

# **An Integrated Wipe Sample Transport/Autosampler to Maximize Throughput for a DART<sup>®</sup>/oa-TOFMS**

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## **Abstract**

A wipe sample transport was designed and built to meet two objectives: to simplify collection, storage, and transport of cotton swab wipe samples and to provide a sample train of 72 wipe samples nearly ready for analysis when the swabs reach the laboratory. The cotton swabs are mounted on an aluminum (Al) rod that is the sample support for an autosampler used to perform Direct Analysis in Real Time (DART<sup>®</sup>)/orthogonal acceleration, Time-of-Flight Mass Spectrometry (oa-TOFMS) analyses. The goal is for one analyst to analyze 1000 wipe samples mounted on 14 Al rods in one 8-hr shift.

## **Introduction**

Hundreds of analyses by a rapid, simple, and rugged analytical technique will be necessary to characterize contaminated sites after accidental, deliberate, or weather-related dispersive events. In addition, identification of the contaminants will be required to assess the health risk posed by each site. Direct Analysis in Real Time (DART) [Cody, et al. 2005a, 2005b, Fernandez, et al. 2006, Grange, 2007, Jones, et al. 2006, Laramée, and Cody, 2007, AccuTOF] and Desorption Electrospray Ionization (DESI) [Chen, et al. 2005, Cotte-Rodríguez, et al. 2005, Fernandez, et al. 2006, Kauppila, et al. 2006, Rodriguez-Cruz, 2006, Takáts, et al. 2004, 2005, Williams and Scrivens, 2005, Direct] ion sources produce ions from analytes on surfaces in open air, which then enter a mass spectrometer for analysis. When samples are analyzed without prior extraction, extract clean-up, or chromatography, and when evacuation of the ion source after a sample is introduced is not required, very rapid mass analyses and high sample throughputs are possible.

Although direct analyses of contaminated surfaces might be preferred, cotton swab wipe samples eliminate the need to chip pieces of concrete, asphalt, plaster, or other materials from surfaces for analysis. Mounting odd-shaped chips into a sample holder would be slow, difficult, and the orientation of the contaminated surface relative to the ionizing beam would greatly affect the ion abundances observed. Recently, an inexpensive, variable-speed autosampler was built to

acquire mass spectra for individual cotton swabs in 6 s using a DART/orthogonal acceleration, Time-of-Flight Mass Spectrometer (oa-TOFMS) [Grange, 2007]. A 91-cm (3-foot)-long, 0.64-cm (1/4")-square, aluminum rod with holes drilled to hold cotton swab heads was mounted on two N-scale model railroad flat cars and pulled along N-scale railroad track through the ion source by a 7-rpm motor. Each mounted sample was exposed to the ionizing beam sequentially.

For cotton swab wipe samples, the time required by the analyst to open packaging around each swab, insert the swabs through the Al rod, trim most of the 15 cm (6") stick from the swabs, and record sample labels and corresponding rod locations in a log book would exceed the time required for data acquisition by at least a factor of 10. This paper describes an inexpensive wipe sample transport with the Al rod, sample support from the autosampler at its core to reduce the sample manipulation time in the laboratory to a value similar to the data acquisition time and to provide a convenient means for field samplers to collect and transport cotton swab wipe samples.

### Design, Dimensions, and Fabrication

The two goals of the wipe sample transport design were to provide the analyst with a long sample train of wipe samples ready for analysis and to provide a rapid, simple, and convenient means for field samplers to collect the samples. To accomplish both goals, cotton swabs were substituted for the squares of cotton cloth, gauze, or other materials often used to collect wipe samples (ASTM; Billets, 2007; CFR, 2004; Frame; 1999, Opstad, et al, 1999) and the Al rod sample support was made the core component of the wipe sample transport. Drawings of portions of the transport are shown in Figure 1 from three orthogonal angles. The cotton swabs were easily mounted through the Al rod, whereas pads could not be easily affixed to the rod. Opstad, *et al.* reported that Q-tips<sup>®</sup> provided higher recoveries of chemical warfare agents than cotton cloths, felt, or filter paper (Opstad, et al. 1999). Cotton swabs atop 15-cm sticks provide a field sampler wearing gloves a longer handle than Q-tips for rolling a swab across a surface. Inexpensive and readily procured construction materials were employed to minimize cost, development time, and fabrication time for the wipe sample transport. Prototypes of four designs were made, with each later design becoming simpler to use, easier to fabricate, and more protective of the cotton swab heads. Only the final design will be discussed.

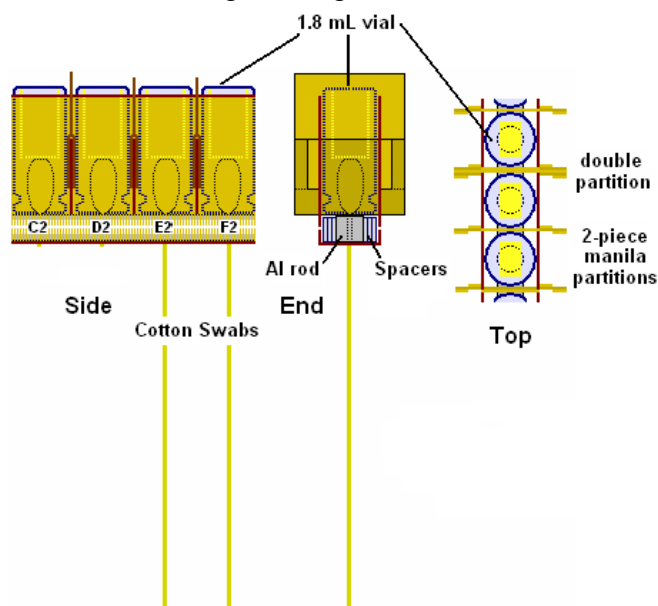


Figure 1. Orthogonal views of the Al rod, vials, and linear cell array used to transport protected cotton swabs and to acquire wipe samples.

**Swab Head Protection.** Through each Al rod (Small Parts, Inc., Miami Lakes, FL), 76 0.28 cm (7/64") holes were drilled at 1.2 cm (15/32") intervals. This interval was chosen to enable using 1.16 cm (0.455")-wide, 1.8 mL, wide-mouth, clear-glass vials (VWR, West Chester,

PA) to cover each swab head. Clean 15 cm-long cotton swabs (Puritan Medical Products, Guilford, Maine) are stored in the Al rod within the glass vials prior to a dispersive event. The glass vials protect the swabs from damage or contamination before and after each wipe sample is collected. The swabs taken directly from 100-swab packages provided no prominent background ions, and ions from the analytes on the swabs dominated the mass spectra. However, as a precaution, each new batch of purchased swabs or swabs that have been stored in vials for months should be checked for prominent background ions before they are used to collect wipe samples. The vials are held in place by a linear array of cells constructed from manila folders and 5.1 cm (2")-wide, clear packaging tape. Figure 2 shows portions of three templates that were

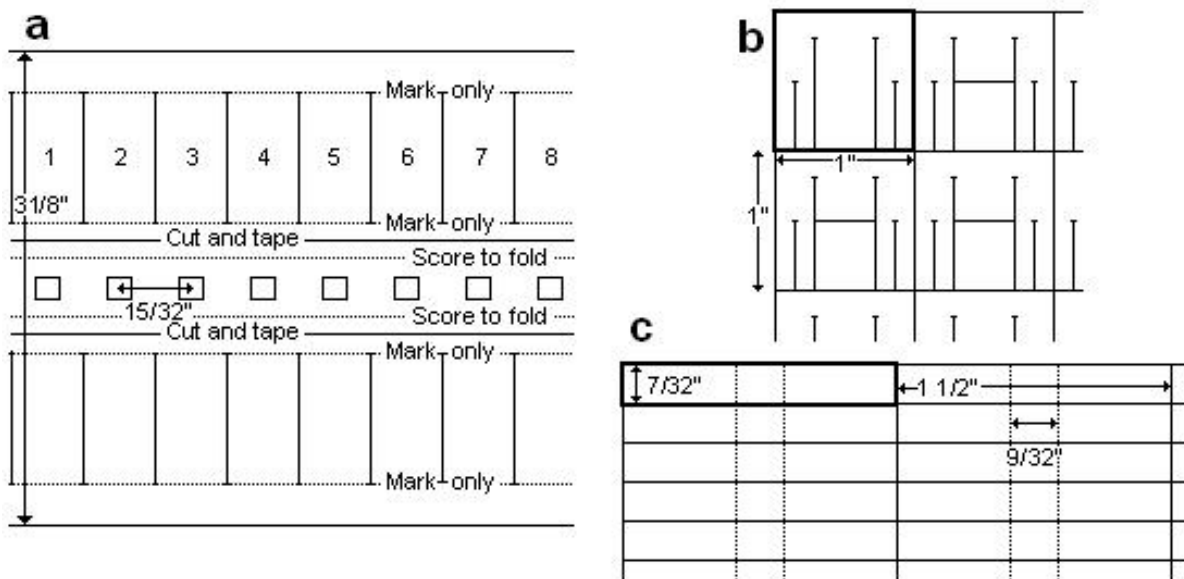
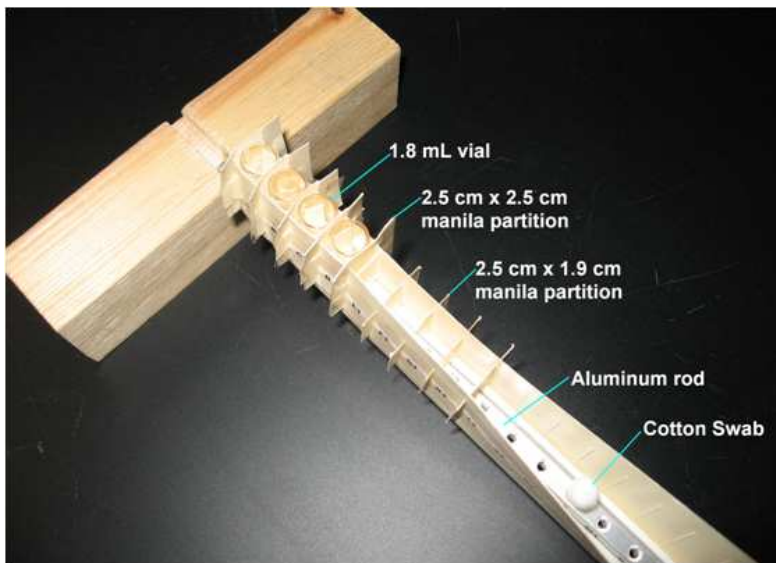


Figure 2. Portions of three templates available from the author used to cut parts of the linear cell array from manila folders: (a) for the outer walls, (b) for the partitions between vials, and (c) for the vial inserts to keep the swabs in place. Solid lines are cut to provide edges of parts or slits. Dotted lines mark a dimension that is scored and in some cases then folded. The bold lines delineate a single partition in (b) and a single insert in (c).

taped onto manila folders to make two of the three component parts of the cells and a U-shaped vial insert. The templates guided manual cutting with a hobby knife of slits and the small square holes through which the swab sticks pass, cutting of strips with a 30 cm (12") portable paper trimmer (Fiskars, Helsinki, Finland), and cutting of slits with a small pair of scissors. These templates are available from the author. The third part for the cell array was a 2.5 cm x 1.9 cm (1" x 3/4") rectangle that did not require a template. Three 0.08 cm x 30.5 cm x 0.5 cm (1/32" x 12" x 3/16") cardboard spacers cut from overhead transparency frames on each side of the Al rod kept the bottom of the cell assembly centered relative to the rod so that the sticks passed easily through the square holes. A second layer of manila folders under the first protected the desk from the hobby knife. Each cell assembly required about 6 hours to manufacture and assemble.

The assembly steps are illustrated in Figure 3. Wood blocks on each end of the Al rod prevent it from rotating during assembly of the cells. The cotton swab passing through a hole in the rod and the underlying hole in the U-shaped cell walls aligns the rod with the cell walls.

Eleven 2.5 cm x 1.9 cm rectangles have been pushed through the nine pairs of slits on each side of the U-shaped cell walls: two each through the first and fifth pairs starting from the end of the rod. A 2.5 cm x 2.5 cm (1" x 1") partition produced using the template in Figure 2b has been pushed downward over the first five rectangles to provide partitions nearly as high as the vials to hold them in place. One each of the two tabs of the 2.5 cm x 2.5 cm partitions outside each cell wall fits in front of and in back of the rectangle. The tabs are taped in place against the rectangle to provide a sturdy two- or three-piece partition between the vials. Construction time is minimized by inserting all of the smaller partitions first, followed by the larger ones.



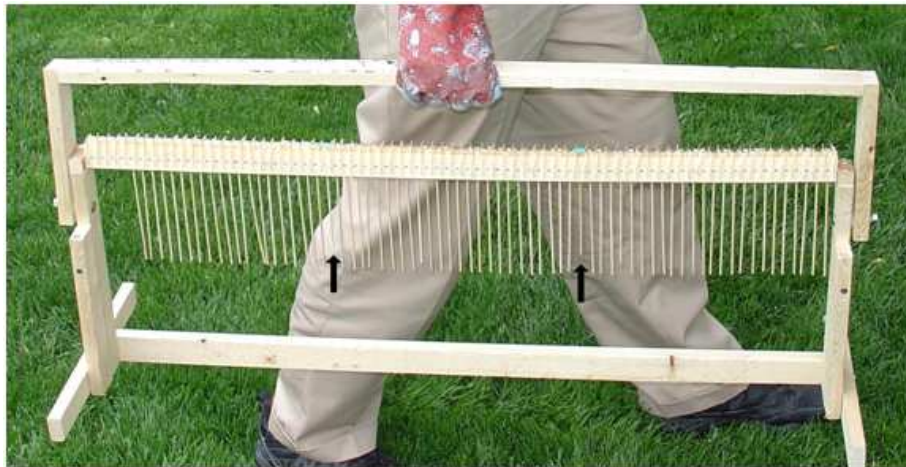
*Figure 3. Photo of a partially assembled Al rod-cotton swab-vial-cell assembly showing the two types of partitions between adjacent vials. The holes are countersunk to aid insertion of the swab sticks through the rod.*

**Sample Labeling.** Each cell position and the underlying rod position have the same label. If a grid pattern of samples were acquired outdoors or surfaces were sampled within a building, each field sampler would be provided a list of the sample labels and corresponding coordinates. The field sampler would not be required to write sample labels on plastic bags, on the list, or elsewhere. The analyst would not have to interpret illegible labels on the plastic bags, open the bags, insert each swab stick through the correct hole in the correct Al rod, and snip off the stick at the bottom of the rod. This time-consuming and error-prone sample preparation task would be eliminated.

This field sampling methodology places a premium on speed, rather than on exhaustive documentation. The rapidly prepared documents would include the field sampler's list of labels and coordinates, a table of ion abundances at each sampling location for each ion of interest, the elemental compositions of the ions of interest, and dissemination maps with four semi-quantitative levels of each analyte (non-detect, low, medium, or high). This information would be sufficient to characterize a contaminated site prior to any necessary remediation. Post-remediation sampling would document the thoroughness of the clean-up of the contaminated areas. The aim is to provide the essential data for disseminated chemicals of concern in many samples, rather than exhaustive characterization, precise quantitation, and documentation for all compounds present in a very limited number of samples.

**Sample Collection.** Figure 4 shows the wipe sample transport containing the Al rod, 74 vials containing swab heads, and the linear cell assembly. Several 15-cm swabs on the left have been clipped at the bottom of the Al rod to simulate prior collection of wipe samples. Clipping

the stick prevents reusing the swab for another wipe sample and prepares the swab for analysis in the laboratory. The rod contains 76 holes, but the first and last extend into the clamping blocks at each end of the rod. Swabs dipped into calibrant solutions will be placed into these two positions



*Figure 4. Photo of the portable wipe sample transport. The shortened sticks of two cotton swabs (arrows) indicate these are calibration standard positions. These two extra vials and swabs are provided in case one or two sample vials or swabs are damaged in the field.*

to these two positions prior to analysis. The structure was made from nominal 1 x 2 (1.9 cm x 3.8 cm x 244 cm) [3/4" x 1.5" x 8"] pine sticks, assorted wood screws, and two double nipped bolts to affix the rotatable handle. A field sampler would carry the support to a pair of coordinates, rotate the handle downward, push upward on the bottom of the cotton swab stick located in the position corresponding to the grid coordinates until the vial was free of its surrounding cell, remove the vial and store it in one of two holes provided in the wipe sample transport, collect the wipe sample, place the vial back over the cotton swab head, insert the stick through the hole in the Al rod, push the vial back down into its cell, and finally, clip off the stick close to the bottom of the Al rod. The 15 cm-long swabs provide convenient sample collection when wearing protective gear including bulky gloves such as the pair visible in Figure 4. Pre-labeled cell and rod locations will minimize a field sampler's exposure to chemicals at each collection site by eliminating the need to affix or write labels. Removing and replacing a 1.8 mL vial is quicker and easier than unwrapping a swab and later inserting it into a plastic bag.

**Recoil.** The diameters of the swab sticks vary from 0.23 cm to 0.25 cm (0.092" to 0.099"). When sticks are clipped with a wire cutter just below the Al rod, the sticks become oblong at the clip site, and larger diameter sticks no longer pass easily through the hole in the Al rod. This oblongation prevents the heads from moving upward due to the recoil associated with clipping. After analysis, less than 20% of the heads (those attached to the narrower sticks) fall out of the rod when the rod is turned upside down and shaken. During clipping of these narrower sticks, recoil propels the cotton heads upward. A 0.56 cm (7/32")-wide U-shaped vial insert made from a manila folder within the vial prevents the swab head from moving upward within the vial until it would exit the Al rod. If this occurred, later removal of the vial would also remove the swab head within the vial, after which the swab might fall out of the vial onto the lab bench or the floor. This impact could lose analyte from the swabs, contaminate the swabs, create sample position uncertainties or errors if several or more swab heads fell, and consume time as the swab heads were reinserted into the Al rod.

A second problem with clipping recoil is that powder loosely attached to the cotton can be dislodged from cotton swabs by the jolt as the wire cutter truncates the stick. Wetting of the swab immediately prior to sampling with an appropriate solvent will dissolve analytes, which will adhere better to the cotton fibers after the solvent dries. Wetting swabs also increases the amount of analytes picked up by the swabs (Frame and Abelquist, 1999, Billets, 2007). Finally, when wet swabs are rolled on a surface, they become more compact and matted, which greatly reduces the likelihood of the He stream blowing cotton tufts loosened from the swabs into the cone orifice.

**Hole Configuration.** How the holes are labeled depends on the experiment. The labels provided by a laser printer on copier paper are affixed to the rod and surrounding cells with transparent tape. First responders will acquire numerous wipe samples to characterize the direction and distance of chemical dissemination. They will also seek to identify the dispersed chemicals based on the exact masses and relative isotopic abundances of ions provided by the oa-TOFMS (Grange, 2006). After any required remediation, a much larger set of samples will be collected to thoroughly document the completeness of the clean-up. The hole configuration for our first experiment will correspond to a 25 x 40 grid for a total of 1000 samples requiring 14 Al rods. During analyses, the 1<sup>st</sup>, 26<sup>th</sup>, 51<sup>st</sup>, and 76<sup>th</sup> holes through each rod will contain cotton swabs dipped into a polyethylene glycol solution or other external standards for mass calibration. In the field, however, spare vials and 2-3 cm shorter cotton swabs are provided at positions 26 and 51 as shown in Figure 4. Colored tape on the bottom of the two vials reminds the field sampler that these are positions for external standards and that no wipe sample is required at these two Al rod locations. This arrangement provides two spare swabs in case cotton swab sticks break during sampling.

The DART/oa-TOFMS might eventually be placed in a van for transport to sites near dispersive events. Use of four swabs for the calibrant ensures that the time difference between acquisition of each analyte swab mass spectrum and the calibration mass spectrum never exceeds 1.3 min when the Al rod is pulled through the ion source at 0.20 cm/s (Grange, 2007). Calibration drift should not be significant on this time scale, even on partly cloudy days when the temperature within the van could vary as the sun intermittently warms outer van surfaces or is covered by a cloud. Fourteen Al rod and cell assemblies have been prepared. To test the speed, simplicity, and ruggedness of the wipe sample transport/autosampler, the swabs will be dipped into solutions of analytes at three different concentrations to simulate field collection and four-color dissemination maps will be prepared from the results. The goal is for one analyst to analyze the 1000 swabs in one 8-hr shift.

**Pre-analysis Sample Preparation.** After a field collector has collected all 72 wipe samples, the Al rod, vials, and cell assembly will be removed from its field support, wrapped in bubble wrap, and sent to the laboratory. Alternatively, the assemblies would be handed to an analyst within an on-site van. The vials and cells must be removed prior to mounting the Al rod and swab heads onto the N-scale flat cars of the autosampler used to transport the swabs through the ion source. To facilitate doing so within 5 min, slits on both sides of the cell assembly 0.3 cm (1/8") below the cell partitions were pre-cut and taped. As illustrated in Figure 5a, the tape

spanning these slits is easily cut using a hobby knife after samples are collected. The bottom of the cell assembly falls away and the cells and vials are then lifted directly upward as shown in Figure 5b. The wide-mouth vials ensure the swab heads remain in the Al rod, when the swab heads are first wetted with a solvent and rolled across the sampled surface. The size and shape of fresh cotton swabs heads are variable with diameters between 0.6 - 0.7 cm (1/4

- 9/32"). Because the diameter of the wide-mouth of the vials is 0.62 cm (0.244"), a few dry swab heads can be lifted by the vial mouths. About 5% of the dry swabs offer substantial resistance when pushed into a vial and are not used. The permanent compression observed for a rolled, wet swab decreases the swab diameters to less than 0.62 cm. After removing the cell assembly and vials, the rod and swabs are placed onto the flat cars, calibrant swab heads are placed into positions 1, 26, 51, and 76, and the rod is run through the He ionizing beam for mass analysis. The bottom of the cell assembly can be re-taped to its top to prepare it for sample collection with another rod, fresh set of swabs, and washed vials for a later dispersive event.

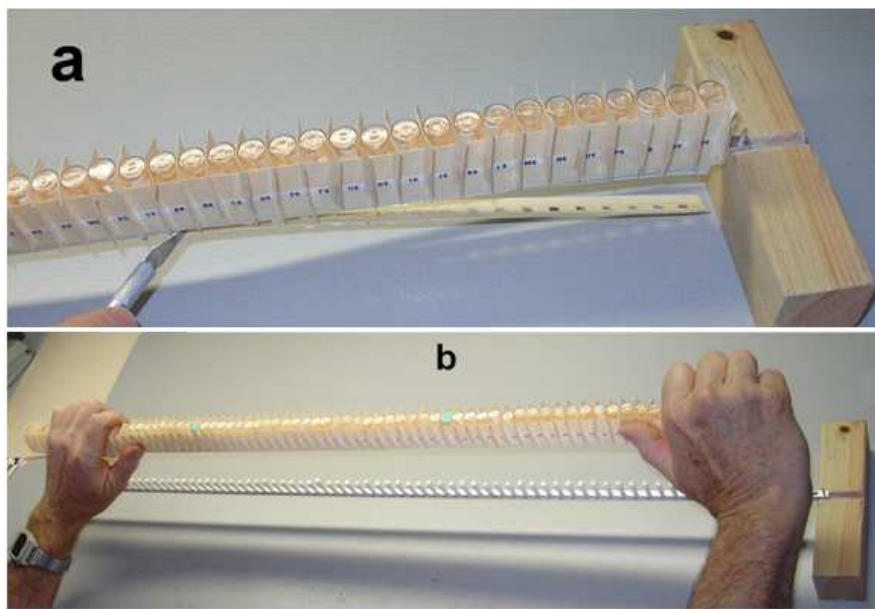


Figure 5. (a) Packaging tape over two pre-cut slits is sliced with a hobby knife to remove the bottom of the linear cell array. (b) The linear cell array and all vials are lifted above the cotton swabs mounted in the Al rod.

**Vial Contact Pressure.** The pressure on the vials provided by the cell walls must be sufficient to hold the vials within the cells if the wipe sample transport is tipped over and to remain in the cells when the cell assembly is lifted upward. At the same time, the pressure should not be so great as to make it hard for the field sampler to push the vial upward out of the cell by pushing on the bottom end of the swab stick. If too much pressure is required, the U-shaped vial insert that prevents the swab from recoiling upward into the vial would be crushed and lose its effectiveness. A suitable pressure was provided by using two of the smaller partitions, rather than one, between every fourth pair of vials as illustrated in Figure 1.

**Compact Transport for 1000 Wipe Samples.** Figure 6 shows 1000 pre-labeled cotton swabs in 14 Al rods ready for a simulated dispersive event stored in a compact frame made from 1 x 2 (1.9 cm x 3.8 cm) [3/4" x 1.5"] pine sticks. The construction method described above is based on the experience gained by manufacturing the 14 rod-swab-vial-cell assemblies.

## Conclusion

An easily transported frame was built to carry 14 sets of Al rods, each supporting 72 cotton swabs for acquiring wipe samples. The swabs are protected by 1.8 mL, wide-mouth vials, and a linear cell assembly to hold the vials in place. A portable wipe sample transport was made to carry single rod-swab-vial-cell assemblies into the field to collect wipe samples. The cell assembly was designed for removal of the vials and cells within 5 min. After addition of four swab heads previously dipped into a calibrant solution, the Al rod and 76 swab heads are placed onto N-scale model railroad flat cars for transport through the He ionizing beam of a DART ion source. Exact masses of the ions are provided by an oa-TOFMS.



*Figure 6. Carrier for 14 pre-labeled Al rods and pre-labeled linear cell array containing 1000 cotton swabs ready for field sampling*

The inexpensive, simple, and rugged integrated wipe sample transport/autosampler described should simplify wipe sample collection, reduce sample labeling errors, and greatly reduce sample preparation times when a DART or other open air ion source with sufficient space for an autosampler that uses a 1/4"-square rod as the sample support is used.

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