electrospray Apparatus Prepared by Adam Malcolm on March 27, 2009.

The following is a summary of the results from a series of ion current measurements at the electrode plates/cones inside the electrospray apparatus. In all cases, methanol was used as the electrosprayed solvent since it has the most reproducible Taylor cone. In all of the primary tests, the first electrode plate was set to -400 V, while the capillary was set to $\sim +2600$ V. Methanol flow was fixed at 0.375 cc/hr (~ 6.2 uL/min). Measurements of the current were taken at both the plate and capillary for 30 minutes, with and without spraying methanol. The results are shown in the figure below.



From the above figure, capillary and plate currents read steady at 1 and 0 uA respectively when no methanol is flowing (Cap. NF and Plate NF curves). However, when methanol is spraying, the currents jump up to 5 and 3 uA for the capillary and electrode plate respectively (Cap. Flow and Plate Flow curves). Even though no methanol was spraying, the capillary still read 1 uA, which means the capillary is suffering from a 1 uA current leak, though we are unable to detect where this leak is. If the current at the capillary when methanol is spraying is corrected for this, it only sees a jump of 4 uA in ion current, not 5 uA. Thus when spraying, 4 uA of ion current are leaving the capillary, and 3 uA are reaching the electrode plate, so an extra 1 uA of ions are either being lost to the walls or the apparatus, or are traveling through the first skimmer cone to the second skimmer cone. For the sake of completeness, the same experiment was run with the electric field reversed. So the capillary and electrode plate potentials were set to -2600 V and +400 V respectively. No current change was observed for this case, as both the capillary and plate currents measured 2 uA and 0 - 1 uA respectively, with and without methanol spray, as seen in the figure below.



While the base current for the capillary in both cases increased to 2 uA, there is no difference in current between spraying and not spraying methanol, indicating a lack of ions at the plate. This result in conjunction with the previous result implicates a flow of ions when spraying methanol only when the electric field is oriented in positive ion mode (towards the electrode plate). It should be noted that electrospray can work in both positive and negative ion mode, so the fact that methanol isn't generating ions in negative ion mode may mean the apparatus is flawed, or that methanol is not just capable of producing negative ions. Most of the literature read deals with methanol work in positive ion mode.

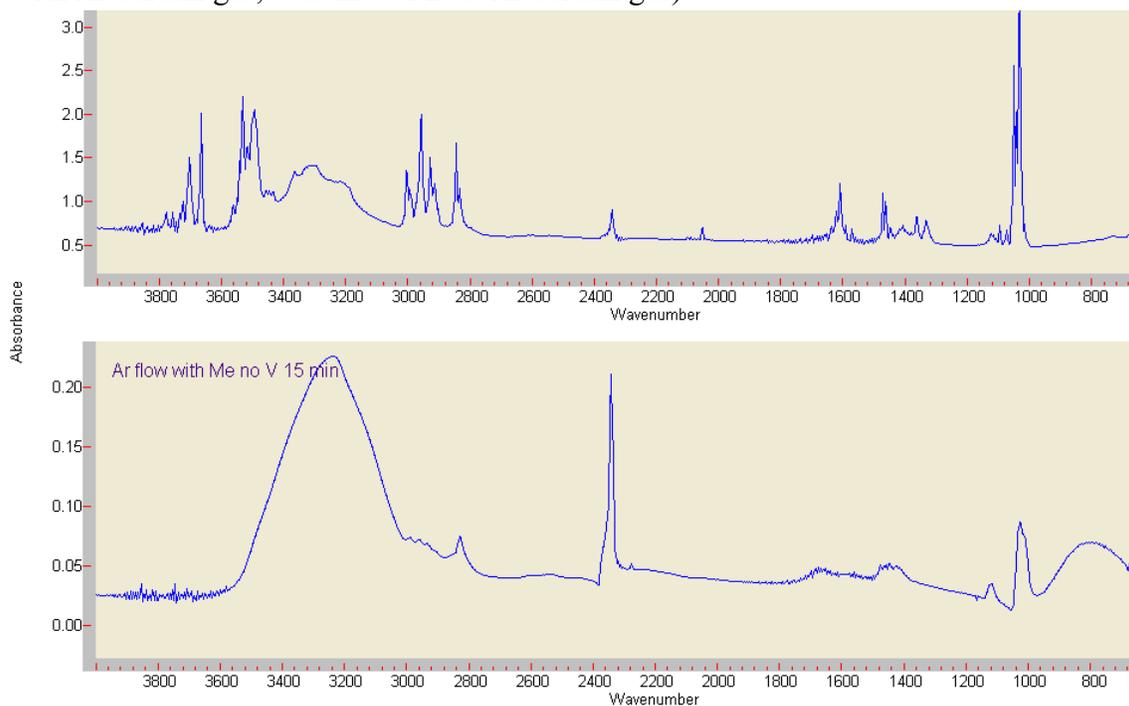
In a series of other measurements, the current at the second skimmer cone (-800 V applied potential) was found to be 0 uA for all conditions, including those where methanol flow was reduced to 3 and 1 uL/min, in which a drop in ion current was observed for the second skimmer cone, electrode plate and capillary. What can be discerned from all of these current tests, is that at that normal operating conditions, an ion current is being produced, indicating that we are generating a stream of ions, however they aren't making it past the first skimmer cone, which could arise from a number of factors. For one, we are unable to detect if we are generating a Taylor cone jet, which is what causes a rapid ejection of ions from the capillary liquid. If we are generating the Taylor cone jet, the electric field isn't allowing the ions to travel with a perfectly linear path of motion. The light at the end of the tunnel is that we are detecting an ion current, so some ions are indeed making it to the first cone. A preliminary literature search has begun to determine if the measured currents are within normal range for the type of electrospray we are doing.

Proof of Curtain Gas Requirement

Report of Electrospray of Methanol into an Ar Matrix with Inert Curtain Gas Prepared by Adam Malcolm on April 3, 2009

Due to the difficulty of electrospraying Au nanoparticles, and the lack of understanding of what is indeed happening inside the apparatus when it is operating, we have decided to electrospray simple substances first, which are well studied and will serve as background for electrospraying Au NPs. We have started with spraying pure methanol, as it has a low surface tension and low Taylor cone formation potential. A recent modification has also been made to the apparatus, where a curtain gas inlet has been created, such that a counter-flow of inert gas will keep air from being drawn into matrix isolation apparatus. Tests were run with a 3000 V potential difference, ~ 6.2 $\mu\text{L}/\text{min}$ methanol capillary flow, and experiments were run for 10 – 15 minutes.

In a first run of 15 minute tests, Ar was used as a curtain gas. Two experiments were done, with and without the electric field potential. It was shown that without the Ar counter flow, the resulting spectrum was messy and indecipherable with many broad and rotational bands overpowering the smaller, sharper absorptions of the methanol. However, with the Ar counter flow, the great majority of these bands were eliminated, and a clean spectrum of methanol was produced. Below are the associated spectra (top: With Ar curtain gas, bottom: Without Ar curtain gas).

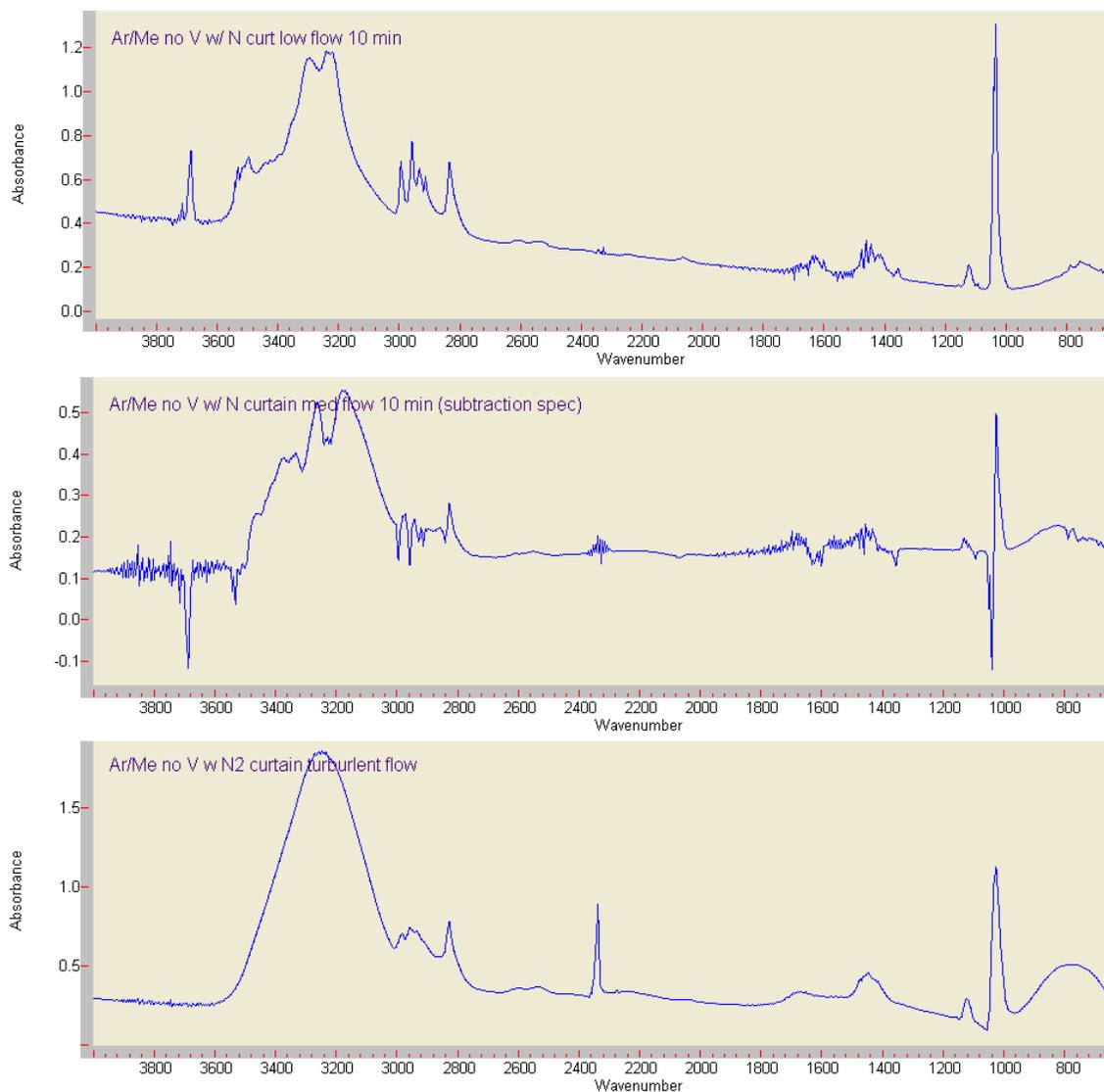


Furthermore, without the Ar curtain gas, the cold cathode pressure began to increase slowly, but steadily until it was up in the 10^{-3} Torr regime, due to increased thermal load on the tip. With the curtain gas the pressure remained in the 10^{-6} Torr regime. This confirmed 2 things for us: the Ar counter flow was preventing the ingress of air (with CO_2 and H_2O), while still allowing methanol vapour to be pulled through via high vacuum into the matrix. The same tests were to be done with the potentials applied

following this, however the capillary began to discharge rapidly and successively; giving the effect of lightning in a bottle. Because of this, the Ar curtain gas was changed to N₂.

The same set of initial tests were repeated as above with N₂ for only 10 minutes (as this time was found to be adequate), and it was found that N₂ also prevented air from making its way into the matrix, much like Ar, however the pressure at the cold cathode very slowly increased, but only into the high 10⁻⁵ and low 10⁻⁴ Torr regime, and only near the end of the experiment. Electrospray experiments were then done, and no discharge of the capillary was observed. Again, electrospray of methanol with and without an N₂ showed that the counter flow kept air out, while letting methanol through. Dealing only with experiments with N₂ counter flow, it was noted that significantly more methanol was observed in the resulting spectrum for the electrospray than without. This indicates that even though methanol vapour will be drawn into the matrix regardless of electrospray, using the electrospray causes more methanol to make it to the cold window, indicating that our apparatus can successfully get analyte from the capillary to the window, however this may be due to the fact that the Taylor cone is better at vaporizing methanol and thus more background methanol vapour will get through.

We decided it would be worthwhile to get the background methanol vapour close to zero, such that when we electrospray, the methanol we observe in the spectrum will only be from the spray. This was done by starting with a low flow N₂ counter flow and working up to high flow. Since we currently have no flow meter on the only N₂ source we have, it is impossible to be quantitative in this regard. Though as a preliminary set of subjectively qualitative tests, low, medium and high N₂ flow experiments were done, without the electric field to determine the amount of methanol vapour let through. Compared to low flow, the medium N₂ flow let in much less vapour. High flow on the other hand, allowed much more vapour through, as well as some atmospheric air. See figure below for associated spectra, top: low flow N₂, middle: medium flow, bottom: high/turbulent flow).



It is thought that due to the turbulence of such a flow, methanol vapour is released from the capillary much faster, and mixes thoroughly with the air and N₂ gas counter flow, such that the gas pulled through the flight path is a mixture of atm gases, N₂ and excess methanol vapour. The resulting spectrum contained strong broad and rotational bands, making analysis difficult. It has thus been determined that some intermediate flow of gas will minimize the amount of methanol vapour that passes through without electro spray, however it will never be eliminated, meaning background solvent in future experiments should be expected as normal.

In summary, a curtain gas inlet was created for the electro spray to keep out warm atmospheric air containing CO₂ and H₂O. It was successfully shown that this inlet performs adequately, and that N₂ is the ideal curtain gas, as it does not promote electrical discharge of the capillary onto the electrode plate like Ar. As well, it has been determined that more methanol makes it onto the cold window with the electric field applied, than

without, implying the successful delivery of methanol into a matrix via Taylor cone electrospray. Lastly, we showed that a medium flow of gas (a subjectively qualitative term) will minimize the amount of background solvent vapour. It is our plan to quantify the N₂ gas flow, as well as electrospray an analyte which is already ionic, such as ammonium acetate. If we can determine that ammonium and acetate are indeed reaching the window, then we know that the apparatus is without a doubt delivering ions to the window via the applied electric field, as such substances have no vapour pressure.

Rate of Matrix Gas Flow (as a result of testing for ammonium acetate)

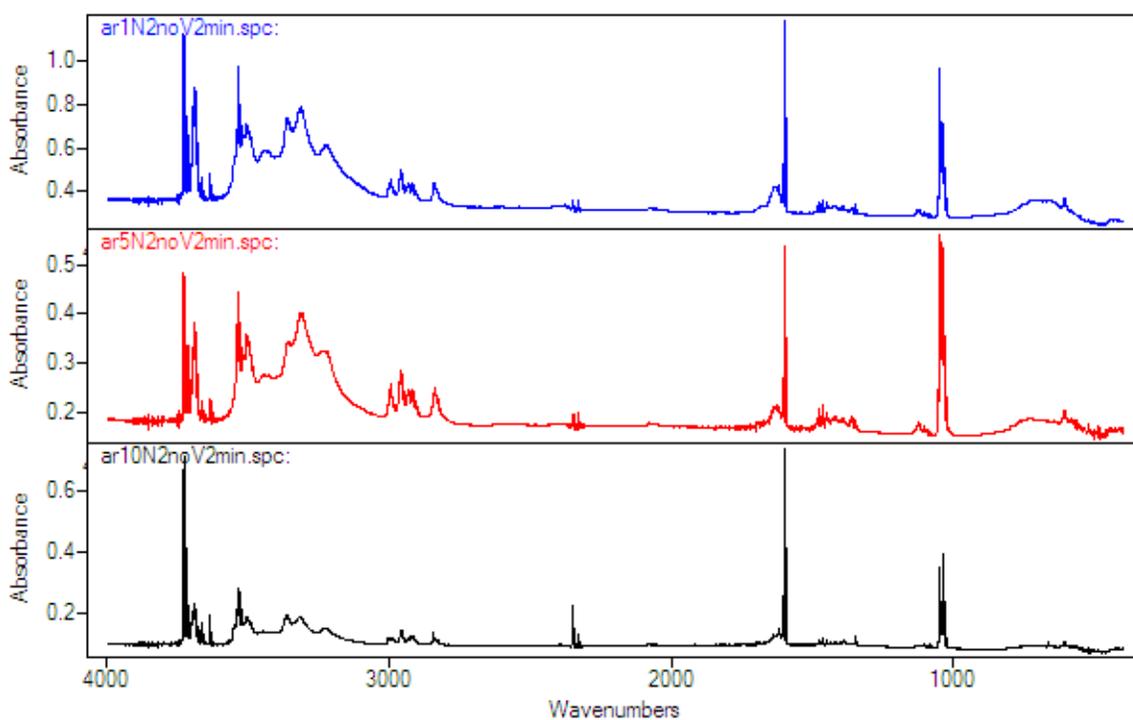
Report on Electrospray of Ammonium Acetate in Ar matrices

Prepared by Adam Malcolm on April 27, 2009

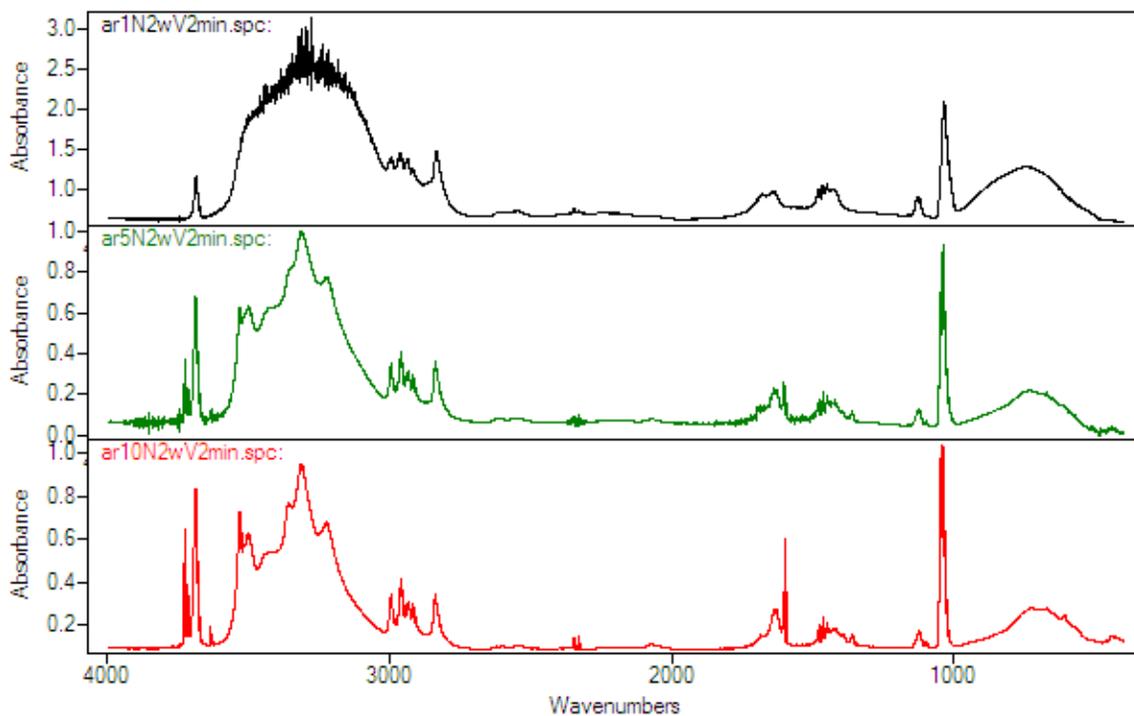
Due to the lack of proof of delivery of ionic methanol from the electrospray apparatus into an Ar matrix, we decided to electrospray an ionic solution of Ammonium Acetate into an Ar matrix. A previous suggestion was made to bias the tip of the matrix apparatus to determine how much methanol reaching the cold window was ionic. This proved to be problematic as setting this up would have been quite difficult and time consuming, as we weren't certain the bias potential would be enough to overcome the kinetic energy of any ions, since they would be traveling with over 3000 eV of kinetic energy. That idea was left behind in order to electrospray something that, if detected, would be known to have been ionic. The series of experiments conducted used a 10 mM solution of Ammonium Acetate (NH₄OAc) in a 60/40 MeOH/H₂O mixed solvent. The capillary flow was kept at ~6 uL/min, and the applied potential was 2480 V in positive ion mode. The N₂ curtain gas was run at ~540 L/hr during 2 minute, while the Ar matrix gas was run at varying flow rates (1 sccm, 5 sccm, 10 sccm, 15 sccm, and 20 sccm). This was done to determine which flow rate offered the best host/guest interactions in order to obtain the cleanest spectrum, as the high pressure from the electrospray inlet causes the resulting matrix to be formed poorly if at all without proper matrix gas flow.

Because we were running in positive mode, we were looking for evidence of the NH₄⁺ ion in the matrix. We found no evidence of this in any of the experiments done, however does not indicate that no ions were electrosprayed. The IR bands of NH₄⁺ are overpowered by the bands of MeOH which is always in great supply, shadowing any of the regions in which NH₄⁺ would show up. This work was however useful for determining the optimal matrix gas-flow conditions, and the reproducibility of results.

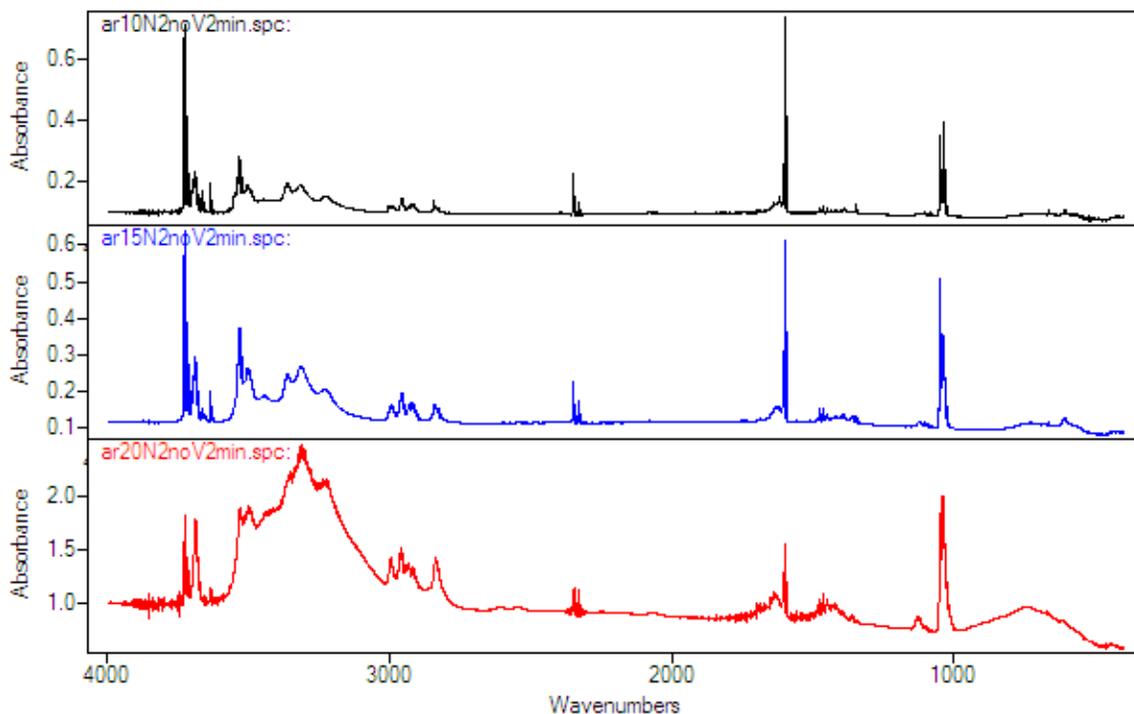
The following spectra show the background MeOH/H₂O solvent vapour at Ar gas-flow rates increasing from 1 sccm, 5 sccm and 10 sccm (top to bottom), without any electrospray.



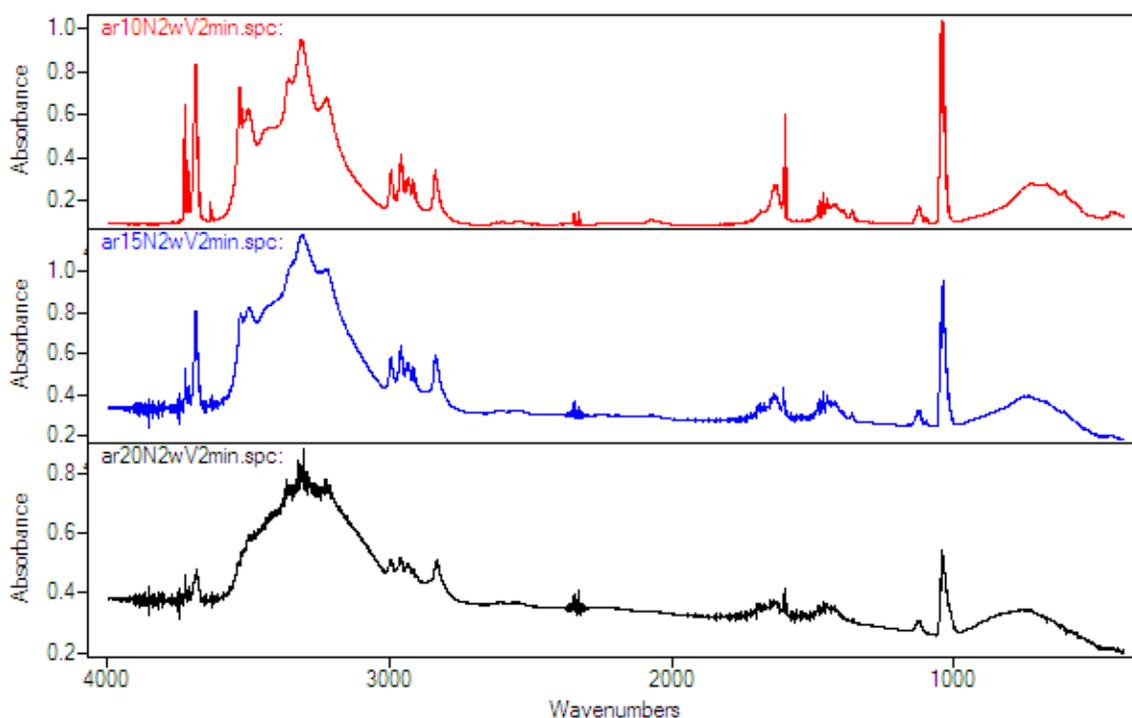
It can be seen that as the Ar flow rate increases, the breadth and amplitude of the absorptions decreases, increasing the clarity of the result. This pattern is made clearer in the figure below for the same experiments, with the electrospray on.



As the flow rate is increased, the peaks of known methanol dimers, trimers and tetramers in the 2500 – 3500 cm^{-1} range become resolved and decrease in intensity. By increasing the flow rate, we are creating a better matrix for the large quantities of gas being poured into the matrix apparatus from the electrospray apparatus. From here, the Ar flow rate was increased to 15 sccm and 20 sccm (which represents the flow threshold of the mass-flow controller). The figure below shows the spectra of the solvent vapour at these flows without electrospray (top: 10 sccm, bottom: 20 sccm).

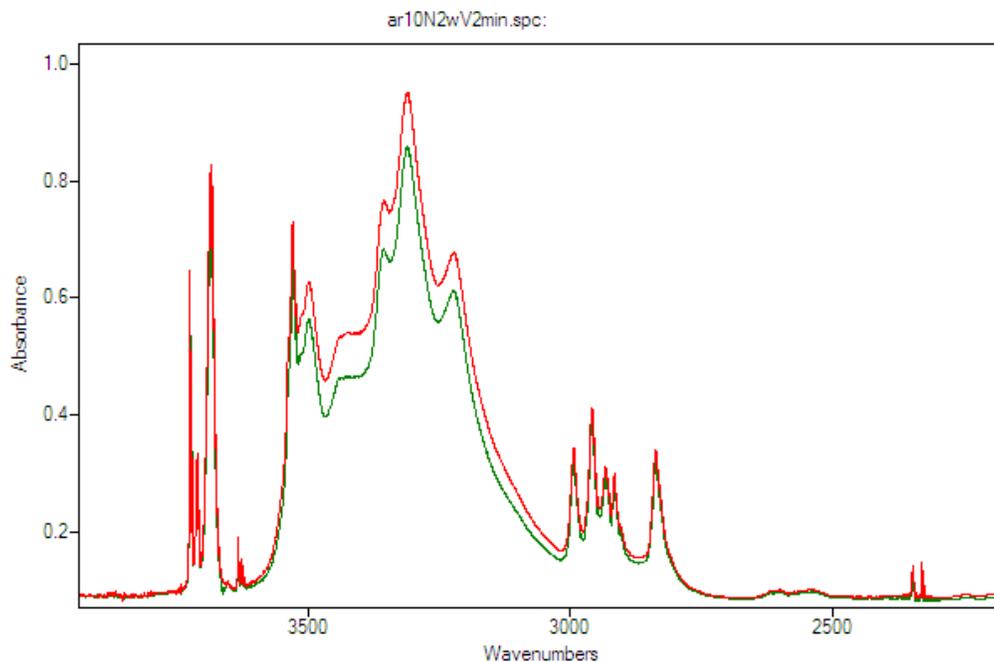


It can be seen that as the Ar flow rate increases to very high flows, the spectra become less resolved due to the increased thermal load on the system. While 15 sccm is almost comparable in clarity to 10 sccm, the peaks associated with the solvent are about twice as intense. At 20 sccm, the system can no longer handle the gas load being applied, and the resulting matrix is poorly formed. The same pattern is again seen in the same experiments with the electrospray on (figure below; top: 10 sccm, bottom: 20 sccm).



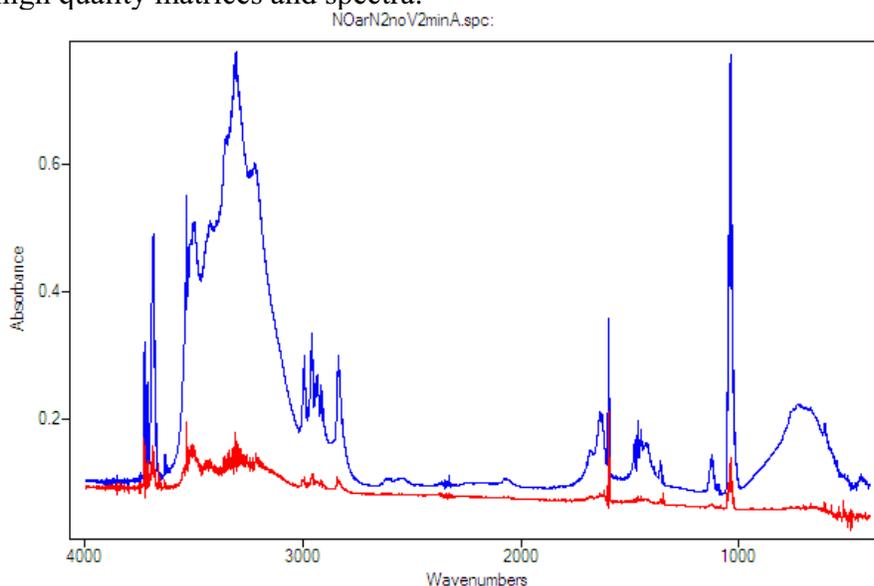
It is clear that above 10 sccm Ar flow, the resulting matrix degrades to a point where peak differentiation is difficult in some regions and impossible in others. With these results in hand we know that the optimal matrix gas flow rate is around 10 sccm, as it offers the greatest resolution while minimizing the concentration of the solvent gas in the matrix. It should be noted that even at this optimum condition, the amount of solvent making its way into the matrix (especially with electrospray) is considerably high. This is currently an artifact of the apparatus we must live with.

In order to determine the reproducibility of the results here, a duplicate experiment with electrospray at 10 sccm was run. The figure below compares the 2000 – 4000 cm^{-1} region for the two experiments (red being the duplicate experiment).



There is obviously some comparability between the two experiments; however some error does still exist for some peaks, as slightly more material must have made it into the matrix the second time, even though the experimental conditions were kept identical. For our purposes currently, this reproducibility will suffice as we haven't yet entered into any analytical work.

In one last set of experiments, we wanted to determine if the material being isolated was forming in an Ar or N₂ matrix, as a considerable amount of N₂ makes its way to the window from the curtain gas inside the electrospray apparatus. The inlet from the electrospray to the window was opened without any Ar flowing both with and without electrospray. The spectra below (red: no electrospray, blue: with electrospray) show that N₂ forms a very poor matrix for the material; we thus need a 10 sccm flow of Ar in order to obtain high quality matrices and spectra.



From here we are going to electrospray a solution of glycine in a series of experiments similar to those outlined here. Being an amino acid, it is amphoteric and should electrospray in both positive and negative ion modes, and contains a carbonyl group which should show a stretching band in the $1600 - 1800 \text{ cm}^{-1}$ region, which in our work is relatively unhindered by background solvent vapour.

Testing for the Presence of Glycine and Myoglobin (May 2009)

Tests identical to those in the previous sections were conducted using a solution of 10 mM glycine and very dilute myoglobin (in 60/40 MeOH/H₂O). The presence of neither substance was verified. Myoglobin was used as a test protein because it is relatively small, and if the electrospray was indeed generating ions that made it to the cold window, would generate numerous absorption bands, as myoglobin consists of thousands of bonds per molecule, making each represent about 200 small organic molecules. Any presence would have surely been identifiable. So far identification of isolated has been done unsuccessful.

Use of a Faraday plate testing for the presence of ions (May, June 2009)

A Faraday plate was crafted to sit inside the flight path assembly to collect ions and deliver a read out of the current they generated. The plate was placed directly behind both skimmer cones and in neither case was there any detection of ions, even on the order of 0.1 pA. The tests were performed both at atmospheric pressure and vacuum (approx. 1×10^{-3}), and in neither case was an ion current generated. This ultimately led us to believe the current tests done in section 4.2 were false readings or some kind of electronic drift in the power supplies that were used to make the measurements. This is ultimately where the project was ended and repair has since ceased.

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See also the report written by Taylor Stock (sent as a physical copy) titled “Charge-Reduced Electrospray Ionization Molecular-Beam Source (CR-ESI-MBS)”.

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This report contains all of the academic reports made on the testing and operation of an electrospray apparatus designed to spray nanoparticles out of solution and introduce them into a vacuum environment for deposition in solid rare-gas matrices. Research and reports were performed and written in the laboratory of Dr. J. Mark Parnis at Trent University.

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nanoparticles, aerosol, electrospray, matrix isolation

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