New ways of ambient ionization

T. Pradeep
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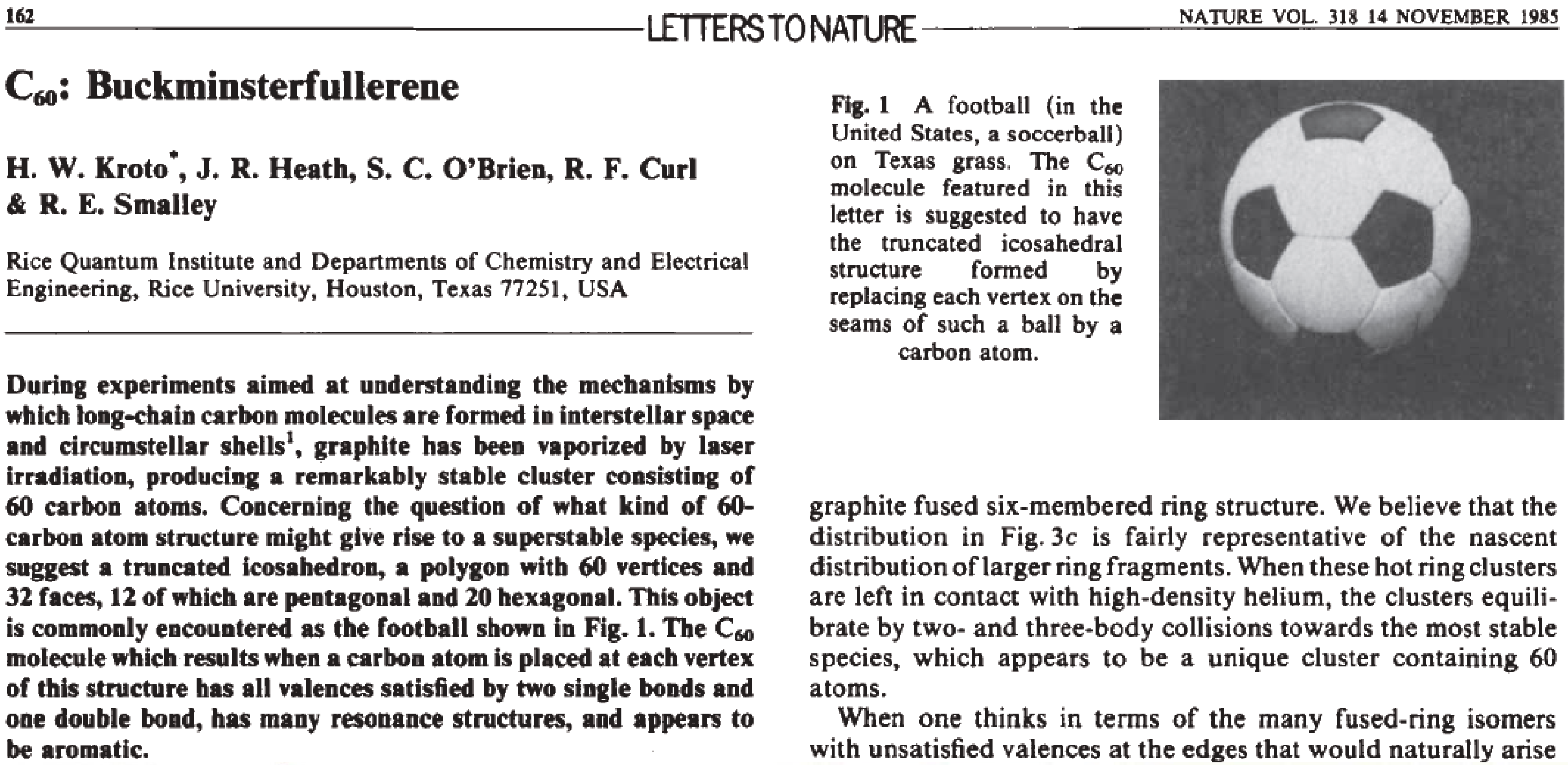
Thanks to Heon Kang

Department of Chemistry, Seoul National University, October 29, 2014



162 LETTERS TO NATURE NATURE VOL. 318 14 NOVEMBER 1985

C_{60}: Buckminsterfullerene

H. W. Kroto*, J. R. Heath, S. C. O'Brien, R. F. Curl & R. E. Smalley

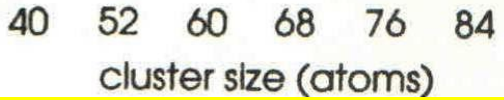
Rice Quantum Institute and Departments of Chemistry and Electrical Engineering, Rice University, Houston, Texas 77251, USA

During experiments aimed at understanding the mechanisms by which long-chain carbon molecules are formed in interstellar space and circumstellar shells[1], graphite has been vaporized by laser irradiation, producing a remarkably stable cluster consisting of 60 carbon atoms. Concerning the question of what kind of 60-carbon atom structure might give rise to a superstable species, we suggest a truncated icosahedron, a polygon with 60 vertices and 32 faces, 12 of which are pentagonal and 20 hexagonal. This object is commonly encountered as the football shown in Fig. 1. The C_{60} molecule which results when a carbon atom is placed at each vertex of this structure has all valences satisfied by two single bonds and one double bond, has many resonance structures, and appears to be aromatic.

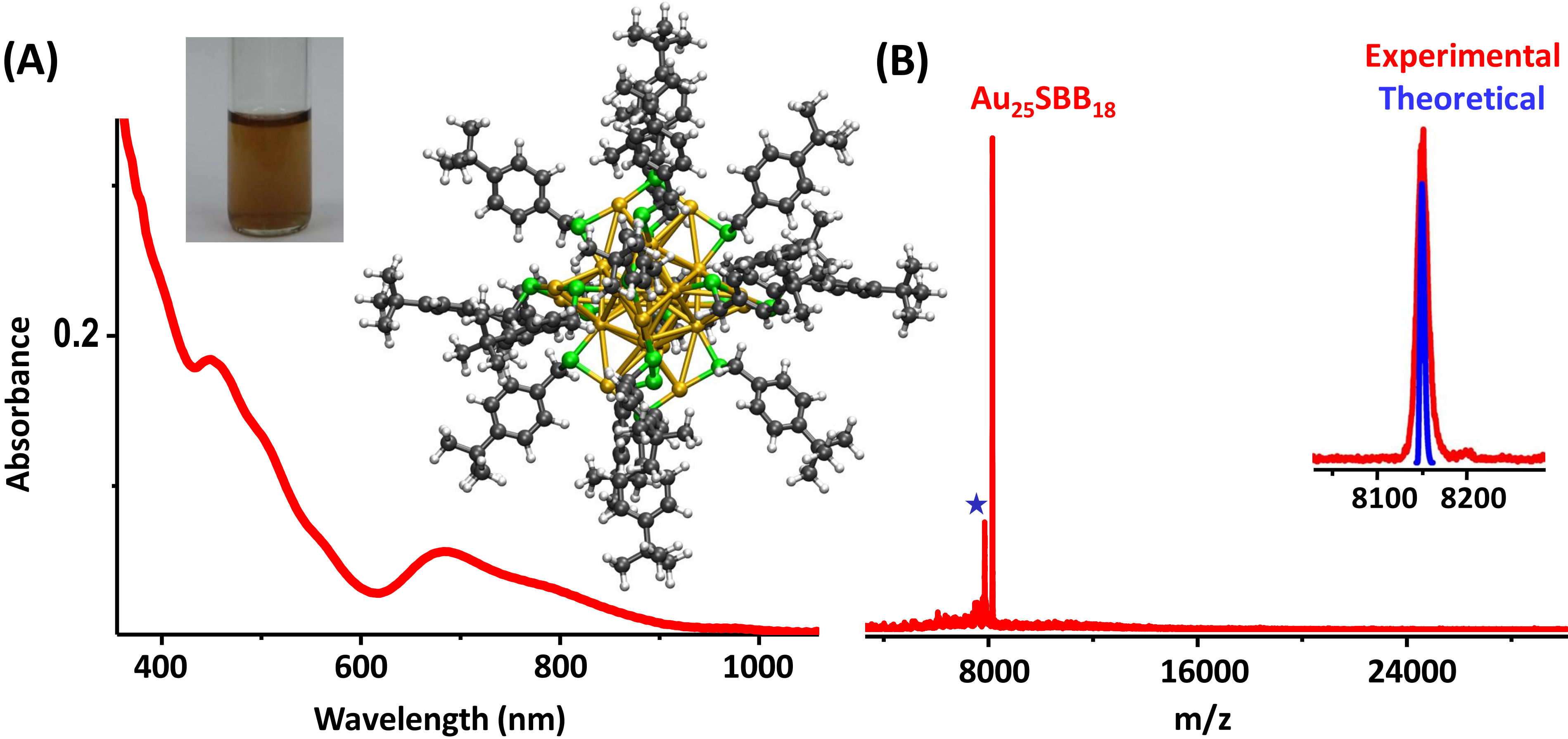
Fig. 1 A football (in the United States, a soccerball) on Texas grass. The C_{60} molecule featured in this letter is suggested to have the truncated icosahedral structure formed by replacing each vertex on the seams of such a ball by a carbon atom.

graphite fused six-membered ring structure. We believe that the distribution in Fig. 3c is fairly representative of the nascent distribution of larger ring fragments. When these hot ring clusters are left in contact with high-density helium, the clusters equilibrate by two- and three-body collisions towards the most stable species, which appears to be a unique cluster containing 60 atoms.

When one thinks in terms of the many fused-ring isomers with unsatisfied valences at the edges that would naturally arise

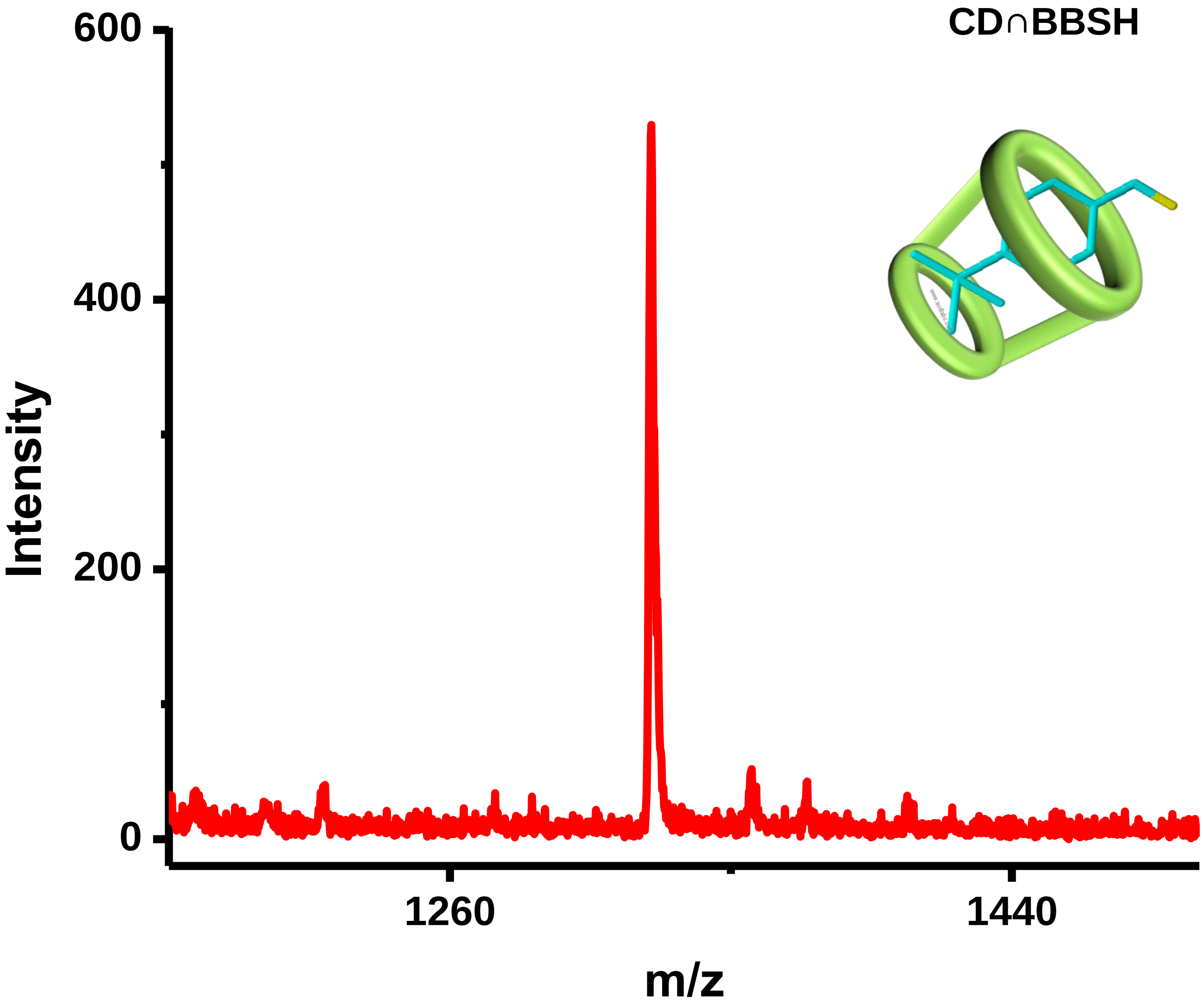


Mass spectrometry in materials science

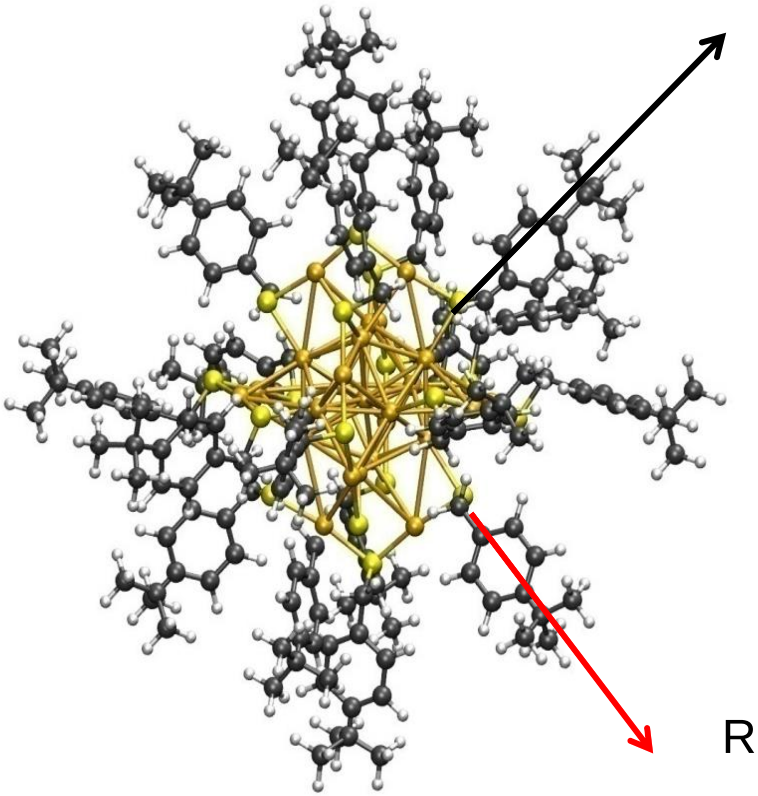


Ammu Mathew et al. ACS Nano 2014.

Positive mode MALDI MS of BBSH∩CD complex



Substitution chemistry of clusters



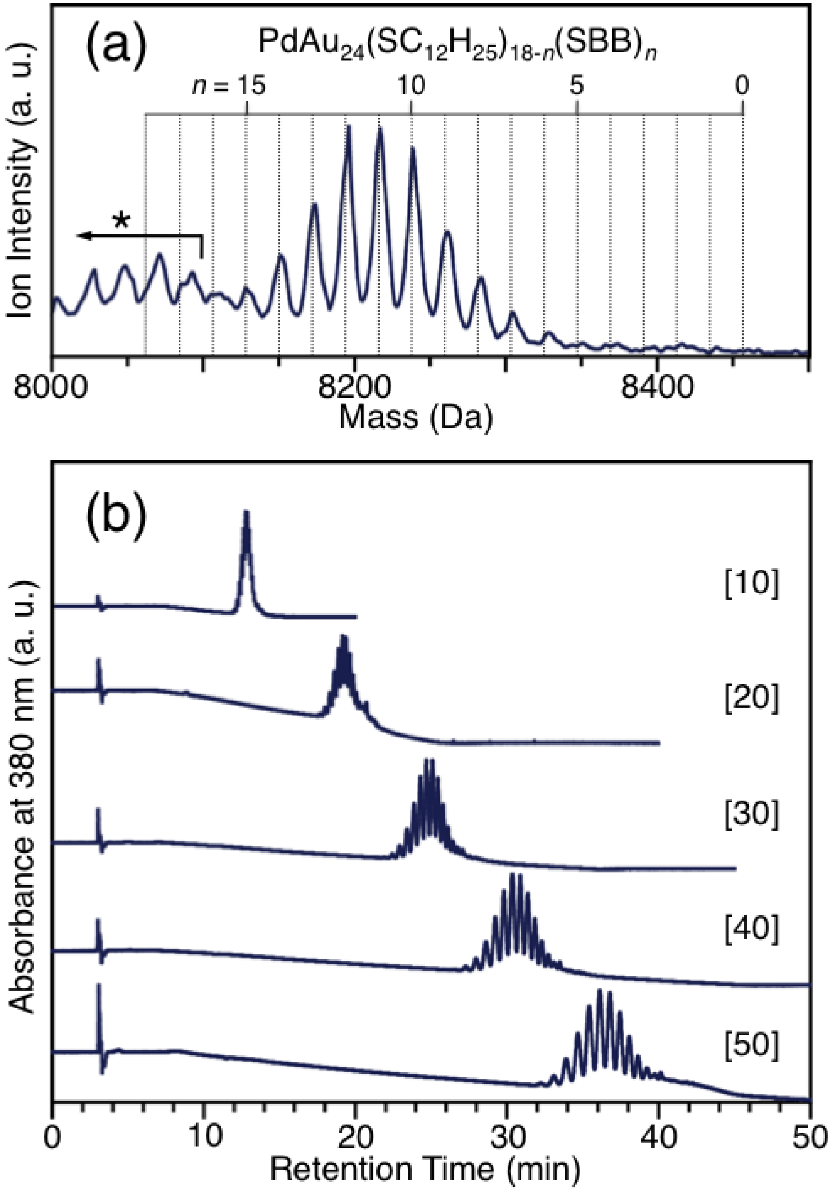
R1

Mixed functionalised clusters

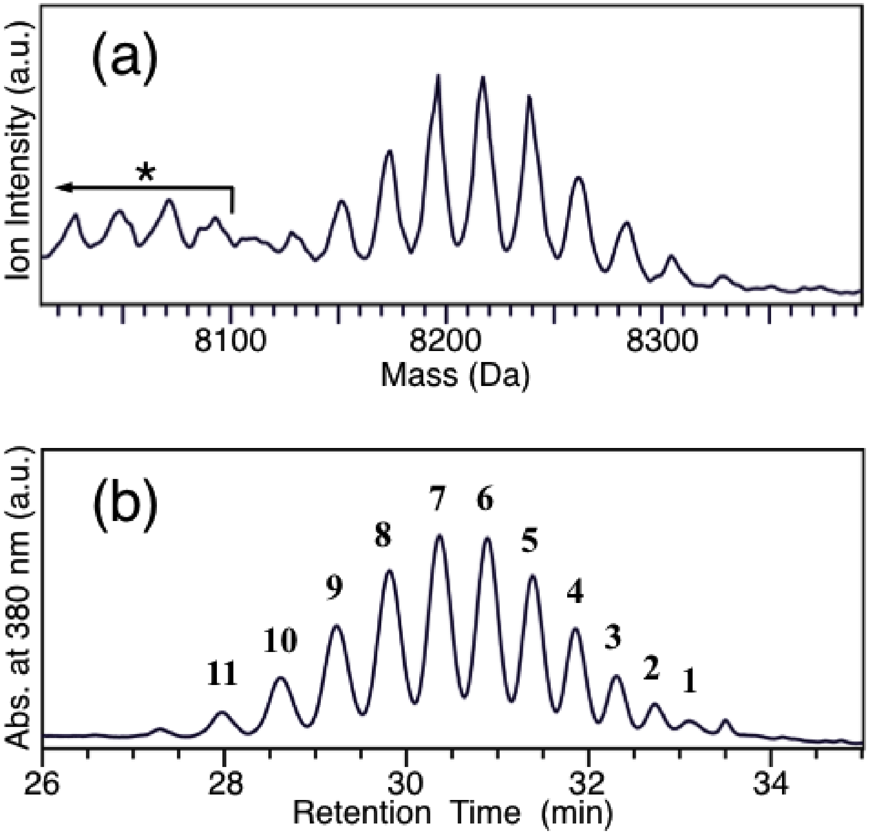
R2

Yoshiki Niihori, Miku Matsuzaki, T. Pradeep and Yuichi Negishi, J. Am. Chem. Soc., 135 (2013) 4946-4949

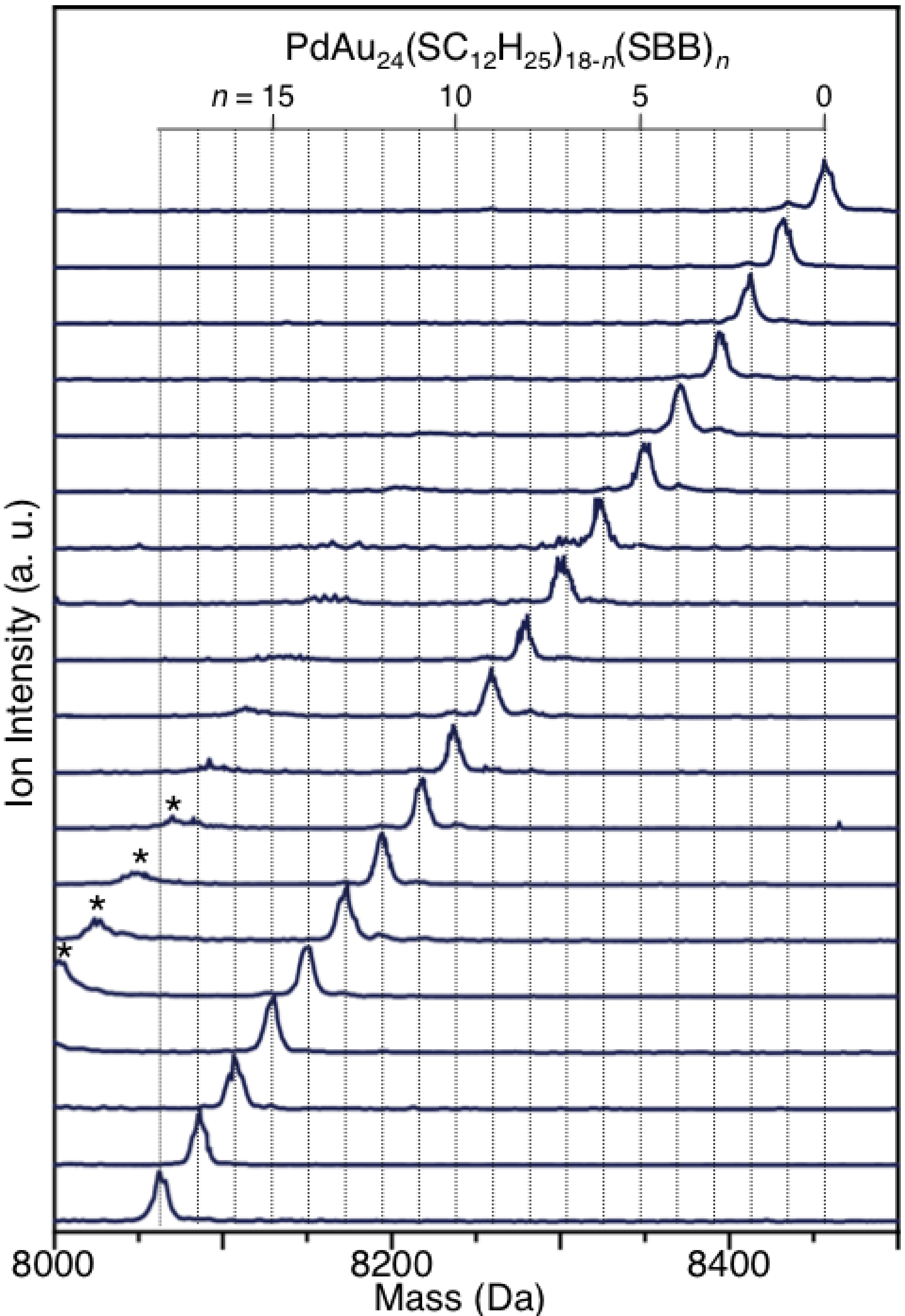
Separation of precise compositions of noble metal clusters protected with mixed ligands



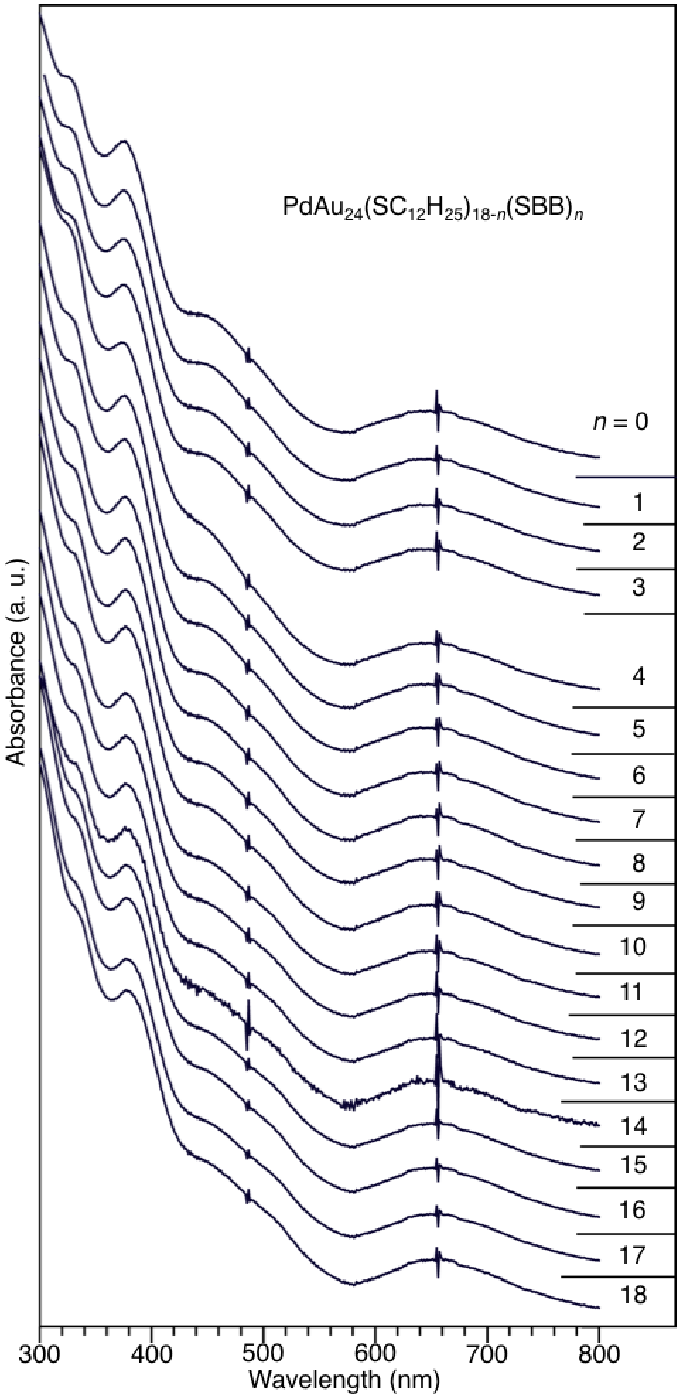
Ligand exchange chemistry – Substitution chemistry



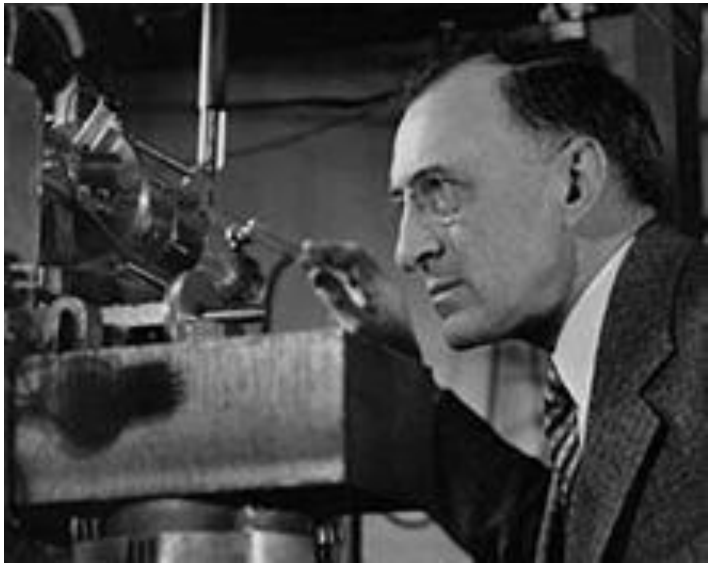
With Niihori and Negishi, Tokyo University of Science



$PdAu_{24}(SC_{12}H_{25})_{18-n}(SBB)_n$
n = 15
10
5
0
*
*
*
*
Ion Intensity (a. u.)
8000
8200
8400
Mass (Da)



$PdAu_{24}(SC_{12}H_{25})_{18-n}(SBB)_n$
n = 0
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
Absorbance (a. u.)
300
400
500
600
700
800
Wavelength (nm)



Arthur J. Dempster

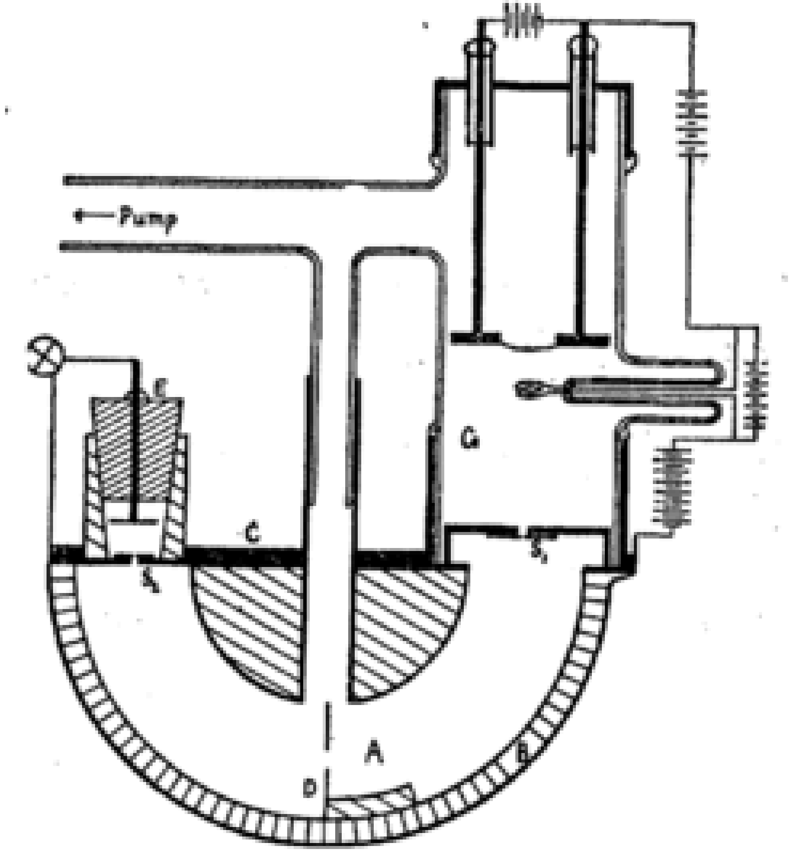


Fig. 1.

Dempster's 1918 mass spectrometer

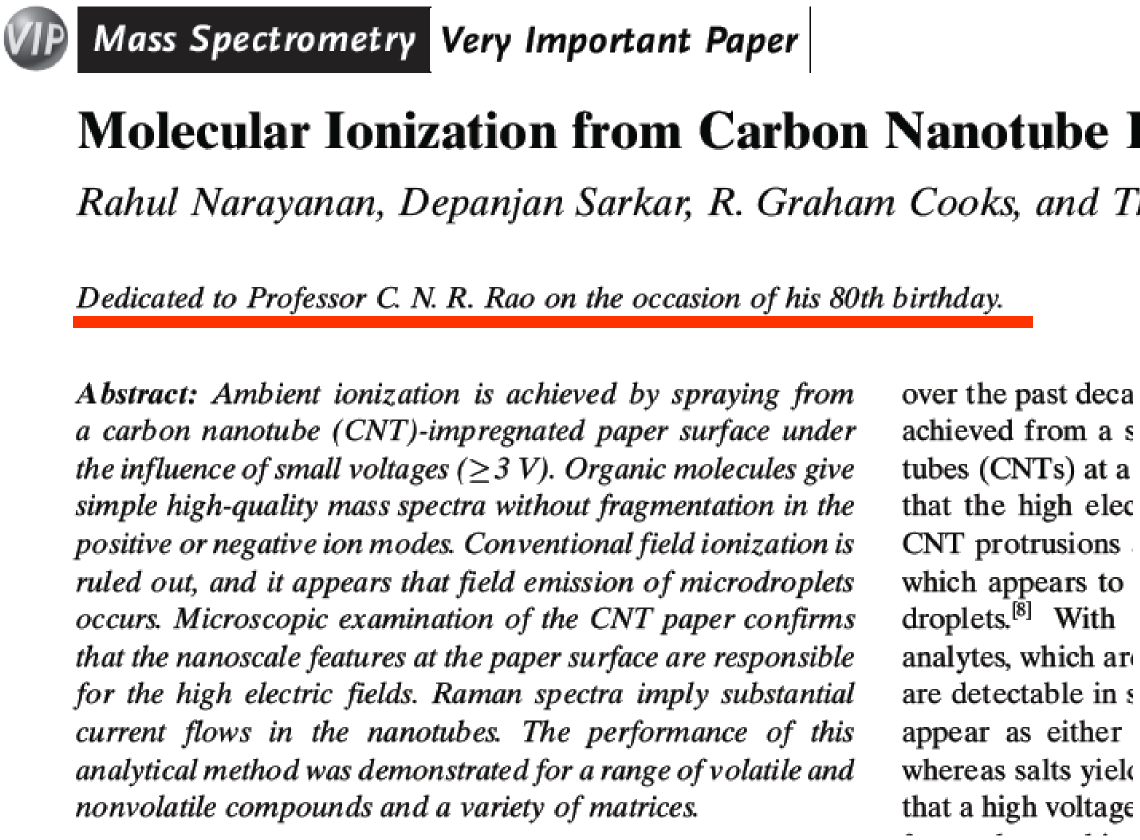
Brief history of mass spectrometry

1886 Eugen Goldstein observes canal rays. - gas discharge
1898 Wilhelm Wien demonstrates that canal rays can be deflected using strong electric and magnetic fields.
1898 J. J. Thomson measures the mass-to-charge ratio of electrons.
1901 Walter Kaufmann uses a mass spectrometer to measure the relativistic mass increase of electrons.
1905 J. J. Thomson begins his study of positive rays.
1906 Thomson's Nobel Prize in Physics
"in recognition of the great merits of his theoretical and experimental investigations on the conduction of electricity by gases"
1906 Gehrke and Reichenheim first produced anode rays by surface ionization (SI).
1913 Thomson is able to separate particles of different m/e ratios. He separates the ^{20}Ne and the ^{22}Ne isotopes, and identifies the *m/z* = 11 due to doubly charged ^{22}Ne particle.
1918 Dempster in his first experiment with his 180 degree spectrometer, used SI as the ionization method.
1919 Francis Aston constructs the first velocity focusing mass spectrograph with mass resolving power of 130.
1922 Aston's Nobel Prize in chemistry "for his discovery, by means of his
mass spectrograph, of isotopes, in a large number of non-radioactive elements,
and for his enunciation of the whole-number rule."
1931 Woodstock first observed secondary ion formation from bombarding ions. Herzog (1942) filed a German patent application for a SIMS analyzer.
1934 Josef Mattauch and Richard Herzog develop the double-fo
1936 Arthur J. Dempster develops the spark ionization source.
1937 Aston constructs a mass spectrograph with resolving powe
1937-47 Nier develops instruments for precise isotope ratio mea
1939 Lawrence receives the Nobel Prize in Physics for the cyclo
1942 Lawrence develops the Calutron for uranium isotope separ
1943 Westinghouse markets its mass spectrometer and proclaim
1946 William Stephens presents the concept of a time-of-flight mass spectrometer.
1954 A. J. C. Nicholson (Australia) proposes a hydrogen transfer reaction that will come to be known as the McLafferty rearrangement.
1954 Inghram and Gomer first demonstrated field ionization from a point source.
1959 Researchers at Dow Chemical interface a gas chromatograph to a mass spectrometer.
1964 British Mass Spectrometry Society established as first dedicated mass spectrometry society. It holds its first meeting in 1965 in London.
1966 F. H. Field and M. S. B. Munson develop chemical ionization.
1968 Malcolm Dole develops electrospray ionization.
1969 H. D. Beckey develops field desorption.
1974 Comisarow and Marshall develop Fourier Transform Ion Cyclotron Resonance mass spectrometry.
1976 Ronald MacFarlane and co-workers develop plasma desorption mass spectrometry.
1984 John Bennett Fenn and co-workers use electrospray to ionize biomolecules.
1985 Franz Hillenkamp, Michael Karas and co-workers describe and coin the term matrix-assisted laser desorption ionization (MALDI).
1987 Koichi Tanaka uses the "ultra fine metal plus liquid matrix method" to ionize intact proteins.
1989 Wolfgang Paul receives the Nobel Prize in Physics "for the development of the ion trap technique".
1999 Alexander Makarov presents the Orbitrap mass spectrometer.
2002 John Bennett Fenn and Koichi Tanaka were awarded one-quarter of the Nobel Prize in chemistry each "for the development of soft desorption ionisation methods ... for mass spectrometric analyses of biological macromolecules."

Ionization in mass spectrometry

1886 Eugen Goldstein observes canal rays due to gas discharge.
1906 Gehrke and Reichenheim first produced anode rays by surface ionization (SI).
1931 Woodstock first observed secondary ion formation from bombarding ions. Herzog (1942) filed a German patent application for an SIMS analyzer.
1936 Arthur J Dempster develops the spark ionization source.
1937-47 Nier develops instruments for precise isotope ratio measurements, which became the model for EI sources.
1954 Inghram and Gomer first demonstrated field ionization from a point source.
1966 F. H. Field and M. S. B. Munson develop chemical ionization.
1968 Malcolm Dole develops electrospray ionization.
1969 H. D. Beckey develops field desorption.
1976 Ronald MacFarlane and co-workers develop plasma desorption mass spectrometry.
1984 John Bennett Fenn and co-workers use electrospray to ionize biomolecules.
1985 Franz Hillenkamp, Michael Karas and co-workers describe and coin the term matrix-assisted laser desorption ionization (MALDI).
1987 Koichi Tanaka uses the “ultra fine metal plus liquid matrix method” to ionize intact proteins.

Method 1



Mass Spectrometry *Very Important Paper*

DOI: 10.1002/anie.201311053

Molecular Ionization from Carbon Nanotube Paper**

*Rahul Narayanan, Depanjan Sarkar, R. Graham Cooks, and Thalappil Pradeep**

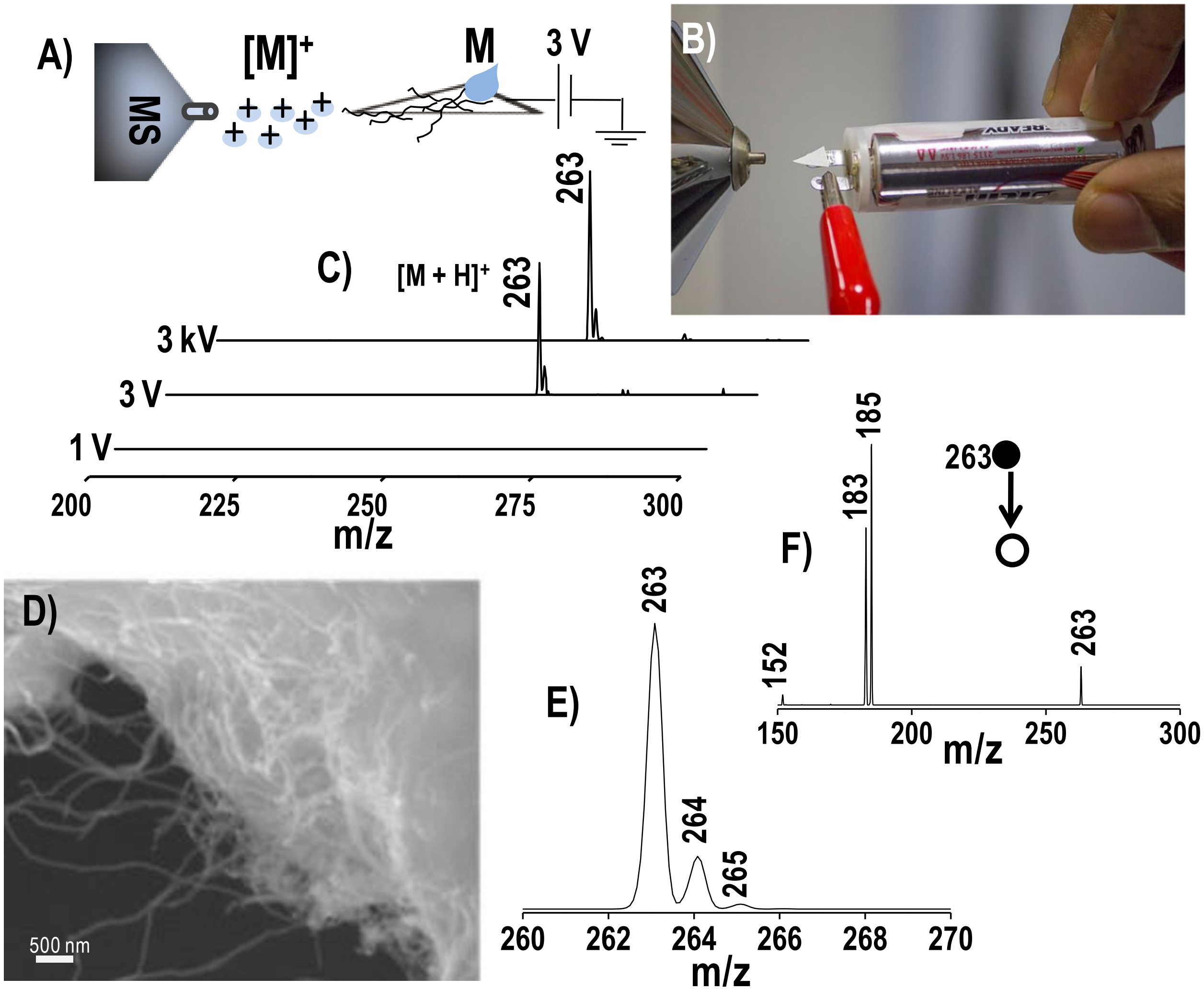
Dedicated to Professor C. N. R. Rao on the occasion of his 80th birthday.

Abstract: *Ambient ionization is achieved by spraying from a carbon nanotube (CNT)-impregnated paper surface under the influence of small voltages (≥3 V). Organic molecules give simple high-quality mass spectra without fragmentation in the positive or negative ion modes. Conventional field ionization is ruled out, and it appears that field emission of microdroplets occurs. Microscopic examination of the CNT paper confirms that the nanoscale features at the paper surface are responsible for the high electric fields. Raman spectra imply substantial current flows in the nanotubes. The performance of this analytical method was demonstrated for a range of volatile and nonvolatile compounds and a variety of matrices.*

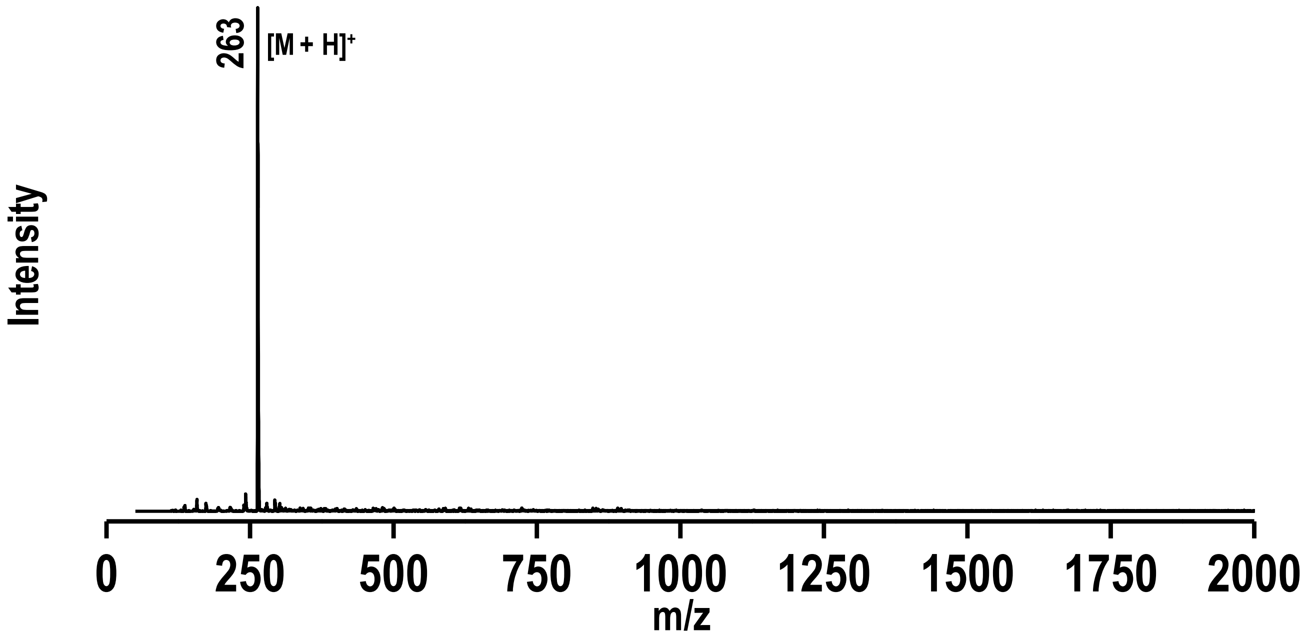
over the past deca
achieved from a s
tubes (CNTs) at a
that the high elec
CNT protrusions
which appears to
droplets.[8] With
analytes, which ar
are detectable in s
appear as either
whereas salts yiel
that a high voltage

A piece of paper ...

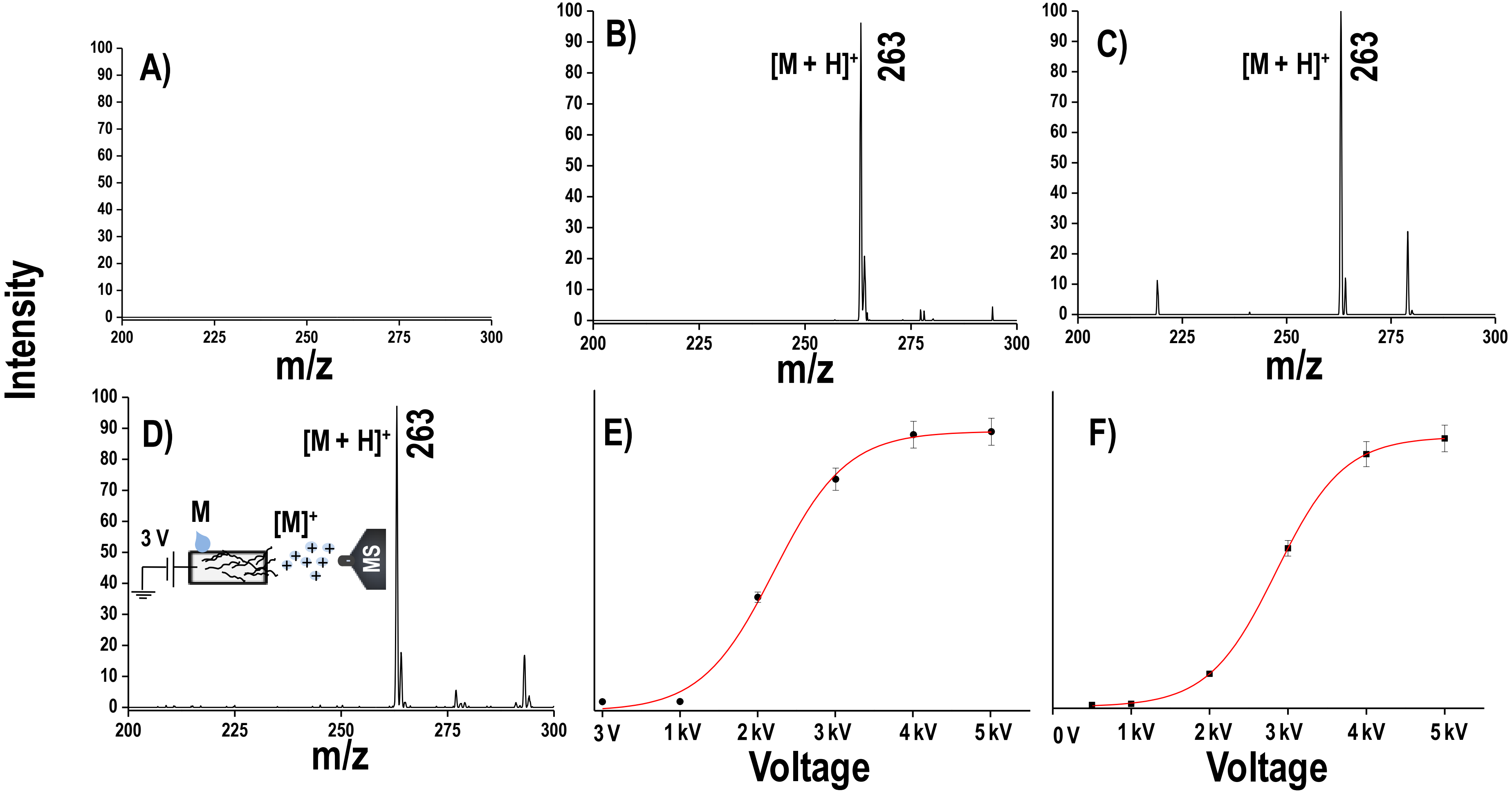
... that is impregnated with multi-walled or single-walled carbon nanotubes generates ions from diverse analytes at voltages as low as 3 V, as T. Pradeep et al. show in their Communication on page 5936 ff. This miniaturized ion source is held in front of a mass-spectrometer inlet to collect the mass spectrum. Common pesticides from the surface of an orange, active molecules from tablets, and a variety of analytes, such as amino acids, can been characterized.



A) Schematic diagram of ionization from CNT paper, B) Optical photograph of the ionization source C) mass spectrum of triphenylphosphine (M) at 3 kV, 3 V and 1 V from wet CNT paper, D) FE-SEM image of CNT-coated paper, E) isotope distribution of the protonated molecule at 3 V and F) product ion MS^2 of *m/z* 263.

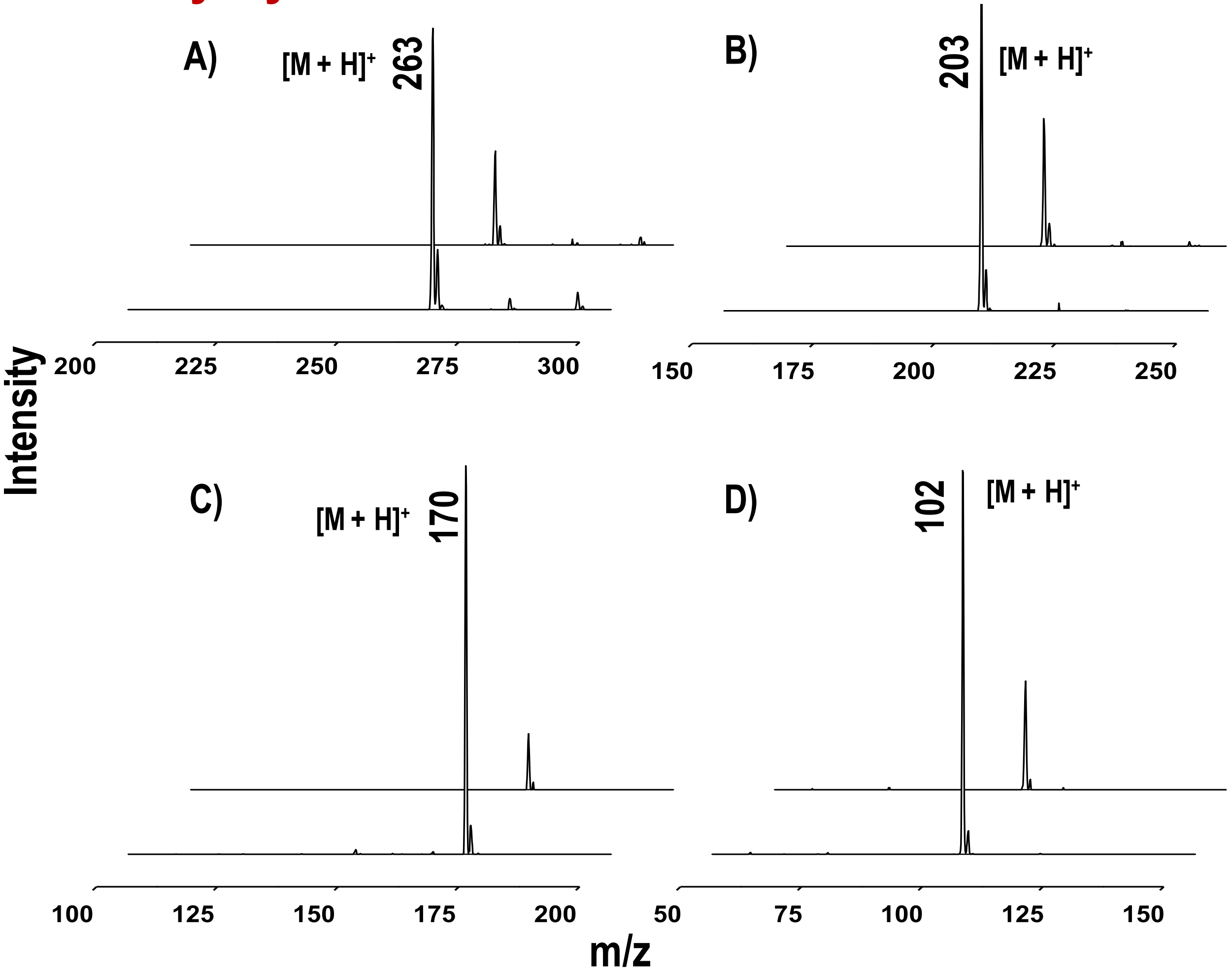


Full range mass spectrum of triphenylphosphine at 3 V



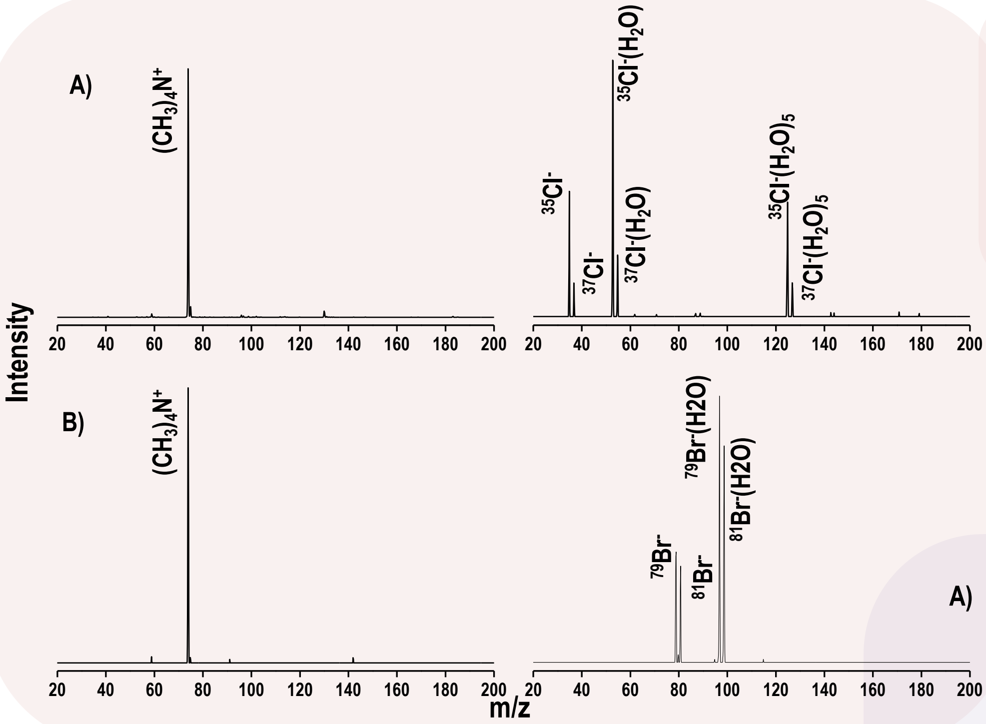
A) Mass spectrum of TPP below 500 V using normal paper B) spectrum of TPP at 3 V using CNT-coated paper C) spectrum at 500 V from a normal paper, D) spectrum using rectangular CNT-coated paper and the inset shows the schematic, E) variation of intensity of the m/z 263 peak with voltage for CNT-coated paper and F) the same for normal paper.

Increase in intensity by the addition of acid:

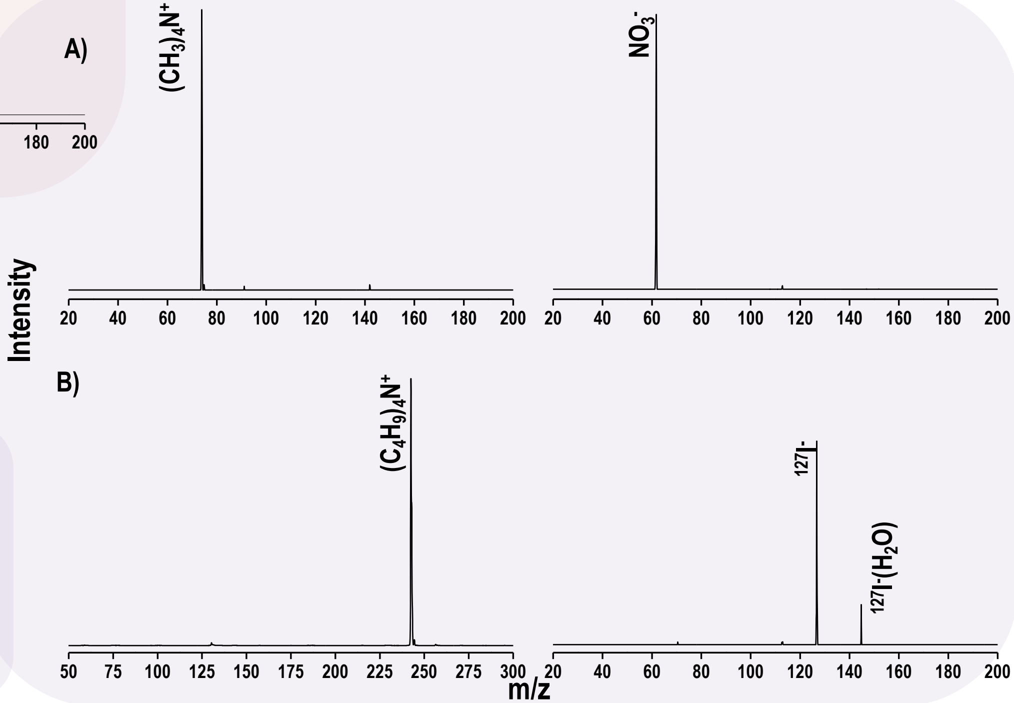


Intensity enhancement upon the addition of dil. HCl for various analytes (M) at 3 V, A) triphenylphosphine, B) tributylphosphine, C) diphenylamine and D) triethylamine. The top spectrum is without HCl addition.

Analysis of preformed ions:

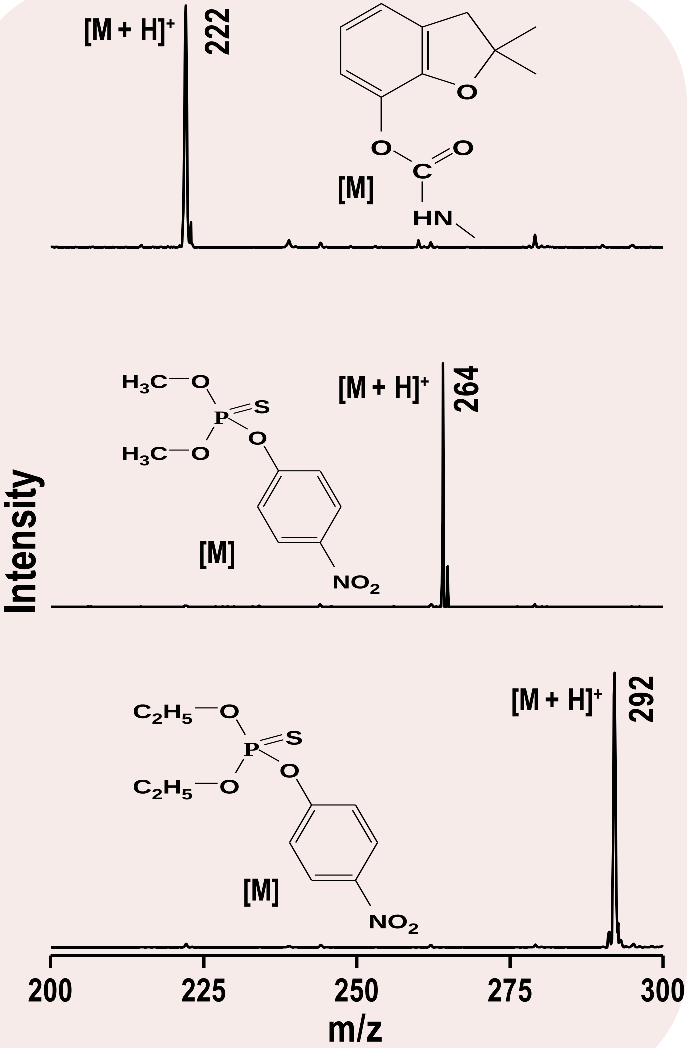


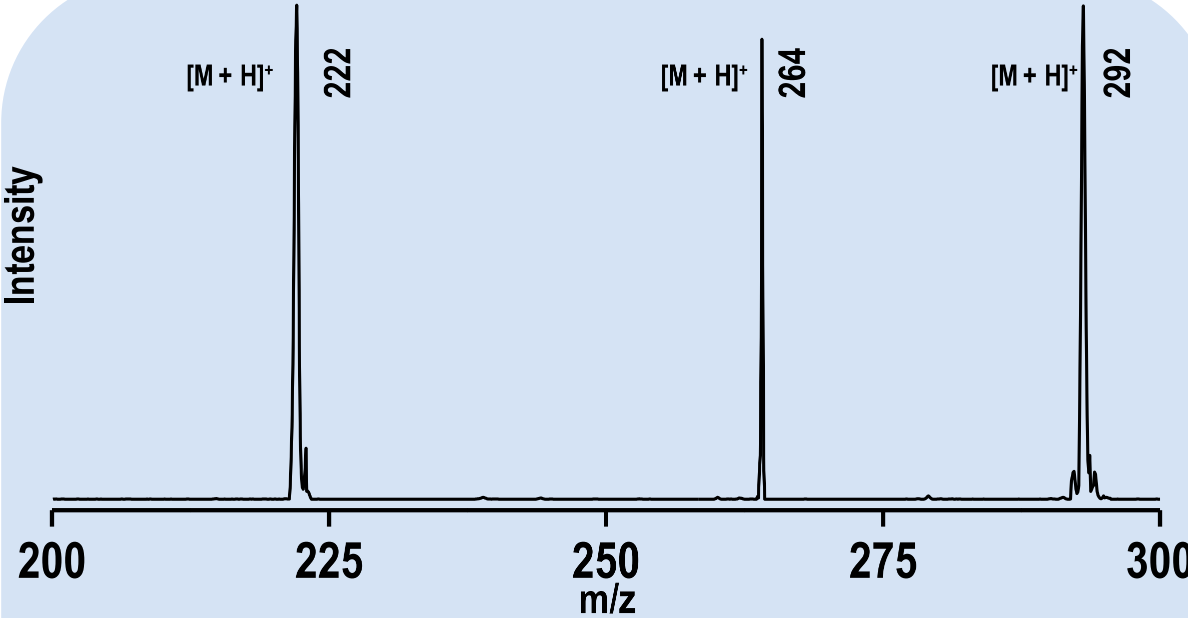
Analysis of preformed ions (positive and negative ion modes) at 3 V; A) tetramethylammonium chloride and B) tetramethylammonium bromide.



Analysis of preformed ions (positive and negative ion modes) at 3 V; A) tetramethylammonium nitrate and B) tetrabutylammonium iodide.

Detection of pesticides from an orange surface:

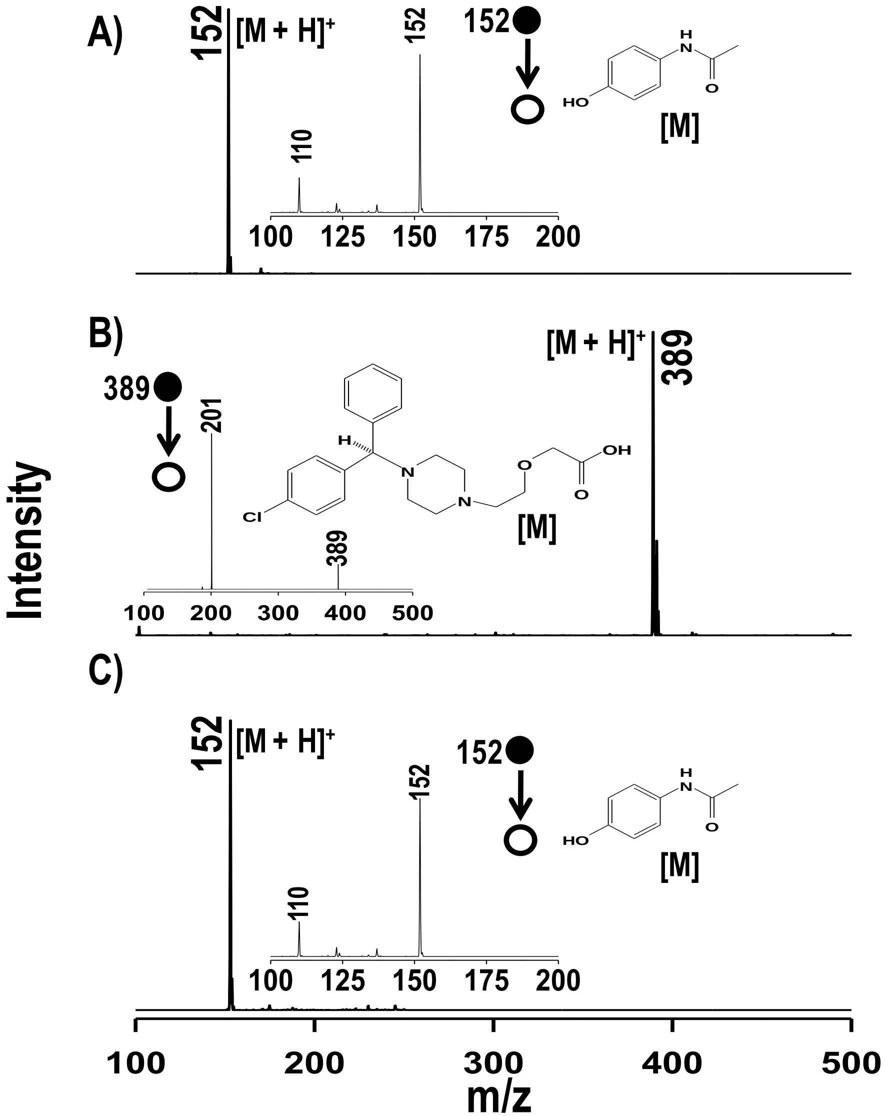




Analysis of a pesticide mixture at 3 V from the surface of an orange. Isotopic distribution of the peaks is not clearly visible due to low intensity.

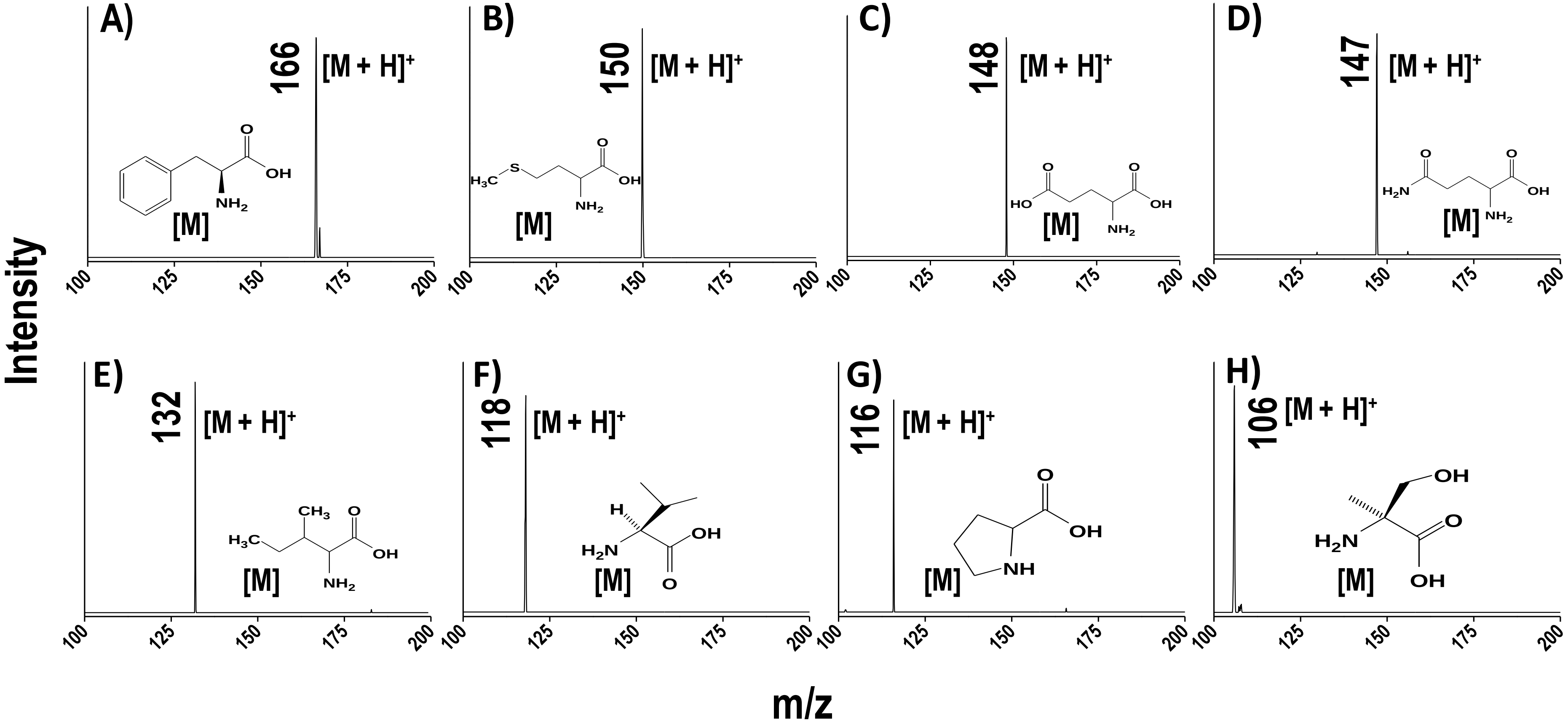
Detection of pesticides examined individually from the surface of an orange. A) Carbofuran, B) methyl parathion and C) parathion.

Analysis of commercial tablets:



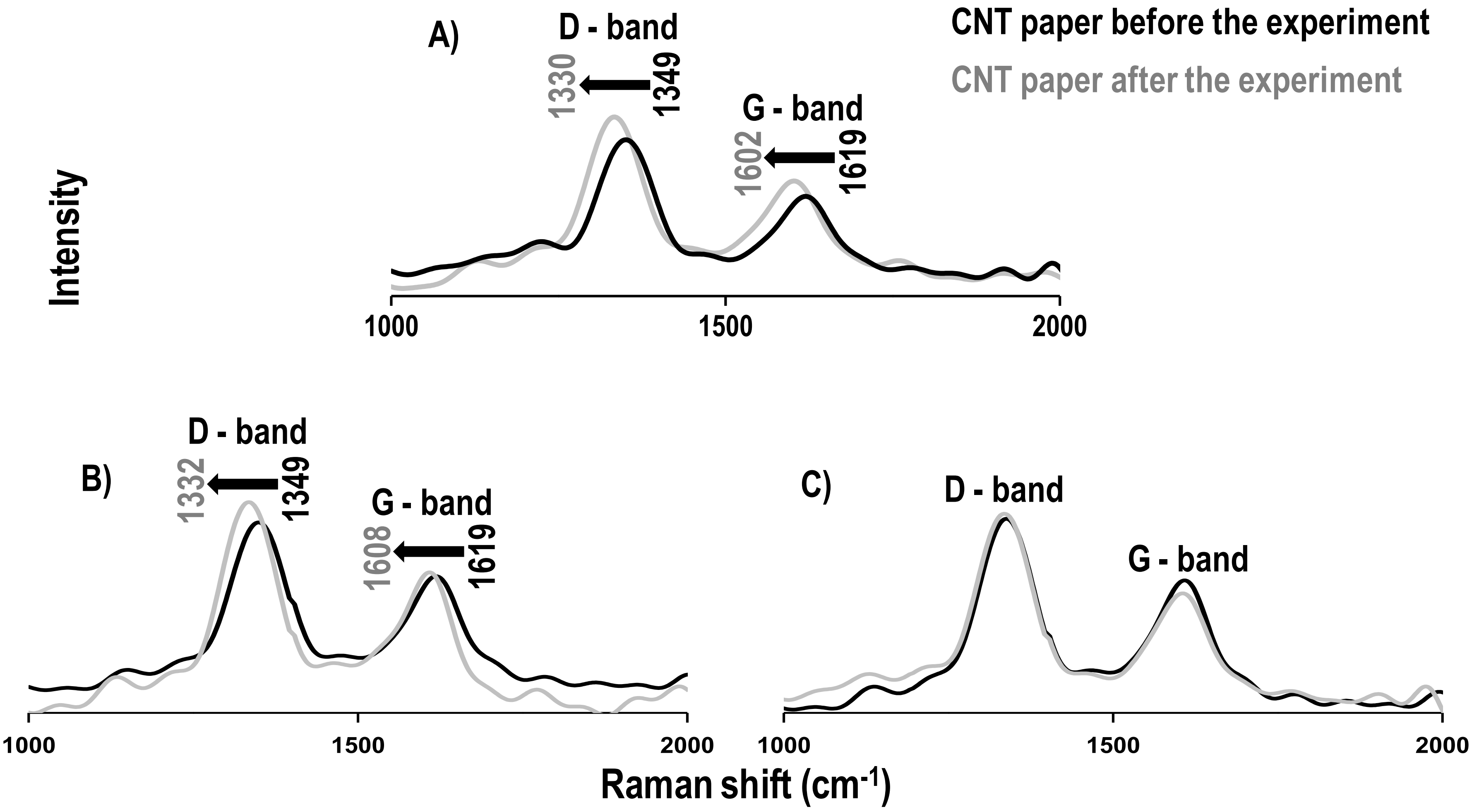
Analysis of tablets from CNT-coated paper at 3 V with their mass spectral and MS^2 data. A) Crocine (paracetamol), B) xyzal (levocetirizine dihydrochloride) and C) combiflam (paracetamol).

Analysis of amino acids:

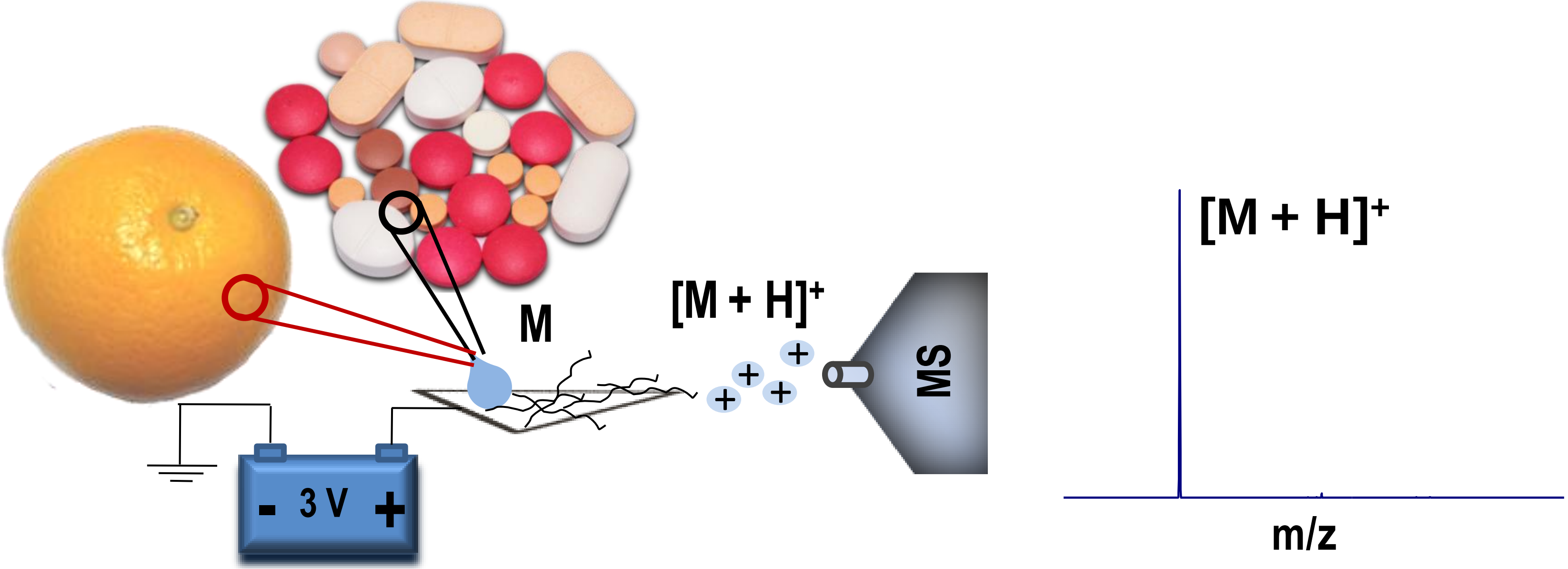


Detection of various amino acids (90 ng) loaded on CNT-coated paper and spectra recorded at 3 V: A) phenylalanine, B) methionine, C) glutamic acid, D) glutamine, E) isoleucine, F) valine, G) proline and H) serine.

Raman analysis of the paper:

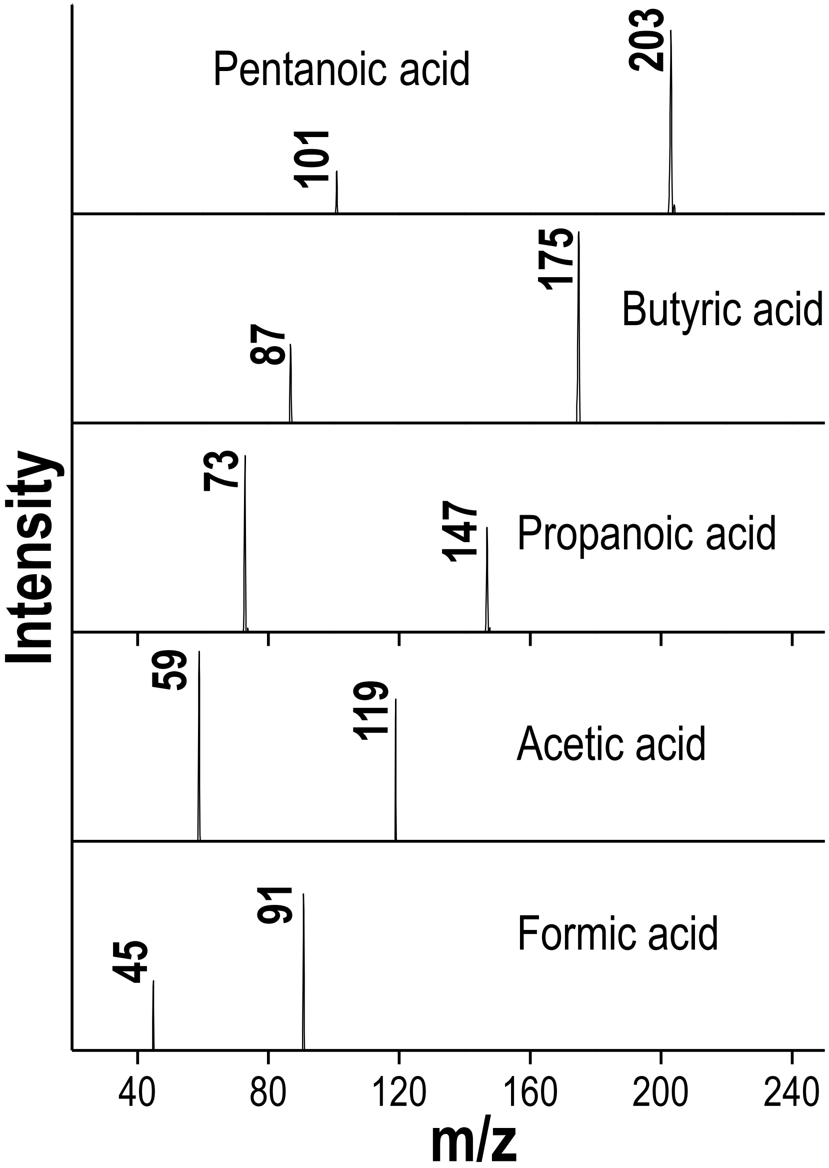


Raman measurement of CNT-coated paper before and after ionization. A) Neutral molecules (30 ppm TTP in MeOH/H_2O) and preformed ions (tetramethylammonium bromide) in B) positive and C) negative ion modes.

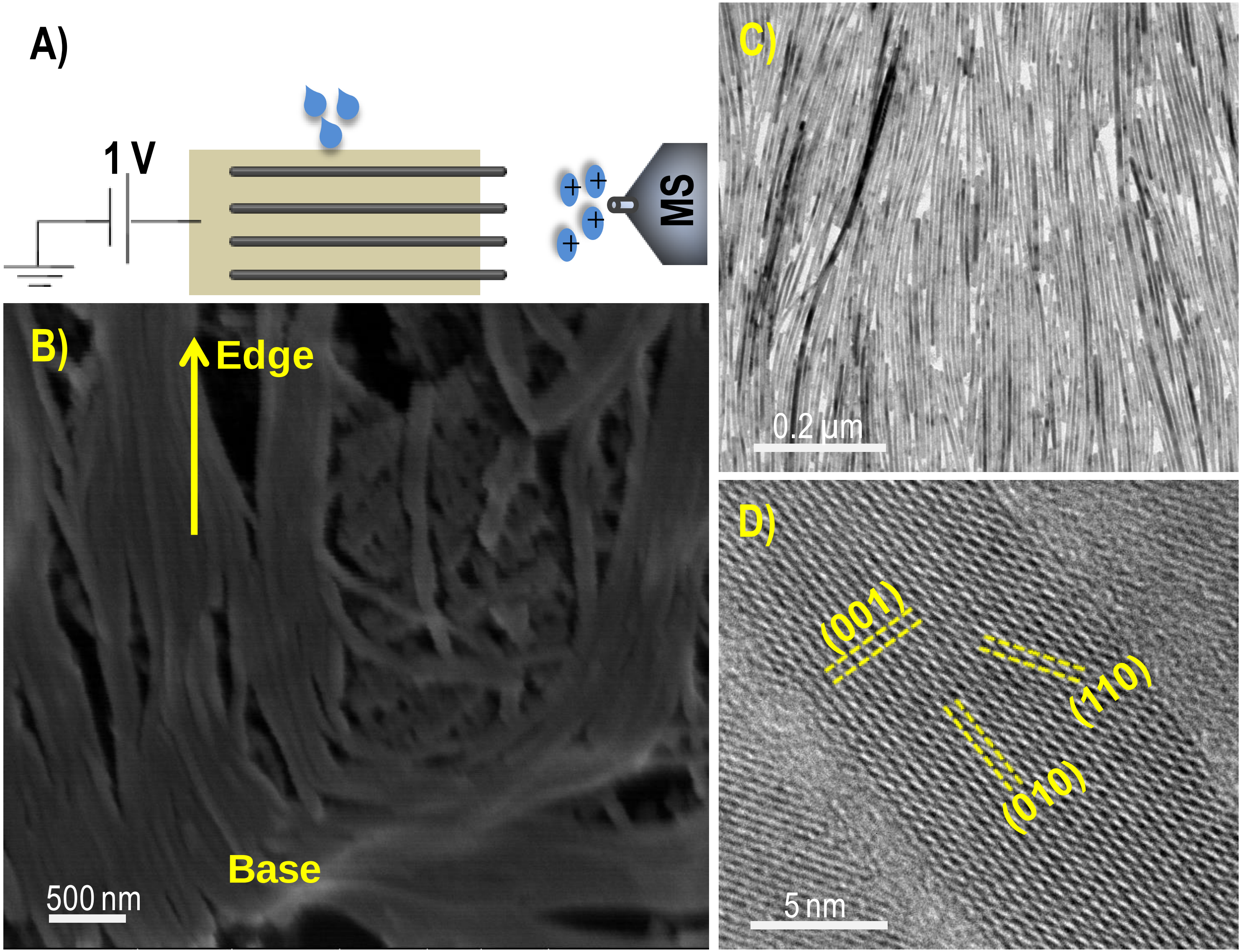

M
[M + H]+
MS
- 3 V +
[M + H]+
m/z

Internal energy

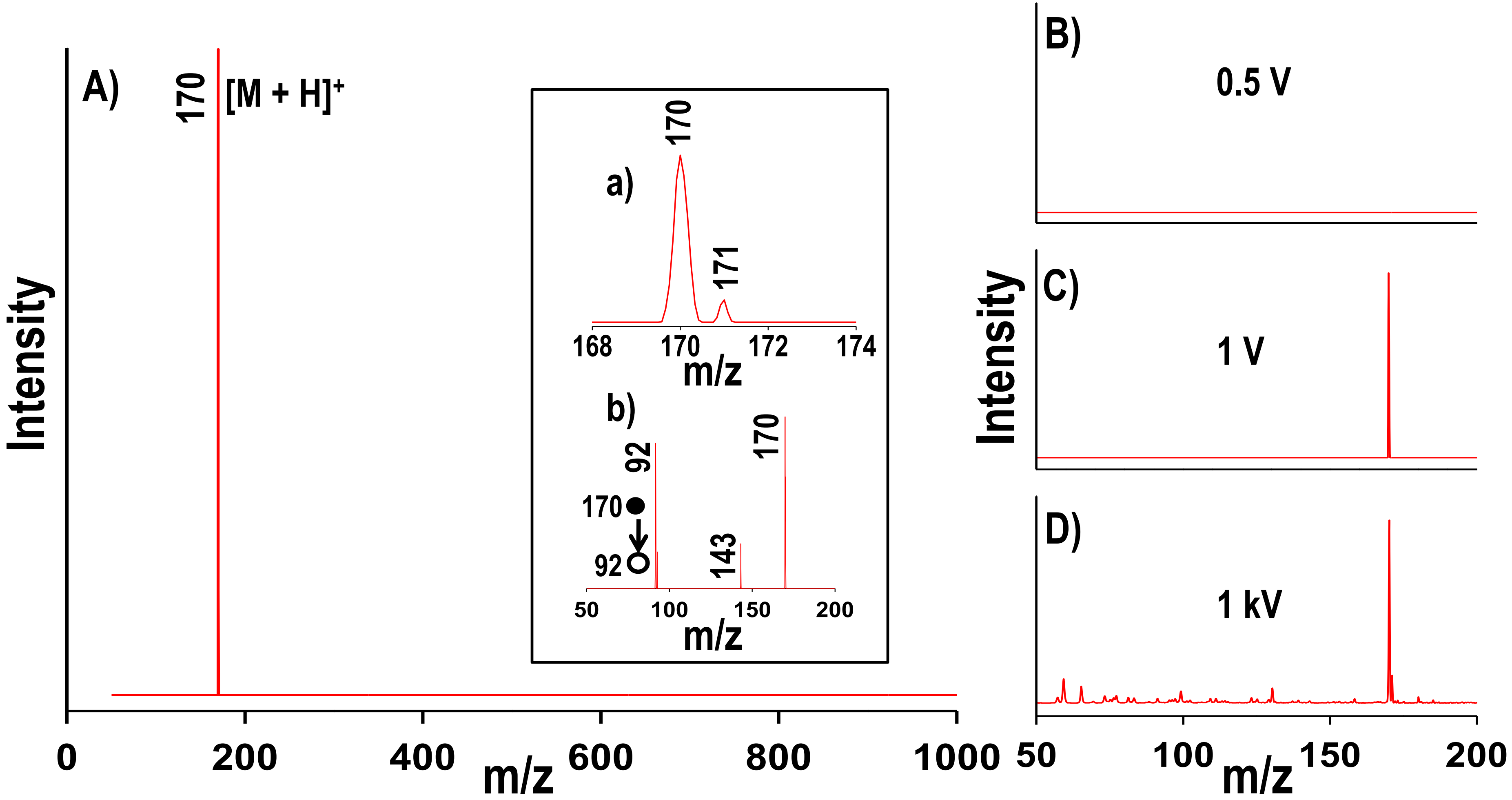
3 V


Pentanoic acid
203
101
Butyric acid
175
87
Propanoic acid
73
147
Acetic acid
59
119
Formic acid
91
45
Intensity
40
80
120
160
200
240
m/z

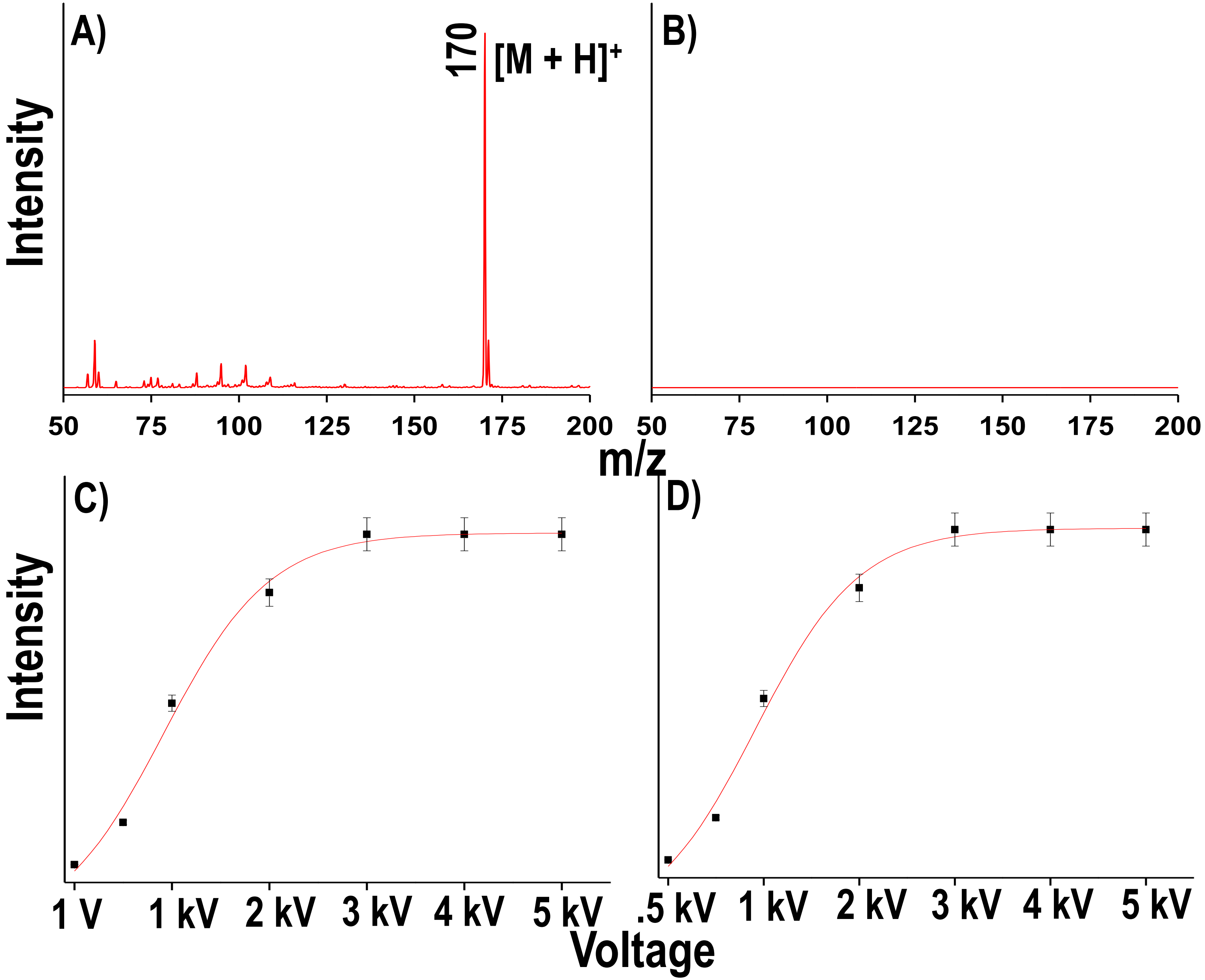
Molecular ionization at 1 V from Te nanowires



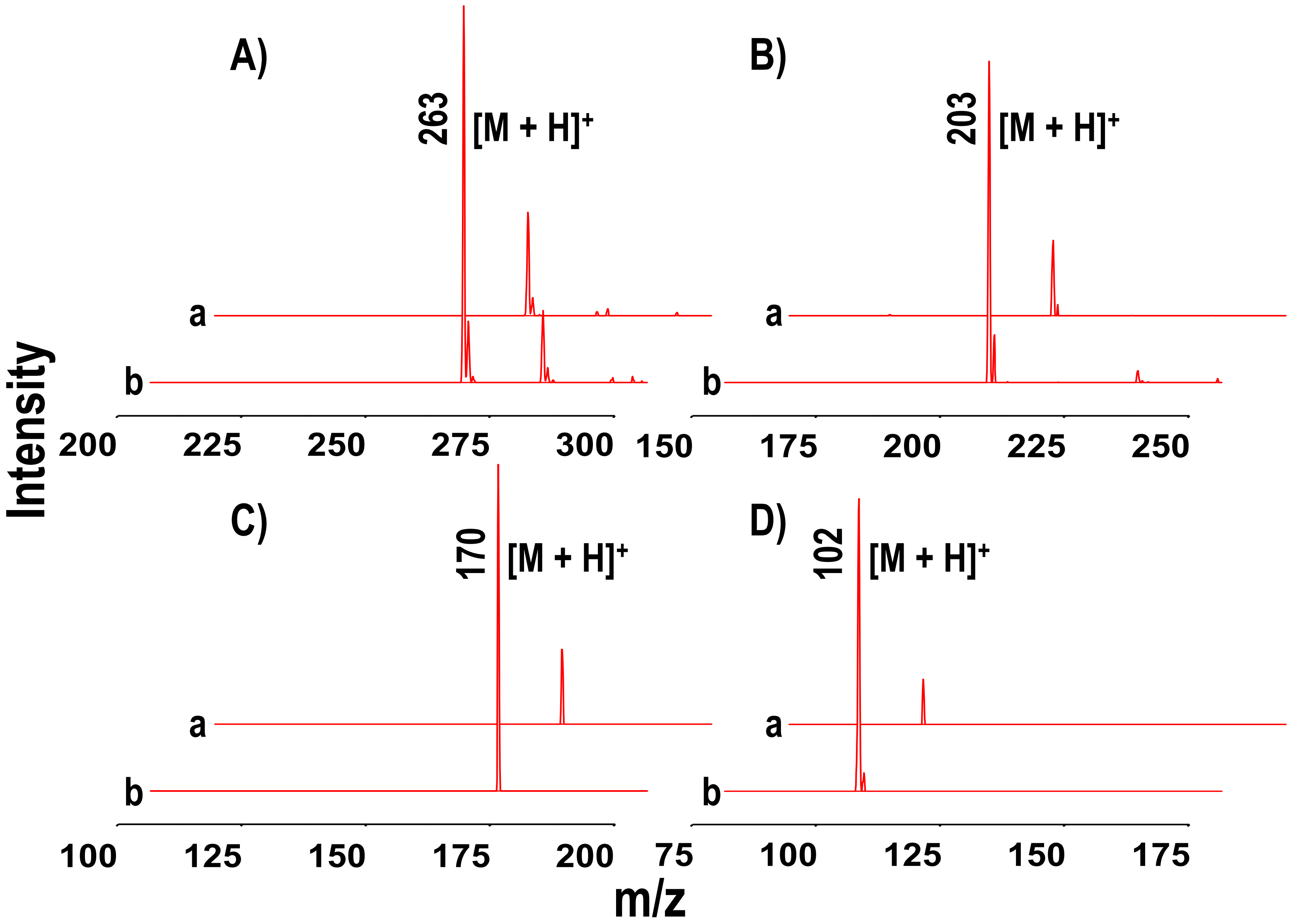
A) Schematic of the ionization process, B) FE-SEM image of aligned Te NWs-coated paper, C) low magnification TEM image of aligned Te NWs and D) high magnification HRTEM image of Te NWs. Various lattice planes observed are marked. Edge of the paper substrate is marked in B. The NWs coat the surface uniformly and a thin coating changes the color of the paper.



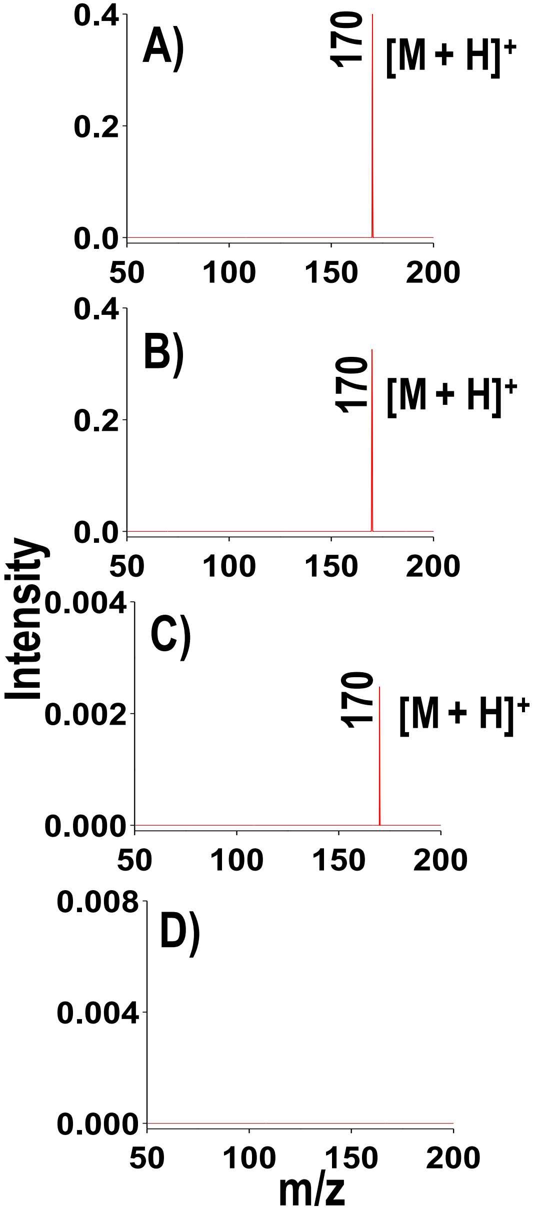
A) Full range mass spectrum of DPA (isotopic distribution of the molecular ion peak (a) and MS^2 of molecular ion peak (b) are shown in the inset) and B), C), and, D) mass spectra of DPA at 0.5 V, 1 V and 1 kV, respectively.



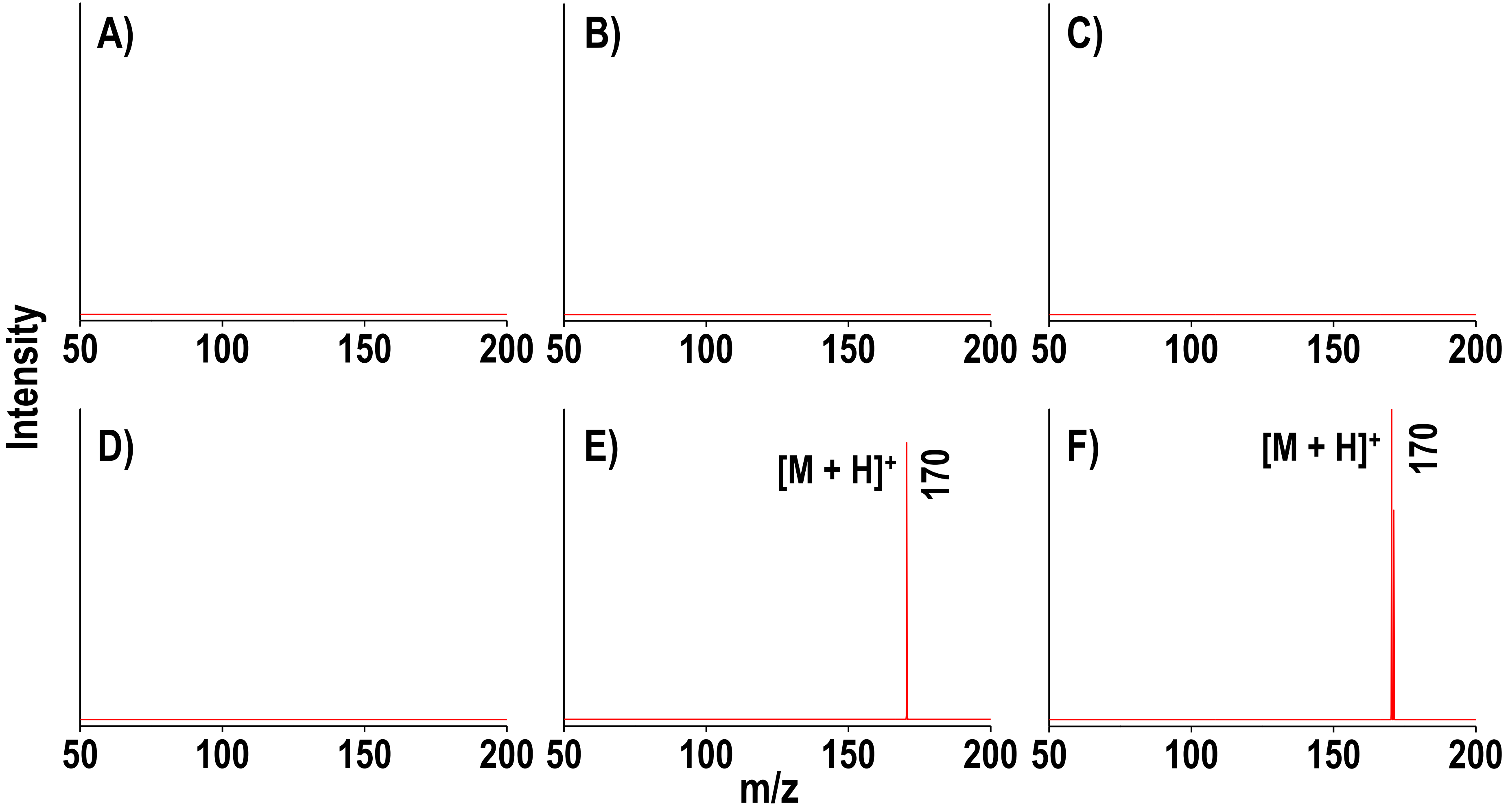
A) Mass spectrum of DPA from a normal paper at 500 V, B) spectrum below an applied potential of 500 V, C) variation of signal intensity with voltage from a Te NW-coated paper, and D) variation of signal intensity with voltage from a normal filter paper.



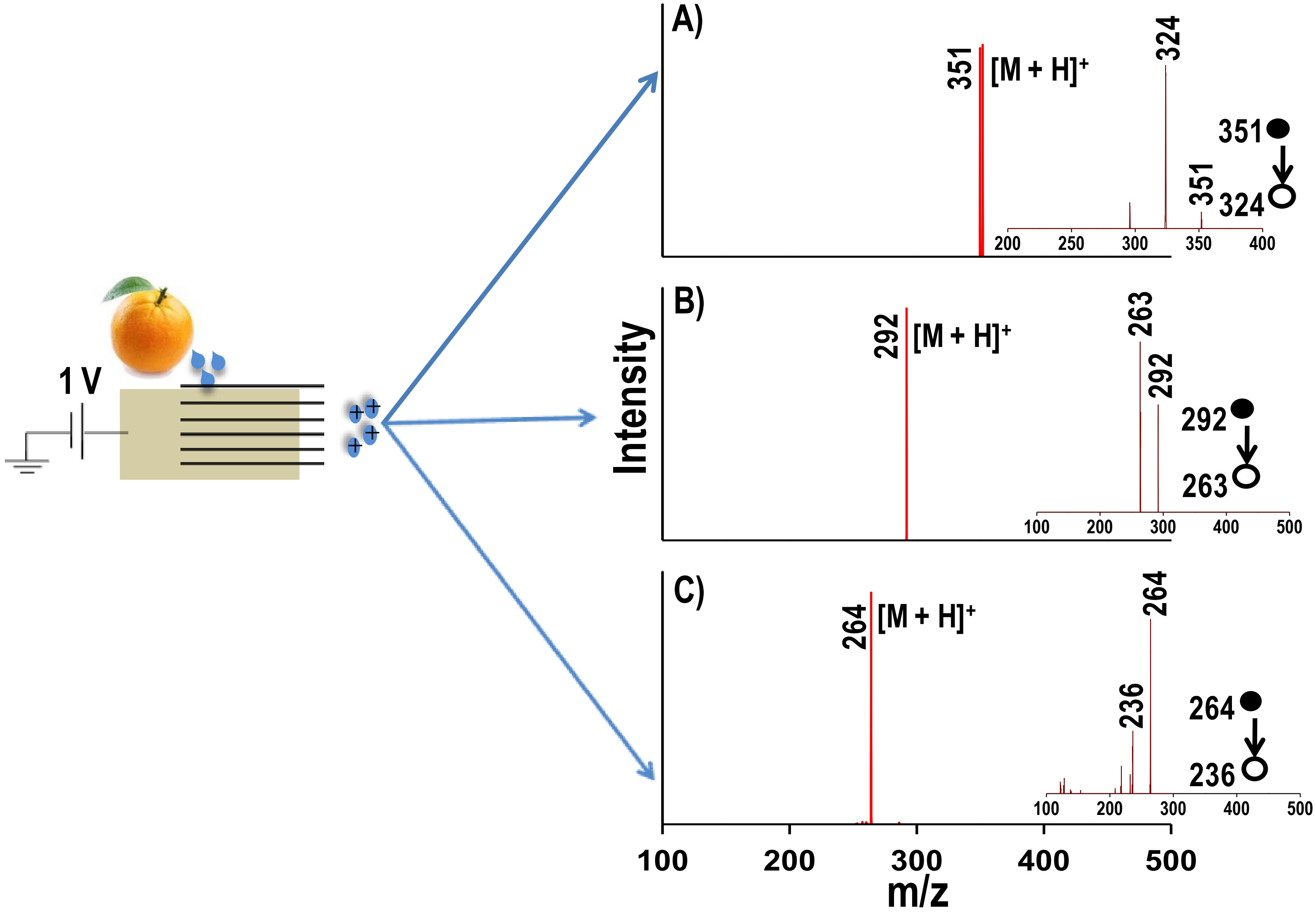
Signal intensity enhancement for various analytes (a – before adding HCl, and b – after adding HCl). A) Triphenylphosphine, B) tributyl phosphine, C) diphenylamine, and D) triethylamine.



Mass spectrum of diphenylamine in different solvents. A) Methanol, B) ethanol, C) propanol, and D) butanol.

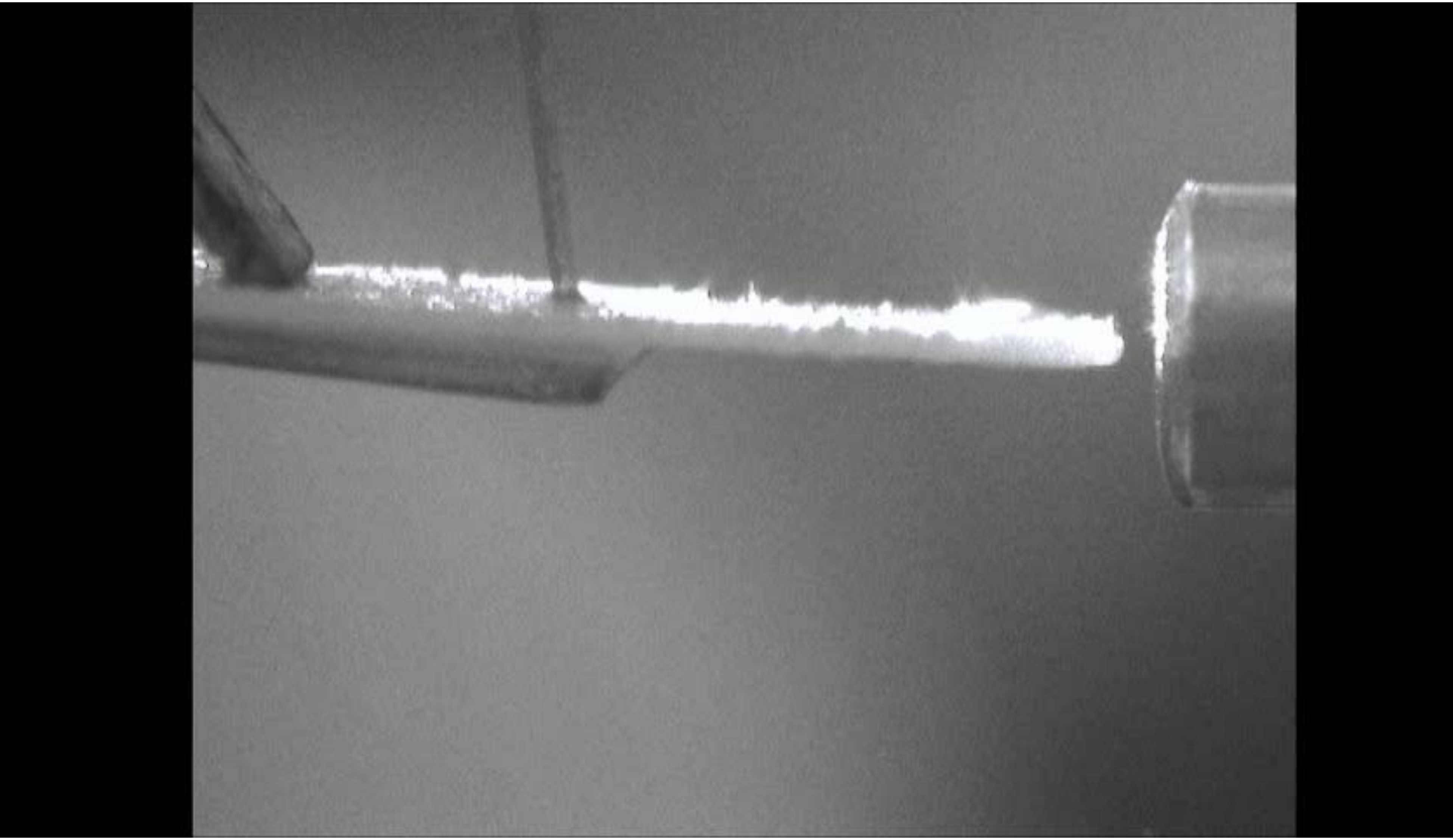


Mass spectrum of diphenylamine in butanol of different conductivity. A) Butanol without sodium acetate, B) butanol with 1 ppm sodium acetate, C) butanol with 10 ppm sodium acetate, D) butanol with 50 ppm sodium acetate, E) butanol with 100 ppm sodium acetate, and F) butanol with 500 ppm sodium acetate.



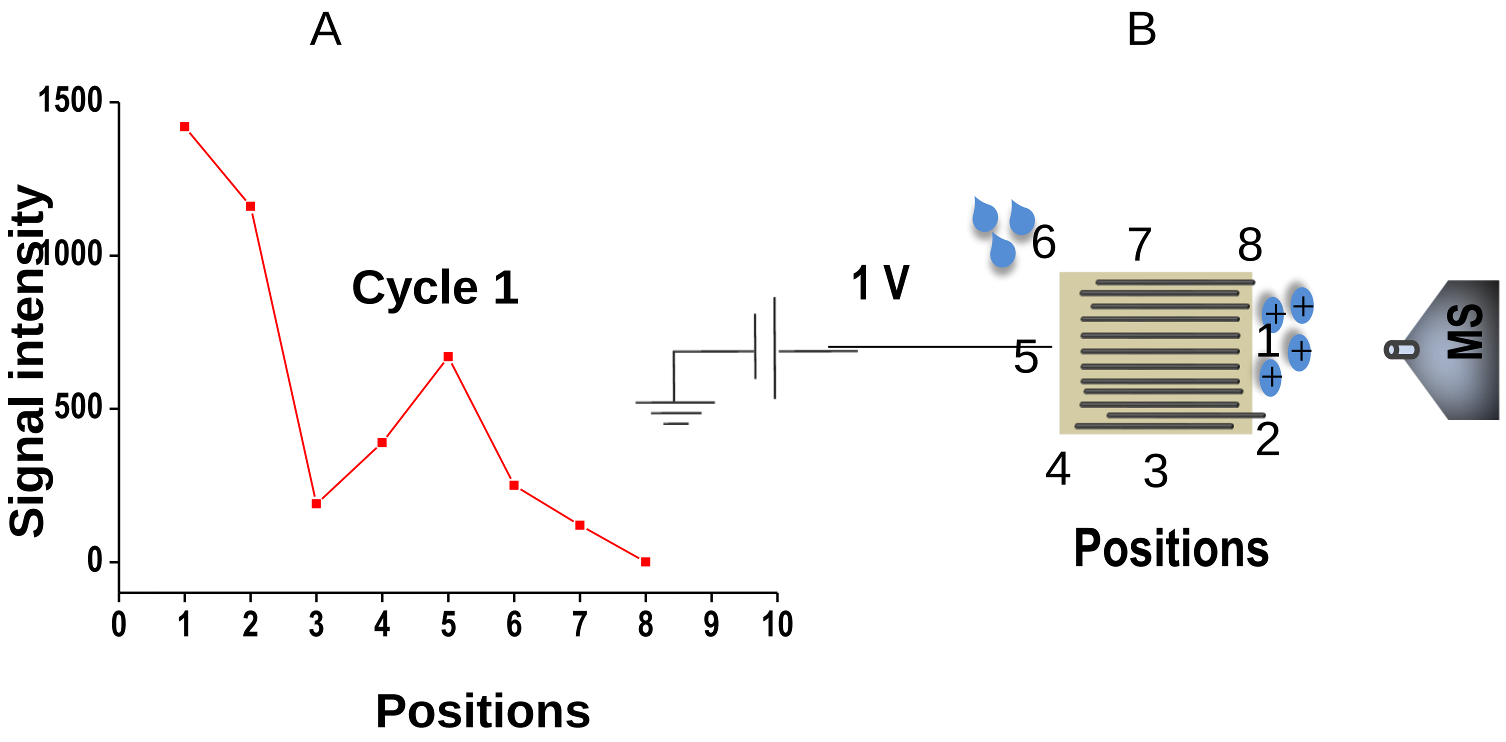
Detection of various pesticides from the surface of an orange at 1 V using Te NW-coated paper. A) Chloropyrifos, B) parathion and C) methylparathion. MS^2 data are given along with the mass spectra.

Rahul, Depanjan, et al. Unpublished



From R. G. Cooks

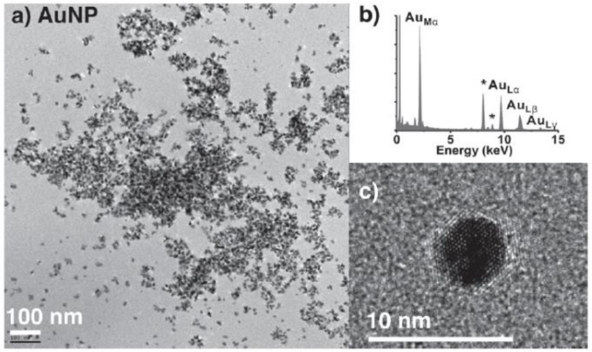
Ionization is aniosotropic

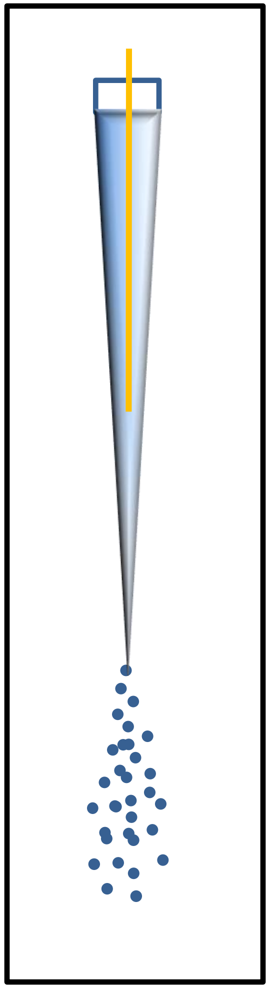


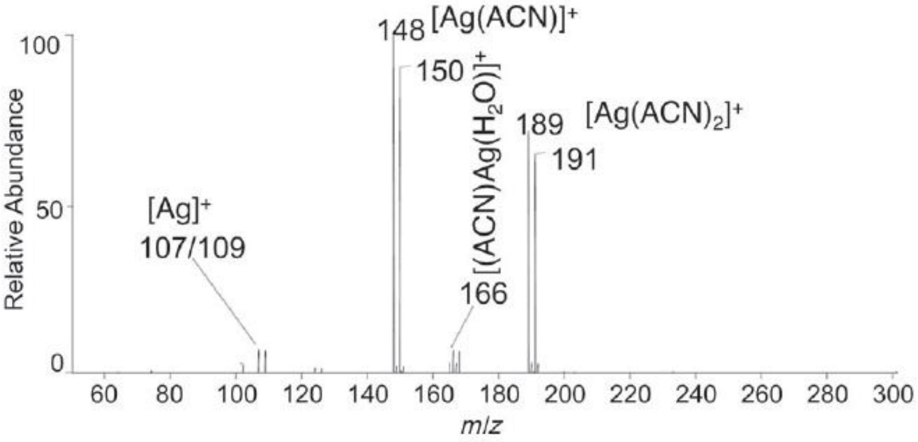
A) Variation of molecular ion intensity of diphenylamine with different positions of nanowire coated glass substrate (positions are marked in the schematic shown in the inset). B) Schematic of the alignment of nanowires with positions.

Method 2

Electrolytic spray deposition

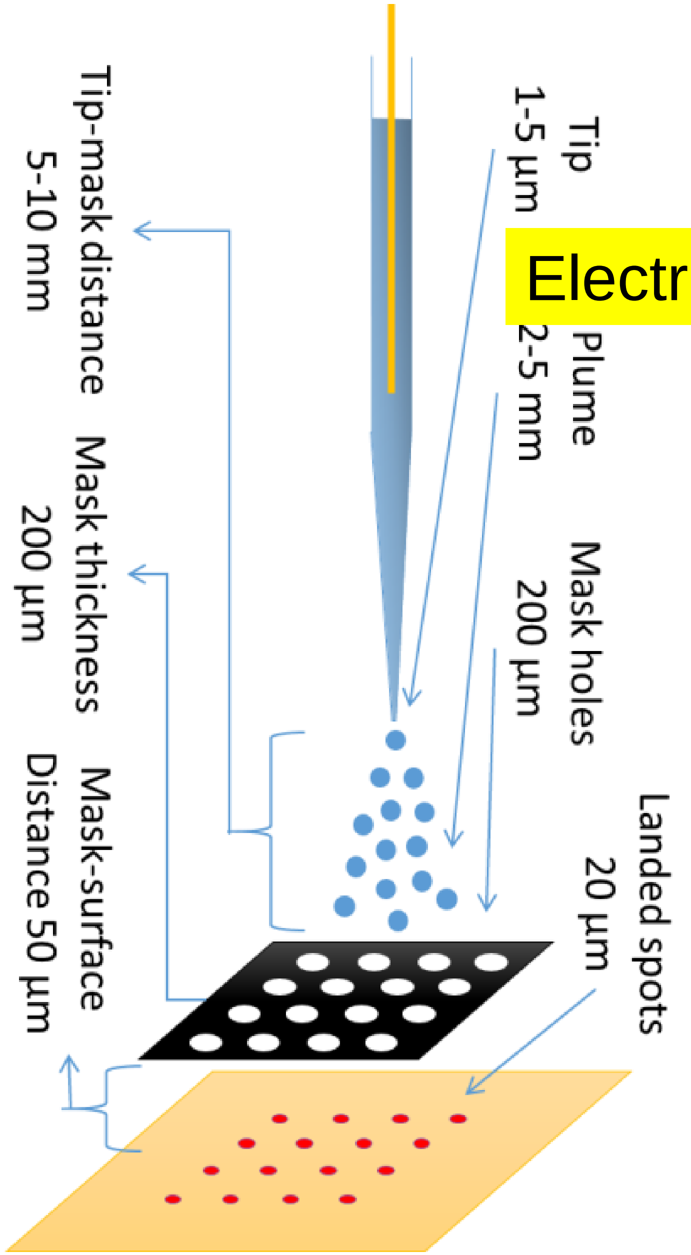




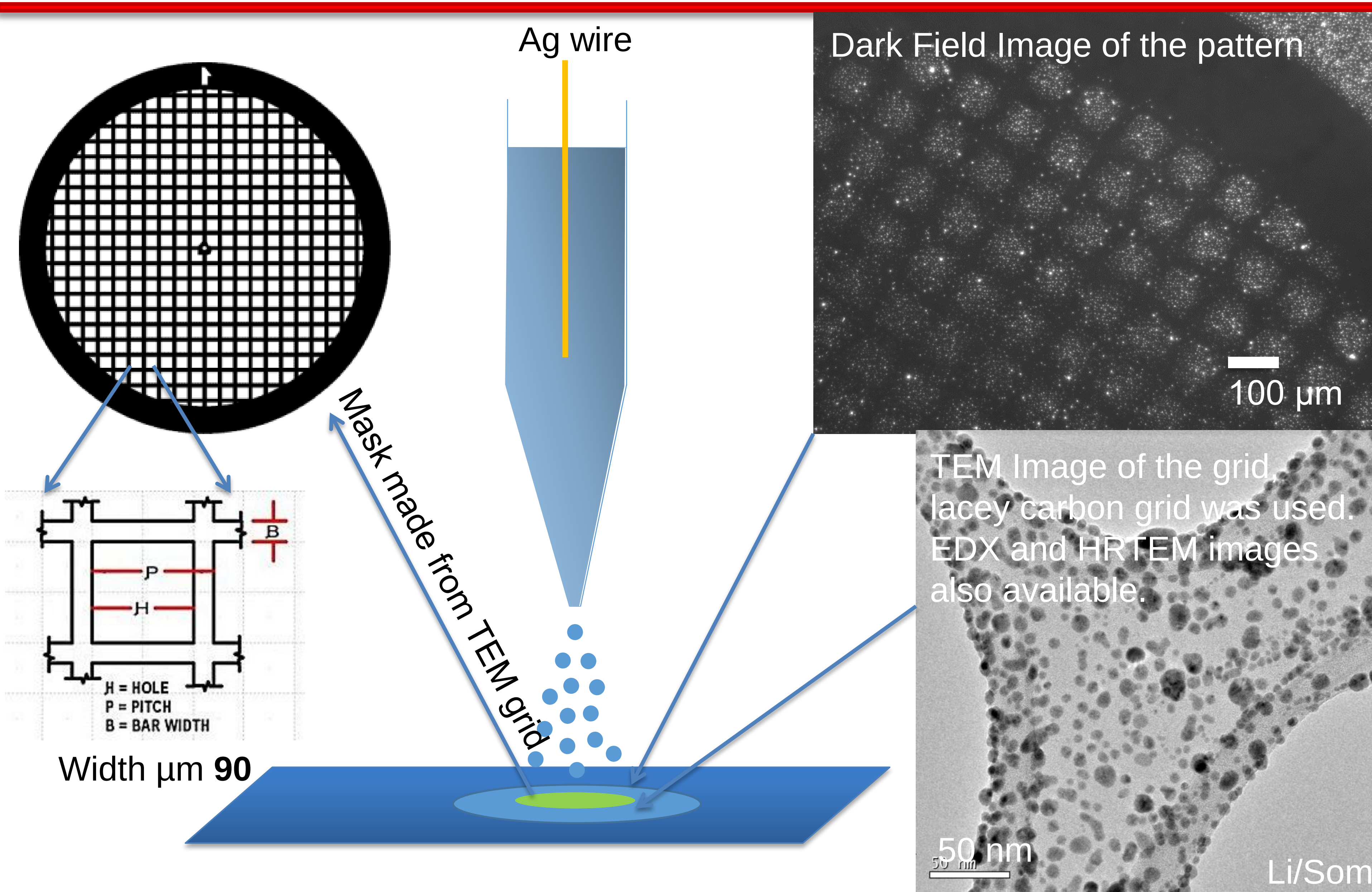


A. Li, Q. Luo, S.-J. Park and R. G. Cooks, Angew. Chem. Int. Ed., 53 (2014) 3147–3150.

Electrolytic spray for patterning



Patterning of nanoparticle regions

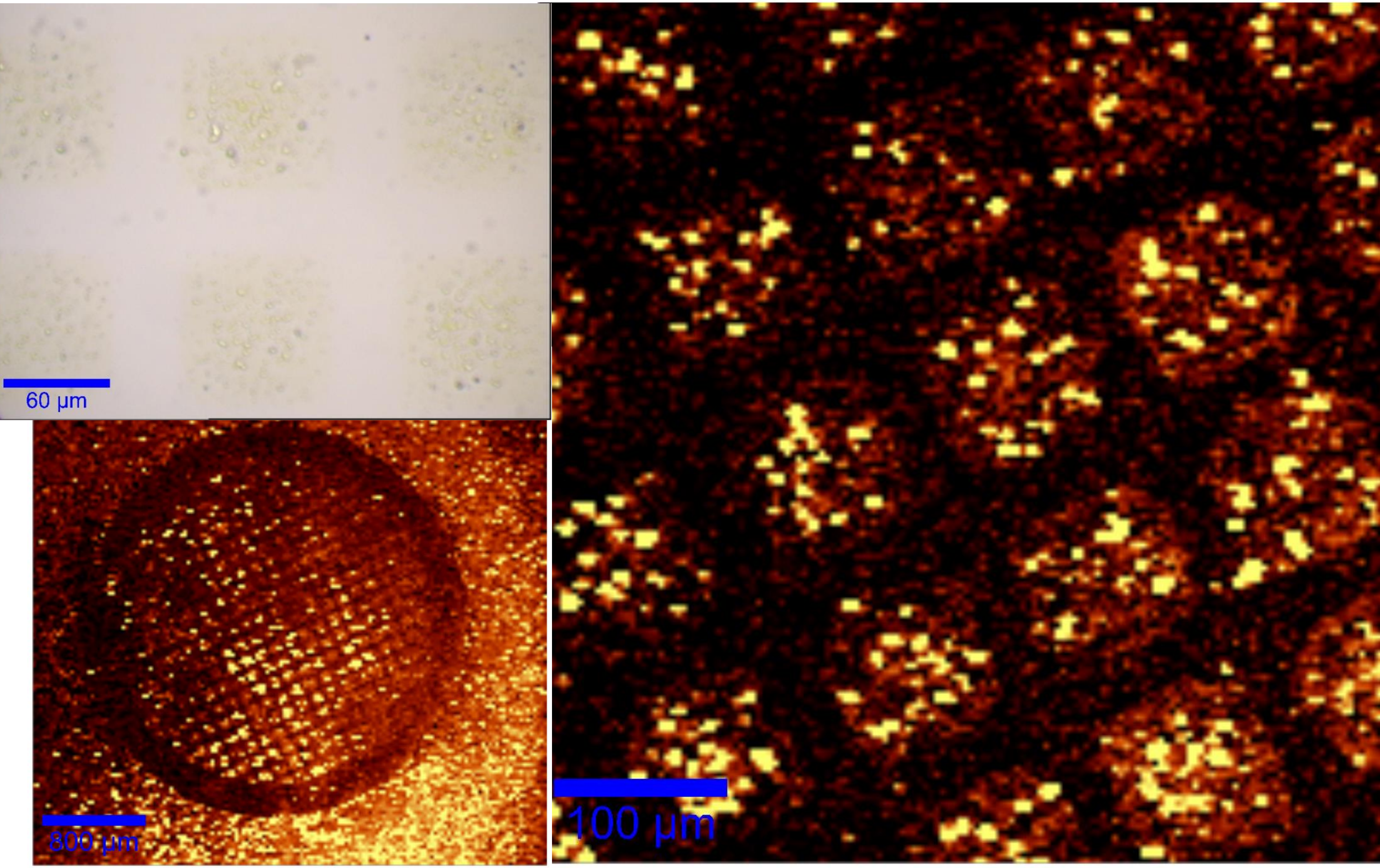


Width µm **90**

Dark Field Image of the pattern

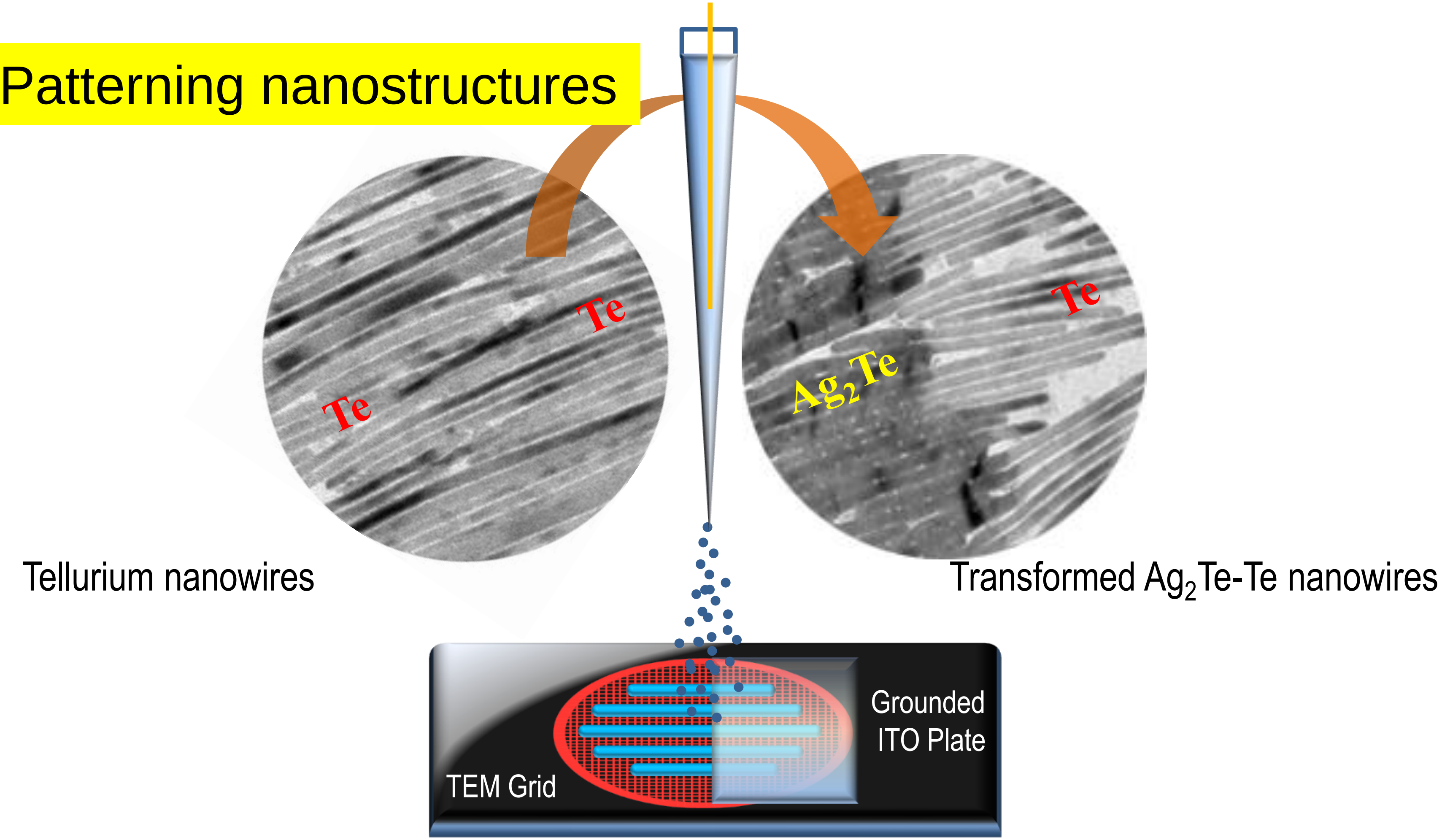
TEM Image of the grid, lacey carbon grid was used. EDX and HRTEM images also available.

Li/Som

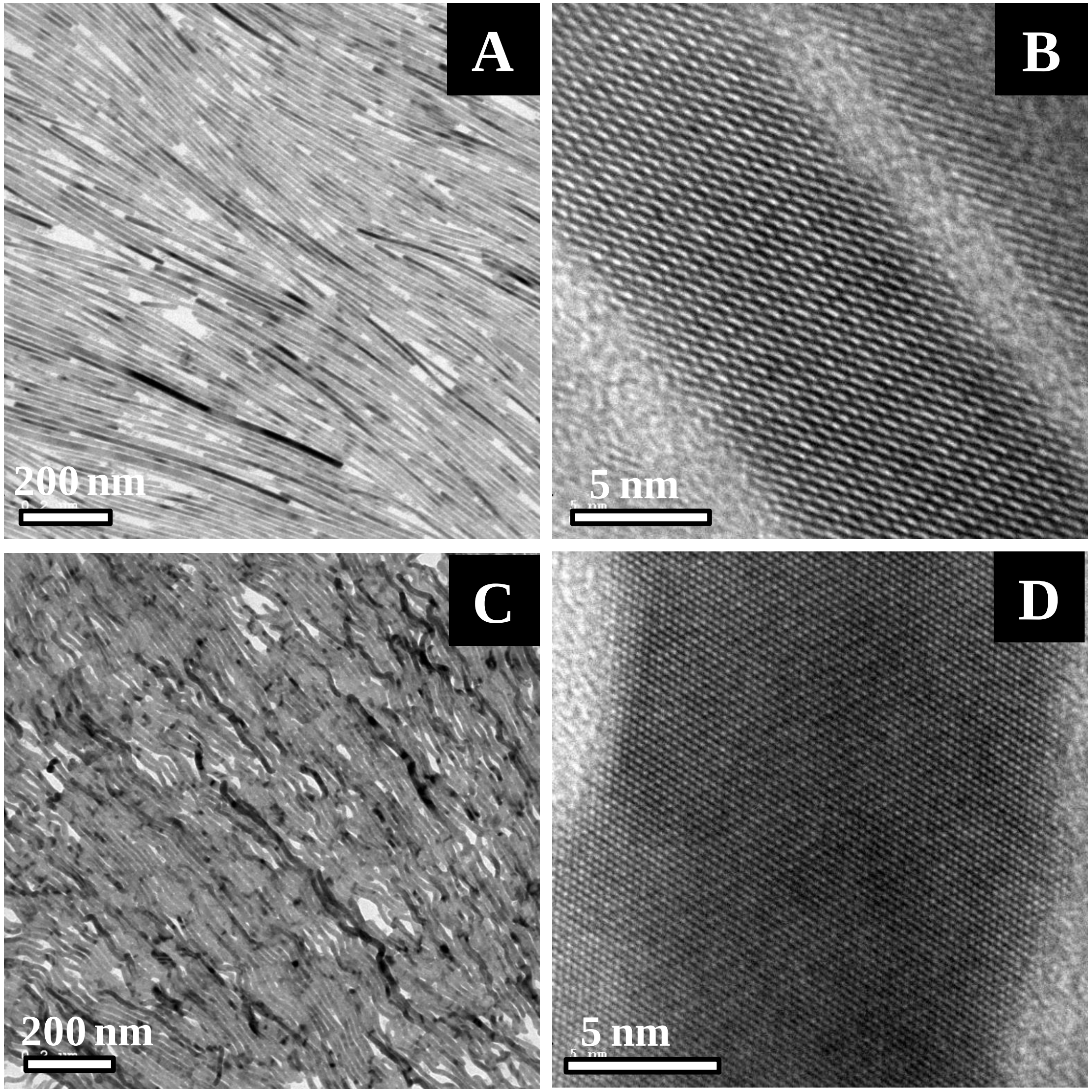


A. Li, Z. Baird, S. Bag, D. Sarkar, A. Prabhath, T. Pradeep and R. G. Cooks,
B. Angew. Chem. Int. Ed., (2014) (DOI: 10.1002/anie.201406660).

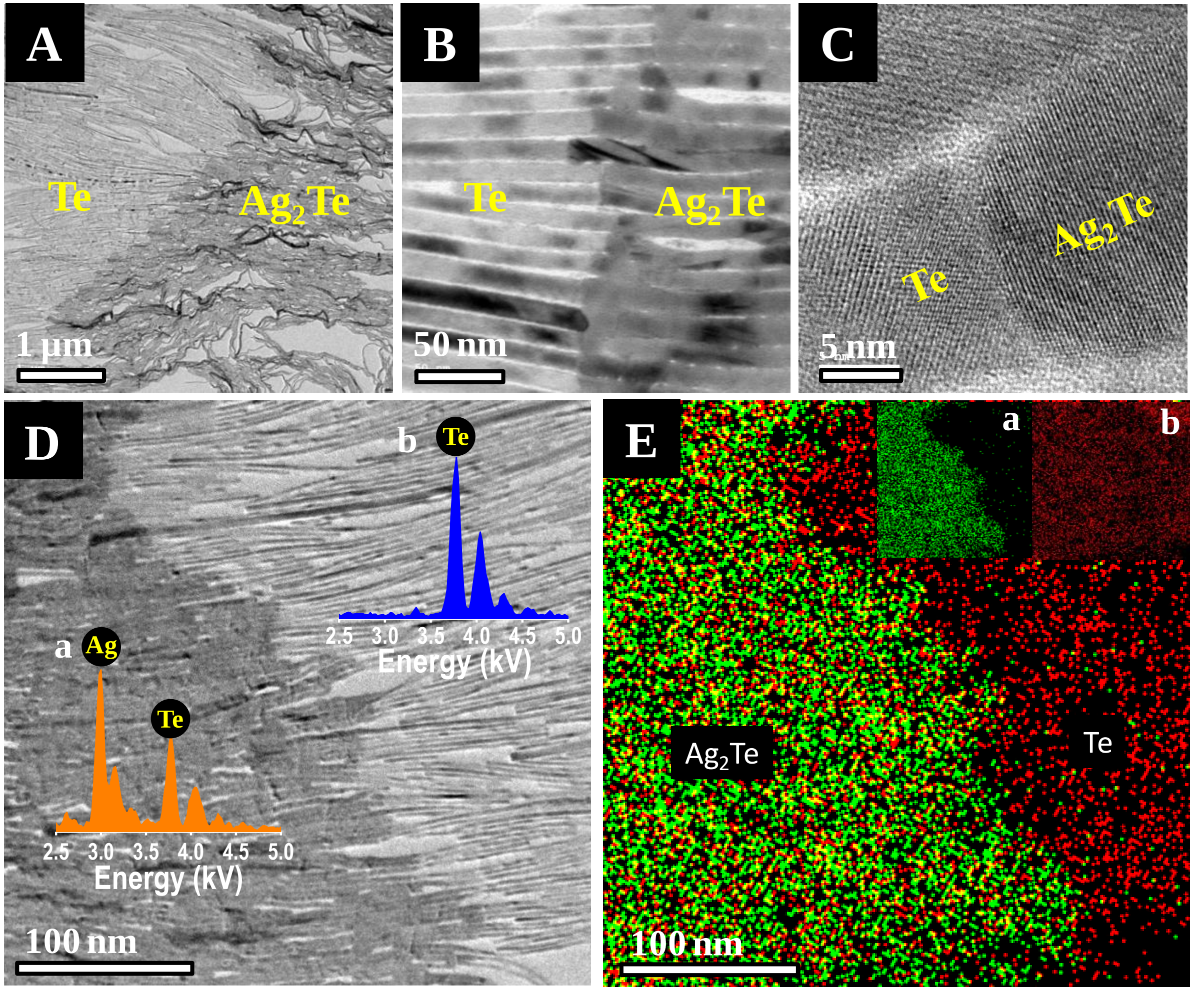
Patterning nanostructures



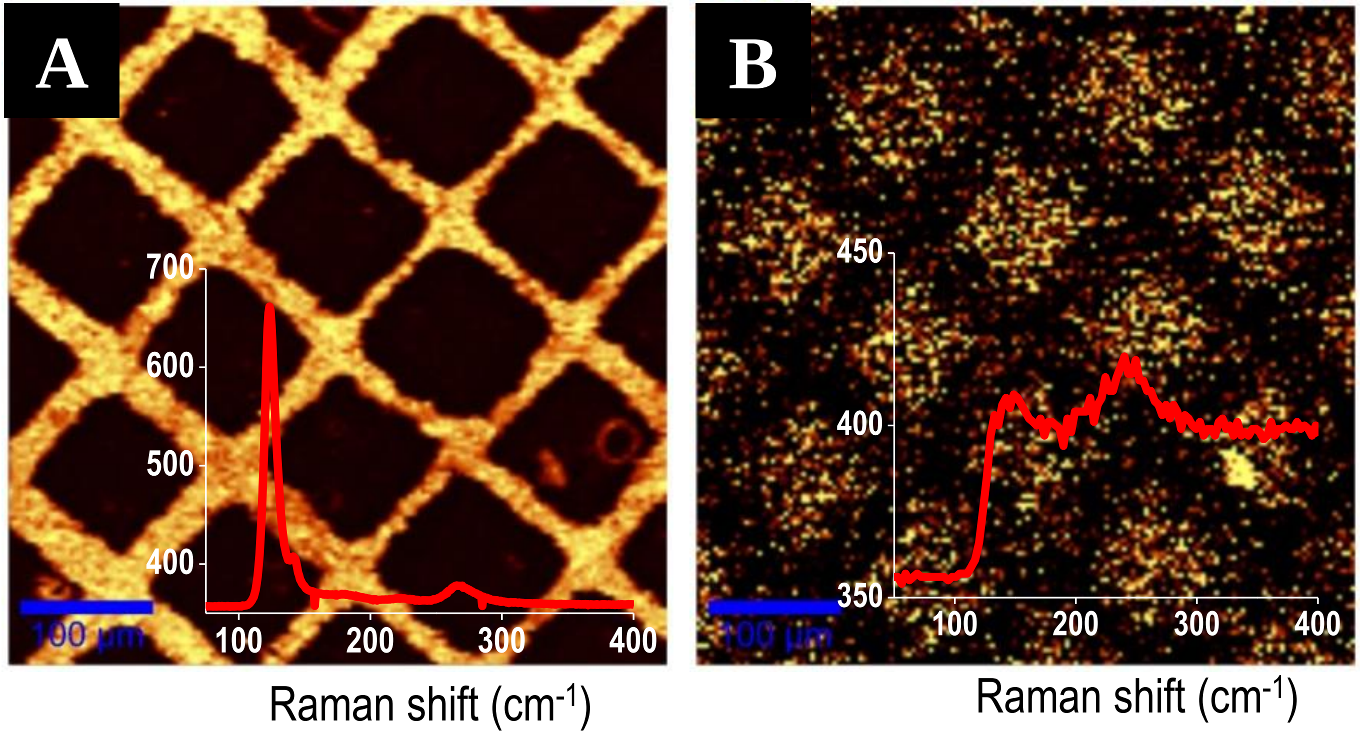
Schematic representation of the transformation of Te nanowires into Ag$_2$Te-Te nanowires through electrolytic deposition. TEM images show aligned Te nanowires before and after transformation.



A) Large area TEM image of aligned Te nanowires, B) HRTEM image of a single Te nanowire, C) large area TEM image of Ag_2Te nanowires obtained from aligned Te nanowires by electrolytic deposition under ambient conditions and D) HRTEM image of a single Ag_2Te nanowire.



A) Large area TEM image showing localized transformation, B) Magnified view of the nanowires showing the presence of Te and Ag_2Te phases in each of the nanowires, C) HRTEM image of such a nanowire (a sharp interface between the two crystalline phases is seen), D) TEM image of the boundary area selected for EDS mapping, insets a and b are the EDS spectra taken from the respective areas indicating elemental composition and E) Combined EDS map of Ag and Te with insets a and b showing the Ag and Te maps alone.



A) Raman image of the patterned surface selecting Te features, B) Raman image of the patterned surface selecting Ag_2Te features. Patterning of the surface was done using an empty TEM grid. Corresponding Raman spectra of Te and Ag_2Te regions are shown as insets.

Honey, I shrunk the mass spectrometer

IIT Madras team was able to create ions from any sample even at one volt

R. PRASAD

INNOVATIVE: *Rahul Narayanan of the Department of Chemistry, IIT Madras and the lead author of the paper performs an experiment using nanotube-coated paper.* – PHOTO: SPECIAL ARRANGEMENT

Mass spectrometers that are as small as a smart phone and require as little as one volt — a 3,000-time reduction in potential — to create an electric field which would turn a sample into ions for identification of composition may soon become a reality.

The feat of shrinking the ion source that requires very little voltage was achieved by a team led by Prof. T. Pradeep of the Department of Chemistry, IIT Madras. The results were published last week in the *Angewandte Chemie International* journal.

Conventionally, a solution of the sample is electrosprayed at 3,000 volts to create charged droplets that become ions. The ions are, in turn, analysed to find the composition or chemical constituents in the case of a sample mixture.

The massive reduction in voltage requirement became possible by using carbon nanotube-impregnated paper to act as a substrate on which the sample was deposited. If the conventional method uses very high voltage to create a strong electric field, the sharp protrusions of the carbon nanotubes help in creating the high electric field by using very low voltage.

"One volt over a few nanometres creates an electric field equivalent to 10 million volts over a centimetre," Prof. Pradeep explained. "The whole idea was to keep the nanotubes separated from each other. Normally they get bundled."

Once nanotubes get bundled, they turn out to become large wire-like structures thereby increasing the voltage required to create an electric field. "Earlier experiments [by others] using carbon nanotubes failed as the nanotubes were bundled," he said. In fact, standard procedures are available to disperse the nanotubes.

Incidentally, the order in which Prof. Pradeep's experiment progressed was unusual. "I had been after this method for a long time. I knew ionisation is possible and can be detected using low voltage. But the answer came first," he recalled. "I understood that by using the nanotube dispersion technique I could get ions. So the ions came first, and I looked at why this happened." And he soon figured it out. "I realised that ions were observed as the nanotubes were separated," he said.

"All good science is commonsense," he noted. "When you look back, [the way] many science breakthroughs [happen] look simple... quite silly. But if you had told this [miniaturizing mass spectrometer] 20 years ago, people would not have believed you."

A few puzzles remain to be solved. The researchers are yet to decipher where the samples get charged — along the entire length of the nanotube or just at the tip. It is also not clear why molecules present in the air don't get ionised and create their own signals (technically called as noise).

Earlier, scientists succeeded in shrinking the size of the analyser and detectors to 1 cm^2 each. Now, by shrinking the size of the ion source, the possibility of simplifying mass spectrometry for analysing various substances opens up.

"If you have a good vacuum system and controlled electronics, we can shrink a mass spectrometer to smart-phone size... we can simplify it. That's the importance of this discovery," he emphasised.

He foresees a day not too far away when gently rubbing the nanotube-coated paper on any object — an apple or a tablet — will be sufficient to collect samples for analysis in a lab. The nanotube-coated substrate can also be reused. In all, there is a real possibility of completely rewriting the way sample testing gets done.

"So what it means [is that] you can collect samples remotely and analyse them elsewhere for disease or pollution prevention or any such thing," he noted. "In a sense, we can make a mass spectrometer reach a wider audience." The mass spectrometer is a sophisticated instrument and has been out of bounds to the common man.

Producing a nanotube-coated substrate is also quite simple. Nanotubes can be grown separately and then coated on the substrate and, behold, it is ready for sample loading.

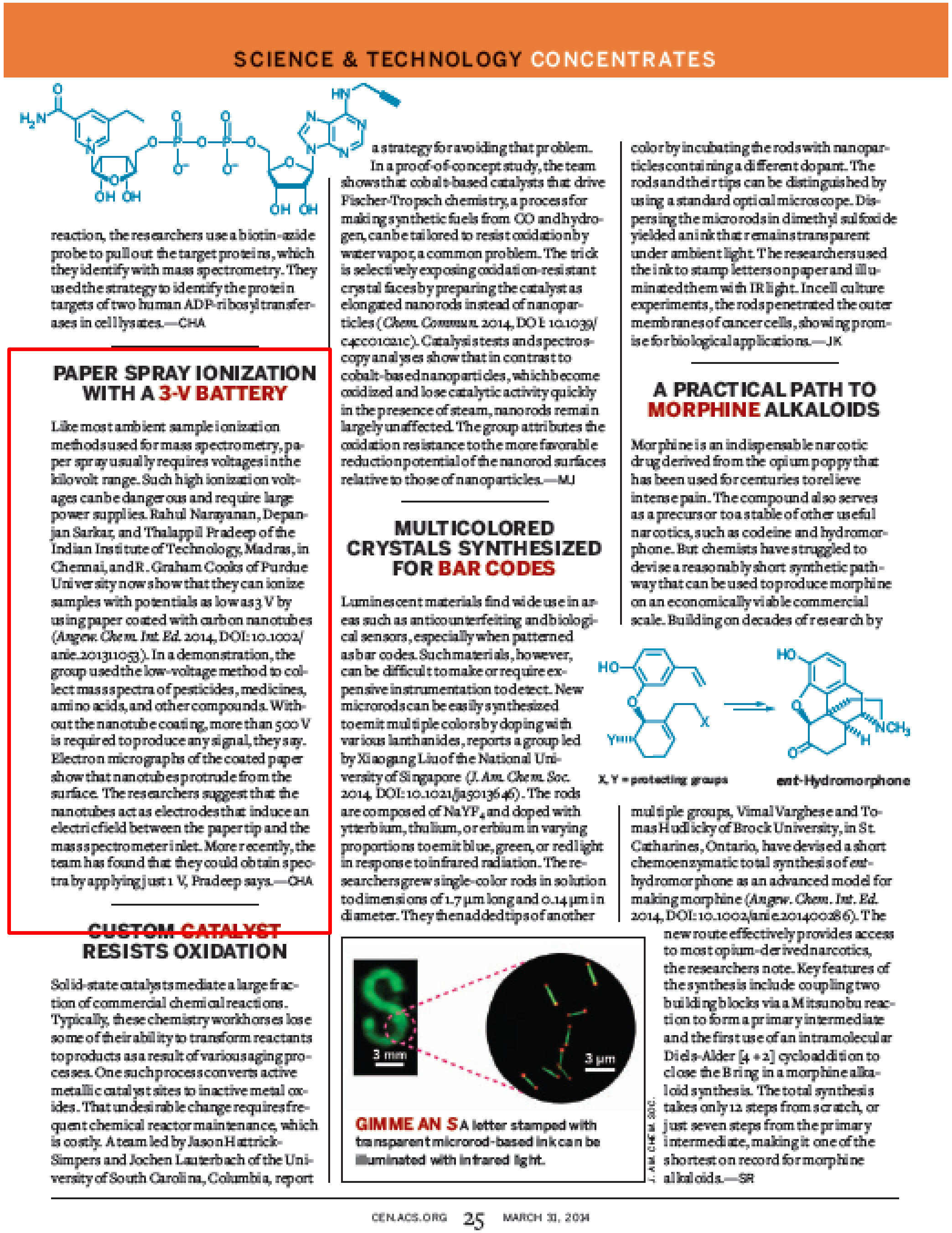
Since samples can be collected by gently rubbing the substrate on the material, there is a possibility of some tubes breaking and sticking to the surface of the material tested. Will such broken nanotubes cause any health hazard?

"We must ensure that the substrate is holding the nanotubes firmly, so no nanotubes stick to the sample tested," he noted.

The Hindu
March 27, 2014

Also featured in several newspapers and websites. Such as The Engineer, PACE, Science Daily, iConnect, Purdue News, etc.

http://www.thehindu.com/sci-tech/science/honey-i-shrunk-the-mass-spectrometer/article5835840.ece



reaction, the researchers use a biotin-azide probe to pull out the target proteins, which they identify with mass spectrometry. They used the strategy to identify the protein targets of two human ADP-ribosyltransferases in cell lysates.—CHA

PAPER SPRAY IONIZATION WITH A 3-V BATTERY

Like most ambient sample ionization methods used for mass spectrometry, paper spray usually requires voltages in the kilovolt range. Such high ionization voltages can be dangerous and require large power supplies. Rahul Narayanan, Depanjan Sarkar, and Thalappil Pradeep of the Indian Institute of Technology, Madras, in Chennai, and R. Graham Cooks of Purdue University now show that they can ionize samples with potentials as low as 3 V by using paper coated with carbon nanotubes (*Angew. Chem. Int. Ed.* 2014, DOI: 10.1002/anie.201311053). In a demonstration, the group used the low-voltage method to collect mass spectra of pesticides, medicines, amino acids, and other compounds. Without the nanotube coating, more than 500 V is required to produce any signal, they say. Electron micrographs of the coated paper show that nanotubes protrude from the surface. The researchers suggest that the nanotubes act as electrodes that induce an electric field between the paper tip and the mass spectrometer inlet. More recently, the team has found that they could obtain spectra by applying just 1 V, Pradeep says.—CHA

CUSTOM CATALYST RESISTS OXIDATION

Solid-state catalysts mediate a large fraction of commercial chemical reactions. Typically, these chemistry workhorses lose some of their ability to transform reactants to products as a result of various aging processes. One such process converts active metallic catalyst sites to inactive metal oxides. That undesirable change requires frequent chemical reactor maintenance, which is costly. A team led by Jason Hattrick-Simpers and Jochen Lauterbach of the University of South Carolina, Columbia, report a strategy for avoiding that problem.

In a proof-of-concept study, the team shows that cobalt-based catalysts that drive Fischer-Tropsch chemistry, a process for making synthetic fuels from CO and hydrogen, can be tailored to resist oxidation by water vapor, a common problem. The trick is selectively exposing oxidation-resistant crystal faces by preparing the catalyst as elongated nanorods instead of nanoparticles (*Chem. Commun.* 2014, DOI: 10.1039/c4cc01021c). Catalysis tests and spectroscopy analyses show that in contrast to cobalt-based nanoparticles, which become oxidized and lose catalytic activity quickly in the presence of steam, nanorods remain largely unaffected. The group attributes the oxidation resistance to the more favorable reduction potential of the nanorod surfaces relative to those of nanoparticles.—MJ

MULTICOLORED CRYSTALS SYNTHESIZED FOR BAR CODES

Luminescent materials find wide use in areas such as anticounterfeiting and biological sensors, especially when patterned as bar codes. Such materials, however, can be difficult to make or require expensive instrumentation to detect. New microrods can be easily synthesized to emit multiple colors by doping with various lanthanides, reports a group led by Xiaogang Liu of the National University of Singapore (*J. Am. Chem. Soc.* 2014, DOI: 10.1021/ja5013646). The rods are composed of $NaYF_4$ and doped with ytterbium, thulium, or erbium in varying proportions to emit blue, green, or red light in response to infrared radiation. The researchers grew single-color rods in solution to dimensions of 1.7 μm long and 0.14 μm in diameter. They then added tips of another color by incubating the rods with nanoparticles containing a different dopant. The rods and their tips can be distinguished by using a standard optical microscope. Dispersing the microrods in dimethyl sulfoxide yielded an ink that remains transparent under ambient light. The researchers used the ink to stamp letters on paper and illuminated them with IR light. In cell culture experiments, the rods penetrated the outer membranes of cancer cells, showing promise for biological applications.—JK

GIMME AN S A letter stamped with transparent microrod-based ink can be illuminated with infrared light.

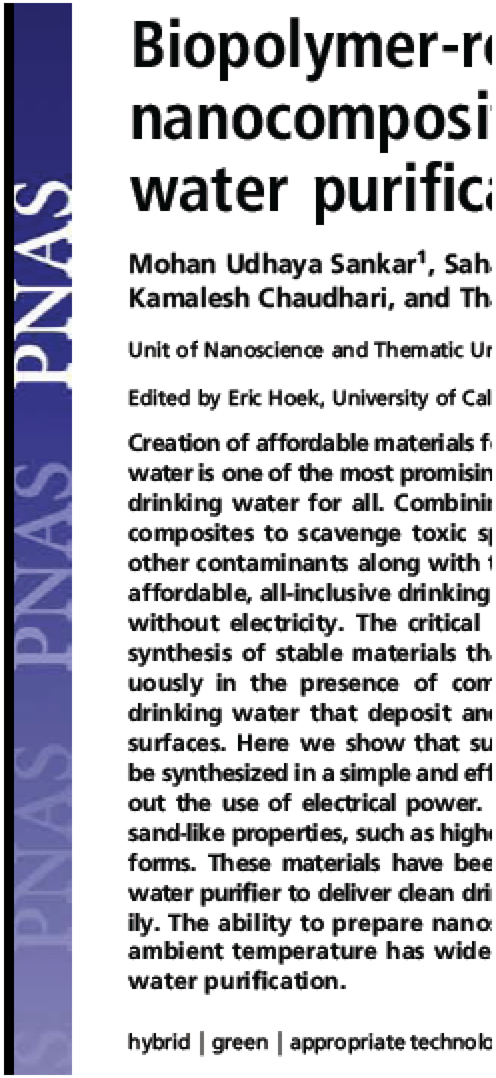
A PRACTICAL PATH TO MORPHINE ALKALOIDS

Morphine is an indispensable narcotic drug derived from the opium poppy that has been used for centuries to relieve intense pain. The compound also serves as a precursor to a stable of other useful narcotics, such as codeine and hydromorphone. But chemists have struggled to devise a reasonably short synthetic pathway that can be used to produce morphine on an economically viable commercial scale. Building on decades of research by multiple groups, Vimal Varghese and Tomas Hudlicky of Brock University, in St. Catharines, Ontario, have devised a short chemoenzymatic total synthesis of *ent*-hydromorphone as an advanced model for making morphine (*Angew. Chem. Int. Ed.* 2014, DOI: 10.1002/anie.201400286). The new route effectively provides access to most opium-derived narcotics, the researchers note. Key features of the synthesis include coupling two building blocks via a Mitsunobu reaction to form a primary intermediate and the first use of an intramolecular Diels-Alder [4 + 2] cycloaddition to close the B ring in a morphine alkaloid synthesis. The total synthesis takes only 12 steps from scratch, or just seven steps from the primary intermediate, making it one of the shortest on record for morphine alkaloids.—SR

X, Y = protecting groups

ent-Hydromorphone

C&E News
March 30, 2014



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Edited by Eric Hoek, University of Cal

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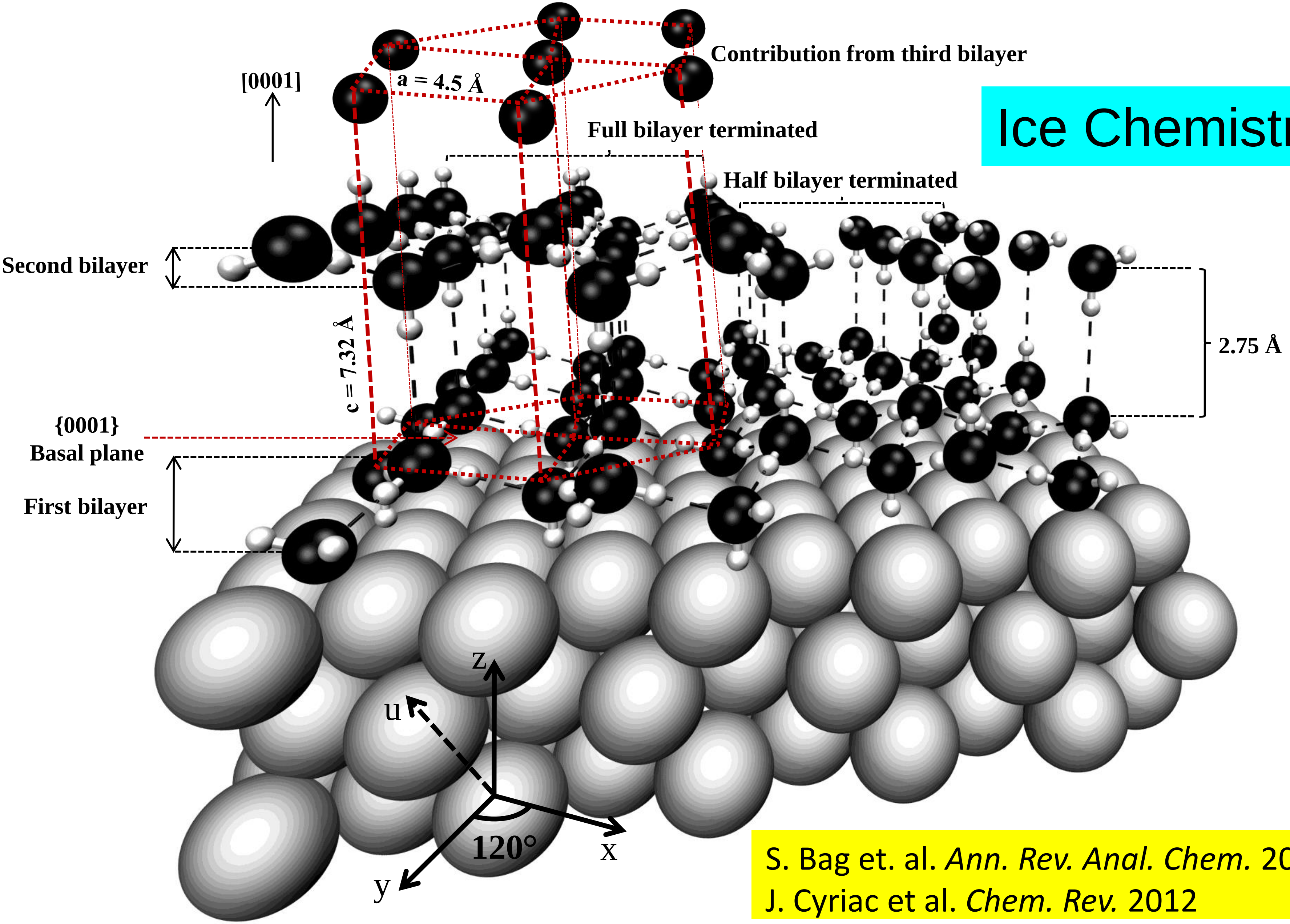
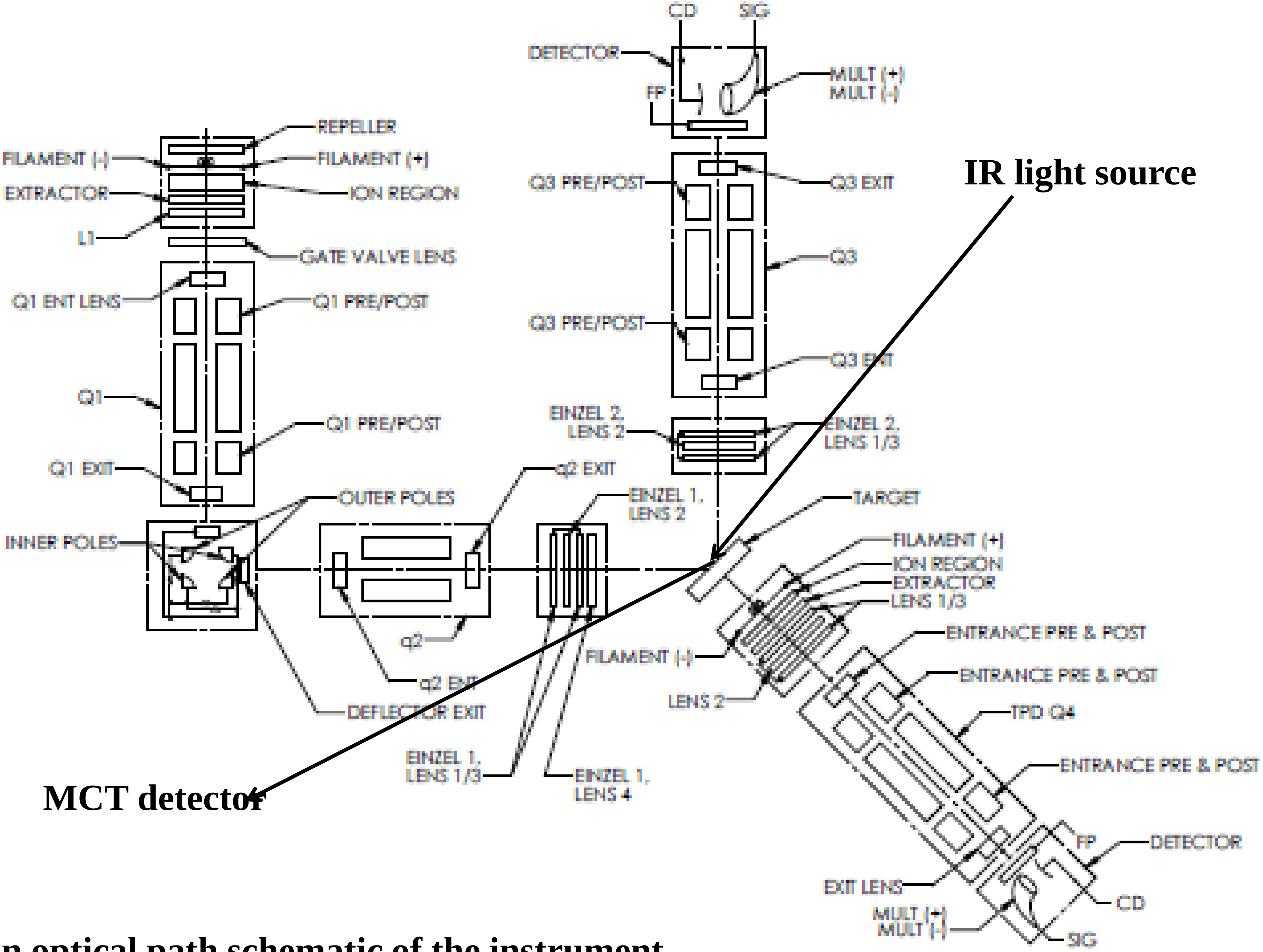
Featured in:
The Guardian, UK
The Hindu, Telegraph, Times of India, etc.
Scientific American
New Scientist
and many others

Ice Chemistry



S. Bag et. al. *Ann. Rev. Anal. Chem.* 2013
J. Cyriac et al. *Chem. Rev.* 2012

A perspective view of Ih ice growth on a Ru(0001) substrate. The grey balls indicate the Ru atoms, the black balls represent the oxygen atoms and the white balls stands for the hydrogen atoms. Hydrogen bonds in the ice structure are shown by black dotted line in between the water molecules. The epitaxial growth of ice is shown.



Ion optical path schematic of the instrument

Soumabha Bag et al. Rev. Sci. Instrum. 2014

Anyin Li, R. G. Cooks

सत्यमेव जयते

Government of India

Nano Mission

Department of Science & Technology